TENSILE PROPERTIES OF NANO Al_2O_3 PARTICULATE-REINFORCED ALUMINUM MATRIX COMPOSITES BY MECHANICAL ALLOYING AND HOT EXTRUSION

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The powder of the micro Al and variant volume fractions of nano Al_2O_3 were milled by a high energy planetary ball-mill. By milling, a homogenous distribution of nano Al_2O_3 particles in the metal matrix were developed. Then the milled powder was cold compressed and sintered at 545°C for one hr. The mold and the sintered sample hold in a furnace until the temperature reached 545°C . Then the hot 27mm diameter sample was extruded to 6mm diameter. From the extruded specimens, tensile, hardness and microstructure of the prepared specimens were determined. By these tests the effect of milling time, the percent of nano-particles and the microstructure were evaluated. The hardness and tensile behaviors of aluminum matrix composites reinforced with nano Al_2O_3 particulate have been found to increase remarkably with the volume fraction of the reinforcement.

Keywords: Nano-composite, planetary ball- milling, hot extrusion, tensile properties.

1. Introduction

In recent years, production and application of composite material especially, metal matrix composites (MMC), was developed considerably. Among the metal matrix composites, the Al matrix composites which was reinforced with ceramic particle, have been much attentioned.¹ Al matrix composites which have high strength and high elastic module are suitable substituting to produce low weight structures.² Reports show that strength of Al matrix composites will be improved about 20% with decreasing reinforcement particle size from micrometric scale to nanometric scale³. To produce metal matrix composites, the production method have an especially important role, because production process has a notable effect on mechanical properties and cost price.⁴ powder metallurgy method is the most common and most suitable production method of composite. Among production method of composites one of the most important advantages of this method compared with molten methods, is to be more low the process temperature, that avoids interaction

between matrix phase and reinforcement, and production of the uninvited phase. In other hands, the uniformity in the reinforcement distribution in matrix causes to improve and increase structural and mechanical properties.⁵ One of the most important factors which have effect on mechanical properties, is the condition of reinforcement phase, such as shape, size, and weight percent of the phase. The reinforcement due to its stiffness and high elastic modulus will be caused not only to reinforce the composite stiffness but also to change in matrix microstructure, and affects the composite properties. See Refs. 6–8 for more details. In this research, the effect of Alumina particles size on mechanical properties of composites, such as hardness, yield stress and change of length to fracture were studied. The effect of milling time on powder morphology and final structure was examined by Scanning electron microscope (SEM).

2. Experimental

The Aluminum powder with particle size 60 μ m, 99% purity, alumina powder with α crystal structure, and particle size 40nm and 3 μ m, and 99.5% purity and stearic acid, were used as a raw material. To consider the effect of alumina particle size on composite properties, 5Vol% Al₂O₃ nano powder with 95Vol% Al, and also 5Vol% Al₂O₃ micro powder with 95Vol% Al were mixed separately. To obtain the raw material, Al and Al₂O₃ powder were introduce together into a horizontal rotating cylindrical mixer operating at a rotation speed of 150 rpm with a mixing time of 1h. The purpose of this step which will be called "conventional mixing" throughout the paper is to mix powders without changing their original characteristics. Then, %1.5wt of stearic acid was added to blend as a process control agent, and mixing was continued for another 30 mine. Mechanical alloying was carried out in a high-energy planetary mill under argon atmosphere (>99.9999% purity). Milling was carried out with 250 rpm speed for times (5,10,15,20,25h). In all experiments 5 balls of 20mm diameter from stainless steel were used and the ball to powder ratio was 10:1. the milling process was stopped after certain times. And powder expelled in a compaction die under argon atmosphere. The blends were uniaxially cold pressed and compacted at 200MPa in a cylindrical die with 27mm diameter and 20mm length. The compacted pieces were sintered to 545°C, then were extruded in extrusion die by press with 0.1mm/s speed, and were obtained the extruded rods with 6mm diameter and 200mm length. Tensile properties of extruded samples were measured according to ASTM B557M-84E1 (4mm diameter and 32mm). Hardness of samples was evaluated by hardness test in Vickers scale by MH1 machine. The phase changes of powders was examined by analysis of x-ray diffraction by Philips PC-RD machine and The fracture surfaces and the microstructures of the milled Al-Al₂O₃ composite powders were examined by Leica Cambridge SEM.

3. Results

3.1. Powder morphology

The effect of mechanical milling on morphology of unreinforcement Al powder and reinforcement Al powder with 5Vol% Al₂O₃ particles was shown in Fig. 1. Initial Al particles were deformed after 25h milling, and changed to irregular shape (Fig. 1a). Micro-welding and fracture between particles are observed. It appears that need more time to obtain equiaxed particles. Fig. 1b shows the Al-5%μAl₂O₃ powder morphology after 25h milling. Decreasing progressive was observed in irregular particle aspect ratio. It is indicating that presence of reinforcement particles accelerates milling process. Fig. 1c shows morphology of Al-5Vol% nAl₂O₃ composite powder at 25h milling. It appears that plastic deformation, welding and fracture of Al matrix have more influence by addition of alumina nano particles to compare with alumina micro particles.

Particle size of unreinforcement Al powder is bigger than particle size of composite powder, and need more time to reach equiaxed state.

Fig. 1. Morphology of Al (a), Al-5vol%μAl₂O₃ (b), Al-5vol% nAl₂O₃ (c) after 25h milling.

Comparing unreinforcement Al powder, the affect micro scale and nano scale on milling processes have important.

3.2. XRD Analysis

The x-ray diffraction pattern of Al-5Vol% Al₂O₃ powder which milled for 20h and pure Al was shown in Fig. 2a. As it can be seen, the Al powder spectrum has high intensity peaks before milling. The intensity of peaks are decreased by addition alumina particles and increasing milling time, and their broadening are increased (Fig. 2b), which reflect decreasing of the particles size and increasing of lattice strain. We can also see, the peaks related to Al are slightly shifts to right side, that represent of impurity element of iron in powders which can caused this shift of the peaks. The analysis of the XRD peaks was performed via the Williamson-Hall method according to the following equation.⁹

$$\beta_s \cos \theta = \frac{K\lambda}{d} + 2\varepsilon \sin \theta. \quad (1)$$

Where β_s is the sample broadening in radians, 2θ is the position of peak maximum, K is Scherrer constant ($K=0.9$), λ is the x-ray wavelength ($\lambda_{Cu}=0.15406\text{nm}$), d is so-

called crystallite dimension, and ε is an approximate upper limit of the lattice distortion. The instrument broadening β_i was removed using the following equation according to Gaussian–Gaussian relationship by stress-free annealed aluminum powder.⁹

$$\beta_s^2 = \beta_e^2 + \beta_i^2 \quad (2)$$

where β_e is the full-width at half-maximum (FWHM) of the measured XRD peak. Table1 shows the crystallite size and lattice strain of Al–5Vol% Al_2O_3 for both nano and micro composite samples which milled for 20h.

Table 1. Crystallite size and lattice strain of milled powders for 20h

Powder	Crystallite size (nm)	Lattice strain (%)
Al-5% μ Al_2O_3	85.6	0.415
Al-5%n Al_2O_3	61	0.521

Fig. 2. XRD plots of Al and Al-5Vol% Al_2O_3 powders milled for 20h.

The effect of mechanical milling for 20h on the microstructure of nano and micro composite powders which are produced by hot extrusion method was shown in Fig. 3. According to previous experiments, particles at earlier times of milling have not an

agglomeration and uniformity distribution in matrix Al, and with increasing milling time to 20h, better distribution were obtained in particles. Fig. 3a shows uniformity distribution of Al₂O₃ micro-particles inside Al matrix which are about 1-3μm. Fig. 3b shows uniformity distribution of Al₂O₃ nano- particles which are about 40-150nm. It is found that Al₂O₃ nano-particles are smeared on surface of Al particles. Meanwhile, it was difficult to determine the difference between the nano alumina particles distribution owing to the limited resolution of SEM.

Fig. 3. SEM micrograph show the distribution of alumina particles in Al-5Vol% μAl₂O₃ (a) and Al-5Vol% nAl₂O₃ (b) powder milled for 20 h.

3.3. Mechanical properties

The mechanical properties of materials produced by powder hot extrusion were shown in Table.2. It appears that alumina particles increased the hardness of Al. To obtain hardness of extruded samples, micro hardness was performed. The main goal of this test was to found the role of both work hardening and alumina particles on micro hardness.

The tensile stress-strain curves of materials are shown in Fig. 4. The ultimate tensile strength (UTS) and elongation to failure (e%) are increased by addition of alumina particles. The increasing of strength in nano composite samples is more noticeable. In other words, the reinforcement nano particles play an effective role to increase the strength in fine Al matrix as compared to reinforcement micro particles.

Table 2. Mechanical properties of Al and Al–5% Al₂O₃ composite

Powder	HV	σ_{UTS} (MPa)	σ_Y (MPa)	σ_Y/σ_{UTS}	ϵ (%)
Al	38.8	116.84	71.57	0.61	23.64
Al-20h-milled	67.3	166.58	106.6	0.63	11.89
Al-5% μAl ₂ O ₃	83.8	210.74	152.38	0.72	4.34
Al-5% nAl ₂ O ₃	113.7	273.48	196.25	0.71	4.68

Fig. 4. Stress–strain curves of Al and Al-5Vol% Al₂O₃ composites.

3.4. Fractography

Fig. 5a shows the tensile fracture surfaces of pure Al and composite samples. The fracture surface of pure Al sample is cap-and-cone. The fracture surface in this sample has adhesion and micro holes and big dimples.

Fig. 5. SEM micrograph of the fracture surface of extruded samples pure Al powder (a), milled Al powder for 20h (b), Al-5Vol% μ Al₂O₃ (c), Al-5Vol% nAl₂O₃(d).

By milling of Al powder for 20h, rate of holes were decreased as compare with pure Al, which is due to fining Al powder particles during milling process. Fracture surface shows that these holes are formed because of separate powder particles from place of sample fracture. The fact that oxide layer formed on Al surface that is more thick by milling process, is exhibited from fine sintering, in result place of powder particles adhesion, are formed route of crack development (Fig. 5d). Fig. 5c shows the fracture surface of micro composite samples. In this state, population rate and fracture of alumina particles are not observed, and alumina particles were developed in boundaries, which is led to increase composite strength. And ductile fracture is little. Fig. 5d shows the fracture surface of nano composite sample which is milled for 20h. the presence fine diffraction in Al_2O_3 matrix interface suggests that relation of interface are good. In nano composite samples fracture is grain-boundary type, and grain-boundaries have important effect on fracture surface. "Recently, simulation and practical research show that fracture surface of nano material is grain-boundary necessity.

4. Discussion

The results of various experiments are shown that by addition of volume fraction of reinforcement particles and reducing their size, the possibility of dislocations interaction with particles are increased at deformation time. Also by reduction of Al_2O_3 particles size from $3\mu\text{m}$ to 40nm , milled particles fracture is occurred earlier which is led to increase the rate of work hardness of Al powder. Also the results of structural consideration by XRD analysis are shown that the average of grain size is decreased by milling is increased which it is one of the reasons to improve the mechanical properties. Also it is shown that speed of grains fining in presence of nano-metric particles is more. the dislocations interaction and nano-metric particles are led to form new dislocation, therefore it is caused to increase dislocation density in reinforced Al structure with nano-metric Al_2O_3 particles. Thus, the nanostructures formation mechanism in nano composite powders is occurred rapidly. According to the stress-strain curve, the sample including 5% of volume of nano alumina particles have higher properties than other three samples. By addition of alumina particles the flexibility is decreased and this reduction of flexibility in micro composite is more as compared with nano-composite, because the micro particles are bigger than nano particles. The dislocation population at behind of micro particles is more and the fracture of micro composite is occurred earlier than nano composite. the samples including reinforcement ceramic particle show higher strength as compared with pure Al. this strength increasing can be justified in this manner that formation of strain in interface particle with matrix is caused to produce more dislocations in composite and these particles are created obstruction on dislocations and are increased the rate of work hardness which it is led to tensile strength addition.

The less strength in these samples resulted reinforcement big particles, undesired diffraction, weaker adhesion of these particle to matrix, and possibility of presence of few porosity in them¹³.

On the other hand, the obtained results of hardness test are shown that hardness of reinforced aluminum with nano-particles is more than pure aluminum of %65.9. Also,

the hardness of nano-composite was increased as compared to reinforced Al with micro particles produced at the same time of %21.06 and milled pure Al at %36.9.

5. Conclusion

1. The reinforced composite powders with Al_2O_3 nano particles are reached to steady state and equiaxed morphology, as compared with micro-particles.
2. By performance of extrusion, it can be obtained to better microstructure properties, such as better distribution of reinforcement particles in matrix and fining them.
3. Mechanical alloying is led to improve the nano composite tensile properties due to it's high capability in homogeneous distribution of reinforcements in matrix and fining of grain size because of consecutive impacts.
4. The hardness of reinforced composite samples with nano particles is more than micro particles due to smaller grains size and effective role on inhibition from dislocations movement.
5. The stress-strain curves of composites show that the role of nano particles to improve the mechanical properties is more as compared with micro particles, and the flexibility of tensile sample in micro composite is less than nano composite.

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