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Towards strong and stiff carbon nanotube-reinforced high-strength aluminum alloy composites through a microlaminated architecture design

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A microlaminated architecture with aligned interlaminar carbon nanotubes (CNT) was elaborated in Al–Zn–Mg–Cu alloy composites fabricated by flake powder metallurgy. The as-designed composites with 2 vol.% CNT exhibit excellent tensile strength of 820 MPa, Young's modulus of 88 GPa and ductility of 5%. It is proposed that the synergistic effect of the dominant ⟨1 1 1⟩ fiber texture and effective load transfer to CNT contributes to the property enhancement over the disordered composites, which supplies a new strategy towards strong and stiff metal matrix composites.

Keywords: Metal matrix composites; Carbon nanotubes; Powder processing; Laminated structure; Texture

High-strength aluminum alloys such as Al–Zn–Mg–Cu (7xxx series) are extensively used in aerospace and other industries owing to their low density, high strength and good workability [1]. However, in many engineering applications where rigid designs are required, a material with higher strength combined with higher elastic modulus is always desired [2]. In this regard, carbon nanotubes (CNT) are considered one of the most promising reinforcements to meet such requirements [3]. Hundreds of papers have been already published on CNT-reinforced pure aluminum (CNT/Al) composites, but very limited research was done for CNT-reinforced aluminum alloy (CNT/Al alloy) composites. This may be due to the higher strength and hardness of Al alloys than pure Al, which makes it much more difficult to get CNT uniformly dispersed and well bonded in Al alloys while maintaining the structure integrity of CNT [4]. Usually, high-energy ball-milling (HEBM) [5,6] was used to incorporate CNT into Al alloy matrix, which induced more serious damage to the CNT than incorporating in pure Al matrix, and thus greatly reduced the enhancement efficiency of the CNT. As a result, the as-obtained CNT/Al alloy composites exhibited mechanical properties far below expectations, with somewhat enhanced tensile strength, but poor ductility and stiffness, which is no doubt a major hurdle to the practical applications of CNT/Al alloy composites.

Recently, the architecture design of laminated structure has been proved to be an effective strategy to mitigate the conflict of strength and ductility through the coordination of the extrinsic (crack-related) and intrinsic (plasticity-related) mechanisms [7]. For example, well-balanced strength and ductility have been achieved in 7075/2024 [8], 7075/1050 [9] and 6014/5754 [10] Al alloy laminated composites fabricated by roll bonding or accumulative roll bonding. In these Al alloy microlaminates, the deformation texture was largely maintained owing to the effect of geometric confinement, which acted as an intrinsic mechanism enabling a higher plastic deformation capacity via dislocation slip behavior different from that in non-textured grains. To date, there have been only a few research studies on CNT/Al composites with laminated structures [11–13], but none of them reported information concerning textures. Therefore, the present study aims to implement a microlaminated structure with textured matrix layers and aligned interlaminar CNT in CNT/Al–Zn–Mg–Cu alloy composites. It is expected to achieve the full potential of both the high-strength Al alloy matrix and CNT reinforcement at the same time.

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The present study reports a new method of fabricating strong and stiff CNT/Al alloy composites through a microlaminated architecture design and texture control. Microflakes of monolithic 7055Al and CNT/7055Al were first prepared by low-energy ball-milling (LEBM), and were then blended and compacted into CNT/7055Al laminated billets, which facilitated a dominant ⟨1 1 1⟩ fiber texture parallel to the extrusion direction (ED) and an effective load bearing of the aligned interlaminar CNT in the extruded CNT/7055Al laminated composites. CNT/7055Al disordered composites with randomly distributed CNT were also fabricated for comparison.

The laminated and disordered composites were fabricated by flake powder metallurgy (flake PM) and conventional HEBM, respectively, as shown in Figure 1a and b. Multi-walled carbon nanotubes (30–50 nm in diameter and 0.5–2 μm long) were dispersed in ethanol, assisted by an ultrasonic shaker for 30 min to get a gel-like dispersion. The 7055Al alloy powders (spray-forming, with the composition of 8.1 wt.% Zn, 2.2 wt.% Mg, 2.2 wt.% Cu, 0.13 wt.% Zr, 0.12 wt.% Fe, 0.10 wt.% Si) were added in the gel-like CNT–ethanol solution, stirred and then dried in vacuum at 343 K for 2 h to obtain CNT/7055Al spherical powders. Afterwards, they were ball-milled at 423 rpm for 2 h under a flowing argon atmosphere (LEBM-1) to obtain CNT/7055Al flakes. The original powders were also ball-milled in ethanol at 252 rpm for 5 h (LEBM-2) to obtain 7055Al flakes. Ball-milling was carried out under circulating cooling water, and the ball-to-powder weight ratio was 20:1. Finally, CNT/7055Al flakes and 7055Al flakes with a weight ratio of 1:1 were blended for 5 h to obtain the laminated composite powders with 2 vol.% CNT. For comparison, 2 vol.% CNT/7055Al spherical powders were ball-milled for 8 h at 423 rpm under the same conditions to fabricate the disordered composite powders. The powders obtained were cold compacted into a column (Ø40 × 30 mm) and then vacuum hot pressed at 600 °C, under a pressure of 10 MPa, followed by hot extrusion at 440 °C with an extrusion ratio of 25:1. The extrusion rods were solution treated with a three-step program of 455 °C/2 h + 475 °C/1 h + 485 °C/20 min, and afterwards quenched immediately into room-temperature water and aged for 24 h to obtain the laminated and disordered composites.

Tensile specimens 5 mm in diameter and 25 mm in gauge length were machined from the extruded rods, and tensile tests were carried out on a Shimadzu Autograph AG-I (50KN) at an initial strain rate of 3×10^{-4} at room temperature. The Young's modulus was calculated from the slope of the initial linear part of the tensile stress–strain curves. The microstructure of the powders and the composites was characterized by field emission scanning electron microscopy (FE-SEM) on a LEO Supra 55 FE-SEM electron microscope and by high-resolution transmission electron microscopy (HRTEM) on a JEOL 2010F microscope. To examine the texture feature of the composites, electron backscattered diffraction (EBSD) was carried out on a FEI Nova 400. The Raman spectroscopy was collected using the 532 nm line of an Ar^+ laser as the excitation source to validate the structural integrity of CNT. The reaction between CNT and the matrix was evaluated using X-ray diffraction (XRD; Rigaku D/max-2550/PC) with a Cu K_α radiation source.

Figure 1c shows the morphology of CNT/7055Al spherical powders with CNT absorbed on the surface. Figure 1d shows that CNT/7055Al flakes exhibited a two-dimensional morphology with thickness ~6 μm, and the CNT were partially embedded in the matrix. Although the composite flakes have merits such as planar grains and preferred orientation, they are always abandoned because of the densification problem caused by the direct contact of CNT in the neighboring composite flakes [14]. However, in this study, CNT/7055Al flakes served as one of the building blocks, and 7055Al flakes with a larger diameter (Fig. 1e) as another, to provide the best chance to cover the exposed CNT, which supplied a feasible pathway for good consolidation. By contrast, the disordered composite powders exhibited a fragmented morphology with CNT fully embedded in the matrix, as shown in Figure 1f.

Figure 2a shows a typical TEM micrograph of the laminated composites prepared by flake PM. Note that a laminated structure with the thickness of ~2 μm was obtained, which impelled CNT to exhibit an interlaminar distribution along ED. The CNT/7055Al interface is relatively sharp, with an abrupt transition from the

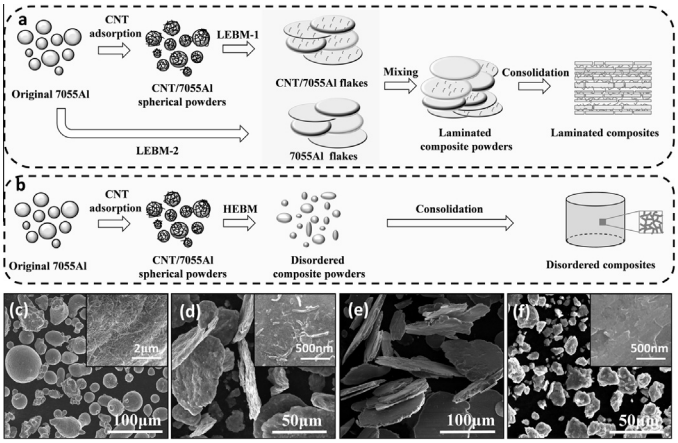

Figure 1. The fabrication process of CNT/7055Al composites: (a) flake PM; (b) HEBM; (c) morphology of CNT/7055Al spherical powders; (d) CNT/7055Al flakes prepared by LEBM-1; (e) 7055Al flakes prepared by LEBM-2; (f) disordered composite powders prepared by HEBM. The inset shows typical surface morphology of the powders.