EFFECTS OF MECHANICAL ALLOYING TIME AND ANNEALING TEMPERATURE ON THE PHYSICAL PROPERTIES OF Al - WC COMPOSITE POWDERS

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Abstract: *In this study, mechanical alloying (MA) of Al-WC powder system was studied to produce aluminium composite powders having finer tungsten carbide fraction in aluminium matrix. For this purpose, elemental mixtures of 70 wt. % aluminium (Al) powder and 30 wt. % of tungsten carbide (WC) powder were mechanical alloyed for the duration of 2, 4 and 8 hrs. MA'ed powders then annealed at 300 ºC, 400ºC and 500ºC for 2 hours under inert atmosphere. Apparent densities of powders were measured in order to characterize both mechanical alloyed and annealed powders. Compressibility of the powders was determined by green density measurements after pressing. Microstructural characterizations were conducted with X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. Strain rates and crystallite sizes were measured according to fundamentals parameter approach (FPA) by applying Lorentzian function using software TOPAS 4.2.*

Keywords: COMPOSITE POWDERS, Al-WC SYSTEM, MECHANICAL ALLOYING, ANNEALING, COMPRESSIBILITY

1. Introduction

Application of high reinforcement volume fractions, obtaining nano/fine particle size, homogeneous distribution of the particles throughout the metal matrix are the main advantages of powder metallurgy in order to manufacture metal matrix composites (MMCs) [1-3]. In addition, during mechanical alloying (MA) technique, the effects of particle size and amount of carbide addition were examined and finer hard particles inside of Al matrix in the presence of high WC amount were observed by the authors [4]. It was also reported by Meyers that ultrafine grain size and high volume fraction of interface have a great impact on the mechanical properties of nanocrystalline materials [5].

In this sense, metal matrix composites (MMCs) have been investigated, developed and applied for various industries due to their unique properties provided by the incorporation of nano/fine hard particles [6]. Among the various kind of MMCs, hard particle reinforced Al-based MMCs are a great of interest owing to their significant mechanical properties which can be possible to be tailored in order to meet specific requirements in particular for aerospace and automotive applications [7, 8]. However, it was shown that nano particles of hard phase may cause to agglomeration and clustering which leads to difficulties processing nanocomposite and decrease the mechanical properties especially when a high volume fraction of the reinforcement is applied [9-11]. Therefore, compressibility and flowability examinations by using various approaches were performed in order to analysis of on-going sintering behaviors of incorporated powders produced via MA [6, 12-18]

In this study, the effects of mechanical alloying (MA) time and annealing temperature on the physical properties of Al - WC composite powders are aimed. The motivation of this study is related with the results of our previous findings during mechanical alloying of Al-WC and Cu-WC systems [4, 19].

2. Experimental Procedure

In this study, Al (Alfa Aesar, -325 mesh, 99.5% purity) and WC powders (Alfa Aesar, -325 mesh, 99.5% purity) were used. Elemental Al and WC powders were blended to constitute the composition of Al –30 wt. % WC (hereafter called as Al30WC). Blended powders were mechanically alloyed (MA'ed) for 2, 4 and 8 h using a Spex™ Duo Mixer/Mill 8000D with a speed of 1200 rpm in a tungsten carbide (WC) vial with WC balls having a diameter of 6.35 mm (1 = 4 inches). The vials were sealed inside a Plaslabs™ glove box under purified Ar gas (99.995% purity) to prevent oxidation during MA. The ball-to-powder weight ratio (BPR) was 10:1. Apparent densities were carried out according to Arnold density measurement. Powders were annealed for 2 hours at 200^{o}C, 300^{o}C and 400^{o}C using Linn^{TM} furnace under H_2 atmosphere. The powder mixtures were uniaxially pressed in steel dies at 2 tons compaction pressure to obtain cylindrical samples of 6 mm diameter using MSE ForceMaster 9110 press machine with the speed of 50 mm/sec. In order to perform microstructural and crystallographic examinations, Bruker™ D8 advance X-ray diffractometer (XRD) with CuK_α radiation (λ=1.542 Å) was utilized. Crystallite size and strain rates were estimated according to fundamentals parameters approach (FPA) by applying Lorentzian function in TOPAS 4.2 (Bruker AXS) software.

The crystallite size was calculated by applying the modified Scherrer's [20] formula based on the XRD peak broadening obtained from XRD data:

$$D = \frac{\kappa\lambda}{\beta cos\theta} \quad (1)$$

Strain rates, which the strain broadening is calculated by following equation:

$$\varepsilon_0 = \frac{\beta}{4\cos\theta} \quad (2)$$

3. Results and Discussion

Theoretical density of Al30WC was calculated as 3.59 g/cm^3. Apparent densities of the MA'ed powders were given in Table 1.

Table 1. True densities of MA'ed powders

Powder	Milling Time (h)	Apparent Density (g/cm^3)
Al30WC	0	0.95
	2	0.99
	4	1.18
	8	1.49
Al30WC annealed at 200^{o}C	2	1.45
	4	1.38
	8	1.19
Al30WC annealed at 300^{o}C	2	1.39
	4	1.37
	8	1.36
Al30WC annealed at 400^{o}C	2	1.55
	4	1.55
	8	1.36

The apparent density is expected to be between 30% and 60% of theoretical density for spherical particles [21]. However, the results were obtained even below 60 % of theoretical density of Al30WC which was discussed as a result of particles in irregular shape and production of fine carbide particles. Besides, as shown from the Table 1, fluctuation in apparent density values was observed by increasing MA time before annealing. This was also thought to be result of small/fine particle existence due to high interparticle friction. However, apparent density of all annealed powders decreased by increasing MA time. Because powders formed by milling are hard, irregular, and exhibit poor flow and packing characteristics, so that annealing affected the density values by deagglomerating particles containing brittle fractions, significantly.

Figure 1 shows the XRD patterns of the 8h MA'ed powders. All the detected peaks are characteristic of the Al (ICDD No: 04-0787) and WC (ICDD No: 89-2727). Peak intensities decreased by time as presented in the figure.

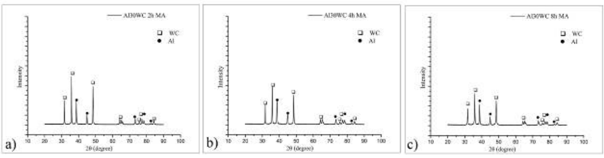

Figure 1. XRD patterns of 8h MA'ed powders

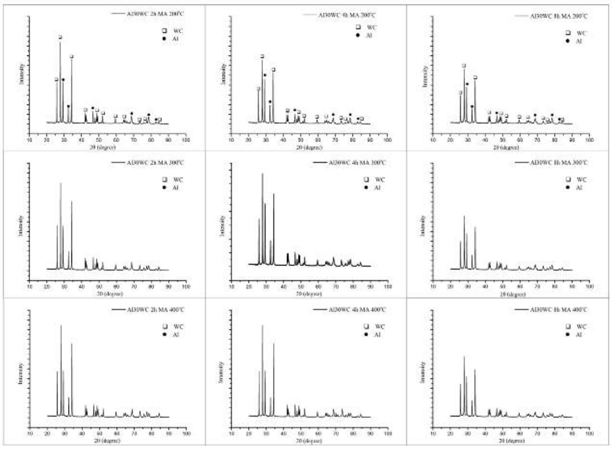

Figure 2. XRD patterns of MA'ed powders after annealing at different temperatures

Peaks belong to Al and WC powders can be seen clearly from the Figure 2 after annealing at various temperatures. Despite there is not any significant change in peak intensity and broadening observed, the peaks have become sharper and narrower by annealing temperature. Peak intensities increased for all powders after annealing. One can say that atomistic rearrangement and relieving of residual stress of lattice has caused this situation which is also good agreement with crystallite size and strain rates summarized in Table 2. Crystallite sizes increased and strain rates decreased after annealing for all powders as it was expected. It should be noted that high temperature of annealing (400^{o}C) was more effective on the removal of residual strains inside the powders.

Green densities of before and after annealed 8 h MA'ed powders are illustrated in Figure 3. As shown from the figure, green densities were found closer to theoretical density and increased by increasing MA time for annealed powders. On the other hand, as a result of high deformation rate, non-heat treated powders have a reverse situation. The green density of powders decreased by MA which is considered to be result of plastically deformed particles formed in irregular shape. In addition, one should be noted that high temperature was more effective on compaction comparing to lower ones. These results are also in good agreement with compressibility graphs of powders given in Figure 4. Sharp change in line (displacement-force graph) was observed for non-heat treated and annealed at 200^{o}C powders. However, the compaction seemed to be performed without any obstacle and initiated at the beginning for the powders annealed at 300^{o}C and 400^{o}C. This situation was considered as result of stress-relieving of particles that saturated for fracturing and became to the balance between fracturing and cold-welding.

Table 2. Crystallite size and strain rates (%) in Al peaks and before and after annealing against milling time.

Sample	MA time (h)	Annealing temperature (^{o}C)	Crystallite size (nm)	Strain (%)
Al30WC	2	-	89.1	0.3324
		200	142.7	0.1736
		300	144.5	0.1734
		400	199.6	0.1264
	4	-	79.4	0.4469
		200	139.9	0.1778
		300	133.2	0.1892
		400	138.8	0.1651
	8	-	85.0	1.2901
		200	156.1	0.1620
		300	154.1	0.1628
		400	154.0	0.1563

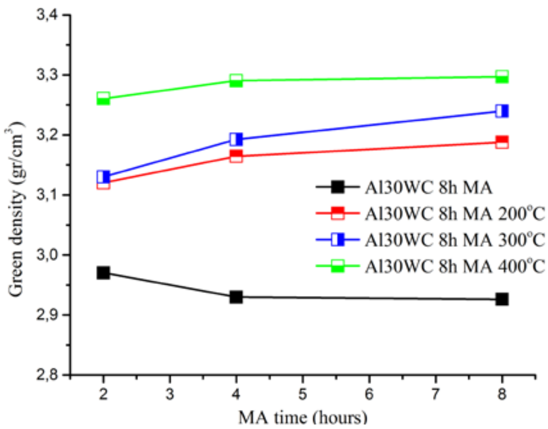

Figure 3. Green densities of 8h MA'ed powders before and after annealing at different temperatures.

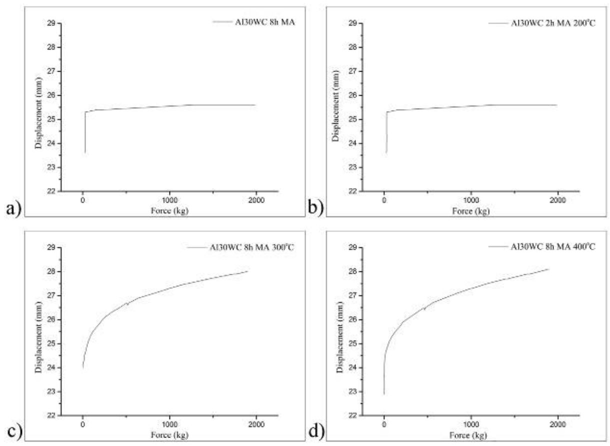

Figure 4. Compressibility of 8h MA'ed Al30WC powders **a)** only milled, **b)** annealed at 200^{o}C, **c)** annealed at 300^{o}C, and **d)** annealed at 400^{o}C.

Scanning electron microscope (SEM) back scatter images of non-heat treated and annealed 8h MA'ed powders were given in Figure 5. From the Figure 5a, the particles were seemed to be more agglomerated and the gaps between the particles were observed larger in distance comparing to annealed powders shown in Figure 5b. This was concluded as a consequence of reducing internal stresses and increasing ductility of metallic alloys (here Al). It was seen that the number of separate WC particles increased after annealing which affects the flowability of Al matrix. The reason to this phenomena was considered to be result of deagglomeration of brittle compounds after anneling.

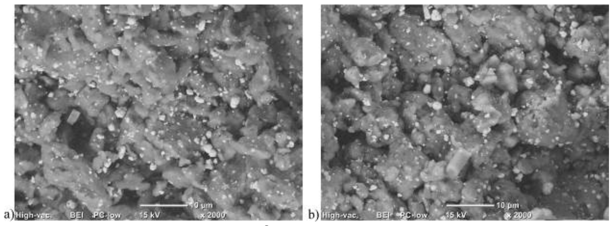

Figure 5. SEM images of 8h MA'ed **a)** non-heat treated and **b)** annealed at 400^oC powders.

4. Conclusions

In this study, the physical properties of mechanical alloyed Al reinforced with 30 % WC powders were investigated. According to results of the experiments these conclusions were obtained:

1. Higher green density values were obtained for annealed samples compared to non-heat treated counterparts. Annealing clearly improved the compressibility of the mechanical alloyed powders. Since the compressibility of powders increased by increasing annealing temperature an optimization will be necessary for different systems.
2. Contrary to compressibility, the strain value decreases after annealing, and the annealing at 400^oC was found more appropriate than the other lower temperatures studied.
3. It should be noted that with increasing annealing temperature, crystallite size of MA'ed powders increases simultaneously.

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