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Fabrication of Al/AlN in-situ nanocomposite through planetary ball milling and hot extrusion of Al/BN: Microstructural evaluation and mechanical behavior

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ABSTRACT

In this study, Al/AlN in-situ nanocomposites were fabricated using Al/BN as the starting composite powders. The impact of adding hexagonal boron nitride (BN) to the Al matrix of commercial purity on the microstructure and mechanical behavior of the fabricated in-situ nanocomposites was investigated. Samples including 1, 2, and 4 wt.% boron nitride nanoparticles were produced by planetary ball milling of the composite powders and a post-process of hot extrusion. Scanning transmission electron microscopy revealed that boron nitride nanoparticles dissolved as a solid solution of B and N in the Al matrix at the as-milled state. Through the process of hot extrusion, AlN as the in-situ phase was formed by a reaction between Al and N. These led to improve the mechanical properties as well as grain refinement of Al/AlN nanocomposite. The average grain size of the fabricated composites with the use of 1, 2 and 4 wt.% BN was measured about 910, 823, and 760 nm respectively. It was found that combined strengthening mechanisms of grain refinement, a solid solution of mostly B and AlN in-situ phase formation improved the mechanical properties of Al/AlN nanocomposite. With the use of 1, 2, and 4 wt.% BN, the tensile strength of nanocomposite samples increased approximately 40, 56, and 57% in comparison with pure Al, respectively. The remarkable change in microstructure and mechanical properties of the nanocomposite was obtained when the content of BN is up to 2 wt.%.

1. Introduction

Recently, the fabrication of bulk Al-matrix composites with use of BN particles have strongly been considered due to their light weight (lower than pure Al), superior strength at room temperature even at low contents of BN as a reinforcement, and good thermal stability at elevated temperatures [1–3]. Consequently, these composites are good candidates for automotive and aerospace applications in comparison to the other Al matrix composite materials.

Hexagonal boron nitride (h-BN) has exceptional features in comparison to other ceramic particles. These exceptional features are its low density (2.3 g/cm^3 and lower than pure Al), its ability to dissolve in the Al matrix during high energy milling and its ability to be formed in-situ phases during the annealing state of fabrication (sintering, spark plasma sintering and hot extrusion) [4,5].

The structure of Al/BN composite powders and their solid solution formation during planetary ball milling were studied by a number of researchers [5–7]. It is well documented that high energy planetary ball milling can lead to the dissolution of BN in the Al matrix as a solid solution. On the other hand, it has been suggested that the possible AlN and AlB_2 as the in-situ phases are created during a heating state in the Al matrix as the following [4,5].

$$3Al + 2BN \rightarrow 2AlN + AlB_2 \quad (1)$$

Hot extrusion and spark plasma sintering have been used successfully to fabricate Al/BN bulk samples from composite powders [1–3,8]. For instance, Firestein et al. [1] synthesized Al/BN nanocomposite by the homogeneous mixing of composite powders and spark plasma sintering. In this study, the nanocomposite samples with 4.5 wt.% of BN nanoparticles showed 50% increase in the

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tensile strength compared with the pure Al as a reference sample. In the other study, Firestein et al. [2] have fabricated in-situ nanocomposite by planetary ball milling to prepare Al/BN powder and then spark plasma sintering. They found that AlN and AlB_2 as the hard inclusions and the grain refinements due to ball milling process enhanced the tensile strength by about 130% compared with the pure Al.

An alternative for the process of spark plasma sintering is the process of hot extrusion that has several potential advantages including: the low cost of production, the easy manufacturing, no size limitation [9,10], the high shearing stresses during the process which is enough to break the nanometric oxide layer covering powder particles, and the occurrence of the dynamic recrystallization during the process [11]. Gostariani et al. [3], fabricated nanocomposite by planetary ball milling of Al/BN composite powders following a conventional hot extrusion. The tensile strength of the nanocomposite containing minimum content of BN (1 wt.% BN) improved by 40% in comparison with the pure Al. In another research, Gostariani et al. [12], investigated the hot deformation behavior of mechanically milled and hot extruded Al-1 wt.% BN using hot compression tests at different temperatures and strain rates. It is found that the dynamic recrystallization is responsible for the fine-grained microstructure of the hot deformed samples. The nanocomposite resisted against abnormal grain growth and led to the stability of ultrafine structure after hot deformation.

Most of the researches on the Al-matrix composite with use of BN particle have focused on the improvement of mechanical properties wherein the microstructure evolution and strengthening mechanisms are rarely evaluated. Therefore, the present study aims to correlate the microstructure evolution to the mechanical behavior of Al/AlN in-situ nanocomposites contain different amount of BN and fabricated by planetary ball milling and hot extrusion.

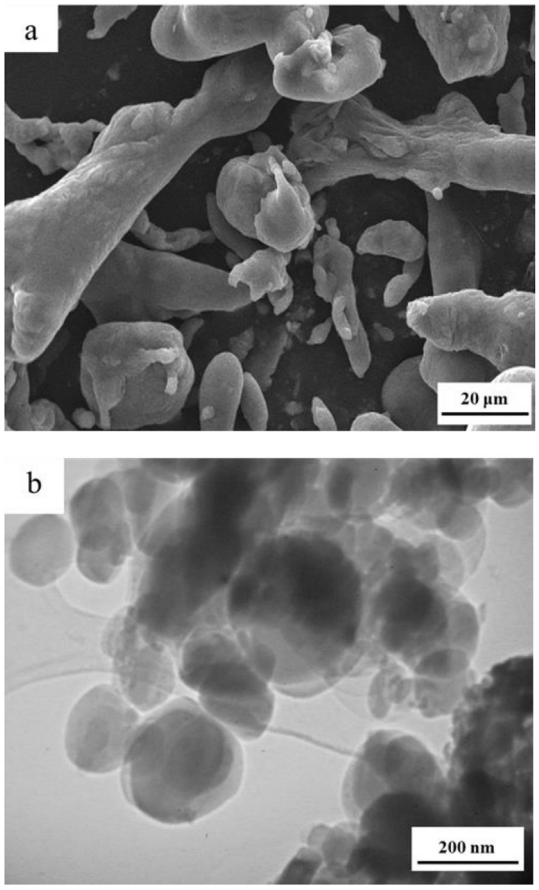

Fig. 1. (a) SEM image of the pure Al powders (b) TEM image of the boron nitride (BN) nanoparticles.

2. Experimental procedure

Commercial pure Al powders with the purity of 99.75% having irregular morphology with an average particle size of 45 μm were employed as the matrix material (Fig. 1 (a)). Hexagonal boron nitride (h-BN) nanoparticles (Lower Friction Co. 99% purity, the average size of 70 nm) were added as the ceramic particle to fabricate Al/AlN in-situ nanocomposite (Fig. 1(b)).

Mixtures of composite powders contain the Al powders with different content of boron nitride (1, 2 and 4 wt.%), and 1 wt.% stearic acid as process control agent (PCA) were prepared using a Fritsch P5 planetary ball mill. The composite powders were loaded with balls (10 mm diameter made of 100Cr6 with density of 7.81 g/cm^3) in a hardened chromium steel round-ended cup. The full volume, diameter, and deepest height of cup are 130 ml, 70 mm, and 45 mm respectively. Also, 50% of the cup is left empty during milling process. The milling process was carried out at room temperature in pure argon atmosphere for 300 min milling time with rotational speed of 430 RPM and powder to ball weight ratio of 1:20. In order to increase compressibility, the milled powder was annealed at 200 °C for 60 min. Then, the composite powders were pressed by a compressive pressure of 190 MPa in a cylindrical die with 35 mm diameter. The pressed samples were sintered at 580 °C for 45 min to fabricate the final dense samples with 10 mm diameter. Finally, these dense samples were extruded with the speed of 0.2 mm/s and extrusion ratio of 12:1. According to the 1, 2, and 4 wt.% of BN in the initial composite powders, the extruded nanocomposites named Al-1 wt.% BN, Al-2 wt.% BN, and Al-4 wt.% BN respectively. A pure Al specimen was prepared with the similar fabrication method (ball milling and hot extrusion processes) to use as a reference sample for comparison of mechanical properties.

A scanning electron microscope (SEM) of type Philips XL 30 and a transmission electron microscope (TEM) of type Philips 100 kV were used to the investigation of the particle size and morphology of pure Al powders and boron nitride particles, respectively. The microstructures of the extruded samples were studied by a TEM/STEM (STEM, JEOL JEM-2100F) equipped with an energy-dispersive X-ray spectrometry (EDS) and the operating voltage of 200 kV. Thin foils for TEM observations were prepared with mechanical polishing to the thickness of 100 μm. Then the thin foils were twin-jet polished using the TenuPol-5 facility (Struers Co. Ltd.) in an electrolyte solution of 20 vol % $HClO_4$ acid and 80 vol % of ethanol with the voltage of 35 V at −20 °C. The specimens were finally polished by ion beam using Gatan 691 precision ion polishing system (PIPS) for ~1 min.

Electron back-scattering diffraction (EBSD) was used for the quantitative analysis of the microstructure. The EBSD observations were performed by a JEOL 7001 F scanning electron microscope (FE-SEM) equipped with a field emission gun operating at 20 kV. For the EBSD measurements, the INCA suite 4.09 software package was used. EBSD analysis was carried out on an area of $45 \times 60\ \mu m^2$ at pixel $0.052\ \mu m^2$. Misorientations below 2° were not measured in the post-processing data analysis. Before the observations, the samples were sectioned through the extrusion direction and then ground using different SiC abrasive papers followed by final polishing with diamond compounds and the polishing cloths. Electropolishing of all EBSD samples were performed in a solution of 20 vol % of $HClO_4$ and 80 vol % of ethanol with a DC voltage of 35 V for 20 s at −20 °C.