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Bi-modally structured pure aluminum for enhanced strength and ductility

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ABSTRACT

Nanostructured metals are known to suffer from reduced ductility which limits their industrial applications. In this work, the ductility of nanostructured aluminum is enhanced by developing a bi-modal microstructure comprised of mechanically milled hard aluminum particles dispersed in a soft aluminum matrix. Both low energy (96 RPM) and high energy (200 RPM) solid-state mixing techniques are employed to blend the hard and soft powders. Both optical microscopy and electron backscatter diffraction (EBSD) revealed that a finer bi-modal structure is achievable upon using high energy ball milling and results in improved performance. The capacity for strain hardening is also enhanced and attributed to the observed dislocations in the coarse-grained regions of the material, as confirmed by transmission electron microscopy (TEM) analysis. The results of this work demonstrate that high strength bi-modally structured pure Al with enhanced ductility can be obtained using conventional P/M techniques.

1. Introduction

Aluminum's many exceptional properties promote it as a strong candidate for several applications in the aerospace, automotive, building, and packaging industries. Recently, refining its internal structure to reach the nano-scale has been fostered; an approach motivated by the promise of better strength and ductility [1–11]. A successful technique to strengthen Al is mechanical milling (MM) [12] including cryomilling [13–15]. In spite of the intensive research efforts, the poor ductility of milled Al remains a challenge which limits its applications [15–17]. This has been attributed to limited dislocation activity in the nanograins [15]. One approach aiming to reach a balance between the strength and ductility of nanostructured metals is to develop hierarchical [18] or multi-modal microstructures [13–15]. For example, Wang et al. [7] annealed copper samples that have been cold worked at liquid nitrogen temperature and observed an enhancement in ductility which they attributed to the development of a bi-modal structure upon annealing. The authors reported that the majority of the grains remained in the nanocrystalline/ultrafine scale, and only 25% of the grains were coarsened during secondary recrystallization [7]. Another approach to develop a bimodal structure is to embed micrometer-sized grains in a matrix of nanometer-sized grains [4]. The larger grains help stabilize the tensile deformation in the bi-modal structured material in coherency with the nanocrystalline grains yielding enhanced ductility.

In addition to the few examples of successful bi-modally structured materials, a tri-modally structured material was developed by Ye et al. [14]. They [14] used cryomilling to produce 20 wt% B_4C – 80 wt% nanocrystalline Al5083, which was then mixed with equal amounts of coarse-grained Al5083. A yield strength of 1065 MPa was reported and attributed to the different micro-structural features, such as B_4C particles strengthening, reduced grain size of nanocrystalline Al, and Orowan strengthening from the oxides, nitrides and carbides. With regards to ductility, the authors [14] reported enhancing the strain-to-failure from 0.8% to 2.5% by annealing their composite.

As reported in the previous paragraphs, multi-scale structures can lead to combined strength and ductility [18]. Misra et al. [19] asserted that the use of bimodally structured composites provides the nano and ultrafine-grained materials with higher plasticity agreeing with Xu et al. [20]. In the same fashion, Rajabi et al. [21] added nano-sized stainless steel particles to the conventional micro-sized ones to enhance the ductility without drastically losing the strength. Multi-scale structures can either be produced through thermo-mechanical treatments or through introducing different size-scale powders and consolidating the mixture into bulk [14,22]. To the best of our knowledge, this promising approach has not been applied to pure Al and is thus the subject

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of the current research in which different size-scale Al powders are mixed to produce bi-modal aluminum (BM Al) samples. The microstructures of the BM Al are controlled by means of the powders mixing technique. The mechanical and fracture behaviors are evaluated and related to the produced microstructures.

2. Experimental procedure

Commercial purity Al powders (99.7% pure) supplied by Aluminum Powder Company Ltd. (UK) were mechanically milled for 12 h (MM-12h) using a high-energy ball mill under argon atmosphere (200 RPM, BPR: 10:1). During milling, 2400 μL of Methanol were added as a process control agent (PCA). The amount of process control agent was investigated in an earlier study by the authors [16] to determine the amount that yields the highest tensile strength. As-received and milled Al powders were characterized by X-ray diffraction using Cu Kα in a Scintag XDS 2000 powder diffractometer, operated at 40 kV and 30 mA to study phase analysis as well as the crystallite size. In addition, a Leo Supra 55 Field Emission Scanning Electron Microscope (FESEM) was used to investigate the effect of mechanical milling on the morphology of the powders.

Equal weights of as-received and MM-12h Al powders were mixed using: (1) a turbula mixer for 30 min at 96 RPM and (2) high-energy ball milling for 1 h at 200 RPM and BPR 5:1. Both processes were conducted under an argon atmosphere. The mixed powders were cold compacted under a pressure of 475 MPa for 30 min then sintered for 3 h at 500 °C followed by extrusion using an extrusion ratio of 10:1. The extrudates were machined to produce tension and compression test specimens. Tensile test specimens were dog-bones with 20 mm gauge length and 4 mm gauge diameter. Compression test specimens were cylindrical billets with 12 mm length and 6 mm diameter. The densities of the consolidated samples were measured using a Mettler Toledo densitometer. A JOEL 2010 analytical transmission electron microscope (TEM), having an LaB6 electron gun, was used to study the microstructure of the bulk samples. The samples were prepared on an ULTRATOME microtome at room temperature. Tension tests were conducted following the ASTM E8 [23] standard and the compression tests were carried out according to the ASTM E9 [24] standard. Tension and compression tests were conducted using strain rates of 4×10^{-4}/s and 6×10^{-4}/s, respectively. The fracture surfaces of the tensile specimens were studied using a Leo Supra 55 FESEM. A Bruker GADDS micro diffractometer equipped with a Cu X-ray tube was used to evaluate the crystallite size of the extruded samples and to identify any secondary phases that could have developed during thermo-mechanical processing. The extruded samples were also studied using electron backscatter diffraction (EBSD) by means of an FEI Nova 600 Nanolab FIB/FEG-SEM. The system was operated at 10 kV with a nominal current of 23 nA. The EBSD data was collected with a Hikari XP (EDAX) camera at 6 × 6 binning at a collection speed of 150 fps. The data was collected and analyzed using OIM version 7.1 (EDAX). Maps were displaced at image quality and image quality with IPF overlaid. All patterns were indexed to an aluminum structure file.

3. Results

XRD analysis of the mechanically milled (MM) powders confirmed the refinement that has taken place as demonstrated by the observed broadening and shortening of the peaks in the MM-12h Al spectrum (Fig. 1). Scherrer's equation was used to determine the average crystallite size – based on broadening for Gaussian shaped peaks – which was found to be 16.7 nm for the MM-12h powders compared to 30.1 nm for as-received powders. Upon subjecting the powders to thermo-mechanical processing (compaction and extrusion), the crystallite size increased by almost three folds for both the as-received and the MM 12h extruded bulk samples compared to their powders. The crystallite size for the extruded bulk samples showed a finer size for the samples mixed using the ball mill (see Table 1). This is derived by the additional deformation to which the material is subjected during mixing by the ball mill. The micro-strain measured from XRD showed higher strain in the sample mixed using the ball mill for the same reason. The micro-strains of the samples mixed using the turbula mixer and ball mill were 4.74×10^{-4} and 12.05×10^{-4}, respectively.

The insets in Fig. 1 show SEM micrographs comparing the morphology of as-received Al powders to MM-12h Al powders in which the refinement of the powders due to the milling process is apparent. Some large particles can also be observed. These particles were found to be clusters of very fine powders. The clustering is believed to be due to their large surface areas.

Tensile testing results showed that the MM-12h Al samples exhibited a tensile strength that is four times that of the samples made from as-received powders. However, this elevated strength was at the expense of the ductility of the material as can be observed in the representative stress–strain diagrams in Fig. 2. Also included in Fig. 2 are representative stress–strain curves comparing the behavior of bi-modal (BM) Al samples mixed using the two aforementioned techniques (turbula mixing and ball milling) to the behavior of their two constituents (MM-12h Al and as-received Al). Although commonly used for blending powders, the use of the turbula mixer yielded poor results. On the other hand, using the high-energy ball mill to mix the powders produced samples which exhibited higher strength and enhanced ductility. Increased work-hardening capacity compared to the MM-12h Al samples was also observed. The tensile strength of the bi-modal material was found to be 315 MPa which is a reduction of only 31.5% compared to the MM-12h Al sample. In addition, the ductility was enhanced from 2.35% (±0.45) to 6.94 % (±1.34) which is higher than typical nanocrystalline metals [15].

Average tensile and compressive strengths values (based on 3 samples per set) for all samples are presented in Table 2. As evident, the compressive behavior of the samples agreed with the tensile results. The bi-modally structured Al samples produced using the high-energy ball mill demonstrated a significantly higher compressive strength compared to the turbula-mixed powders. However, the compressive strength is reduced compared to the MM-12h Al with no change in ductility.

The bi-modal Al samples demonstrated good consolidation as density was found to be 98.20 % ± (0.07) for the turbula mixed samples and 98.60 % ± (0.13) for the ball-milled ones. FESEM micrographs for the fracture surfaces of the BM Al samples mixed using the turbula mixer and the ball mill are shown in Fig. 3(a) and (b), respectively. Also, for the sake of comparison, the fracture surfaces of the as-received Al and MM-12h Al samples are presented in Fig. 3(c) and (d) with the former showing evidence of failure in ductile mode and the latter demonstrating a distinctive brittle fracture. Fig. 3(a) shows that the hard particles of MM-12h Al are in the form of clusters surrounded by the ductile Al matrix. In addition, deformation and yielding seem to be limited to the soft Al matrix. On the other hand, the sample mixed using the ball mill, Fig. 3(b), shows better dispersion of the two constituents and more uniform yielding. It is believed that the premature failure in the case of the turbula-mixed samples is due to the nucleation and growth of voids at the interface between the yielded matrix and the hard particles due to the difference in their deformation behavior. Optical microscopy images in Fig. 4(a) and (b) are consistent with the SEM results. They show a much more refined