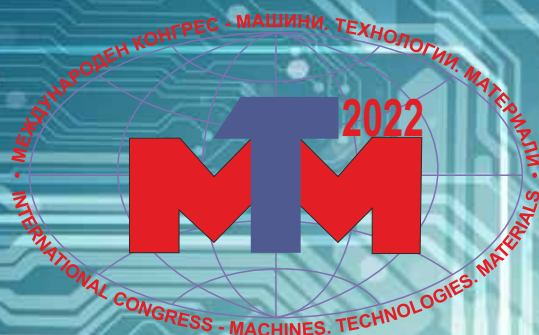


**XIX INTERNATIONAL SCIENTIFIC CONGRESS**

**WINTER SESSION**

**09 - 12.03.2022, BOROVETS, BULGARIA**



**MACHINES.**  
**TECHNOLOGIES.**  
**MATERIALS 2022**  
**PROCEEDINGS**

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**BULGARIA**

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# **PROCEEDINGS**

**YEAR V, ISSUE 2 (22), BOROVETS, BULGARIA 2022**

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# Создание экспериментальной установки для реализации комбинированного процесса

## Creation of an experimental installation for the implementation of a combined process

PhD Volokitin A.V.; d.t.s., professor Naizabekov A.B.; PhD Volokitina I.E., Lavrinyuk D.N.

Rudny industrial institute, Rudny, Kazakhstan  
e-mail: dyusha.vav@mail.ru

**Abstract:** The design of the main deforming elements, tooling and installation for the implementation of a combined process of high-pressure torsion and cryogenic cooling has been carried out. Based on the analysis of scientific and technical literature and modeling in the Deform software package, design drawings were developed. At the first stage of creating an experimental installation for the implementation of the combined process, the design of tools and equipment for this installation was carried out. In particular, several parts were designed and manufactured: the upper striker, which is informed by the translational movement from the press; the lower striker, which is informed by the torque from the translational movement of the upper striker and the matrix; the matrix and the upper and lower holders. At the second stage, for the deformation of piston rings, the design and creation of an installation for the implementation of a combined process of high-pressure torsion and cryogenic cooling was carried out.

**Keywords:** SEVERE PLASTIC DEFORMATION, HIGH-PRESSURE TORSION, STAMP DESIGN, SIMULATION.

### 1. Введение

Одной из основных задач физического материаловедения является обеспечение требуемого сочетания технологических и эксплуатационных свойств современных материалов. Механические свойства зависят от таких структурных параметров, как размеры зерен, плотность дислокаций, типы межкристаллитных границ. Наиболее простым и эффективным способом получения материалов с ультрамелкозернистой структурой является термомеханическая обработка (ТМО), основанная на сочетании больших пластических деформаций и отжига. Варьируя режимы ТМО, можно получать структуры с различными параметрами в результате развития тех или иных рекристаллизационных процессов, что позволяет за счет микроструктурного дизайна управлять свойствами материалов в широких пределах.

Методы интенсивного пластического деформирования (ИПД), в отличие от традиционных методов обработки металлов давлением, направленных преимущественно на формообразование, используются для значительного изменения структуры, фазового состава, физических и механических свойств. Наиболее исследуемым из всех методов ИПД является метод кручения под высоким давлением. Данный метод был разработан П.У. Бриджменом (Нобелевская премия по физике в 1946 г.) в 1950-х гг. и в дальнейшем развит в работах Л.Ф. Верещагина, Н.С. Ениколопьяна, В.Е. Панина и других ученых из России, США, Австрии, Японии и Китая. Этот метод позволяет проводить непрерывную деформацию материалов, даже хрупких и прочных, до очень высоких степеней, причем можно изменять температуру деформирования в широком диапазоне. В последние годы отмечается повышенный интерес к использованию возможностей интенсивной деформации при температуре жидкого азота или близкой к ней. Такая обработка не сопровождается динамическим возвратом или рекристаллизацией, что позволяет более эффективно измельчить зёрненную структуру [1-3].

В связи с этим представляет интерес применение интенсивного охлаждения в жидком азоте при деформации, что позволит получить близкое к оптимальному сочетание прочности и пластичности материала.

Геометрическая форма образца такова, что основной объем материала деформируется в условиях квазигидростатического сжатия под действием приложенного давления и давления со стороны внешних слоев образца. В результате деформируемый образец, несмотря на большую степень деформации, не разрушается. Методом кручения под высоким давлением также возможна обработка заготовки в форме кольца, по предложенной С. Эрбелем схеме [4].

Данный метод имеет развитие и улучшения направленные на повышение различными путями однородности получаемой структуры в центре и на периферии образца, технологичность и расширение типов производимых материалов.

### 2. Разработка чертежей новой конструкции

Как известно, в процессе кручения под высоким давлением движение деформирующего инструмента состоит из двух типов: поступательного и вращательного. В качестве рабочего механизма для реализации данного способа идеально подходят прессы, позволяющие создать высокое гидростатическое давление при сжатии. Однако главной трудностью является необходимость осуществления операции кручения вдоль оси заготовки. Для этого необходимо сообщить определенный крутящий момент деформирующему инструменту, что зачастую является невозможным из-за особенностей конструкции большинства прессового оборудования. Поэтому единственным возможным вариантом в этом случае остается обеспечение кручения деформирующего инструмента при неизменном прямолинейном движении пуансона прессы. Решение этой технической задачи может реализовано на практике только при наличии составного деформирующего инструмента, включающего в себя как блоки перемещения, так и вращения.

Для реализации процесса кручения под высоким давлением на существующем оборудовании лаборатории кафедры МиГД была разработана специальная конструкция, позволяющая реализовать процесс скручивания под высоким давлением, за счет прямолинейного движения бойка относительно станины. Прямолинейное движение верхнего бойка, с закрепленным на нем нижним бойком, за счет сил контактного трения направленных под наклонным углом к ответной части матрицы, передает ей крутящий момент, в результате чего прямолинейное движение превращается в движение кручение.

На основе анализа научно-технической литературы и моделирования в программном комплексе Deform [5-6] были разработаны чертежи конструкции. Конструкция состоит из нескольких частей: верхнего бойка (рис. 1), которому сообщается поступательное движение от прессы; нижнего бойка (рис. 2), которому сообщается крутящий момент от поступательного движения верхнего бойка и матрицы (рис. 3) в которой находится заготовка в виде кольца.

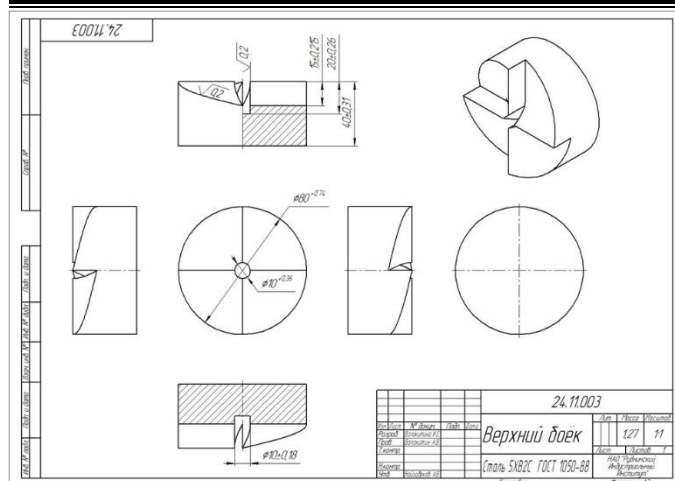


Рис. 1 Чертеж верхнего бойка

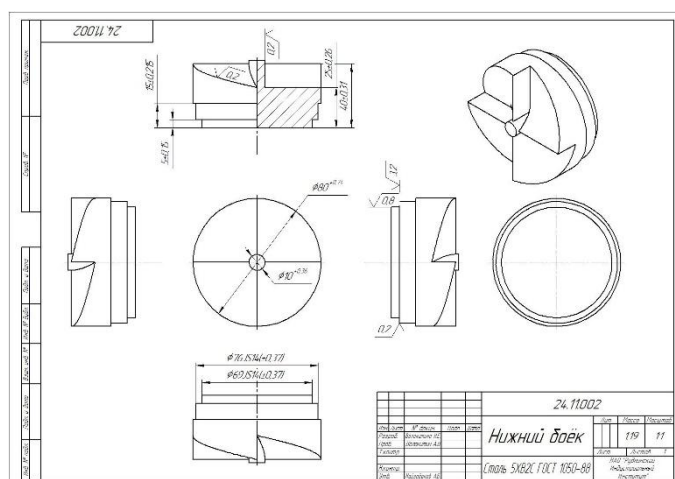


Рис. 2 Чертеж нижнего бойка

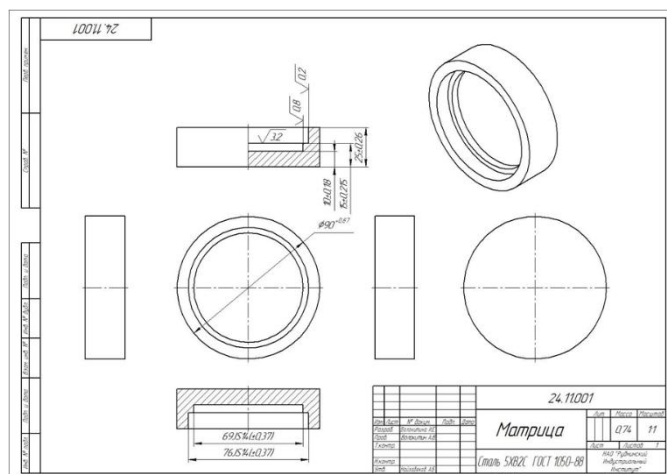


Рис. 3 Чертеж матрицы

На нижней грани верхнего бойка созданы 4 периодических выточки спиральной формы. При этом в центре верхнего бойка предусмотрено цилиндрическое отверстие для штока деформирующего элемента и обеспечения соосности обоих бойков.

Нижний боек имеет несколько ступеней. Данное конструктивное решение является необходимым, поскольку в данном случае речь идет о деформировании кольцевой заготовки, а не дисковой. Первый переход (вторая промежуточная ступень) обеспечивает своей боковой гранью соприкосновение с боковой гранью стакана, в который закладывается заготовка. Второй переход (третья нижняя ступень) обеспечивает контакт с заготовкой по ее внутреннему

радиусу, попутно полностью замыкая ее поперечное сечение. В соответствии с этим принципом, внутренняя форма матрицы также должна иметь ступенчатую форму. Ширина ступени должна соответствовать ширине обрабатываемой кольцевой заготовки.

Для закрепления конструкции на гидравлическом прессе были разработаны удерживающие элементы: верхний держатель (рис. 4) и нижний держатель (рис. 5).

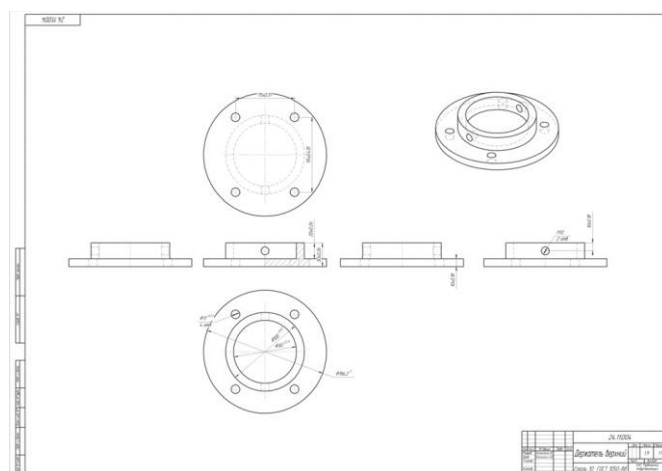


Рис. 4 Чертеж верхнего держателя

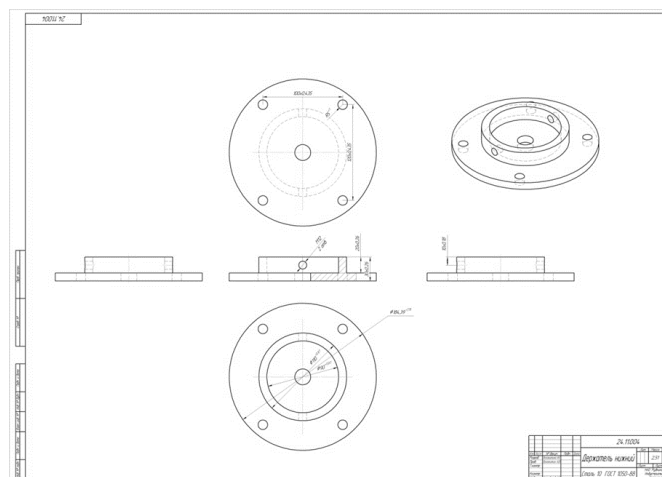


Рис. 5 Чертеж нижнего держателя

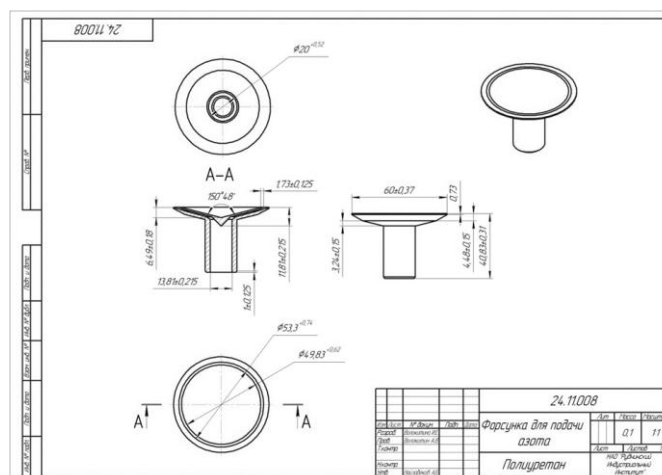


Рис. 6 Форсунки сплинклерного типа

Для реализации процесса кручения под высоким давлением с криогенным охлаждением в нижнем бойке выполнено технологическое отверстие, в которое установлена форсунка для подачи жидкого азота в камеру деформации заготовки. В ходе проведения моделирования было принято решение в использовании форсунки сплинклерного типа, позволяющие

реализовать равномерную подачу азота на всю поверхность кольца. При использовании форсунок обычного типа распыление азота осуществлялось не равномерно по поверхности кольца. Данная форсунка будет изготовлена на 3d принтере из полиуретана (рис. 6).

### 3. Создание экспериментальной установки для реализации комбинированного процесса

Экспериментальная оснастка для КВД труднодеформируемых материалов должна отвечать требованиям, предъявляемым к горячему прессовому инструменту процессов ОМД. Для формообразующих элементов оснастки необходимо использовать комплексно-легированные классы штамповых сталей. Например, хорошо противостоят тепловым ударам, износу и воздействию высоких напряжений широко используемые комплексно-легированные хромистые стали повышенной теплостойкости и вязкости марки 5XB2C. Конструкцию изготавливали в условиях ТОО «Цифровой мир» из стали марки 5XB2C из ковальной отожжённой заготовки на токарно-фрезерном станке с ЧПУ. Составные части конструкции после изготовления были подвержены специальной термической обработке для повышения прочностных свойств.

После изготовления всех элементов конструкции, производили ее сборку. Так нижней держатель был установлен на основании плиты гидравлического пресса и закреплен стяжными болтами через удерживающие уголки (рис. 7). В нижний держатель установлен нижний боек, с камерой для деформации колец, верхняя матрица устанавливается сверху на образец.



Рис. 7 Нижний держатель с установленной матрицей

Верхний боек был неподвижно закреплен в верхнем держателе с помощью распорных болтов. Верхний держатель закреплен болтами к верхней плите гидравлического пресса (рис. 8). В исходном состоянии матрицы разведены. На первом этапе проводим центрирование осей бойков для их совпадений. Проверяем центрирование сближением бойков. Только после холостого прохода осуществляем полный цикл деформационного упрочнения.



Рис. 8 Верхний держатель с установленным в него верхним бойком

### 4. Выводы

Проведено проектирование основных деформирующих элементов, оснастки и установки для криогенного охлаждения. На основе анализа научно-технической литературы и моделирования в программном комплексе Deform были разработаны чертежи конструкции. Конструкция состоит из нескольких частей: верхнего бойка, которому сообщается поступательное движение от пресса; нижнего бойка, которому сообщается крутящий момент от поступательного движения верхнего бойка и матрицы. На нижней грани верхнего бойка созданы 4 периодических выточки спиральной формы. При этом в центре верхнего бойка предусмотрено цилиндрическое отверстие для штока деформирующего элемента и обеспечения соосности обоих бойков. Нижний боек имеет несколько ступеней. Данное конструктивное решение является необходимым, поскольку в данном случае речь идет о деформировании кольцевой заготовки, а не дисковой. На втором этапе работы для деформирования поршневых колец было осуществлено проектирование и создание установки для реализации совмещенного процесса кручения под высоким давлением и криогенного охлаждения.

### 5. Финансирование

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# Structural Network for the Implementation of “Industry 4.0” in Production Processes

Isak Karabegović<sup>1</sup>, Raul Turmanidze<sup>2</sup>, Predrag Dašić<sup>3,4</sup>

<sup>1</sup>Academy of Sciences and Arts of Bosnia and Herzegovina (ANUBiH), Bistrik 7, 71000 Sarajevo, Bosnia and Herzegovina

<sup>2</sup>Georgian Technical University (GTU), Kostava 77, 0175 Tbilisi, Georgia

<sup>3</sup>Academy of Professional Studies Šumadija (ASSŠ) – Department Trstenik, 37240 Trstenik, Serbia

<sup>4</sup>SaTCIP Publisher Ltd., 36210 Vrnjačka Banja, Serbia

**Abstract:** In the last ten years, the development and implementation of robotics, sensor and digital technology in the world from introduced us the third industrial revolution to the fourth industrial revolution. In its development strategy, Germany introduces digital technologies in production processes called “Industry 4.0” in 2011. The German example is followed by the following countries: USA, Great Britain, Sweden, Japan and other. Almost all technologically developed countries strive to introduce advanced technologies into production processes in order to maintain their competitive positions in the market. The implementation of the fourth technological revolution depends on a number of new and innovative technological achievements, i.e., the implementation of patents for the basic technologies of Industry 4.0. It is necessary to integrate production processes in all phases of product development, as well as monitoring during its working life through the use of advanced technologies. When automating production processes, we must include intelligent sensors, intelligent robots and connect everything to the Internet using digital technologies that closely monitor the product development process. It is also required to collect large amounts of data through network communication used during the production process. The paper presents a structural network with all levels for the implementation of Industry 4.0 in the production process of the metal industry.

**Key words:** Industry 4.0, robot, smart sensor, digital technology, production process, smart automation.

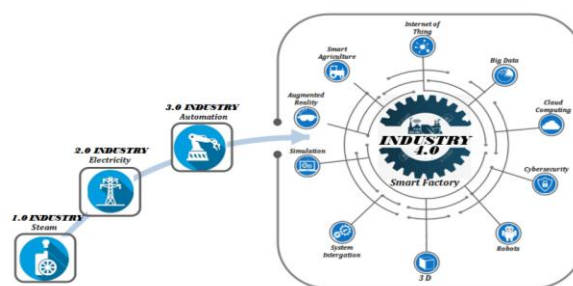
## 1. Introduction

In the last 20 years, there has been a development of digital technologies that are implemented in the production processes themselves. We are at the beginning of the fourth industrial revolution because many industrial development strategies of countries in the world that are considered technologically advanced advocate the transformation of industrial production through the implementation of all advanced technologies, especially digital technologies. Germany first announced part of its High-Tech Strategy 2020 program in Hanover in 2011 under the title “Industry 4.0”, while in the USA, in 2012 the Advanced Manufacturing Partnership Steering Committee provided recommendations for positioning the USA for long-term leadership in advanced technology. Similarly, in 2013, the UK Government Office for Science highlighted the strategy under the title “The Future of Manufacturing - A New Era of Opportunity and Challenge for the UK”, while China under the title “Made in China 2025” wants to become the most technologically advanced country in the world; The Government of Japan presented its strategy under the name “Robot Revolution” [1, 12-14, 20-21], and the Swedish Government's strategy for new industrialization is to strengthen the capacity of companies for change and competitiveness through automation and digitalization. Industry 4.0 is creating radically new approaches that will involve individuals and institutions in a revolutionary way. The Industry 4.0 platform aims to match supply and demand in a very affordable way by providing the customer with a wide range of products without both parties interacting. The inclusion of digital technologies with other advanced technologies will transform the production processes in industrial production due to the fact that the price of this technology is constantly declining in the market and are increasingly widespread in production processes, and the second reason is that ICT technologies are implemented with other technologies and enable digital transformation of production processes [1-2, 7, 9, 16-19, 24-32]. When it comes to the transformation of production processes, it is impossible to imagine without the inclusion of robotics,

digital and sensor technology. The second generation of industrial robots has been developed, and new generations of robots must be intelligent and autonomous, thus increasing the reliability of the production process, as well as reducing the time to produce the finished product, as well as precision and adaptability beyond human capabilities.

## 2. The implementation of patents in technologies that are the basis for Industry 4.0 expands the application of Industry 4.0

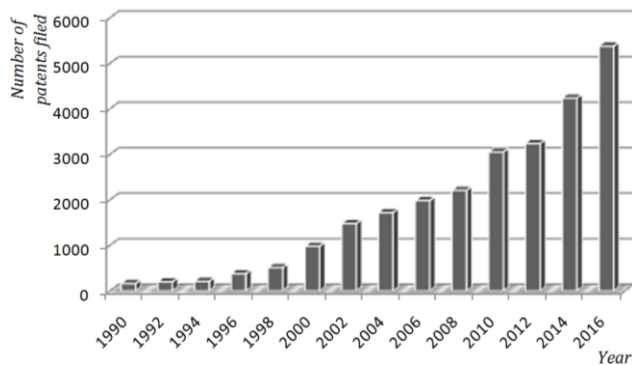
To date, we have had three industrial revolutions in the world: the invention of the steam engine was marked as the first industrial revolution, and the invention of electricity as the second industrial revolution. The rapid development of informatics and automation electronics was marked as the third industrial revolution, while the world today is on the brink of the fourth technological revolution “Industry 4.0” as shown in Fig. 1.



**Fig. 1.** Industrial revolutions in the world and their evaluation

Industry 4.0 is a digital transformation of production/related industries and the process of creating new value [3-5, 22-23, 28, 30-32]. Digital transformation must be integral, comprehensive, involving all actors in global politics, from the public and private sectors to civil society and academic community [6, 29]. Within the fourth industrial revolution, a new value chain is being formed that relies primarily on Cyber-Physical Systems

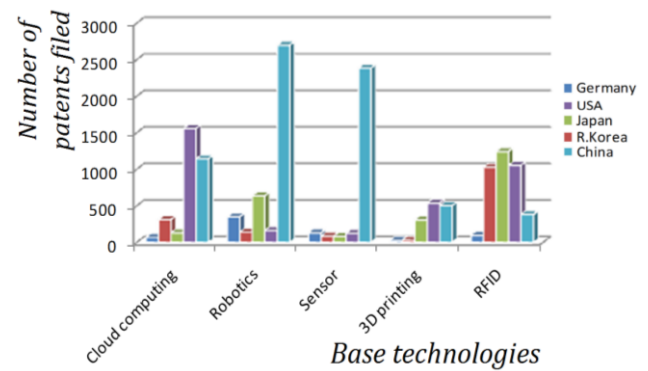
(CPS) which is also another name for the Internet of Things (Internet of Things), and its associated service most often implemented in the cloud (Cloud Computing). The implementation of Industry 4.0 is taking place through the massive use of the Internet of Things (IoT). Some studies estimate that by 2025, there will be 26.30 billion devices in homes equipped with sensors, processors and embedded software and connected to the Internet of Things (IoT). Numerous patents are responsible for this trend of implementation of basic technologies from Industry 4.0, as shown in Fig. 2 [25], which shows the trend of applying for patents of the fourth industrial revolution - Industry 4.0, which were applied to the EPO's free database "European Patent Office (EPO)" in the European Union until 2016.



**Fig. 2.** Number of filed patents of the fourth industrial revolution - Industry 4.0 in the European Union [25]

By analysing Fig. 2, we come to the conclusion that we have a continuous growth of patents from the basic technologies of the fourth industrial revolution in the European Union. From 1990 to 2016, it can be roughly concluded that the trend of patent filing and applications follows an exponential function, so that in 2016 the number of patents reached 5.380. We see that the latest available data on filed patents signal the implementation of Industry 4.0. In order to create an image of which countries in the world and which are the base technologies of Industry 4.0 where the largest number of patents filed, we need to do analyse Fig. 3 [8]. By analysing Fig. 3 we come to the conclusion that in the first place in the world is robotic technology in terms of reported and implemented innovations, where China is in first place, Japan in second place. In second place is RFID technology, then in third place is cloud computing, in fourth place are smart sensors and the last fifth technology 3D printing by reported and implemented innovations.

The trend of applying Industry 4.0 innovations allows only limited conclusions about patents and their implementation, as there are a huge number of low-quality patents from Industry 4.0 in China. The second reason why this trend of innovation in China should be taken with a grain of salt is the dysfunctional government policy that rewards the registration of innovations regardless of their quality and the third reason is that many reporting innovations from China's industry seem lower quality their acceptance rate is significantly lower.

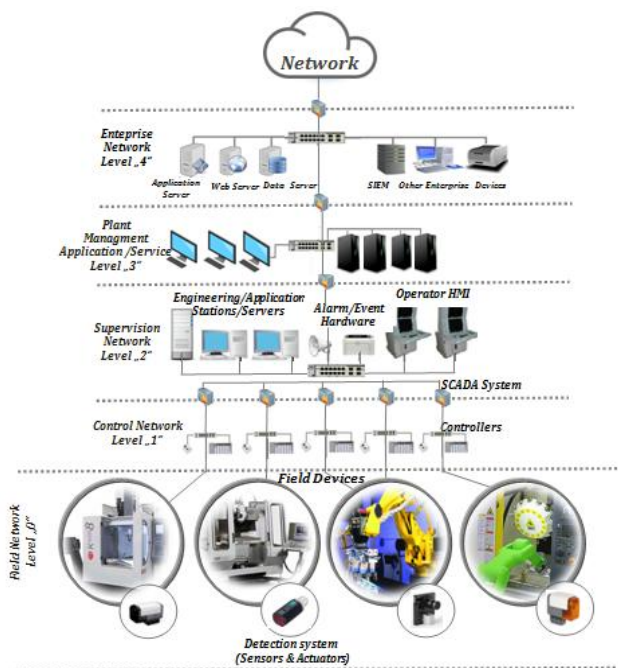


**Fig. 3.** Trend of patent applications from the five basic technologies of Industry 4.0 in technologically developed countries [8]

### 3. Structural Network for the Implementation of Industry 4.0 in Industrial Production Processes of the Metal Industry

For the implementation of "Industry 4.0", regardless of the principles on which the scenario is based, it is necessary that the application of innovation, software and hardware structure comes first. After that, in second place is the education of users - consumers, i.e., training programs for the general population. In order to implement Industry 4.0 in third place it is necessary to integrate with new technologies that we have listed as shown in Figure 4. It is necessary to enable CPS to monitor industrial production processes, connect data obtained through implemented sensors in production processes with virtual and simulation production models, in other words, create a virtual copy of the physical production process. The development and implementation of powerful computers enables the CPS to make its own decisions within the production process of the "smart factory", in other words, decentralization is performed, there is no need for central planning and supervision. In order to implement all of the above, it is inconvenient to establish an Industry 4.0 value chain, which means between new technologies such as IoT, ICT, CPS, .... etc., with consumers, devices and tools. Many research institutions around the world are working on the development and implementation of various "Industry 4.0" implementation scenarios. In Germany prof. M. Herman and his colleagues identify project principles such as: interoperability, decentralization, virtualization, real-time capability, service orientation and modularity, on which the Industry 4.0 implementation scenarios are based. Until recently, we had a traditional linear supply chain, but today many product supply chains are being transformed from a static array into a dynamic array that is evolving to an optimal state. Development and Implementation of Industry 4.0 in the production processes of the industry - Smart factory is characterized by the following most important characteristics: *connectivity, optimization, transparency, proactivity and agile flexibility*. The connectivity of the production process is one of the most important characteristics. By implementing a large number of smart sensors that provide continuous data sets, ensure that data is constantly updated and provide current operating conditions, and the integration of data from operating and business systems, as well as suppliers and customers allows us to view the input and output supply chain processes and in this way we are

able to increase the overall quality of the supply network itself with our products. Another feature is the optimized production process that allows us high reliability, optimization of energy input from the input, production and output chain, minimal manual intervention, automation during the production process, reducing costs and waste, etc. The data captured by smart sensors is transparent, allowing greater visibility throughout the production chain, allowing the organization to make the right decision or make autonomous decisions through analysis provided by tools such as real-time displays, alerts and notifications. The production process becomes a proactive process where employees and systems are able to anticipate and act on time before a problem arises. This feature includes the identification of anomalies, by having the ability to predictively solve the problem of quality in the production process of the product, as well as monitoring safety and maintenance. The agile flexibility itself enables the production process to adapt to the schedule and changes of the product with minimal intervention. Industry 4.0 implementation allows independent configuration of equipment and material flows depending on the product being produced and scheduled changes that we can monitor in real time, and the structural network of Industry 4.0 implementation in the metal industry production process is shown in Fig. 4. [10, 12, 14, 18].



**Fig. 4.** Structural network scheme with all levels for the implementation of Industry 4.0 in the production process of the metal industry [10, 12, 14, 18]

Fig. 4 shows us that the structural implementation network of Industry 4.0 in any production, including the textile industry, consists of five levels. Zero level is a network of production processes of the textile industry where sensors are installed to monitor production processes and to detect systems. After the zero level comes level 1, i.e., the control network, where we can use controllers to act to eliminate errors that occur in the production process. The third level is the supervision network that monitors everything, and gives a signal/alarm

if something is wrong. In the third new is the operations management network, and in the last level 4 is the company network that is connected to all possible information via the cloud as shown in Figure 4. Industry 4.0 not only serves large companies; currently small companies can compete with large companies on a way they had never been able to before.

#### 4. Conclusion

We can conclude that new technologies include: IoT (Internet of Things), robotics, cloud computing, smart sensors, radio frequency identification, cyber-physical systems and big data, are key in the application of the Industry 4.0 concept, in all industries, and the same so in the metal industry as shown by the structural network with all levels for the implementation of Industry 4.0, because it implies complete digitalization of all production processes, also when creating an idea about a product, product engineering, production organization, process control and industrial services. There is a continuous increase in the application of Industry 4.0 in the world, and this is confirmed by the trend of applying for patents from Industry 4.0 from basic technologies: Cloud Computing, Robotics, Sensor, 3D printing, RFID, both in the European Union and in the world. With the application of Industry 4.0 in production processes, the company has advantages such as: increasing efficiency, increasing the speed of production, increasing product quality, increasing flexibility, increasing operational safety. In addition, with the implementation of Industry 4.0, the company expands its access to the global economic market because it has a wide user base. There are three challenges when it comes to the Industry 4.0 application in the company. The first challenge relates to methods and methodology, especially how to integrate machines and devices within the production process. The second challenge is education, i.e., how to prepare new generations of engineers who need to work and implement Industry 4.0. The third challenge is about society, primarily how to explain to people that Industry 4.0 creates new opportunities and brings a better quality of life for all of us, without having to be afraid of change and everything that Industry 4.0 brings. Great attention in the world is paid to Industry 4.0 and the role of man in integrated factories, which are getting closer to realization day by day.

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## Изменение свойств сталемедной проволоки в процессе «РКУП-волочение»

Changing the properties of steel-copper wire in the process of "ecap-drawing"

PhD Volokitina I.E.; d.t.s., professor Naizabekov A.B., PhD Volokitin A.V., Panin E.A.

Rudny industrial institute, Rudny, Kazakhstan  
e-mail: irinka.vav@mail.ru

**Abstract:** The purpose of this study is to study changes in the microstructure and properties of steel-coated wire in the process of "ECAP-drawing". An important result of the study, demonstrating the high manufacturability and practical value of the process, is that when combining equal-channel angular pressing with traditional drawing, the structure of bimetallic wire obtained by equal-channel angular pressing is preserved. Thus, as a result of three passes of deformation by the ECA -drawing method, a fragmented structure with equiaxed grains of the order of 9 microns is formed in the steel core, and the copper shell is crushed to 13 microns. The combination of ECAP with drawing allows not only to strengthen the composition, but also to simultaneously increase the important plastic properties of products regulated by a number of documents. This circumstance favorably distinguishes the method under study from other (traditional) types of deformation treatment, accompanied by modification of the structure and properties of the material.

**Keywords:** BIMETALLIC WIRE, MICROSTRUCTURE, DRAWING, STEEL, COPPER.

### 1. Введение

В настоящее время многие технические и промышленные проблемы не могут быть решены с использованием только отдельных материалов на основе одного металла или сплава. Это связано с тем, что в данных условиях эксплуатации материал должен обладать высокой скоростью теплопередачи при минимальных тепловых потерях, а также противостоять любой форме коррозии в рабочей среде. Сочетание различных механических и физико-химических свойств подразумевает не только многофункциональность готового продукта, но и сложность, а в некоторых случаях и невозможность сочетания металлов и сплавов. Один из металлов такой пары расположен в центре в виде сердечника, а второй - на периферии в виде концентрической оболочки, плотно прилегающей к сердечнику. Такая проволока производится в основном прокаткой или прессованием с последующим волочением биметаллической заготовки. Поэтому прочность соединения между сердечником и оболочкой особенно важна для биметаллической проволоки. Если сцепления недостаточно, то процессы прокатки и волочения будут затруднены, а качество проволоки будет низким. Поэтому в производстве предпочтение отдается тем методам, которые обеспечивают максимально возможную прочность сцепления для этой пары металлов.

Среди различных биметаллических композитов биметаллическая медноалюминиевая проволока достаточно хорошо известна своим уникальным применением в распределительных шинах электрической энергии, автомобильной проводке, бронированных кабелях и катушках телевизионного ярма, а также в обмотках автомобильных генераторов переменного тока [1-3]. В этих композитах медная оболочка повышает электропроводность, а алюминиевый сердечник снижает стоимость и вес биметалла [3].

Одним из наиболее перспективных методов повышения прочностных и пластических свойств металлов является формирование ультрадисперсных структур в материалах методами деформации [4-6]. Однако, несмотря на многочисленные разработки, современные методы имеют ряд существенных ограничений с точки зрения непрерывности и производительности технологических схем [7-8]. Одним из наиболее эффективных методов получения ультрамелкозернистой структуры являются методы интенсивной пластической деформации [9-10].

Равноканальное угловое прессование (РКУП), которое, вероятно, является наиболее широко используемой технологией интенсивной пластической деформации, рассматривается как перспективный метод достижения желаемых высоких степеней деформации [11-13]. В последнее время на основе этого метода появилось большое

количество его модификаций [14-16], некоторые из которых позволяют получать сортовые изделия [16-18].

Однако, несмотря на многочисленные разработки, проблема создания новой схемы деформации, максимально приближенной к промышленным условиям и представляющей научный и практический интерес с точки зрения массового производства, остается нерешенной. Одной из таких схем может быть новая непрерывная схема равноканального углового прессования и волочения (РКУП-волочение) [19-21]. Его ключевая особенность заключается в том, что, в отличие от других комбинированных методов, здесь отсутствует стадия прокатки. Непрерывность деформации обеспечивается процессом волочения, который происходит сразу после процесса РКУП. Благодаря такой уникальной схеме деформации в поперечном сечении заготовки развивается достаточно высокий уровень растягивающих напряжений, и неправильный подбор технологических параметров приведет к обрыву деформируемой проволоки.

Для пластической деформации биметаллических разнородных металлов необходимо выполнить, по крайней мере, два условия [21]:

- содержание мягкого компонента должно быть минимальным, так как чем выше энергия деформации мягкого металла (т.е. чем больше его объем), тем больше вероятность разрывов и разрывов твердого менее пластичного металла;
- условия деформации мягкого компонента должны быть как можно более сложными, что может быть достигнуто путем создания неблагоприятной схемы напряжений путем выбора величины деформации и формы рабочего инструмента.

При неблагоприятных условиях в процессе волочения оболочка может быть содрана с поверхностных слоев, могут возникнуть разрывы и другие дефекты. В настоящее время было проведено множество исследований взаимосвязи "структура-свойства" для однородной медной или стальной проволоки методом РКУП-волочения, который доказал свою эффективность [19-21].

Известно ограниченное количество работ, посвященных изменению микроструктуры биметаллической проволоки после деформации, поскольку сталь и медь обладают разными механическими свойствами, эти металлы на границе раздела могут деформироваться по-разному, чтобы оставаться "связанными" друг с другом во время волочения. Поэтому целью данного исследования является изучение изменения свойств сталемедной проволоки в процессе РКУП-волочения, как методом моделирования, так и экспериментальным путем.

## 2. Материал и методика исследования

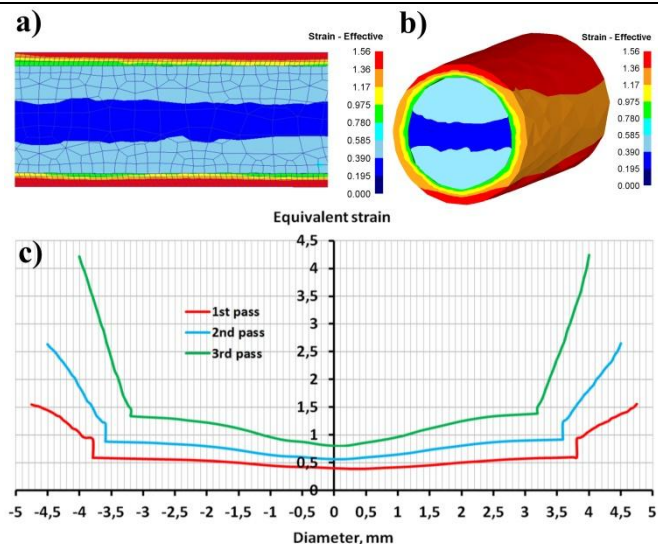
Для компьютерного моделирования комбинированного процесса было выбрано программное обеспечение Deform. Исходная заготовка представляла собой биметаллическую проволоку с соединением "сталь-медь" диаметром 10 мм, диаметр стального сердечника составлял 8 мм. В качестве основного материала была выбрана сталь AISI-1566. Материалом корпуса был медный сплав CDA 110 (содержание меди 99,9%). Механические свойства обоих материалов были взяты из базы данных материалов Deform. При каждом проходе диаметр РКУ-матрицы соответствовал диаметру проволоки, а диаметр волокна обеспечивал сжатие 0,5 мм за каждый проход. Поскольку оба материала неподвижны относительно друг друга в биметаллической проволоке, между ними был установлен жесткий неразрывный контакт. На контакте медной оболочки и обоих инструментов был установлен коэффициент трения 0,1. Деформация производилась при температуре окружающей среды.

Лабораторный эксперимент проводился на промышленном волочильном стане В-1/550 М по методике, описанной в работе [10]. В качестве исходной заготовки использовали биметаллическую проволоку состоящую из стального сердечника - сталь 1566 и внешнего слоя из меди М1, диаметром 10 мм, диаметр стального сердечника составлял 8 мм. Деформирование осуществляли в три прохода. Для волочения применяли твердосплавные волокна с полированными каналами, уменьшенными углами конусности рабочей зоны и плавными переходами от одной зоны к другой. Подготовку поверхности биметаллической проволоки к волочению проводили по обычной для проволоки из стали технологии, в качестве смазки использовали смесь порошков мыла и серы.

Подготовка образцов для металлографического анализа проводилась на установке электролитической пробоподготовки Struers. Все образцы были исследованы в средней плоскости образца, чтобы избежать влияния периферийных областей. Полученные образцы рассматривались в двух сечениях: поперечном и продольном. Металлографический анализ всех исследованных образцов проводили с помощью электронного просвечивающего микроскопа JEM2100.

## 3. Результаты эксперимента

После трех циклов деформирования комбинированием РКУП и волочения увеличение деформации в оболочке достигает  $\epsilon=1,56$ , центральная зона сердцевины обрабатывается до  $\epsilon=0,4$ , поверхностные слои - до  $\epsilon=0,6$  (Рис. 1а). Однако при рассмотрении поперечного сечения заготовки было выявлено неравномерное распределение деформации в вертикальном и горизонтальном направлениях (рис. 1б). Этот эффект является результатом влияния соединений каналов в РКУ-матрице – заготовка получает основную деформацию сдвига в зонах поверхности вертикального направления при прохождении каналов матрицы.



**Рис. 1** Эквивалентная деформация в сталемедной проволоке после процесса "РКУП-волочения": а – продольный разрез; б – поперечный разрез; в – распределение эквивалентной деформации в вертикальном направлении по проходам

Рассматривая распределение эквивалентной деформации при многопроходной деформации (рис. 1с), можно отметить, что общий уровень деформации увеличивается с увеличением количества циклов обработки. Также существует характерная разница в накоплении деформации слоями: деформация в слое более мягкой оболочки имеет более интенсивный уровень роста, уровень деформации в стальной сердцевине намного ниже, с распределением градиента (максимум на поверхности, низкий в центре). По мере увеличения общего уровня деформации разница эквивалентных значений деформации в зоне контакта оболочки и сердечника (вертикальные зоны графиков) значительно уменьшается. Так, если после 1 прохода разница достигает 50% (0,6/0,9), то после 3 проходов эта разница составляет всего 8% (1,38/1,49).

Микроструктурные исследования проволоки показали, что в исходном состоянии медная оболочка имеет крупнозернистую структуру с большим количеством двойников и средним размером зерна 50 мкм (рис. 1а). Исходная микроструктура стального сердечника соответствует характерной структуре высокоуглеродистой доэвтектоидной стали марки 1566 и представляет собой смесь феррита с большим количеством перлита, средний размер зерна составляет 18 мкм. При деформировании методом РКУП-волочения после 1 прохода можно наблюдать сильно удлиненные зерна меди вблизи границы раздела сталь-медь, зерна центральной области стали менее деформированы, что указывает на меньшее напряжение в центральной зоне. После 3 проходов наблюдается интенсивное диспергирование структуры медной оболочки, размер зерен меди измельчается до 13 мкм (рис. 1б, верхняя часть). Процесс фрагментации носит кумулятивный характер, т.е. степень дисперсности зерна и толщина измельченного слоя от прохода к проходу непрерывно возрастает. Структура стального сердечника после 3 проходов состоит из изогнутых и волнообразных зерен феррита, границы которых неравновесны и имеют сложный дислокационный характер, кроме того, наблюдается дробление перлитных участков. Так после 3-х проходов деформирования в стали формируется фрагментированная структура с равноосными зернами размером порядка 9 мкм (рис. 1б нижняя часть).

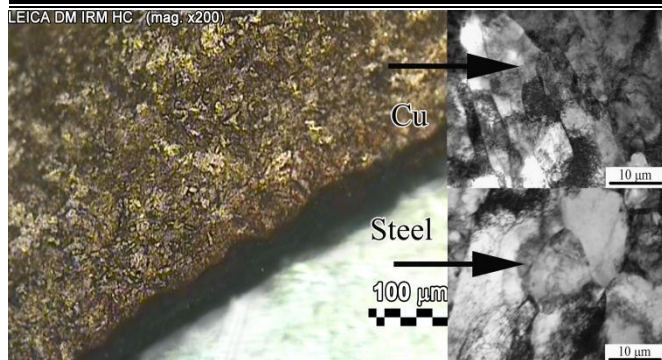


Рис. 2 Микроструктура стале-медной проволоки: а – исходное состояние, б – после трех проходов методом

Предел прочности ( $\sigma_B$ ) увеличивается от 580 МПа в исходном состоянии до 812 МПа после третьего прохода. Предел текучести ( $\sigma_{0.2}$ ) увеличивается от 380 МПа в исходном состоянии до 562 МПа после третьего прохода. И как видно по кривым, основной прирост прочности приходится на первые два прохода деформации. Так на первом проходе предел прочности увеличивается на 22%, а предел текучести на 23%. Пластические характеристики с увеличением количества проходов наоборот снижаются, так относительное удлинение с 34% в исходном состоянии до 22% после 3 проходов.

Анализ изменения величины микротвердости от центра проволоки к периферии показал, что этот параметр находится в прямой зависимости от уровня накопленной деформации. Так полученные значения твердости меди превышают твердость отожженного меди на 315 МПа, вследствие наклепа при деформировании методом РКУП-волочение и составляют 663 МПа. Микротвердость стального сердечника в исходном состоянии составляет 600 МПа, а после трех проходов деформации – 985 МПа.

## 5. Финансирование

Данное исследование было профинансировано Комитетом науки Министерства образования и науки Республики Казахстан (Грант № AP08052852).

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# Design and 3D printing of holders for XRF analysis requiring small volumes of sample

Liliya Tsvetanova<sup>1\*</sup>, Nikola Kuvandjiev<sup>1</sup>

<sup>1</sup>Institute of mineralogy and crystallography "Acad. Ivan Kostov", Acad. Georgi Bonchev Str.  
Bulgarian Academy of Science, Sofia, Bulgaria  
lilicvetanova79@abv.bg

**Abstract:** The preparation of XRF tablets with small amounts of samples produced from various synthesis is a challenging operation. Zeolites and other types of samples with minimal volumes are frequently provided for analysis by X-ray fluorescence spectroscopy. Here we present three types of holders made of PLA (polylactic acid) using commercially available 3D printing technology. The employed material is a bioactive thermoplastic aliphatic polyester extracted from renewable resources and is biodegradable. The holders have reduced diameters of the measured area and can accommodate powders, sheets or solid state samples. The holders are basically three-dimensional plastic models that can be designed and printed based on the provided sample. They are low cost and can be easily applied in practice. The design of the first type of holders is targeting powder samples, allowing the reduction of the required amount from 5-10 g per sample to 1 g. The second type of holders aimed the XRF analyses of textile like materials while the third type of design aimed metal alloys. Examples of the use of the holders are the analyses of coins (here 2 leva is shown) and textile samples containing different metals (e.g. zinc).

**Keywords:** 3D PRINTING, XRF HOLDER, SAMPLE PREPARATION

## 1. Introduction

The 3D printer we used is commercially available and is from the Creality Ender-3 V2 FDM 3D Printer series (Fig. 1).

The software used allows the selection of the appropriate thickness of the production layers in terms of accuracy / resolution and speed of production. Each axis can be controlled individually and the distance of each step/movement can be set (0.1mm / 1mm / 10mm).

The integrated all-metal body of the printer is equipped with Carborundum Glass Platform. It allows the printing bed to heat up faster and the prints stick without distortion. The Creality Ender-3 V2 printer is equipped with proprietary hardware that includes a 32-bit motherboard.

Fused Deposition Modeling (FDM) is a form of 3D printing technology in which the material is applied by laying down consecutive layers. Supported printing materials are threads of PLA [1-7], TPU [8-10], carbon fiber [11-14] and other linear threads [15].



Fig. 1 3D printer Creality Ender-3 V2.

## 2. Materials and methods

Software used for preparing the 3D models for printing is Ultimaker Cura. Another open-source 3D printing software – Blender, is used to design virtual models of the real solid objects (holders), that are then transformed into readable format for the 3D printer using Ultimaker Cura software. This process of breaking up a 3D object into a readable format is called slicing. When the process of cutting a 3D object is complete, it generates a readable language for the 3D printer. This language is called G-code. Once the cutting is complete, the exact G-code commands that will be needed to complete the printout are produced. The 3D printer interprets the instructions and then combines (hardens) the material to create the 3D object. The 3D printer creates objects by printing

them, layer by layer, until they are finished. 3D printing software has many platforms that are completely free to use. Some of them are open source cross-platform tools.

The material used to make the three types of holders is thermoplastic aliphatic polyester PLA (polylactic acid), commercially available. The optimization of the production of different types of holders is necessary due to the different types of samples for analysis. Fig. 2 shows a holder suitable for analysis of tablets of powder samples, Fig. 3 - holder for textile like materials and Fig. 4 - holder for metal alloys.

The tested materials are powder sample of titanosilicate ETS-4, textile sample with zinc content and metal alloy – e.g. 2 leva coin. They are characterized by X-ray fluorescence spectroscopy (WDXRF) and these results are supported by powder X-ray diffraction (PXRD).

X-ray fluorescence analysis was used to determine the chemical composition of the different type of samples. The chemical elemental composition was determined by a Supermini200 spectrometer - Rigaku, Japan. Data collection was performed by wave-dispersed X-ray fluorescence at 50 kV and 4.00 mA. The data processing is performed using the ZSX software package. The sample is placed in a holder with an irradiated area of 20 and 20 mm in diameter.



Fig. 2. Holder for tablets

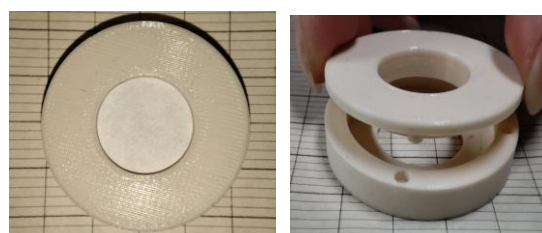


Fig. 3. Holder for textile



Fig. 4. Holders for metal alloys

Powder X-ray diffraction (XRD) was used for phase identification, that was also used for the 3D printed holders. The samples were measured at room temperature on an Empyrean diffractometer MalvernPanalytical, the Netherlands. Data collection was performed using Cu radiation ( $\lambda = 1.5406 \text{ \AA}$ ) at 40 kV and 30 mA and a PIXcel3D detector in the range  $2 - 90^\circ 2\theta$ , step  $0.013^\circ 40 \text{ sec/step}$ .

### 3. Results and discussion

The results of chemical analysis performed on the basis of X-ray fluorescence spectroscopy (WDXRF) using a 3D printed powder sample holder are presented in Table 1. From Table 1 it can be seen that the difference between the analysis using a 3D holder and with conventional holder is in the range of 2-4%, which is an expected result. This difference is due to the reduction of the measuring area by 1 cm. In this way the required amount of sample is reduced from 5 g to 1 g. This holder is very suitable for small amount samples.

Table 1: Chemical composition on powder zeolite sample measured by small 3D printed powder holder and ordinary holder.

| Cd-ETS-4 small 3D printed powder holder |          | Cd-ETS-4 ordinary holder |         |
|-----------------------------------------|----------|--------------------------|---------|
| elements                                | mass %   | elements                 | mass %  |
| Cd                                      | 11.9337  | Cd                       | 9.5992  |
| Na                                      | 2.4532   | Na                       | 2.4395  |
| K                                       | 1.6994   | K                        | 2.2764  |
| Ti                                      | 38.1830  | Ti                       | 36.6379 |
| Si                                      | 44.8179  | Si                       | 48.3618 |
| Total                                   | 99.30872 | Total                    | 99.3148 |

The results of the chemical analyses of the second type 3D holder suitable for textile samples are presented in Table 2.

Table 2: Chemical composition of a textile samples containing Zn, measured with a 3D printed holder for textil.

| Textile Samples     |         |         |          |
|---------------------|---------|---------|----------|
| Initial sample – Ch |         | Ch Zn   | Zn Ch Zn |
| elements            | mass %  |         |          |
| Mg                  | 2.0489  | 3.3095  | –        |
| Al                  | 2.0315  | 1.0582  | 0.9888   |
| Si                  | –       | 1.6520  | 2.1623   |
| S                   | 6.4242  | 1.6520  | 1.1701   |
| Cl                  | 12.2734 | 1.5989  | 1.9027   |
| K                   | 6.2269  | 1.5899  | 3.0173   |
| Ca                  | 6.8172  | 5.2794  | 7.4254   |
| Ti                  | 60.2514 | 6.9571  | 13.0993  |
| Fe                  | 3.9265  | 0.4572  | 1.1555   |
| Zn                  | –       | 77.1993 | 69.0784  |

As it can be seen from Fig. 3, the holder is ideally suited for this purpose. This is because the “Lego like click” holder allows the textile sample to be pressed between the two parts of the holder thus it does not tear in a vacuum environment.

The use of a 3D printed holder for solid (alloy) sample allows the measurement the sample on small and large diameter focusing the X-ray beam on the desired area (Table 3. and Fig. 4) The table shows that with a 3D the printed holder with a small diameter of about 20 mm, the value of nickel decreases by about 3%. This is an expected result considering that the measurement was performed only on the yellow area of 2 leva. In the analyzing the alloy with the help of a large 3D printed holder (25 mm), the measurement was performed on the full area of 2 leva (white and yellow part).

Table 3: Chemical composition of metal sample (2 leva), measured with a 3D printed holders for metal alloys with different diameter.

| Holder   | Small diameter 20 mm | Large diameter 25 mm |
|----------|----------------------|----------------------|
| Elements | Mass %               |                      |
| Al       | 0.1115               | 0.1139               |
| Si       | 0.2683               | 0.2629               |
| P        | 0.0469               | 0.0393               |
| S        | 0.0943               | 0.0547               |
| Cl       | 0.5087               | 0.3066               |
| K        | 0.5830               | 0.4907               |
| Ti       | 0.6270               | –                    |
| Mn       | 0.0506               | 0.0936               |
| Fe       | 0.0582               | 0.0619               |
| Ni       | 5.6433               | 8.9648               |
| Cu       | 75.0869              | 74.3068              |
| Zn       | 16.9214              | 15.3048              |

For the determination the phases by powder X-ray diffraction (PXRD) again we used the 3D printed holder for the textile sample (Figs. 5 and 6) and the holder for the alloy sample – 2 leva (Figs. 7 and 8).

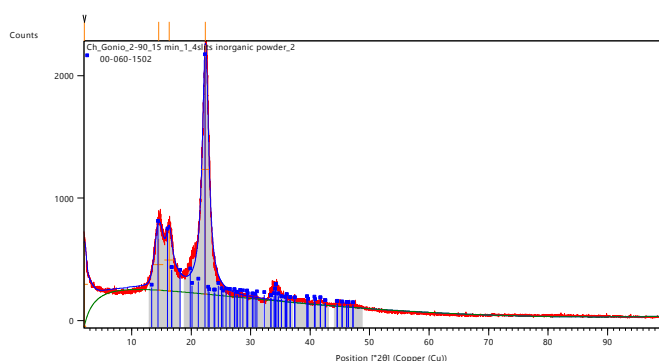


Fig. 5. Diffraction pattern of textile sample Ch measured with 3D holder

Table 4: Pattern List of textile sample Ch

| Ref. Code   | Compound Name | Displ.[°2θ] | Scale Fac. | Chem. Formula                                                 |
|-------------|---------------|-------------|------------|---------------------------------------------------------------|
| 00-060-1502 | Cellulose-Iβ  | -0.386      | 0.846      | (C <sub>6</sub> H <sub>10</sub> O <sub>5</sub> ) <sub>n</sub> |

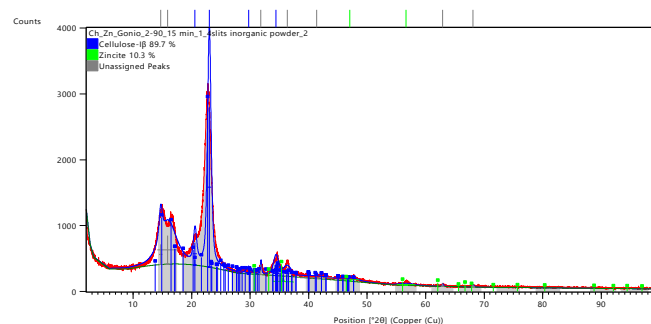


Fig. 6. Diffraction pattern of textile sample ChZn measured with 3D holder

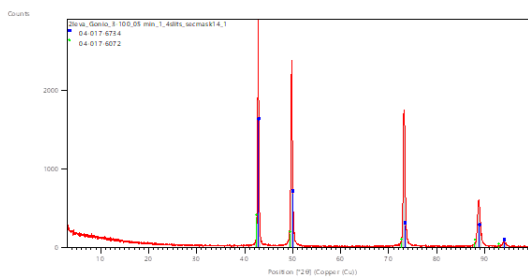
**Table 5:** Pattern List of textile sample Ch Zn

| Ref. Code   | Compound Name | Displ.[°2θ] | Scale Fac. | Chem. Formula                                                 |
|-------------|---------------|-------------|------------|---------------------------------------------------------------|
| 00-060-1502 | Cellulose-Iβ  | 0.058       | 0.634      | (C <sub>6</sub> H <sub>10</sub> O <sub>5</sub> ) <sub>n</sub> |
| 00-001-1136 | Zinc Oxide    | -1.132      | 0.045      | Zn O                                                          |

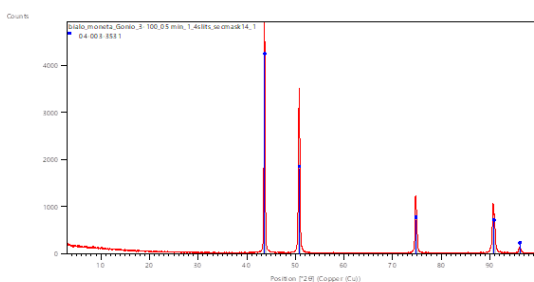
X-ray phase analysis of textile samples confirms the data from the chemical analysis for the presence or absence of zinc in the tested samples (**Tables 4, 5**).

For examination of the phase composition of the alloy sample - 2 leva the same holders were used as those used in the WDXRF chemical analysis of the alloys (**Fig. 4**)

The results from PXRD confirm those of WDXRF e.g. that the sample in a 3D holder with a diameter of 25 mm is an alloy of Cu, Mn, Ni and Fe (**Fig.7, Tables 3,6**). Phase analysis shows that the sample measured by the conventional holder only on the white part contains Fe and Ni (**Fig. 8, Table 7**). The results of the chemical analysis show mainly copper, zinc and a little nickel, but it should be noted that the study was conducted only on the yellow part of the 2 leva with 3D holder, a diameter of 20 mm (Table .3).

**Fig. 7.** Diffraction pattern of alloys from 2 leva measured with 3D holder – 25mm diameter**Table 6:** Pattern List of alloy sample 2 leva the yellow part

| Ref. Code  | Compound Name | Displ.[°2θ] | Scale Fac. | Chem. Formula      |
|------------|---------------|-------------|------------|--------------------|
| 04-017-734 | Copper        | 0.000       | 0.553      | Cu <sub>0.10</sub> |
|            | Manganese     |             |            | Mn <sub>0.40</sub> |
| 04-017-072 | Nickel        | 0.000       | 0.130      | Ni <sub>0.50</sub> |
|            | Iron          |             |            | Fe                 |

**Fig. 8.** Diffraction pattern of alloys from 2 leva measured with 3D holder – 20mm diameter**Table 7:** Pattern List of alloy sample 2 leva the white part

| Ref. Code   | Compound Name | Displ.[°2θ] | Scale Fac. | Chem. Formula                       |
|-------------|---------------|-------------|------------|-------------------------------------|
| 04-003-3531 | Iron Nickel   | 0.000       | 0.851      | Fe <sub>0.5</sub> Ni <sub>0.5</sub> |

## 4. Conclusions

Our experiments have shown that commercially available 3D printers and materials for it can be successfully applied in the field of science research. The advantages are the low price and

affordability of the products. Another indisputable positive is that a holder can be made according to the type and size of the sample.

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# Optimization of the synthesis of mesoporous titanosilicate ETS-10: Problems and unexpected difficulties

Liliya Tsvetanova<sup>1</sup>, Borislav Barbov<sup>1\*</sup>

Institute of mineralogy and crystallography "Acad. Ivan Kostov", Acad. Georgi Bonchev Str., bl. 107, 1113 Sofia, Bulgaria  
e-mail: lilicvetanova79@abv.bg

**Abstract:** We focused on the synthesis and subsequent use of mesoporous titanosilicate ETS-10 for industrial water purification. Numerous attempts have been made to optimize the conditions and reagents used for the synthesis of the ETS-10. The main problem we faced in the synthesis of ETS-10 is the production of "pure" phase and it was related to the type of titanium source. Various sources of titanium were used - titanium tetrachloride, anatase (anatase 92% and rutile 6%), nanosized titanium dioxide (Degussa P25) etc. The use of liquid titanium tetrachloride, due to its high hydrophilicity, leads to difficulties in reproducible control of the quantities of used reagents, which leads to the formation of unwanted crystalline phases, especially if the synthesis is carried out in small volumes. In addition, the optimized by us synthesis attempts included a reduced reaction time - up to 24 hours - and lower temperatures (up to 200 °C). For comparison standard synthesis is carried out for 72 h at 230 °C. As a result of the experiments, ETS-10 with minimal SiO<sub>2</sub> residues was obtained in 24 hours at 200 °C using nanosized anatase.

**Keywords:** ETS-10, SYNTHESIS, TITANOSILICATE, MESOPOROUS

## 1. Introduction

The interest in titanium-silicate microporous and mesoporous zeolite materials is huge. This is due to their wide application in catalytic reactions [1], as sorbents [2], ion exchangers [3] etc. Therefore, a lot of research is being done on optimizing their synthesis. Our main two goals are to synthesize a pure phase of mesoporous titanosilicate ETS-10, and exploit the sorption properties of this material.

ETS-10 was first synthesized by Engelhard in 1989 [4, 5]. It is one of the titanosilicates molecular sieves that have octahedrally coordinated titanium in its frame. Indeed, the building blocks of the ETS-10 structure are SiO<sub>4</sub> tetrahedra and TiO<sub>6</sub> octahedra. The bonding of these units through a shared/bridging oxygen produces a 7- and 12-membered rings. In turn those rings arrange to produce three-dimensional framework containing smaller and bigger channels with pore size of about 0.5 and 0.8 nm respectively. Only the channels generated by 12-membered rings are easily accessible and thus suitable for ion exchange. The basic formula of ETS-10 is (Na,K)<sub>2</sub>TiSi<sub>5</sub>O<sub>13</sub> [6]. The theoretical Ti/Si ratio is 0.2 but due the presence of structural defects it is generally higher (> 0.25). This framework allows the adsorption of cations with larger ionic radii which attracted our interest [7].

A number of different methods of synthesizing ETS-10 are known from the literature. First, syntheses with different sources of Ti and Si are possible: tetraethyl orthosilicate (TEOS), Sodium silicate aqueous solution [8], TiCl<sub>3</sub> [9], TiCl<sub>4</sub> [10], TiF<sub>4</sub> [11], Ti<sub>2</sub>O, commercial Degussa P25 [8, 12], sodium hydroxide, (TiSO<sub>4</sub>)<sub>2</sub> [13], (TiSO<sub>4</sub>)<sub>3</sub> [14] NaF, NaCl and potassium hydroxide, KF, KCl, ash from rice husks as a source of silicon etc. [7, 11, 12, 15-21]. Then, mesoporous titanosilicate ETS-10 can be synthesized with or without the addition of organic template molecules [16, 22, 23]. The organic template is environmentally undesirable and unprofitable because the removal of clogged templates requires calcination e.g. high temperature, and can damage the framework. Finally, time and temperature can be varied. Thus it is desirable to synthesize ETS-10 in the absence of organic template agents, at lower temperatures and reduced time.

## 2. Materials and methods

### Synthesis.

For the low-temperature hydrothermal synthesis of ETS-10, a number of experiments were performed with different sources of titanium, silicon, sodium, and potassium according to procedures described in the literature. The experiments aimed at optimizing the synthesis conditions e.g. without template and optimizations of temperature and time conditions.

The reagents used in our study for the synthesis of ETS-10 are: NaOH, KOH, KF, TiCl<sub>4</sub>, TiO<sub>2</sub> (Degussa P25) and (anatase 92% and rutile 6%), nanosized Si<sub>2</sub>O and Si<sub>2</sub>O with size < 10 μm. The optimized synthesis system from us is: 0.42Na<sub>2</sub>O-0.03K<sub>2</sub>O-0.1TiO<sub>2</sub>-0.5SiO<sub>2</sub>-5.5H<sub>2</sub>O, in which the reaction conditions such as

alkali cations, content water and ratio Si/Ti, pH, etc. were changed. A typical recipe for synthesis of ETS-10 used in this work is presented below.

Synthesis procedure: 3.36 g of NaOH are added to 8 ml of water, stirred until clear (first solution). A second solution was prepared in parallel - 0.797 g of TiO<sub>2</sub> (anatase) and 1.6 H<sub>2</sub>SO<sub>4</sub> were added to 2 ml of water. 3.30 g of SiO<sub>2</sub> (nanosized) are then added to solution 1 and stirred for 20 minutes. Then the two solutions (Ti: Si) are slowly mixed, stirred for about 20 minutes on a magnetic stirrer without heating and the pH measured. Then 0.871 g of KF were added, with stirring for additional 20 minutes. After that the gel mixture is transferred in stainless steel Teflon lined autoclave and the autoclave placed in preheated oven heated to 200 °C for 22 hours. The obtained product is centrifuging and dried at 90 °C for 2-3 hours.

### Methods.

The main method for characterizing the phases is powder X-ray diffraction (PXRD). Samples were measured at room temperature on an Empyrean MalvernPanalytical diffractometer, the Netherlands. Data collection was performed with Cu radiation (λ = 1.5406 Å) at 40 kV and 30 mA and PIXcel3D detector in the range 2 - 90° 2θ and step mode 0.013° 80 seconds and experiment duration 35 minutes.

Wave-dispersive X-ray fluorescence spectroscopy (WDXRF) was used to determine the chemical composition of the phases. The spectrometer is Rigaku Supermini200, Japan. Data collection was performed operating mode at 50 kV and 4.00 mA. The sample is crushed and then pressed. The ratio of sample amount (in grams) to the amount of glue (Acrawax C powder) is 5:1 g. The sample is placed in a holder with an irradiated area of 30 mm in diameter. The analysis used a semi-quantitative method to determine the elemental composition. The results obtained by the "SQX" semi-quantitative analysis method allow the determination of the chemical composition of samples without the need for reference materials (standards). One WDXRF measurement has a duration 30 minutes.

Scanning electron microscopy (SEM) is used to determine the morphology and size of the crystals. Scanning electron microscopy (SEM) was performed on a Philips 515 microscope operating at 20 keV in secondary electron mode. The samples were mounted on a sample holder with a conductive carbon coating.

## 3. Results and discussion

### Titanium tetrachloride synthesis

Figure 1a shows a diffraction pattern of synthesis with the following molar composition: 0.59Na<sub>2</sub>O-0.69K<sub>2</sub>O-0.1TiO<sub>2</sub>-3.95SiO<sub>2</sub>-267H<sub>2</sub>O. The initial pH is about 12. Nano-sized SiO<sub>2</sub> was used. The synthesis temperature is 200 °C and the duration is 24 hours.

On the Figure 1b the diffraction pattern with molar ratio: 0.29Na<sub>2</sub>O-0.35K<sub>2</sub>O-0.1TiO<sub>2</sub>-1.98SiO<sub>2</sub>-133H<sub>2</sub>O is presented. The initial pH is about 13. As Si source SiO<sub>2</sub> with crystallite size <10μm

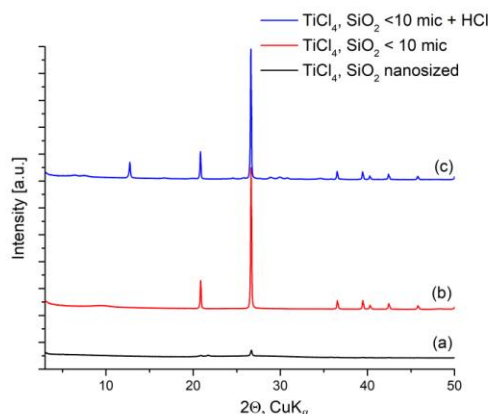
was used. The synthesis temperature is 200 °C and the duration is 24 hours.

In a later synthesis of ETS-10 was used following molar composition:  $0.59\text{Na}_2\text{O}-0.69\text{K}_2\text{O}-0.1\text{TiO}_2-3.95\text{SiO}_2-267\text{H}_2\text{O}$ . The initial pH is about 11. For source of Si was used micrograin  $\text{SiO}_2$  with size of crystallites less than  $10\mu\text{m}$ , as well hydrochloric acid. The synthesis temperature is 200 °C and the duration is 24 hours. (Fig. 1c).

In the synthesis of ETS-10 with different amounts of titanium tetrachloride for 24 hours, at 200 °C and different sources of  $\text{SiO}_2$  (nanosized and micrograins  $< 10\mu\text{m}$ ), as well as the use of hydrochloric acid from Fig. 1 one can see that mostly quartz and ETS-4 are obtained.

For the conditions in Fig. 1a the amount of titanium tetrachloride is very low and the source of  $\text{SiO}_2$  is nanosized in contrast to those in figure 1b. Under such conditions, quartz begins to form. Using low amounts of  $\text{TiCl}_4$ , micrograin  $\text{SiO}_2$  and HCl (to control the pH) the amount of quartz increases at the expense of ETS-4 (Fig. 1c).

These results are an indication that at 200 °C for 24 hours and with the selection of the listed reagents it is impossible to obtain ETS-10, instead ETS-4.



**Fig. 1** XRD ETS-10 synthesized with  $\text{TiCl}_4$  and nanosized  $\text{SiO}_2$  under the same conditions -200°C/24h: **a)** Quartz; **b)** Quartz~80% and ETS- 4~20%; **c)** Quartz~85% and ETS- 4~15%

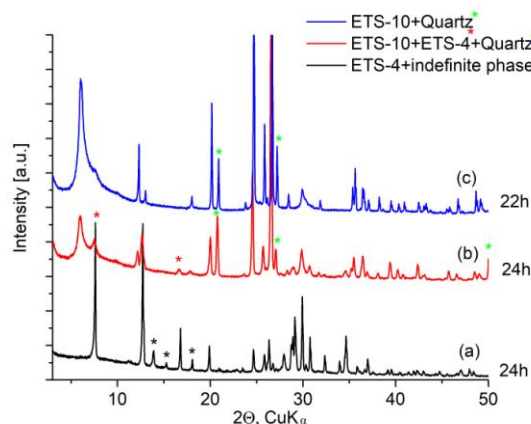
Additional syntheses with a source of Ti -  $\text{TiCl}_4$  were performed, in which both the amount of titanium and water were changed.

The characteristics of the synthesized phases under different conditions of the system are presented in Fig. 2. The results show that by hydrothermal synthesis at 200 °C for 24 h, the use of increased amounts of titanium tetrachloride and less water ETS-4 with impurities is obtained (Fig. 2a). When the amount of titanium tetrachloride is decreased and the amount of water increased, a mixture of ETS-10 / ETS-4 / Quartz is obtained (Fig. 2b).

#### Titanium dioxide – anatase synthesis

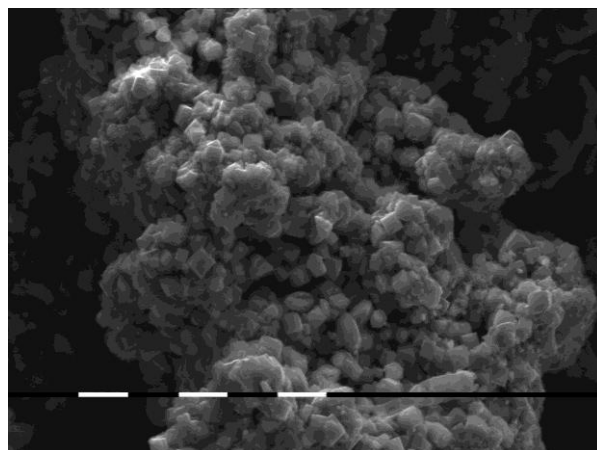
The system and procedure described below was used for the synthesis of ETS-10 with minimal amounts of quartz (Fig. 2c).

The most optimal of our tested conditions are those using nanosized titanium dioxide - anatase, nanosized silicon dioxide, increased amounts of sodium hydroxide and the use of potassium fluoride as sources of alkalis, sulfuric acid, pH=10.6 and reducing the synthesis time to 22 hours (Fig. 2c).



**Fig. 2** XRD ETS-10 synthesized at different times with different precursors; **a)** ETS-10 -200°C/24h,  $\text{TiCl}_4$ , **b)** ETS-10 -200°C/24h,  $<\text{TiCl}_4>\text{H}_2\text{O}$ ; **c)** ETS-10 -200°C/22h,  $\text{TiO}_2$  (anatase).

The result of the SEM images (Fig.3) shows that the ETS-10 crystals morphology is cubic and the size of the crystallites is  $\sim 5\mu\text{m}$ . This is a characteristic morphology for the synthesis of ETS-10 with nanosized titanium dioxide - anatase, established by Rocha in 1998.



**Fig. 3** SEM images of the ETS-10 (particle size  $5\mu\text{m}$ )

The chemical results from WDXRF are presented in Table. 1 and correspond to the stoichiometric formula:  $(\text{Na}_{1.10}\text{K}_{0.28})\text{Ti}_{0.91}\text{Si}_5\text{O}_{13}$  which is in agreement with  $(\text{Na,K})_2\text{TiSi}_5\text{O}_{13}$ . The table shows that no significant amounts of chlorine were found in the product.

**Table 1.** Chemical composition of the final product (ETS-10) synthesized by nanosized  $\text{TiO}_2$

| Oxide                | Mass %  |
|----------------------|---------|
| $\text{N}_2\text{O}$ | 10.2057 |
| $\text{K}_2\text{O}$ | 3.6023  |
| $\text{SiO}_2$       | 72.4312 |
| $\text{TiO}_2$       | 13.7135 |
| Cl                   | 0.0472  |

To determine the Si/Ti ratios, an elemental analysis of WDXRF was performed (Table 2). The results show that the Si/Ti ratio is 2.6, which is less than reported by Rocha 5-6 for pure ETS-10 phase. In our experiments for the synthesis of ETS-10, we found that with such a ratio (2.6) we obtain the phase with a small contamination with quartz, which contradicts Rocha's statement that quartz is formed mainly at a Si/Ti ratio above 6 [8].

**Table 2:** Elements composition of the final product (ETS-10) synthesized using TiO<sub>2</sub> (anatase, temperature 200°C/22h)

| Elements  | Mass %  |
|-----------|---------|
| Na        | 10.3135 |
| K         | 7.3170  |
| Si        | 59.4409 |
| Ti        | 22.7390 |
| Cl        | 0.0985  |
| Si/Ti     | 2.6140  |
| (Na+K)/Ti | 0.7753  |

#### 4. Conclusion

From the experiments conducted so far, it is clear that the ETS-10 synthesis system is sensitive to many of the parameters such as titanium source, alkali, pH, content water, temperature, time of synthesis. For example, when using titanium tetrachloride, as it is hydrophilic and changes the whole stoichiometry, the amount must be accurately estimated, because otherwise it has a negative effect on the final product.

We achieved the preparation of the ETS-10 phase at 200 °C for 22 hours, with minimal impurities of quartz without the use of SDA. The ETS-10 phase can be obtained with minimal impurities only when using a combination of reagents nanosized source of TiO<sub>2</sub> - anatase, nanosized source of SiO<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>, KF and NaOH.

To obtain a pure phase, the sources of titanium and alkali must be carefully selected and the temperature raised to 230 °C.

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# Ultra-short laser patterning of silk fibroin thin films - a potential scaffold platform in tissue engineering applications

Albena Daskalova, Liliya Angelova

Institute of Electronics at Bulgarian Academy of Science, Sofia, Bulgaria 72, Tzarigradsko Chaussee Blvd., 1784-Sofia, Bulgaria  
albdaskalova@gmail.com, lily1986@abv.bg

**Abstract:** Silk-based scaffolds are specifically investigated in various tissue engineering applications, including for cartilage, bone, nerve, muscle, skin, or corneal regeneration. Guiding muscle cell growth is a challenging task in muscle tissue engineering. In this work, a self-assembled silk fibroin thin films were processed by ultra-short laser radiation to investigate its potential for guiding muscle cell proliferation. The ultra-short laser processing of silk fibroin (SF) have produced micro channels which are suitable for self-assembling and orientation of muscle cells and provides a niche for its attachment. Silk fibroin is an excellent candidate as a biomaterial for tissue engineering applications. Moreover, bacterial biofilm formation on surfaces are associated with persistent microbial contamination. Thus, recently new approaches are needed to impede bacterial surface colonization. Using femtosecond laser irradiation (wavelength 800 nm), laser-induced surface microstructuring is applied to achieve non-thermal, precise, and crack free surface processing with different topographical designs on silk fibroin thin films in order to enhance repelling of bacteria attachment.

**KEYWORDS:** BIOMATERIALS, TISSUE ENGINEERING, LASER PATTERNING, ANTIMICROBIAL, CELLULAR ADHESION

## 1. Introduction

Tissue engineering is a field in biotechnology that deals with the creation, regeneration, and improvement of the function of biological tissues. The purpose of the matrix is to create a biomimetic environment that stimulates cell adhesion, differentiation, and proliferation. In this way, the cells can be reorganized into new three-dimensional tissue. In the process of tissue regeneration, the matrix itself decomposes, leaving only the new tissue. The main focus of this study is on the creation of laser-structured, tissue matrices based on silk and fibrin for the regeneration of muscle and nerve tissue. Silk has specific amino acid motifs (RGD motifs), which are necessary for stable cell adhesion and are part of the natural extracellular matrix surrounding the cells in vivo [1]. The main disadvantages of biomaterials are the weak mechanical properties and the possibility of an immune response from the recipient after implantation. Thanks to its main building block, the protein fibroin, silk fibers have extremely good mechanical properties (strength, toughness, elasticity, strength), which presents the main features which are the base in the use of silk matrices for the regeneration of tissues, subjected to mechanical stress under normal physiological conditions. [1]. In order to be biocompatible, the outer layer of silk thread made up of the protein sericin is removed, thus the material does not cause an immune reaction from the body in which it is placed. Another biological material with extensive application in tissue engineering is fibrin, which is a biopolymer that plays a major role in blood clotting and wound healing. Most often, fibrin is applied in tissue engineering in the form of a hydrogel, and its application gives the best results in the regeneration of muscle, skin, and cartilage tissues. In addition, the fibrin-based hydrogel stimulates angiogenesis and the growth of dendrites from neurons [2].

Most chemical methods use organic chemicals to control the properties of the matrices, leaving residual toxic traces. In addition to chemical methods, the properties of various cell matrices can be controlled by physical methods, the most promising of which are laser sintering and laser-based surface modification. Laser techniques are non-contact and do not require the use of chemicals for additional treatment of the matrix. Irradiation with ultra-short laser pulses is an alternative approach by which these shortcomings can be overcome, as they possess extremely high processing accuracy and do not lead to the formation of thermal deformations on the processed material due to their extremely short pulse durations. Research in recent years has shown that surface modifications through the application of ultra-short pulse laser pulses change the topography of the material and thus can improve its properties and functions. Specific to tissue engineering, this type of modification on cell matrices leads to the creation of micro and nanostructures on the surface of the material, which in turn can strongly affect cell adhesion, orientation, and differentiation and subsequently can enhance bacterial rejection. For example, for

proper differentiation and orientation, muscle cells must occupy a certain parallel conformation. This structuring of the cells is extremely important, as it generates the uniaxial force required for proper muscle development. Obtaining certain parallel patterns by applying laser-induced modeling can direct muscle cells to occupy the correct conformation and thus stimulate the creation of muscle tissue. In addition, the surface laser treatment leads to a change in the wettability of the material, which allows control over its interaction with living cells and extracellular structures.

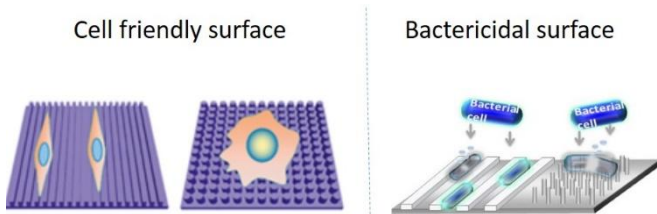
Silk, is a natural biopolymer material, and has become a promising hi-tech material. Silk fibroin (SF) origin is from Bombyx mori silkworm. It represents a fibrillar protein composed of three parts: heavy chain fibroin (391 kDa), light chain fibroin (27 kDa), and P25 (25 kDa). Silk fibroin possess high level of biocompatibility, and tunable biodegradation rate, as well as strong mechanical characteristics. SF can be processed into versatile forms, thus it becomes a favorable material for development of various drug delivery platforms, medical devices, and tissue-engineering matrices SF in an aqueous state or dissolved in organic solvents is explored to synthesizing films, and nanofibers, for diverse biomedical applications Fig.1.



**Fig. 1** Types of scaffolds based on silk fibroin.

Besides the vast of excellent biocompatibility properties that SF possess and makes it a perfect candidate for a wide range of applications, a major drawback with SF is related to the absence of antibacterial properties. Bacterial infection is of considerable importance and when an implantable biomaterial is applied into the body. Biomaterial contamination represents a great treat and can evoke difficulties to heal infections, thus leading to risk to the

patient's health status. Despite the huge impact that medical implants have on patients' health and quality of life, the possibility of side effects after implantation remains. One of the most common complications is the development of a bacterial infection associated with the implant itself. *S. aureus* and *S. epidermidis* have been listed as the main pathogens that colonize implants and lead to severe inflammation [3]. When the implant is inserted at the site of injury, both human and bacterial cells compete for a place to attach to the material's surface. The initial adsorption of bacteria onto an inert and abiotic implant is unspecific and directed mainly by electrostatic, hydrophobic and van der Waals forces [4]. After they are attracted to the implant, microbes could bind irreversibly to the surface itself or to various serum proteins that have already been randomly adsorbed to the implant surface. Once the pathogens have attached to the surface, they proliferate and interact with each other in order to form an extracellular polysaccharide matrix—the bacterial biofilm [5]. The microbes, being embedded in such a layer, could become resistant to common antibiotic treatments. Consequently, the task of eliminating the developed infection becomes extremely hard. The effects of topographical features and roughness profile of different surfaces on bacterial attachment have been widely explored Fig. 2. Generally, with the increase in surface roughness, the surface area suitable for microbial adhesion also increases, which, in turn, results in a higher chance of a biofilm formation. Despite this general observation, it has also been discovered that surfaces with higher roughness allow the adsorption of serum proteins. In consequence, a thin coating is formed which masks the rough nature of the surface and impairs the interplay between the bacteria and the material. The surface topography and morphology are two key factors that influence bacterial behavior and are strongly correlated to the surface roughness.



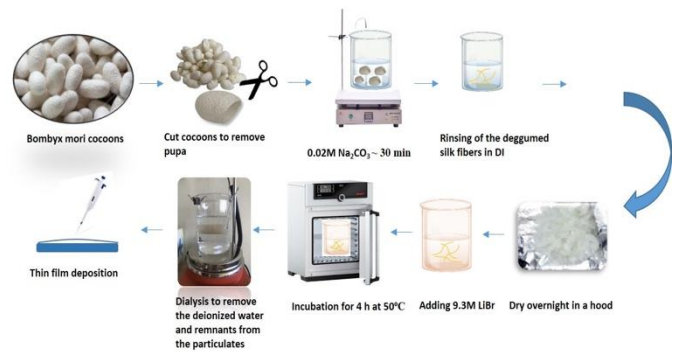
**Fig. 2** Textured surfaces : Cell attractive for enhanced cellular adhesion and proliferation; Antibacterial structuring for prevention of microbial adhesion - biomimetic surface texturing.

Silk doesn't possess the cell adhesive components, such as arginine-glycine-aspartic acid (RGD) sequence, which enhances cell adhesion. Thus additional laser based treatment applied to thin SF films will introduce a morphological change expressed in deviation in surface roughness, making the surface from one hand bacteria repellent and on the other hand more cell friendly.

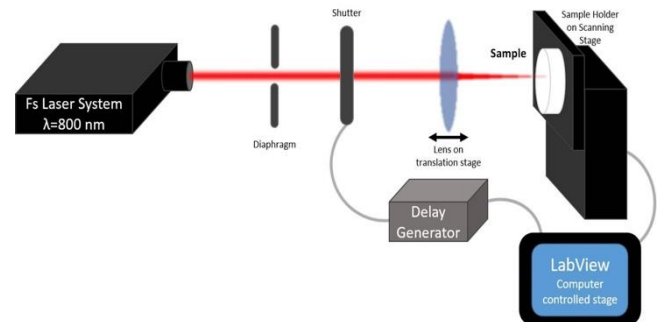
## 2. Materials and methods

Silk fibroin was extracted from *Bombyx mori* cocoons. The *Bombyx mori* cocoons were cut into small pieces and degummed in 0.2 M  $\text{Na}_2\text{CO}_3$  solution at 100 °C for 1.5 h under stirring. The resulted degummed silk was rinsed with deionized water to assure the removal of sericin. And last step was to dissolve the sericin free silk in 9.3M LiBr solution for 3h at 60°C. The obtained viscous solution was finally dialyzed and centrifuged to remove the deionized water and remnants from the particulates. Subsequently, silk thin films were dried at drying oven at 37° C for 48h. Aqueous silk solutions were used to make thin films on the surface of glass slides Fig. 3.

Laser processing was carried out using  $\lambda = 1030$  nm wavelength,  $\tau = 150$ fs duration pulses (Ti:sapphire mode-locked Quantronix-Integra-C system) operating at a frequency of 500 Hz. Fig.4.



**Fig. 3** Process of extraction of silk fibroin (SF).



**Fig. 4** Schematic of the femtosecond laser processing setup.

The samples were positioned on XY translation stage. The irradiations were performed in air. The number of pulses (N) delivered at each laser spot was defined by the speed of the translation stage, and the distance between each two consecutive laser-created spots ( $d_x$ ) and the distance between two separate rows ( $d_y$ ) was controlled between 32  $\mu\text{m}$  and 45  $\mu\text{m}$ . The laser fluence (F) was varied between 0.4 and 1.25 J/cm<sup>2</sup>.

## 3. Results and discussion

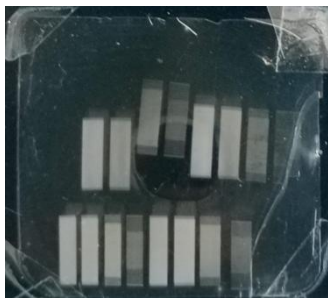
The ability to control the surface properties of biomaterials and improve the adhesion of different types of cell cultures and biomolecules sets out the basic requirements for the development and use of new generation biomaterials for the purposes of tissue engineering and regenerative medicine. Various methods and approaches are used to improve the surface and volumetric properties of different type scaffolds, combining all kinds of knowledge from various fields such as physics, chemistry, biology, medicine.

The work of various research groups, clearly shows that the texturing of biomaterials has a significant effect on cellular development, compared to untreated "smooth" and non-textured surfaces. The literary reference shows that dependencies of cell behaviour are observed according to the characteristics of the given surfaces on which they are seeded. For example, Alexandre Cunha et. al. [6] show that by controlling the output laser parameters (energy, number of laser pulses, radiation polarization), different sized textures of Ti-6Al-4V surfaces are produced by laser processing with ultra-short pulses. The resulting structures exhibit hydrophilic behaviour and high affinity for HBSS solution (Hank's balanced salt solution). The surfaces thus treated, are characterised by an increase in surface wettability and can be used to control the behavior of hMSCs cells by exploring the change in their cytoskeletal form and proliferation. Another research group of Liang et.al., treated surface of NiTi alloys by femtosecond laser irradiation and observed growth of osteoblasts cells, oriented along the laser created grooves, as a result, large amount of synapses and filopodia were formed due to the grooves, holes and nanoparticles on the alloy surface. Moreover, as a result of the femtosecond laser treatment, noticeable improvement of biocompatibility due to

formation of micro-patterns of NiTi alloys was achieved. Surface patterning on micro and nano- scale is critical for distinguishing the effects of cell shape, focal adhesion, and ligand input for improving cell functions. Thus, the different kinds of structures, such as grooves, grate, ladder and pit will lead to the observation of different effects of cell behaviour by altering their geometry [7].

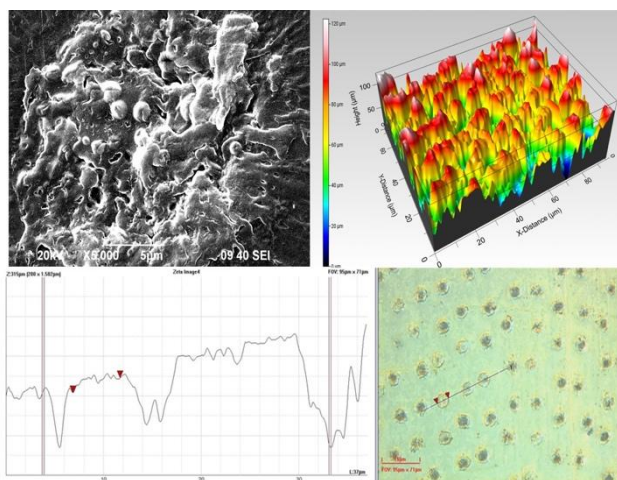
One of the recent concepts of creating scaffold-type structures for tissue growth is based on seeding well-selected cell cultures, providing mechanical attachment and “directed cells growth” on artificial grafts [8, 9]. The creation and modeling of the so-called “scaffold ” for the needs of tissue engineering is a major challenge due to requirements related to the mechanical and morphological properties of the surface as well as their biodegradability [10,11].

Femtosecond laser processing can also provide other types of surface geometries (foam and bubble – like structures) by varying translation stage scanning speed, applied laser energy, pulse number and sample positioning. The films of silk fibroin were irradiated by a fs-laser beam Fig. 5.



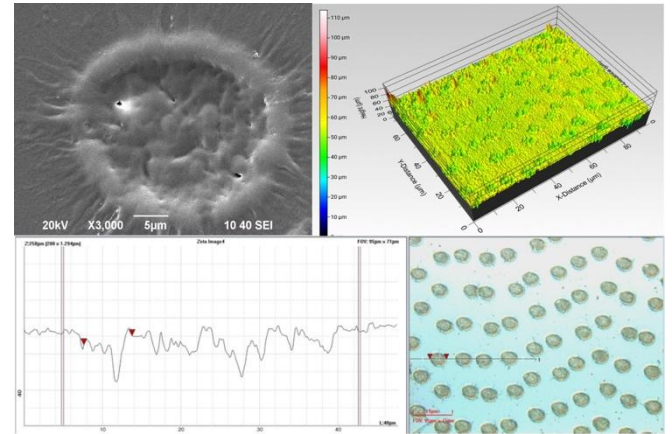
**Fig. 5** Image of femtosecond laser patterned thin film of silk fibroin. Creation of modification zones under irradiation with a range of laser parameters.

It can be observed that the laser irradiation of the silk fibroin films induces different modifications expressed also in the color of the processed areas, due to effect the local heat distribution and pressure waves generated by the laser interaction. The surface morphological characteristics of silk fibroin films can be tailored by (i.e. ridges, grooves structures) by single and multiple pulses from femtosecond laser irradiation and varying the scanning velocity. A typical SEM image of a laser - induced porous foam formation on the surface of silk thin film after irradiation with single pulse is shown on **Fig. 5**.



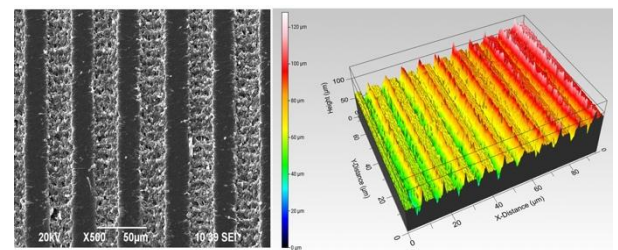
**Fig. 6** SEM image and optical profilometer surface mapping of silk fibroin film after laser exposure to  $\lambda = 800\text{nm}$ ,  $\tau = 150\text{fs}$ ,  $N=1$ ,  $E = 0.4\text{J}/\text{cm}^2$ .

Surface modification in the form of foam formation produced after irradiation by fs-laser pulses under high repetition rate, is a complex phenomenon, which involves interplay between strong energy deposition localization, low heat conductance, and surface tension effects. Thus modification process is triggered via strong ionization and electron ejection from the surface. After increase of the applied laser energy ( $E$ ) to  $0.8\text{J}/\text{cm}^2$  formation of ridge is observed at the ablation zone surroundings **Fig. 7**.



**Fig. 7** SEM image and optical profilometer surface mapping of silk fibroin film after laser exposure to  $\lambda = 800\text{nm}$ ,  $\tau = 150\text{fs}$ ,  $N=1$ ,  $E = 0.8\text{J}/\text{cm}^2$ .

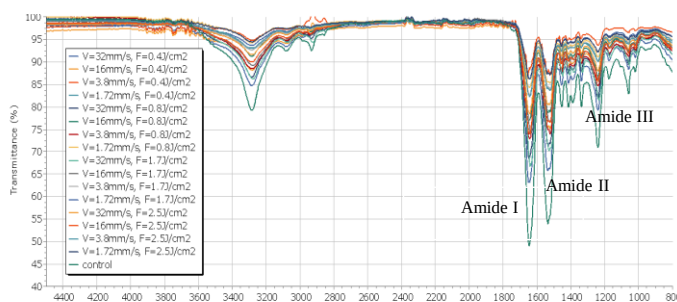
The porosity of the ablation zone is diminished, leading to formation of a melted layer and filling of the pores. In the case of ‘bubble’ types of structures, the roughness parameter ( $S_a$ ) of the irradiated zone was measured to vary from  $28\mu\text{m}$  to  $17\mu\text{m}$  for single pulse irradiation, along with increasing laser fluence values. The characteristic ‘sponge-like’ structure transforms to morphological structure with folded central part, leaving area with edges.



**Fig. 8** SEM image and optical profilometer surface mapping of microchannels silk fibroin film produced after laser exposure to  $\lambda = 800\text{nm}$ ,  $\tau = 150\text{fs}$ ,  $N=5$ ,  $E = 0.4\text{J}/\text{cm}^2$ .

Laser patterning in the form of micro channels results in a precise removal of a material, and formation of clean stripe – like channels **Fig.8**. The direct ablation of silk fibroin thin film hasn’t induced damage to the surroundings of the processed zone. The proposed geometry is suitable for muscle and nerve cell ‘Contact guidance’, which is of main importance to control cellular morphology, orientation and direct cell migration. This type of patterning will permit the cells to occupy the bottom of the microchannels and align along the shapes of the produced microchannels. Micro-patterning technology, can enable the geometric control of muscle and neuronal cell alignment.

We have used the Fourier Transform Infrared Spectroscopy (FTIR) in transmittance mode to evaluate the molecular and crystalline structures of the silk fibroin thin films before and after irradiation with a range of laser fluences and scanning velocities **Fig.9**.



**Fig. 9** FTIR transmittance spectra of silk fibroin films before and after laser irradiation with a range of energies between  $E = 0.4 \text{ J/cm}^2 - 2.5 \text{ J/cm}^2$  and scanning speed in the range of  $V = 1.7 \text{ mm/s}$ ,  $3.8 \text{ mm/s}$ ,  $16 \text{ mm/s}$ ,  $32 \text{ mm/s}$ .

FTIR spectroscopy was employed to analyze the conformational changes that are established during laser processing process. The  $\alpha$ -helix is represented by a strong absorption bands at  $1656 \text{ cm}^{-1}$  (amide I) (C=O stretching vibrations),  $1540 \text{ cm}^{-1}$  (amide II) (secondary N–H bending), and  $1230 \text{ cm}^{-1}$  (amide III) (C.N stretching vibrations and N.H bending). The FTIR measurements demonstrate that the silk fibroin matrix is not affected by the applied laser radiation, the main amide groups are present in all spectra. The only difference is observed in the decrease of the peaks intensity in comparison to untreated sample.

## Conclusion

Laser patterning of micrometer dimensions of silk fibroin thin films is demonstrated using fs-laser exposure to diverse energies and scanning speeds. Obtaining porous microstructured thin-film surfaces with porous characteristics from naturally derived polymers (silk) could be used for muscle and nerve cells engineering and/or in cell directional growth and tissue regeneration. The obtained design of processed silk fibroin with topographical and chemical features suitable for engineering biointerfaces with multiple functionalities is a main factor in creation of effective topographical bioplatfroms equipped with a possibility to regulate cells fate.

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# Симулационно моделиране коефициент на реституция

## Simulation Modelling of Coefficient of Restitution

Nikolay Stoimenov \*, Miglena Paneva, Gabriela Kotseva, Blagoy Sokolov  
Institute of Information and Communication Technologies at the Bulgarian Academy of Sciences,  
Acad G. Bonchev str. block 2, 1113 Sofia, Bulgaria  
\*nikistoimenov@gmail.com

**Abstract:** This article discusses types of mills, used to reduce the size of grinding materials. An analysis of methods for determining the restitution coefficient was made, after that a method for determining the restitution coefficient was chosen. Input parameters have been experimentally determined, enabling proper modeling and simulation. The process is modeled and then simulated in a 3D software environment working on the of discrete elements method. 3D printed materials were used as input data on the restitution coefficient in the software. The obtained data is compared with the experimentally established restitution data from the real experiments.

**Key words:** Mills, milling processes, coefficient of restitution, 3D modelling, 3D simulation, 3D printing.

### 1. Въведение

Добиването на полезни изкопаеми, които в състава си имат достатъчно съдържание на полезни минерали, се обработва, след което се получава т.нар. изходен продукт. Осъществяването на ефективен и качествен обогатителен процес изисква изходния продукт да претърпи технологична обработка за осредняване на минералния и химичния състав на продукта. Най-често този процес се осъществява в мелници (топкови, автогенни, полу-автогенни).

В индустрията все още се използват топкови мелници със сферични тела. Терминът топка мелница е ограничен до тези, които имат съотношение дължина към диаметър от 2 до 1 и по-малко. Сферичните мелници, при които съотношението дължина към диаметър е между 3 и 5, са обозначени тръбни мелници [1-3].

Процесите на смилане в топкови мелници са изключително трудни за изследване. Поставянето на сензори и уреди води до увреждането им, поради агресивната работна среда. Симулирането на процеса би допринесло за разбирането на важни фактори, влияещи на производителността, качеството на изходния продукт, използваната електроенергия и други [4]. Има данни, че при симулиране на различни процеси, чрез точно моделиране, разходите могат да бъдат намалени с над 50% [5].

Целта на настоящата статия е моделиране и симулиране на коефициент на реституция с цел верификация на получени резултати от опитно установяване.

### 2. Коефициент на възстановяване

Коефициентът на възстановяване (реституция) се използва при характеризирание на скоростта на разсейване на енергията, включващи сблъсъци на повърхности, най-често със сфери. Предизвикателството при измерването на коефициента на възстановяване е влиянието на фактори, наблюдавано в експерименталните данни, което е резултат от различни свойства на обектите, форма и грапавост на повърхността, както и от несъвършенства в експерименталната техника. Коефициентът на възстановяване зависи от много елементи, като геометрията на телата в контакт, скоростта на приближаване, свойствата на материала, продължителността на контакта и евентуално триенето. Коефициентът на възстановяване е често срещан и популярен параметър, използван в много модели на сблъсък, особено за сблъсък без триене поради неговата простота и обикновено се прилага като постоянни стойности между нули до единица [6-9].

Един от методите за определяне на коефициента на възстановяване е чрез височини при свободно падане на сфера, като се измерва височината от която пада, както и височината на отскока от повърхността, с която сферата има контакт. Друг

метод е чрез определяне на скорост, като съотношението на скоростта след удар към скоростта скорост преди удара. Изследвания показват, че определянето на коефициента според тези методи е съпоставимо [6-9].

Загубите на енергия по време на тези сблъсъци са важни за правилното моделиране и симулиране на динамиката на процесите в топковите мелници.

### 3. Експериментална подготовка

За верифициране на моделирането и симулирането на коефициент на реституция са използвани данни от опитно определен коефициент на реституция. Коефициента е определен, като са отчетени височините преди и след удар. Сфера, свободно падаща е пусната от височина 540 mm. Сферата пада върху повърхност, след което с помощта на високоскоростна камера NAC MEMRECAM HX-6 [10, 11] е записан отскока. Камерата позволява снимки от 2000 до 370 000 кадъра в секунда, разрешаваща способност регулируема според броя на кадрите в секунда:

- при резолюция 1920x1080 до 2330 кадъра в секунда;
- при резолюция 640x480 до 14 150 кадъра в секунда;
- при минимална резолюция 320x24 до 370 330 кадъра в секунда.

Камерата разполага с вътрешна бърза памет 32 GB, външна синхронизация, 3 различни обектива (вкл. варио обектив), температурна калибровка, допълнително осветление от два прожектора по 1 KW, управляващ софтуер и софтуер с възможност за измерване на позиция, скорост, ускорение и ъглови параметри. Може да работи с различни програмни продукти, разполага с дистанционно управление, с възможност за playback от самата камера, USB и Ethernet интерфейси.

С помощта на софтуерен продукт Vicasso 2009 [12, 13] са отчетени височините. Софтуерът позволява възпроизвеждане на единичен видеоклип, с точен достъп до всяка част от видеото чрез лента за проследяване или бутони за единичен кадър. Самото изображение може да се увеличава или намалява. Поддържа сравнение на няколко видеоклипа. Богат набор от инструменти за обработка на изображения позволява на изображението да бъде подобро, за да се премахне шума или да се увеличи контраста, така че да могат да бъдат разкрити специфични детайли. Тези подобрения могат да бъдат приложени към целия видео поток, така че тези функции да

могат да бъдат следвани във видео и подобрено видео да бъде запазено във файл. По-нататъшният анализ на изображението се подпомага от редица инструменти за калибриране и измерване, включително предоставянето на решетки и линии за калибриране и измерване. Друга предоставена ценна функция е анотацията на видео изображението, било то с текст, решетки, линии и информация за времето или лог, което помага за персонализиране на видеото.

Сферата и повърхността, върху която пада свободно са 3D принтирани с материал PolyLactic Acid (PLA). Сферата е 3D принтирана с височина на слоя 0.16 mm, запълване 100%. Данни за характеристиките на сферата са предоставени в таблица 1. Повърхността е принтирана със същата височина на слоя, като запълването е 20%. Плътните слоеве между запълването са по 5 броя. Таблица 2 показва параметрите на повърхността, върху която пада сферата. Получените резултати от експерименталното определяне на коефициента на възстановяване са представени в таблица 3. Направени са 3 опита, след което е взета средната стойност. За определяне на коефициента на възстановяване, е използвана формулата [6]:

$$e = \frac{v_2}{v_1} = \left(\frac{h_2}{h_1}\right)^{\frac{1}{2}} \quad (1)$$

,където:

$e$  – коефициент на възстановяване;

$v_2$  – скорост след отскок;

$v_1$  – скорост преди отскок;

$h_2$  – височина преди отскок;

$h_1$  – скорост преди отскок.

Таблица 1. Характеристики на 3D принтирана сфера.

|                               |            |
|-------------------------------|------------|
| Материал                      | PLA        |
| Обем [m <sup>3</sup> ]        | 3.817e-7   |
| Плътност [kg/m <sup>3</sup> ] | 785.957558 |
| Маса [kg]                     | 0.0003     |
| Размер [D, m]                 | 0.009      |

Таблица 2. Характеристики на 3D принтирана плоча.

|                               |                |
|-------------------------------|----------------|
| Материал                      | PLA            |
| Обем [m <sup>3</sup> ]        | 7.260260224e-5 |
| Плътност [kg/m <sup>3</sup> ] | 418.717        |
| Маса [kg]                     | 0.0304         |
| Ширина [m]                    | 0.06016        |
| Дължина [m]                   | 0.119416       |
| Височина [m]                  | 0.01           |

Таблица 3. Характеристики на 3D принтирана сфера.

| Основа/<br>сфера       | № експери-<br>мент | Начална<br>височина<br>[mm] | Висо-<br>чина<br>на<br>отскок<br>[mm] | Коефициент<br>на възста-<br>новяване |
|------------------------|--------------------|-----------------------------|---------------------------------------|--------------------------------------|
|                        | 1                  | 542                         | 141,17                                | 0.510354048                          |
|                        | 2                  | 542                         | 213,44                                | 0.627535448                          |
|                        | 3                  | 542                         | 136,49                                | 0.501823244                          |
| Средна<br>стойнос<br>т |                    |                             | 163,7                                 | 0.549572125                          |

#### 4. Симуляционно моделиране

За симуляционното моделиране е използван софтуерен продукт, работещ по метода на дискретните елементи – EDEM Software. Програмният пакет включва модули за: изграждане на модели на обекти EDEM Creator, симулация на взаимодействие между много обекти EDEM Simulator, анализ на резултати, графики, отчитане на ъгли, брой мелещи тела, износване и др. EDEM Analyst.

С EDEM Creator може лесно и бързо да се създават модели на насипен материал. Предвидени са инструменти, с които лесно да се моделират формата, плътността и контакта на частиците. EDEM Simulator е средата, в която се конфигурира и симулира представянето на движението и взаимодействието на множество частици в поток (струя). Паралелната ефективност увеличава скоростта при работа с големи модели. EDEM Analyst предоставя инструменти за последваща обработка, анализ, визуализация и сваляне на симулационни данни. EDEM осигурява бърза 3D визуализация на системата за насипване на голям брой частици.

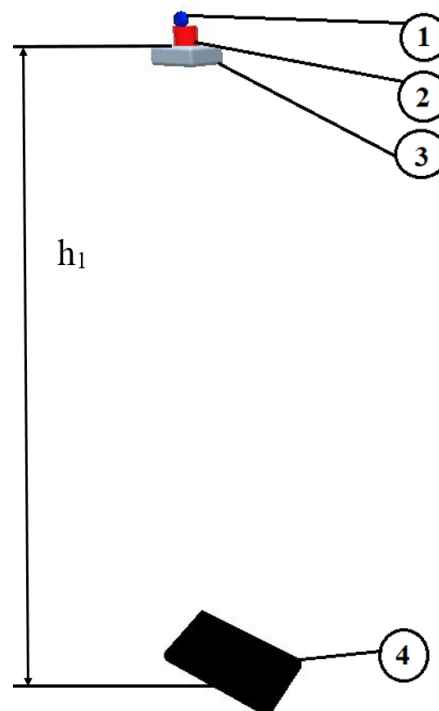
Приложения: за симулация на движение на мелещи среди в топкови мелници, движение на насипни материали, фармацевтика, автомобилостроене, тежка механизация и други.

Преди започване на симулирането, в софтуерния продукт е необходимо да се зададат входни параметри за частици (играещи ролята на сфера) и материал на оборудване.

При материала на частиците се задават входни параметри: размери, плътност, обем, маса, коефициент на възстановяване, коефициент на статично триене, коефициент на триене при търкаляне и др.

Зададените входни параметри за характеристиките на сферата и плочата са идентични, като тези, описани в таблица 1. В данните от таблица 2 е променен размерът до идеален - 120x60mm. В таблица 3, първоначалната височина, от която свободно пада сферата е зададена на 540mm. Данните за коефициенти на възстановяване, триене, триене при търкаляне са нанесени, спрямо опитно установените, при провеждането на експериментите [14].

Опитната установка за провеждане на експериментите е показана на фиг. 1. 3D принтираната сфера (1) се поставя в улей (2), след което застава на плоча (3). При премахване на плочата (3), сферата (1) пада свободно от височина  $h_1 = 540$  mm върху 3D принтираната плоча 4.



Фиг. 1. Схема на опитната установка.

Целта на симулационното моделиране е да се верифицира максимално получената височина на отскока след контакт с плочата, получен в софтуера спрямо височината на отскока при опитното установяване на коефициента на реституция.

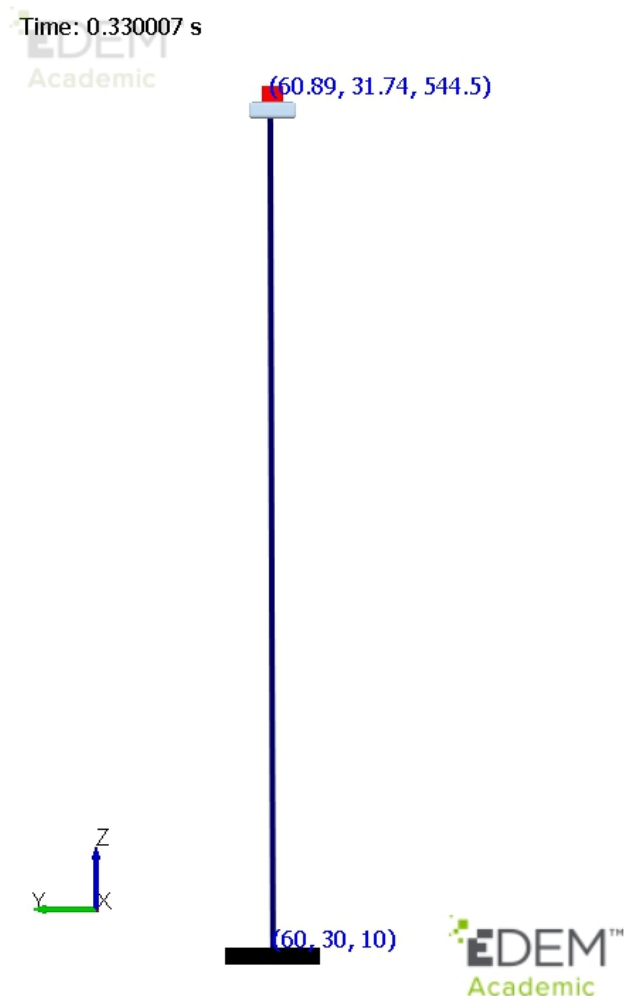
Софтуера има възможност за следене на сферата през цялото и движения, в координати XYZ. За начална точка на следенето е зададена сферата, а за крайна точка е зададена плочата.

При първоначалния момент, разстоянието между сферата и плочата е 544,5 mm, показано на фиг. 2. Разликата от 4,5 mm се дължи на факта, че софтуера взима центъра на сферата, която е с радиус 4.5 mm. При крайната точка за следене спрямо повърхност, ръчно е зададена горната повърхнина на плочата, описана в координатите  $X = 60$  (средата от дължината 120 mm),  $Y = 30$  (средата от ширината 60mm) и  $Z = 10$  – височината на плочата, която е 10 mm.

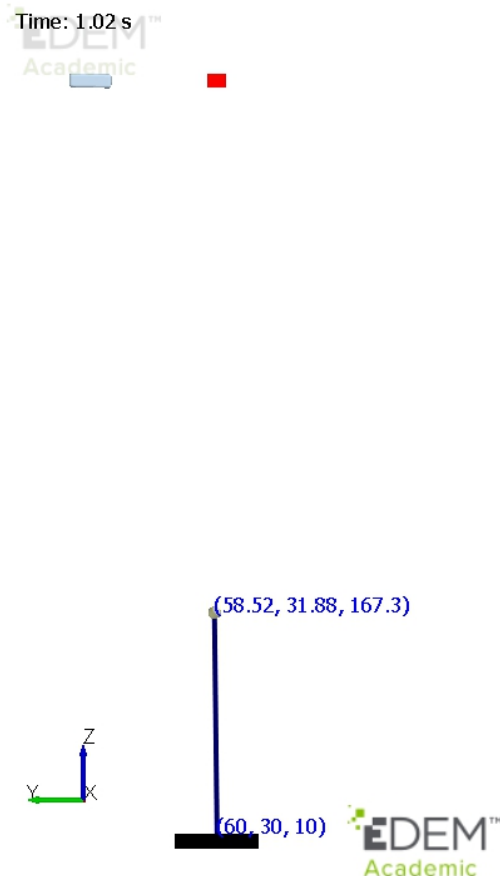
На фиг. 3 е показан моментът, в който сферата е на най-голямото разстояние от плочата, а именно 167,3 mm. В този момент сферата се намира в покой.

Допълнително, в софтуера е отчетена и скоростта на сферата, като в момента на отскока, преди отново да смени посоката скоростта е  $=0$  m/s.

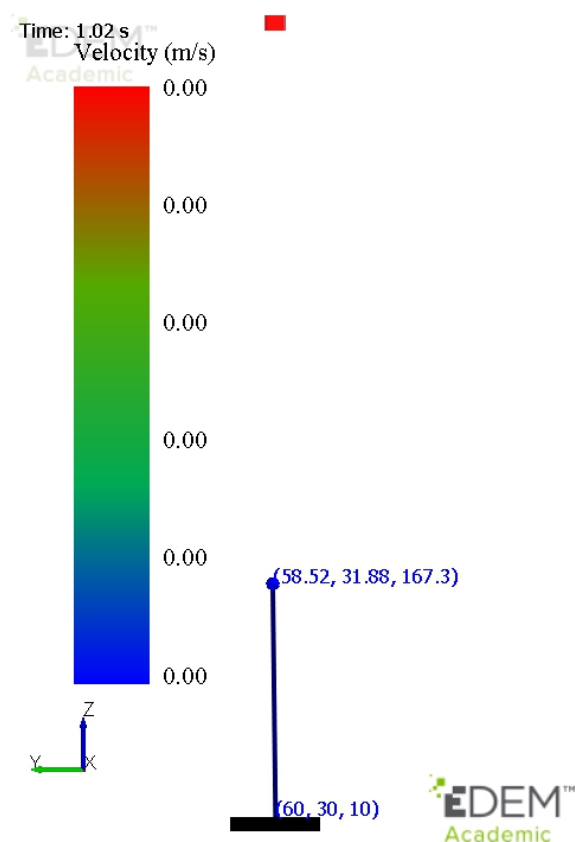
Максимално достигнатата скорост по време на симулационното моделиране е 3,19 m/s, показано в графиката на фиг. 5. Фиг. 6 показва динамиката на отскоците и затихването.



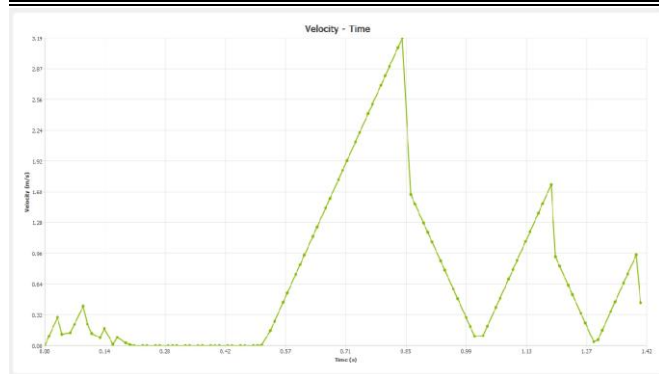
Фиг. 2. Първоначален момент от симулацията.



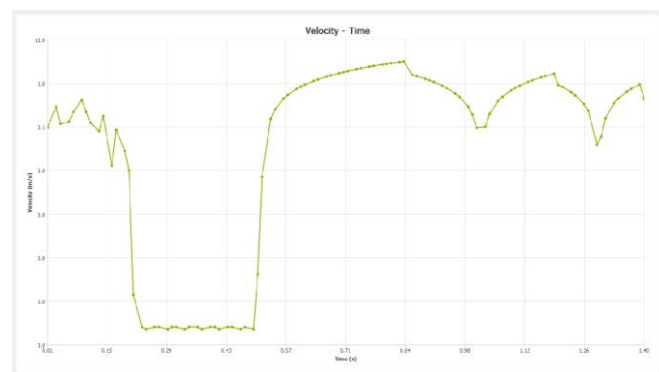
Фиг. 3. Разстояние на отскок.



Фиг. 4. Скорост в момента, преди сферата да смени посоката си.



Фиг. 5. Скорост при свободно падане на сфера.



Фиг. 6. Динамика на отскока при свободна падане на сфера.

## 5. Заключение

Получените резултати от симулационното моделиране се доближават до отчетените средни стойности при провеждането на опитните експерименти. Получената средна височина на отскок от експерименталното изследване е  $h_2 = 163,7$  mm. Отчетената височина на отскок от симулационното моделиране е 167,3 mm. От полученият резултат следва да се извади радиуса на сферата от 4,5 mm и се получава височина на отскок при симулационното моделиране от  $h_2 = 162,8$  mm.

Получените стойности при коефициент на реституция от проведеното експериментално изследване е  $e = 0.5495$ , а от симулационното моделиране е  $e = 0.5490$ .

При такава разлика може да се счита, че верификацията, получена при симулационното моделиране отговаря на опитно установения коефициент на възстановяване.

## Благодарности

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# Deposition of $\text{ZrO}_2$ thin films obtained by sol-gel method on monolithic composite with layered structure of Nb30Ti/Al

Vladimir Petkov, Mihaela Aleksandrova, Vladimir Blaskov

Bulgarian Academy of Sciences, Institute of Metal Science, Equipment, and Technologies with Center for Hydro- and Aerodynamics "Acad. A. Balevski", Shipchenski Prohod Blvd. 67, 1574 Sofia, Bulgaria, vladimir2pe@yahoo.com

**Abstract:** The present publication is aimed at obtaining a stable sol from  $\text{ZrO}_2$  stabilized with  $\text{Y}_2\text{O}_3$ . The substrate on which the layers are deposited was developed by a team from Russia and is intermetallic with a layered structure Nb30Ti/Al. Deposition was performed by spinning with a Spin Coater Ossila L2001A3. The deposition sequence of the layers lasts 30 seconds. The finished carrier is dried at  $120^\circ\text{C}$  in an oven. It is heated in a laboratory furnace at  $350^\circ\text{C}$ . The results were characterized by XRD and SEM analyzes.

**Key words:** SOL-GEL, LAYERS,  $\text{ZrO}_2$ ,  $\text{Y}_2\text{O}_3$ , DEPOSITION

## 1. Introduction

Undoped zirconia is a typical insulator with a wide band gap (5.1–7.8 eV) [1].  $\text{ZrO}_2$  is characterized by 3 polymorphic modifications: monoclinic, tetragonal and cubic. It has been proven that the individual phases are most often stable in the following temperature ranges: monoclinic up to  $1170^\circ\text{C}$ ; tetragonal in the range  $1170 - 2370^\circ\text{C}$ ; cubic at temperatures above  $2370^\circ\text{C}$ . By appropriate chemical or physical treatment, as well as by doping mentioned temperature ranges can be significantly changed. High temperature phases can be stabilized at room temperature by replacing.  $\text{ZrO}_2$  is characterized by 3 polymorphic modifications: monoclinic, tetragonal and cubic. It has been proven that the individual phases are most often stable in the following temperature ranges: monoclinic up to  $1170^\circ\text{C}$ ; tetragonal in the range  $1170 - 2370^\circ\text{C}$ ; cubic at temperatures above  $2370^\circ\text{C}$ . By appropriate chemical or physical treatment, as well as by subsidizing the temperature ranges that exist modifications can be significantly changed. High temperature phases can be stabilized at room temperature by replacing of Zr (up to 15%) with larger cations [2].

$\text{ZrO}_2$  thin films are widely used in various fields of human activity due to its outstanding properties: excellent mechanical strength, chemical and corrosion resistance, corrosion resistance coatings, high thermal stability, high wear resistance and tribological properties, gas sensors properties, remarkable optical properties, interesting photo-luminescence characteristics [3-11] etc. Unfortunately, when heating samples of undoped zirconium dioxide, the unwanted phase transition 1 genus (martensite type) takes place. Tetragonal modification - Monoclinic modification (in the range  $930-1220^\circ\text{C}$  by heating and  $950^\circ\text{C}$  by cooling) by cooling. [12] The difference in the volumes of the two phases reaches 4.5%, which leads to a significant reduction in the hardness of  $\text{ZrO}_2$  products and they are often unusable. It is especially true for the ceramic operating at high temperatures as well as in aggressive mediums. In this case the fracture of materials can be induced by small damages (pore, scratch, defects) [13, 14]. This serious temperature-dependent problem is overcome by appropriate doping of  $\text{ZrO}_2$  with oxides:  $\text{Y}_2\text{O}_3$ ,  $\text{CeO}_2$ ,  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{Gd}_2\text{O}_3$  and others [15] phase transformations. It is proved that bending strength and thermal shock resistance of the samples are significantly improved. That is why they are suitable for application in industry as extreme environments materials [16]. Special attention was paid to the doping with yttrium oxide [15-18] which leads not only to the enhancement of the mechanical strength (fracture toughness) but also to a series of other excellent physicochemical properties of the obtained samples [15-18]. The substitution of  $\text{Zr}^{4+}$  with  $\text{Y}^{3+}$  in the crystal lattice of zirconium dioxide forms 1 oxygen vacancy for every two Y atoms as a charge compensating mechanism [19]. Vacancies have been found to be essential for the functioning (operation) of gas sensor devices and solid oxide fuel cells [19-21]. Characteristic of  $\text{Y}_2\text{O}_3$  doped  $\text{ZrO}_2$  films is also their high

temperature stability. At a weight concentration of  $\text{Y}_2\text{O}_3$  relative to  $\text{ZrO}_2$  6-8%, thin layers are formed which are very suitable as thermal barrier coatings [22].

Thin films of  $\text{Y}_2\text{O}_3\text{-ZrO}_2$  stabilized are synthesized by a series of chemical and physical methods: sol-gel, electron-beam evaporation, atomic layer deposition, pulsed-laser deposition, magnetron sputtering, rf sputtering, etc. [23-28]. Of particular interest in obtaining these films is the use of the chemical sol-gel method, which is characterized by some of the following advantages [29-32]: (i) a cheap and simple method compared to other methods. simpler and cheaper than others), (ii) significant reduction of the synthesis and sintering temperatures as well as a possibility for operation at atmospheric pressure operation (iii) very fine (homogeneous) mixing of the components on micro- and macro-level (iv) synthesis of nanosized particles and structures (v) allows easy control of the thickness and porosity of the films (vi) synthesis of very thin films, which increases the adhesion of the film to the substrate (vii) preparation of a very large number of materials and compounds (viii) application of inorganic and organic precursors, etc. [29-32]. These advantages encouraged us to use sol-gel technology for films synthesis. [33-35]

## 2. Experimental part.

In the present work, sol-gel technology was used to obtain  $\text{Y}_2\text{O}_3$  doped  $\text{ZrO}_2$  thin films for sample coatings in order to improve the thermal and corrosion resistance of the material. The process includes the following stages: synthesis of stable sol; mixing of the zirconium sol with solutions of additives and organic binders; application of dip coating and spinning methods for deposition of the layers to different types of substrates; as well as drying and calcination of coatings. The synthesis of zirconium sol proceeds in the following sequence.

Zirconium oxalate sol was prepared in the following sequence:  $0.1\text{M ZrOCl}_2 \cdot 8\text{H}_2\text{O}$  (Alfa Aesar) was dissolved in 31 ml of absolute alcohol and carefully homogenized on a magnetic stirrer for 30 minutes at room temperature. As a hydrolysis process was observed, 0.8 ml of  $\text{HNO}_3$  was added slowly dropwise. The process continues until the solution is clear. 0.3 ml of acetyl acetone is added to stabilize the solution. Dissolve yttrium nitrate,  $\text{Y}(\text{NO}_3)_3$  (Alfa Aesar) in 0.3 ml  $\text{HNO}_3$ . Homogenize for 15 minutes. The yttrium nitrate solution is mixed with the stable zirconium sol at room temperature for 30 minutes.

The sample labeled Nb Ti/Al (20 - 12 mm) was pre-treated mechanically, after which the samples are immersed in a solution of  $\text{C}_2\text{H}_5\text{OH}$  in order to degrease the surface. Spin Coater Ossila L2001A3 applies five layers. The deposition of thin films involves

three steps. The spinning speed is 1300 rpm and the stay time in the sol is 30sec.

The deposited layers are dried in the dryer at 120°C. After that a heat treatment in a laboratory furnace Veb Electro Bad Frankenhausen L M 312.11 at 350°C and gradually controlled temperature is realized. The thermal treatment time is 10 hours.

### 3. Results and discussions

The results of the XRD analysis show that in sample 1 three phases are revealed -  $\text{Al}_2\text{O}_3$ ,  $\text{AlNb}_3$ ,  $\text{TiNbO}_4$ , while in sample 2-  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ ,  $\text{AlNb}_2$  are observed. In the second sample  $\text{ZrO}_2$  from the inorganic precursor  $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$  is in quantities that are in the range of sensitivity of X-ray phase analysis. Fig. 1

According to XRD data, sample 1 is characterized by the following peaks: 24.3, 26.8, 35, 35.6, 38.12, 38.33, 39.16, 41.58, 43.33, 45.83, 51.66, 54.58, 57.91, 60.17, 64.62, 68.4, 72.08, 74.58, 77.08, 77.2

Sample 2 is characterized by peaks: 25.62, 28.33, 32.08, 33.33, 35, 35.41, 37.1, 37.5, 37.92, 38.75, 40.42, 41.58, 42.08, 42.50, 43.42, 45.62, 52.5, 54.16, 57.5, 58.3, 59.58, 61.54, 61.77, 65.77, 66.66, 68.44, 68.66, 70.22, 71.11, 76.88, 77.33, 79.5. Fig. 2

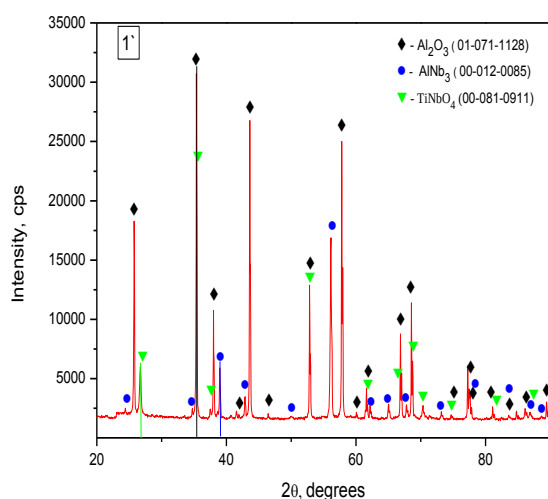


Fig.1 XRD of the sample 1

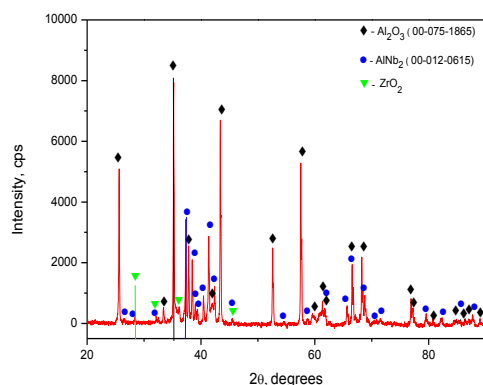


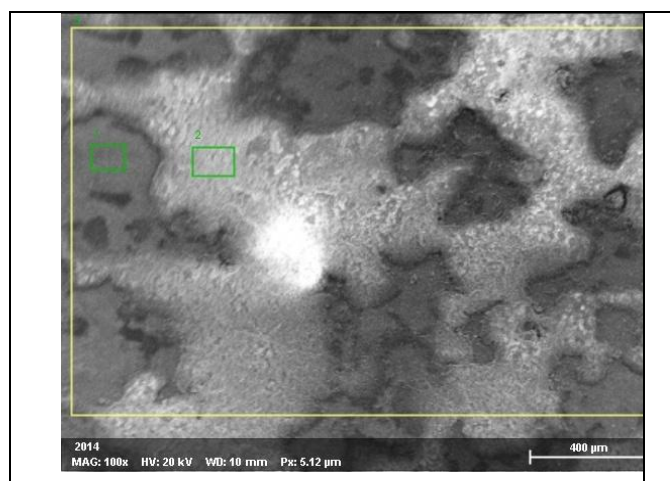
Fig.2 XRD of the sample 2

The study was performed using a scanning electron microscope "HIROX SH-5500P" with EDS system "QUANTAX 100 Advanced" with the following parameters: SEM HIROX SH-5500P. Specifications: Window 10 compatible, 5-axis fully

motorized stage, Stage tilt from - 45 to + 90 degrees, Maximum sample size: diameter = 80 mm, thickness = 40 mm, Maximal beam resolution: 5 nm (in SE mode), Maximal magnification: x 150 000, 4 variable apertures and continuous variable spot size, Two detectors: SE and BSE, Accelerating voltage: 1 to 30 kV Integrated EDS – QUANTAX 100 Advanced – Bruker. Integrated silicon drift detector (SDD) – nitrogen free, Detection from Boron to Uranium, Qualitative and Quantitative analysis, Multi-point and area spectrum acquisition, Line scan profile, X-ray mapping.

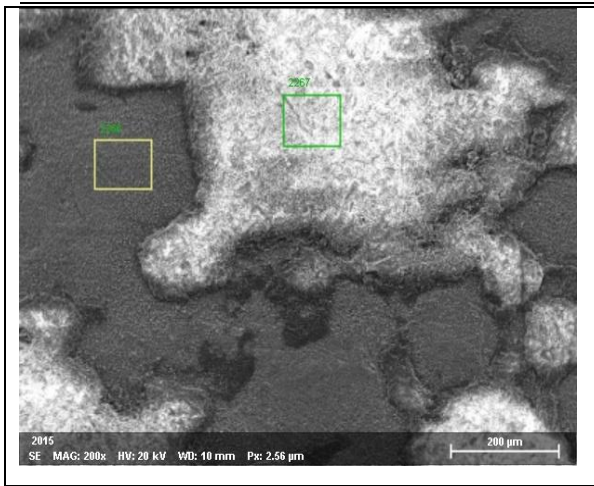
The samples were tested in the "secondary electrons" mode at an accelerating voltage of 20 kV. To characterize the microstructure, zone analyzes were performed at different magnifications at different points of the samples. In sample N1, two separate phases can be distinguished, namely: (1) Ti-Nb-O-Al and (2) Al-O-Si-Zr. The phases are shown in the photo below, and the results of chem. analysis are presented in the tables below the photos number 2, 3, 4.

Fig. 4 shows the simultaneous presence of 2 areas - well covered deposited layer, as well as uncovered. In Fig. 4 well covered area is very small and predominantly is uncovered. In fig 3 only the uncovered area exist. The sample shown in Fig. 3 reveals the presence of Ti, Nb, Al, O, Y, Na, Fe, Si. The sample of Fig. 5 is characterized by the presence of Ti, Nb, Al, O, Zr, Y, Si. In the sample of Fig.3 the presence of Ti, Nb, Al, O, Zr, Y, Si, Fe is established.



| 1       |         |       |          |                |          |                             |
|---------|---------|-------|----------|----------------|----------|-----------------------------|
| Element | At. No. | Netto | Mass [%] | Mass Norm. [%] | Atom [%] | abs. error [%]<br>(1 sigma) |
| Ti      | 22      | 13341 | 42.13    | 39.23          | 32.97    | 1.26                        |
| Nb      | 41      | 10014 | 39.39    | 36.67          | 15.89    | 1.56                        |
| O       | 8       | 588   | 17.29    | 16.10          | 40.50    | 4.61                        |
| Al      | 13      | 2242  | 7.11     | 6.62           | 9.88     | 0.43                        |
| Zr      | 40      | 256   | 1.07     | 1.00           | 0.44     | 0.11                        |
| Y       | 39      | 43    | 0.19     | 0.18           | 0.08     | 0.06                        |
| Si      | 14      | 63    | 0.15     | 0.14           | 0.20     | 0.05                        |
| Fe      | 26      | 14    | 0.07     | 0.06           | 0.04     | 0.04                        |
|         |         | Sum   | 107.40   | 100.00         | 100.00   |                             |

Fig.3 SEM image of the monolithic composite Nb30Ti/Al with  $\text{ZrO}_2$  thin films



1

| Element | At. No. | Netto | Mass [%] | Mass Norm. [%] | Atom [%] | abs. error [%]<br>(1 sigma) | rel. error [%]<br>(1 sigma) |
|---------|---------|-------|----------|----------------|----------|-----------------------------|-----------------------------|
| Ti      | 22      | 12913 | 42.42    | 40.64          | 35.47    | 1.27                        | 3.00                        |
| Nb      | 41      | 10233 | 39.54    | 37.88          | 17.03    | 1.56                        | 3.95                        |
| O       | 8       | 511   | 15.40    | 14.75          | 38.52    | 4.29                        | 27.85                       |
| Al      | 13      | 1714  | 5.52     | 5.29           | 8.19     | 0.35                        | 6.36                        |
| Zr      | 40      | 282   | 1.16     | 1.11           | 0.51     | 0.12                        | 10.23                       |
| Y       | 39      | 51    | 0.22     | 0.21           | 0.10     | 0.06                        | 27.48                       |
| Si      | 14      | 54    | 0.13     | 0.12           | 0.18     | 0.05                        | 35.84                       |
| Fe      | 26      | 0     | 0.00     | 0.00           | 0.00     | 0.00                        | 0.64                        |
|         |         | Sum   | 104.38   | 100.00         | 100.00   |                             |                             |

*Fig.5 SEM image of the monolithic composite Nb30Ti/Al with ZrO<sub>2</sub> thin films*

#### 4. Conclusion

In this work, the synthesis of stable yttrium doped ZrO<sub>2</sub> sol was shown. Coating by spinning (rotation) and characterization of results by XRD and SEM analyzes were realized. The thermal treatment at 350°C was reached after 1 hour and a delay of 10 hours were implemented in order to achieve the desired result. The results of the SEM analysis show in some places a well-deposited coating, while in some places there are not well-covered parts due to the uneven treatment of the substrates.

#### Acknowledgement

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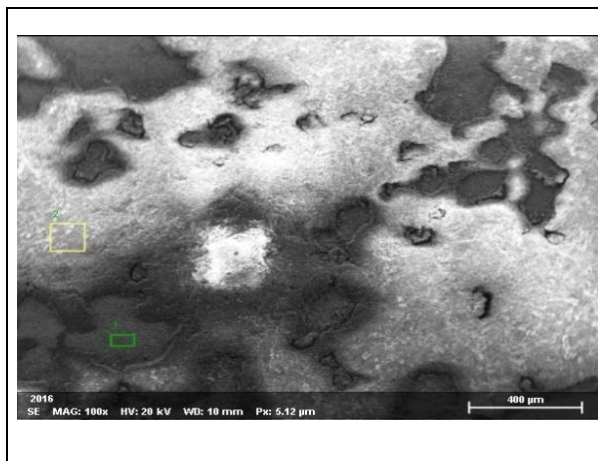
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| Element | At. No. | Netto | Mass [%] | Mass Norm. [%] | Atom [%] | abs. error [%]<br>(1 sigma) | rel. error [%]<br>(1 sigma) |
|---------|---------|-------|----------|----------------|----------|-----------------------------|-----------------------------|
| Ti      | 22      | 8792  | 43.38    | 42.24          | 38.27    | 1.34                        | 3.08                        |
| Nb      | 41      | 7278  | 40.57    | 39.50          | 18.45    | 1.64                        | 4.03                        |
| O       | 8       | 293   | 13.24    | 12.90          | 34.97    | 4.45                        | 33.57                       |
| Al      | 13      | 852   | 4.28     | 4.17           | 6.71     | 0.31                        | 7.32                        |
| Na      | 11      | 55    | 0.66     | 0.64           | 1.21     | 0.14                        | 21.80                       |
| Y       | 39      | 54    | 0.34     | 0.33           | 0.16     | 0.08                        | 23.04                       |
| Fe      | 26      | 24    | 0.16     | 0.15           | 0.12     | 0.06                        | 37.30                       |
| Si      | 14      | 21    | 0.07     | 0.07           | 0.11     | 0.04                        | 59.39                       |
| Zr      | 40      | 0     | 0.00     | 0.00           | 0.00     | 0.00                        | 1.79                        |
|         |         | Sum   | 102.70   | 100.00         | 100.00   |                             |                             |

*Fig.4 SEM image of the monolithic composite Nb30Ti/Al with ZrO<sub>2</sub> thin films*



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# Technology for production of reduced yellow paving stones on the basis of modified marl clay from the Alexandra quarry, the village of Lovets, Bulgaria (Part 1)

Gergana Mutafchieva, Lyuben Lakov, Mihaela Aleksandrova, Gabriel Peev

Bulgarian Academy of Sciences, Institute of Metal Science, Equipment, and Technologies with Center for Hydro- and Aerodynamics "Acad. A. Balevski", Shipchenski Prohod Blvd. 67, 1574 Sofia, Bulgaria, cl.creativeline@gmail.com

**Abstract:** Due to the specifics of marl clays, research to date has shown the need to stabilize these clays with respect to their critical melting point. The research is based on a previous analysis of pure raw material from marl clay, located in the area of the village of Lovets, which is modified by various additives. On the one hand, the modification is used to stabilize the ceramic mass and on the other hand, by adding various agents, the aim is to lighten the composition and obtain the required color pattern. For the purposes of production, the experimental part includes laboratory development of a scale model of yellow paving stone. The development presents the entire process of creating instrumental equipment – model making and matrix construction, and molding of finished products - pressing process for paving stones production from modified marl clay. The technological cycle that can be implemented in the real conditions for yellow paving stones production will rely on the present study.

**Keywords:** PRESSING OF CERAMIC PRODUCTS, YELLOW PAVING STONES, MODIFIED MARL CLAYS.

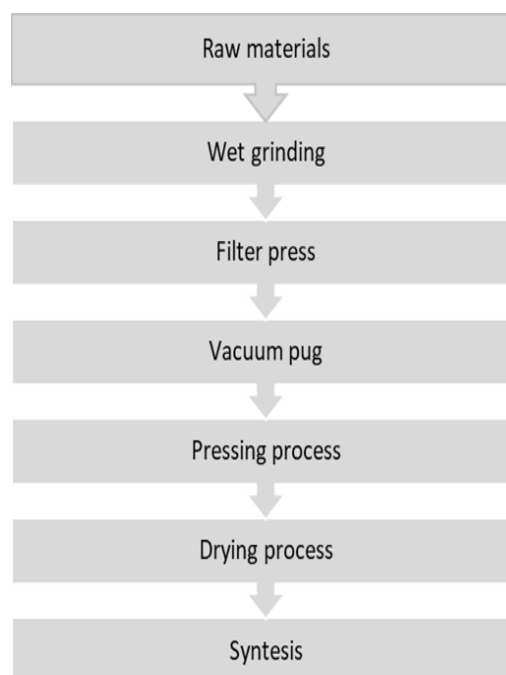
## 1. Introduction

The production of yellow paving stones is associated with a laborious and complex cycle in terms of both the technological process and the raw material. This is mainly due that the finished products have to meet specific physical and mechanical parameters. These requirements should be corresponded to the European standard EN 1344: 2013, which has the status of BDS from 2014-03-17. Requirements include indicators such as compressive strength, bending strength, high wear resistance, abrasion resistance, slip/skid resistance, transverse breaking load, minimum unit value, freeze/thaw resistance, high density, and low water absorption of the composition used, and last but not least color characteristics [6]. Studies show that marl clays are able to meet these parameters, but due to their chemical composition, they need to be modified with additives in order to increase their melting point and stabilize the glass phase at the final temperature. However, there are not many deposits in Bulgaria that meet these conditions and are available not only for experimental research but also for production purposes. Research so far shows that a suitable quarry is located in the Popovo region, but due to the cessation of production in the region, access to the field is limited [7]. Its necessitated new studies of suitable marl clay, which was studied as a raw material from the area of the village of Lovets, Shumen district. The analyzes showed appropriate results for the development of the material by the addition of modifying agents. Due to the indicators of the mass used, the development includes a laboratory technological process in the production of reduced yellow pavers. This covers the entire production cycle from the quarry to the final product, on the basis of which an adequate and analogous structural process for the production of exterior paving from yellow pavers will be created. The technological development includes the production of a model - gypsum prototype of paving stone in scale 1: 2 with dimensions 117 x 61 x 46 mm.; production of a matrix form for pressing ceramic pavers - construction of a metal frame with a mold of polymer-modified gypsum mixture [8]; preparation and processing of modified semi-dry marl clay - grinding, mixing, extrusion and vacuuming of the raw material with the addition of fireclay, kaolin K2, glass,  $ZrO_2$ , and  $ZnO$ ; molding of ceramic pavers - pressing production of semi-dry ceramic mass; drying and firing of the formed pavers - modes of drying, heating and temperature synthesis with isothermal retention in several main temperature processes [2-3].

## 2. Experiments

The research part presents an experimental complete process for the production of scaled ceramic products for exterior flooring - yellow pavers, from quarry to finished synthesized product. The production cycle begins with the preparation of raw materials and obtaining the necessary plasticity of the mass for pressing ceramic pavers. The main components are fireclay from fired marl, clay, and kaolin as the plasticity of the mass is determined by the ratio of

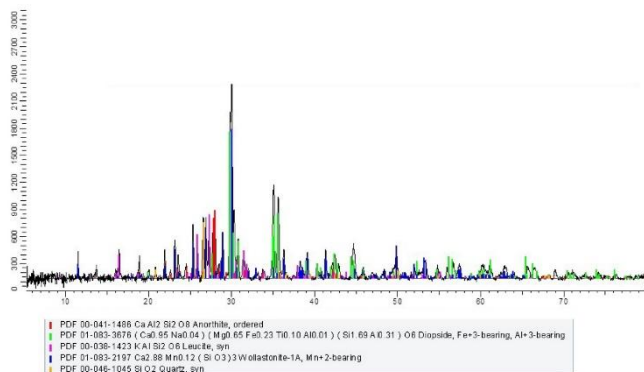
fireclay fraction to clay substance, and residual moisture in the mass after extrusion is 20-25%. A scheme of the production process from mass preparation to temperature synthesis is presented in fig. 1. The experimental part develops the already studied indicators of marl clay from the village of Lovets, presented at the VI International Scientific Conference Industry 4.0, held from 08 to 11.12.2021. According to the obtained data, the studied marl clay is from group III (acidic) with content below 15%  $Al_2O_3$  high carbonate, the content of alkaline earth oxides over 17 - 18% and high content over 3%  $Fe_2O_3$ . The total amount of alkaline earth oxides is 21.82% by weight, according to the classification of Sitin, which characterizes it with a relatively narrow sintering interval. Therefore, it was necessary to modify the marl we used in order to stabilize the material at the final temperature. Table 1 shows the chemical analysis of a sample, which in X-ray phase analysis gives suitable peturgical phases anorthite  $CaAl_2Si_2O_7$  and diopside  $MgCaSi_2O_6$ , which is presented in Figure 2 [1,4,5].



**Fig. 1.** Scheme of technological process for the production of reduced ceramic blocks for external flooring from mass preparation to the synthesis of the finished product.

**Table 1.** Quantitative analysis of the content of oxides in a synthesized sample of marl clay from the village of Lovets.

| Composition                    | Quantity% |
|--------------------------------|-----------|
| Na <sub>2</sub> O              | 0.7       |
| MgO                            | 2.24      |
| Al <sub>2</sub> O <sub>3</sub> | 11.36     |
| SiO <sub>2</sub>               | 35.19     |
| P <sub>2</sub> O <sub>5</sub>  | 0.1       |
| K <sub>2</sub> O               | 2         |
| CaO                            | 19.58     |
| TiO <sub>2</sub>               | 0.47      |
| MnO                            | 0.11      |
| Fe <sub>2</sub> O <sub>3</sub> | 4.85      |
| LOI                            | 23.21     |
|                                | 99.82     |
| Cu                             | 0.06      |
| Zn                             | 0.073     |
| Sr                             | 0.053     |

**Fig. 2.** XRD of the sample synthesized at 1140°C with the presence of peturgical phases anorthite and diopside.

To modify and stabilize the ceramic mass is experimented with test specimens in different proportions of fireclay and clay, as well as brightening additives presented in the table. 2. The best indicators presented correspond to sample XIX in the ratio by weight of clay - fireclay 30% - 60% and the addition of kaolin 10%. The reduction of the fireclay fraction increases the plasticity of the mass, which makes it suitable for the molding process – pressing process. The residual humidity after the vacuum extruder is 20-25%. The addition of kaolin stabilizes the liquid phase and brightens the mass to obtain a color corresponding to the pattern. The X-ray phase analysis of the synthesized sample shows the presence of peturgical phases (Fig. 1) as the temperature regime is consistent with DTA and is presented in the table. 3 [7].

**Table 2.** The percentage ratio of clay - fireclay and modifying additives of marl clay from the Alexandra quarry, the village of Lovets.

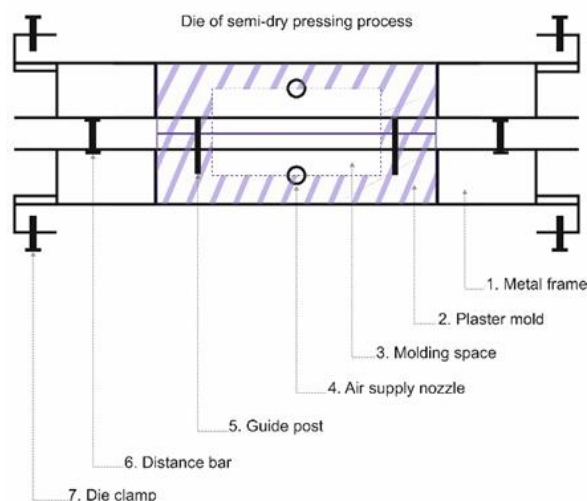
| №    | Fire clay | Marl | ZrO <sub>2</sub> | ZnO | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> 2SiO <sub>2</sub> 2H <sub>2</sub> O |
|------|-----------|------|------------------|-----|------------------|--------------------------------------------------------------------|
| II   | 55        | 40   | 5                | -   | -                | -                                                                  |
| III  | 55        | 40   | -                | 5   | -                | -                                                                  |
| VI   | 50        | 40   | 5                | -   | 5                | -                                                                  |
| VII  | 50        | 40   | -                | 5   | 5                | -                                                                  |
| XIII | 35        | 55   | -                | -   | 10               | -                                                                  |
| XIV  | 35        | 60   | -                | -   | 5                | -                                                                  |
| XIX  | 30        | 60   | -                | -   | -                | 10                                                                 |
| XX   | 30        | 55   | -                | -   | -                | 15                                                                 |

**Table 3.** Temperature regime for synthesizing ceramic blocks with temperature retention at several points.

| Temperature °C | Retention in min. |
|----------------|-------------------|
| 150            | 60                |
| 650            | 60                |
| 840            | 180               |
| 1140           | 180               |

The technological process for obtaining form for pressing ceramic blocks includes a model making and an author's applied development of two-part matrix construction making, which represents a metal frame and a gypsum mold. The model is made on a scale of 1 to 2 compared to the original ceramic block and shown in figure 3, figure 4 presents a scheme of the matrix structure.

**Fig. 3.** Gypsum model of a reduced block for outdoor flooring on a scale of 1:2 compared to the original paving block - yellow paving stone.



**Fig. 4.** Scheme of the matrix construction for pressing process of reduced ceramic blocks for paving.

The gypsum model is made with an expansion of 14% compared to the compressibility of the semi-dry mass used so that after the temperature synthesis the ceramic block retains its scale ratio to the original one. Once the model is ready, the construction of the matrix is started to make. The first stage includes the production of metal frames according to the pressing facility used: Russian hydraulic press П-125 is used. Each of the two metal frames has an enclosed area in the middle where the model is put and the gypsum mold will be cast, and each part of the mold is cast consequently. Metal frames include: clamping elements that fix each structural part (metal frame + gypsum mold) to the working plates of the press (note 7. Fig. 4); fixing guides of the two metal frames (note 5. Fig. 4); spacer distance, which provide the necessary distance and centering between the working surfaces of the matrix (note 6. Fig. 4); openings with mounted nozzle for the supply of compressed air in order to form microscopic capillaries in the gypsum structure, which helps faster and easier separation of the molded product from the matrix (note 4. Fig. 4). Each structural part contains a gypsum form - a mold (note 2. Fig. 4.) and a form-forming space (note 3. Fig. 4.). The gypsum composition used is a polymer modified mixture KERADUR S4P in the ratio of parts by weight of gypsum to water 3 to 1 [8]. The volume of the required amount of gypsum powder and water is calculated by the formula:

$$\frac{v}{2} = X_1$$

$$X_2 = X_1 \cdot 3$$

where  $x_1$  is the required amount of water in liters;  $x_2$  is the required amount of gypsum in kg,  $v$  is the volume of the mold forming space.

The liquid solution of the polymer-modified gypsum mixture is being homogenized by continuous mechanical stirring until complete dissolution of the polymerizing agent in the order of 20 to 30 minutes. The formation of capillaries in the cast by supplying compressed air begins when the gypsum is heated to a temperature of 37 ° C. The air supply starts at 0.5 bar/min to 3 atmospheres, then the supply increases by 0.5 bar / 30 sec to 5 atmospheres. The retention during the continuous supply of 5 atmospheres of compressed air holds to a total time of 40 minutes. During that time the maximum water content in the gypsum mold was evaporated and the formation of the capillary principle was obtained. The whole production process for the matrix construction is presented in fig. 5.



**Fig. 5.** Technological process in the production of a matrix structure for pressing ceramic products – reduced paving stones from semi-dry modified marl clay.

### 3. Conclusion

The experimental development shows very good results both in terms of the technological process and in terms of indicators of the marl clay used. In summary, the results of the present study can be defined as comprehensive coverage of all stages of the production cycle - from mass preparation to temperature synthesis of the final product. This allows control at each stage of the process, which should be used in the production of real-sized blocks for paving pavement through the already developed technological principle. The performed analyzes will be used for the implementation of the technological framework in production.

### Acknowledgement

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# Innovative technologies for the production of high-oxide corundum and boron carbide ceramics

Lyuben Lakov, Mihaela Aleksandrova, Gergana Mutafchieva, Gabriel Peev

Bulgarian Academy of Sciences, Institute of Metal Science, Equipment and Technologies with Hydro- and Aerodynamics Centre "Acad. A. Balevski", Shipchenski Prohod Blvd. 67, 1574 Sofia, Bulgaria, e-mail: mihaela.krasimirova@mail.bg

**Abstract:** In this publication, the authors present an in-depth justification on the technology for the production of high-oxide ceramics. In-depth research on international markets on the supply and use of raw materials is presented. The various design developments related to the ways of pressing products are described. The physico-mechanical parameters of the synthesized corundum and boron-ceramic ceramics were also studied.

**Keywords:** CORUNDUM CERAMIC, BORON CARBIDE CERAMIC, FORMING METHODS

## I. Introduction

### High-oxide corundum and boron carbide ceramics

The beginning of the production and use of  $\text{Al}_2\text{O}_3$ -based ceramics is considered to be 1905. At present, the corundum ceramics occupies the largest share of all other technical ceramics produced globally - about 40 %. Its application is for refractory products, in the vacuum technique, as constructive elements as well as for chemically resistant and electrically insulating products. Over the past decades due to its low cost, the high-oxide corundum ceramic products have been applied for protection against high-speed kinetic and cumulative impacts on moving and stationary objects, as well as for the production of multilayer ceramic panels for personal protective armor vests. In general, the corundum ceramics plays an important role in modern science and technology. Areas of its application are constantly expanding as its properties are continually improving through the use of new production technologies.

Corundum ceramics is a densely sintered polycrystalline structure. The feedstock is  $\alpha\text{-Al}_2\text{O}_3$  (corundum). A basic requirement for the raw material is its high purity. The degree and constancy of impurities predetermines the purpose of the ceramics. Another requirement is a well defined sedimentary composition (finely milled feedstock), obtained without contamination during milling. Alumina powders with close particle size (mono fraction powders) are required for preparation of high-alumina corundum ceramics intended for special applications. In these cases chemical methods are used - chemical coagulation or sol-gel method. These methods are characterized by the advantage that they achieve a high dispersion, purity and activity of the ceramic powders, as well as homogeneity of the constituent masses, and hence of the resulting ceramics. Using these methods sintered materials can be obtained easily at low temperature.

The direct use of corundum powder as a raw material for ceramics production is inappropriate since it is non-plastic. In order to obtain a product, the ceramic powder must acquire bonding properties, which is achieved by adding organic composition as a technological bond. Substances that act as a temporary bonds are called plasticizers. They give formability to the ceramic mass for fabrication of products and the mechanical strength of the material prior to firing and in the process of firing the bond burns without residue. Most often these are water-soluble organic binders such as polyvinyl alcohol and the like. In order to improve the wetting of the mineral components and to increase the surface activity of the organic bond, a small percent of surfactant is added - about 0.5 wt % of the amount of corundum. The most common surfactant is oleic acid ( $\text{C}_{17}\text{H}_{33}\text{COOH}$ ).

Pure boron carbide products have an extremely wide range of physical, chemical and mechanical properties, which make them unmatched in many areas of technology. Their high hardness (the third after those of the diamond and cubic boron nitride), modulus of elasticity, sound velocity, and low relative weight allows it to occupy an important place in the construction of multilayer ceramic-polymer systems for protection against high-speed kinetics and cumulative impacts.

In nuclear power plants, cylindrical profiles of boron carbide are used to regulate the course of nuclear reactions because they contain boron atoms  $^{10}\text{B}$  in which the capture of neutrons is extremely high.

Nozzles for sandblasters, cutting and grinding tools and more are also produced from boron carbide.

Despite of the extremely high chemical resistance of boron carbide products to various aggressive environments, due to technological difficulties in the manufacturing of large-scale products for the chemical industry, they are not yet widely used.

Currently, studies on the synthesis of pure boron carbide and the production of articles based on it have received much attention. Particular attention is paid to improving the method using hot pressing.

At present, a lot of attention is played in various smaller and larger countries to the research on the synthesis of pure boron carbide and the manufacture of articles on this basis. Particular attention is paid to refining the method of hot pressing.

### 1. Requirements for corundum masses, preparation of press powders, forming methods of preforms and development stages

As noted, high alumina corundum ceramic masses are non-plastic and because of this their masses can be thermoplastic (paraffin bonding used) or semi-dry (if polyvinyl alcohol with surfactant - oleic acid as bonding is used). Prior to their production, the corundum (most commonly electrically melted) is grinded. Grinding is often wet. Afterwards, the powder, with the required granulometric and sedimentary composition, is sieved (most often with vibratory sieves) and the iron impurities are removed by magnetic separation. The fractions are blended, plasticizer is added as per the technology of the respective product and the mass is homogenized. The material should be very well compacted after pressing. This is why the grain size distribution is essential to provide a maximum packing. Surfactants are also added which, in addition to being adhesive additive, play the role of a grain sealer, reducing friction between them and the mold. These additives are called lubricants. Prior to the addition of plasticizers and surfactants, the masses are crumbly, easily spreadable in layers with different density and do not "flow" well when filling the molds if not properly prepared. All this is avoided as the masses are aggregated into larger granules of approximately the same size and with round shape, which are durable, do not stick together and fill the mold well. The main method used for this purpose is spray drying. It is done after an initial homogenous mixing of the corundum powder with the organic binder and its humidification. The resulting material is injected in the direction of its movement or against it (counter-current) in the so-called spray dryers.

The main idea in developing the present technology is to achieve specific composition and specific technical conditions for the production of corundum ballistic ceramics through the study of a number of corundum compositions. For this purpose the most up-to-date information was obtained on the globally used ballistic ceramics and in particular of that based on corundum. A number of technological issues related to the preparation of corundum

compositions with a certain particle size were studied: the addition of different type and quantity of modifiers, the granulation and the molding methods - compacting by a hydraulic press, thermoplastic casting, slip casting, isostatic pressing, detonation pressing. The issue of the production of corundum ballistic ceramics was solved by the development of the technology.

First, seven compositions with different  $\text{Al}_2\text{O}_3$  content were prepared and experimented under laboratory conditions. The most promising was determined. Mixtures of the various corundum compositions according to the applied molding method were prepared. The densities, the temperature mode of firing, respectively the coefficients of shrinkage after firing, of the semi-finished product after molding were determined in order to produce tools for the final products - square and rectangular plates with different thickness. Various physicochemical properties, such as final density, speed of sound passage, Poisson coefficient, bending strength, modulus of elasticity, relative share of the absorbed kinetic energy, hardness, etc. were examined of the various laboratory samples.

The technological process of ballistic corundum ceramics production includes the following operations:

1. Use of pure raw materials obtained by chemical methods. In this case  $\text{Al}_2\text{O}_3$  powder.
2. Dosage of the raw materials and weight mixing with additives.
3. Preparation of a ceramic granulate, thermoplastic masses or dispersed aqueous colloid system.
4. Forming of the obtained products.
5. Drying and firing of the preform bodies.
6. Mechanical processing if needed.
7. Quality control of the obtained products.

## 2. Standards referring to corundum ceramics properties

The object of the technology is to achieve maximum protection of the corundum products, in particular plates with a maximum  $\text{Al}_2\text{O}_3$  content with, used as a front part of a multilayer ceramic-polymer hard plate. The chemical composition of the ceramics corresponds well with the corundum ceramic used for electro technical purposes described in the International Standards "IEC" of the European Committee CENELEC. With the introduction of unified European regulations, these standards are already listed as: EN60672-1 "Specification for Ceramic and Glass Insulation and Materials, Part 1": Definitions and classification and EN60672-3 "Specification for Ceramic and Glass Insulation and Materials, Part 3": Materials.

As an example, we list some of the most important indicators to be met by the best corundum ceramics containing  $\text{Al}_2\text{O}_3 > 99\%$  of the group C799, namely:

- porosity - 0.0%,
- density  $> 3.7 \text{ g/cm}^3$ ,
- bending strength  $> 300 \text{ MPa}$ ,
- elasticity modulus 380 GPa.

These values are minimal, each manufacturer guarantees in his technical specification additional and possibly better properties for his products.

## 3. Standards related to the control of material characteristics (in this case the corundum ceramic tiles), defining the protective properties of the final product.

There are various methods developed to determine the material's properties, such as density, tensile bending and compression strength, hardness, impact strength, elasticity modulus, etc. Applicable for these purposes are EN ISO12680, EN658, ENV14186, EN1094, EN993 series standards. There are non-standardized methods, known in the literature, to make an indirect

assessment of the protective properties of the material under certain test conditions.

## 4. Market study at home and abroad for supplying the necessary raw materials for regular production.

The market study for the supply of the necessary raw materials is consistent with the stages of production of high alumina corundum ceramics, namely in the following order:

4.1. The worldwide suppliers of 99.99% pure aluminum oxide with a defined sedimentary and granular composition or of granules with a defined chemical composition for dry pressing, are as follows:

- company Martinswerk - Germany,
- company Nabaltek - Germany,
- company Alcoa - USA.

For this purpose, inquiries were made to the respective suppliers, and we received their offers as follows:

- Nabaltek has submitted a quotation for pure aluminum oxide as well as ready-made granulate brand KMS96 and brand NM9620;
- Martinswerk submitted an offer for MR70 / S ready-made granulate.

4.2. Preparation of alumina oxide press powder for isostatic pressing.

The market for suppliers of alumina with a 99 % content and average grain size of 5 microns was studied.

We received offers from the suppliers quoted in item 5.1, as follows:

Nabaltek has provided an offer for alumina oxide brand ND313.

Martinswerk provided an offer for alumina oxide brand MR52.

To improve the physico-mechanical properties and to reduce the sintering temperature, a market study of suppliers of magnesium and other oxides was required. We have sent a request for offer to the firm May Chem from the Netherlands.

For improvement of the parameters of the finished product related to liquefiers and plasticizers, a request for offer was sent to the companies Zchimmer and Schwarz from Germany. The German company has offered the following brands: Dolapix PC67 and OptaPix PAF2.

## 5. Development of semi-dry, impulse and detonation pressing molds.

### 1.1. Development of semi-dry pressing mold

The study concerns a press tool for hydraulic press DORST TPA 50/2 designed for pressing ceramic plates made of alumina-based ceramic material with dimensions 50 x 50 x 8 mm and 50 x 50 x 10 mm.

#### 5.1.1. Description of the equipment

It is an instrument consisting of a mold, a top and a bottom punch and connecting elements. The following materials are defined for making the basic elements of the instrument taking into account the specifics of the ceramics dry pressing.

- mold - body of hard metal - Ceratizit H40S/HIP,
- top and bottom punch - steel X12 thermally treated to the necessary hardness,

- connecting elements – steel C45.

The dimensions of the connecting elements are tailored with the DORST TPA 50/2 automatic press adapter. The dimensions of the molding parts are defined as follows: by performing a technological trial using the same press at similar technological parameters but with another pressing tool to determine the compressibility coefficient of the ceramic granulate. This coefficient determines the dimensions of the raw body or of the shaping parts of the finished bodies.

## 1.2. Development of equipment for impulse and blast compression.

These equipment was not designed for a specific product of definite dimensions but only for model samples to determine the effect of this compression on the physico-chemical and mechanical characteristics of the finished product without pursuing a long-term use.

## 6. Manufacture of proper size rubber molds for isostatic (hydrostatic) pressing.

The manufactured rubber molds are for isostatic pressing of 50 x 50 x 8 mm and 50 x 50 x 10 mm preform ceramic plates. This is done by the following scheme:

6.1. Calculation of the dimensions of the green plate of the respective composition with a compression coefficient of  $\sim 1.166$ , which means that the preliminary dimensions should be 58.3 x 58.3 x 9.32 mm and 58.3 x 58.3 x 11.66 mm respectively.

### 6.2 Calculation of the mold

#### a) Internal diameter of the mold

Considering that the coefficient of compaction at 1,300 bar is  $K_{com} \approx 1.3$  and that the square plates dimensions of 58.3 x 58.3 x 9.32 mm and 58.3 x 58.3 x 11.66 mm may fit in a circle with a diameter 90 mm, the internal diameter of the mold must be  $\approx 90 \times 1.3 = 117$  mm. Considering that a further 10-13 mm are required for the preform to become a right-sized cylinder after machining, the required internal diameter of the mold must be 130 mm.

#### b) Length of the mold

Thicknesses of one plates are 9.3 mm and 11.66 mm respectively. Four mm for preliminary machining and 5 mm for cutting of a single plate (this is the thickness of the turning knife) should be added to these dimensions. The required length of the green body becomes at least 230 mm for 12 plates 8 mm thick and 10 plates 10 mm thick. Considering that the coefficient of compaction is approximately 1.3 for isostatic pressing, and the sleeve is closed with rubber stoppers at 40 mm depth, the required length will be 380 mm. The thickness of the mold's wall is 10 mm. Two molds are needed for full loading of the isostatic press.

## 7. Injection molding

7.1. Preparation of a technological binder and a thermoplastic slip. Forming of details.

5.65% bees wax is added to 1 kg of hard paraffin. The two materials are placed in a vessel and heated in water bath at  $80 \div 90^\circ \text{C}$  to melt and are homogenized. The resulting binder is poured into metal molds and, after cooling and curing, is stored for further application for preparation of thermoplastic slip. This is done by placing in a heated water bath at  $80 \div 90^\circ \text{C}$  the preliminary cleaned from the binder quantity (16.5%) and waiting for it to melt.

The alumina mixture is added in small portions under continuous stirring with a propeller stirrer.

After the entire material is added in the heated water bath, the stirring continued for another 15-20 minutes. The resulting slip is aged for 2-3 hours in an electric dryer at a temperature of  $65-80^\circ \text{C}$ , then poured as a thin jet in suitable metallic molds. The casting is stored in a clean and dry place. The details are molded by hot casting under pressure from 1 to 3 atmospheres in metallic molds. The working temperature of the slip varies in the range  $60 \div 75^\circ \text{C}$ . After casting the products and hardening the slip, the mold is opened and the items are removed very carefully. Exerting an effort, such as pressure or tension on the product before it has solidified, is not desirable as it may result in deformation.

### 7.2. Removing the technological binder

The removing of the technological binder is carried out in the temperature range of  $50 - 1050^\circ \text{C}$  in an electric resistance furnace. The products are placed in high-alumina saggars and filled with adsorbent. As an adsorbent is used  $\text{Al}_2\text{O}_3$  with a grain size of  $40 \div 50$  microns. The rate of the temperature rise is as follows:

- $50 \div 200^\circ \text{C} - 10^\circ \text{C}$  per each 8 hours,
- $200 \div 300^\circ \text{C} - 25^\circ \text{C}$  per hour,
- $300^\circ \text{C} -$  holding for 3 hours,
- $300 \div 450^\circ \text{C} - 25^\circ \text{C}$  per hour,
- $450 \div 1050^\circ \text{C} - 100^\circ \text{C}$  per hour.

After the furnace is cooled down, the items are removed and cleaned with compressed air. Their final firing is carried out by placing them on refractory plates in a high-temperature electric furnace, at a temperature determined for each composition in the temperature range  $1550-1650^\circ \text{C}$ .

Only samples for physico-chemical and mechanical tests were produced by the presented technology.

### 7.3. Compression of ceramic products by explosion

For this purpose, a metal mold with a punch is used in which a certain amount of press powder is placed. The compression is carried out in a blast chamber, the explosive being placed around the punch. The disruptive explosive has a "pyromex" density with detonation velocity of  $D = 7,200 \text{ m/sec}$  and density  $R_0 = 1.48 \text{ g/m}^3$ . The explosion is carried out by an EAMN - 25 detonator.

The parameters of the blast on the punch are:

- Initial speed  $U_0 = 2,900 \text{ m/sec}$ ,
- speed of the particles produced by the explosion  $U_f = 1,823 \text{ m/sec}$ ,
- density of the explosion products  $\rho_f = 1,980 \text{ kg/m}^3$ ,
- speed of sound in the explosion products  $d_f = 5,377 \text{ m/sec}$ ,
- pressure of the wave front  $p_f = 0.131 \cdot 10^{11} \text{ Pa}$ ,
- ratio between the pressure of the wave front and the pressure on the punch  $p_f/p_1 = 1.31 \cdot 10^6 \text{ Pa}$ .

It is determined experimentally that for the same explosive the detonation velocity  $D$ , [m/sec] and the specific energy of blast transformation depend on the density of the charge  $\rho_0$ .

The high density of the samples obtained after the explosion compaction and their incredibly high increase in strength, despite the instantaneous impact of the pressure, shows the perspective of using this method to obtain preforms from of any technical ceramics.

## 8. Essence of the isostatic pressing

The essence of the method is based on the following: the granulate is packed in the rubber molds and vibrates with them, then it is placed in cylinders filled with liquid (water emulsion, glycerin or oil). A pump system creates pressure in the liquid around the sleeve in the cylinder with a value of up to 2,000 MPa - in our case 1300 MPa. The granulate is pressed from all sides by the impact of the liquid on the rubber mold. An irregular cylindrical preform with uniformly distributed density and isotropic texture is obtained. The duration of the hydrostatic (isostatic) compression process depends on the size of the preforms and lasts for few minutes. Typical for this method is that there is no friction of the press powder on the mold's walls. It is compressed from all sides, producing a uniformly dense preform without the presence of layers or sections of different density. The densification is 35-39 % better in comparison with the classic automatic hydraulic press method, which results in an increase rate of solid-phase reactions during the firing process at the same temperature and reduces the shrinkage of the articles. In this sense, the method is more energy efficient. Due to the short movement of the high pressure there are profound changes in the structure of the crystalline substances.

### 8.1 Essence of the conventional molding method

The conventional molding method is the most common one. Articles from all masses regardless of their chemical and mineral composition can be produced by it. Density depends exclusively on the ratio, amount and size of the individual powder fractions and on the pressure and the molding conditions. The density of the bulk material upon free backfilling depends on the size, shape, particle size distribution, state and the structure of the particles. To obtain a dense package, the most suitable is a multi fraction composition with a maximum particle size of less than 1  $\mu\text{m}$ . Numerous studies are known concerning the particle size distribution and particle size of the material. Practically granulates are produced in the form of a press mass, which regulates pressing. The essence of the process is based on the following: low moisturized powdery granulate of  $\alpha\text{Al}_2\text{O}_3$  with particle diameters less than 1  $\mu\text{m}$  and modifiers with plasticizers are poured in the mold. Then the mass is pressed by a punch at one side (one-sided matrix) or by two punches moving oppositely (two-sided matrix). The pressure can be applied continuously during the entire compression process (one-stage compression) or with steps (stepped pressing). The continuation of the steps is of the order of a second or parts of a second. Stepped pressing is preferred as it helps the equalization of the pressure and the removal of the air from the mold. After the final pressing, the articles are pushed out of the die and the compression cycle is completed. By this method different samples of different compositions can be formed at different pressures.

### 8.2. Essence of the impulse compaction

This method is based on the effect of pulsating forces multiplied by value, with magnitude of up to a maximum of 100,000 tons at values of impulse body displacement up to 10 mm. This is achieved by means of a power pulse generator, a machine for transforming constant forces and moments into pulsating ones. The generator is implemented on a purely mechanical basis. It consists of a shell and impulse body, and in the cavity between them is a built-in bar mechanism, in which the circular joints and rod arms are replaced with segmental shoulders and joints. By this replacement, it is possible to obtain a large area of the press bearing and the dimensions and the mass of the power impulse generator can be reduced many times.

According to the described scheme, samples were pressed in the mold, giving extremely high physical and mechanical indices.

### 8.3 Sintering synthesis of test specimens obtained from different compositions at various methods and compression pressures

The corundum is difficult to sinter. The diffusion mechanism, including the recrystallization process, depends on the temperature and duration of firing, dispersity of the corundum, the activity of the crystalline chemical status of the material, the initial density of the green product, the presence of impurities or specially introduced modifiers, and last but not least the gas environment. The maximum grain size of  $\text{Al}_2\text{O}_3$  capable of solid-phase sintering must not exceed 3 - 5  $\mu\text{m}$ . We use finely dispersed  $\text{Al}_2\text{O}_3$  - up to 1  $\mu\text{m}$ . During the sintering process, recrystallization of the dispersed corundum ceramics occurs, which is of great importance for increasing or decreasing the size of the crystals, which affects the different properties. During our studies we achieved a crystal size of 1.2 to 2.5  $\mu\text{m}$ , average 1.75  $\mu\text{m}$ . The degree of perfection of the corundum crystal lattice also has a very strong influence on the firing temperature and the degree of sintering. The crystalline grid of electro fused corundum has a high degree of lack of defects and therefore, with identical dispersity, the articles made from it without the use of modifiers are much more difficult to sinter than those obtained from technical  $\text{Al}_2\text{O}_3$ .

### 8.4. Investigation of the physico-mechanical properties of the obtained corundum ceramics

Experimental corundum ceramic samples of different compositions obtained by the above-described formation methods, at different compaction pressures and sintered at specified temperatures and duration of the isothermal temperature retention are studied and a number of physico-chemical indicators are determined. The results are analyzed and plates with dimensions 50 x 50 x 10 mm - 20 pcs and 50 x 50 x 8 mm - 20 pcs are produced at industrial conditions, corresponding to the conditions of the preparation of the best samples. An excerpt of the best results is presented in Table 1.

**Table 1.** An excerpt of the physico-mechanical properties of corundum ceramics, obtained by different forming (pressing) methods

| №                                                                                              |                              | 1  | 1                                               | 2                                               | 3                                               | 4                                                 | 5                            |
|------------------------------------------------------------------------------------------------|------------------------------|----|-------------------------------------------------|-------------------------------------------------|-------------------------------------------------|---------------------------------------------------|------------------------------|
| Composition                                                                                    |                              | 2  | 3                                               | 1                                               | 2                                               | 1                                                 | 4                            |
| Forming method                                                                                 |                              | 3  | Conventional method<br>P = 10 MN/m <sup>2</sup> | Isostatic pressing<br>P = 130 MN/m <sup>2</sup> | Impulse pressing<br>P = 2.786 GN/m <sup>2</sup> | Explosive pressing<br>P = 24.00 GN/m <sup>2</sup> | Thermo-plastic pressing      |
| Firing temperature and time                                                                    |                              | 4  | 1,570 °C, 2h                                    | 1,580 °C, 2h                                    | 1,570 °C, 2h                                    | 1,560 °C, 2h                                      | 1,600 °C, 2h                 |
| Initial density (after forming)                                                                | $\rho_0$ , g/sm <sup>3</sup> | 5  | 16.2                                            | 20.0                                            | 20                                              | 25                                                | —                            |
| Relative share of density after forming in respect to the theoretical density of pure corundum | %                            | 6  | 40.50                                           | 47.5                                            | 55.75                                           | 58.00                                             | —                            |
| Final density (after firing)                                                                   | $\rho$ , g/sm <sup>3</sup>   | 7  | 3.75                                            | 3.85                                            | 3.90                                            | 3.95                                              | 3.70                         |
| Relative share of density after firing in respect to the theoretical density of pure corundum  | %                            | 8  | 87.00                                           | 93.85                                           | 94                                              | 96                                                | —                            |
| Transmission of the speed of sound                                                             | $V_i$ , m/s                  | 9  | 9,780.5                                         | 10,272.3                                        | 10,300.5                                        | 10,350.00                                         | 9,800.00                     |
| Poisson's coefficient                                                                          | $\mu$                        | 10 | 0.20                                            | 0.22                                            | 0.22                                            | 0.23                                              | 0.20                         |
| Bending strength                                                                               | $\sigma$ , MPa               | 11 | 320                                             | 420                                             | 425                                             | 480                                               | 330                          |
| Modulus of elasticity                                                                          | E, GPa                       | 12 | 370.2                                           | 390.00                                          | 390.00                                          | 400                                               | 380                          |
| Relative share of absorbed kinetic energy                                                      | DE, Jm <sup>2</sup> /kg      | 13 | 46                                              | 63                                              | 70                                              | 72                                                | —                            |
| Knoop Hardness                                                                                 | HRC                          | 14 | 10 GPa at a load of 1,000g                      | 11 GPa at a load of 1,000g                      | 11.5 GPa at a load of 1,000g                    | 11.5 GPa at a load of 1,000g                      | 10.2 GPa at a load of 1,000g |

## II. Boron carbide ceramics

### 1. Materials and influence of their properties on the quality of the boron carbide products.

The properties of boron carbide products depend on the properties of the starting material (boron carbide powder) and the production technology. The properties of the powder are structurally sensitive and depend on the porosity, shape and size of the grains, their orientation, the type and amount of impurities, as well as their volume distribution. In addition, boron carbide has a phase-varying composition that depends on the ratio of components B and C. Boron carbide is produced in a number of countries - USA, Germany, Russia, China and others. For now, China is the largest producer of  $B_4C$  and at the lowest price. The purest  $B_4C$  is produced in the US and Germany, but at a very high price.

The quality of the products, as noted, depends on the purity of boron carbide and the molding and sintering technology. Assuming that hot pressing is the best technology for a given sedimentary and chemical composition of  $B_4C$  powder, the question arises as to which factor determines cost and how to improve it. Generally hot pressing is performed in rigid graphite molding equipment in an argon or vacuum environment. Regardless of the quality of graphite and the atmospheric conditions, the molding equipment breaks down and pollutes the surface layer of the articles changing the B:C ratio.

In view of the unlimited number of suppliers of different chemical and chemical compounds, our task was to buy ready-to-use powder from  $B_4C$  and to focus on the main part of the technology- molding, temperature regime and clinkering of the products.

When developing our technology, we use boron carbide, bought from Thermo Fisher - Germany, accompanied by a Certificate of analysis of the boron carbide 99 +% product. The product has the following composition presented in the following table:

**Table 2** Certificate of analysis of the boron carbide 99 +% product

|                      |           |
|----------------------|-----------|
| Particle size        | -325 mesh |
| Boron Carbide        | 99.01 %   |
| Total Boron + Carbon | 99.75 %   |
| Total Boron          | 78.41 %   |
| Free Boron           | 0.09 %    |
| Boron Anhydride      | 0.12 %    |
| Boron Compound       | 78.32 %   |
| Total Carbon         | 21.34 %   |
| Carbon Compound      | 20.69 %   |
| Iron                 | 0.10 %    |

### 2. Factors influencing the production of $B_4C$ -based products

The process of molding of preforms from  $B_4C$  is one of the most important. To a great extent it determines very important properties of the sintered products, especially the mechanical strength. Various molding methods have been developed which are also applied to other ceramic materials. Compaction in steel molds, isostatic pressing, hot-pressing with simultaneous sintering, slip

casting (thermoplastic and in the form of aqueous dispersion colloid systems) are widely used for the forming of products from high melting point powders. The latter two methods are very rarely used. Forming in metal molds is primarily for modified boron carbide compositions. It is widespread and productive. Isostatic pressing produces good results but is economically ineffective. Top quality products are produced by hot pressing with simultaneous sintering, but they are very expensive and are used for special purposes only. The essence of the pressing process is in obtaining preforms with definite shape by reducing the volume of boron carbide powder used by exertion of pressure resulting in increased contact between the particles. In the free fill state the contact area between the powder particles is very small. In the initial stage of compaction, the particles are moved and fill the empty spaces, and thus increase the area of the contacts. Deformation begins to occur at the points of contact where the pressure is transmitted. Gradually, these contacts begin to appear in small groups of particles and finally throughout the volume of compressed powder. As a result, the powder particles begin to deform plastically if the temperature is high or broken into small pieces if the temperature is low.

Compaction of powders by pressure is a complex process that depends on the properties of the pressed material and of the particles and on the manner and conditions of the pressing. Before compression of powders of difficult-to-melt compounds such as  $B_4C$ , plasticizers are added to obtain the necessary molding plasticity. Rubber dissolved in gasoline, bakelite dissolved in alcohol, polyvinyl alcohol and the like are used as plasticizers. The plasticizers are mixed and homogenized with the powder and then the plasticized material is granulated. It should be noted that the plasticizer facilitates compression, but adversely affects the quality of the product, as it enters the gaps, closes the air and limits the possibility of obtaining high-density products. This requires that the minimum required amount of plasticizer should be determined experimentally, depending on the pressing method. There are two technologies that we have studied and are using in our practice to obtain  $B_4C$  products. One involves the compression of a modified  $B_4C$  based composition with 99% purity plasticized with gasoline-dissolved rubber into a metal die using a hydraulic press, and the other is isostatic pressing of preforms with the same composition and with reduced plasticizer content followed by machining until raw materials are obtained before sintering. These technologies are described in detail above for the production of high alumina corundum ceramics. Thus, 50 x 50 x 10 mm and 50 x 50 x 8 mm tiles of boron carbide ceramics were obtained. In order to avoid labor-intensive machining after isostatic pressing, the articles are first compressed into a metal die in a hydraulic press and then are placed in thin-walled elastic cuffs and pressed isostatically to eliminate structural differences in the raw material volume, which are the main reason for deformation of the products during the sintering process. The heat treatment was performed in a high temperature furnace in argon medium. The articles are placed in a closed box made of high-density graphite covered with  $B_4C$  powder of the same composition as the test pieces. The high temperature heat treatment time is 30 min at 2,250°C. The physico-mechanical parameters of the obtained boron carbide ceramics are shown in Table 2.

**Table.3.** Physical and mechanical parameters of boron carbide ceramics obtained by hydraulic pressing and isostatic additional pressing

|                                                                                                                                       |                                |          |                                                                |
|---------------------------------------------------------------------------------------------------------------------------------------|--------------------------------|----------|----------------------------------------------------------------|
| <b>№</b>                                                                                                                              |                                | <b>1</b> | <b>1</b>                                                       |
| <b>Composition</b>                                                                                                                    |                                | 2        | 3                                                              |
| <b>Forming method</b>                                                                                                                 |                                | 3        | Hydraulic pressing<br>$P = 10 \text{ MN/m}^2$                  |
| <b>Sintering time and pressure</b>                                                                                                    |                                | 4        | Isostatic additional pressing<br>$2,250^\circ\text{C}$ , 1/ 2h |
| <b>Initial density (after forming)</b>                                                                                                | $\rho_0$ , $\text{g/sm}^3$     | 5        | 12.30                                                          |
| <b>Relative share of density after forming in respect to the theoretical density of pure corundum <math>\text{B}_4\text{C}</math></b> | %                              | 6        | 35.60                                                          |
| <b>Final density after sintering</b>                                                                                                  | $\rho$ , $\text{g/sm}^3$       | 7        | 2.47                                                           |
| <b>Transmission of the speed of sound</b>                                                                                             | $V_i$ , m/s                    | 8        | 12,490                                                         |
| <b>Bending strength</b>                                                                                                               | $\sigma_i$ , MPa               | 9        | 430                                                            |
| <b>Modulus of elasticity</b>                                                                                                          | $E$ , GPa                      | 10       | 395                                                            |
| <b>Relative share of absorbed kinetic energy</b>                                                                                      | $DE$ , $\text{Jm}^2/\text{kg}$ | 11       | 72                                                             |
| <b>Knoop Hardness</b>                                                                                                                 | HRC                            | 12       | 14 GPa at a load of 1,000g                                     |
| <b>Poisson's coefficient</b>                                                                                                          | $\mu$                          | 13       | 0.19                                                           |

### III. Conclusion

In conclusion, we can summarize that a literature and marketing study was conducted on the standards and raw materials for the production of corundum and boron carbide ceramics. In-depth information is presented on the indicators that must be met by both types of ceramics related to the production of materials with desired properties. The constructive developments for molds and the production of rubber bellows for isostatic pressing are presented. The physico-mechanical parameters of corundum and boron carbide ceramics compositions have been studied and described, and the best values are presented in a table.

### Acknowledgements

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## Конструкции за влагане и хомогенизация на елементи в стопилки от алуминий и алуминиеви сплави

### Constructions for introduction and homogenization of elements in melts of aluminum and aluminum alloys

Chief Assistant PhD Boyko Krastev, Assoc. Prof. PhD Valentin Manolov, Assoc. Prof. PhD Angel Velikov  
Institute of Metal Science, Equipment and Technologies with Hydroaerodynamics Center "Acad. A. Bolevski" –  
Bulgarian Academy of Sciences, Sofia, Bulgaria

Гл.ас.д-р Бойко Кръстев, доц.д-р Валентин Манолов, доц.д-р Ангел Великов  
Институт по металознание, съоръжения и технологии с център по хидро- аеродинамика "Акад. А. Балеvски"  
Българска академия на науките, София, България

**Резюме:** Разработени са два броя конструкции за въвеждане в стопилката на елементи, както следва:

- за въвеждане на калций (Ca) на гранули, за повишаване на вискозитета и хомогенизация чрез механично разбъркване;
- за въвеждане на  $TiH_2$ , хомогенизация, последващо формиране на порестата структура и кристализация.

Проведени са изследвания за подаване на елементите в стопилката. Експериментирани са различни скорости на въртене на пропелера, съответно 300-400 об/мин при влагане на Ca и 300-600 об/мин при влагане на  $TiH_2$ .

**Ключови думи:** конструкции, хомогенизация

**Abstract:** Two constructions for the introduction of elements into the melt have been developed:

- for introduction of granulated calcium (Ca), for the viscosity increasing and for homogenization by mechanical stirring;
- for the introduction of  $TiH_2$ , homogenization, subsequent formation of the porous structure and crystallization.

Studies have been carried out for feeding the elements into the melt. It has been experimented different speeds of propeller rotation, respectively 300-400 rpm at Ca introduction and 300-600 rpm at  $TiH_2$  introduction.

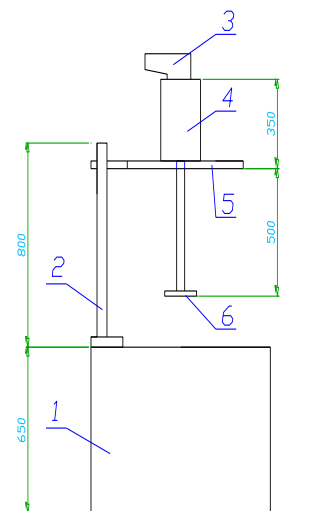
**Key words:** constructions, homogenization

#### 1. Въведение

Получаването на алуминий и алуминиеви сплави с висока порестост или наричани още пени е предмет на работата на редица изследователи. Тези материали имат ниска плътност с голямо количество разпределени пори. Намират приложение за топлоизолация и за защита при пожар, а също така за звукоизолация и за филтриране на течности. В настоящата работа са представени две конструкции за влагане на алуминий и алуминиева сплав A356.

2. Конструкция (установка) за влагане на Ca в стопилка  
На фиг.1 схематично е показана установка за влагане на Ca в стопилка от алуминий или алуминиева сплав A356. Установката е неподвижно монтирана към съпротивителна пещ 1, в която се осъществява стопяването. Установката се състои от колона 2, бормашина 3, защитен цилиндър 4, защитна плоча 5 и пропелер 6. Показана установка

Задвижването на пропелера се осъществява посредством бормашината, към която е монтирано устройство, определящо обороти 300-400. Устройството е изработено от фирма TOMIS EOOD и чрез него се регулират оборотите (скоростта) на въртене.



Фиг.1. Установка за влагане на Ca в стопилка

На фиг. 2 е нагледно е показан външния вид на установката.



Фиг. 2. Външен вид на установката.

Калцийт, във вид на гранули, е предварително опакован в алуминиево фолио и е прикрепен към пропелера. След стопяването на материала в пещта, калцийт се внася в стопилката и се задвижва пропелера. Разбъркването трае 6 минути, като по този начин се повишава вискозитетът на стопения материал (фиг.3).



Фиг.3. Хомогенизиране на калций

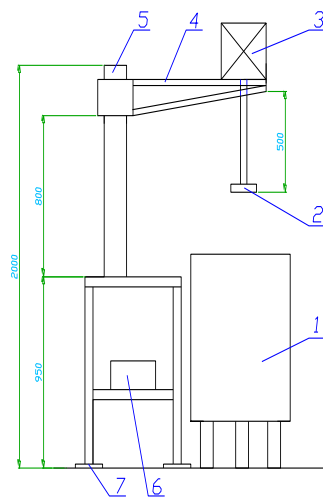
След хомогенизирането на стопилката, тя се прелива в метална пота, предварително подгрята във втора пещ (фиг.4)



Фиг.4. Преливане на стопилка

3. Конструкция (установка) за влагане на  $TiH_2$  в стопилка

На фиг.5 схематично е показана установка за влагане на  $TiH_2$  в стопилка от алуминий или алуминиева сплав А356. Установката е подвижна и е монтирана в близост до съпротивителна пещ 1, в която се осъществява стопяването. Установката се състои от носеща маса 7, Устройство за регулиране на оборотите 6, колона 5, носеща рейка 4, електродвигател 3 и пропелер 2.

Фиг.5. Установка за влагане на  $TiH_2$  в стопилка

Фиг.6. Външен вид на установката.

След преливането на стопилката се задвижва пропелера и започва разбъркването на  $TiH_2$ , предварително опакован в алуминиево фолио, в разтопения материал (фиг.7).



Фиг.7. Хомогенизиране на  $TiH_2$ .

Тази операция продължава 90 секунди. След приключване на хомогенизацията сплавта се разпенва. Експериментирано е интензивно охлаждане, с цел фиксиране на разпенения материал (фиг.8).



Фиг.8. Принудително охлаждане.

На фиг.9 е показана получена разпенена отливка.



Фиг.9. Разпенена отливка.

#### 4.Заклучение

Разработени и изработени са две конструкции (установки) за влагане на  $Ca$  и  $TiH_2$  в алуминий и алуминиева сплав. Установките ще бъдат използвани и при следващи експерименти за влагане на наноматериали в стопилка. Работата е подкрепена от проект по договор № КП-06-Н47/12 от 03.12.20г., финансиран от Фонд за научни изследвания, България. Оборудването и експерименталните единици бяха финансирани от Европейския фонд за регионално развитие в рамките на ОП „Наука и Образование за интелигентен растеж 2014-2020, проект СЕ „Национален център по мехатроника и чисти технологии“, № BG 05M2OR001-1.001-0008-С8.

# Study of the microstructure evolution during forging in a new design strikers implementing alternating deformation by finite element method

Lezhnev Sergey Nikolaevich<sup>1</sup>, Naizabekov Abdrakhman Batyrbekovich<sup>1</sup>, Panin Evgeniy Aleksandrovich<sup>2</sup>, Kuis Dmitry Valeryevich<sup>3</sup>, Stepankin Igor Nikolaevich<sup>4</sup>

<sup>1</sup>Rudny industrial Institute, 50 let Oktyabrya str. 38, Rudny, 115000, Kazakhstan; e-mail: sergey\_legnev@mail.ru

<sup>2</sup>Karaganda Industrial University, Republic av. 30, Temirtau, 101400, Kazakhstan; e-mail: cooper802@mail.ru

<sup>3</sup>Belarusian State Technological University, Sverdlova str. 13a, Minsk, 220006, Belarus; e-mail: dmitrykuis@mail.ru

<sup>4</sup>Pavel Sukhoi State Technical University of Gomel, Oktyabrya av. 48, Gomel, 246746, Belarus; e-mail: igor-stepankin@mail.ru

**Abstract:** The study of the microstructure evolution during forging in a new design strikers implementing alternating deformation based on computer modeling by the finite element method was carried out. The influence of the flat face inclination angle was studied, for which models with an inclination angle of 0, 15, 30 and 45 degrees were studied. It was established that the most optimal option is the use of strikers with an angle of 30°. On this strikers configuration the influence of the workpiece heating temperatures and the punch movement speeds was studied. The analysis of these technological parameters on the microstructure evolution showed that both parameters affect the structure grinding intensity, while the influence of the heating temperature is more significant.

**KEYWORDS:** FORGING, BROACHING, SHEAR DEFORMATION, COMPUTER MODELING, MICROSTRUCTURE EVOLUTION.

## 1. Introduction

In traditional metal forming operations, to improve the metal quality by grinding the structure to a fine-grained state, it is necessary to significantly change the workpiece sizes, which leads to significant energy and labor costs. However, it is often necessary to obtain a blank, the dimensions of which differ slightly from the initial one. The implementation of these two principles in practice is possible due to the intensification of shear and alternating deformations [1]. At the same time, for a high-quality processing of the initial structure, it is necessary to ensure such a technological process of deformation that sufficient shear or alternating deformation occurs in the entire deformable volume [2]. Currently, to solve this problem, a number of new forging processes and tools for their implementation have been developed [3-7], which allow implementing a high level of shear or alternating deformations.

In the work [8] the technology of broaching in lock strikers was developed and studied, which allows to realize intensive shear deformation over the entire volume of metal, and a forging tool for its implementation was proposed. This technology showed very good results, which consisted in improving the quality of the metal of forgings [9]. But this technology has its own small drawbacks - this technology is mainly applicable for forgings of only rectangular cross-section, and there are concentrators of tensile stresses due to the angles in this design.

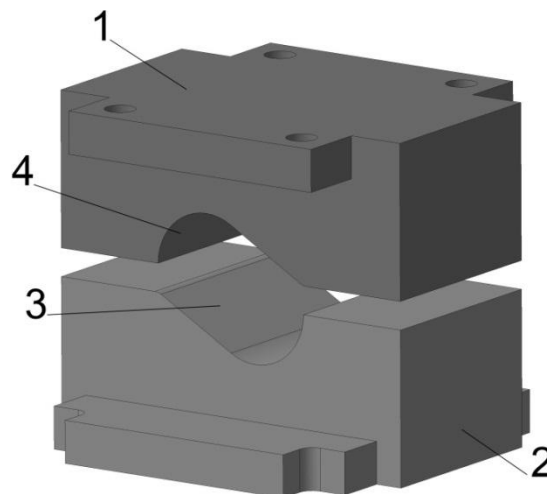
Therefore this tool has been improved so that it is possible to use it for round forgings and to avoid the occurrence of even small concentrators of tensile stresses. At the same time, the design of this tool (Fig. 1) will allow for more significant alternating deformations over the entire volume of the deformed workpiece.

The purpose of this work is to study the influence of the faces inclination angle of the strikers shown in Figure 1, as well as kinematic and temperature factors on the microstructure evolution.

## 2. Computer simulation

Computer simulation of the forging process with an additional macroshift was carried out using the Deform software package. 5HV2S steel was chosen as the billet material. The initial billet had a diameter of 45 mm. The deformation was carried out at a temperature of 1000 °C. A non-isothermal calculation type was set for the simulation. The vertical speed of the upper striker was 1 mm/sec. When the workpiece came into contact with the strikers,

the value of the friction coefficient was set to 0.25. An absolute tetrahedral grid was built on the blank, condensed on the surface for better rendering of the round shape. The minimum size of the element was set to 0.3 mm, the maximum size of the element was set to 0.6 mm, the parameters for the grid remeshing were set by default.



**Figure 1** - Tool for broaching: 1 – upper striker; 2 - lower striker; 3 - flat working surface of the striker; 4 - working surface of the striker, made in the form of a circle segment

During computer modeling, the influence of the flat face inclination angle, as well as kinematic and temperature factors on the microstructure change was studied. The value of 60 microns was taken as the initial grain size. 4 deformation cycles were carried out with the workpiece turning at 90° after each compression. When analyzing each model, the study of the selected parameters was carried out on the last cycle.

First, the influence of the flat face inclination angle was studied. For this purpose, models with an inclination angle of 0, 15, 30 and 45 degrees were built. In order to identify the most rational strikers design, it is necessary to introduce a criterion of optimal values. The following criterion for grain size was adopted - "less is better".

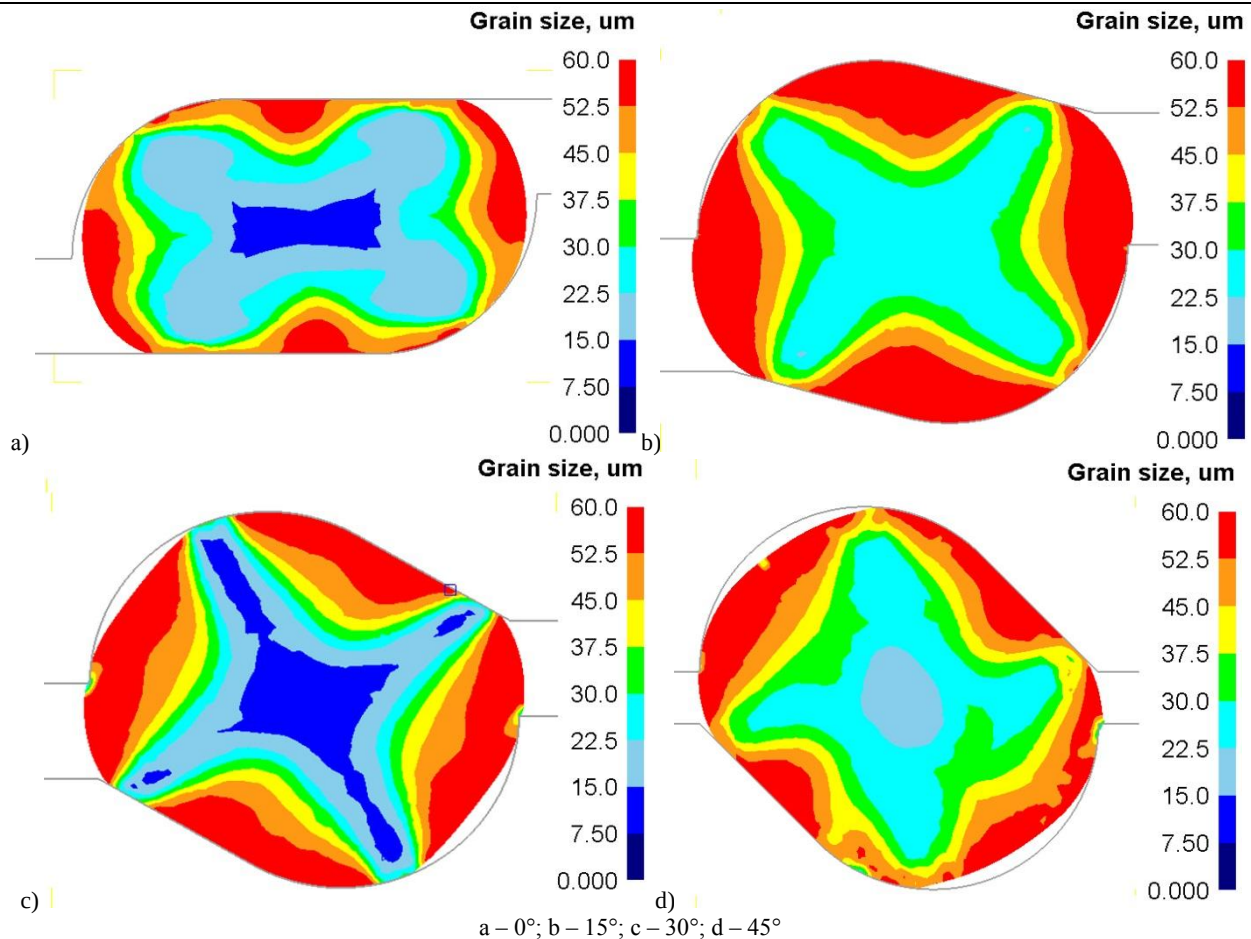


Figure 2 – Grain size change in strikers with different inclination angles of a flat face

The analysis of the microstructure evolution (Figure 2) showed that in all variants the grain grinding area covers most of the cross-section of the workpiece. At the same time, the initial grain is crushed most intensively in strikers with flat face inclination angle of  $0^\circ$  (11 microns) and  $30^\circ$  (13 microns). Based on the accepted optimality criterion of the grain size "less is better", the following sequence was identified in descending order:  $15^\circ \rightarrow 45^\circ \rightarrow 30^\circ \rightarrow 0^\circ$ .

Thus, the most optimal option would be to use strikers with an angle of  $30^\circ$ , since in this case there is a fairly extensive distribution of strain across the cross-section in the workpiece (the second in terms of cross-section coverage after strikers with an angle of  $0^\circ$ ) with a significantly lower deformation force [10]. The result is the second most intense level of grain grinding.

For further analysis of the influence of the technological parameters in the new strikers on the microstructure evolution, it was decided to use strikers with an inclination angle of flat faces of  $30^\circ$ . The temperature of metal heating and the speed of the punch movement were chosen as variable parameters, since the values of these parameters are easy to change in real conditions. The resulting model is shown in Figure 6b at a temperature of  $1000^\circ\text{C}$  and the upper striker speed of  $1\text{ mm/s}$  was taken as the base model. As a result, to study the influence of the technological parameters in new strikers on the microstructure evolution, the following models were additionally constructed:

- with heating temperatures of the workpiece up to  $1200^\circ\text{C}$  and up to  $800^\circ\text{C}$ ;
- with upper striker speeds of  $0.1\text{ mm/s}$  and  $10\text{ mm/s}$ .

Figure 3 shows the results of calculating the change in grain size for various technological parameters. When considering models with different values of the heating temperature (Figure 3 b-c), it was found that this parameter has a very significant effect on the grinding intensity of the structure. When the heating temperature increases to  $1200^\circ\text{C}$ , the grain grinding intensity decreases significantly, which is the result of an increase of static and

dynamic recrystallization. When the heating temperature decreases to  $800^\circ\text{C}$ , on the contrary, the intensity of structure grinding increases significantly. This is due to the fact that for steel 5HV2S, the temperature of the beginning of recrystallization is  $775^\circ\text{C}$  (point AC1). Heated to  $800^\circ\text{C}$ , the workpiece gradually cools down, bypassing the AC1 point, as a result, the recrystallization processes are completely suppressed.

When considering models with different values of the upper striker speed, it was found that this parameter affects the intensity of structure grinding, although not as significant as the heating temperature. When the upper striker speed increases to  $10\text{ mm/s}$ , the intensity of grain grinding decreases, and when the upper striker speed decreases to  $0.1\text{ mm/s}$ , the intensity of structure grinding increases. This phenomenon is also associated with recrystallization processes. The recrystallization intensity depends on the workpiece temperature, the intensity of change of which depends on the strain rate. At a reduced upper striker speed, the total duration of the deformation process increases. As a result, the workpiece has time to cool down more, which affects the level of grain grinding. At an increased strain rate, the intensity of cooling of the workpiece decreases, which leads to a decrease in the level of grain grinding.

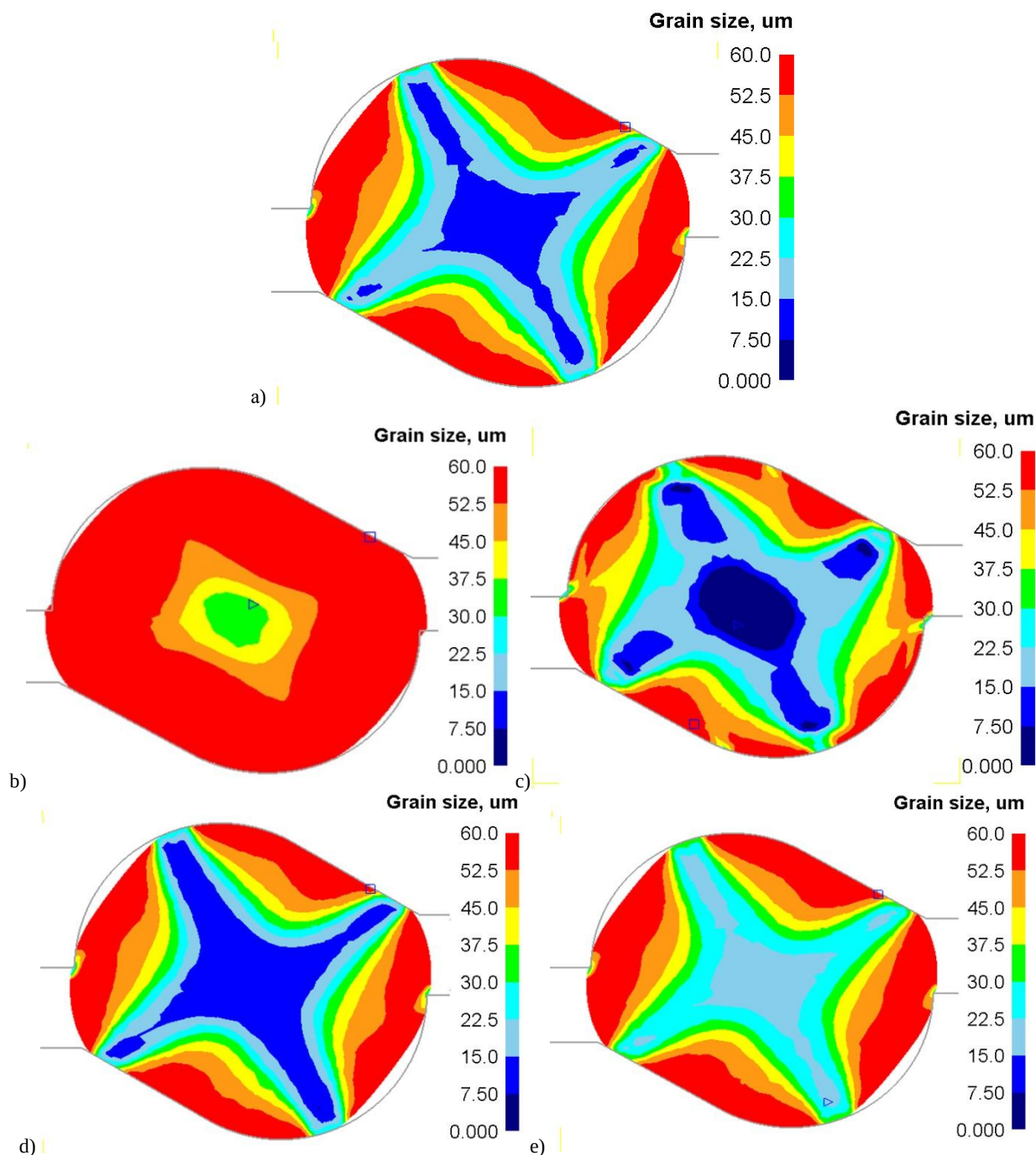
Based on the data obtained, the most optimal conditions from the point of view of the final grain size are the heating temperature  $800^\circ\text{C}$  and the upper striker speed  $0.1\text{ mm/s}$ . However, in order to approve these parameters, it is necessary to evaluate the resulting deformation forces in the models under consideration, since this parameter is critical at the stage of implementation of the developed technology. Summary data on grain size and deformation force are presented in Table 2.

It can be seen that despite the minimum grain size obtained, the option of lowering the heating temperature of the workpiece to  $800^\circ\text{C}$  is not optimal, since in this case, due to cooling and lowering the plasticity of the metal, the force level increases almost 2.5 times compared to the basic level. At the same time, all three options for the upper striker speed can be recommended for the

implementation of this process in practice. The key factor in choosing the speed here will be the rated power of the pressing equipment. With a sufficient margin of safety, it is desirable to conduct the deformation process at reduced deformation rates, which will lead to additional grinding of the initial grain.

**Table 2** - Grain size and deformation force in models with different technological parameters

|            | Base model       | 1200 °C          | 800 °C          | 0,1 mm/s         | 10 mm/s          |
|------------|------------------|------------------|-----------------|------------------|------------------|
| Grain size | 13 $\mu\text{m}$ | 36 $\mu\text{m}$ | 6 $\mu\text{m}$ | 10 $\mu\text{m}$ | 19 $\mu\text{m}$ |
| Force      | 202 kN           | 164 kN           | 495 kN          | 278 kN           | 185 kN           |



a - base model; b - at 1200 °C; c - at 800 °C; d - at 0,1 mm/s; e - at 10 mm/s  
**Figure 3** - Grain size change in models with different technological parameters

## Conclusion

The paper presents the results of computer modeling of the microstructure evolution during forging of round-section blanks in new design strikers. The analysis of models with different inclination angles of flat faces showed that the most optimal option is to use strikers with an angle of 30°. The analysis of the influence

of technological parameters on the microstructure evolution showed that both parameters (upper striker speed and heating temperature) affect the structure grinding intensity, while the influence of the heating temperature is more significant. The most optimal heating temperature of the workpiece in terms of grain size and the resulting force is the base value of 1000 °C, while all three options for the speed of movement of the punch can be recommended for the implementation of this process in practice.

### **Acknowledgments**

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# Influence of the forging process in step-wedge-shaped strikers on the destruction nature of the deformable metal

Volokitin A.V.<sup>1</sup>, Tolkushkin A.O.<sup>2</sup>, Panin E.A.<sup>3</sup>, Pishchikov V.<sup>1</sup>

<sup>1</sup>Rudny industrial Institute, 50 let Oktyabrya str. 38, Rudny, Kazakhstan; e-mail: dyusha.vav@mail.ru

<sup>2</sup>Ural Federal University named after the First President of Russia B.N. Yeltsin, Ekaterinburg, Russia; e-mail: a.o.tolkushkin@urfu.ru

<sup>3</sup>Karaganda Industrial University, Republic av. 30, Temirtau, Kazakhstan; e-mail: cooper802@mail.ru

**Abstract:** The paper investigated the effect of cyclic deformation on the destruction nature of workpieces during broaching in step-wedge strikers. AISI 1020 was chosen as the workpiece material. According to the experiment results, the influence of turning and cycles number on the values of impact strength was carried out. Fractography analysis of the fracture surface structure was also conducted.

**KEYWORDS:** BROACHING, STEP-WEDGE STRIKERS, CYCLIC DEFORMATION, IMPACT STRENGTH.

## 1. Introduction

At the present time, many energy-saving technologies are known in the world that allow to obtain high-quality metal products by rolling, forging, pressing and other methods of metal forming. When forging, this problem is successfully solved when deforming metals in special tools that implement shear and alternating strains throughout the entire volume of the deformed metal. Since it is the implementation of intense shear strains in the forging process of forgings from ferrous and non-ferrous metals that leads to a significant change in the initial microstructure and an increase in the physical and mechanical properties of the deformed metal. In this regard, the production of blanks by new forging methods with a significant reduction in energy and labor costs improving the quality of forgings is economically advantageous. One of the ways to achieve this goal is the use of deformation methods and tools that implement shear and alternating strains in the entire volume of the metal.

Previously, various investigations have already been conducted to study the stress-strain state, forces, mechanical properties and microstructure during broaching in step-wedge strikers [1-4]. The main purpose of determining the impact strength during bending is to assess the performance of the material under difficult loading conditions and its tendency to brittle fracture [5].

## 2. Experimental part

The experiment was carried out as follows: blanks of AISI 1020 steel with dimensions of 30x60x250 mm were heated in a resistance chamber furnace to a temperature of 1200°C, and then deformed in step-wedge strikers with an angle of inclination of 30° and with a wedge angle of 160° (Fig. 1) with different angles of turning.

The first batch of blanks was deformed without turning - workpiece immediately after alternating deformation in step-wedge strikers was fed to a flat section of these strikers for straightening. After the 2nd, 4th and 6th deformation cycles, standard samples were made from deformed workpieces for impact strength testing (GOST 9454-60).

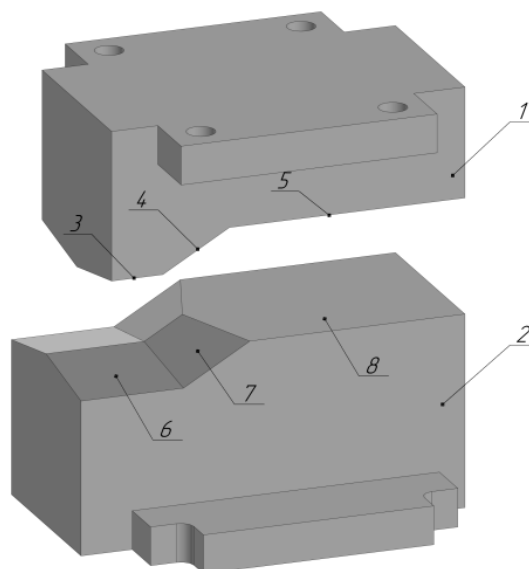
The second batch of blanks was deformed in the same tool, but with 180° turning, i.e. after each pass, the workpiece was turned by 180° and alternating deformation was performed. Standard samples for impact strength and compression testing were also made from deformed workpieces. Impact strength tests were carried out using a pendulum.

The impact strength was calculated by the formula [5]:

$$a = A / F \quad (1)$$

where F – the cross-sectional area of the sample at the incision zone before the test;

A - the work spent on deformation and destruction of the sample.



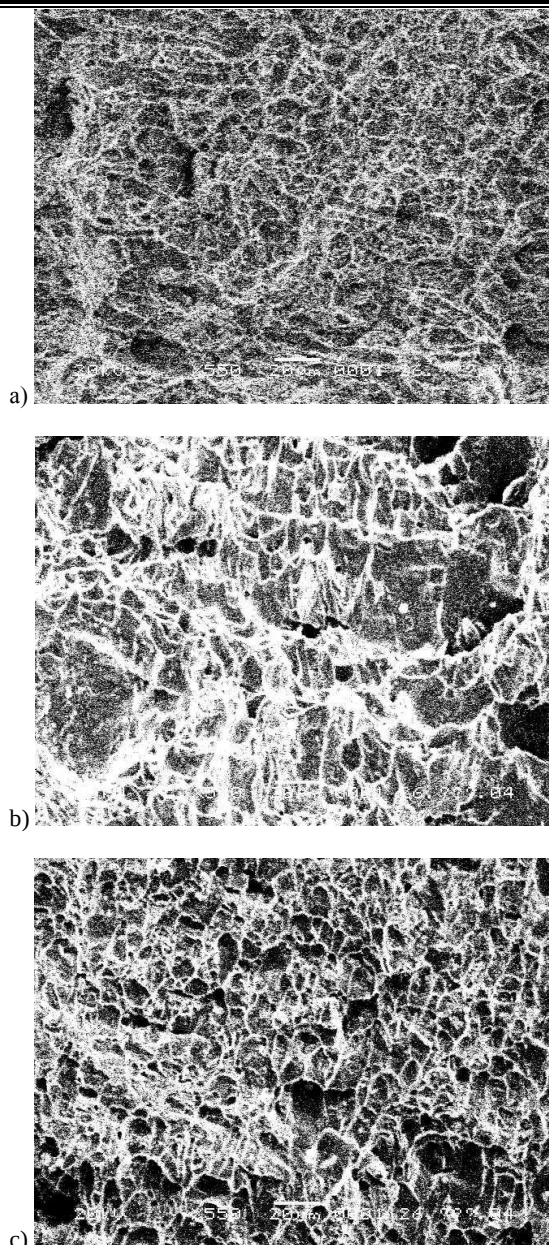
**Figure 1** - Step-wedge-shaped strikers

1 - upper striker; 2 - lower striker; 3 - smaller stage with wedge of the upper striker; 4 - inclined section with wedge of the upper striker; 5 - large flat stage of the upper striker; 6 - smaller stage with wedge of the lower striker; 7 - inclined section with wedge of the lower striker; 8 - large flat stage of the lower striker

It is known from the works [6, 7] that the more dislocation sliding systems are simultaneously involved in the plastic metal flow, the higher the mechanical and technological properties of the material. The maximum increase in the number of dislocation sliding systems involved can be achieved with cyclic deformation of metal in step-wedge strikers due to the implementation of alternating strains, both in the longitudinal and transverse directions. In addition, the desired effect is achieved faster if the workpieces are deformed with 180° turning. Thus, with an increase in the number of deformation cycles of workpieces in step-wedge strikers according to the proposed schemes, the impact strength of the metal progressively increases. The results of the calculated values of impact strength are presented in Table 1.

**Table 1** - Results of calculated values of impact strength

| Method of deformation                    | Number of cycles | Average value of impact strength, N/mm |
|------------------------------------------|------------------|----------------------------------------|
| In step-wedge strikers without turning   | 2                | 176,69                                 |
|                                          | 4                | 191,71                                 |
|                                          | 6                | 206,61                                 |
| In step-wedge strikers with 180° turning | 2                | 188,03                                 |
|                                          | 4                | 202,57                                 |
|                                          | 6                | 212,56                                 |



**Figure 2 - Fracture structures**

- a – fracture structure after two deformation cycles; b - fracture structure after eight deformation cycles without turning; c - fracture structure after eight deformation cycles with 180° turning*

The experiment results were also confirmed by a fractography study of the fracture surface, which showed that at the initial stages of processing in step-wedge strikers (Figure 2a), the fractures of the samples, in addition to the fibrous component of the viscous fracture, have a sufficiently large proportion of the brittle component. As the deformation cycles increase (Figure 2b), the fraction of the brittle component decreases due to an increase in the plastic characteristics of the material [5]. The same effect is enhanced when deforming samples with edging (Figure 2c).

## Conclusion

The test of workpieces formed in step-wedge strikers for impact strength was carried out. According to the experiment results, the influence of the turning and the number of broaching cycles on the impact strength values was investigated. A fractography study of the fracture surface of the workpieces showed that with an increase in the number of cycles, the plastic characteristics of the metal increase with a simultaneous decrease in brittle ones.

## Acknowledgments

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# The technology of obtaining copper-ferrochrome composite material

Lyudmila Ryabicheva<sup>1,\*</sup>, Aleksandr Dyadichev<sup>1</sup>  
Vladimir Dahl Lugansk National University, 20-A Molodezhnyi Sq., Lugansk, Ukraine<sup>1</sup>  
ryabic@gmail.com

**Abstract:** The research of the technology of obtaining new copper-ferrochrome composite material has been done. The results of its structure and properties analysis are presented. The material structure consists of the copper base, non-dissolved coarse inclusions of ferrochrome, diffused zones, forming around these inclusions and phases, having formed in the place of completely dissolved inclusions of ferrochrome fine particles. The material has good mechanical properties and a high wear resistance due to the formed solid solutions of the carbides in the copper.

**Keywords:** COMPOSITE MATERIAL, COPPER, FERROCHROME, PRESSING, SINTERING, STRUCTURE, CARBIDES, HARDNESS, STRENGTH, WEAR RESISTANCE

## 1. Introduction

To manufacture antifriction and construction details the alloys on the copper base – bronze and brass – are widely used. The products from these materials are obtained by casting and pressure treatment of compact materials [1] or from metallic powders by pressing and sintering [2]. The mechanical properties of the powder materials depend on the chemical composition, obtaining technology, porosity, dispersion of the initial powder components: having the same porosity, they increase with the decrease of the size of the powder particles. The promising tribotechnical materials are powder bronzes, being alloyed with chrome [3]. The disadvantage of cast and powder bronzes is their comparatively low carrying ability and a high price for the tin. Due to this, the development of the technology for obtaining composite material on the base of copper, and being reinforced by the dispersed inclusions, is of great interest.

## 2. Premises and means for the problem solution

The works [4, 5] present the results of researching the properties of the composite materials tin bronze – ferrochrome FC 800 with the particles' size of 40-50  $\mu\text{m}$ . It is ascertained that during the sintering of the samples with low porosity the more increase in the sizes occurs the more the content of Fe-Cr is, the highly porous samples undergo the shrinkage. At the porosity of 15 % the increase of Fe-Cr content up to 3 % leads to the increase of the yield point and to the decrease of the unit elongation. With the further increase of Fe-Cr content the strength and the plasticity decrease. The composition, containing 5% of Fe-Cr, has the maximum tribotechnical characteristics. The researches have shown that to work in the loaded units it is efficient to use pore-free composite material on the copper base, containing ferrochrome and a solid lubricant, i.e. graphite. Such material can be obtained by the technology of the powder metallurgy which includes the following actions: mix material preparation, pressing the porous workpieces, sintering, compaction up to the pore-free state. Moreover, considering a high cost of the copper powder, it is reasonable to use a cheaper copper powder, got from the industrial waste.

The given paper presents the results of researching the structure and properties of the composite material on the copper powder base, obtained from the copper electric conductor waste, mixed with ground ferrochrome.

## 3. The problem solution

The samples for the research were made from the mixture of copper powders 93.8 %; ground ferrochrome of FC-400A brand 5 % (ISO 5448-81) and 1.2 % fine-dispersed graphite. The ferrochrome and the copper powder were sifted through the sieve with the cells of 80  $\mu\text{m}$ .

According to the conducted chemical analysis, the ferrochrome contains 54.97 % Cr, 3.8 % C, the rest is iron. When introducing the mix material of FeCr – 5%, C – 1.2 %, Cu – 93.8% into the composition the content of the components in the mixture will be as follows: Cr – 2.75%, Fe – 2.06% and C – 0.19%. The density of the powder pore-free composite material, calculated by the additivity formula, is 8.46  $\text{gr}/\text{cm}^3$ .

The workpieces with the dimensions 12.6x54.3x10.0 mm and 10% porosity were pressed from the obtained mix material in the moulds in the hydraulic press. The lubricant – molybdenum disulfide – was applied on the matrix surface. The mass of the powder sample for pressing workpieces, which was calculated considering the mass loss during the pressing and sintering, was 53.7 gr. The pressing pressure was taken as equal to 600 MPa. The rated force of the pressing was 410.5 kN.

The pressings were sintered in the synthesis gas environment in the furnace according to the stepped regime with equalizing during 25 minutes at the temperatures 200-220°C and 300-320°C, the growth up to 400-420°C, equalizing during 90 minutes, heating up to the temperature 500-520°C and 600-620°C with the equalizing of 25 minutes at each temperature, the rise up to the temperature of 900-920°C and equalizing of 90 mins.

The sintered workpieces were stamped in the closed stamp with the cross flow of the metal with a fringe for 90 °, and then were sintered in the same environment at the temperature of 780-820°C for 30 minutes. The stamping pressure was 800 MPa, and the stamping strength was 535.7 kN. The samples for tension testing with the diameter 5 mm and the initial calculating length was 25 mm were turned from the prismatic samples.

While doing the researches the visual examination was done, the mass and the sizes were controlled. The samples for pressing workpieces were weighed on the laboratory scales with the precision up to 0.05 gr, the mass of the produced samples was determined by weighing on the electronic scales with the precision up to 0.0001, the density was calculated.

The hardness was measured by Brinell hardness test with the ball of 5 mm under the load of 2452 N. The structure was researched on the metallographic sections before the etching, and after the etching in the iron acid the structure was studied by the microscope MIM8 (МІМ8). The microhardness was determined by the device PMT-3 under the load of 200 N.

## 4. Results and discussion

The visual examination of the samples after pressing, sintering and stamping showed that cracks, tightening, fissures and other defects of the shape were absent.

The composite has a high compression rate. The density of the compressed workpieces was 7.74-7.59  $\text{g}/\text{cm}^3$ , this corresponds to the calculating porosity 6-10%. However, during sintering there was a growth of the samples, it is explained by the “hydrogenous disease” of the copper. The decrease of the sizes was 1.3% in length, 1.6% in the width, 8.7% in the height. As a result the porosity increased up to 19-20%.

The density of the stamped samples was 8.44  $\text{gr}/\text{cm}^3$ , i.e. practically pore-free material was obtained (the calculating porosity does not exceed 0.4 %). The hardness of the material after stamping was HB 68-72. As a result of annealing the sizes and the density of the samples did not change, while the hardness decreased and was HB 42-47. The limit of the strength under tension was 117 MPa, the unit elongation was 1.6 %, it is lower than of the chrome-tin bronzes.

To determine the wear resistance the tests were done on the friction machine according to the scheme “disc-block” of the tested

material and a known antifriction powder material on copper base (APMCP), containing 5-10.5% of chrome [4]. The counterbody from steel 45 had the hardness HRC 43. The pressure on the samples was 5.25 MPa, the rubbing speed was 2.1 m/s. Before the tests the samples were weighed on the analytical balance, and were well-lubricated, the lubrication consisted of molybdenum disulfide and machine oil. During the test the lubrication was not supplied to the friction zone. After the test end the samples were washed out by acetone, then dried and weighed again. The wear resistance was evaluated by the intensity of the wear:

$$(1), \quad I = \frac{W}{L},$$

where:

$W$  is the value of the linear wear of the friction samples;

$L$  is the friction length of the given sample, the friction length corresponds to the wear  $W$ , m.

While determining the wear by weighting, the value of the linear wear  $W$  was determined by the formula:

$$(2), \quad W = \frac{\Delta G}{\rho A},$$

where:

$\Delta G$  is the change of the samples' mass under the test, kg;

$\rho$  is the density of the worn out materials, kg/m<sup>3</sup>;

$A$  is the contour contact area of the samples, m<sup>2</sup>.

The friction length was defined by the ratio:

$$(3), \quad L = \pi d N,$$

where:

$N$  is the amount of full disc turns (determined by the counter registration of the turns' number on the friction machine);

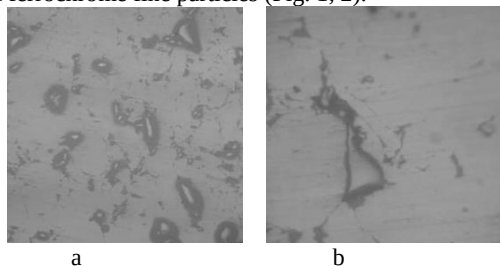
$d$  is the diameter of the roller, mm.

As it is evident from the tests' results, presented in Table 1, the wear resistance of the researched material is by 1.9 times higher and it wears out the counterbody by 1.7 times faster.

**Table 1:** The results of the trial on the friction machine

| Material                                            | The intensity of the wear, micron/km |                      |
|-----------------------------------------------------|--------------------------------------|----------------------|
|                                                     | Counterbody                          | The tested material  |
| Antifriction powder material on copper base (APMCP) | $5.22 \cdot 10^{-6}$                 | $2.62 \cdot 10^{-8}$ |
| The tested one                                      | $2.98 \cdot 10^{-6}$                 | $1.39 \cdot 10^{-8}$ |

The structure of the obtained material is heterophase, and consists of the copper base, non-dissolved coarse inclusions of ferrochrome, diffused zones, forming around these inclusions and phases, having formed in the place of completely dissolved inclusions of ferrochrome fine particles (Fig. 1, 2).

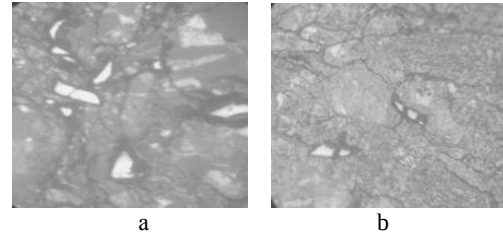


**Fig. 1.** The microstructure of the non-etched samples from the copper-ferrochrome composite material: a – 320x, b – 500x

According to the chemical analysis, ferrochrome contains 3.8 % of carbon. Apparently, it contains a great amount of carbides of the following type  $(Cr, Fe)_7C_3$  and  $(Cr, Fe)_{23}C_6$ . Besides, it may contain solid and fragile  $\sigma$ -phase (intermetallic compound FeCr). The carbides  $(Cr, Fe)_7C_3$  and  $(Cr, Fe)_{23}C_6$  have a complex crystalline lattice. Such carbides easily dissolve in the steels under heating in  $\gamma - Fe$  [6]. The information about dissolubility of carbides in the

steel is not found in the reference literature. It is possible to presume that carbides dissolve in the copper well due to the fact that  $\gamma - Fe$  has a face-centered cubic lattice with the period 0.3645 nm and the atomic radius 0.1411, the copper also has a face-centered lattice with the period 0.3608 nm and the atomic radius 0.1413.

Table 2 presents the results of measuring the microhardness of different phases of the composite material. The microhardness of powder copper without alloying additions is given for comparison. As it is seen, the hardening of the copper base happens. It is possible to presume that the copper hardening takes place due to the formed solid solutions of the carbides in the copper. The structure of the diffused zone can be presented by a mechanical mixture of solid solutions on the basis of copper and carbides.



**Fig. 2.** The microstructure of the samples from the copper-ferrochrome composite material after etching: a – 320x; b – 500x

**Table 2.** The results of the microhardness' measurement

| Material                                     | Limits of the microhardness values, MPa | Average value, MPa |
|----------------------------------------------|-----------------------------------------|--------------------|
| Unalloyed powder copper before annealing     | 748-944                                 | 845                |
| Unalloyed powder copper after annealing      | 228-412                                 | 345                |
| Copper as the base of the composite material | 616 – 719                               | 706                |
| Diffused zone                                | 1463 -1738                              | 1553               |
| Ferrochrome in the composite                 | 10478 – 19855                           | 14955              |

The carried out researches have demonstrated that the copper-ferrochrome composite material has very good mechanical properties and a high wear resistance. Such material can be used for manufacturing details, working in the friction units. The obtained results also indicate that further enhancement of the composite properties is possible due to the use of more dispersed ferrochrome particles, and also due to the thermal material processing, i.e. hardening and aging.

## 5. Conclusion

The research of the new copper-ferrochrome composite material has been done. The material structure consists of the copper base, non-dissolved coarse inclusions of ferrochrome, diffused zones, forming around these inclusions and phases, having formed in the place of completely dissolved inclusions of ferrochrome fine particles. The material has sufficient strength properties and high wear resistance. The reason for this is hardening of the copper due to the formed solid solutions of the carbides in the copper.

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# ТЕХНОЛОГИЯ И ПРИСПОСОБЛЕНИЯ ЗА СГЛОБЯВАНЕ И МЕХАНИЧНО ОБРАБОТВАНЕ НА КРАНОВА КОЛОНА

## TECHNOLOGY AND FIXTURES FOR ASSEMBLING AND MACHINING OF A CRANE COLUMN

Asst. Prof. Dr Stankov N.  
University of Ruse, 8 Studentska Str., 7017 Ruse, Bulgaria  
nstankov@uni-ruse.bg

**Abstract:** The paper presents the developing of a technology and the designing of fixtures for assembling and machining a crane column. The whole process related to the production of a certain crane column is considered - from the 3D model to the real product. A technology and fixtures for assembling and machining the column have been developed. A control card to control the dimensions at each stage of the production of the column has been made. A control programme for machining using a specific CNC machine has been developed. The 3D models are designed with the CAD system SolidWorks. Complete design documentation is created for each of the fixtures and the process is automated by using SolidWorks template files.

**Keywords:** CRANE COLUMN, CRANES, DEVELOPMENT, DESIGN, CONTROL, 3D MODELS, CAD SYSTEM, SOLIDWORKS TEMPLATE FILES, DESIGN DOCUMENTATION

### 1. Въведение

В доклада е представен съвместно разработен проект, свързан с конструирането и внедряването в производството на специализирана нестандартна технологична екипировка за изработването на кранови колони. Разработката е на екип от Русенски университет „Ангел Кънчев“ и фирма „СЛ Индъстрис“ ЕООД, град Русе [17]. Фирмата е производител на различни заваръчни конструкции (шасита, стабилизатори, предни и задни мостове, колони и стрели за кранове), машинно обработени детайли и изделия, компоненти и специализирана екипировка за кранове, мотокари, товарачи, влекачи, багери и други машини. Фирмата произвежда различни видове грайфери и кофи за насипни материали.

В доклада е разгледан процеса на изработване на един от основните компоненти на крановете, това е крановата колона. Крановата колона се монтира на различни видове кранове, автокранове, гондоли за скрап, лекотоварни автомобили и други специализирани машини.

Разнообразието на колони е голямо и основно зависи от предназначението им и тавароносимостта на конструкцията. Колоните представляват специализирана конструкция, устойчива на голямо натоварване. Монтирането ѝ към крана става чрез зацепване на зъбно колело със зъбна рейка. На тялото на колоната се монтират хидравлични цилиндри, които са закрепени на крановите стрели и осигуряват съответния им ход. Колоната осигурява завъртането на телескопичната стрела по време на работа. Това позволява по-голям обхват на действие на стрелата и разширява възможностите на крана.

На фиг. 1 е показана крановата колона, процесът на изработване, на която се разглежда в доклада [18].



Фиг. 1. Кран на фирма ATLAS с колоната.

Основен проблем при изработването на кранови колони е сложната форма и конструкция, която имат. Необходимо е постигането на точното разположение на детайлите им, за да се получат зададените в конструкторската документация форма и размери на колоната. Това важи особено за детайл зъбно колело, който е с точно определена ориентация в колоната.

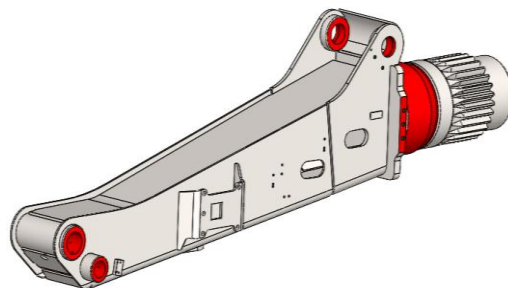
Съгласно изискванията на чертежите се прави механично обработване на функционалните повърхнини, за да може да се извърши монтаж на изделието и то да изпълнява предназначението си.

Това налага за сглобяването и механичното обработване на колоната да се разработят и използват при производството ѝ специализирани приспособления или т. нар. нестандартна технологична екипировка.

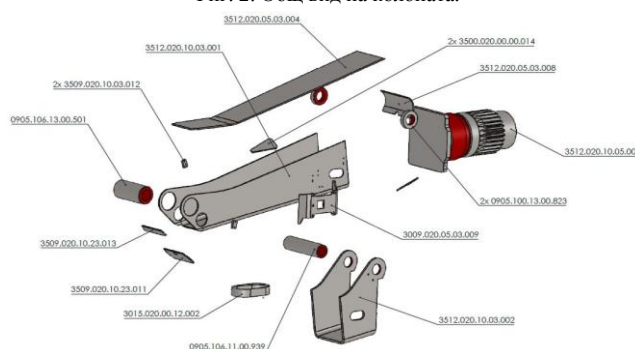
### 2. Предпоставки и средства за решаване на проблема

#### 2.1. Общи сведения за крановата колона

Различните видове кранови колони се различават по габаритните си размери, ориентацията в машината, конструкцията и натоварването, което могат да понесат. На фиг. 2 е показан общият вид на колоната, която ще бъде разгледана. Изделието е част от производствена програма на фирмата.



Фиг. 2. Общ вид на колоната.



Фиг. 3. Съставни елементи на колоната.

Съгласно разработената технология, за сглобяване на колоната се използва специализирано приспособление. Чрез приспособлението се извършва ориентиране и установяване на детайлите на колоната в определено положение. За позициониране на някои от детайлите се използват шаблони.

## 2.2. Технологична подготовка на детайлите на колоната

Съгласно документацията на изделието, предоставена от фирма ATLAS, се налага предварително да се подготвят технологично детайлите му и самото изделие, за да бъдат изработени в условията на фирмата.

Етапите от технологичната подготовка на изделието са следните:

- създаване на тримерни модели на детайлите и на изделието;
- разработване на технологията за изработване на детайлите и на изделието [16];
- създаване на нови чертежи на детайлите и на изделието;
- подготовка на файловете за разкрой на детайлите от листов материал;
- създаване на възли в изделието;
- разработване на технология за сглобяване на изделието;
- разработване на технологична екипировка за сглобяване;
- разработване на технология за заваряване на изделието;
- разработване на технология за механично обработване на изделието;
- разработване на технологична екипировка за механично обработване;
- избор на машина за извършване на механичното обработване;
- разработване на методика за контрол на изделието;
- разработване на ремонтна технология на изделието.

Създаването на тримерни модели е важна стъпка от процеса на разработване на дадено изделие. Тримерните модели могат да се използват за създаване на нова конструкторска документация за нуждите на фирмата. В тримерните модели се задава необходимата информация чрез атрибути, която може да се използва за управление на документацията при налична PDM система, както и от другите отдели във фирмата при налична ERP система [4, 5, 11, 12].

На база моделите се определя и технологията за изработване на детайлите – добавяне на отвори, прибавки за механично обработване, предварителна обработка на определени повърхнини. Това налага създаването на нова конструкторска документация, която да се използва в производството. След уточняване на всички особености, тримерните модели могат да се използват за създаване на файловете за разкрой на детайлите от листов материал.

За да се намали времето за изработване, много често се прилага метода на разделяне на изделието на възли, които предварително да се изработят, след което да се използва приспособление за сглобяването им в крайното изделие.

Последен етап е създаването на необходимата екипировка за сглобяване и за механично обработване на изделието.

В случая за разглежданата колона са разработени тримерните модели на детайлите, направени са технологични промени, свързани с производството им и са създадени нови възли от изделието.

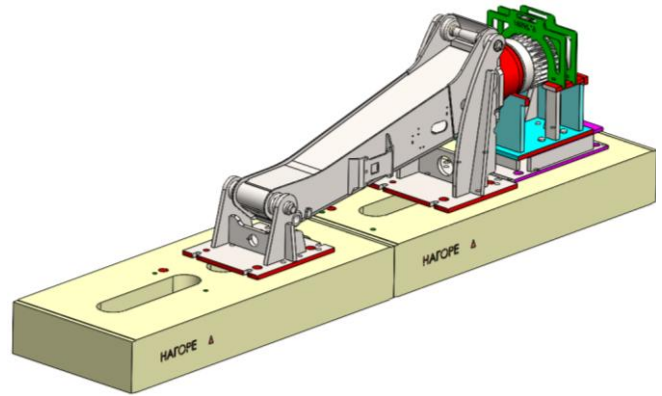
Тримерните модели са разработени с CAD системата SolidWorks [1, 2, 3, 6, 7, 19].

## 3. Решение на разглеждания проблем

### 3.1. Приспособление за сглобяване на колоната

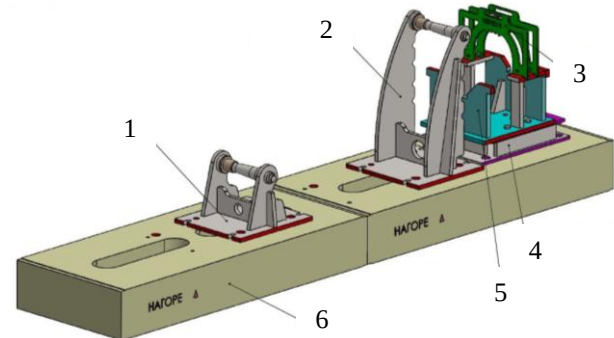
След като е уточнена технологията за изработването на детайлите, се разработва технологията за сглобяване на колоната и необходимата екипировка за реализиране на тази технология.

Приспособлението е разработено чрез CAD системата SolidWorks и на фиг. 4 е показан общият му вид с установени детайли на колоната.



Фиг. 4. Общ вид на приспособлението за сглобяване.

На фиг. 5 е показано приспособлението с всичките му съставни елементи. То осигурява позициониране на детайлите в определено положение и с точно определени разстояния между тях [8, 9, 10, 13, 14, 15].



Фиг. 5. Съставни елементи на приспособлението.

- 1 – Стойка предна, 2 – Стойка средна,  
3 – Шаблон за ориентиране на зъбното колело,  
4 – Основа, 5 – Призми, 6 – Маса двоена 2x1670 mm

За реализиране на технологията за сглобяване на колоната е разработено приспособление, състоящо се от няколко възела. Те се установяват на механично обработена двоена маса с размери 2x1670 mm, върху която са направени центрови отвори Ø43 mm и проходни резбови отвори M20 за центроване и закрепване на възлите.

На всеки един възел от приспособлението се извършва механично обработване на показаните с червен цвят повърхнини.

С механичното обработване се постига точност на размерите и на взаимното разположение на функционалните повърхнини на приспособлението.

Приспособлението осигурява висока производителност, не е сложно за манипулация, с цел да се избегнат всякакви грешки от субективен характер.

На фиг. 6 е показано изработеното приспособление.



Фиг. 6. Приспособление за сглобяване на колоната.

### Технологията на сглобяване на колоната е следната.

Сглобяването на колоната започва с установяване на зъбното колело 3512.020.10.05.000 върху призмите. Ориентирането на зъба от зъбното колело спрямо центъра на колоната става с шаблон. Втулките с номер 0905.100.13.00.823 се прихващат към детайл 3512.020.10.03.002 и заедно се установяват на приспособлението с дорник, минаващ през отворите им. Следващият детайл, който се установява е 3512.020.10.03.001. Към него се прихващат втулките с номера 0905.106.13.00.501 и 0905.106.11.00.939. Те се центроват към приспособлението с дорник, минаващ през отворите. След установяване на основните елементи на колоната се добавят и всички останали детайли, показани на фиг. 3. Всички детайли се закрепват в съответното им положение чрез прихващане.

На фиг. 7 е показана колоната, изработена с приспособлението.



Фиг. 7. Колоната след сглобяване с приспособлението.

### 3.2. Заваряване на колоната

След като е уточнена технологията за сглобяване, се разработва технологията за заваряване на колоната.

Заваряването на колоната е сложен процес и се изпълнява съгласно разработените за всяка една колона заваръчни планове. Заваряването може да бъде ръчно или роботизирано.

Заваръчният апарат трябва да бъде за МИГ-МАГ заваряване.

Зоните на заваряване трябва да бъдат почистени и на определени места предварително подгряти до определена температура.

След изваждане на колоната от приспособлението следва цялостното ѝ заваряване съгласно изискванията на заваръчния чертеж. След заваряване, колоната се почиства от пръски. Извършва се контрол на функционалните ѝ размери и контрол на разположението на детайлите ѝ. Ако има някакви отклонения в размерите и в разположението на детайлите, се извършват корекции за отстраняването им.

Извършва се контрол на качеството на изпълнение на заваръчните шевове с ултразвук, като се проверява за наличието на дефекти от типа на пукнатини и шупли.

След като колоната е заварена, може да се пристъпи към следващия етап от технологията за изработването ѝ – механичното ѝ обработване. За установяването на колоната на машината с ЦПУ също се използва специализирано приспособление.

### 3.3. Приспособление за механично обработване на колоната

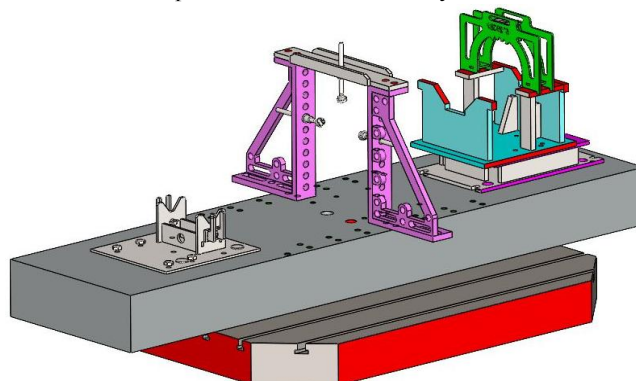
След като е уточнена технологията за сглобяване и технологията за заваряване, се разработва технологията за механично обработване на колоната и се разработва необходимата екипировка.

Механичното обработване ще се извърши на една установка. Описват се операциите и повърхнините, които се обработват, използваните металорежещи инструменти, и режимите на рязане.

За реализиране на технологията за механично обработване на колоната и за да се намали времето за базиране, закрепване и центроване на колоната върху машината, се използва специализирано приспособление за установяването ѝ на машината с ЦПУ.

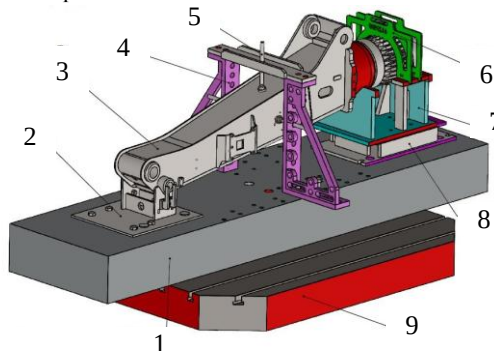
Колоната се базира и закрепва в специализирано приспособление, предварително установено, центровано и закрепено на машината.

Приспособлението е разработено чрез CAD системата SolidWorks и на фиг. 8 е показан общият му вид.



Фиг. 8. Общ вид на приспособлението за механично обработване.

На фиг. 9 е показано приспособлението с всичките му съставни елементи [8, 9, 10, 13, 14, 15]. То осигурява позициониране на колоната в определено положение и с точно определена ориентация.



Фиг. 9. Съставни елементи на приспособлението.

- 1 – Механично обработена маса 2675 mm, 2 – Стойка предна, 3 – Кранова колона, 4 – Ъгъл притискач (2 бр.), 5 – Притискач, 6 – Шаблон за ориентирване на зъбното колело, 7 – Призми, 8 – Основа, 9 – Маса на машината

На приспособлението за механично обработване не се прави допълнителна обработка, защото се използва основата и призмата от приспособлението за сглобяване (те са механично обработени). Предната стойка е изработена от детайли от ламарина с дебелина 8 mm. Детайлите се изрязват на лазер, който постига точност на ряза до 0,1 mm.

Взлите на приспособлението се установяват върху предварително обработена маса, като се центроват с щифтове  $\varnothing 43$  mm. На масата са направени центрови отвори  $\varnothing 43$  mm и проходни резбови отвори M20 за центроване и закрепване на взлите. Направен е центрови отвор по средата на масата на приспособлението  $\varnothing 50$  mm за центроването ѝ към масата на машината. След поставяне на масата на приспособлението върху масата на машината, тя се центрова към нея с ос  $\varnothing 50$  mm, но това не я ограничава напълно. Втората стъпка е масата да се центрова перпендикулярно на шпиндела. Центроването става с измервателен часовник, отклонението на масата трябва да е в рамките на 0,02 mm.

Върху стойката 2 се базира втулката, която центрова колоната по ос X до 0,01 mm. Ограничението по ос Z става с две вертикални планки на страна. Ограничението варира до 0,20 mm. След като колоната е центрована тези вертикални планки се премахват, за да не пречат на обработването на втулката. Те са закрепени с имбусен болт M16.

Чрез използване на приспособлението се гарантира неизменност на положението на обработваните повърхнини.

На фиг. 10 е показана колоната след механичното ѝ обработване.



Фиг. 10. Колоната след механичното обработване.

След приключване на механичното обработване се извършва контрол на изделието.

### 3.4. Контрол на колоната

След като е уточнена технологията за сглобяване, технологията за заваряване и технологията за механично обработване, се разработва методика за контрол на колоната.

След приключване на механичното обработване на колоната се извършва цялостен контрол на обработените повърхнини съгласно документацията на изделието. Контролът се извършва от контрольор по качеството (ОТКК), като за целта се попълва контролна карта. На фиг. 11 е показана контролната карта на колоната.

| SL Industries Ltd                      |                                           | Address: 71 Tutarakan Str. Town: Ruse 7003 |                    | Country: Bulgaria |                | tel.: + 359 82 84 10 14 |  |
|----------------------------------------|-------------------------------------------|--------------------------------------------|--------------------|-------------------|----------------|-------------------------|--|
| QUALITY CERTIFICATE № 8.2.4 ФК 03 / 01 |                                           |                                            |                    |                   |                |                         |  |
| Part № 6129580 / Order №               |                                           |                                            |                    |                   |                |                         |  |
| №                                      | Required dimension according drawing      |                                            | Measured dimension |                   |                |                         |  |
|                                        | [inch]                                    | [mm]                                       | Tacking [mm]       | Weld [mm]         | Machining [mm] | Quality Control [mm]    |  |
| 1                                      |                                           | A=550                                      |                    |                   |                |                         |  |
| 2                                      |                                           | B=1886                                     |                    |                   |                |                         |  |
| 3                                      |                                           | 80 $+0.5$<br>$-0.5$                        |                    |                   |                |                         |  |
| 4                                      |                                           | 80 $+0.5$<br>$-0.5$                        |                    |                   |                |                         |  |
| 5                                      |                                           | 412 $+0.5$<br>$-0.5$                       |                    |                   |                |                         |  |
| 6                                      |                                           | 1317 $+1$<br>$-1$                          |                    |                   |                |                         |  |
| 7                                      |                                           | Φ50H7 $+0.025$<br>$0$                      |                    |                   |                |                         |  |
| 8                                      |                                           | Φ65H7 $+0.030$<br>$0$                      |                    |                   |                |                         |  |
| 9                                      |                                           | 218 $+0.5$<br>$-0.5$                       |                    |                   |                |                         |  |
| 10                                     |                                           | 286 $+0.5$<br>$-0.5$                       |                    |                   |                |                         |  |
| 11                                     |                                           | Φ60F8 $+0.076$<br>$+0.030$                 |                    |                   |                |                         |  |
| 12                                     |                                           | 167 $+0.3$<br>$-0.3$                       |                    |                   |                |                         |  |
| 13                                     | Teeth exactly in the middle of the column | Teeth must be straight in the column       |                    |                   |                |                         |  |
| 14                                     | YES                                       | DA                                         |                    |                   |                |                         |  |
| 15                                     | NO                                        | NE                                         |                    |                   |                |                         |  |
| 16                                     |                                           |                                            |                    |                   |                |                         |  |
| 17                                     |                                           |                                            |                    |                   |                |                         |  |
| 18                                     |                                           |                                            |                    |                   |                |                         |  |

NOTE: COATING: GOOD ☐ SUFFICIENT ☐ POOR ☐ READINGS: THICK ☐ Avg ☐ Min ☐ Max ☐

Production Manager: \_\_\_\_\_  
Welding inspecting coordinator: \_\_\_\_\_  
Quality Controller: \_\_\_\_\_  
Date: \_\_\_\_\_

/Conclusion: General rules for welding manufacturing are accomplished/

Фиг. 11. Контролна карта на колоната.

Контролната карта е направена така, че размерите, които трябва да се изпълнят съгласно чертежа се контролират на всички етапи от производството на изделието. Това са – етап сглобяване, след прихващане на детайлите, след заваряване, след механичното обработване и окончателен контрол.

При констатиране на отклонения на контролираните размери извън допуска, се извършват корекции за отстраняването им съгласно разработената ремонтна технология.

Задължителен при производството на колоната е контролът на качеството на изпълнение на заваръчните шевове. С ултразвук се проверява за възникнали дефекти като пукнатини и шупли в зоните на заваряване. При констатиране на възникнали дефекти в заваръчния шев, се извършват корекции за отстраняването им, съгласно разработената ремонтна технология.

### 3.5. Разработване на управляваща програма

За механичното обработване на колоната е разработена управляваща програма. Използван е програмен език FANUC.

Обработката ще се извърши на машинен център с ЦПУ AWEA BL2018S, показан на фиг. 12.



Фиг. 12. Машинен център AWEA BL2018S.

Програмата е разработена чрез използване на специализиран софтуер CIMCO Edit V6 [20]. Програмата съдържа координатите на характерни точки от изделието, металорежещите инструменти, движенията им и режимите на рязане.

Създаването на управляващи програми чрез използване на софтуер води до избягване на грешките, допуснати в програмирането, които биха довели до разрушаване на инструментите или повреждане на обработваното изделие. Значително се намалява нужното време за създаване на програмите.

Софтуерът решава и обратната задача – от готова управляваща програма, се извеждат координатите на характерни точки от траекторията на движенията, които извършват инструментите, т.е. може да се симулира реалния процес на механично обработване.

### 3.6. Особенности при моделирането на примерните модели в CAD системата SolidWorks

За разработените приспособления е направена пълна конструктивна документация. Процеса на създаването ѝ е автоматизиран, чрез използване на шаблонни файлове на документите, които използват CAD системата SolidWorks.

При създаването на примерните модели на детайлите и на сглобените единици са използвани шаблонни файлове за Part и Assembly документите. За всеки един тримерен модел на детайл или сглобена единица, от меню File Properties, в прозореца Summary Information, в секцията Custom се избират необходимите атрибути. При разработването на конструктивната документация са използвани шаблонни файлове за Drawing документите, които представляват стандартните чертожни формати – A4, A3, A2, A1 и A0 [4, 5, 6, 7, 11, 12].

В шаблона на всеки един тримерен модел на детайл или сглобена единица се задава необходимата информация чрез атрибути – потребителски и служебни. Тази информация е нужна при разработването на конструктивната документация, тъй като излиза автоматично в таблиците на чертежите. Информацията от атрибутите може да се използва за управление на документацията при налична PDM система, както и от другите отдели във фирмата при налична ERP система.

#### 4. Заключение

Въз основа на направеното по представената разработка, може да се направят следните изводи:

1. Разработена е технология и приспособление за сглобяване на колоната. Приспособлението осигурява точно позициониране на детайлите на колоната, не е сложно за манипулация, с цел да се избегнат всякакви грешки от субективен характер.

2. Разработена е технология и приспособление за механично обработване на колоната. Чрез използване на приспособлението се намалява времето за базиране, закрепване и центроване на колоната върху машината.

3. Приспособленията за сглобяване и механично обработване могат да се използват за изработване на различни видове кранови колони. За целта е необходимо само да се заменят определени базирани и закрепващи елементи и възли.

4. Направена е пълна конструктивна документация на разработените приспособления. Процесът на създаване на конструктивната документация е автоматизиран чрез използването на шаблонни файлове за Part, Assembly и Drawing документите.

5. Така създадената база данни може да се използва при решаване на сходни проблеми при подобни изделия.

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# МОДЕЛ НА СЕИЗМИЧЕН ПИЕЗОЕЛЕКТРИЧЕСКИ СЕНЗОР

Доц. д-р Бойчев Я. Д.  
ИМСТЦХА - БЪЛГАРСКА АКАДЕМИЯ НА НАУКИТЕ, 67 ул. Шипченски проход, 1574 София, България

## A MODEL OF SEISMIC PIEZOELECTRIC SENSOR

Associate professor Boychev Y. D.  
Institute of Metal Science Equipment and Technologies with Hydroaerodynamics Centre "Acad. A Balevski" at Bulgarian Academy of Sciences, 67 Shipchenski Prohod Street, 1574 Sofia, Bulgaria

E-mail: [y\\_boichev@abv.bg](mailto:y_boichev@abv.bg)

**Abstract:** One of the most common seismic sensors are piezoelectric, which are used to monitor the seismic situation in industrial areas, to build security systems, to monitor landslides, etc. The main task in their design and operation is to determine the relation between mechanical parameters of the sensor and its response to seismic impacts. To solve this problem, a mechanical-mathematical model of a piezoelectric sensor and an approach for determining its response to vibration with a frequency of 1 Hz to 200 Hz and applied force with a maximum acceleration of 0.1 g are proposed.

**Keywords:** piezoelectric sensors, seismic waves, mathematical modeling

### 1. Увод

Придвижването на наземната военна техника е съпроводено с шум и вибрации. Акустичните емисии са свързани с вида на двигателя, геометрия на ауспуха, вид на гумите, пътната настилка. Сеизмичните сигнали зависят от типа на транспортното средство, неговото окачване, начина на задвижване (колесно или верижно) вида на почвата и пр. Разпространението на генерираните сеизмични вълни в почвата в зависимост от нейния вид е разгледано в [1-7]. Потенциалът на повърхностните и Релееви сеизмични вълни за идентификация на военна техника са изследвани от Succì et al. [8], Tian et al [9].

При разработването на системи за автоматична детекция и идентифициране на наземна военна техника, базирани на анализ на генерираните от нея сеизмични вълни, основен елемент са пиезоелектрическите сензори. Поради това, както и поради усъвършенстването на технологиите за обработване и анализиране на първичната информация получавана от сеизмичните сензори е изключително важно да се познават техните амплитудно-честотни характеристики (АЧХ). За избягване на трудоемките натурни тестове, както и за разширяване на диапазона на сеизмичните въздействия е направен механо-математичен модел на пиезоелектричен сеизмичен сензор и получените резултати са посочени в дадената работа.

### 2. Цел на изследването

Целта на изследването е да се определи влиянието на физикометричните характеристики на разработвани в ИМСЦХА-БАН виброакустични сензори върху регистрираните от тях данни за повърхностните сеизмични вълни, генерирани от различни видове наземна техника. За оценка е създаден механо-математичен модел на виброакустичен сензор.

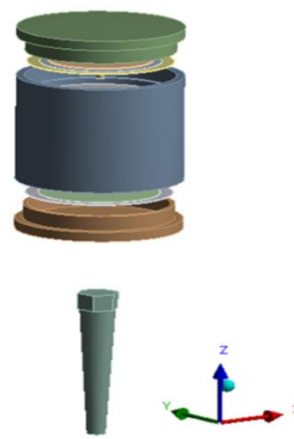
### 3. Механо-математичен модел

Разработеният модел се базира на решаването на последователност от гранични задачи свързани със следните типове анализ:

- Статичен анализ на сензора при натоварване от собствено тегло (т.е. при липса на външни въздействия) и фиксирано закрепване на основата;
- Модален анализ за определяне на първите 30-50 бр. собствени честоти при фиксирано закрепване на основата;
- Спектрален анализ на реакцията на модела на вибрационно въздействие с честота 1Hz до 200 Hz и акселерограма приложена сила по оста с ускорение 0,1g.

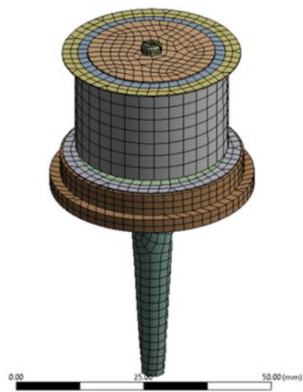
За оценка на влиянието на сеизмичните колебания върху амплитудно-честотните характеристики на сензорите беше извършено моделиране, базирано на 3D модел на пиезо електрически сензори с използване на програмен продукт ANSYS Mechanical. Предназначен е за решаване на широк клас задачи в механиката и широко използван при проектиране и разработване на съвременни продукти в индустрията работещи при сложни механични натоварвания, вълнови въздействия, електромагнитни въздействия и др.

За нуждите на моделирането е разработен геометричен модел (Фиг. 1) като е използвана екзекутивната документация на сензора разработен в ИМСЦХА – БАН която съдържа чертежи на конструкцията. Направени са някои опроставяния (основно свързани с пренебрегване на малки закръгления в отделните детайли), които не водят до отклонение в резултатите, но облекчават изготвянето на крайно-елементен модел.



Фиг. 1. Геометричен модел на пиезоелектрически сензор

Моделът е съобразен с възможностите на метода на крайните елементи (МКЕ) и е приложен за реализация в комерсиален пакет ANSYS по МКЕ, като са използвани крайни елементи тип Solid KE Solid Shell KE за тънкостенните мембрани (Фиг. 2). Използвани са крайни елементи с 6 степени на свобода.



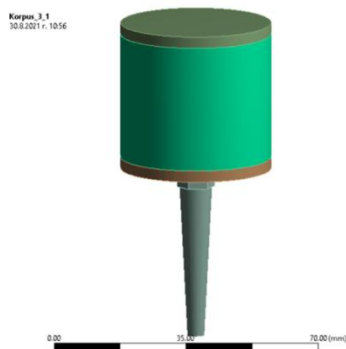
Фиг. 2. Крайно-елементен модел на пиезоелектричен сензор използващ представяне чрез тънкостенни мембрани.  
Брой възли – 31363, брой елементи – 9328

Материалните характеристики на отделните детайли са взети от документацията на продукта, а данни за модула на еластичност, коефициент на Поасон и плътност на материала се изменят за четири различни конструктивни материала - на корпуса, на масата и на еластичната мембрана с пиезоелемента.

За целите на моделирането са необходими входни данни отнасящи се до плътност, еластичен модул, и коефициент на Поасон, които са дадени в Табл. 1.

Табл.1 Входни данни

| Детайл                 | Плътност<br>[ $kg\ m^{-3}$ ] | Еластичен<br>модул<br>[Pa] | Коефициент на<br>Поасон |
|------------------------|------------------------------|----------------------------|-------------------------|
| Корпус (фиг.3)<br>D16T | 2770                         | 7.2E+10                    | 0.3                     |
| Капак (фиг.4)          | 7850                         | 2E+11                      | 0.3                     |
| Маса (фиг.5)           | 10660                        | 2E+11                      | 0.3                     |
| Мембрана<br>(фиг.6)    | 8400                         | 9E+10                      | 0.31                    |



Фиг. 3 Корпус (зелено)



Фиг. 4 Капак (зелено)



Фиг. 5 Маса (зелено)

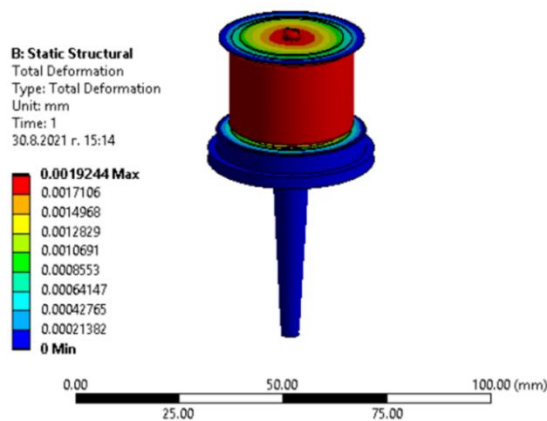


Фиг. 6 Мембрани (зелено)

Разработеният модел дава възможност за адекватно моделиране на сензорите при различни акселерограми, които са резултат от различни тежки машини при движението им върху различни терени. Успешно могат виртуално да се изследват различни конструктивни решения за сензорите при различни сеизмични въздействия. Сеизмичните въздействия се задават като акселерограми в честотният спектър.

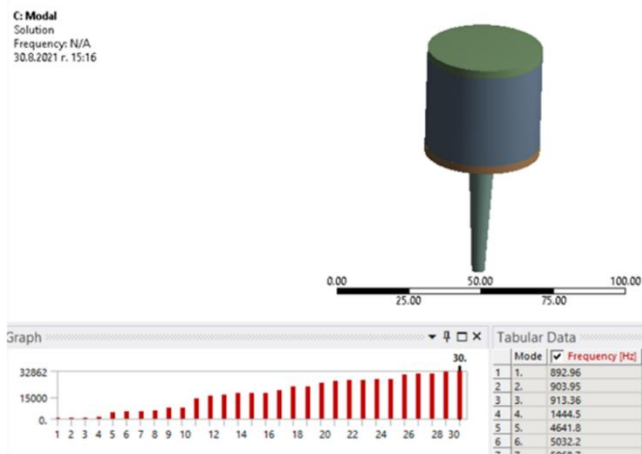
#### 4. Резултати

За валидиране на методиката и модела за проведени серия от числени симулации. Резултати от симулациите са представени на следващите фигури:



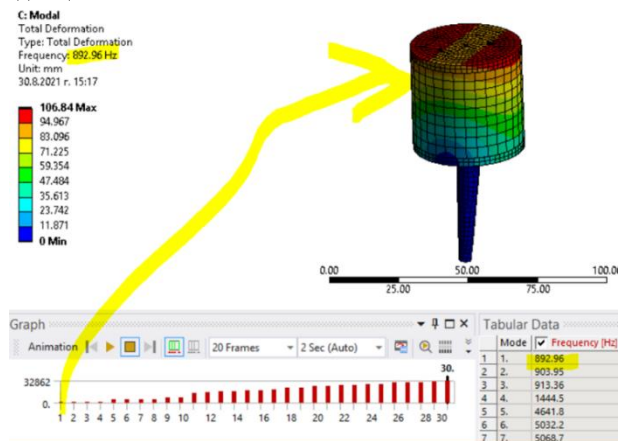
Фиг. 7 Статичен анализ. Деформирано състояние от собствено тегло

Деформираното състояние от собствено тегло позволява да се оцени провисването на мембраните при натоварване от собствено тегло без сеизмична въздействие. Това е състояние на “покой” на сензора. При мембрани с различна коравина може да се оцени дали това провисване е допустимо за функционалното предназначение на продукта.

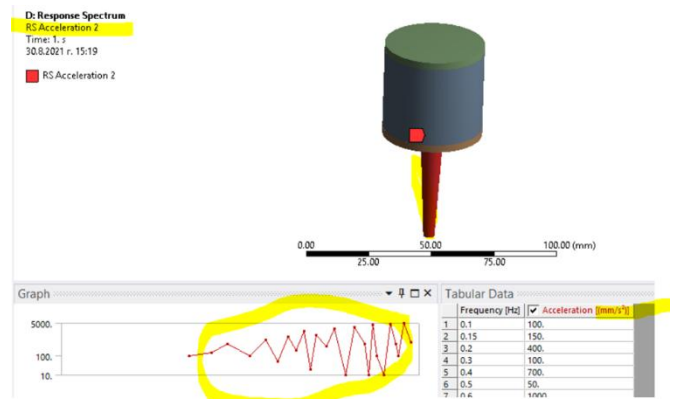


Фиг. 8 Модален анализ. Графично са представени 30 собствени честоти от анализа.

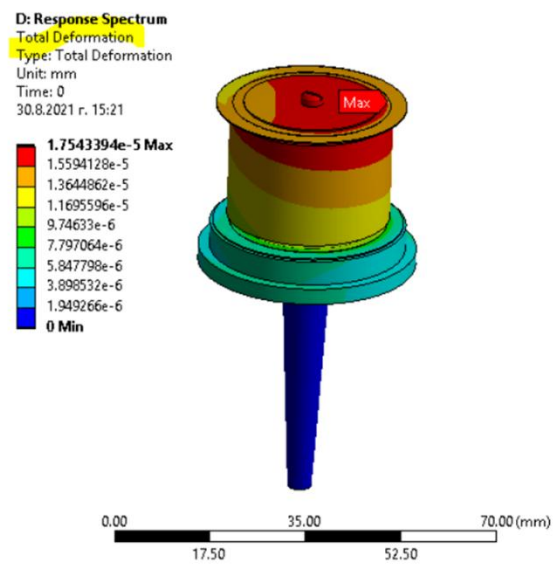
Модалният анализ е необходим за получаването на достатъчно собствени честоти, които се използват при последващия анализ.



Фиг. 9 Модален анализ. Първа собствена честота

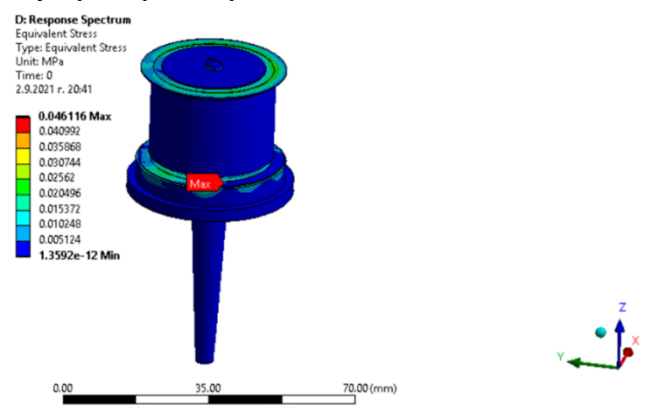


Фиг. 10. Спектрален анализ при зададена тестова акселерограма и гранични условия съответстващи на закрепването на терен на сензора



Фиг. 11. Спектрален анализ SRSS method. Точката(зоната) на максималната деформация

Използван е SRSS(square root of the sum squares) method за сумиране при спектралният анализ.



Фиг. 12. Спектрален анализ. Напреженията в отделните елементи на сензора.

#### 4. Заключение

Разработеният изчислителен модел дава възможност за адекватно моделиране на сензора при различни акселерограми, които са резултат от различни тежки машини при движението им върху различни терени. Успешно могат виртуално да се изследват различни конструктивни решения за разработване.

Моделът позволява да се параметризира, което улеснява оптимизация на конструкцията при различни критерии дефинирани от потребителя. Използването на съвременни методи за проектиране позволяват скъсяване времето от проектно решение до търговската реализация. Успоредно с това, могат да се анализира влиянието на физикометричните характеристики на виброакустичните сензори разработвани в ИМСТЦХА – БАН върху регистрираните от тях данни.

Резултатите от това изследване са насочени и към изпълнение на Работен пакет 2 „Интелигентни системи за сигурност“ на проект BG05M2OP001-1.002-0006 „Изграждане и развитие на Център за компетентност “Квантова комуникация, интелигентни системи за сигурност и управление на риска (Quasar)“, който е получил финансиране от Европейския фонд за регионално развитие чрез Оперативна програма „Наука и образование за интелигентен растеж“ 2014-2020.

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# Analysis of specific absorption rate of electromagnetic field, generated by smartphone

Magdalena Garvanova  
University of Library Studies and Information Technologies, Sofia, Bulgaria  
m.garvanova@unibit.bg

**Abstract:** This article analyses the Specific Absorption Rate (SAR) and outlines the thermal effect in depth of the human head. SAR is the most important dosimetric value when the human body is irradiated with signals in the radio frequency range. For this purpose, a computer model is created using COMSOL Multiphysics software, taking into account the specific characteristics of the human head and the parameters of a working smartphone. This model is suitable for studying the effects of radio waves on humans.

**Keywords:** Specific Absorption Rate, computer model, thermal effect

## 1. Introduction

Creating a model to study the process of absorbing electromagnetic energy, emitted by a mobile phone and the subsequent thermal effect in the human body, is crucial in analyzing the effects, caused by the use of wireless smart devices. The purpose of the model is to facilitate the research and understanding of the processes leading to an increase in the temperature of the human head as a result of the absorbed radiation, emitted by a mobile device [1].

Biophysical effects such as tissue heating and nervous stimulation of the body by strong electromagnetic fields are well-known to scientists, as radio frequency radiation is not ionizing and therefore cannot directly cause damage to human DNA [2], [3]. According to researchers, the most significant effect of radio waves is the heating of the tissues of the human head. Harmful thermal effects cause cataract, but there are also non-thermal effects that are controversial [4]. It should be noted that not all biological effects necessarily lead to negative consequences on human biophysical conditions.

In addition to the characteristics of the electromagnetic fields, the thermophysiological processes of the human body and the parameters of the environment such as the specific heat capacity of the tissues, the cooling of the blood circulation and the ambient temperature should be taken into account. The aim of the present study is to create a computer model of the Specific Absorption Rate of the electromagnetic field, generated by a working mobile phone. Its verification will be carried out through author's experimental research, done via thermal images from a thermal imaging camera and electroencephalogram – EEG.

## 2. Specific Absorption Rate

As already mentioned, SAR is a characteristic that describes the absorption of the power of the electromagnetic field that propagates in different types of tissues [5]. Local SAR is defined as the loss of power  $dP_1$ , absorbed in an infinitesimally small mass  $dm$ , and can be described by the following equation:

$$SAR = \frac{dP_1}{dm} = \frac{\sigma_{eff} E_{rms}^2}{\rho} = \frac{J_{rms}^2}{\rho \sigma_{eff}} \quad (1)$$

where  $E_{rms}$  and  $J_{rms}$  are the root mean square values of the electric field strength and the density of electricity, respectively  $\sigma_{eff}$  is the effective conductivity of human brain tissue and  $\rho$  is the tissue density. As it is known, the energy from the electromagnetic fields in the radio range is absorbed into the tissue of the human body and transformed into heat. This leads to another definition of SAR, namely:

$$SAR = c_p \frac{\Delta T}{\Delta t} \quad (2)$$

where  $c_p$  is the specific heat capacity of the tissue and  $\Delta T$  is the change in temperature over a period of time  $\Delta t$ .

A distinction between the instantaneous SAR and the permissible SAR should be made, where an average value is measured for a given mass of tissue and a specified period of time.

The increase of the temperature can be estimated by the equation [5]:

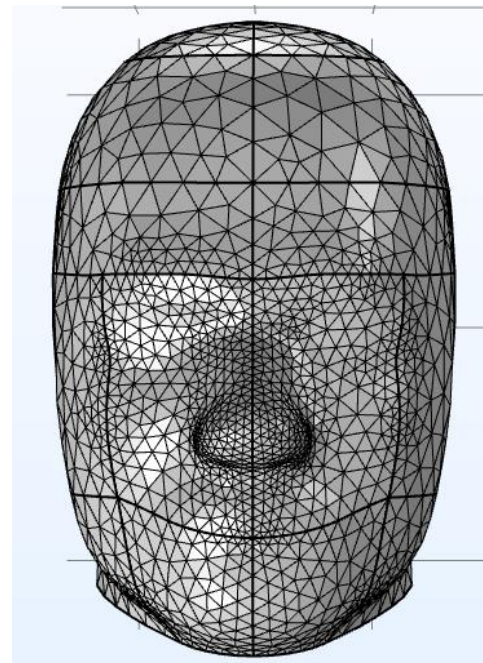
$$\Delta T = \frac{SAR \tau}{c_p} (1 - e^{-t/\tau}) \quad (3)$$

where  $\tau$  is the thermal time constant. The created computer model examines the interaction of heat transfer between tissues due to the presence of two heat sources. It does not take into account the influence of vascular geometry on heat transfer.

## 3. Computer model of SAR

The created model is a combination of a model of a human head and a model of an antenna built into a mobile phone. The human head pattern includes layers of varying thickness, skin, skull, and brain tissue patterns. This model allows to simulate the processes of penetration and absorption of electromagnetic energy from the tissues of the head, as well as to observe the thermal processes, caused by high-frequency fields. The characteristics of the human head and the parameters of the biological tissues, used in the model, are as close as possible to the real ones.

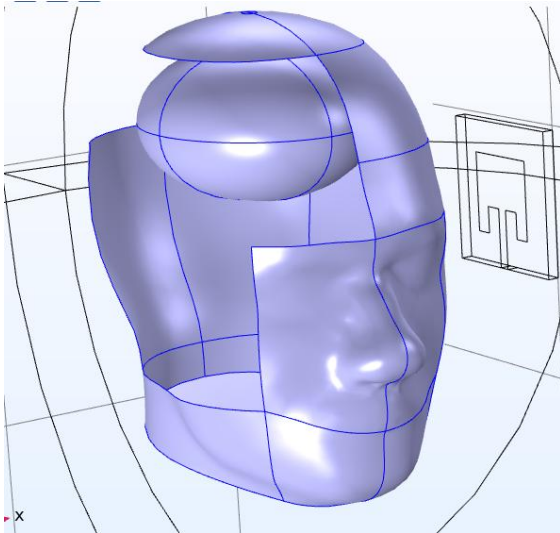
The design and geometry of the human head are provided by the IEEE, and the model was imported into the COMSOL Multiphysics software from a file named `sar_in_human_head.mphbin` (Fig. 1).



**Fig. 1.** Human head model

The source of electromagnetic radiation is a model of a mobile phone that was added manually (Fig. 2). The electromagnetic parameters of the biological tissues of the human head are modelled

using an interpolation function that applies the characteristics of a particular tissue inside the human head.



**Fig. 2.** Human head model and GSM

The output data for this function is directly taken from a system file that is created using multiple magnetic resonance images of human head sections. The model is obtained from 109 images, each with a size of 256 by 256 pixels. The `sar_in_human_head_interp.txt` system file can also be used to visualize the dielectric permittivity and perfusion inside the head.

The pattern of propagation of the electromagnetic field is simulated by a mobile phone, located one centimeter on the left side of the human head (Fig. 2). The distribution of the strength of the electric field  $E$  is obtained by solving the equation [6]:

$$\nabla \times \frac{1}{\mu_r} \nabla \times E - k_0^2 \varepsilon_r E = 0 \quad (4)$$

where  $\mu_r$  is the relative permeability,  $k_0$  is a wave vector and  $\varepsilon_r$  is the permittivity of the tissues.

The depth of penetration of the electric field into the biological tissue of the human head depends on the frequency of electromagnetic waves and is determined by the formula:

$$\delta = \frac{1}{\omega} \left( \frac{\varepsilon_r \mu_r}{2} \left( \sqrt{1 + \frac{\sigma_{eff}^2}{\omega^2 \varepsilon_r^2}} - 1 \right) \right)^{-1/2} \quad (5)$$

At the frequency of the electromagnetic field at  $f = 900 \text{ MHz}$  the depth of penetration for different tissues of the human body is shown in Table 1.

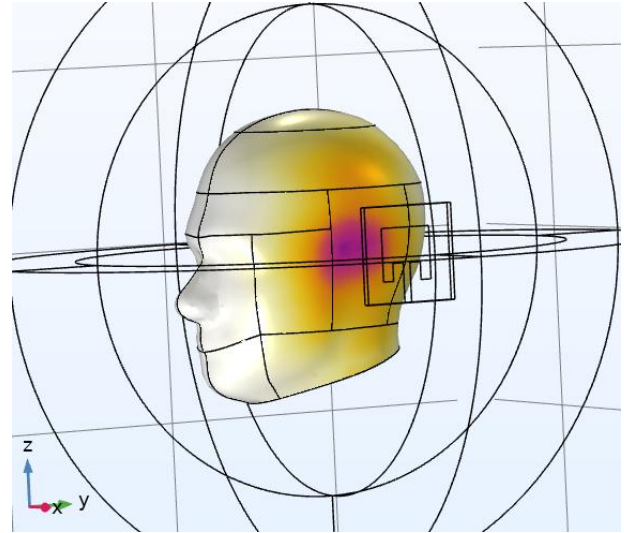
**Table 1.** Depth of penetration of the electromagnetic field

| Tissue              | Skin | Fat   | Muscle | Skull | Brain |
|---------------------|------|-------|--------|-------|-------|
| $\delta[\text{mm}]$ | 40.2 | 164.5 | 42.3   | 131.8 | 47.5  |

#### 4. Results

The simulation of the created computer model in the environment of COMSOL Multiphysics allows to estimate SAR on any shape and tissue of the human body. The local SAR value in a human head at an electromagnetic field frequency of 900 MHz is demonstrated in Fig. 3. When a human head is irradiated with an electromagnetic wave, it penetrates the tissue and gives part of its energy to the head or the wave is absorbed into the head tissue. Electromagnetic energy affects the particles in the tissue due to the

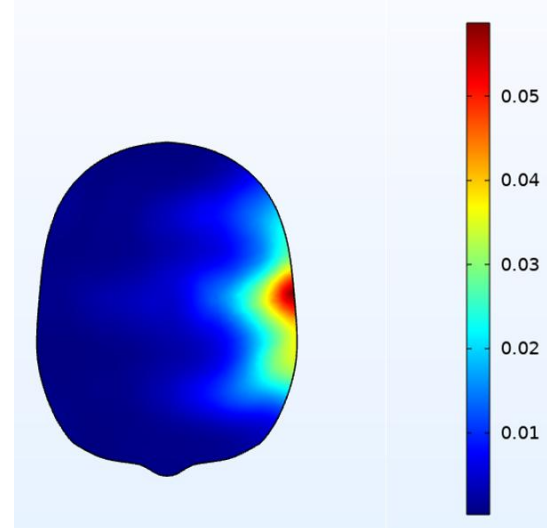
electrical and magnetic components of the electromagnetic field. SAR values are higher in the surface layers of the human head.



**Fig. 3.** Human head SAR model

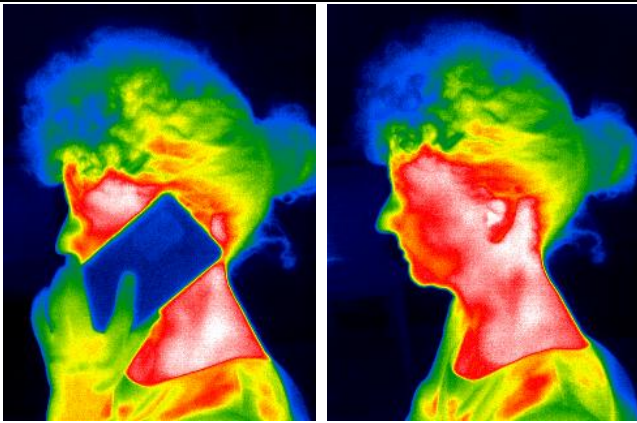
The created computer model calculates only the local value of the SAR parameter. The maximum value of the local SAR is always higher than the maximum value of the average SAR value.

The propagation of electromagnetic waves in biological tissues takes the form of absorbed energy. Most of the absorbed energy is converted into heat, which leads to an increase in temperature in biological tissues. The thermal effect, obtained from electromagnetic radiation of a mobile phone, is most pronounced on the surface of the head skin (most often the area of a person's ear) [2]. The increase in temperature depends on the physiological properties of the tissues (blood perfusion) and can be calculated using a biothermal equation that takes into account heat loss due to blood circulation in the human body. The thermal effect on and in the human head for a frequency of 900 MHz is shown in Fig. 4.



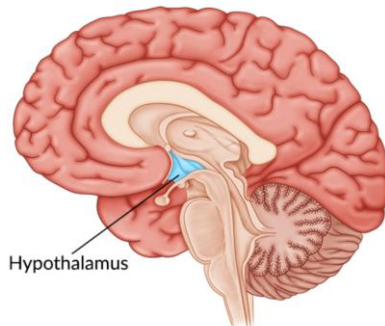
**Fig. 4.** Thermal effect in the depth of the head

It can be seen that the temperature is highest in the area near the source of electromagnetic radiation, where the mobile phone is positioned. The temperature of the brain tissue rises about  $0.6^\circ\text{C}$  and decreases inside the head. The validity of this computer model was experimentally confirmed using a thermal imaging camera and EEG. The results of the experiment for the distribution of the temperature on the surface of the head after using a mobile phone are outlined in Fig. 5.



**Fig. 5.** Temperature distribution at the surface after 15-minute phone call

Temperature distribution at the surface of human head is in correlation with ambient temperature while the temperature inside biological tissues does not depend on it. It is interesting to know that the microwave change in the temperature of the human head can lead to an increase in the temperature of the hypothalamus (Fig. 6) by about 0.2-0.3°C, which may cause health problems [7].



**Fig. 6.** Hypothalamus

The hypothalamus has some basic functions such as controlling body temperature, hunger, thirst, libido, heart rate, releasing hormones from the pituitary gland, producing digestive juices, balancing body fluids, etc. As the body's various systems send signals to the brain, they alert the hypothalamus to any imbalances. As a result, the hypothalamus responds by releasing the right hormones to balance the body.

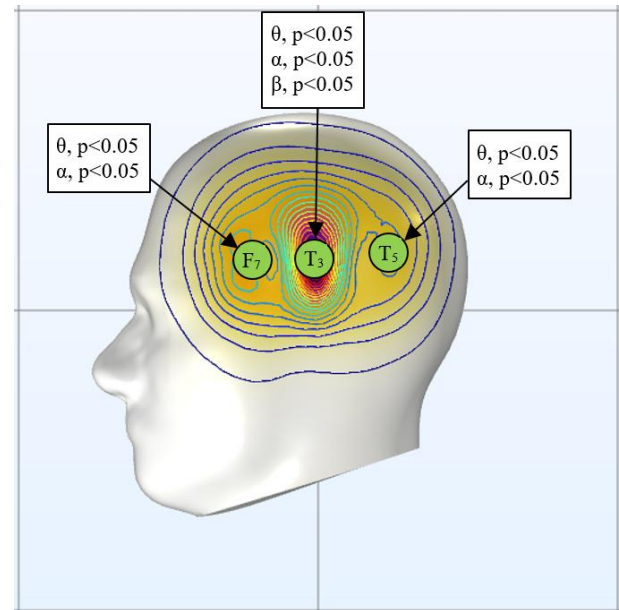
The influence of mobile phone exposure on the nervous response was studied in [8], [9] by analysing electrophysiological measurements of brain wave activity. EEG test measurements were taken in two situations when the participant was not using and when using a mobile phone (Fig. 7).

A series of paired samples t-test analysis on spectral power, conducted over the EEG spectral band delta –  $\delta$  (2-4 Hz), theta –  $\theta$  (4-8 Hz), alpha –  $\alpha$  (8-13 Hz), and beta –  $\beta$  (13-32 Hz) for all points was applied. The results with p-values less than 0.05 indicate statistically significant differences in EEG power in T3 ( $\theta$ ,  $\alpha$ , and  $\beta$ ), T5 ( $\theta$  and  $\alpha$ ), and F7 ( $\theta$  and  $\alpha$ ) points as shown in Fig. 8.

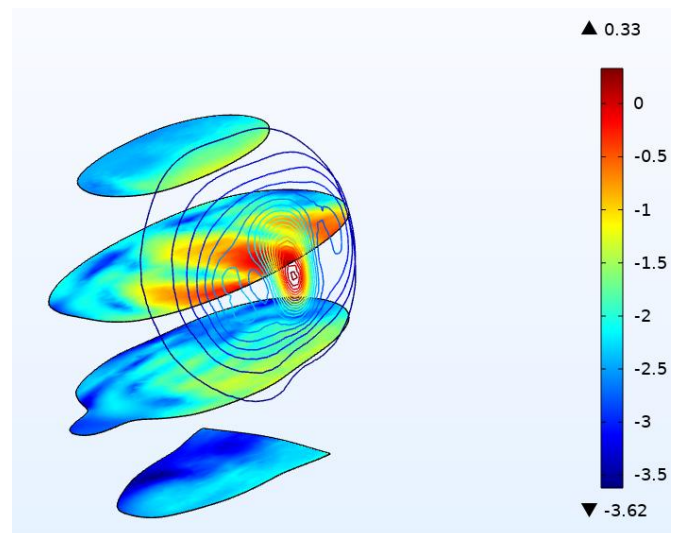
In the points, which are in the immediate vicinity of the GSM device with highest absorption of the electromagnetic field, a significant change of the signal spectrum is observed (Fig. 8), as revealed our previous research [8], [9], [10].



**Fig. 7.** A participant during the experimental procedure



**Fig. 8.** SAR model and points of change in human brain activity



**Fig. 9.** Distribution of SAR in the human head

A section of the head at the level of maximum absorption (Fig. 9) shows coincidence of the sites of change in brain activity with the maximum SAR levels. The change in brain activity due to prolonged use of a mobile phone is a prerequisite for loss of concentration, memory problems, feelings of fatigue, anxiety, and intense affective states [11], [12].

## 5. Conclusion

This article examines the Specific Absorption Rate (SAR), as well as the thermal effect, caused by the use of a mobile phone. The study is conducted using a computer model via COMSOL Multiphysics software, which takes into account the specific characteristics of the human head and the parameters of a mobile phone. The advantage of the simulation compared to the thermal imaging camera is possibility to analyse temperature distribution through head and inside biological tissues. The validity of the created computer model is confirmed by a series of experimental studies using thermal images and EEG.

The results of research show that the temperature to which the tissues of the head are heated depends on the amount of energy, absorbed by the human head. The resulting high SAR values on the skin surface are not biologically hazardous, as peripheral blood circulation and thermal conductivity to the environment effectively cool the skin. More worrying is the fact that prolonged use of a mobile phone leads to a change in the brain activity of the user. Our future research efforts are oriented towards creating a comprehensive model for studying the risk factors of excessive use of smart technologies among children and adults.

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# An evaluation of wind energy potential in Topoja area, Albania.

Elena Bebi<sup>\*</sup>, Majlinda Alcani<sup>2</sup>, Lorenc Malka<sup>2</sup>, Artan Leskoviku<sup>3</sup>  
 Department of Production and Management, Faculty of Mechanical Engineering,  
 Polytechnic University of Tirana, Albania<sup>1</sup>  
 ebebi@fim.edu.al  
 Department of Energy, Faculty of Mechanical Engineering,  
 Polytechnic University of Tirana, Albania<sup>2</sup>  
 Renewable Energy Directorate,  
 National Agency of Natural Resources, Albania<sup>3</sup>

**Abstract:** Climate change and efforts to reduce greenhouse gas emissions, have significantly increased interest globally in implementing technologies utilising renewable energy sources such as wind energy. This rise in interest it also in countries in which there is a lack of data showing longitudinal measures of wind speed. Balkan Wind Atlas was developed from Sander&Partner Institute in October 2014. It offers wind maps that are suitable to identify the windiest areas covering a surface of 325 000 km<sup>2</sup> and a database for secured planning of wind energy for Albania, Kosovo, North Macedonia, Montenegro and Serbia. This study investigated the high level of consistency of empirical wind measurements conducted on-site, compared to the ones offered in Balkan Wind Atlas. We also explore the ways of utilising the atlas through the WasP software to study wind potencial even in those areas where there are no on-site measurements, thus lowering costs for investments in wind farm projects. The evaluation of the wind energy potential in Topoja area was taken as a case study.

**Keywords:** WIND ENERGY DATA, BALKAN WIND ATLAS, WASP, ALBANIA.

## 1. Introduction

Renewable power generation is becoming the default economic choice for new capacity. Solar PV and onshore wind projects are able to produce power for less than the cheapest new fossil fuel-fired cost project. It has become clear that renewables will become the backbone of the electricity system and help decarbonize electricity generation [1]. For the most of world's population, electricity production from new renewables is more cost effective than from new coal-fired power plants [2]. 2020 was the best year in history for the global wind industry with 93 GW of new capacity installed, driven by China and the US [3]. For onshore wind projects, the global cumulative installed capacity between 2010 and 2020 grew from 178 GW to 699 GW [1].

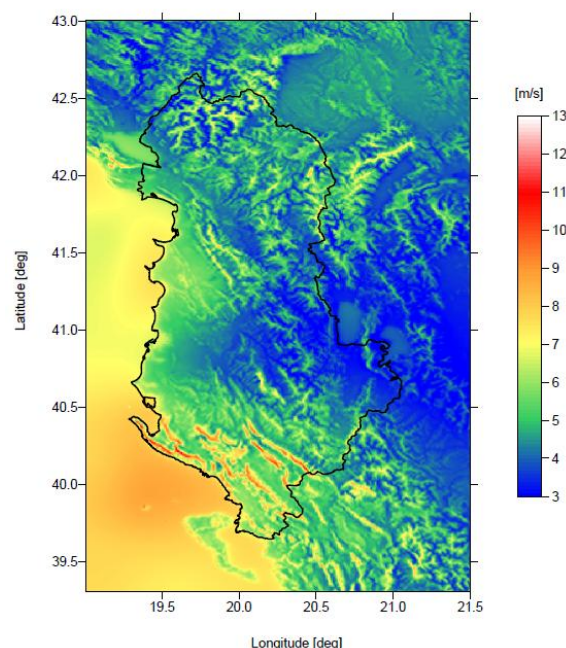
In most of Europe, the increase in utilization of wind power for electricity has resulted in increased economic stability against fluctuating fuel prices. Renewable energy sources are able to reduce the European Union's dependence on foreign energy imports, also meeting sustainable objectives to tackle climate change and to enhance economic opportunities[4].

One of the major economic advantages of harvesting wind energy in Albania is the reduction in the country's economic dependency on hydropower for producing electricity. Based mostly on one source of the energy resources, Albania has been facing severe energetic crisis which express visibly the high scale of the hydrological risk in the stability of electricity generation for the uninterrupted supply of customers of the Electricity System[5]. Albanian government has considered the promotion of renewable energy use as an important tool of energy policies for the increase of the security for energy supply, economic development, energy sector sustainability and environment protection[6].

But the lack of long-term measurements by the standards required by the assessment of wind farm projects, is among other reasons, that has hindered the development of this energy sector in Albania. The history of wind speed measurements in Albania is mainly related to meteorological studies for weather forecasting or for air and naval services. Many researchers have noted that a major barrier to deployment in many regions of the world is a lack of reliable and detailed wind resource data[7]. On the other hand, wind data analysis and accurate wind energy potential assessment is critical for proper and efficient development of wind power application and is highly site dependent. The wind potential assessment of a site requires the knowledge of statistical characteristics, prediction of wind speed, persistence, availability, diurnal, and monthly variation, etc.[7].

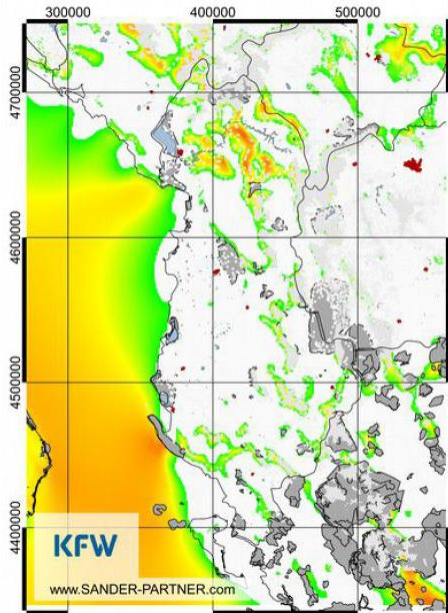
The most serious effort for wind resource assessments in Albania was the study conducted from CETMA Consortium in

cooperation with Department of Physics in University of Genova, based on an agreement between the Albanian government and the Italian Ministry for Environment, Land and Sea (IMELS), on 30 July 2008 in Tirana[8]. Figure 1 shows the map of average wind distribution over the territory of Albania assessed at a height 50m above the ground level.



**Fig. 1** The map of the distribution of average wind speed at 50m above ground level, over Albanian territory.

And undoubtedly an important development was the publication in October 2014, with the support of the Bank KfW which has financed many energy projects in Albania, the Wind Atlas for Balkan Peninsula, by the Sander & Partner Institute(Fig.2). This atlas offers wind maps that are suitable for identifying windy areas. It includes wind data for more than 40,000 locations in the form of 30-year time series data with a 3km resolution, including wind speed and direction, temperature and air pressure, and solar irradiation. The Wind Atlas for the Balkans offers wind maps and data for it covering the territories of Albania, Kosovo, North Macedonia, Montenegro, and Serbia. The weather model calculates the wind under the influence of the regional topography and other regional features like vegetation or the water exchange from the soil to the atmosphere(Wind-Atlas-Balkan, Workshop, October 23, 2014, Tirana).



**Fig. 2** Map of areas with wind energy potential, good wind conditions, no conflicts with inhabited areas and respecting natural parks.

In this study, data collected over a 15-month period (March 19, 2013, to June 14, 2014) in Mamaj, Tepelena, were compared with those obtained from Wind Balkan Atlas for the closest point to this area in 2013 which make up the last year of the atlas data. By this comparison was investigated the high level of consistency of empirical wind measurements conducted on-site, compared to the ones offered in Balkan Wind Atlas. Furthermore, using the longitudinal data from Balkan Wind Atlas is very useful for investors. By using the WAsP (Wind Atlas Analysis and Application Program) software, investors can develop wind potential distribution maps (resource grids) at 50m above ground level for the site of interest. The initial evaluations of the wind conditions are typically based on numerical products, such as wind atlases, which generally provide convenient, fast, and easy access to estimations of the characteristics wind conditions at a site[9].

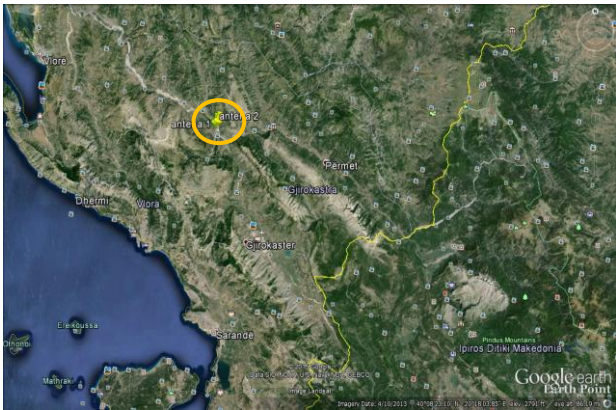
Wind Atlases are produced for many countries including Egypt, South Africa, Finland, Germany, Greece, Russia, Iceland[9], Oaxaca(Mexico)[10], Armenia[11], etc. They are used for assessment of wind energy potential in several topographic regions all over the world, for determining the location of areas with high wind potential and for setting up wind farms, such as: Eritrea[12], Thailand[13], Spain[14], Qatar[15], etc.

The goal of this study is to raise awareness to both Albanian and foreign investors on the usefulness of Balkan Wind Atlas’s data for assessing wind potential in areas of interest. This data would help investors find the appropriate location of anemometric towers to run on-site measurements of wind speed. Furthermore, considering that areas of interest for Albanian investors are relatively small, as well as that the Energy Regulatory Authority(ERE), approved Feed-in-Premium tariffs of 76€/MWh for wind farms up to 3MW installed power on July 27<sup>th</sup>, 2017, there is a high incentive for wind farm engineers to utilize data from the Balkan Wind Atlas as well as the WAsP software. Making use of these tools would allow them to correctly choose the exact location and type of wind turbines to produce the highest capacity factor, as well as cut down on significant costs.

**2. Materials and Methods**

In the area of interest in setting up a wind farm, in Mamaj, north of Tepelenë, Albania (Fig.3), measurement campaign started on March 19, 2013, and lasted until June 14, 2014. Wind speed data were acquired through anemometers with 3 cone shaped cups installed on 40m, 50m and 60m height and averaged every 10min.

Wind direction was acquired through wind vanes mounted at 45m and 55m height. Air temperature was measured on height 13m.



**Fig. 3** The site of Mamaj, Tepelenë, Albania

The meteorological measurement data were obtained from 2 measurement stations installed in two separate towers(Valley and Hill tower) with a height of 60m each and in a distance of about 800m from each other(Fig. 4). The geographical details of the data measurement stations are given in Table 1. State-of-the-art wind data analysis tool Windographer(2011) was used for obtaining the statistical summaries of all the data: diurnal and seasonal variation of the wind speed, seasonal Weibull shape and scale parameters, wind power density, vertical wind shear, etc.



**Fig. 4** The location of the measurement towers in the site of Mamaj, Tepelenë, Albania

**Table 1:** Geographical Details of Data Collection Station.

| Station | Latitude (°) | Longitude (°) | Altitude (m) |
|---------|--------------|---------------|--------------|
| Valley  | 20.0166E     | 40.3233N      | 158.3        |
| Hill    | 20.0090E     | 40.3090N      | 311          |

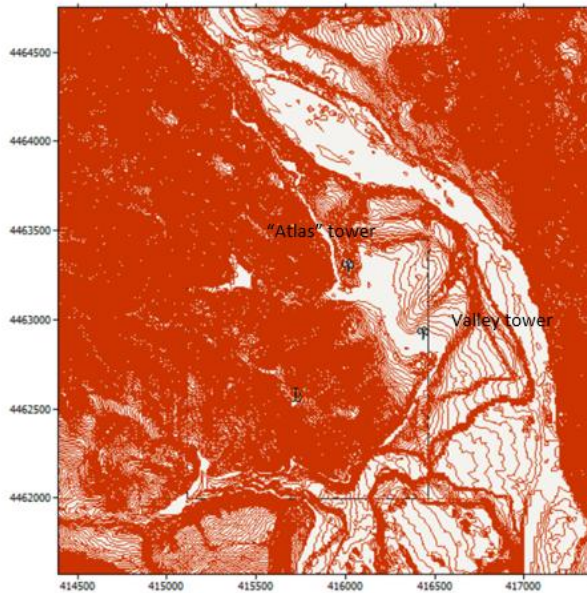
The data collected for the period March 2013-December 2013 from the anemometric tower installed in the valley, were compared to those extracted from the hypothetical anemometric tower of Balkan Wind Atlas for this area in 2013.

Geographical coordinates of the location of these two towers are shown in the Table 2. The distance between them is 580m.

**Table 2:** Geographical Details of Data Collection “Atlas” and Valley tower.

| Station | Latitude (°) | Longitude (°) | Altitude (m) |
|---------|--------------|---------------|--------------|
| Valley  | 20.0166E     | 40.3233N      | 158.3        |
| “Atlas” | 20.0120E     | 40.3160N      | 147          |

Figure 5 shows the reciprocal position of Valley tower and the hypothetical anemometric “Atlas” tower which is closest point to this area obtained from Balkan Wind Atlas.



**Fig. 5** The location of the measurement towers in the site of Mamaj, Tepelenë, Albania

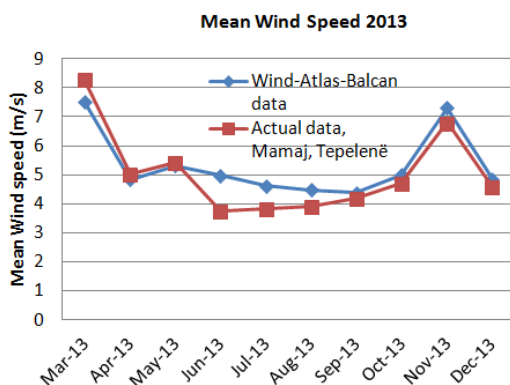
In Table 3 are shown the monthly mean wind speed values for the period 19 March 2013-December 2013, based on measurements carried out by the anemometric tower in the valley as well as that hypothetical "Atlas".

**Table 3:** Mean monthly wind speed respectively by the "Atlas" and Valley tower for the period March 2013-December 2013.

|                         | Mean wind speed (m/s) by "Atlas" tower | Mean wind speed(m/s) by Valley tower |
|-------------------------|----------------------------------------|--------------------------------------|
| March 2013              | 7.52                                   | 8.3                                  |
| April 2013              | 4.84                                   | 5.04                                 |
| May 2013                | 5.31                                   | 5.43                                 |
| June 2013               | 4.98                                   | 3.77                                 |
| July 2013               | 4.63                                   | 3.84                                 |
| August 2013             | 4.48                                   | 3.92                                 |
| September 2013          | 4.38                                   | 4.2                                  |
| October 2013            | 5                                      | 4.7                                  |
| November 2013           | 7.31                                   | 6.8                                  |
| December 2013           | 4.86                                   | 4.602                                |
| Overall Mean Wind Speed | 5.331                                  | 5.0602                               |

The graph (Fig. 6) reveals a noticeable trend where the data from Balkan Wind Atlas matches the measured data.

The greatest data discrepancy pertains to months June, July, and August.



**Fig. 6** Mean monthly wind speeds in 2013, by Balkan-Wind-Atlas and actual data measurement in Mamaj, Tepelenë, Albania

Many records (about 4%) from on-site measurements conducted during these months were considered erroneous and were deleted. We stress that the cone-shaped anemometers lacked a calibration date. But in terms of overall average wind speed for this period, the mismatch was about  $\pm 5\%$ .

WAsP (Wind Atlas Analysis and Application Program) was used to determinate Observed Wind Climate(OWC), by using the wind speed and direction information. The WAsP is a PC software that extrapolates vertical and horizontal statistics regarding wind climate[16]. WAsP simulation program is a program that can conduct an analysis by using the wind speed data obtained from meteorological stations and analyzes the data by modeling regarding the direction of wind on the land to establish wind farms[17].

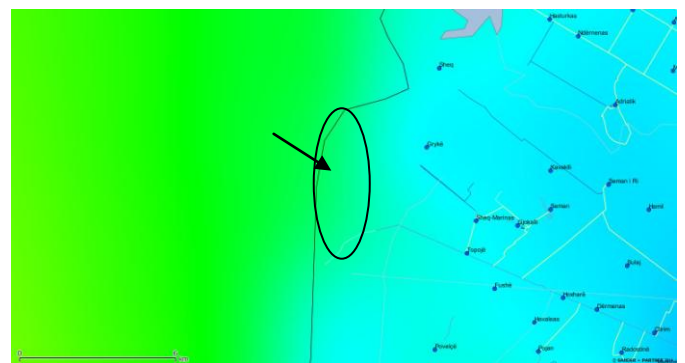
According to the data coming from Observed Wind Climate (OWC) from each anemometric tower, the average wind speed at 50m above ground level, for Valley tower was  $U=5.32\text{m/s}$  from March 2013 to June 2014, while the average wind speed for "Atlas" tower was  $U=5.52\text{m/s}$  from January 2013- December 2013. In this case the mismatch rate was  $\pm 3.7\%$ .

Therefore, the long-term data coming from Balkan Wind Atlas can be used with high confidence especially in non-complex terrains of the Western Lowland of the country. According to National Agency of Natural Resources of Albania, 4 investment company are licensed to develop wind farms with 3 MW installed power each of them in the Topoja area which lies in the Western Lowland, near the Adriatic Sea (Fig. 7).



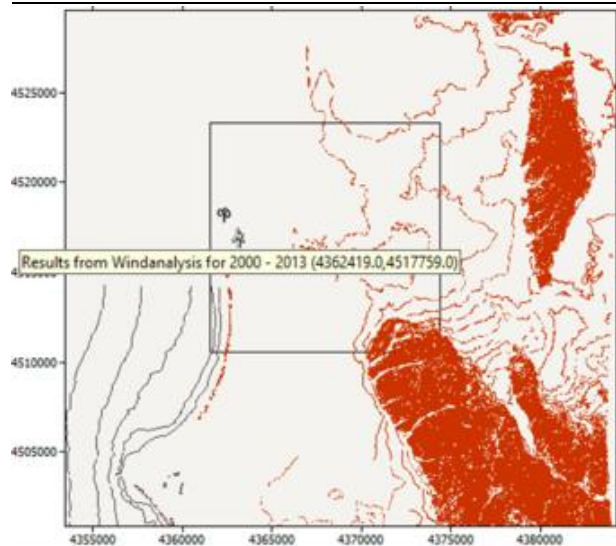
**Fig. 7** The Topoja area, Fier, Albania.

The best wind potential area is shown in Figure 8.



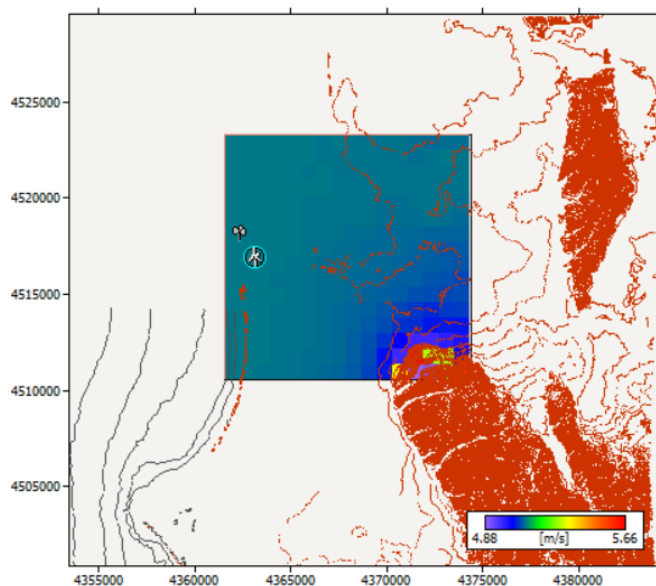
**Fig. 8** The windiest site in Topoja area, Fier, Albania.

This is an area of  $5.6\text{km}^2$  and 2-2.5m above sea level. Data obtained from Balkan-Wind-Atlas for the period 2000-2013, were used to study wind climate in the area of interest in Topoja, Fier, Albania for the hypothetical anemometric tower with the coordinates  $40^{\circ}47'57.5''\text{N}$ ,  $19^{\circ}22'08.7''\text{E}$  whose position on the topographical map is shown in Figure 9.

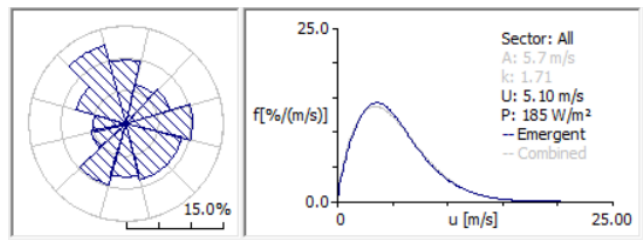


**Fig. 9** The hypothetical anemometric tower in Topoja area, Fier, Albania.

According to the Observed Wind Climate (OWC) of the hypothetical anemometric atlas tower, mean wind speed and the power density in 50m a.g.l. were found to be 5.08m/s and 150.27W/m<sup>2</sup> respectively. Turbine with 50m height hub was placed in the area and by using WAsP software wind rose and Weibull distribution were studied (Fig. 10), (Fig. 11). It is noted that the probability of having wind speeds above 5.5m/s in 50m a.g.l is 36.6%.



**Fig. 10** Resource grid of Topoja area for 50m a.g.l..



**Fig. 11** Frequency wind distribution and Weibull distribution in the windiest potential area of Topoja.

According to the wind turbine, the average wind speed, and the wind power density at 50m a.s.l are 5.10m/s and 185 W/m<sup>2</sup>. This identifies the Topoja area as class 1 wind power[7].

However, of interest will be comparing these results with those of real measurements that are being carried out on the site.

Furthermore sensitivity economic analysis would help the decision-makers to estimate the sensitivity of important financial indicators in relation to key technical and financial parameters in order that the investments in Topoja area to consider acceptable [19].

### 3. Conclusions

This study supports the idea that the use of Balkan Wind Atlas data is an important tool for identifying areas with wind energy potential especially for countries where long-term measurement data is lacking. To increase reliability in Balkan Wind Atlas, data collected over a 15 month period (March 19, 2013 to June 14, 2014) in Mamaj, Tepelena, were compared with those obtained from Wind Balkan Atlas for the closest point to this area in 2013. This reveals a noticeable trend where the data from Balkan Wind Atlas matches the measured data. In this case the mismatch rate was  $\pm 3.7\%$ .

The long-term data coming from Balkan Wind Atlas were used to evaluate the wind energy potential in one of the non complex terrains of the Western Lowland of the country, in Topoja area. The wind turbine, was located by WAsP in the windiest area given by Balkan Wind Atlas. The average wind speed and the wind power density at 50m a.s.l are 5.10m/s and 185 W/m<sup>2</sup> respectively. This identifies the Topoja area as class 1 wind power.

Provided with this context, there is high interest in comparing these data to those of real measurements being carried out on the ground. The comparison will further increase the reability of Balkan Wind Atlas data which will eventually serve as a tool for conducting initial assessments of wind potential in areas of interest to investors in the field.

Conducting a thorough analysis on wind potential in areas of interest is particularly important in a country like Albania where no wind farms have been established yet. Assessing wind energy potential as well as conducting an economic sensitivity analysis will be crucial for guaranteeing success for future investors.

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## Identifying Photovoltaic Water Pumping (PVWP) Systems Opportunities in Albanian's Agriculture Context.

<sup>a</sup>Ilirian Konomi, <sup>b</sup>Lorenc Malka, <sup>a</sup>Ermonela Rrapaj

<sup>a</sup>Department of Hydraulics & Hydrotechnics, Faculty of Civil Engineering, Polytechnic University of Tirana, Albania

<sup>b</sup>Department of Energy, Faculty of Mechanical Engineering, Polytechnic University of Tirana, Albania

ikonomi64@gmail.com, lmalka@fim.edu.al

**Abstract:** A lot of economic analyses have been conducted in the last ten years to establish the most cost-effective solution for irrigation and evaluation of the project profitability. The benefits generated by the PVWP providing water by a submersible pump located inside a deep well have been highlighted for Divjaka region. The solar potential in the site is quite enough to be used to pump water from the deep well into the tank positioned at an effective altitude which can provide the water quantity and pressure by gravity. The study shows that installing a PVWP system represents the best technical and economic solution to drive a water pump that provides water for sprinkler irrigation. The economic benefits have been also addressed, evaluating the energy production and distribution throughout the year and the specific cost per m<sup>3</sup> of water supplied (€/m<sup>3</sup>). Renewables are the key to enhance food and water security, drive agri-food productivity, leading to socio-economic benefits in recovering from post-Covid-19. By combining our knowledge, data collected, surveys together can contribute to economic growth of our community-ensuring access to clean and affordable energy and raising the standard of living of rural and most vulnerable communities. In the area there are used two types of water pumping for irrigation purposes: Diesel driven water pumps and electricity powered water pumps. Both systems are very costly due to the high fuel cost and on the other hand self-investment to bring electricity from the national distribution lines are needed.

**Keywords:** PVWP, PVsyst, RES, Irrigation

### 1. Introduction

The development of renewable energy sources as a means of meeting the global energy demand and simultaneously replacing fossil fuels as one of the key drivers of climate change has become one of the major societal challenges of our time. In this context, photovoltaic (PV) systems offer great potential and are considered even more efficient in capturing sunlight energy than photosynthesis [1]. Energy and agri-food systems are deeply connected, and it is needed at every stage of agri-food systems, its current use in their development is unsustainable. On the other hand, about 30% of the world's energy is consumed within agri-food systems, mainly in the form of fossil fuels, and this energy is responsible for a third of their greenhouse gas emissions [2]. Renewable Energy Sources is essential for agri-food systems transformation and development, climate resilience and net-zero strategies by 2030 in Albanian context as the majority of the rural population lies their economy on agriculture. Over 2.5 billion people worldwide rely on agriculture for their livelihoods making the sector a key driver for development. These findings are becoming even more relevant, as water demand for irrigation is expected to increase in prospective future climatic conditions [3-4]. Hence, a properly and well-designed irrigation system addresses uniform water application quantities in a timely manner while minimizing losses and damage to soil and crops as well. The design of a proposed gravity sprinkler irrigation system will smooth water characteristics and application rates in respect to the demand and the right time.

In the other hand physical characteristics of the area to be irrigated must be considered in locating the lines and spacing the sprinklers or emitters, and in selecting the type of mechanized system.

The location of the water supply, capacity, and the source of water will affect the size of the pipelines, irrigation system flow rates, and the size and type of pumping plant to be used. The power unit selected will be determined by the overall pumping requirements and the energy source available. The use of PVWP technology for irrigation is considered an innovative and sustainable solution with the aim to provide cost-effective solution within off grid PV concept. Such systems can promote the use of agriculture land, especially in remote areas of Albania.

The combination of PVWP technology with water saving irrigation techniques and sustainable management of the groundwater resources can lead to several benefits. From the technical point of view this system can offer the improvement of grid reliability and limitation of power outages, protection of critical loads, independence from national grid supply, and increased energy security coupled with a fixed energy cost which is immune to future tariffs and fossil fuel costs increases. This article presents a real

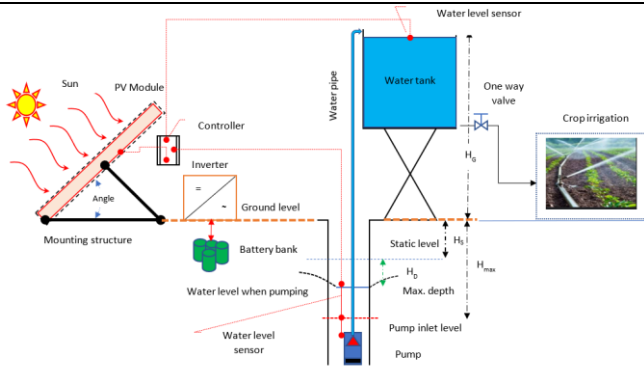
application “**Photo Voltaic Water Pump**” (PVWP) installed in Divjaka region to provide water for irrigation 2.0 ha.

### 2. Off - Grid PV systems applications

Solar irrigation, among the most mature applications, is being widely adopted to improve access to water, thus enabling multiple cropping cycles and increasing resilience to changing rainfall patterns. The use of solar irrigation pumps has raised farmers' incomes by 50% or more in India compared to rain-fed irrigation. In Rwanda, smallholder farmers' yields have grown by about a third. The use of solar irrigation also displaces current and future fossil fuel use as the land area under irrigation expands. In so doing, it lowers emissions. Bangladesh's Nationally Determined Contribution under the Paris Agreement, for example, identifies solar irrigation as a key measure to mitigate climate change. Life-cycle emissions for solar-powered water pumping are estimated to be 95% to 98% lower than for pumps powered by grid electricity or diesel fuel [5]. Today, PV is one of the fastest-growing renewable energy technologies and is ready to play a major role in the future global electricity generation mix and a contribution for some 3.8 million jobs, or nearly a third of the sector total [6.]. Using solar PV to power mini-grids is an excellent way to bring electricity access to people who do not live near power transmission lines, particularly in developing countries with excellent solar energy resources and reducing the negative effect on environmental.

The cost of manufacturing solar panels has plummeted dramatically in the last decade, making them not only affordable but often the cheapest form to be replaced and integrated in existing power systems. Solar panels have a lifespan of roughly 30 years and come in variety of shades depending on the type of material used in manufacturing.

Off grid PVWP systems applications have been studied to cover a lot of issues, especially to provide water for drinking purposes in the areas that suffer the lack of electricity. Nevertheless, the drastic fall in prices of PV modules due to the new-born production and costless technologies of the PV lead to increased interest on research and development of off grid PV systems, encouraging greater system flexibility and large-scale integration and new applications especially in Albania. It is also essential to identify economic, technical, and environmental constraints that can negatively affect the 3 operations of PVWP systems.



**Figure 1:** Integrated proposed PVWP system.

Both systems can be applied using open or under pressure water tank. The pump can be immersed or on the ground level. The water is pumped into a storage tank, according to sun availability. Pressure (head) is mainly related to the difference between the input and output levels. The pump should at least provide a total head which as a result of several contributions. In PVsyst the ground level served as a reference, hence:

1

where:

$H_G$ =head due to the height of the outlet pipe above the ground (assuming that outlet pressure is negligible).

$H_S$ =static head due to the depth of the water level in the well, in absence of any pumping.

$H_D$  = dynamic "drawdown" head: in a borehole well, the effective water level is dynamically lowered by the water flow extraction

$H_F$ =friction losses in the piping circuit, which depend on the flowrate. Solar PV offers better benefits and reliable solutions for consumers in rural areas who do not have access to the grid [7]. The economic benefit is assessed based on the LCOE which represents a good starting point to compare benefits and competitiveness of different technologies. Photovoltaic systems are cost-effective in small off-grid applications, providing power, to rural homes in developing countries, off-grid cottages and motor homes in industrialised countries, and remote telecommunications, monitoring and control systems worldwide.

The studies have demonstrated that a solar PV combined with diesel engine (hybrid) has relatively lower LCOE than a pure diesel generator-only. The IEA estimates that to achieve the goal of universal electricity access, 70% of the rural areas that currently lack electricity will need to be connected using mini grid or off-grid solutions. Photovoltaic systems can be combined with fossil fuel driven (Genset) motors. Off-grid applications include both stand-alone systems, and hybrid systems, which are similar to stand-alone systems but also include a fossil fuel generator (Genset) to meet some of the load requirements and provide higher reliability. The studies have demonstrated that a solar PV combined with diesel engine (hybrid) has relatively lower LCOE than a pure diesel generator-only. Nevertheless, the capital cost of the battery, which is one of the most significant components in LCOE evaluation aims to be reduced to more than 60% by 2030 [8]

### 3. Photovoltaic theory and PVWP calculations.

In this paper the off-grid applicability of the PVWP project altering the existing genset/electric water pump options is investigated for irrigation. Thus, an accurate methodology comprehending in-depth analysis of the benefits must be applied and always required. The need for a pile of datas including physical characteristics, financial viability, environmental advantage, carbon credits, social or other self-interests of the project, will help the energy planners to a mature decision. For this work PVsyst model is chosen. The model uses a computerized system with integrated mathematical algorithms and top to bottom approach. It provides a cost technical and economic analysis and also some financial summary. The PVsyst Software

has been developed to overcome the barriers to clean energy technology implementation at the preliminary feasibility stage.

First it is analysed the capacity and structure of the actual water demand and then choosing the right PVWP system. For the chosen water demand and pumping water system it is easy to select from the database the most suitable module type and model, respectively matching on recommendations and trends. The water is provided from surveys in the site, from the literature of the field from recommendations and also from the owner declarations.

### 4. Basics of Solar Energy Theory

Before entering into the details of the PV model, it will be useful to review briefly some basic concepts of solar energy engineering. Many of the variables derived in this section will be used in several parts of the model. For the most part, the equations in this section come from a standard textbook on the subject, Solar Engineering of Thermal Processes, by Duffie and Beckman (1991) [9], to which the researchers can address various technical aspects.

### 5. Declination

The declination is the angular position of the sun at solar noon, with respect to the plane of the equator. Its value in degrees is given by Cooper's equation:

$$\delta = 23.45 \sin \left( 2\pi \frac{284 + n}{365} \right) \quad (2)$$

where  $n$  is the day of year (i.e.  $n=1$  for January 1,  $n=32$  for February 1, etc.). Declination varies between  $-23.45^\circ$  on December 21 and  $+23.45^\circ$  on June 21.

### 6. Solar hour angle and sunset hour angle

The solar hour angle is the angular displacement of the sun east or west of the local meridian, morning negative, afternoon positive. The solar hour angle is equal to zero at solar noon and varies by 15 degrees per hour from solar noon. The sunset hour angle  $\omega_s$  is the solar hour angle corresponding to the time when the sun sets and given by equation 3:

$$\omega_s = -\tan \varphi \tan \delta \quad (3)$$

$\varphi$  represents the latitude of the site specified by the user.

### 7. Extraterrestrial radiation and clearness index

Solar radiation outside the earth's atmosphere is called extraterrestrial radiation. Daily extraterrestrial radiation on a horizontal surface,  $H_0$ , can be computed for day  $n$  from the following equation:

$$H_0 = 86400 \frac{G_{sc}}{\pi} \left( 1 + 0.33 \cos \left( 2\pi \frac{n}{365} \right) \right) \quad (4)$$

$$(\cos \varphi \cos \delta \sin \omega_s + \omega_s \sin \varphi \sin \delta)$$

where  $G_{sc}$  is the solar constant equal to  $1,367 \text{ W/m}^2$ , and all other variables have the same meaning as explained in equation 1 and 2. Before reaching the surface of the earth, radiation from the sun is attenuated by the atmosphere and the clouds. The ratio of solar radiation at the surface of the earth to extraterrestrial radiation is called the clearness index, defined in equation 4:

$$\bar{K}_T = \frac{\bar{H}}{H_0} \quad (5)$$

where  $\bar{H}$  is the monthly average daily solar radiation on a horizontal surface and  $\bar{H}_0$  is the monthly average extraterrestrial daily solar radiation on a horizontal surface.  $\bar{K}_T$  values depend on the location and the time of year considered; they are usually between 0.3 (for very overcast climates) and 0.8 (for very sunny locations).

## 8. Calculation of average efficiency

The array is characterized by its average efficiency,  $\eta_p$ , which is a function of average module temperature  $T_{a_c}$ :

$$\eta_p = \eta_r [1 - \beta_p (T_c - T_r)] \quad (6)$$

where  $\eta_r$  is the PV module efficiency at reference temperature  $T_r$  (25°C), and  $\beta_p$  is the temperature coefficient for module efficiency,  $T_c$  is related to the mean monthly ambient temperature  $T_a$  through Evans' formula (Evans, 1981) [10]:

$$[T_c - T_a] = (219 + 832 \bar{K}_t) \frac{NOCT - 20}{800} \quad (7)$$

where NOCT is the Nominal Operating Cell Temperature and  $\bar{K}_t$  the monthly clearness index.  $\eta_r$ , NOCT and  $\beta_p$  depend on the type of PV module considered. Such values can be manually entered into the model, but for "standard" technologies, assumed values are given in Table 1.

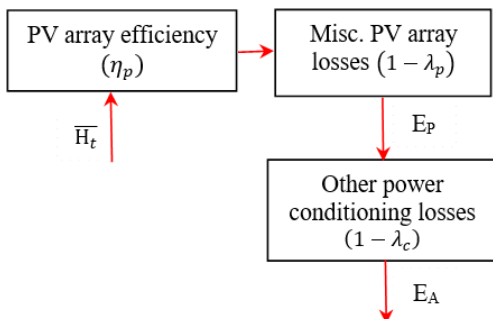
**Table 1:** PV module characteristics for standard technologies.

| PV module type | $\eta_r$<br>(%) | NOCT<br>(°C) | $\beta_p$<br>(%/°C) |
|----------------|-----------------|--------------|---------------------|
| Mono - Si      | 13.0            | 45           | 0.4                 |
| Poly - Si      | 11.0            | 45           | 0.4                 |
| a-Si           | 5.0             | 50           | 0.11                |
| CdTe           | 7.0             | 46           | 0.24                |
| CIS            | 7.5             | 47           | 0.46                |

The equation above is valid when the array's tilt is optimal (i.e. equal to the latitude minus the declination). If the angle differs from the optimum the right side of equation (8) has to be multiplied by a correction factor  $C_f$  defined by:

$$C_f = 1 - 1.17 \times 10^{-4} (s_m - s)^2 \quad (8)$$

where  $s_m$  is the optimum tilt angle and  $s$  is the actual tilt angle, both expressed in degrees.



**Figure 2:** Flowchart for PV Array Model

## 9. Other corrections

The energy delivered by the PV array,  $E_p$ , is simply:

$$E_p = S \cdot \eta_p \cdot \bar{H}_t \quad (9)$$

where  $S$  is the area of the array. It has to be reduced by "miscellaneous PV array losses"  $\lambda_p$  and "other power conditioning losses"  $\lambda_c$ .

$$E_A = E_p (1 - \lambda_p) (1 - \lambda_c) \quad (10)$$

where  $E_A$  is the array energy available to the load and the battery. The overall array efficiency  $\eta_A$  is defined as:

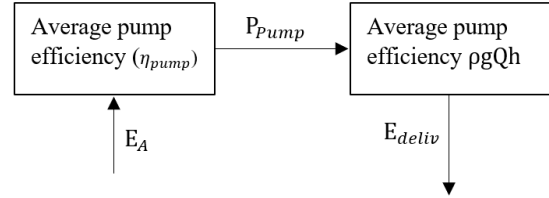
$$\eta_A = \frac{E_A}{S \cdot \bar{H}_t} \quad (11)$$

## 10. Off-Grid Model

Off-grid renewable power can come from a variety of sources, ranging from large-isolated power grids to solar lights and solar home systems. In addition to households, off-grid renewables provide power for water pumping, street lighting,

telecommunications towers, rural schools and clinics, as well as for remote commercial and industrial facilities and other uses [11].

The off-grid model represents stand-alone systems with a battery backup, with or without an additional genset. The conceptual framework of the model is shown in Figure 3. Energy from the PV array is either used directly by the load or goes through the battery before being delivered to the load. The remainder of the load is provided by the genset if there is one, that is, stand-alone and hybrid systems differ only by the presence of a genset that supplies the part of the load not met directly or indirectly by photovoltaics.



**Figure 3:** Energy transformation workflow.

The water pumping model is based on the simple equations found in Royer et al. (1998) [12] shown schematically in Figure 16. The daily hydraulic energy demand  $E_{hydr}$ , in J, corresponding to lifting water to a height  $h$  (m) with a daily volume  $Q$  (m<sup>3</sup>/d) is:

$$E_{hydr} = 86400 \rho g Q h (1 + \eta_f) \quad (12)$$

where  $g$  is the acceleration of gravity (9.81 m s<sup>-2</sup>),  $\rho$  the density of water (1000 kg/m<sup>3</sup>), and  $\eta_f$  is a factor accounting for friction losses in the piping. This hydraulic energy translates into an electrical energy requirement  $E_{pump}$ .

$$E_{pump} = \frac{E_{hydr}}{\eta_{pump}} \quad (13)$$

where  $\eta_{pump}$  is the pump system efficiency. If the pump is AC, this equation has to be modified to take into account the inverter efficiency  $\eta_{inv}$ :

$$E_{pump} = \frac{E_{hydr}}{\eta_{pump} \eta_{inv}} \quad (14)$$

Energy delivered is simply:

$$E_{deliv} = \eta_{pump} \min(E_{pump}, E_A) \quad (15)$$

where  $E_A$  is the energy available from the array (this quantity should be multiplied by  $\eta_{inv}$  in the case of an AC pump), and daily water delivered is obtained from:

$$Q_{deliv} = \frac{E_{deliv}}{86400 \rho g Q h (1 + \eta_f)} \quad (16)$$

Suggested array size is calculated simply by inverting the above equations and is therefore equal to  $E_{pump}/\eta_A$  where  $\eta_A$  is the overall array efficiency (see equation 10). This quantity is calculated on a monthly basis and the maximum over the season of use is the suggested array dimension. In the case of an AC pump, suggested inverter capacity is simply taken equal to the nominal array power. This is the only method possible since it is assumed that the pump power rating is not known (only the energy demand is known)

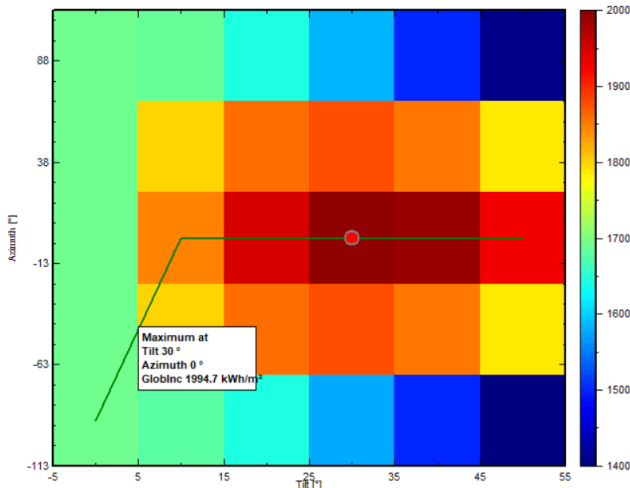
## 11. PVWP system in PVsyst environment

The selection of the PV must meet different criteria simultaneously:

- generate high quality electricity according to specific standards.
- withstand the high variability of solar radiation characteristics.
- require less maintenance interventions and costless.
- compete economically with other energy sources.
- fast and cost-effective compliance.
- Guarantee the water demand in time and reducing the unused energy level.

From the PVsyst database and technical information obtained from the manufacturer, comparisons were executed to determine the most efficient PV module among five alternative types taken in this study.

After creating the data file in the PVsyst for the selected area the first step is to define the orientation of the solar plane. The quick optimization of the solar energy should be accordingly with respect to function and aim of the proposed PV system. As our system will be used to provide water for irrigation purposes in summer period and for the fixed tilted plane type the maximums fall in the 23° tilted angle and azimuth 0°. For the summer period (April to September) the transposition factor resulted 1.06, loss in respect to optimum is -0.2% and global yield (in summer) on collector results 1307kWh/m<sup>2</sup> (see table nr 2).



**Figure 4:** Optimization tilt and azimuth angle at the proposed PVWP system.

PVsyst enable the users to perform automatically a set of simulations, where one or more parameters are varied systematically according to a specified range. Optimization tools of the 'Orientation' suggest a tilt angle of 30° and azimuth0°. In our calculation the tilt angle is suggested 23° up 30°. But in our case is approximated to 23° as the aim is to provide water during summer period global yield on collector is 1309 kWh/m<sup>2</sup> while for 30° is 1294kWh/m<sup>2</sup>.

**Table 2:** The preliminary parameters chosen for the PVWP model.

| Transposition factor | Loss in respect to optimum (%) | Global yield (in summer) on collector (kWh/m <sup>2</sup> ) |
|----------------------|--------------------------------|-------------------------------------------------------------|
| 1.06                 | -0.2%                          | 1307                                                        |

In the second step it is obligatory to define the "Water needs" button selecting "Pumping Hydraulic Circuit" and one can choose among one of the three available systems (refer figure 4):

1. Pumping from a deep well, to a tank storage,
2. Pumping from a lake or river, to a tank storage,
3. Pumping into a pressurized tank, for water distribution.

In our case study pumping from a deep well to a tank storage is defined. The well can be 5 meters up to 15 meters deep. From the farmers statement, normally 10 meters deep well can guarantee the water demand referring the driest month (July) for more 10 hours for irrigation of carrots or potatoes. The actual well is around 15 meter and the water amount is able to provide 8 hours irrigation per day and the specific drawdown of -0.08m/m<sup>3</sup>/h with a maximum flowrate of 62.5m<sup>3</sup>/h, dynamic lower level -10m, the tank volume is defined 200m<sup>3</sup> which can easily provide the water amount per each irrigation day with a reserve of 40%. Feeding altitude is selected 6.5m above ground level calculated as a result of the water parameters needed at the end of the circuit line.

**Table 3:** Main parameters from a deep well to a tank storage.

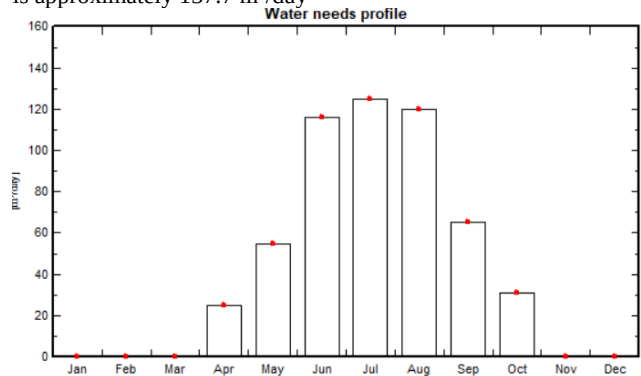
|                       | Unit               | Value |
|-----------------------|--------------------|-------|
| Static level          | m                  | -5    |
| Specific drawdown     | m/m <sup>3</sup> h | -0.08 |
| Maximum flowrate      | m <sup>3</sup> /h  | 62.5  |
| Lower dynamic level   | m                  | -10   |
| Hydraulic circuit     | m                  | 250   |
| Number of elbows      | pieces             | 30    |
| Other friction losses | %                  | 3-10  |
| Tank volume           | m <sup>3</sup>     | 200   |
| Tank diameter         | m                  | 6.5   |
| Water full height     | m                  | 6.03  |
| Feeding altitude      | m                  | 6.5   |
| Pipe                  | "                  | 4     |

The tank diameter volume is defined taking into account the daily water quantity demand referring the worst case (the most dry month of the year results July), crop type and soil type. The references for the water needs profile are provided form the literature given below in figure 5. Sprinkler irrigation systems are well adapted to carrot and potatoes irrigation. In the region, sprinkler designs of 12x12 m were common in carrot irrigation. Sprinkler flow rates varies from 0.3-4m<sup>3</sup>/h. Similar result, 2.57 m<sup>3</sup>/h sprinkler having flow rate at 2.38 atm pressure with 4.5/4.8 mm nozzle diameter was also mentioned elsewhere [13] in same region. Durations for using sprinklers with 0.3-1.0 m<sup>3</sup>/h and 1.5-2.5 m<sup>3</sup>/h flow rates were 9-10 h and 5-6 h. Yield or quality of carrot root is highly affected by carrot cultivar, fertilizer management, cultural practices in vegetation period and correct selection or management of irrigation system. In our study, carrot root yield was in the range between 60 and 85 m<sup>3</sup>/ha. Irrigation water was obtained from the ground water source in research region. Average electricity consumption for pumping unit volume of water and seasonal electricity consumption were determined as about 0.305 kwh/m<sup>3</sup> and 5338 kwh/ha (17500x0.305), respectively. In research of, energy consumption of such pump was about 0.340 TL/kwh. It was calculated as 1815 TL/ha or \$485/ha. In generally water requirement for vegetable crop can be calculated by using the following formula [14]:

$$W_{req} = \frac{A * PE * P_c * K_c * w_a}{E_u} \quad a$$

Where:  $W_{req}$ =Peak water requirement (m<sup>3</sup>/day); A=crop area (20 000m<sup>2</sup>); PE =Pan Evaporation rate (mm/day) converted to m/day; (8-10 mm/day);  $P_c$ =Pan Coefficient (0.7 to 0.9);  $K_c$ =Crop Coefficient (0.8 to 1);  $w_a$ =wetted area (%) taken (90% for sprinkler irrigation);  $E_u$ =Emission uniformity of sprinkler irrigation (0.8).

By substituting the mentioned above values, the water requirement is approximately 137.7 m<sup>3</sup>/day



**Figure 5:** Water demand profile for the proposed case study.

As it can be seen from the water needs profile given in figure 5 the average yearly water demand is around 45 m<sup>3</sup>/d or 16 334 m<sup>3</sup> for the whole year for the given surface in figure number 1.

The next step is select the most suitable water pump based on the water needs profile. This selection should take into the consideration the well type, control type and solar radiation potential as well. The simulation should be performed to provide the best solution in terms of specific energy, system and pump efficiency and reducing fraction of unused energy produced by the PV system. The result from the simulation carried out are summarized in table 4.

**Table 4:** Main technical parameters of the water pump

| Centrifugal Multistage (Grundfos SOF 9-3 v30-300V) |        |      |      |
|----------------------------------------------------|--------|------|------|
| Maximal power                                      | 1400 W |      |      |
| Voltage                                            | 170 V  |      |      |
| Max.Current                                        | 8.2 A  |      |      |
| Head min/Nom/Max (meterW)                          | 5      | 15   | 25   |
| Corresp.Flowrate                                   | 17     | 14.4 | 11.0 |
| Corresp. Power                                     | 1400   | 1400 | 1400 |
| Efficiency                                         | 16.6   | 41.9 | 53.7 |

**Table 5:** Main technical parameters of the PV module used in the study

|                                                                 | Technology                               | Specification                                                         |
|-----------------------------------------------------------------|------------------------------------------|-----------------------------------------------------------------------|
| <b>PV System</b>                                                | Generic/Si-poly                          | 285Wp 30V                                                             |
| <b>Sizing voltages</b>                                          |                                          | V <sub>mpp</sub> (60°C)<br>31.2V<br>V <sub>oc</sub> (-10°C)<br>49.9 V |
| <b>Modules in series</b>                                        |                                          | 3                                                                     |
| Operating conditions                                            |                                          |                                                                       |
| <b>V<sub>mpp</sub></b>                                          | (60°C)                                   | 94V                                                                   |
| <b>V<sub>mpp</sub></b>                                          | (20°C)                                   | 111V                                                                  |
| <b>V<sub>oc</sub></b>                                           |                                          | 150 V                                                                 |
|                                                                 | Plane irradiance 1000 kWh/m <sup>2</sup> |                                                                       |
| <b>I<sub>mpp</sub></b>                                          |                                          | 7.9A                                                                  |
| <b>I<sub>sc</sub>/I<sub>sc</sub> (STC)</b>                      |                                          | 8.4A/8.4A                                                             |
| <b>Maximum operating power at 1000 W/m<sup>2</sup> and 50°C</b> |                                          | 0.8kW                                                                 |
| <b>Array nominal power (STC)</b>                                |                                          | 0.9kW                                                                 |

Inverter type for this case study is provided from the model universal controller 1000W and suggested control type MPPT - DC converter.

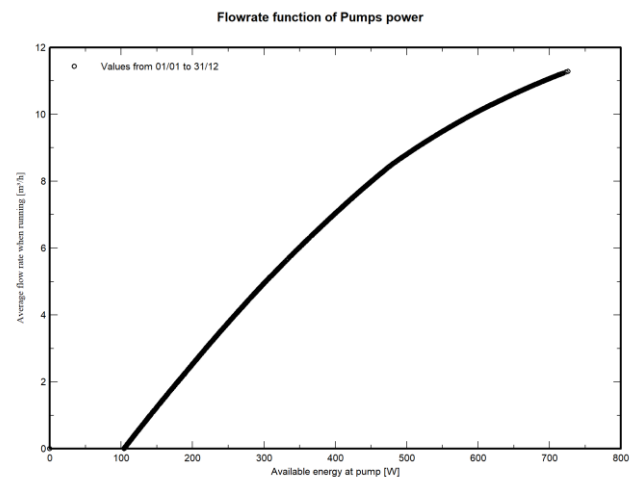
## 12. Results and analysis

From the simulation of the proposed PVWP system the final results for the efficiency of the system and other techno economic features.

In figure 6 it is given the flowrate as a function of available energy delivered at the pump. At the maximum head of 25m flowrate reaches the level of **11.0 m<sup>3</sup>/h**

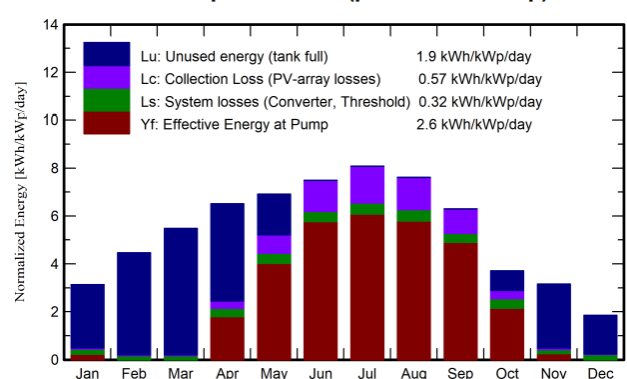
The pump results DC motor and the maximum flowrate is 17 m<sup>3</sup>/h but in nominal condition the water flowrate then can be pumped is 14.4 m<sup>3</sup>/h. The water needs can be provided if the pump works 6-8 hour per day then 115m<sup>3</sup>/day can be discharged into the tank. That

is the water demand that system can be provided in one day referring the worst case (July period).



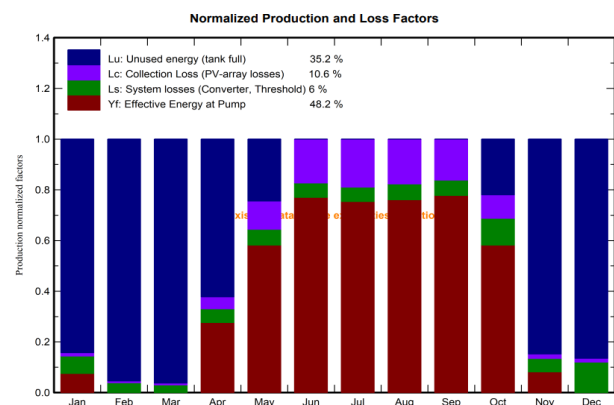
**Figure 6:** Flowrate function of pumps power.

**Normalized productions (per installed kWp)**



**Figure 7:** Normalized Energy (kWh/kWp/day).

In figure 7 the normalized production (kWh/kWp/day) per each month is given. The maximum production fall in July while in March the unused energy happen due to the zero water demand profile. In January up to May the fraction of unused energy prevails. In summer period from April to October the unused fraction becomes zero as the water demand is higher. The collection losses (PV-array losses) are higher during the summer period reaching the maximum value in July (0.57kWh/kWp/day). Converter losses are in the range of 0.32 (kWh/kWp/day).



**Figure 8:** Normalized Production and Loss Factors.

In figure 8 the unused energy fraction results 35.2 % due to the water profile demand during winter season. PV array losses are 10.6% while effective Energy at pump results 48.2%.

**Table 7:** Main balances and results from the simulation of the selected PVWP system.

|           | GlobEff<br>kWh/m <sup>2</sup> | EAnMPP<br>kWh | E_PmpOp<br>kWh | ETKFull<br>kWh | H_Pump<br>meterW | WPumped<br>m <sup>3</sup> | W_Used<br>m <sup>3</sup> | W_Miss<br>m <sup>3</sup> | EArray<br>kWh | PR<br>ratio |
|-----------|-------------------------------|---------------|----------------|----------------|------------------|---------------------------|--------------------------|--------------------------|---------------|-------------|
| January   | 94.0                          | 76.6          | 6.3            | 61.0           | 11.43            | 100                       | 0                        | 0                        | 76.6          | 0.076       |
| February  | 121.5                         | 97.1          | 0.0            | 88.3           | 0.00             | 0                         | 0                        | 0                        | 97.0          | 1.000       |
| March     | 164.9                         | 128.9         | 0.0            | 118.5          | 0.00             | 0                         | 0                        | 0                        | 128.9         | 1.000       |
| April     | 190.1                         | 144.6         | 46.1           | 85.8           | 11.52            | 741                       | 750                      | 0                        | 144.5         | 0.276       |
| May       | 205.2                         | 156.1         | 106.6          | 36.4           | 11.81            | 1698                      | 1705                     | 0                        | 156.0         | 0.562       |
| June      | 219.2                         | 159.0         | 148.0          | 0.0            | 11.63            | 2394                      | 2571                     | 909                      | 158.9         | 0.770       |
| July      | 244.1                         | 173.5         | 161.4          | 0.0            | 11.67            | 2649                      | 2657                     | 1218                     | 173.5         | 0.754       |
| August    | 230.1                         | 166.2         | 153.6          | 0.0            | 11.70            | 2537                      | 2537                     | 1183                     | 165.9         | 0.761       |
| September | 184.0                         | 135.4         | 125.7          | 0.0            | 11.63            | 2074                      | 1931                     | 19                       | 135.4         | 0.778       |
| October   | 111.7                         | 86.2          | 57.2           | 17.8           | 11.47            | 907                       | 961                      | 0                        | 86.1          | 0.582       |
| November  | 91.8                          | 73.2          | 6.6            | 58.8           | 11.64            | 111                       | 0                        | 0                        | 73.1          | 0.082       |
| December  | 55.5                          | 45.0          | 0.0            | 36.7           | 0.00             | 0                         | 0                        | 0                        | 44.9          | 1.000       |
| Year      | 1918.2                        | 1441.8        | 811.6          | 503.5          | 11.62            | 13211                     | 13111                    | 3330                     | 1440.8        | 0.482       |

**Legends**

GlobEff Effective Global, corr. for IAM and shadings

EAnMPP Array virtual energy at MPP

E\_PmpOp Pump operating energy

ETKFull Unused energy (tank full)

H\_Pump Average total Head at pump

WPumped Water volume pumped

W\_Used Water drawn by the user

W\_Miss Missing water

EArray Effective energy at the output of the array

PR Performance Ratio

In table 7 Balances and main results per each month of the year are given. The water used throughout the year results 13111 m<sup>3</sup> while the missing water results 3330 m<sup>3</sup>. Having in mind that system suggested to incorporate a water tank with 125 m<sup>3</sup> which is taken 200 m<sup>3</sup> providing a reserve of 30% more than the water needs. Other important outputs are carried out for evaluation in the case if the system changes (water pump or well type).

### 13. Conclusion

The present paper address various aspects related to PVWP energy systems for irrigation purposes in agriculture sector in Divjaka district. Nowadays, benefits coming from the PVWP system is becoming extremely beneficial from both technical and cost point of view and of course environmentally friendly. The perspective of a wide use of green power especially in agriculture sector motivates the policy makers in Albania to evaluate the possibility of funding the proposed systems.

The simulation from the study shows that for 2.0 ha, referring to the chosen PVWP system, the energy at pump becomes 812 kWh, specific energy 0.06kWh/m<sup>3</sup>, system efficiency 56.3%, pump efficiency 52.2% while the unused energy fraction results 34.9% (503kWh). The missing water rate is evaluated at a range of 19.6% which is covered by the extra dimension of the water tank.

The study shows very good results compared to the existing water pump systems (totally based on fossil or electricity from the grid) applied for irrigation purposes in Albania. Further investment in RES is essential for agri-food systems transformation and development, climate resilience and net-zero strategies by 2030 in Albanian context, as the majority of the rural population lies their economy on agriculture. The use of this kind of system could have an important contribution in the diversification of energy sources, mitigation of GHG, social and economic development of our country.

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# Electricity generation from PV plants up to 2MWp in Albania. Case study: Tërbuf-Divjaka Municipality

Ilirian Konomi<sup>1</sup>, Lorenc Malka<sup>2</sup>, Ermonela Rrapaj<sup>1</sup>

Department of Hydraulics & Hydrotechnics, Faculty of Civil Engineering, Polytechnic University of Tirana, Albania<sup>1</sup>

Department of Energy, Faculty of Mechanical Engineering, Polytechnic University of Tirana, Albania<sup>2</sup>

lmalka@fim.edu.al<sup>2</sup>, ikonomi64@gmail.com<sup>1</sup>

**Abstract:** Albania has a significant potential of Renewable Energy Sources (RES) and therefore represents a promising energy source for Albania. The use of PV for electricity generation supports the long-term objective of the country's energy policies set out in the national energy strategy 2018-2030, including support for the development of economic sectors, increasing security of energy supply and environmental protection. The use of Renewable Energy Sources (RES) will help reduce dependence on energy imports and increase the security of energy supply, with quality and without interruption. The possible benefits generated by the PV system implementation for the selected region should be highlighted, as well as the effects of the most sensitive parameters. The solar potential in Tërbuf-Divjakë showed that PV can be the best solution to provide electricity installed on saline non-agricultural lands near the Adriatic coast.

**Keywords:** PV-PUMP STATION, CO<sub>2</sub>, GHG, RETScreen Expert.

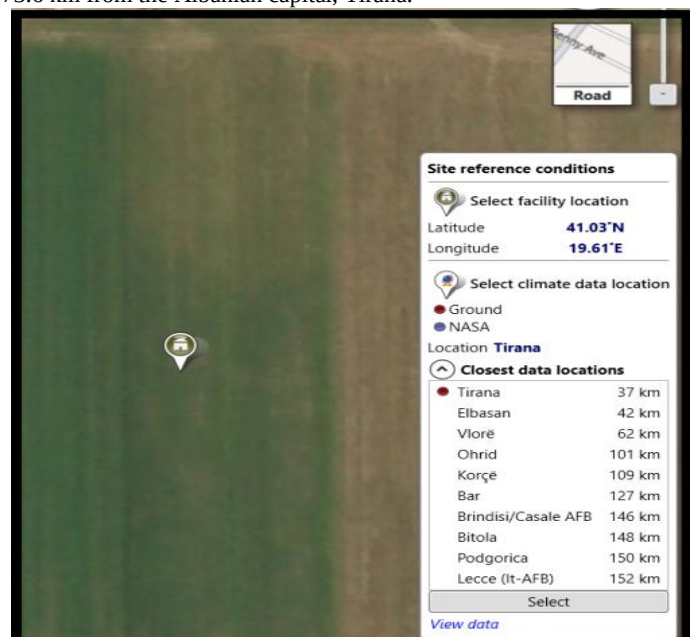
## 1. Introduction

The physical phenomenon responsible for converting light to electricity-the photovoltaic effect-was first observed in 1839 by a French physicist, Edmund Becquerel who noted that a voltage appeared when one of two identical electrodes in a weak conducting solution was illuminated. The PV effect was first studied in solids, such as selenium, in the 1870s. The photovoltaic (PV) effect is the basis of the conversion of light to electricity in photovoltaic, or solar, cells. Described simply, the PV effect is as follows: Light, which is pure energy, enters a PV cell and imparts enough energy to some electrons (negatively charged atomic particles) to free them. A built-in-potential barrier in the cell acts on these electrons to produce a voltage (the so-called photovoltage), which can be used to drive a current through a circuit. In the 1880, selenium photovoltaic cells were built that exhibited 1 up to 2% efficiency in converting light to electricity. Selenium converts light in the visible part of the sun's spectrum; for this reason, it was quickly adopted by the then-emerging field of photography for photometric (light-measuring) devices. Even today, light-sensitive cells on cameras for adjusting shutter speed to match illumination are made of selenium. In the 1920s and 1930s, quantum mechanics laid the theoretical foundation for our present understanding of PV. A major step forward in solar-cell technology came in the 1940s and early 1950s when a method (called the Czochralski method) was developed for producing highly pure crystalline silicon. In 1954, work at Bell Telephone Laboratories resulted in a silicon photovoltaic cell with a 4% efficiency. Bell Labs soon bettered this to a 6% and then 11% efficiency, heralding an entirely new era of power-producing cells achieving an overall efficiency of 20% for on-grid electricity generation. Considerable, interest in renewable energy sources (RES) and significant increases in cost of imported oil have compelled various countries to search for low-cost energy sources and improved technologies such, wind turbines, and synergies between systems to achieve lower cost of electricity generation. The world is now moving towards reliable and feasible hybrid renewable energy affected by the depletion of fossil fuel reserves and the negative effect on the environment as well as the potential attributes to merits of hybrid combinations. The share of renewable energy in the world is expected to increase from 19% in 2008 to about a third in 2035 while Albania is expected to increase its share of renewable energy in the total energy consumption to 42% by 2030 [1]. Solar and wind energy largely depends on the climatic conditions in a particular location, they depend on the availability of sunshine and wind flow respectively, hence it's very important in the renewable energy sector to conduct feasibility studies before implementing a project.

In the beginning of 2022 Albania is facing with energy crisis leading to high electricity cost. It is therefore important to give a boost to Albanian energy policy by increasing the RES share in the country's energy mix to drive down the high cost of energy generation and to build a climate friendly environment for the future. This requires considerable research, planning and development to help integrate renewable energy into the existing transmission and distribution system.

## 2. Site background and installation of proposed PV plant

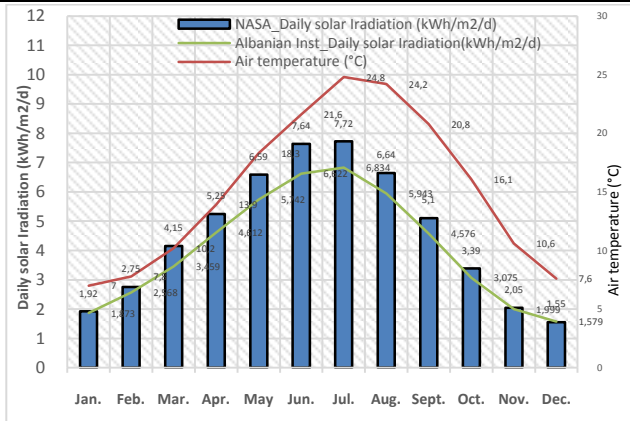
The 2MW photovoltaic plant will be installed on the ground in Tërbuf. The property is located 14.8 km from the city of Divjaka, 73.6 km from the Albanian capital, Tirana.



**Figure 1:** Location of the 2MW PV project. Village of Tërbuf, Divjakë Municipality, Albania.

From the model and data provided from Albanian Meteorological Institute it is shown that this region has a high solar potential.

From [2] and its Earth Science research program has long supported satellite systems providing important weather data capable to be fully integrated in RETScreen model. These data include long-term climatologically averaged estimates of meteorological quantities and surface solar energy fluxes. These satellite and model-based products have been shown to be accurate enough to provide reliable solar and meteorological resource data over regions where surface measurements are sparse or non-existent. The highest values are observed during the summer season of the year, while the lowest values are observed in the winter months. The highest solar radiation value 6.834 kWh/m<sup>2</sup>/d is reached in July, while the lowest value 1.55 kWh/m<sup>2</sup>/d hits in December. According to Albanian Institute of Geo-Sciences, Energy, Water and Environment the annual mean solar radiation for Divjaka region is 4.082 while referring to NASA and 4.56 kWh/m<sup>2</sup>/d (11% lower).



**Figure 2:** Comparative climate data between data obtained from NASA and meteorological station, Tërbuf village, Divjakë Municipality, Albania. Daily solar radiation kWh/m²/day at the proposed location.

**Table 1:** The site-specific solar energy data. Divjaka region. [3]

As shown in Table 1, the specific photovoltaic energy produced by each kWp installed produces approximately 1554.8 kWh of specific electricity (kWh/kWp). However, these values remain to be confirmed by the energy model selected for this feasibility analysis through RETScreen Expert. The suggested optimal angle of inclination of the photovoltaic module is 34°.

| Specific photovoltaic power output         | PV <sub>OUT</sub> specific | 1554.8 | kWh/kWp            |
|--------------------------------------------|----------------------------|--------|--------------------|
| Direct normal irradiation                  | DNI                        | 1725.2 | kWh/m <sup>2</sup> |
| Global horizontal irradiation              | GHI                        | 1640.9 | kWh/m <sup>2</sup> |
| Diffuse horizontal irradiation             | DIF                        | 620    | kWh/m <sup>2</sup> |
| Global titled irradiation at optimum angle | GTI                        | 1912.5 | kWh/m <sup>2</sup> |
| Optimum tilt of PV modules                 | OPTA                       | 34/180 | °                  |
| Air temperature                            | TEMP                       | 17.4   | °C                 |
| Terrain Elevation                          | ELE                        | 2      | m                  |

From Global Solar Atlas, energy planners can generate suitable site information for preliminary studies in EU-28 countries as they consider default values for many factors that are important for a design of a photovoltaic system. For more professional and detailed estimation it is used PVsyst tool that allow configuration of the proposed projects using more detailed solar and weather data as primary inputs to the simulation.

### 3. Albanian's Energy System overview.

Having in mind the fact that electricity comes from RES (almost 100% hydro sources), a balanced configuration of the energy system dominated by fossil fuel powered vehicles (100%), will be preferred by an energy system with opposite fuel portions. This fact, together using technical approach of the current study will help in the selection of the optimal RES configuration in the transport sector. Combining different technologies that support new fuel types in compliance with the main two objectives of the national energy strategy and sector strategic action plan is mandatory. This analysis is very hard and may require the use of technologies that are still in the early stages of development. The total final energy consumption in Albania referring 2018 is 24 TWh (2081ktoe) where the consumptions by different sectors in Albania is given in table 2. Indeed, the transport sector is by far the biggest consumer of energy sharing 40% of PES in Albania. According to in 2018, Albania's total primary energy supply (TPES) amounted to 2131(ktoe).

**Table 2:** Distribution of primary energy supply by demand sector, 2018. Source: [1]

| Sector      | %  |
|-------------|----|
| Transport   | 40 |
| Residential | 24 |
| Industry    | 20 |
| Services    | 10 |
| Other       | 6  |

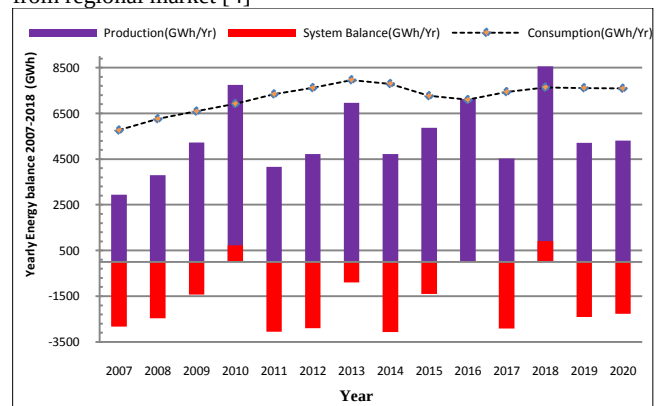
**Table 3:** Fuels going through Final Energy Consumption in Albania, 2018. Source [1]

|                          | %          |
|--------------------------|------------|
| Crude, NGL and Feedstock | 57         |
| Hydro electricity        | 25         |
| Solid fuels              | 9          |
| Biomass (Fuelwood)       | 8          |
| Solar energy             | 1          |
| NG                       | Negligible |
| Derived heat             | 0          |

Distribution of energy by fuel type going to the final energy consumption in Albania is given in table 3. Crude oil cover around 57% of the final energy consumption in the country followed by hydroelectricity 25%, biomass as wood fuel type 8%, solid fuel 9% and the rest (1%) comes from solar energy. Albania's electricity demand grew rapidly in 1995- 2000. This was due to demographic, economic and social trends, including rural-to-urban migration, increased use of electricity for space heating and cooling and rising living standards [16].

The Albanian power system is dominated by hydropower, representing more than 95% of the country's installed capacity with a total of 2,506 MW installed. The majority of the installed capacity (1448MW) 58% is owned by the Albanian Power Corporation (KESH) and the rest 42% belongs to the private producers (1058MW) [4] (where the installed capacity of independent producers is 436 MW, while 622 MW belongs to the producers with priority). According to [4] the installed hydropower capacity comprises mainly large hydropower installations (i.e., above 10 MW in size) constituting 1904MW, while small hydropower installations amount to 192MW. The country has a 98MW fossil-fuel thermal power plant representing 4% of the total installed capacity that has not been put into use since its construction in 2011 due to a failure in its cooling system.

The remaining 1% (10 MW) of installed capacity in 2018 comprised small-scale (i.e., each less than 2 MW) solar photovoltaic (PV) plants. The electricity import varies and depend on weather conditions. Historically about 3TWh/year of electricity is imported from regional market [4]



**Figure 3:** Yearly electricity balance in Albania (GWh), 2007-2020. [4]

The net domestic electricity production of 5313 GWh is registered during 2020. As can be seen in the graph in figure 3, the lowest electricity consumption recorded in our country is in 2007 with 5,767 GWh and the highest electricity consumption is the one recorded in 2013 with 7855 GWh. For 2020, electricity consumption in our country is 7,588 GWh. Compared to 2019, there is a slight decrease in electricity consumption in the country by 23.4 GWh. Generation of electricity for 2020 is about 9.7% lower than average production for the 2009-2020 period. Despite the increase in production capacity during 2020 due to a bad hydrological year, the electricity production for this year has resulted in the amount of 5,313 GWh or 569 GWh less than average electricity production in the 2009-2020 period. This indicator clearly represents the high degree of hydrological risk in the production stability of electricity in our country, which bases the production of electricity mainly on hydro sources.

Technical and non-technical losses still remain critical issues for the national power system accounting for around 21.7% of the total electricity consumption in 2019. Between 2014 and 2018, the amount of net energy imported varies from 12% of the TPES in 2015 up to 46% in 2017.

#### 4. Solar energy progress.

The Paris Agreement sets a goal to limit the increase in global average temperature to well below 2°C above pre-industrial levels and to attempt to limit the increase to 1.5°C. Implicit in these goals is the need for a transition to a low-carbon energy sector, which accounts for two-thirds of global emissions. RES, coupled with energy efficiency gains, can provide 90% of the CO<sub>2</sub> emissions reductions in the roadmap to 2050. Renewable energy is therefore a key component of Nationally Determined Contributions (NDCs)—the central implementation tool for countries under the Paris Agreement. At present, the level of detail contained in NDCs differs from country to country, with little in-depth analysis and limited quantitative information about the role of renewable energy in meeting greenhouse gas (GHG) emission reduction targets [5]. Based on the targets projected in global level Albania is making efforts to reduce import of electricity, improve its security of supply and to attain the Paris Agreement. The Albanian ministry of Energy and Transportation and its dependency institutions has compiled the *"The National Energy Strategy 2018-2030"*, consisting on 6 possible scenarios of energy's transition process toward a sustainable and reliable energy by shifting Albania to decentralized renewable energy market, and energy efficiency. This strategy requires reaching a RES share of 42% to the total energy consumption and also reducing CO<sub>2</sub> (referring 2016) level up to 11.5% by the end of 2030. The first goal can be achieved by large scale integration of RES capacities, especially wind generation capacities [1]. The RES share in global electricity generation reached almost 27% in 2019, renewable power as a whole still needs to expand significantly to meet the SDS share of almost half of generation by 2030 which requires the rate of annual capacity additions to accelerate [5].

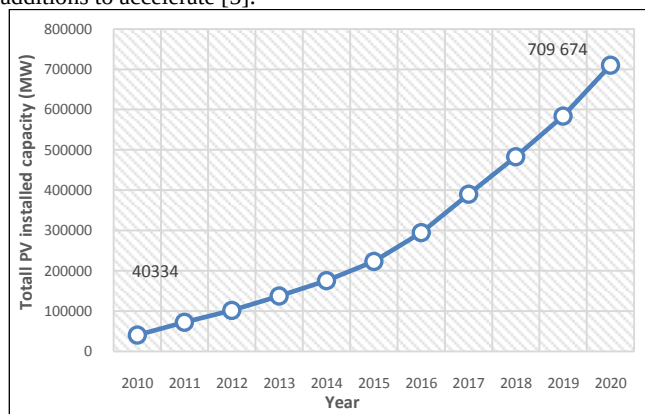


Figure 4: The global installed capacity trends of the PV systems.

In the graph in figure 4 the global installed PV capacity is given. The growth of PV capacity worldwide tends an exponential progress and results 18 times more in 2020 compared to 2010. The second national energy goal, compared to the baseline scenario in 2016, should be fully in line with EU objectives, its commitment is to reach a reduction of 11.5% of CO<sub>2</sub> emissions by the end of 2030. Considerable interest in RES and significant increases in cost of imported oil and very frequent services of related power generation technologies have compelled various countries to search for low-cost energy sources and improved technologies based on RES, PV and synergies between systems to achieve lower cost of electricity generation.

#### 5. Economic aspects of PV systems

Three key factors are essential when designing PV power plants. **First** there must be a sufficient source of solar potential in the proposed region,

**Second**, PV technology must be promising as well as cost effective. Studies has shown that it is cost-effective for small loads (less than 10 kWp) need lower capital costs than grid extension and have lower O&M.

**Third**, possibility for shortest distribution lines and access to the national distribution-transmission lines.

Any factor that leads to lower total lifecycle costs, or that yields greater kWh over the chosen analysis period, lowers the LCOE of a PV system. The total lifecycle cost in the numerator is a function of the initial capital cost (which primarily includes the module, the installation hardware and labour, and transaction costs for system installers and financiers), as well as ongoing operation and maintenance expenses (which oftentimes includes inverter replacement) and decommissioning costs including module collection and recycling. The global weighted-average LCOE of utility-scale PV plants declined by 82% between 2010 and 2019, from around \$0.378/kWh to \$0.068/kWh in 2019, with a 13% reduction year-on-year in 2019. At an individual country level, the weighted average LCOE of utility-scale solar PV declined by between 66% and 85% between 2010 and 2019.

#### 6. PV Project Cost distribution

Although the cost of PV panels energy has dropped dramatically in the last 10 years, technology requires a higher initial investment than traditional fossil fuel generators. Approximately 55% of the cost goes to module, 15% to inverter and the rest is belongs to BoS and installation costs (see graph in figure 5).

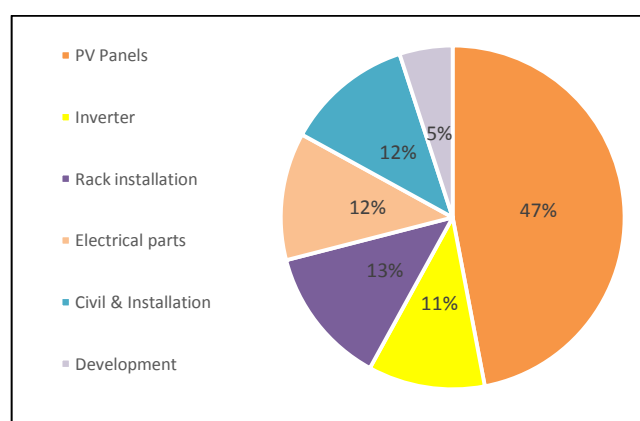
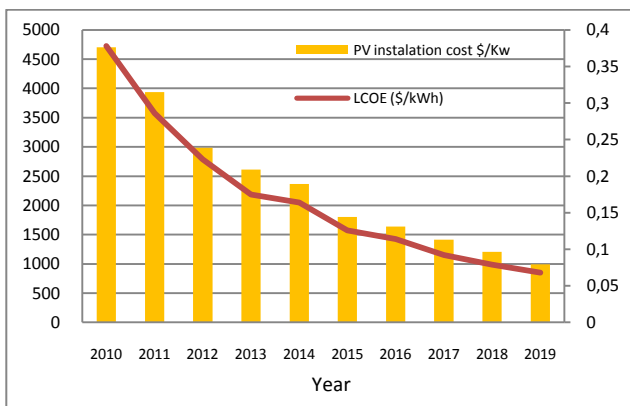


Figure 5: Cost breakdown of PV system components (%).

#### 7. Capital Investment Cost

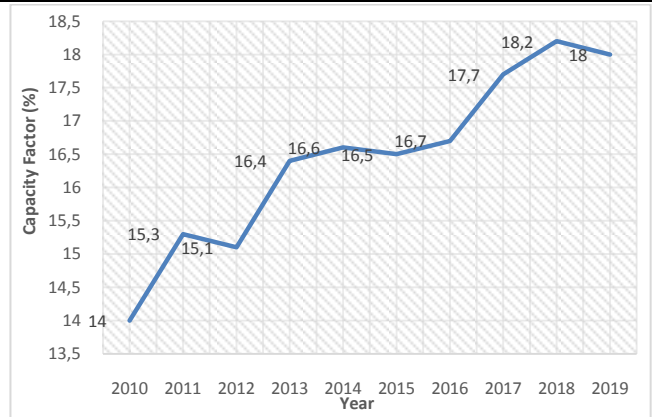
With very rapid reductions in solar PV module and balance of system costs, utility-scale solar PV is now increasingly competing head-to-head with alternatives and without financial support. Lower solar PV module prices and ongoing reductions in balance of system costs remain the main driver of reductions in the cost of electricity from solar PV. The costs for renewable energy

technologies reached new lows again last year. Solar and wind power have emerged as the most affordable power source for many locations and markets, with cost reductions set to continue into the next decade. The latest improved manufacturing processes and enhanced module efficiency enabled are the key drivers of lower module costs. In addition, as project developers gain more experience and supply chain structures continue to develop in more and more markets, declining BoS costs have followed. This has led to an increased number of markets where PV systems are achieving competitive cost structures and resulted in falling global weighted-average total installed costs [1]. In 2019, significant total installed cost reductions have occurred across all the major markets such as China, India, Japan, Republic of Korea and the United States. An increasing number of cost competitive projects in India led to weighted average total installed costs of \$618/Kw in 2019, around a fifth lower than in China. However, competitive cost structures are not confined to established markets anymore. Between 2010 and 2019, total installed costs have declined between 74% and 88% in markets where historical data is available back to 2010. The global capacity weighted-average total installed cost of projects commissioned in 2019 was \$995/kW, 18% lower than in 2018 and 79% lower than in 2010 (see graph in figure 6). Based on the costs of the developed projects around the globe, yearly variation of total installation cost and LCOE is given graphically in figure 6.



**Figure 6:** Global weighted average total installed costs, capacity factors and LCOE for PV, 2010–2019 [6].

An important driver of improved competitiveness historically, the downward trend in solar PV module costs continued during 2019. By the end of 2009 and 2019, crystalline silicon module prices declined between 87% and 92% for modules sold in Europe, depending on the type. The weighted average cost reduction could be in the order of 90% during that period. More recently the cost of mainstream module technology declined 14% between December 2018 and December 2019, reaching \$ 0.27/W. A wide range of costs exists, however, depending on the type of module considered, with costs for December 2019 varying from as low as \$0.21/W for the lower cost modules to as high as \$0.38/W for all black modules. The cost of high efficiency crystalline modules at \$0.37/W was slightly above thin film offerings, which sold for \$0.36/W during that period. These costs declines and the advances in the ability to securely operate the grid with high shares of variable renewables are not only decarbonizing the electricity sector but are unlocking low-cost decarbonization in the end-use sectors in conjunction with increased electrification. On average, in 2019, balance of system costs (excluding the module and inverter) made up about 64% of total installed costs. In 2019, total BoS costs ranged from a low of 48% in India to a high of 76% in the Russian territories. Overall, soft cost categories for the evaluated countries made up around 40% of total BoS costs and about a quarter, on average, of the total installed costs. In 2016, these values were a third and 17% respectively.



**Figure 7:** Global weighted average capacity factors for utility-scale PV systems by year of commissioning, 2010–2019 [6].

The global weighted-average capacity factor for new, utility-scale solar PV, increased from 13.8% in 2010 to 18.0% in 2019. This was predominantly driven by the increased share of deployment in sunnier locations. A constant increased between 2010 and 2018 of the capacity factor is given in Figure 7. The development of the global weighted-average capacity factor is a result of multiple elements working at the same time. Higher capacity factors in recent years have been driven by the shift in deployment to regions with higher irradiation, the increased use of tracking devices in the utility-scale segment in large markets and a range of other factors that have made a smaller contribution (e.g., reduction in system losses).

## 8. Operation and Maintenance Costs

The Operation and Maintenance costs of utility-scale solar PV plants have declined in recent years. However, in certain markets, the share of O&M costs in total LCOE has risen, as capital costs have fallen faster than O&M costs. O&M cost declines have been driven by module efficiency improvements leading to reduced surface area required per MW of capacity. At the same time, competitive pressures and improvements in the reliability of the technology have resulted in system designs optimised to reduce O&M costs and improved O&M strategies that take advantage of a range of innovations from robotic cleaning to “big data” analysis of performance data to identify issues and preventative interventions ahead of failures driving down O&M costs and reducing downtime. For the period 2018-2019, O&M cost estimates for utility-scale plants in the USA have been reported at between \$(10-18)/kW per year. Recent costs there seem to be dominated by preventive maintenance and module cleaning, with these making up as much as 75% and 90% of the total, depending on the system type and configuration. The rest of the O&M costs can be attributed to unscheduled maintenance, land lease costs and other component replacement costs. The current benchmarks without inverter replacement are \$11.5/kW/year (residential), \$12.0/kW/year (commercial), \$9.1/kW/year (utility-scale, fixed-tilt), and \$10.4/kW/year (utility-scale, tracking), significantly below previous O&M, only benchmark estimates. Average utility-scale O&M costs in Europe have been recently reported at \$10/kW/year, with historical data for Germany suggesting O&M costs came down 85% between 2005 and 2017, to \$9/kW/year.

## 9. Results and discussion

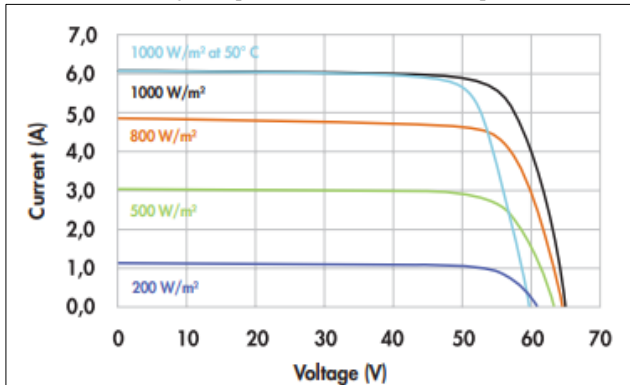
PV configuration to supply on grid electricity in Albania was presented. The perspective of a wide use of green power motivates the scientific community to study the possibility of fabricating PV modules providing on grid electricity production. The technical aspects of module type selection are of particular importance as a fairly technical choice of PV module type and type has a direct impact on the annual revenue generated. Based on credible references such as IRENA; IEA etc. PV should achieve CF at least over 16%. In our case study, the proposed PV project located in

Tërbuf village part of Divjaka Municipality referring the chosen PV module, SunPower module performs satisfactorily and achieves an efficiency of 19.3%.

**Table 4:** Main technical parameters (electrical and physical) of the PV module.

| Model Type                                                                                                                                                 | SunPower model mono-Si - E19-310-COM |           |
|------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------|-----------|
| Nominal Power (+5/-3%) (P <sub>nom</sub> )                                                                                                                 | W                                    | 310       |
| Efficiency                                                                                                                                                 | %                                    | 19        |
| Rated Current (I <sub>mpp</sub> )                                                                                                                          | A                                    | 5.67      |
| Rated Voltage (V <sub>mpp</sub> )                                                                                                                          | V                                    | 54.7      |
| Short Circuit Current (I <sub>sc</sub> )                                                                                                                   | A                                    | 6.05      |
| Open Circuit Voltage (V <sub>oc</sub> )                                                                                                                    | V                                    | 64.4      |
| Nominal operating temperature of cells (NOCT)                                                                                                              | °C                                   | 45 ±2     |
| Maximum System Voltage                                                                                                                                     | V                                    | 1000      |
| Standard Operation Temperature (T <sub>c</sub> )                                                                                                           | °C                                   | 25        |
| Specific energy (E)                                                                                                                                        | W/m <sup>2</sup>                     | 1000      |
| Dimensions                                                                                                                                                 | mm                                   | 1559x1046 |
| Weight                                                                                                                                                     | kg                                   | 18.6      |
| Technology                                                                                                                                                 | mono-Si                              |           |
| Class                                                                                                                                                      | Class A                              |           |
| Impact Resistance                                                                                                                                          | Hail – 25 mm at 23 m/s               |           |
| Max load-550 kg/m <sup>2</sup> (5400 Pa), front (e.g. snow) w/specified mounting configurations 245 kg/m <sup>2</sup> (2400 Pa) front and back - e.g. wind |                                      |           |
| Frame-Anodised aluminium alloy type 6063 (silver); stacking pins                                                                                           |                                      |           |

In the above table the electrical and mechanical features of the chosen cell type are given. The type of module is chosen based on the overall efficiency and prices available from the producer.



**Figure 8:** Current/voltage characteristics with dependence on irradiance and module temperature.

It can be clearly seen that the cell output voltage related with the temperature and the cell output current is affected by irradiation level. Due to these characteristic dependencies, extraction of maximum power from PV modules mainly changed by the temperature and irradiance level. Furthermore, for specifying rating of power electronics equipment, these variations should be taken into consideration. Open circuit voltage ( $V_{oc}$ ) is primarily affected by temperature and the relationship between them is inversely proportional whereas the current is only slightly dependent. This relationship can be extracted by using Equation (1).

$$V_{oc} = V_{oc-SGT} - \gamma \cdot (T - T_{STC}) \quad 1$$

$\gamma$  is a constant which can be obtained from datasheet of a PV module. It is a negative value and shows change of open circuit voltage by increasing or decreasing ambient temperature for 1 °C.

$$I_{sc} = V_{sc-SGT} \cdot (1 + \alpha \cdot (T - T_{STC})) \cdot \frac{G}{G_{STC}} \quad 2$$

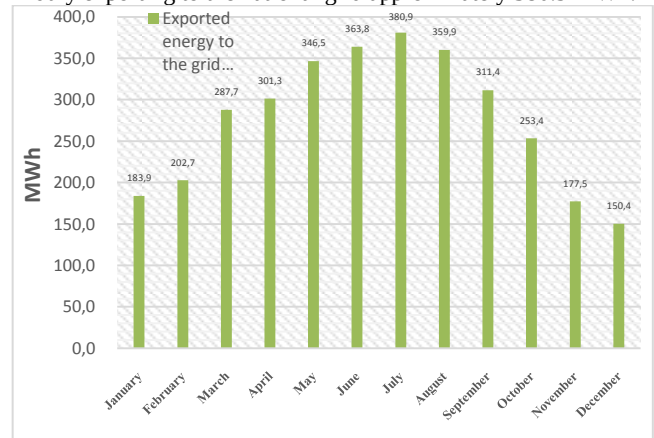
Short circuit current ( $I_{sc}$ ) is mostly affected by irradiation level and the relationship between them is directly proportional as shown in Equation 2.

**Table 5:** Summary of the proposed On-grid PV system.

| Sunpower mono-Si - E19-310-COM |                |       |
|--------------------------------|----------------|-------|
|                                | Unit           |       |
| Power                          | Wp             | 310   |
| Number of modules              | Pieces         | 6452  |
| CF                             | %              | 19.3  |
| Annual energy production       | MWh            | 3319  |
| Area                           | m <sup>2</sup> | 1.538 |
| Weight                         | kg             | 18.6  |
| Module Surface                 | m <sup>2</sup> | 10364 |
| Total soil area                | m <sup>2</sup> | 20728 |
| Inverter 30KW 450-700V         | Pieces         | 52    |
| Max 900V LF Tr 50Hz            |                |       |

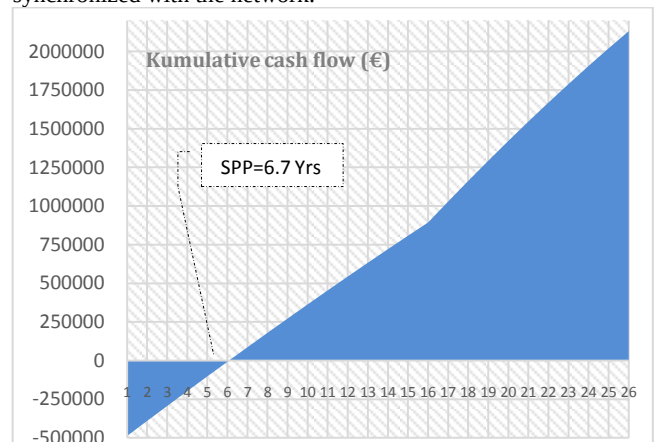
As it can be seen from table 5 the capacity factor of the chosen module is 19.3%. For proposed PV system of 2MW of capacity is needed approximately 6452 PV modules and 52 inverters.

To design a power electronic inverter for grid-tied PV system, an overview of rules and regulations should be investigated in order to be allowed to connect to the grid. There are various grid codes, standards and related documents are available. As it can be seen from in figure 9 the proposed PV system of 2MW of capacity can generate 3319 MWh per year. The maximum of its production falls in July exporting to the national grid approximately 380.9 MWh.



**Figure 9:** Monthly electricity generation (MWh) from the proposed PV plant.

Before making a network connection of a PV system, it should be evaluated to show how it affects the network. From the simulation in RETScreen Expert the annual energy production is 3319MWh. Further investigation needs to be performed on how the system synchronized with the network.

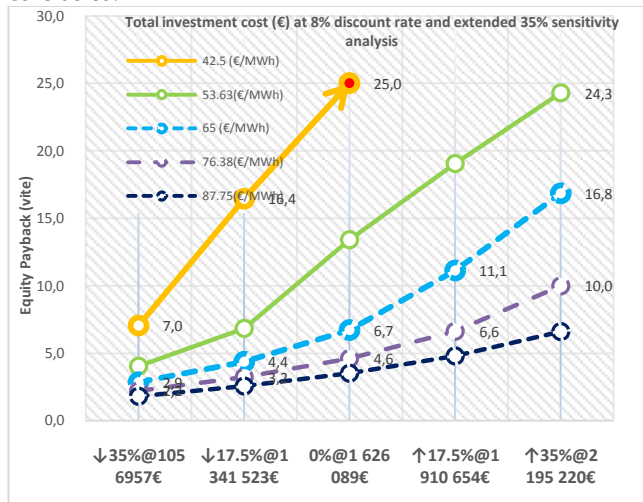


**Figure 10:** Pay back period of the proposed PV power plant in years.

From the cash flow analysis it results that the investment return period occurs within 6.7 years. This is a positive indicator of the

investment of the photovoltaic plant with a capacity of 2MW and annual energy generated 3319MWh.

For a chosen total investment of 743€/kW and a benchmark of electricity rate the SPP results 6.7 years. In the sensitivity analyses it is clearly shown that the total investment cost and electricity export rate are the most important parameters that affect other economical indicators of the proposed system. In our calculation other costs such as the grid extension cost and O&M costs is considered.



**Figure 11:** Total investment cost (€) at 8% discount rate and extended 35% sensitivity analysis

## 10. Conclusion

The supply of electricity from inland production is sometime risky and highly affected on weather conditions. In the current situation, more than ever, it is necessary to encourage and realize investments in the field of electricity generation to overcome energy crises in our country. From the official statistics on energy balance especially

to that of electricity generation and import ratio, in the last 10 years our country has survived a positive balance only in 2010, 2016 and 2018, Albania has imported electricity from the region at a high price, aggravating the economic situation of energy supply (refer graph in figure 3) in the last 4 years, our country has imported 2915GWh, 913GWh, 2406GWh and 2276GWh electricity. The maximum annual electricity that we can produce by the system is 3319 MWh and SPP results 6.7 years if the total installation cost is assumed 743€/kW and electricity export rate 65€/MWh.

Hence, the implementation of small PV on grid power plants is very important for the diversification of the energy sector in Albania, will push the economy opening new jobs and reducing the electricity import.

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# MODELING OF FUNCTIONAL DEPENDENCES BY HERMITIAN SPLINES WITH EXPONENTIAL-POWER EXPRESSION IN LINKS

Bohdan Markovych<sup>1,2</sup>, Ihor Medynskyi<sup>3</sup>, Yaropolk Pizyur<sup>1,4</sup>

<sup>1</sup> Lviv Polytechnic National University, Lviv, Ukraine

<sup>2</sup> bohdan.m.markovych@lpnu.ua,  <https://orcid.org/0000-0002-8813-9108>

<sup>3</sup> ihor.p.medynskyi@lpnu.ua,  <https://orcid.org/0000-0002-0651-4198>

<sup>4</sup> yaropolk.v.pizyur@lpnu.ua,  <https://orcid.org/0000-0001-8001-7256>

**Abstract:** The definitions of Hermitian splines with nonlinear expressions in the links are given. Formulas for the parameters of Hermitian splines with exponential-power links with four parameters are derived. The definition of the balance approximation of functions by splines is given. The formula for the balance approximation error for Hermitian splines with four parameters are obtained. The error of approximation of functions by polynomial Hermitian splines is compared with the error of approximation of functions by Hermitian splines with exponential-power expressions in the links.

**Keywords:** HERMITIAN SPLINES, BALANCE APPROXIMATION, APPROXIMATION ERROR, APPROXIMATION ERROR KERNEL

## 1. Introduction

Approximation of functional dependencies is necessary for practical calculations in many fields of technology and in research. Analytically given functions, which are represented by a complex expression, often need to be replaced by a simpler expression, so that their properties are preserved. This is required when calculating functions on a computer.

To increase the accuracy of the representation of functional dependencies, various approximation methods are used: minimax approximations, least squares method, spline approximation of different types and so on. A number of problems require approximation not only of the function itself, but also its derivatives. Hermite splines are used for this purpose in papers [1,2,3]. To improve the accuracy of the approximation of functions in the spline links, you can use not only polynomials, but also nonlinear expressions

$$(1) \quad F(A; x) = F(a_0, a_1, \dots, a_m; x).$$

In works [4,5,6] expressions are got for the parameters of Hermitean splines with exponential and logarithmic links, by links as a sum of exponent and polynomial, relation of polynomial to the linear function. In works [7,8] formulas are shown out for the errors of approximation of functions by polynomial Hermitean splines. By analogy to the minimax approximation [9] in the article [4,10] a theorem that allows to take approximation nonlinear Hermitean splines with difficult expressions in links (for example superposition of power function and a polynomial) to polynomial Hermitean is proved.

## 2. Formulation of the problem

Let  $\alpha$  and  $\beta$  be given real number, such that  $0 < \alpha < \beta < \infty$ . Denote by  $X = \{x \in [\alpha, \beta] : \alpha = x_0 < x_1 < \dots < x_r = \beta\}$  the set of nodes of interpolation.  $\theta(u) = \begin{cases} 1, & u \geq 0 \\ 0, & u < 0 \end{cases}$  – the characteristic

function of half-interval  $[0, \infty)$ .  $C^n[\alpha, \beta]$  – the set of functions  $f : [\alpha, \beta] \rightarrow R$  which have continuous derivatives  $f^{(k)}(x)$ ,  $k = \overline{0, n}$ . Assume that values of the function  $f(x) \in C^n[\alpha, \beta]$  and its derivatives up to  $n$  are given on the set  $X$ . It is needed to build a Hermitian spline with the links of the form

$$(2) \quad F_1(A; x) = a_0 \exp(a_1 x) x^{a_2 + a_3 x}, \quad x > 0,$$

$$(3) \quad F_2(A; x) = a_0 \exp(a_1 x + a_2 x^2) x^{a_3}, \quad x > 0,$$

where  $a_i$  – parameters of links of spline,  $i = \overline{0, 3}$ , i. e. quantity of parameters is even  $m + 1 = 4$ .

The aim of the article is to construct Hermitian splines with exponential-power links with four parameters and to obtain a formula for estimating the error of approximation for functions by these splines.

## 3. Definition of Hermitian splines with nonlinear expressions in the links

We give the definition of Hermitian splines with nonlinear expressions in the parameters (below we shall use the shot form nonlinear Hermitian splines) with an even number of parameters..

**Definition 1.** Suppose that functions  $f(x)$ ,  $F(A; x) = F(a_0, a_1, \dots, a_m; x) \in C^n[\alpha, \beta]$ . A function

$$(4) \quad S(A; x) = \sum_{k=1}^r F(A_k; x) * \theta((x - x_{k-1})(x_k - x)),$$

which satisfy the system of equations

$$(5) \quad f^{(i)}(x_k) - S^{(i)}(A; x_k) = 0; \quad i = \overline{0, n}; \quad k = \overline{0, r},$$

where  $A_k$  - parameters  $k$ 's link of spline, is called the nonlinear Hermitian spline with even quantity of parameters  $m + 1$  ( $m = 2n + 1$ ).

From system (5) we get  $S(A; x) \in C^n[\alpha, \beta]$ . An expression  $F(A_k; x)$  is called the link of Hermitian spline. Approximation error of function  $f(x)$  by using Hermitian spline  $S(A; x)$  is characterized weighting distance (function of error)

$$(6) \quad \rho(x) = [f(x) - S(A, x)] / w(x),$$

where weighting function of approximation  $w(x) \in C^n[\alpha, \beta]$ ,  $w(x) \neq 0$  for  $x \in [\alpha, \beta]$ .

From definition 1 follows, that for determination of parameters for every link of a nonlinear Hermitian spline it is necessary solve the system of equations (5).

## 4. Derivation of formulas for the parameters of Hermitian splines with exponential-power links

First, we construct a Hermitian spline with a link, which is represented by expression (2). Since the approximate expression (2) does not change the sign, this expression can be used to

approximate functions that do not change the sign. Suppose for specificity that  $f(x) > 0$ .

According to definition 1 parameters of the link of Hermitian spline (4) with the link (2) satisfy a system of equations (5). In this case we have the following system:

$$(7) \quad \begin{cases} f_0 - a_0 \exp(a_1 x_0) x_0^{a_2 + a_3 x_0} = 0 \\ f'_0 - a_0 \exp(a_1 x_0) x_0^{a_2 + a_3 x_0 - 1} \times \\ \quad \times (a_3 x_0 (1 + \ln x_0) + a_2 + a_1 x_0) = 0 \\ f_1 - a_0 \exp(a_1 x_1) x_1^{a_2 + a_3 x_1} = 0 \\ f'_1 - a_0 \exp(a_1 x_1) x_1^{a_2 + a_3 x_1 - 1} \times \\ \quad \times (a_3 x_1 (1 + \ln x_1) + a_2 + a_1 x_1) = 0 \end{cases},$$

where  $x_0$  – left limit and  $x_1$  – right limit of the link,  $x_0 < x_1$ ,  $f_i^{(j)} = f^{(j)}(x_i)$ ,  $i = 0, 1$ ,  $j = 0, 1$ . Let us solve the system (7) for unknown parameters  $a_i$ ,  $i = \overline{0, 3}$ .

From the first and third equations of the system (7) we find the expressions for the parameter  $a_0$

$$(8) \quad \begin{aligned} a_0 &= f_0 \exp(-a_1 x_0) x_0^{-(a_2 + a_3 x_0)}, \\ a_0 &= f_1 \exp(-a_1 x_1) x_1^{-(a_2 + a_3 x_1)}. \end{aligned}$$

We equate the expressions for  $a_0$  and get the expression  $a_1$

$$(9) \quad a_1 = \frac{1}{x_1 - x_0} \left( \ln \frac{f_1}{f_0} - (a_2 + a_3 x_0) \ln x_0 - (a_2 + a_3 x_1) \ln x_1 \right).$$

Substitute the first expression for  $a_0$  and the expression for  $a_1$  into the second equation of system (7) and obtain a linear equation with respect to unknown parameters  $a_2$  and  $a_3$

$$(10) \quad \eta_1 a_2 + \theta_1 a_3 = \gamma_1,$$

where

$$\begin{aligned} \eta_1 &= 1 + \frac{x_0}{x_1 - x_0} (\ln x_0 - \ln x_1), \\ \theta_1 &= x_0 \left( 1 + \ln x_0 + \frac{x_0}{x_1 - x_0} \ln x_0 - \frac{x_1}{x_1 - x_0} \ln x_1 \right), \\ \gamma_1 &= x_0 \frac{f'_0}{f_0} - \frac{x_0}{x_1 - x_0} \ln \frac{f_1}{f_0}. \end{aligned}$$

Substitute the second expression for  $a_0$  and the expression for  $a_1$  into the fourth equation of system (7) and obtain the second linear equation with respect to unknown parameters  $a_2$  and  $a_3$

$$(11) \quad \eta_2 a_2 + \theta_2 a_3 = \gamma_2,$$

where

$$\begin{aligned} \eta_2 &= 1 + \frac{x_1}{x_1 - x_0} (\ln x_0 - \ln x_1), \\ \theta_2 &= x_1 \left( 1 + \ln x_1 + \frac{x_0}{x_1 - x_0} \ln x_0 - \frac{x_1}{x_1 - x_0} \ln x_1 \right), \end{aligned}$$

$$\gamma_2 = x_1 \frac{f'_1}{f_1} - \frac{x_1}{x_1 - x_0} \ln \frac{f_1}{f_0}.$$

We have obtained a system of two linear equations (10) and (11) with respect to two unknowns parameters  $a_2$  and  $a_3$ . Solving it we get expressions for the parameters  $a_2$  and  $a_3$

$$(12) \quad a_2 = \frac{\gamma_1 \theta_2 - \gamma_2 \theta_1}{\mu_1 \theta_2 - \eta_2 \theta_1},$$

$$(13) \quad a_3 = \frac{\gamma_1 - \eta_1 a_2}{\theta_1}.$$

From formulas (8) – (9) and (11) – (12) for the parameters  $a_i$ ,  $i = \overline{0, 3}$  follow that the necessary condition for the existence of the Hermitian spline approximation with link (2) is the fulfillment of the condition  $x > 0$  and.

We derive the expressions for the Hermitian spline with the link (3). According to definition 1, the parameters of the link for the Hermitian spline (4) with the link (3) satisfy the system of equations (5). We have the following system:

$$(14) \quad \begin{cases} f_0 - a_0 \exp(a_1 x_0 + a_2 x_0^2) x_0^{a_3} = 0 \\ f'_0 - a_0 \exp(a_1 x_0 + a_2 x_0^2) x_0^{a_3 - 1} \times \\ \quad \times (a_3 + x_0 (a_1 + 2a_2 x_0)) = 0 \\ f_1 - a_0 \exp(a_1 x_1 + a_2 x_1^2) x_1^{a_3} = 0 \\ f'_1 - a_0 \exp(a_1 x_1 + a_2 x_1^2) x_1^{a_3 - 1} \times \\ \quad \times (a_3 + x_1 (a_1 + 2a_2 x_1)) = 0 \end{cases},$$

where  $x_0$  – left limit and  $x_1$  – right limit of link,  $x_0 < x_1$ ,  $f_i^{(j)} = f^{(j)}(x_i)$ ,  $i = 0, 1$ . Let us solve system (14) with respect to the unknowns parameters  $a_i$ ,  $i = \overline{0, 3}$ . The process of deriving formulas for parameters is similar to deriving formulas for parameters of Hermitian spline with link (2). Therefore, we omit the detailed conclusion and present the final result. The parameters  $a_1$  and  $a_2$  are the solution of a system of two linear equations and are calculated by formulas

$$(15) \quad a_1 = \frac{\gamma_3 \theta_4 - \gamma_4 \theta_3}{\eta_3 \theta_4 - \eta_4 \theta_3},$$

$$(16) \quad a_2 = \frac{\gamma_3 - \eta_3 a_1}{\theta_3}.$$

where

$$\eta_3 = \frac{x_1 - x_0}{\ln \frac{x_0}{x_1}} + x_0, \quad \theta_3 = \frac{x_1^2 - x_0^2}{\ln \frac{x_0}{x_1}} + 2x_0^2,$$

$$\gamma_3 = x_0 \frac{f'_0}{f_0} + \frac{\ln \frac{f_1}{f_0}}{\ln \frac{x_0}{x_1}}, \quad \theta_4 = x_1^2 \left( 1 + 2 \ln \frac{x_1}{x_0} \right) - x_0^2$$

$$\eta_4 = x_1 \left( 1 + \ln \frac{x_0}{x_1} \right) - x_0, \quad \gamma_4 = x_1 \frac{f'_1}{f_1} \ln \frac{x_0}{x_1} + \ln \frac{f_1}{f_0}.$$

Parameters  $a_3$  and  $a_0$  we calculate by formulas

$$(17) \quad a_3 = \frac{a_1(x_1 - x_0) + a_2(x_1^2 - x_0^2) - \ln \frac{f_1}{f_0}}{\ln \frac{x_0}{x_1}},$$

$$(18) \quad a_0 = f_0 \exp(-a_1 x_0 - a_2 x_0^2) x_0^{-a_3}.$$

It follows from formulas (15) - (18) for the parameters  $a_i$ ,  $i = \overline{0,3}$  that the necessary condition for the existence of the Hermitian spline approximation with link (3) is the fulfillment of the condition  $x > 0$  and  $f(x) > 0$ .

### 5. Error of approximation of functions by Hermitian splines

One of the important characteristics of splines as a means of approximating functions is the error of approximation. Here is the definition of balance (with the same maximum error on each link) approximation of functions by splines.

**Definition 2.** The approximation of a function  $f(x)$  by a Hermitian spline  $S(A; x)$  is called a balance approximation with a given quantity of links  $r$ , if

$$\mu_i = \max_{x \in [z_{i-1}, z_i]} |(f(x) - S(A; x)) / w(x)| = \mu, \quad i = \overline{1, r},$$

where  $\mu_i$  – error of approximation for the link with number  $i$  which define on the segment  $[z_{i-1}, z_i]$ ,  $z_i$  – extreme points of links,  $\{x, z_i\} \subset [\alpha, \beta]$ ,  $i = \overline{1, r}$ .

It is known [1,4] that the error of the balance approximation of functions by Hermitian splines with an even number of parameters is calculated by the formula

$$\mu = \frac{1}{2^{m+1}(m+1)!r^{m+1}} \left( \int_{\alpha}^{\beta} \left| \frac{\eta(f, F)}{w(x)} \right|^{\frac{1}{m+1}} dx \right)^{m+1} \left[ 1 + O\left(\frac{\beta - \alpha}{r}\right) \right],$$

where  $r$  – quantity of links for spline on the segment  $[\alpha, \beta]$ ,  $w(x)$  – weighting function,  $\eta(f, F)$  – kernel of error of approximation. To use formula (19), it is necessary to have an expression for the kernel of error of approximation  $\eta(f, F)$ , which would not depend on the parameters  $a_i$ ,  $i = \overline{0,3}$  the link  $F(A; x)$  of the spline. Expressions for kernels can be found using the properties of kernel of errors that follow from exchange theorems [4,10]. So for links (2) and (3) the kernel of error has the form

$$\eta(f, F_1) = f(x) \frac{d^2 \ln(x^2 (\ln f(x))'')}{x^2 dx^2},$$

$$\eta(f, F_2) = f(x) \frac{d^3 \ln(x (\ln f(x))')}{x dx^3}.$$

It is known [4], that for Hermitian splines with a link in the form of a polynomial of the third degree the kernel of error is equal to  $\eta(f, F_3) = f^{(4)}(x)$ .

### 6. Results and discussion

A number of numerical experiments are performed. Thus, when approximating the function  $\tan(x)$  on the segment  $[0.1, \pi/3]$ , the error of approximation by the Hermitian spline with the link (2) is 0.053638, with the link (3) – 0.032912 and by the Hermitian spline with the link in the form of a polynomial of the third degree – 0.086386. When approximating the function  $\frac{1}{1+x}$  on the segment  $[0.1, 10]$  the approximation error of the Hermitian spline with the link (2) is 0.047731 with the link (3) – 0.103071 and by the Hermitian spline with the link in the form of a polynomial of the third degree – 0.746865. From the given examples the advantage in accuracy of approximation by Hermitian splines with exponential-power links is obvious. The disadvantage of exponential-degree Hermitian splines is that they can approximate only functions that acquire positive values at  $x > 0$ .

### 7. Conclusion

Hermitian splines with exponential-power links of two types with four parameters are constructed. The formula for estimating the error of balance approximation of functions by Hermitian splines is given. Analytical expressions for kernels of errors for Hermitian splines with exponential-power links are obtained. Theoretical results are confirmed by practical calculations. Since the error of approximation of functions by Hermitian splines with exponential-power links is in some cases smaller than that of a polynomial Hermitian spline, it is expedient to use them to approximate functions. The prospect of research in this direction is the construction of Hermitian splines with more parameters in the links. Since physical processes are described by various nonlinear dependences (logarithmic, fractional-rational, in the form of the sum of a polynomial and exponential or power expressions), to obtain more accurate models of the described processes it is advisable to build and study Hermitian splines with other nonlinear expressions.

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