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The effect of the addition of multiwalled carbon nanotubes on the uniform distribution of TiC nanoparticles in aluminum nanocomposites

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Aluminum matrix composites reinforced by TiC nanoparticles (with volume fractions of 1.5% and 3%) or a combination of TiC nanoparticles and carbon nanotubes (CNTs) were fabricated using a combination of ball-milling and sheath-rolling techniques. Hybridization of TiC nanoparticles with a small amount of CNTs (0.7 vol.%) significantly enhanced the distribution of TiC nanoparticles in the matrix, indicating that CNTs can be used as carriers to improve the dispersion of nanoparticles in metal–matrix composites.

Keywords: Ball milling; Rolling; Metal–matrix composites (MMCs); Carbon nanotubes; Aluminum

Nano metal–matrix composites have a strength advantage over conventional composites because the Orowan strengthening effect is greatly enhanced by reducing the size of the reinforcing agents from the micrometer to the nanometer scale [1]. Nanosized reinforcements are also favorable for grain boundary pinning and can hence result in smaller matrix grain sizes in the composites, leading to further improvements in the strength of the composites [2,3].

TiC is thermally a very stable refractory metal carbide and possesses high hardness. Many efforts have been made to fabricate TiC/Al composites using in situ methods [4,5]. In in situ as-cast TiC/Al composites, however, TiC particles often exist in clusters [4,5], and it is difficult to disintegrate these TiC clusters by thermomechanical working because TiC particles are often highly interlinked. Furthermore, TiC particles in in situ composites are typically micron sized. Studies on TiC/Al nanocomposites processed by powder metallurgy have rarely been reported in the literature. Very recently, Senthilkumar et al. [6] reported on the microstructure and mechanical properties of TiC/Al composites containing 0.55–1.12 vol.% TiC nanoparticles synthesized using ball milling followed by hot extrusion.

Hybridization of different reinforcement types is attractive because this approach can be used to tailor the properties of the composites. Thakur et al. [7] used nanosized alumina particles to hybridize carbon nanotube (CNT) reinforcements in a Mg matrix. Instead of using 1 vol.% CNTs, a concentration at which it was difficult to obtain a uniform dispersion of CNTs in the Mg matrix (due to the strong tendency of CNTs to form agglomerates), hybridization with an addition of 0.7–0.3 vol.% CNTs and 0.3–0.7 vol.% alumina particles proved to be a better option to enhance the strength of the CNT/Mg composites.

The main objective of this study was to examine the feasibility of synthesizing TiC/Al composites hybridized by the addition of CNTs using a combination of high-energy ball-milling and sheath-rolling techniques. The microstructure and mechanical properties of the hybrid composites were compared with those of the TiC/Al composites fabricated using the same methods and the results discussed.

Multiwall carbon nanotubes (MWCNTs, 20 nm in diameter and ~10–15 μm in length) were manufactured by Iljin Nanotech Co. Ltd. (Korea). using an arc-discharge method. Atomized commercial-purity aluminum powders (99.5% purity, 100–150 μm in diameter) were produced by Changsung Co. Ltd. (Korea). TiC

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nanoparticles (with an average diameter of 45 nm) were purchased from Hefei Kaier Nanometer Energy & Technology Co. Ltd. (China). Using the MWCNTs, TiC particles and aluminum powders, the composite powders were synthesized using a high-energy ball-milling technique. Ball milling was conducted in an attrition mill at 400 rpm for 6 h with a ball-to-powder weight ratio of 15:1 under an argon atmosphere. The ball-milled powders were placed in a copper tube with an inner diameter of 26 mm and degassed for 1 h at 673 K; then, the ends of the tube were sealed. Three types of composite powders were prepared. Two powders contained 1.5 and 3 vol.% TiC particles, and the other contained 1.5 vol.% TiC particles and 0.7 vol.% CNTs. The amount of the added CNTs was determined based on the observation of the ineffective dispersion of CNTs in the matrix at a CNT amount larger than about 1 vol.% [7].

Ball-milled pure aluminum powders were also prepared for reference. The tube samples were held at 723 K for 40 min and then subjected to rolling to reduce the thickness to 2 mm in eight passes. The surface temperatures of the upper and lower rolls were maintained at 473 K. The composite layers obtained after the removal of the copper tube layer were similar for all three composites (0.85–0.90 mm). The density of the rolled nanocomposites was measured using Archimedes' principle. The densities of the pure aluminum, 1.5 vol.%, 3 vol.% TiC, and hybrid composites were 99.3%, 99.5%, 99.4% and 99.4% of the theoretical densities, respectively, indicating that almost full densities were obtained in all the materials after sheath rolling.

The microstructures of the composites on the normal direction (ND)–rolling direction (RD) and the RD–transverse direction (TD) planes were examined via optical microscopy (OM, Olympus BX51 M) and field-emission transmission electron microscopy (TEM, JEM 2100F). Tensile tests were conducted at an initial strain rate of $1 \times 10^{-3}\ s^{-1}$ using specimens with a gauge length of 10 mm cut out from the rolled plate along the RD, using the electrodischarge machining process.

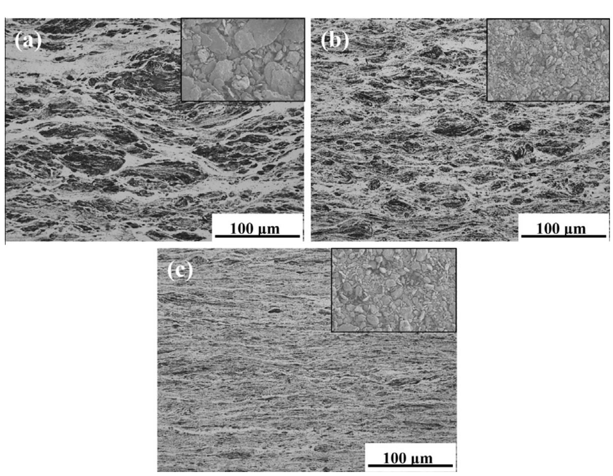

Figure 1. Optical micrographs of the 1.5 vol.% TiC (a), 3 vol.% TiC (b) and hybrid (c) composites obtained after rolling. (d) Raman spectra of the raw CNTs, the ball-milled hybrid powders and the hybrid composite. The insets show the morphology of the obtained after ball milling.

The insets in Figure 1a–c present SEM micrographs of the three types of composite powders fabricated using ball milling. For the pure aluminum and 1.5 vol.% TiC/Al powders, coarse and flattened powders are dominant. However, small powders are dominant for the 3 vol.% TiC/Al and 1.5 vol.% TiC/CNT/Al powders. These observations suggest that the extra addition of TiC or CNTs accelerated the milling process by increasing the work-hardening rate of the aluminum powders. Most of the TiC particles or CNTs appear to have been implanted into the aluminum powders because only a small portion of TiC and CNTs were observed to be on the surface of the powders.

Figure 1a–c presents the OM micrographs of the three composites obtained after the sheath rolling. The TiC particle distribution over the matrix in the 1.5 vol.% TiC/Al composite is heterogeneous. The microstructure consists of two different zones: a thick and elliptical zone with a high density of TiC particles (20–200 μm in length) and a zone with a relatively low density of TiC particles filling the spacing between the zones with high-density TiC particles. The 3 vol.% TiC/Al composite exhibits a heterogeneous microstructure with similar features; however, the size of the zone with high-density TiC particles is much smaller (20–40 μm). This microstructural difference between the two TiC/Al composites may result from the different behaviors that occur during the ball-milling process used here. By adding a higher volume fraction of TiC particles, the break-up of the deformed powders into smaller powders was accelerated and, thus, more uniform blending of the powders occurred, leading to the size reduction of zones with a high density of TiC particles. Another more important observation is that the addition of CNTs significantly enhanced the TiC particle distribution in the hybrid composite (Fig. 1c), leading to the formation of a notably more homogeneous microstructure compared to the two TiC/Al composites.

According to the Raman spectra of the initial CNTs, the ball-milled hybrid powders and the rolled hybrid composite (not shown here), each material exhibits two distinct peaks, which represent a G band due to vibrational modes and a D band arising from disorder-induced modes. The ratio of the intensities of the D and G bands (I_D/I_G), which provides an estimate of the number of defects in graphite structures [8], increased from 0.68 to 1.53 after ball milling. This result implies that the CNTs were damaged during ball milling. No appreciable further increase in the intensity ratio, however, occurred after rolling.

The microstructure of the pure aluminum observed by TEM (not shown here) was homogeneous and the grain size was 0.5 μm. Figure 2a–d shows TEM micrographs of the three composites. The TiC/Al composites exhibit smaller grains than the pure aluminum, but their microstructures are inhomogeneous. Figure 2a,b illustrates the typical microstructures of the zones where the densities of TiC particles are relatively low in the two TiC/Al composites. The number of particles in the matrix is higher in the 3 vol.% TiC/Al composite. In both of the two composites, the TiC particles are uniformly dispersed in very fine-grained matrix (with typical grain sizes of 0.3–0.5 μm). Figure 2c shows a