Microstructure and Tensile Properties of an Ultrafine Structured Al-5vol. %Al_2O_3 Nanocomposite Produced by Using a Powder Metallurgy Processes

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Abstract

Ultrafine grained Al-5vol%Al_2O_3 metal matrix composite powders were produced from a mixture of Al and nano Al_2O_3 powders using high energy mechanical milling (HEMM). The composite powders produced were first hot pressed at 300°C with a pressure of 240 MPa to produce cylindrical powder compacts for the forging part and composites powder were cold compacted under the 1 GPa pressure to produce cylindrical compacts for the extrusion one. Severe plastic deformation process was utilized to consolidate the powder compacts into nearly fully dense forged disks and extruded bars. With the same volume fraction of Al_2O_3 the average microhardness of the forged disks was 117HV and 133HV for the extruded bars. The tensile strength of the forged disks increased to 362 MPa and 371 MPa for the extruded composite. Al-5vol%Al_2O_3 forged disk showed a macroscopic plastic yielding, while the Al-5vol%Al_2O_3 extruded bars did show a macroscopic plastic yielding with a small plastic strain to fracture (<1%).

Key words: *Metal matrix composite, Nanostructure, High energy mechanical milling, Forging, Extrusion, Tensile strength*

1. Introduction

Al based metal matrix nanocomposites (MMNC`s) are an important group of materials due to their lightness and the potential of offering good ductility, good fracture toughness, high formability and high strength [1-6]. By using a dispersion of ceramic nanoparticles(diameter <100 nm) instead of micrometer sized particles, high fracture toughness offered by the ductile aluminium matrix can be maintained since the ceramic nanoparticles are very difficult to be fractured through stress concentration on the ceramic particles due to their small sizes and lack of flaws[7-10] . By refining the microstructure of the matrix to nanostructured level or ultrafine structured level, the strength of the matrix can also be significantly increased.

There has been a substantial amount of research which has been done on fabrication, microstructure and mechanical properties of Al based MMNC`s produced by powder metallurgy processes which combines high energy mechanical milling and powder consolidation [1, 4, 5, 8, 11-13]. It has been shown that a uniform distribution of Al_2O_3 or other ceramic reinforcement nanoparticles and significant grain refinement of the Al matrix can be successfully obtained in the microstructure of the bulk nanocomposite samples [1, 4, 8, 11]. Due to the fine grains and uniform distribution of the ceramic nano particles within the Al matrix, the mechanical properties, the yield strength and ultimate tensile strength were increased from 130 and 200 to 260 and 370 MPa and the plastic strain to fraction was in the range of 2-5%. For powder consolidation, powder compact extrusion and powder compact forging has been used [2, 8, 14, 15]. It is worthwhile to mention that powder compact forging and powder compact extrusion of Al based MMCs has been studied by many researchers. For instance, Ogel et al.[15] Produced an Al-Cu-SiC metal matrix composites by using a conventional hot pressing and they found that the yield strength and tensile strength of the material were improved while the ductility reduced with increasing the amount of SiC particles. Monazzah et al.[16] milled atomized Al powder in a planetary mill . The produced powder was de-gassed and extruded at extrusion ratio of 16:1.the creep behaviour of the extruded billet in the direction of extrusion was examined at constant applied load. Balog et al.[17] formed 25 samples of extruded Al–Al_2O_3 composites by using in situ. It was found that the properties of the compacts stemmed from the extraordinary grain boundary strengthening effect of the ultrafine-grained compacts due to their microstructures and was enhanced by the presence of the nano-metric Al_2O_3.

Although a substantial amount of research work on nanostructured and ultrafine structured Al based MMNCs produced by powder metallurgy processes has been done and published in open literature, the mechanical properties (especially those under tension) of the samples produced are not as good as expected. It is believed that this is mainly due to the lack of good quality consolidated samples. In this study, we aim to produce high quality ultrafine structured Al based MMNCs samples with a homogenous dispersion of 5vol. % γ- Al_2O_3 nanoparticles by a combination of high energy mechanical milling and rapid powder consolidation using powder compact forging and powder compact extrusion. This paper is to report the microstructure and tensile properties of the bulk ultrafine structured Al-5vol. % Al_2O_3 nanocomposite samples produced in this study and compare them with those of other Al based MMNCs reported in the literature.

2. Experimental procedure

The starting powders used for producing ultrafined structured Al-5vol%Al_2O_3 nanocomposites using high energy mechanical milling (HEMM) were an aluminium powder (99.5%pure; average particle size of 40 µm) and gamma alumina nanopowder (99%pure; average particle size ~50nm). The milling was done under high purity argon using a hardened steel vial with a cylindrical cavity of 60 mm in depth and a 100 mm in diameter, stainless steel balls with a diameter of 12 mm, and a PM 4000 Restch planetary ball mill. Before milling, 1wt% stearic acid was added to the powder charge in the vial as a process control agent. The ball to powder weight ratio was 5:1. The starting powders were first mixed for 6 hours at a rotation speed of 100 rpm, and then milled for a net total time of 12 hours with a rotation speed of 400 rpm. The ultrafine structured Al-5vol%Al_2O_3 nanocomposite powder produced by milling was consolidated by using powder compacts forging and powder compact extrusion respectively. For powder consolidation using powder compact forging(PCF), the powder was first compacted by using uniaxial hot pressing at 300 ºC for 15 minutes under a pressure of 240 MPa using a cylindrical H13 steel die(internal diameter: 25mm) . The powder compacts were heated to 450 ºC using induction heating under argon atmosphere, and then forged using an open die kept at room temperature , a 100-ton hydraulic press with a ram travelling speed of 7.7 mm/s. Circular disks were produced from the powder compact forging experiments. For powder consolidation using powder compact extrusion (PCE), the powder was compacted by using uniaxial cold pressing at room temperature for 5 minutes under a pressure of 1000 MPa using the same die as mentioned above. The powder compacts were then heated to 500 ºC using induction heating under argon atmosphere, and subsequently extruded using an extrusion die set kept at 450 ºC and the same 100-ton hydraulic press. The extrusion ratio was 7:1. Cylindrical bars were produced from the powder compact extrusion experiments. Flat dog-bone shaped tensile testing specimens with a gauge length of 9.5 mm for the forging samples and 20 mm for the extrusion samples, were cut from the powder forged disks and extruded bars respectively using an electrical discharge machining (EDM) wire cutter. The density of the consolidated samples was determined using the Archimedes method. Tensile testing of the specimens was done at room temperature using an Instron 4204 testing machine with a strain rate of $1.8x10^{-4}$/s. Scanning electron microscopy (SEM)(Hitachi S4700), transmission electron microscopy (TEM) (FEI CM200) and X-ray diffractometry (XRD)(Philip X-pert) were used to characterize the milled powders, the consolidated bulk samples and the fractured specimens. The Vickers microhardness of

the milled powder particles, forged disks and extruded bars was measured using Vickers microhardness tester (LECO LM700) with a load of 25 g and a loading duration of 15 s.

3. Results

3.1 Microstructure

The XRD patterns of the milled Al-5vol%Al_2O_3 nanocomposite powder and the bulk samples produced by powder compact forging and powder compact extrusion respectively are shown in Fig.1. The XRD patterns only show the diffraction peaks of Al matrix which are significantly broadened. The average grain size of the Al matrix was determined using the Williamson-Hall method. Fig. 2 shows $\beta^*\text{Cos}\ \theta/\lambda$ vs $\text{Sin}\ \theta/\lambda$ data points corresponding to the peaks in the XRD patterns of the milled powder and bulk samples produced by powder compact forging and powder compact extrusion respectively and the best fit lines. The values of R^2 for the curve fitting were in the range 88-95% indicating that the quality of the curve fitting was high. From the slopes and the intercepts of the best-fit lines, it was determined that the grain sizes were 167 nm, 500 nm, 111 nm, while the lattice strain were 0.28%,0.46% and 0.15% for Al-5vol%Al_2O_3 powder, and bulk samples produced by powder compact forging and powder compmact extrudion respectively.

The microstructures of the milled Al-5vol.%Al_2O_3 nanocomposite powder particles and the bulk consolidated samples were examined using TEM, and Figs.3 show the typical TEM bright field and corresponding dark field images and corresponding selected area electron diffraction patterns(SAPDs) obtained from the examination. As shown in Fig. 3, the grain sizes of the Al matrix in the as milled Al-5vol.%Al_2O_3 nanocpmosite powder particles were in the range of 50-300 nm, those in the bulk sample produced by powder compact forging were in the range of 100-500 nm, and those in the bulk sample produced by powder compact extrusion were in the range of 100-400 nm. The TEM bright field and dark field images also show that the Al matrix grains either contain a high density of dislocations or have a nanometer sized substracture.From the TEM images and SAPDs, it was clear that the Al_2O_3 nanoparticles were homogeneously distributed within the Al matrix, but the contrast showing the Al_2O_3 nanoparticles was not very clear due to the very small size of the Al_2O_3 nanoparticles and the heavy contrast of the Al matrix containing a high density of dislocations and substructures.

Fig. 5 shows the SEM micrographs of the samples obtained from the central region of forged disk and extruded bar taken. SEM examination showed that both were almost fully dense with the volume fraction of pores being <1%. In contrary the relative density was found to be 94% for the forging compacted samples and 99.5% for the extruded samples calculated by using the Archimedes method.

3.2 Mechanical Properties and Fracture Behaviour

The microhardness of the milled Al-5vol. % Al_2O_3 nanocomposite powders and the corresponding bulk samples produced by powder compact forging and powder compact extrusion respectively are listed in Table 1, and they were 0.89 GPa, 1.15 GPa and 1.3 GPa, respectively. The typical tensile engineering stress-strain curves of the specimens cut from the bulk Al-5vol. % Al_2O_3 nanocomposites samples produced by powder compact forging and powder compact extrusion of the mechanically milled powders compact are shown in Fig.6, and their tensile properties are listed in Table1. As shown in Table 1, the yield strength ($\sigma_{0.2}$), ultimate tensile strength, and plastic strain to fracture of the bulk samples produced by powder compact forging were 343.7 MPa, 362.2 MPa, and 8% ,respectively, and those of the bulk samples produced by powder compact extrusion were 318.2 MPa, 371.1 MP and 8%, respectively. The engineering stress-strain curves show strain softening.

To reveal more information about the nature of interparticle bonding in the consolidated bulk samples, the longitudinal surfaces of the tensile tested speciemns after fracture was also examined using SEM. As shown by the SEM images in Figs.7, just a few cavities(indicated by the arrows in Fig.7 formed near the fracture surface during tensile deformation and fracture of the specimens cut from the bulk consolidated samples produced by powder compcat forging and powder compact extrusion. The shape of the cavities do not confirm whether they were caused by separation of neighbouring powder particles due to weak interparticle bonding. In the mean time, the regions away from the fracture surfaces were ffee of any cavities, this indicates that the cavities near the fracture surfaces were formed by nucleation in the solid material, rather than due to separation of weak bonded particles.Fig. 8 show the fracture surfaces of the tensile tests specimens which indicate that the fracture of the specimens occurred through ductile fracture of the Al matrix (as reflected by the dimples).

4. Discussion

This study shows that with adding five volume fractions of Al_2O_3 nanoparticles in the starting Al/Al_2O_3 powder mixture, the sizes of the powder particles change, resulting in the

formation of nanograins and ultrafine microstructure as shown in Fig.3. With applying the thermomechanical processing; powder compact forging (PCF) and powder compact extrusion (PCE), the Al lattice parameter is increased as shown in Fig.2 and the lackness of clear particle shaped contrasts in the TEM image of the 12 hours milled Al and Al-5vol. %Al_2O_3 after PCF and PCE suggested that the Al_2O_3 nanoparticles have been dissolved within the Al ductile matrix. The XRD patterns for the milled powder, powder compact forging, and powder compact extrusion are shown in Fig.1. A very Weak diffraction peaks belonging to the Al_2O_3 phase can be detected, which is likely due to the relatively small fraction of Al_2O_3 nano particles in the composite powder, and the low level of crystallinity of the Al_2O_3 nanoparticles , showing that none or negligible grain growth of the reinforcing phase has occurred in the current consolidation conditions .The detection of only one phase in the pattern of the composite indicates no reaction between matrix and reinforcement to form additional phases took place during the consolidation. The results indicates of higher grain size refining of Al in the presence of nanometric Al_2O_3 particles especially after powder compact extrusion and reaeched 111 nm.

The theoretical densities of the Al-5vol%Al_2O_3 composites were calculated by using the rule of mixture, while the actual densities of the powder compacts were calculated by dividing their weights by corresponding volumes. It was found that the relative densities for both samples produced by powder compact forging and powder compact extrusion were in the range of 94-99.5%. This is likely because of the increased hardness of the powder particles caused by work hardening during milling and by the volume fraction of the hard Al_2O_3 nano particles in the composite structure. It was observed that the samples produced by powder compact extrusion (PCE) has a more finer microstructure than that produced by powder compact forging (PCF), average grain size estimation using the Williamson-Hall method and the TEM examinations in Fig.3.This is important to notice, because it shows that the composite after PCE process with higher operating temperature refined rather than coarsened. This grain refinement can be due to recrystallization of the heavily and plastically deformed the ultrafined grained Al matrix during heating to 500 ºC.

The fracture surface of the Al-5vol. %Al_2O_3 composites produced by powder compact forging exhibit dimples which indicate a ductile fracture. Fig.8.a micrograph reveals that fractured particles were surrounded by ductile regions described as tear ridges and voids. In the other hand, with the powder compact extrusion the fracture surface shows the brittle fracture which dominates the inter-particle regions as shown in Fig.8.b. The fracture surface

resulted from decohesion between the adjacent particles due to incomplete densification of the composite and resulted in lackness of ductility <1%.

The ultimate tensile strength of the tensile test samples after powder compact forging and powder compact extrusion was 362.2 and 371.1MPa, respectively. It's important to mention that the mechanical properties obtained in this work is slightly better to what was reported by Hesabi [8] ;where it was reported a yield stress of 263 MPa and an ultimate tensile stress of 356 for Al-5vol. % Al_2O_3 nanocomposite produced by extrusion. As it is expected, the strength increased with adding 5vol. % Al_2O_3 nano reinforcement and the composite became more brittle. That can be explained by the constrained plastic deformation of the aluminium metal matrix by the act of the nanoparticles [8, 9], grain boundary embrittlerment [11], and increased porosity and less formability [13].

5. Conclusions

Al-5vol. %Al_2O_3 nanocomposite powders have been produced using two severe plastic deformation processes; High energy mechanical milling (HEMM) followed by consolidation by a combination of hot pressing at 300ºC and powder compact forging at 450ºC and cold pressing at room temperature and extrusion at 500 ºC and extrusion rate of 7:1. The microstructure of the forged disks consists of fine Al_2O_3 particles homogenously distributed in an ultrafine grained Al matrix as shown in Fig.3. The average microhardness of the forged composite disks increased from 91 for Al milled to 133HV with adding five volume fraction of Al_2O_3. The tensile strength for the produced samples of Al composite was 362 MPa and 371.1 MPa for Al-5vol. %Al_2O_3 forging and extrusion process, respectively. The Al-5vol. %Al_2O_3 forged disk show macroscopic plastic yielding, while the Al-5vol. %Al_2O_3 extruded bars showed macroscopic plastic yielding with a small plastic strain to fracture (~1%).

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