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## Mechanical behavior of ultrafine-grained Al composites reinforced with B<sub>4</sub>C nanoparticles

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The influence of nanoscale reinforcement on the mechanical behavior of ultrafine-grained composites was studied. Al 5083 (Al-4.5 Mg–0.57Mn–0.25Fe) composites, with grain size of 115 nm and  $B_4C$  reinforcement size of 38 nm, were fabricated via cryomilling and consolidation. The result reveals that the presence of nanoparticles enhances strength by interacting with dislocations, while simultaneously retarding grain growth. Furthermore, the nanoparticles-reinforced composite exhibits enhanced plasticity relative to the same material reinforced with micrometric particles. The underlying mechanisms are discussed. © 2011 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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Ultrafine-grained (UFG) aluminum matrix composites (grain size 100-500 nm) are of interest for weight-critical applications, given their potential to improve fuel efficiency and limit carbon dioxide emissions, particularly in light of recent reports that suggest that it may be possible to develop novel materials with ultrahigh strength ( $\geq 1$  GPa) [1,2]. The published results suggest that the elevated strength in a UFG matrix is mostly derived from the Hall-Petch strengthening mechanism; however, it is typically achieved at the expense of tensile ductility due to the limited work hardening that is associated with the ultrafine grain sizes, which ultimately causes early strain localization. Moreover, it is indicated that the presence of micrometric ceramic particles negatively affects the resistance of the UFG matrix to sustain strain localization because the micrometric ceramic particles are prone to act as stress concentrators that promote early nucleation of cracks and voids [3]. One approach that has been successfully implemented to enhance plasticity and contain strain localization with minimal strength degradation involves the introduction of a bimodal grain size distribution (referred to as a trimodal composite) [4,5]. However, even in these composites large plastic strains were only reported for samples tested under compression, or at high strain rates

 $(>10^3 \text{ s}^{-1})$ . In fact, for the samples tested in tensile tests, the elongation was found to be less than 1% [4,6,7], while monolithic UFG Al alloys can display a tensile ductility of up to 10% [8,9]. These results invite the question whether it is possible to further refine a bimodal grain architecture by reducing stress/strain localization in a trimodal composite and thereby retrieve the plasticity of the bimodal grain matrix? To that effect, spherical nanoparticles have been successfully incorporated into coarse grained (grain size  $>1 \mu m$ ) composites to augment resistance to lattice dislocation glide via the Orowan mechanism, as well as to diminish stress localization from the particle size reduction, leading to a simultaneous increase in yield strength and ductility in coarse grained Al/Si<sub>3</sub>N<sub>4</sub> ( $\sim$ 15 nm) [10] and Al/Al<sub>2</sub>O<sub>3</sub> ( $\sim$ 90 nm) [11].

Recent studies have revealed that nanostructured metals may sustain tensile elongation if strain localization is effectively hindered. Xiang et al. [12] demonstrated that nanostructured Cu films that are bonded well on a polymer substrate can sustain tensile strains of up to 10% without any appreciable cracks and up to 30% with discontinuous microcracks. By contrast, poorly bonded Cu films form channel cracks at strains of about 2%. More recently, Fang et al. [13] showed that nanostructured Cu films confined by a coarse-grained Cu substrate can sustain a tensile true strain exceeding 100% without cracking through suppressing strain localization with a gradient grain size transition.

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In view of the above studies, the present work was undertaken to provide insight into the viability to retrieve plasticity by increasing resistance to dislocation glide and limiting stress/strain localization in a UFG matrix, thereby decreasing the size of the reinforcement phase to the nanometer scale and changing its morphology. B<sub>4</sub>C particles were selected because this material is the third hardest ceramic, ranked just after diamond and cubic BN, and possesses a low density of 2.51 g cm<sup>-3</sup> (20-40% lighter than SiC, Al<sub>2</sub>O<sub>3</sub>, Si<sub>3</sub>N<sub>4</sub>, AlN, cubic BN or diamond). These unique characteristics, along with other attractive properties such as high impact and wear resistance, and a high capacity for neutron absorption, renders it a good reinforcement candidate material [14,15]. In this study, UFG Al 5083 matrix composites reinforced with B<sub>4</sub>C nanoparticles were fabricated through a powder metallurgy process. We first demonstrate that the high-strength UFG composite can undergo large plastic strain under compression and then the tensile deformation behavior is investigated.

Gas-atomized Al 5083 (Al-4.5 Mg-0.57Mn-0.25Fe, in wt.%) powder with a particle size of  $<45 \,\mu m$  (<325mesh) was V-blended with 5 vol.% B<sub>4</sub>C nanoparticles. The  $B_4C$  nanoparticles (designated  $n-B_4C$  hereafter) were fabricated and supplied by ARDEC (Armament Research, Development and Engineering Center, Picatinny, NJ), and had an average three-dimensional particle size of  $\sim$ 38 nm (or an average cross-sectional particle size of 31 nm with a standard deviation of 11 nm), as shown in Figure 1. The powder blend was cryomilled in liquid nitrogen for 12 h. Cryomilling was conducted in a modified Svegvari attritor at a speed of 180 rpm with a ball-to-powder ratio of 32:1. After cryomilling, the average grain size of the composite powder was determined to be 30 nm by X-ray diffraction line profile analysis. The cryomilled, nanostructured A15083/5 vol.% n-B<sub>4</sub>C composite powder was hot vacuum degassed at 400 °C for  $\sim$ 16 h (designated as sample  $5-nB_4C$  hereafter). For the trimodal composite samples (with coarse grain addition), 30 vol.% unmilled Al 5083 powder (with a grain size on the order of  $1 \mu m$ ) was V-blended with the cryomilled composite powder prior to degassing, to give a final composition of 3.5 vol.% *n*-B<sub>4</sub>C, 66.5 vol.% UFG grains and 30 vol.% coarse grains (designated as sample 3.5-nB<sub>4</sub>C-30CG hereafter). The degassed powder was then consolidated by hot isostatic pressing (HIP) at 400 °C, followed by extrusion at the same temperature with an extrusion ratio of 10:1. The microstructures of the samples were characterized using a Phillips CM-12 operating at 100 kV for grain size statistical analysis. High-angle angular dark-field imaging was conducted to examine

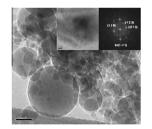
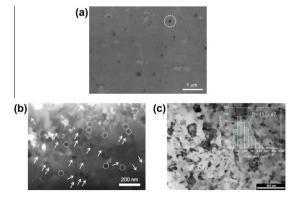


Figure 1. TEM bright-field images of B<sub>4</sub>C nanoparticles.

the B<sub>4</sub>C nanoparticle dispersion using a JEOL JEM-2500SE microscope operating at 200 kV. The transmission electron microscopy (TEM) specimens were mechanically thinned to a thickness of 20 µm and then perforated using a Gatan PIPS 691 ion-milling machine. Cross-sections of the composites and fracture surfaces of tensile tested samples were examined using scanning electron microscopy (SEM). The tensile tests were carried out along the extrusion direction with an Instron 8801 universal testing machine using dog-bone-shaped specimens with a gauge length of 12 mm and a diameter of 3 mm. The compression tests were conducted using cylindrical specimens 8 mm in height and 5 mm in diameter. All the mechanical testing was performed at a strain rate of  $10^{-3}$  s<sup>-1</sup> and the strain was measured using a video extensometer with a resolution of  $5 \,\mu m$ .

A SEM micrograph of the cross-section of the  $5-nB_4C$ sample in the extrusion direction is shown in Figure 2(a). The image clearly indicates that the  $B_4C$  nanoparticles (dark particles, centered around 31 nm) are homogeneously dispersed in the Al 5083 matrix. The light particles are determined to be Al<sub>6</sub>(Mn, Fe) that exists as a non-heat-treatable intermetallic phase in the Al 5083 alloy [16]. There are occasional voids left over from consolidation (marked with a circle in Fig. 2(a)). Such residual microscale voids, although seen only rarely in our studies, may promote the initiation of shear localization and the onset of fracture, as is further addressed below. It can also be seen that a small fraction of B<sub>4</sub>C particles in the range of 100-200 nm is present. A high-angle angular dark-field image, revealing the finer B<sub>4</sub>C particles as well as matrix grains, is displayed in Figure 2(b). Agglomeration of reinforcing nanoparticles on grain boundaries, which was reported in micrometric-grained Al composites exceeding 4 vol.% nanoparticles [11], was not observed in our studies. Moreover, we did not observe regions denuded of reinforcing particles [7,17], which have been reported to evolve as a consequence of matrix diffusion into interparticle regions during consolidation of nanocomposite powders. Figure 2(c) is a bright-field TEM image of the Al 5083 matrix (along the extrusion direction), with the inset showing the histogram of the grain size distribution for 375 grains. The grains are mostly equiaxed, with an average size of 115 nm. The geometric mean grain



**Figure 2.** Microstructure of Al5083/5 vol.% n-B<sub>4</sub>C composite: (a) SEM cross-section; (b) high-angle angular dark-field image; (c) TEM bright-field image. The n-B<sub>4</sub>C particles at grain boundaries and within grains in (b) are marked with circles and arrows, respectively.

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