

MAGNETIC PULSE COMPACTION AND SUBSEQUENT SPARK PLASMA SINTERING OF NANOSTRUCTURED ALUMINA

МАГНИТНО-ИМПУЛЬСНОЕ ПРЕССОВАНИЕ И ПОСЛЕДУЮЩЕЕ СПАРК-ПЛАЗМЕННОЕ СПЕКАНИЕ НАНОСТРУКТУРНОГО ОКСИДА АЛЮМИНИЯ

Kovaleva I.¹, Zholnin A.², Grigoryev E.³, Olevsky E.^{4,5}
NRNU MEPhI, Russia^{1,2,3,4}, San Diego State University, USA⁵

Abstract: The purpose of this paper was to study the regularities of formation of ultrafine structure in alumina by magnetic pulse compaction (MPC) and spark plasma sintering, and the producing of nanostructured compacts having high density and microhardness. The combined application of two technologies magnetic pulse compaction and spark plasma sintering in the practice of compacting powders is very rare and unique. We have studied the microstructures of consolidated alumina samples. The anomalous zones present in volume of magnetic pulse compacted and spark plasma sintered samples of both types α and δ phases of alumina. The microstructure of the fracture surface between anomalous zones depends on the phase state of the particles of the initial powder. MPC of δ -alumina leads to a more uniform distribution of anomalous zones along diameter compact after SPS. MPC of α -alumina leads to an increase of the microhardness on the surface of compacts.

KEYWORDS: MAGNETIC PULSE COMPACTION, SPARK PLASMA SINTERING, NANOSTRUCTURE, ALUMINA, MICROHARDNESS

1. Introduction

The purpose was to study the regularities of formation of ultrafine structure in Al_2O_3 ceramics by magnetic - pulse compression and spark plasma sintering, and getting out of Al_2O_3 nanopowder compacts having high density and microhardness, observed experimentally in [1]. Overview magnetic pulse compaction and spark plasma sintering of powder materials [2] has showed that the combined application of these two technologies in the practice of compacting powders is very rare and unique.

The studies were conducted on the powder Al_2O_3 , obtained by the explosion of aluminium wire in the oxygen-containing gas produced by "Advanced Powder Technology", Tomsk. The specific surface area is 35-40 m^2/g . The mean particle size of 36 nm, an average size from the surface - 45 nm, a mass median size - 54 nanometers. Most of the powder was in a state of δ -phase. The part of the powder was heated in air to a temperature of 1300°C to transfer from unstable low temperature phases to stable α -phase.

2. Experimental results

Spark plasma sintering was performed in vacuum with LABOX-Sinter Land 625 in the graphite mold with an inside diameter of 10 mm at a pressure of 50 MPa. Temperature was determined by pyrometer. The mass of alumina powder was 0.4 g. The thickness of the resulting compacts was a little over 1 mm. Heating was performed at a rate of 100°C / min to 1400°C and staying 10 minutes at this temperature.

In conventional pressure compaction was 185 MPa. Pressure at MPC was defined by high voltage discharge. For δ - Al_2O_3 we have used a single pulse with a discharge voltage of 1.8 kV, and for α - Al_2O_3 we have used five pulses with a discharge voltage of 2.0 kV. Selected compaction modes correspond to the boundary of the overpressing. If MCP compact was cracked, then it was ground in a mortar and this powder was loaded into a mold for SPS.

SPS results of α and δ phases alumina powders with four kinds of pre-treatment are shown in Figures 1 and 2.

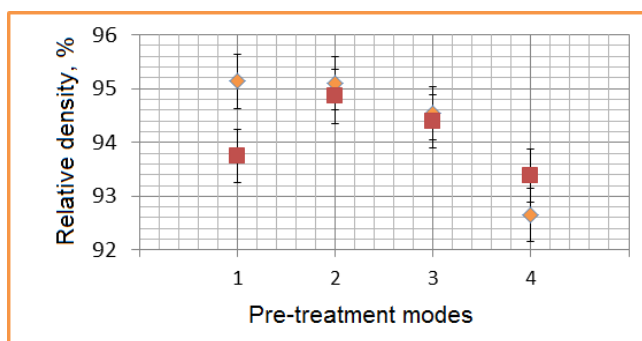


Fig. 1. Relative density of the SPS compact depending on the type of pre-treatment.

The numbers on the x-axis correspond to: 1 – the initial powder material (without training), 2 – powder after the traditional pressing, 3 - after the MPC, 4 – milled powder after MPC. Rhombs correspond to the original δ -phase Al_2O_3 powder, quadrates correspond to the α -phase Al_2O_3 powder or powder compact before the SPS.

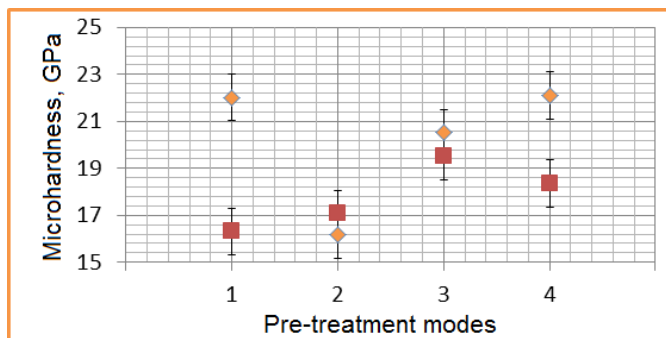


Fig. 2. Microhardness on the surface of SPS compacts depending on the type of pre-treatment.

At the fracture surface of the sintered pellets for both types α and δ phases of alumina powders present anomalous zones (AZ) a bimodal structure with length of about 300 microns with a microhardness much higher than on the surface microhardness of compacts (Fig. 3).

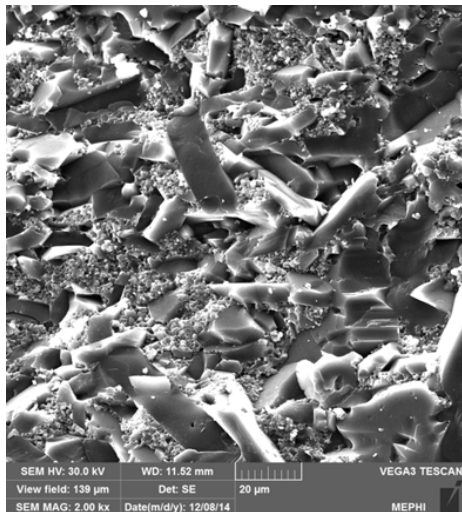


Fig. 3. Microstructure zones bimodal structure.

Dimensions of large crystallites in anomalous zones reach 40 microns. The proportion of fracture surface occupied by the anomalous zones increases from the edge to the center of the sample. In the center of the proportion of AZ is up to 40%. MPC leads to a more uniform distribution of AZ along the diameter of compact.

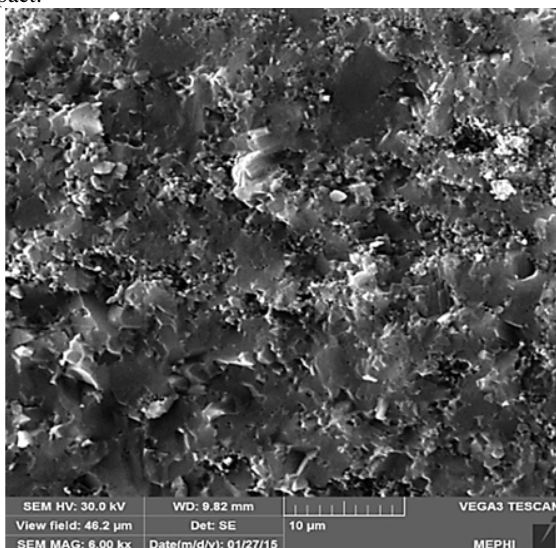


Fig. 4. Microstructure with alternating coarse and fine-grained layers (mainly for compacts of α -phase Al_2O_3).

The microstructure of the compact fracture surface between AZ depends on the phase state of the particles of the initial powder. The homogeneous microstructure of δ - Al_2O_3 powder regardless of the method of pre-treatment has an average grain size of 0.5-0.8 microns after SPS. Fine grains in anomalous zones have a similar

size. The microstructure of α - Al_2O_3 samples between AZ is bilayer. It consists of layers of large and small grains (Fig. 4).

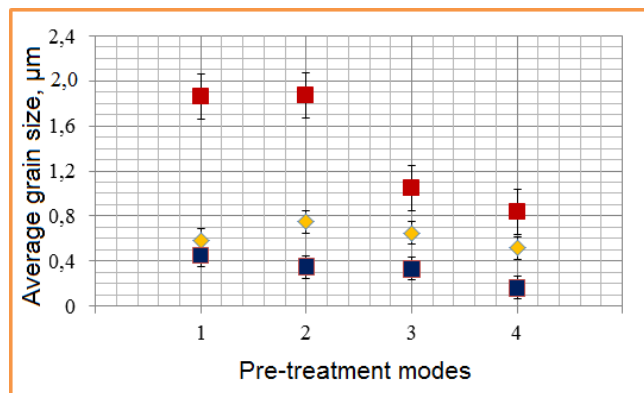


Fig. 5. The average grain size alumina outside the zones with abnormal structure (coarse-grained layers - red quadrates; fine-grained layers - blue quadrates; original δ -phase Al_2O_3 - rhombs)

Their dimensions are shown in Fig. 5.

3. Conclusion

Best results for the microhardness and density for all the studied options were obtained by SPS of initial nanopowder δ - Al_2O_3 without any pretreatment. MPC of δ -alumina before SPS leads to a more uniform distribution of anomalous zones along diameter compact. MPC of α -alumina before SPS leads to an increase of the microhardness on the surface of compacts, leads to equalize the distribution of anomalous zones along diameter compact and leads to reduce the size of the grains in a layered structure between AZ.

Acknowledgements

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References

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