



# Influence of length-scale on stabilization of boron carbide in Al-based metal matrix composites during plasma activated sintering

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## ABSTRACT

We reported an investigation of the influence of length scale on stabilization of boron carbide ( $B_4C$ ) during plasma activated sintering. In the current work, AA7075/ $B_4C$  powders containing two types of  $B_4C$  particles (Micro- $B_4C$  and Nano- $B_4C$ ) were cryomilled for 8 h in liquid nitrogen, respectively. The characterization of as-cryomilled powders indicated that both Micro- $B_4C$  particles and Nano- $B_4C$  particles could distribute homogeneously in the matrix. AA7075/Nano- $B_4C$  powder have a finer particle size and grain size relative to that of the AA7075/Micro- $B_4C$  powder. The influence of length scale on stabilization of  $B_4C$  particles during plasma activated sintering was investigated in details. The statistical grain diameter analysis from transmission electron microscopy observation revealed that the grain size of AA7075/Nano- $B_4C$  bulk (~77.4 nm) is finer relative to those of the AA7075 bulk (~193.5 nm) and AA7075/Micro- $B_4C$  bulk (~184.7 nm). The coupling effect of ex-situ Nano- $B_4C$  particles and in-situ dispersion particles contribute to the enhancement of the stabilization of the AA7075/Nano- $B_4C$  bulk.

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## 1. Introduction

Al-based metal matrix composites (MMCs) reinforced with ceramic particle provide an approach for enhancing the mechanical behaviors of Al/Al alloy without increasing the weight or relative density [1–7]. The development of strong and lightweight Al-based MMCs are of particular interest for use in aerospace, automobile and defense application due to their enhanced specific strength, stiffness, creep and fracture resistance [8–13]. The grain refinement of Al, to form nanostructured Al-based MMCs, is an effective method to further enhance the mechanical behaviors of Al-based MMCs. The unique properties of nanostructured Al-based MMCs are closely related to the fine grain size and high density of grain boundaries [14]. Inspection of the published paper reveals that the cryomilling, similar to conventional ball milling, is representative of a kind of synthesis techniques that obtain the nanostructured state by mechanical alloying in liquid nitrogen medium. Cryomilling could suppress the recovery and recrystallization by taking advantage of the extremely low temperature, which results in finer grain size and more rapid grain refinement. Nevertheless, nanostructured materials are always in an extremely higher energetic state relative to their coarse-grained materials, which results in the grain growth at elevated temperature or even at ambient temperature [15].

It is of utmost importance to enhance the thermal stability of the nanostructured Al-based MMCs. Previous studies have investigated the thermal stability of the pure Al powder, AA5083 powder, and AA5083/ $B_4C$  powder [16–18]. These studies have demonstrated the growth kinetics of the powders, suggesting that the activation energies for grain growth at elevated temperature regime are dominated by the lattice diffusion, whereas various mechanisms control the grain growth in the low temperature regime. Various stabilizing methods of grain size for the nanostructured Al-based MMCs has aroused extensive investigation to inhibit the grain growth during annealing or consolidation, including grain boundary segregation, solute drag, and pinning particles. These methods contribute to the stabilization of the grain size either by reducing the grain boundary energy or by impeding the mobility of the grain boundary. Among these approaches, the pinning particles have been demonstrated as an effective method for the stabilization of nanostructured Al-based MMCs via impeding the mobility of the grain boundary [19]. Both in-situ formed dispersoids (such as oxide, carbide and nitride) and ex-situ reinforcements can be employed [19]. Roy et al. reported the enhanced thermal stability of the AA5083 bulk due to the presence of the in-situ formed dispersion particles [20]. Hashemi-Sadraei et al. revealed the effect of the process-generated nitrides on the thermal stability of the AA5083/ $B_4C$  powder, specifically controlled via cryomilling time. The results indicated that the 24-h as-cryomilled AA5083/ $B_4C$  powder retained the nanostructure even after annealing 24 h at 823 K [21]. Āurišinová et al. investigate the influence of the volume fraction of the ex-situ  $Al_4C_3$  on thermal stability of Al- $Al_4C_3$  bulk

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composites, the results suggest that all the composites are stable up to 773 K [22]. Nevertheless, these studies did not provide fundamental information related to the influence of length scale on stabilization of the nanostructured Al-based MMCs, especially the ex-situ reinforcement. In view of the lack of fundamental information about the influence of reinforcements on the stabilization during consolidation, the goal of the current work is to provide the fundamental insight into the following two questions: (i) How did the length scale of ex-situ reinforcements affect the grain refinement during cryomilling? (ii) How did the length scale of ex-situ reinforcements affect the stabilization of the composites during consolidation?

In order to clarify the aforementioned questions, AA7075/B<sub>4</sub>C composites with two types of B<sub>4</sub>C powders (micron and nanoscale particle size) were chosen as the model system in the current work. For a constant volume fraction, the mixed AA7075/B<sub>4</sub>C powders were cryomilled to form the nanostructured Al-based MMCs, subsequently, the as-cryomilled powders were consolidated via plasma activated sintering. Hence, it is our purpose to investigate the microstructure of the as-cryomilled powders as well as the as-sintered bulks.

## 2. Experimental procedures

Gas-atomized AA7075 (Bai Nian Ying, Zhejiang, China) powder with a particle size of <74 μm was V-blended with 7.5 wt % Micro-B<sub>4</sub>C (Aladdin™, median particle size of ~2.0 μm) and 7.5 wt % Nano-B<sub>4</sub>C (Hefei Kaier Nano, media particle size of ~50 nm), respectively. The morphology and distribution of the raw materials could be found in our prior studies [9,10]. The blended powder was then cryomilled with a stainless steel vessel and balls (~5 mm in diameter) in liquid nitrogen for 8 h, which was conducted in a modified O1-HD attritor at a speed of 600 rpm with a ball-to-powder ratio of 25:1. The cryomilled powders were then consolidated by plasma activated sintering (PAS, ED-PAS III). The compacts with 80 MPa of applied uniaxial pressure were heated up at a rate of 100 °C/min, and then sintered at 450 °C for 5 min. The

AA7075 was cryomilled and consolidated with the similar processing history in the current paper to better understand the length scale of the stabilization of B<sub>4</sub>C particles. The as-cryomilled AA7075, AA7075/Micro-B<sub>4</sub>C, and AA7075/Nano-B<sub>4</sub>C powders are designed as P1, P2 and P3, respectively. The bulk materials consolidated from P1, P2 and P3 are named as S1, S2 and S3, respectively.

The chemical composition of the as-cryomilled powders was obtained via Luvac Inc. The oxygen and nitrogen were measured by inert gas fusion, the content of the carbon was measured by combustion infrared detection, while all others were obtained by direct current plasma emission spectroscopy. Scanning electron microscopy (SEM, Quanta 3D FEG Dual Beam) was used to evaluate the microstructure of the samples. The X-ray diffraction (XRD, Smartlab) was used to identify the phase structure of the cryomilled powders as well as sintered bulk samples. Quantitative analyses of grain diameter and micro-strain were calculated according to the Williamson-Hall method. The full-width at half-maximum (FWHM) of peaks was obtained as a measure of peak broadening, the true peak broadening  $B$  was calculated by  $B = \sqrt{B_o^2 - B_i^2}$ , where  $B_o$  is the observed peak broadening and  $B_i$  is the instrumental broadening. This approach assumes that the broadening includes the grain size broadening and micro-strain broadening using  $B \cos \theta_B = K \frac{\lambda}{d} + \varepsilon \sin \theta_B$ , where  $\lambda$  is the wavelength of Cu K $\alpha$  X-ray,  $K$  is ~0.9,  $\varepsilon$  is the micro-strain, and  $\theta$  is the Bragg angle [23,24]. The dislocation density  $\rho$  could be calculated from the grain diameter  $d$  and micro-strain  $\varepsilon$ , which is given  $\rho = 2\sqrt{3}\varepsilon/db$ , where  $b$  is the Burgers vector (0.286 nm for Al) [23,24]. The microstructure of the samples was also examined by a transmission electron microscopy (TEM, CM-20). The grain size of the Al is determined by TEM observation via measuring Feret diameters of the strongly diffracting grain using image analysis software of ImageJ. More than 200 measurements were conducted for each average value to estimate the statistical accuracy. Samples for TEM studies were prepared using an Ion Milling (Gatan PIPS 691)

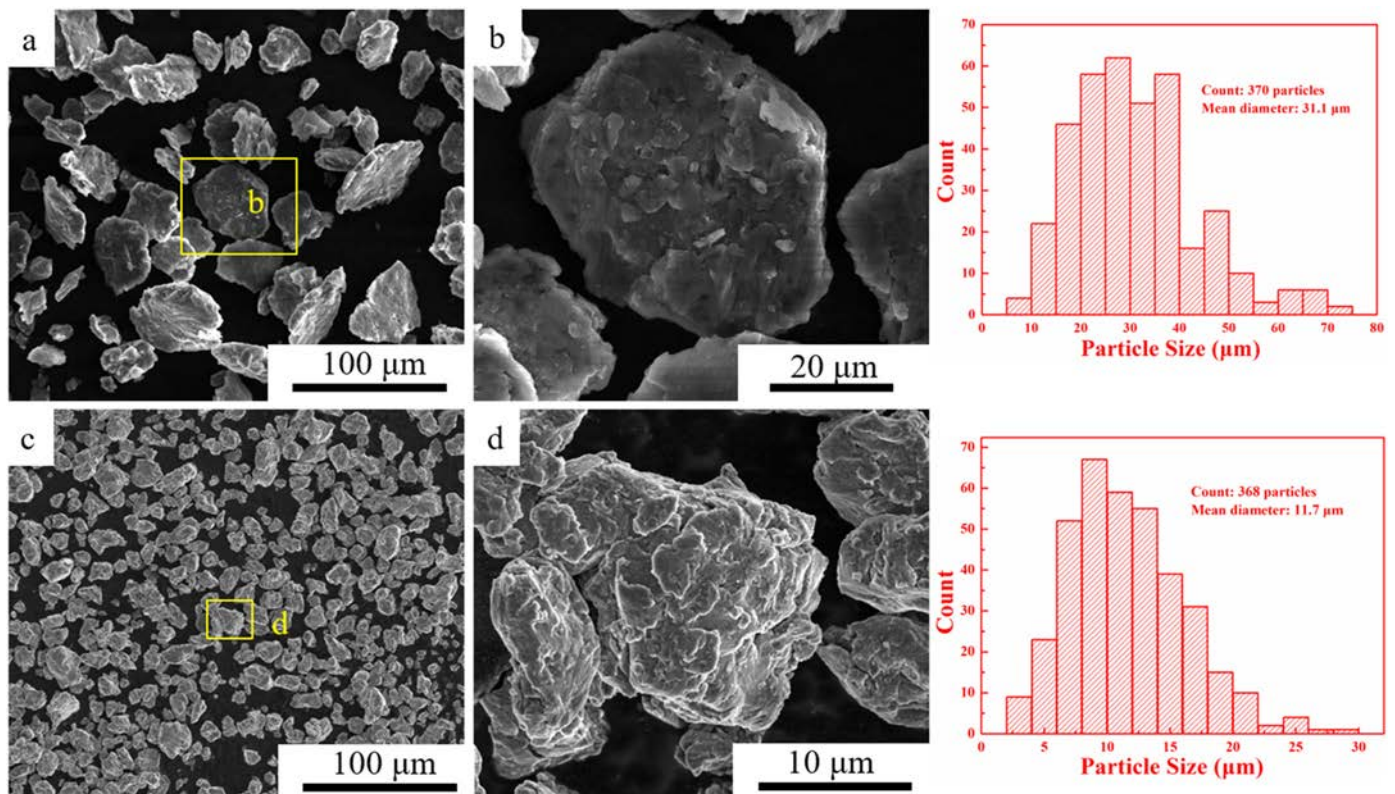


Fig. 1. SEM images showing the morphology and particles size distribution of the B<sub>4</sub>C/AA7075 nano-composite powder synthesized via cryomilling: (a) (b) P2, (c) (d) P3.

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