



Studying on the fatigue behavior of Al- Al₂O₃ metal matrix nano composites processed through powder metallurgy

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ABSTRACT

Excellent mechanical properties and fatigue performance of Al/Al₂O₃ metal-based nanocomposites caused to introduce this material as a good candidate for various applications. In this regard, the preparation and characterization of this composite can be considered as a hot issue for research. The study was carried out in several steps including: (i) preparation of Al/Al₂O₃ metal-based nanocomposites at various Al₂O₃ content as reinforcement (4, 6 and 8 wt.%) using wet attrition milling; (ii) hot forward extrusion process of prepared samples; (iii) determination of mechanical properties of prepared composite by tensile test; (iv) usage of rotating-bending fatigue test for determination of the fatigue performance of prepared composite and (v) analysis of the fracture surfaces of fatigue tests specimens to determine the mechanism/s of failure based on scanning electron microscope analysis. The results showed that the presence of Al₂O₃ nanoparticles up to 6 wt.% enhanced the fatigue strength of Al/Al₂O₃ nanocomposites while the higher amount of reinforcement has a detrimental effect on the fatigue strength. Also, the statistical nature of fatigue data confirmed the higher coherency as well as homogeneity of prepared composites by 6 wt.% of reinforcement. The EDX spectrum confirmed the presence of Al₂O₃ at the origin of crack. As a consequence, the most probable mechanisms for crack initiation through the cyclic loading can be considered as the fracture and/or detachment of reinforcement particles.

Keywords: Aluminum; Nanocomposite; Powder metallurgy; Extrusion; Fatigue strength

1. Introduction

Unique characteristics of metal matrix composites combined with simple production process proposed these materials as good candidates for production at industrial scale. According to the literature [1-10], the type, size, shape, distribution, volume fraction and coherency of reinforcement to the matrix have administrated effects on the fatigue performance of prepared composites. Aluminum matrix nano-composites (Al MMNCs) are a wide category of engineering material that uses Al₂O₃

nanoparticles (with the size lower than 100 nm) as reinforcement and considering as a hot issue for many researches [11-15].

Fatigue failure during the cyclic loading in many engineering instruments is inevitable. In this regards, investigation of fatigue behavior of Al MMNCs during the dynamic loading plays a key role in design, material selection, forecasting the life of components and its durability [16-20].

It is worth to note that, the mechanical properties (using tensile test) and tribological characteristics of

Al MMNCs were the subjects of various researches [21-25] while, in spite of the importance of fatigue behavior of such components, little investigations were carried out in these categories [26]. There are various techniques for the production of metal matrix nanocomposites (MMNCs) including solid-state processes (e.g., high energy ball mill and powder metallurgy techniques) [27]; liquid-state (e.g., casting processes, stir casting, ultrasonic-assisted casting) [28]; semi-solid processing (e.g., a combination of rheo-casting, squeeze casting and semi-solid route stir casting) [29]. Outstanding specifications of hot extrusion including excellent preferential axial alignment of discontinuous fibers and large compressive hydrostatic state of stress [30-33] caused to evolve this technique as the most promising approach for the preparation of MMNCs [34-36]. Senthilkumar et al. [37] fabricated Al/Al₂O₃ composites using hot extrusion process. They used nano and micron size of Al₂O₃ particles as reinforcement. Analysis of low cycle fatigue data of prepared samples revealed that the usage of reinforcement in nano scale showed better fatigue performance respect to the samples that used Al₂O₃ with micron size. The main contributions of the current study can be considered as the investigation of the effect of the presence of various amounts of reinforcement, i.e., Al₂O₃ nanoparticle, on the fatigue behavior of prepared Al/Al₂O₃ nanocomposites and illustration of a comprehensive study on the fatigue behavior (low cycle fatigue and high cycle fatigue) of prepared composites.

2. Materials and Methods

Air atomized commercial pure aluminum powder (powder size of 45 μm) and α-Al₂O₃ powder (the average powder size of 35 nm, Nanostructured & Amorphous Materials, Inc.) were used as raw materials. The samples were prepared using the powder metallurgy technique. Before starting the milling process, the agglomeration of reinforcement, viz. Al₂O₃ nanoparticles, was inhibited by utilization of ultrasonic vibration in ethanol slurry for 60 min.

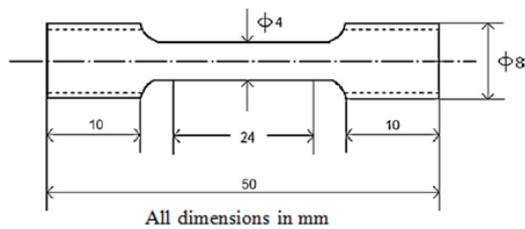


Fig. 1- Schematically representation of tensile test specimens according to ASTM E8.

As the next step, the prepared Al₂O₃ solution was added to the Al powder and milled for 8 hr by attrition ball mill. This study used hardened steel balls with a diameter of 5 mm, the ball to powder ratio of 15/1 and rotational speed of 480 rpm. Various samples including pure aluminum and nanocomposites with 4, 6 and 8 wt.% of Al₂O₃ nanoparticles were prepared. The consolidation process was carried out as follows: (1) drying of prepared samples for 90 min at 150 °C; (2) production of billets using uniaxial single action compaction at the pressure of 140 MPa; (3) final densification process to provide a dense rod with the diameter of 10 mm by employment of hot forward extrusion technique at 600 °C with reduction ratio equal 2.7/1; (4) the surfaces of prepared samples were polished by sandpaper # 600, 1200 and 3000, respectively to remove all surface defects including scratches and key defects. Scanning electron microscope (SEM, Leica Cambridge S360) was employed to investigate the fracture surfaces of prepared samples. The theoretical densities of prepared composites were calculated by mixture rule. The mechanical properties were determined by uniaxial tensile test and micro-hardness test. The load and dwell time of microhardness were supposed as 25 g and 15 sec, respectively. It was worth to note that, the uniaxial tensile tests were done parallel to the extrusion direction according to ASTM E8 using a gauge length of 20 mm. Fig. 1 schematically showed the geometry of the tensile test. Instron 8802 testing machine with a crosshead speed of 0.05 cm/min was used at the room temperature. The fracture surfaces of specimens were investigated by SEM. The relative density of prepared samples determined using Archimedes water immersion method. The fatigue behavior of prepared samples was investigated by the rotating-bending fatigue test based on ASTM E466. Fig. 2 illustrates the geometry of the fatigue test sample. The rotating fatigue machine (HSM19D, P.A. Hilton Ltd) with high-speed testing 6000 rpm was used at room temperature.

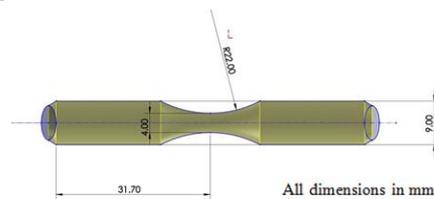


Fig. 2- Schematically representation of fatigue test specimens on the base of ASTM E466.

3. Results and discussion

The relative density of consolidated samples as a function of wt.% of reinforcement was shown in Fig. 3. Accordingly, the relative density showed an increasing trend where the Al₂O₃ wt.% changed from zero to 6 wt.%, while this trend was inverted at following up to 8 wt.%. It was worth to note that, the metallic bonding of Al as matrix has higher tendency for cold work respect to the combination of ionic-covalent bonding of Al₂O₃ as reinforcement. The lower content of Al₂O₃ (up to 6 wt.% Al₂O₃) enhanced the possibility of filling pores of prepared composites. As a consequence, the relative density in these composites enhanced. Since, the agglomeration of nanoparticles is an effective physical process for the reduction of its surface energy, it seems that in the case of 8 wt.% Al₂O₃, agglomeration of reinforcement was intensified and decreased the relative density of prepared samples. Also, the lower range of error bare of the prepared sample with 6 wt.% Al₂O₃ respect to the other samples can be considered as an evidence of higher uniformity of this sample. Fig. 4 shows the micro-hardness of various prepared composites. Accordingly, the presence of Al₂O₃ (up to 8 wt.%) dramatically enhanced the micro-hardness of prepared samples [38-40]. It seems that Al₂O₃ nanoparticles decrease the dislocation motion in prepared samples. Comparison of yield strengths and ultimate tensile strengths in Fig. 4 revealed that the presence of reinforcement effectively enhanced the strength of prepared composites. According to the Orowan bowing mechanism, the tensile strength of metal matrix composites strongly dependent on the interaction between the dislocation and reinforcement particles. Eqs. 1 and 2 showed the correlation between the strength and dislocation

characteristics. In which, G is shear modulus, L, b, d, and V are the interparticle spacing, Burger's vector, grain diameter of the reinforcements and volume fraction of particles, respectively. Since, the matrix and reinforcement of composites have various thermal expansions, enhanced the density of dislocation in the structure. If ρ, ΔC, ΔT defined as dislocation density, thermal mismatch, and temperature changes, respectively, then dislocation density can be calculated using Eq. 3. The strength of prepared composites can be calculated using Eq. 4 in which G and b are the shear modulus of composite and Burgers vector, respectively. Also, α is a constant between 0.5 and 1.

$$\Delta\sigma = 2Gb/L \tag{eq. 1}$$

$$L = 0.6d\left(\frac{2\pi}{V}\right)^{0.5} \tag{eq. 2}$$

$$\rho = 12\Delta T\Delta CV/bd \tag{eq. 3}$$

$$\sigma_d = \alpha Gb\rho^{\frac{1}{2}} \tag{eq. 4}$$

By consideration of the thermal mismatch among the structural component of MMNCs and Orowan bowing mechanism can be concluded that lowering the particle size induced higher strength to the prepared composites. It was necessary to note that, due to the lower particle size of reinforcement, the occurrence of the Orowan bowing mechanism was more probable. On the other hand, large enough variation between the processing and test temperature-induced considerably thermal expansion coefficient mismatch in the prepared composites.

Figure 5 shows the S-N curves of product samples. As can be seen, there is not any clear fatigue limit in all prepared composites. Accordingly, the fatigue strength of prepared samples in 10⁷ cycles

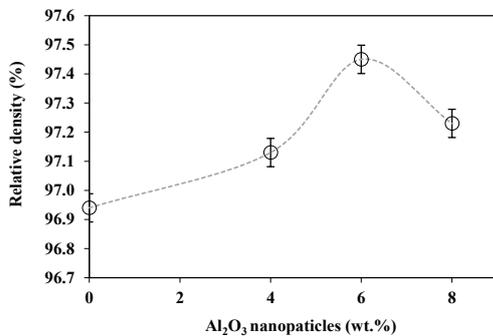


Fig. 3- Relative density of various Al/Al₂O₃ prepared nanocomposites.

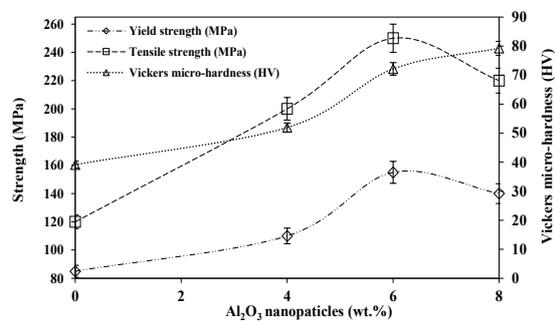


Fig. 4- The changes of strength and Vickers micro-hardness of Al/Al₂O₃ prepared nanocomposites as a function of wt.% of Al₂O₃ as reinforcement.

considered as fatigue strength. The absence of institutional atoms including C, H, N, and O is the main reason for the absence of a sharp fatigue limit. Similar behaviors have been reported in Al alloys [41, 42]. Boxplot used to illustrate the distribution of S-N data for low cycle fatigue (LCF) and high cycle fatigue (HCF) as shown in Fig. 5. As a general trend, at the constant stress levels, all samples showed the wider distribution in HCF respect to LCF. This behavior can be related to the higher effect of microstructure on fatigue behavior in the HCF regime. In these regards, the wider distribution of fatigue data in HCF of Al- 8 wt.% Al₂O₃ can be related to the higher possibility of agglomeration in

this sample respect to the Al- 6 wt.% Al₂O₃. Also, the average number of cycles to the fracture of Al- 6 wt.% Al₂O₃ was nearly the same as the median and confirmed the uniformity of these samples. It was worth to note that, similar to Al- 8 wt.% Al₂O₃, the median of pure Al and Al- 4 wt.% Al₂O₃ were significantly different from the average and can be considered as inhomogeneity of these samples.

Table 1 abbreviated various mechanical properties of prepared composites obtained from tensile and fatigue tests. Accordingly, the presence of Al₂O₃ up to 6 wt.% enhanced the strength and ductility of prepared samples, effectively. Also, the comparison of fatigue strength in 10⁷ cycles showed

Table 1- Mechanical properties of prepared composites

| Sample | Yield strength (MPa) | Tensile strength (MPa) | %Elongation | σ _a (MPa) | Fatigue strength at 10 ⁷ cycles (MPa) |
|-------------------------------------------|----------------------|------------------------|-------------|----------------------|--------------------------------------------------|
| Al- 0 wt.% Al ₂ O ₃ | 85 | 120 | 35 | 80 | 32 |
| Al- 4 wt.% Al ₂ O ₃ | 110 | 200 | 31 | 130 | 48 |
| Al-6 wt.% Al ₂ O ₃ | 155 | 250 | 28 | 165 | 72 |
| Al-8 wt.% Al ₂ O ₃ | 140 | 220 | 25 | 145 | 67 |

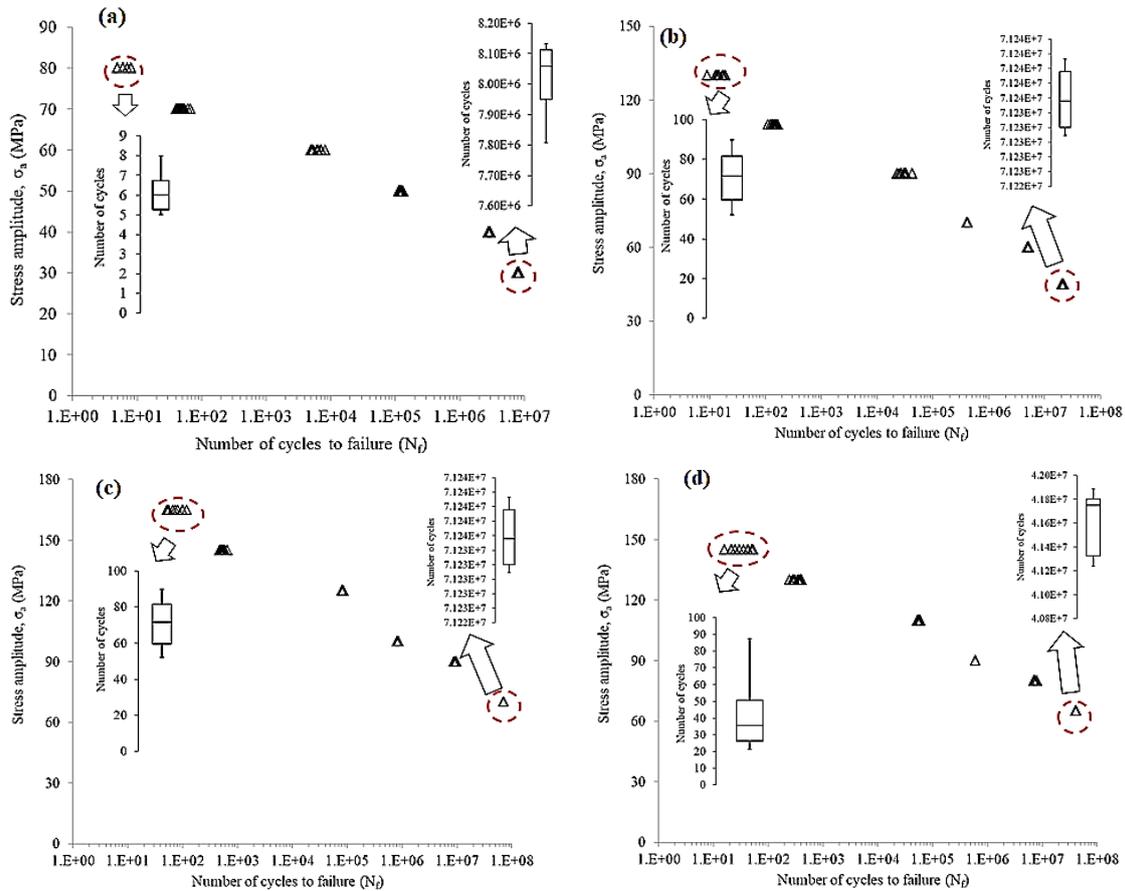


Fig. 5- S-N curves for (a) Al, (b) Al- 4 wt.% Al₂O₃, (c) Al- 6 wt.% Al₂O₃ and (d) Al- 8 wt.% Al₂O₃.

that using 6 wt. % Al_2O_3 as reinforcement can be enhanced the fatigue strength to be about 125% respect to the pure sample. Since, all engineering materials inherently have the micro-cracks, the main part of fatigue life related to the crack propagation respect to the crack initiation. There are three different mechanisms for the control of fracture in metal matrix composites including (i) nucleation of voids and growth, (ii) fracture of reinforcement particles and (iii) interfacial decohesion.

As shown in Fig. 6, all of fatigue cracks initiated from the surface defects and extended to the core of sample. Also, higher values of reinforcement enhanced the possibility of agglomeration as well as the creation of a continuous Al_2O_3 brittle phase through the grain boundary.

According to the literature [43-50], there are four major sources for crack initiation as follows:

i) Through the first period of cyclic loading, due to the intrinsic brittleness of reinforcement particles, micro cracks were initiated due to the fracture of reinforcement particles. The high rate of strain concentration as a consequence of incoherency between the surrounding matrix and reinforcement particles intensified this behavior;

ii) The detachment of reinforcement from matrix especially at the interface between the reinforcement and matrix can be considered as the second possible mechanism for the crack initiation through the cyclic loading. In this case, the cluster of particles can act as single large particles that effectively prevented from the plastic flow through the loading and enhanced the possibility of crack initiation;

iii) Fracture of large foreign particles, e.g. inclusion and/or intermetallic, rather than reinforcement, are the other responsible for the crack initiation. From one hand, the large size of these phases (about 20 to 100 μm) and from the other hand their brittle nature enhanced the possibility of crack initiation in this mechanism;

iv) Crack initiation can be formed due to the aggregation of cavities and porosities through prolonged cyclic loading. These defects were formed typically from incomplete penetration of the matrix material into the particle clusters.

As shown in Fig. 7, the size of dimples that formed in fractured surfaces decreased by the amount of reinforcement. Fig. 7(d), confirmed the formation of micro-cracks in Al- 8 wt.% Al_2O_3 samples. Consequently, due to the presence of Al_2O_3

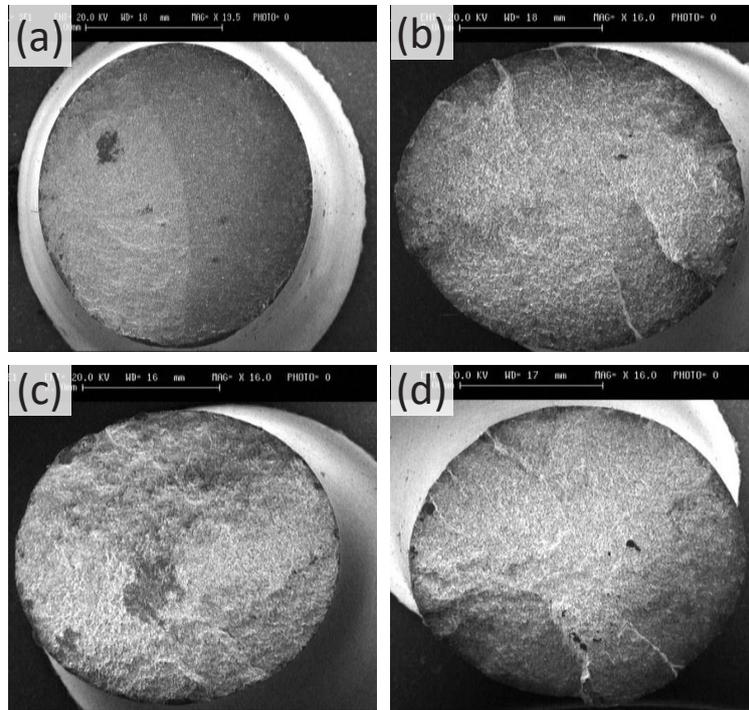


Fig. 6- Fracture surface of the specimens: (a) Al, (b) Al- 4 wt.% Al_2O_3 , (c) Al- 6 wt.% Al_2O_3 and (d) Al- 8 wt.% Al_2O_3 .

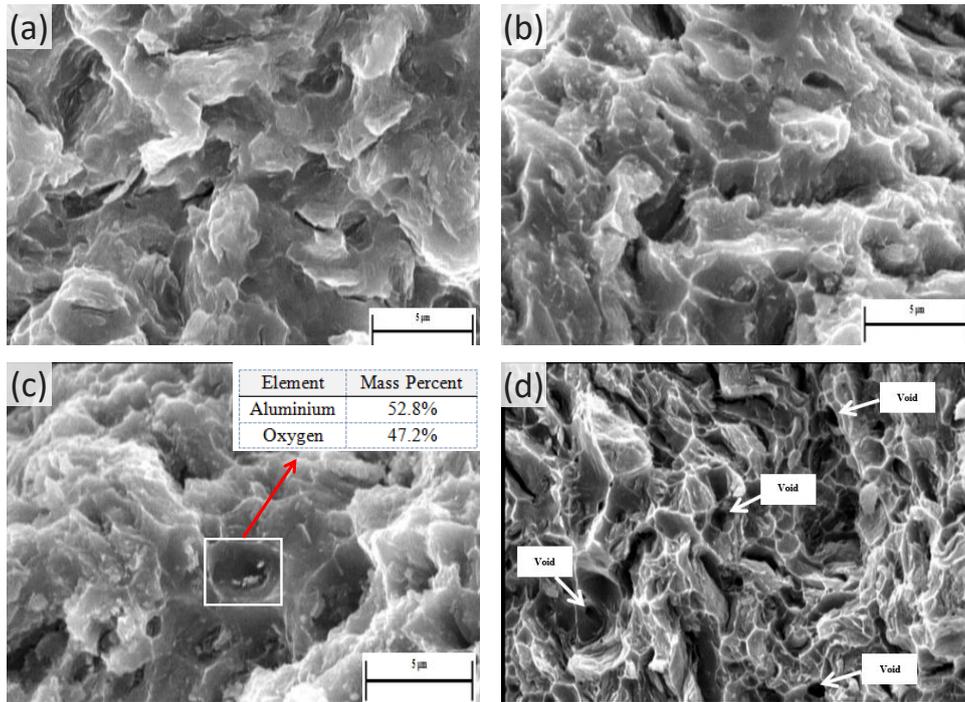


Fig. 7- Fracture surface of the specimens: (a) Al, (b) Al- 4 wt.% Al_2O_3 , (c) Al- 6 wt.% Al_2O_3 and (d) Al- 8 wt. % Al_2O_3 .

phase in the formed micro-cracks that confirmed by EDX spectrum confirmed that the occurrence of the fracture or detachment of reinforcement particles were the most administrated mechanism for the crack initiation through the cyclic loading.

4. Conclusion

Al matrix composites reinforced by Al_2O_3 fabricated using wet attrition milling followed by hot forward extrusion process. The results confirmed that, increasing the amount of nano-sized particulates, induced higher relative density at first (until to 6 wt.% of Al_2O_3) and then decreased the relative density for samples prepared at 8 wt.% of Al_2O_3 . Moreover, the microhardness of composites by increasing the amount of reinforcement enhanced. Similar trend especially observed for the strength of prepared samples until 6 wt.%. The results showed the possibility of the employment of the Orowan bowing mechanism as well as the thermal mismatch between the matrix and reinforcement for the explanation of the behavior of prepared composites. In summary, the presence of higher Al_2O_3 nanoparticles up to 6 wt.%, enhanced the fatigue life of prepared composites. While the higher amount of nanoparticles to 8

wt.% showed adverse behavior. This trend can be related to the agglomeration of Al_2O_3 nanoparticles as well as the generation of continuous brittle phases within the grain boundaries. The results confirmed the higher size of dimples that formed at the surface of fractured specimens with a higher amount of reinforcement. Accordingly, the Al- 6 wt.% Al_2O_3 proposed as appropriate composite for the construction of structure used in cyclic loading.

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