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Strength and ductility enhancement in nanostructured Al6063 with a bimodal grain size distribution

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Abstract. A bimodal grain structure comprising of ultrafine-grained (UFG) and coarse-grained (CG) regions was produced in Al6063 by means of powder metallurgy route. Nanocrystalline Al alloy powder was synthesized by high energy mechanical milling using a planetary ball mill under Ar atmosphere for 22.5 h. The as-milled powder was mixed with 30 vol.% unmilled powder and consolidated by extrusion at 450 °C with an extrusion ratio of 9:1. UFG and CG billets were also fabricated at the same condition by using milled and unmilled Al6063 powders, respectively. The grain structure was studied by scanning electron microscopy (SEM) and electron back-scattered diffraction (EBSD) techniques. The deformation behavior under uniaxial tension was investigated. The bimodal Al6063 showed balanced mechanical properties, including enhanced yield and ultimate strength comparable UFG alloy with reasonable ductility analogous CG material. The fracture surfaces demonstrated a ductile fracture mode, in which the dimple size was related to the grain structure. The ductility enhancement mechanisms in nanostructured Al6063 with bimodal grain structure were discussed.

1. Introduction

Recent advancements in high energy ball milling techniques help to produce very fine grains in metallic microstructures and these materials are termed as nanocrystalline or ultrafine-grained (UFG) materials. These materials demonstrate a noticeable increase in strength compared to coarse-grained (CG) counterparts. Though the high strength is desirable, but the reduction in grain size severely limits dislocation motion, and thus plastic deformation and ductility. It was shown that UFG materials exhibit a limited uniform elongation, usually less than 5 % in tension, making them improper for forming operations or for use in load bearing equipments. The reduced uniform elongation was attributed to the low work hardening capability of UFG metals. According to the Hall-Petch relationship for polycrystalline material (grain size larger than 1 µm) [1], the yield strength increases with increasing the average grain size regardless to the grain size distribution. Nevertheless, in UFG materials, strength depends not only on average grain size but also on grain size distribution. Thus, grain size distribution can effectively used for controlling the mechanical response (strength and ductility) of UFG materials. Various methods to find an optimal level of strength and ductility in UFG materials have been employed. They range from heat treatment [2,3] and varying the manufacturing temperatures [4,5] to precisely-controlled fabrication processes developed to significantly reduce the number of voids, inclusions, and other imperfections [6]. The first two ways coarsen all the grains within the UFG material. The third can maintain the UFG size and thus the strength of the material, but is even more costly and harder to implement on a large scale. One of the promising methods
has been used in the last decade is tailoring the grain structure of UFG metals by introduction of soft inclusions of the same metal in the strong UFG matrix [7]. Bimodal microstructures are frequently produced by mixing mechanically-milled/cryomilled powders with an appropriate amount of un-milled powders followed by consolidation. A composite metal with a bimodal grain size distribution is achieved by this technique. Basically, the UFG region retains its strength while a high extent of deformation can occur in the soft CG regions. For instance, Witkin et al. [8] have prepared Al-7.5%Mg samples with a bimodal grain structure and have reported a 1.7% and 13.6% decrease in the yield strength, but 71% and 286% increase in the elongation in UFG alloy containing 15% and 30% of coarse grains, respectively. This method has been successfully used for fabrication of some Al alloys like Al 5083 [9] and Al-Mg [10-12]. However, to the best of our knowledge this method has not been used for production of Al-Mg-Si alloys. In this paper, we describe the fabrication of a UFG Al6063 with a bimodal grain structure engineered for high strength with enhanced ductility.

2. Experimental
A gas-atomized 6063 aluminum alloy powder was used as starting material. The chemical composition of the powder was determined as (in wt.%): Al–0.92 Mg–0.404 Si–0.329 Fe–0.01 Cu. The powder particles exhibit mainly spherical shape and tear-drop, as shown in Fig. 1. 

Mechanical milling was performed in a planetary ball mill. 1.5 wt.% Stearic acid (CH₃(CH₂)₁₆COOH, Merck) as the process control agent was added to the aluminum powder and mixed for 60 min with rotational speed of 100 rpm. High-chromium steel balls with diameters of 11 and 19 mm were used as milling media. The ball-to-powder weight ratio (BPR) was fixed at 10:1. Mechanical milling was performed under Ar atmosphere with rotational speed of 350 rpm for 22.5 h. 
A mixture of milled and 30 vol.% of unmilled powders were mixed and degassed under Ar flow in a tube furnace at 450°C for 60 min. The powders were filled in 30 mm diameter Al cans under Ar atmosphere in the glove box and then cold compacted to attain a relative density of 0.75. The cans were preheated at 450 °C and finally hot extruded to cylindrical billets 10 mm in diameter. The extrusion ratio of 9:1 was used. Similarly, two billets of 100% milled and 100% unmilled powders were consolidated for comparison purposes.
X-ray diffraction (XRD) analysis was performed by using a Philips X’pert MPD Diffractometer with Cu K\textsubscript{α} radiation source working at 40 kV and 40 mA. Microstructural observations were performed by optical microscope (OM) and field emission scanning electron microscope (FE-SEM, Tescan). For optical microstructure studies, the samples were prepared by common metallography procedure and were etched in Keller’s solution (2.5% HNO\textsubscript{3}, 1.5% HCl, 1% HF, 95% H\textsubscript{2}O). The grain structure was determined by a field emission variable pressure scanning electron microscope (FEVP-SEM, Hitachi, SU-6600) equipped with electron backscattered diffraction (EBSD). The specimens were mechanically ground, pre-polished using diamond pastes and finally polished by colloidal silica suspension. The data processing was carried out using OIM analysis software (version 5.2; EDAX).

Uniaxial tensile test was utilized to evaluate the mechanical behavior of consolidated powders. Flat tensile specimens with gauge length of 9 mm and thickness of 2 mm were machined using electro-discharge machining (EDM). Tensile test was performed according to standard ASTM E8. The fracture surfaces of the samples after tensile test were explored by SEM.

3. Results and discussion

Figure 2 shows an OM microstructure of Al6063 alloy extruded from a mixture of milled and 30 vol.% of unmilled powders along and perpendicular to the extrusion direction. UFG regions are seen dark and CG regions are viewed bright. This can be attributed to the higher reactivity of UFG regions to the etching reagent compared to CG regions as a result of higher fraction of grain boundaries in UFG areas. The coarse grains in the order of several micrometers are evenly distributed within the UFG matrix revealing the formation of a bimodal grain structure. The CG regions are elongated along the extrusion direction and formed narrow bands inside the UFG matrix.

![Fig. 2: Optical micrograph of a bimodal microstructure of Al6063: (a) along the extrusion direction; (b) perpendicular to the extrusion direction.](image)

Figure 3 depicts the inverse pole figure (IPF) and image quality (IQ) maps obtained from EBSD analysis of the bimodal grain structure Al6063 alloy. The dissimilar colors of the grains in the IPF-EBSD map reveal the large difference in orientation of neighbor grains (Fig. 3a). Moreover, a bimodal grain structure consisting of ultrafine grains and coarse grains is observed. The grain size distribution of the UFG regions varied in the range of 80 nm to 1.4 µm and the grain size of the CG regions changed between 1.8-4.1 µm. In the IQ map (Fig. 3b), blue lines represent the place of high-angle boundaries with misorientations greater than 15° and green lines the location of low-angle boundaries with lower than 15° misorientation. The IQ map demonstrates elongated coarse grains with several subgrains within them. Nevertheless, grains in UFG matrix
seem virtually equiaxed that are almost surrounded by high angle boundaries. This can be attributed to the small crystallite size of the milled powders so that their deformation along the extrusion direction is dispensable.

**Fig. 3:** Electron back scatter diffraction maps of Al6063 alloy: (a) inverse pole figure; (b) image quality.

The tensile stress-strain curves for the bimodal and unimodal (CG and UFG) Al6063 alloys are shown in Fig. 4. Detailed tensile properties of the samples are given in Table 1. The CG sample had very little strength and failed noticeably in a ductile manner with slant surfaces aligned ~45° relative to the tensile axis. Thus, a significant necking and plastic strain to fracture of 15% occurred. On the other hand, the UFG material showed a high yield and tensile strength (214 MPa and 291 MPa, respectively) with a limited strain to fracture (6%). Little to no necking occurred and the fracture path was jaggedly perpendicular to the loading direction. The tensile ductility was enhanced in the bimodal sample with a strain to fracture of about 9%. However, the bimodal Al6063 showed a lower strength compared to UFG sample (see Table 1). Fracture surface of the bimodal sample showed a mixed fracture mode with large shear lips and flat
central region. Consequently, the bimodal Al6063 alloy demonstrated an attractive balance of strength and ductility as compared to its unimodal counterparts.

**Fig. 4:** Tensile stress-strain curves for the as-extruded bimodal alloy and unimodal CG and UFG samples.

<p>| Table 1. Mechanical properties of bimodal and unimodal (CG and UFG) Al6063 samples. |
|--------------------------------------------------|------------------|------------------|------------------|</p>
<table>
<thead>
<tr>
<th>Yield strength (MPa)</th>
<th>Ultimate tensile strength (MPa)</th>
<th>Strain to fracture (%)</th>
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<tbody>
<tr>
<td>UFG</td>
<td>214</td>
<td>291</td>
</tr>
<tr>
<td>Bimodal (UFG+30 vol. % CG)</td>
<td>188</td>
<td>266</td>
</tr>
<tr>
<td>CG</td>
<td>93</td>
<td>150</td>
</tr>
</tbody>
</table>

Tensile fracture surfaces of the specimens at a high magnification are shown in Fig. 5. Ductile fracture with dimple-like morphology was observed in the CG Al6063 (Fig. 5a). On the fracture surface of UFG sample (Fig. 5b) many dimples whose sizes are on the order of magnitude of the UFG grain diameters were detected. It is clear that the size of dimples in the UFG material is much smaller than that of the CG alloy. The fracture surface of bimodal material (Fig. 5c and 5d) shows fine dimples related to UFG region and coarse dimples related to CG region. Additionally, the fracture surface exhibited a localized plastic deformation within the CG regions (necking) as well as cavitation at CG/UFG interfaces revealing an extensive plastic deformation prior to fracture (Fig. 5c). Cavitations were also observed along the edge of the sample in the regions of largest deformation concentrated close to the fracture area. They are hypothesized to be the result of un-accommodated strain between the UFGs and CGs. Cavitation and necking can cause plastic instability and eventually a cavitation-controlled failure in the sample [13]. Cavitation/necking-controlled failure usually provides a fracture surface perpendicular to the applied load. Nevertheless, the fracture surface of the bimodal sample exhibited a combined condition, with the fracture surface perpendicular to the applied load at the center, and 45° to the applied load at both edges of the flat specimen, indicating that shear banding occurs in the final stages of failure.
Improved elongation to failure of the bimodal material in comparison to UFG counterpart can be related to the existence of the coarse grains that tend to deform extensively and contribute to the global uniform elongation. In fact, CGs experience substantially more plastic deformation than the UFGs do, and therefore parts of the UFG matrix must displace toward the deforming CG to maintain the cohesion of the CG boundary.

Detection of necking, cavitations (Fig. 5c) and delaminations (Fig. 5d) between CG and UFG regions on the fracture surface introduces a specific fracture mechanism for the bimodal Al6063. The final break occurs in CG regions while the height of failed CG regions is larger than UFG matrix level (bright areas in Fig. 5c). Under the tensile load, firstly CG regions plastically deform without fracture and the UFG matrix bears most of the tensile load elastically. At a higher stress level, UFG regions, after yielding undergo a slight plastic deformation because of the limited dislocation activity in ultrafine grains. Thus, UFG regions carry most of the applied stress and a small amount of the load is transferred to the ductile CG regions. This phenomenon
A bimodal grain size was achieved in the hot extruded Al6063 alloy with a grain size of 0.08-1.4 µm in nanostructured regions and 1.8-4.1 µm in coarse-grained regions. Therefore, large localized plastic strain occurs in CGs. Under increasing tensile load, the mismatch in properties, namely the ductility, across the CG boundary results in the UFG region being stressed beyond its ultimate strength sooner than the CG regions. Voids initiate and grow as cracks in the UFG matrix and especially at CG/UFG interfaces. Cracks propagate away from or around the CGs, but the CGs blunt the crack tip and decrease the stress concentration factor of crack, as illustrated schematically in Fig. 6a. Consequently, cracks are generated within UFG matrix but soft CGs limit their growth which is termed “ductile-phase toughening mechanism” [14]. Also the interface delamination between CG and UFG regions perpendicular to the fracture plane occurs (Fig. 6b). Delaminations represent a severe plastic deformation near the fracture surface during tension and enhance the ductility of a bimodal microstructure [15]. In the final stages of tension under a high applied stress, crack propagation accelerates until the crack spans, running through both microstructural regions. Neeking happens in CGs and eventually the global failure occurs. The mechanism of failure introduced in this study is in a reasonable agreement with the mechanism proposed by Lavernaia et. al [11] on a bimodal Al-Mg alloy.

Fig. 6: Schematic illustration of: (a) crack initiation and crack tip blunting; (b) necking, cavitation and delamination in a bimodal structure.

4. Conclusions
Al6063 alloy was fabricated by consolidation of a mixture of milled nanocrystalline powders and unmilled coarse-grained powders with a volume fraction of 30%. The main findings can be summarized as:

1) A bimodal grain size was achieved in the hot extruded Al6063 alloy with a grain size of 0.08-1.4 µm in nanostructured regions and 1.8-4.1 µm in coarse-grained regions.
2) An enhanced tensile elongation with a high strength was observed in bimodal Al alloy. Under tensile load, CG aggregates deform plastically while the UFG matrix bears most of the tensile load elastically.
3) Enhanced tensile ductility was attributed to the occurrence of crack blunting and bridging as well as delamination during the plastic deformation.

References