



Strengthening mechanisms of graphene- and Al₂O₃-reinforced aluminum nanocomposites synthesized by room temperature milling



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ABSTRACT

Powder metallurgy (PM) is a widely used processing method to synthesize ultrafine and nanometric grain-sized alloys and composites. While most previous work on synthesizing nanocrystalline (NC) metal-matrix nanocomposites (MMNCs) has used cryomilling technique with liquid nitrogen and stearic acid followed by high temperature/high vacuum drying, we employed an advanced room temperature method that involves milling in ethanol followed by low temperature atmospheric drying – a much less expensive process. To understand the strengthening mechanisms of MMNCs, pure NC Al was reinforced with varying concentrations of Al₂O₃ nanoparticles (NPs) and graphene nanoplatelets (GNPs). The results show that i) room temperature milling in ethanol followed by a relatively low temperature drying treatment can produce NC Al and NC Al MMNCs with grain sizes comparable to materials produced by cryomilling, ii) grain boundary strengthening as described by the Hall–Petch relation accounts for the strength of Al–Al₂O₃ MMNCs, and iii) grain boundary strengthening and solute strengthening seem to account for the strength in Al–GNP MMNCs.

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1. Introduction

Nanocrystalline (NC) alloys and metal-matrix nanocomposites (MMNCs) based on lightweight metals such as Al and Mg are currently being heavily investigated in the hopes of producing strengths comparable to those of much denser metals [1–4]. To date, most investigations based on powder metallurgy (PM) have used cryomilling, which is the milling of metal powders using liquid nitrogen as the milling environment under a very low temperature (~80 K) with stearic acid as a process control agent (PCA) [5–8]. Though this cryomilling process is capable of producing grain sizes in the low nanometer range, a high temperature/high vacuum treatment (i.e., degassing process) is necessary to remove the PCA and water of hydration from the powders before they are consolidated first into a green body and then into a dense bulk specimen [9–11].

While it is known that the extremely fine grain size of NC alloys and NC MMNCs is a major contributor to the improved strength of these materials via Hall–Petch-type grain boundary strengthening, there has been speculation that other mechanisms such as Orowan looping, solid solution strengthening, precipitation strengthening, and the coefficient of thermal expansion (CTE) mismatch strengthening may also play an important role in MMNCs [12–16]. For example, Amirkhanlou et al. [17] synthesized AA1050–SiC_p MMNCs by accumulative press

bonding, and they identified the presence of SiC nanoparticles (55 nm) within Al grains (~280 nm) by field-emission scanning transmission electron microscopy (FE-STEM). In their analysis, the most important strengthening mechanisms were Orowan (76.6%) and grain refinement (14.8%). Kim et al. [18] found the opposite to be the case for Mg₁₇Al₁₂–Mg synthesized by high-ratio differential speed rolling (HRDSR). They claimed that the largest contribution to the strength came from grain refinement (~62%) and Orowan (~20%). However, because the majority of work has been carried out on alloys, rather than pure metals, it is difficult to determine the underlying mechanism with any certainty due to the complex structures of the material. Further, in analyses of Mg MMNCs by Ferguson et al. [12] and Al MMNCs by Kongshaug et al. [19], it has been suggested that, in both solidification processed (SP) and PM processed MMNCs, grains size, alloying effects, and increased dislocation density from mechanical post-processing (i.e., work hardening during consolidation) are primarily responsible for the mechanical properties of increased strength and the nanosized reinforcement plays only an indirect role to refine the grain size.

Recently, there has been great interest in exploring the effects of exotic nanoscale reinforcements as potential strengthening agents for NC MMNCs, rather than the more commonly used, spherical Al₂O₃ or SiC nanoparticles [12,13,19]. For example, Maung et al. investigated the mechanical properties of cryomilled pure Al–diamantane composites [20]. In this work, the diamantane particles were added to help prevent coarsening of the grains during consolidation allowing for Hall–Petch strengthening. It was observed that below a grain size of 110 nm, the

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material exhibited an inverse Hall–Petch behavior (i.e., the material softens with decreasing grain size), which the authors claim was due to a change in deformation mechanism; namely, from bulk dislocation movement to grain boundary sliding. It remains to be seen if the inverse Hall–Petch behavior of these materials is due to the extremely fine grain size or is a result of the diamantine addition. Graphene in particular has drawn significant attention as candidate reinforcement due to its reported ultrahigh elastic modulus and strength [21]. In the few studies that have been published on graphene reinforced MMNCs, several strengthening mechanisms have been proposed, including Orowan, CTE mismatch, load transfer, formation of carbides, and strain hardening due to the ball milling [22–24]. However, the main strengthening mechanisms of graphene-reinforced metal-matrix composites (MMCs) remain unclear.

In this work, we investigated the hardness of pure Al and Al reinforced with either Al_2O_3 nanoparticles or graphene nanoplatelets (GNPs) produced by a room temperature ethanol milling procedure. Note that there are many other routes to synthesize composites at room temperature including ECAP (equal channel angular processing) and HPT (high pressure torsion) processing. However, the milling and subsequent compaction method was selected in the present work due to its economic efficiency and ability to produce NC grain sizes. The objectives of this study is to determine i) if room temperature milling in ethanol followed by a relatively low temperature drying treatment can produce NC Al and NC Al MMNCs with grain sizes that are comparable to materials produced by cryomilling, ii) which strengthening mechanisms are active in these materials, and iii) if the inverse Hall–Petch behavior reported for NC Al with grain sizes less than 110 nm is independent of reinforcement type.

2. Experimental procedure

The primary materials used in this investigation were a) 99% pure Al powder (Acros Organics, Waltham, MA) with an average particle size of 75 μm , b) Al_2O_3 powder (Nanophase, Romeoville, IL) with an average particle size of 47 nm, and c) GNPs M5 (XG Sciences, Lansing, MI) with average thickness of approximately 6 nm and average platelet diameter of $\sim 5 \mu\text{m}$. Three different scanning electron microscopes

(SEM) (Hitachi S-4800 Ultra High Resolution Cold Cathode Field Emission Scanning Electron Microscope (FE-SEM), JEOL JSM-6460 LV, and TopCon SM-300 Scanning Electron Microscope) were used for characterization of the samples. Fig. 1 shows the SEM micrographs of as-received 99% pure Al powder (subsequently herein referred to as “pure Al”), Al_2O_3 , and GNPs. To produce NC MMNCs, the reinforcements with various wt.% (GNPs (0.1, 0.5, and 1 wt.%) and Al_2O_3 (1, 3, and 5 wt.%) were dispersed in 99.5% anhydrous ethanol by ultrasonication. Although there is a study to address the effects of the ethanol on the Al during ball milling under cryomilling process [25], it is expected that no influential chemical reaction would occur in the room temperature drying process, as no PCA was used in our work. The Al powder and the reinforcement slurry were added to a Szegvari attritor mill equipped with an alumina reservoir and milled for 6 h at 500 rpm using a ball-to-powder weight ratio of 15:1 (5 mm diameter zirconia balls). The procedure used to synthesize NC GNP and Al_2O_3 -reinforced MMNC powders is schematically illustrated in Fig. 2. Unreinforced pure Al was also produced by the same method to compare with the properties of the composite samples. The milled composite powders were dried at 135 $^\circ\text{C}$ for 1 h to remove the ethanol. The dried powders were consolidated by single action cold compaction in a steel mold with 200 MPa at room temperature followed by single action hot compaction in a steel mold with 500 MPa at 525 $^\circ\text{C}$ in air for 5 min such that a 25.4 mm diameter cylinder with a height of 10 mm was produced. Through density measurement and microstructural evaluation of Fe-2%Cu-0.5%C, Kong et al. [26] showed that, at a certain consolidation pressure, the density of the powder processed samples does not change significantly. Indeed, in a previous work, we have shown that a 500 MPa hot-compaction pressure used in this work is sufficient to consolidate ball milled Al- Al_2O_3 and Al-GNP powders [27]. Selected hot compacted samples were annealed at 535 $^\circ\text{C}$ with measurements of hardness and grain size at 3, 6, 9, 17 and 24 h. Grain size was determined in the hot compacted and annealed samples using the Scherrer equation [28] from X-ray diffraction (XRD) data gathered by a D8 Bruker diffractometer with Cu K α 1 radiation ($\lambda = 0.15406 \text{ nm}$) (scanning from $2\theta = 10^\circ$ to $2\theta = 120^\circ$, step size of 0.02° , counting time of 0.3 s per step). A TEM specimen was prepared using in-situ lift-out technique by an FEI Strata 400 Dual Beam FIB/SEM [29–31]. The sample was imaged in FEI Tecnai

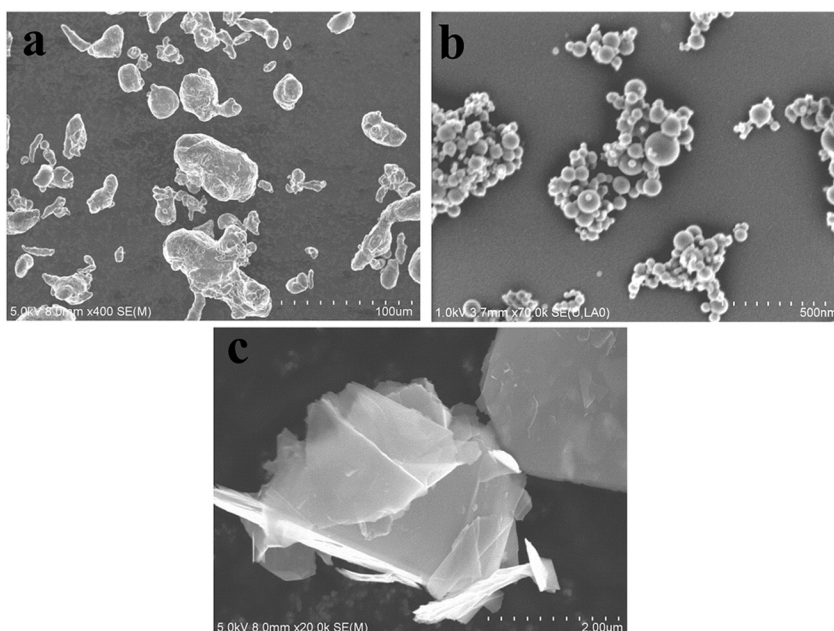


Fig. 1. SEM micrographs of (a) as-received pure Al powder, (b) Al_2O_3 powder, and (c) graphene nanoplatelets (GNPs).

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