



## Metal/ceramic interface structures and segregation behavior in aluminum-based composites



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### ABSTRACT

Trimodal Al alloy (AA) matrix composites consisting of ultrafine-grained (UFG) and coarse-grained (CG) Al phases and micron-sized B<sub>4</sub>C ceramic reinforcement particles exhibit combinations of strength and ductility that render them useful for potential applications in the aerospace, defense and automotive industries. Tailoring of microstructures with specific mechanical properties requires a detailed understanding of interfacial structures to enable strong interface bonding between ceramic reinforcement and metal matrix, and thereby allow for effective load transfer. Trimodal AA metal matrix composites typically show three characteristics that are noteworthy: nanocrystalline grains in the vicinity of the B<sub>4</sub>C reinforcement particles; Mg segregation at AA/B<sub>4</sub>C interfaces; and the presence of amorphous interfacial layers separating nanocrystalline grains from B<sub>4</sub>C particles. Interestingly, however, fundamental information related to the mechanisms responsible for these characteristics as well as information on local compositions and phases are absent in the current literature. In this study, we use high-resolution transmission electron microscopy, energy-dispersive X-ray spectroscopy, electron energy-loss spectroscopy, and precession assisted electron diffraction to gain fundamental insight into the mechanisms that affect the characteristics of AA/B<sub>4</sub>C interfaces. Specifically, we determined interfacial structures, local composition and spatial distribution of the interfacial constituents. Near atomic resolution characterization revealed amorphous multilayers and a nanocrystalline region between Al phase and B<sub>4</sub>C reinforcement particles. The amorphous layers consist of nonstoichiometric Al<sub>x</sub>O<sub>y</sub>, while the nanocrystalline region is comprised of MgO nanograins. The experimental results are discussed in terms of the possible underlying mechanisms at AA/B<sub>4</sub>C interfaces.

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### 1. Introduction

Nanostructured (<100 nm) and ultrafine-grained (100–500 nm) materials are noteworthy because they provide an opportunity to study the influence of microstructural dimensions on mechanical behavior. Remarkable enhancements to mechanical strength and physical performance have been documented for such materials [1–4]. One strategy to produce nanostructured or ultrafine-grained materials involves severe plastic deformation (SPD) using, for instance, equal channel angular pressing, high pressure torsion [5], surface mechanical attrition treatment [6], and ball milling [2,7]. A derivative of ball milling that has been broadly used to prepare nanocrystalline microstructures is

cryogenic ball milling (cryomilling), which takes advantage of cryogenic temperatures to suppress dynamic recrystallization by dissipating heat generated during the milling process [2]. Regardless of the synthesis technique, room temperature ductility and strain hardening of nanocrystalline materials are generally degraded by the limited dislocation activity that occurs at these grain sizes [8–11]. A number of approaches, such as the introduction of coarse grains, precipitate particles and promoting twin formation, have been successfully used to enhance the ductility of nanocrystalline materials [12]. Another approach involves the development of trimodal metal matrix composites, which consist of a coarse grained (CG) structure (larger than 1 μm grain size), an ultrafine-grained (UFG) structure, and micron-sized B<sub>4</sub>C reinforcement particles [13–16]. The CG structure is employed to provide ductility, while the UFG structure facilitates grain boundary strengthening due to, e.g., the Hall–Petch relationship. The B<sub>4</sub>C particles pin the movement of dislocations to further resist plastic

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deformation, lower the material's density and increase the elastic modulus of the metal matrix composite.

Inspection of the published literature shows that the mechanical behavior of trimodal composites has been studied for the past decade [13–15,17,18]. A widely used matrix is Al–Mg partly due to the excellent balance of strength, weldability and corrosion resistance of this family of alloys [19]. The original trimodal composite [14], consisting of 10 wt.% micron-sized  $B_4C$ , 40 wt.% UFG 5083 AA and 50 wt.% CG 5083 AA, was manufactured through cryomilling, blending, cold isostatic pressing and extrusion. This composite exhibited ultra-high compression yield strength of 1065 MPa. Early trimodal composites established themselves as attractive alternatives to conventional armor steel due to their excellent dynamic deformation behavior, with strain-to-failure of 14% at strain rates on the order of  $10^3 \text{ s}^{-1}$  and strength values as high as 1000 MPa [13,20]. However, widespread application of the early trimodal composites was restrained by their limited strain-to-failure (0.8%) when tested under quasi-static compression, and failure prior to yielding when tested under tensile loading.

Modifications of the trimodal composite with a higher content of the CG phase led to tensile yielding but with limited elongation (1–2%) [15]. More recently, Zhang et al. [21] showed that nanometric  $B_4C$  reinforcement suppressed stress/strain localization and thereby improved plasticity. In this particular study a trimodal composite consisting of 3.5 wt.% nano-sized  $B_4C$ , 30 wt.% CG 5083 AA and the balance UFG 5083 AA was reported to exhibit a strength above 630 MPa with a tensile strain of 3% [21].

A critical requirement for the application of trimodal composites is the creation of a strong interfacial bond between ceramic reinforcement and metal matrix to allow for effective load transfer. To that effect, the interfacial structures that form between Al and  $B_4C$  reinforcements in 5083 AA trimodal composite have been studied using high resolution transmission electron microscopy (HRTEM) [8,17,22]. Li et al. [17,22] reported the presence of amorphous layers, with a typical thickness between 5 and 8 nm, separating  $B_4C$  particles and the UFG Al phase. The authors suggested that the interfacial layers likely originated from the native surface oxide of 5083 AA during processing [17], but detailed studies were not conducted to ascertain the origin of these layers. Hence, the possibilities that the interface layers originated from either amorphization of crystalline  $B_4C$  [23] under shock load or from amorphous boron oxide [24] due to surface oxidation were not considered.

The formation of one to three layers of nanocrystalline grains with diameters of approximately 30–50 nm at the Al/ $B_4C$  interface has also been reported previously [8,22]. Segregation of Mg to Al/ $B_4C$  interfaces was demonstrated using electron energy loss spectroscopy (EELS) and energy-dispersive X-ray spectroscopy (EDXS) [17,25]. Vogt et al. suggested that nucleation of micro-cracks approximately 100 nm away from the AA/ $B_4C$  interfaces may have led to failure during compression testing [13]. However, studies of interfacial phenomena focused on only small length fractions of grain boundaries and did not provide a global understanding of the characteristics and possible influence of interfacial composition and structure. For example, no energy-loss near-edge fine structure (ELNES) studies have been performed to provide bonding information for the elements segregated to the interface. Hence, information on the relationship between different interface morphologies (e.g., amorphous layers, nanocrystalline grains, etc.) and their chemical composition profiles remains absent in the literature.

In view of the above discussion, we determine microstructure information from HRTEM, EDXS, EELS, and precession assisted electron diffraction. The experimental results are used to ascertain a fundamental insight into the mechanisms that affect the characteristics of Al/ $B_4C$  interfaces.

## 2. Experimental details

### 2.1. Materials synthesis

The feedstock powders used to produce trimodal Al matrix composites were gas-atomized 5083 AA (4.5 wt.% Mg, 0.57 wt.% Mn, 0.25 wt.% Fe balanced with Al) provided by Valimet, Inc. (Stockton, CA) and  $B_4C$  powder (Tetrabor<sup>®</sup>, F1200) from ESK Ceramics (Saline, MI). The as-received 5083 AA powder had an average particle size below 45  $\mu\text{m}$ . The particle size for the as-received  $B_4C$  powder ranged from 1 to 7  $\mu\text{m}$  [26]. The composite powder was fabricated via cryomilling [2] of 14 wt.%  $B_4C$  and 76 wt.% UFG 5083 AA for 24 h using stainless steel balls as grinding media. Chemical analysis of the cryomilled powder was completed by Luvak, Inc. (Boylston, MA) and Shiva Technologies (Syracuse, NY). Non-metallic elements were measured using combustion infrared detection and inert gas fusion, while metallic elements were measured using direct current plasma emission spectroscopy. The chemical composition analysis of the as-cryomilled powder before mixing with CG 5083 AA is shown in Table 1 [26]. After cryomilling the powder was mixed with CG 5083 AA.

The mixed powder consisting of three different components was compacted to approximately 50% relative density inside a nitrogen glove box and subsequently degassed in a furnace to remove hydrogen and oxygen introduced by the surfactant (stearic acid) used during milling. Degassing was performed at  $4 \times 10^{-5}$  Torr while the sample was heated over 4 h up to 400 °C with a holding time of 8 h. Degassing was considered completed when the pressure reached  $2 \times 10^{-6}$  Torr. Degassed powders were subsequently consolidated by hot isostatic pressing (HIP) at  $400 \pm 4$  °C and  $101.3 \pm 1.7$  MPa for 4 h. Extrusion was used for secondary consolidation at a rate of 48.8 m/min. To aid with the deformation during extrusion, the billets were pre-heated in a furnace at 525 °C for 30 min and then extruded at a ratio of 6.5:1. The final product is designed to consist of 10 wt.%  $B_4C$ , 30 wt.% CG 5083 AA, and the balance of UFG 5083 AA [25]. The grain sizes of these micro constituents after consolidation were determined through TEM to be 400–600 nm, 600–2600 nm, and  $174 \pm 100$  nm, respectively. More detailed information about the synthesis procedure is published elsewhere [14,18,25,26].

### 2.2. Microstructure characterization

The morphology and structure of different phases and their interfaces was investigated by transmission electron microscopy (TEM) using either a JEOL JEM-2500SE instrument (JEOL Ltd., Tokyo, Japan) at University of California, Davis (UCD), or a FEI monochromated F20 UT Tecnai scanning transmission electron microscope (FEI Company, Hillsboro, OR) at the National Center for Electron Microscopy (NCEM). Scanning transmission electron microscopy (STEM) was carried out with a Titan G2 80-200 instrument (FEI Company, Hillsboro, OR) equipped with ChemiSTEM technology and a Fischione high angle annular dark field (HAADF) detector (Fischione, Export, PA) at the FEI Nanoport (Hillsboro, OR). All microscopes were operated at 200 kV.

Electron-transparent TEM samples were prepared with either a FEI Versa 3D focused ion beam (FIB) at the FEI Nanoport, or a FEI Scios FIB at UCD. The starting voltage was 30 kV for trenching and initial thinning. The accelerating voltage of the ion beam was then reduced to 5 kV when sample thickness reached approximately 400 nm. Final thinning was performed at 2 kV when the TEM lamellae became electron-transparent. After final thinning, an ion beam energy of 0.5 kV was used to remove most of the remaining amorphous damage layers from the sample surfaces. The final thickness of the TEM samples was approximately 60 nm.

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