

# Enhancing Strength and Toughness of Aluminum Laminated Composites through Hybrid Reinforcement using Dispersion Engineering

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## Experimental part:

The atomized spherical pure Al powders (~ 6 µm), amorphous boron, and the pristine MWCNTs (1.5 wt.%, diameter: 30-50 nm, length: 0.3-0.5 µm), synthesized by means of chemical vapor deposition (CVD), were used as raw materials. Generally, CVD-grown CNTs have a few defects and significant amorphous carbon coating on the tube surface [1], which promote the formation of an Al<sub>3</sub>C<sub>4</sub> and Al<sub>3</sub>BC. The spherical Al powders is firstly ball milled at 120 rpm for 15 h to obtain the Al flakes with thickness about 300 nm. To accommodate a uniformly coated CNTs and B particles on the Al flakes, CNTs, Al flakes and B particles were firstly mixed in molar ratio of Al/B/CNT= 3/1/1 in an agate mortar, then high-shear pre-dispersion processed (at 1500 rpm, 30 min). The prepared flaky-shaped powders and 2 wt.% stearic acid were then treated by low-speed ball-milling (LSBM: 10 h, 120 rpm) to mechanically activated nanocomposite building blocks (CNT, B @ Al flakes), followed by high-speed BM (HSBM: 0.5 h, 300 rpm) to fabricate Al@ (CNT, B) composite powder. In order to avoid self-ignition, the ground powders were taken out of the jar after the temperature (T) dropped to around room T (~ 26°C). After degassing at 400 °C, the composites powders were compacted, vacuum sintered (600 °C, 2h) and extruded (420 °C, ratio of 25:1). Then to further consolidate the composite, the plate was firstly cold rolled (reduction of 50%) at room T and subsequent annealing (at 800 °C, 3h, flowing argon) to synthesize in-situ Al<sub>3</sub>BC. The final main composite contained 2.8 wt.% Al<sub>3</sub>BC, ~ 0.45 wt.% CNTs, and ~ 2 wt.% Al<sub>2</sub>O<sub>3</sub>. The CNT content in the composite was assessed through Raman spectroscopy. For comparison, the 1.5 wt.% CNT/Al without B addition was also fabricated with the same procedure.

Microstructural analyses were carried out using high-resolution transmission electron microscopy (HR-TEM, Talos F200X G2), scanning transmission electron microscopy (STEM), and electron backscattered diffraction (EBSD, NordlysMax3, Oxford, SEM equipped with EDAX Velocity Super EBSD detector with a length step of 40 nm). Grain diameters were estimated by measuring the length and width at least 350 grains in EBSD images. The Raman spectroscopy using the 532 nm line of an Ar<sup>+</sup> laser as the excitation source and an electrochemical dissolution method [2] were used to evaluate the structural integrity of CNTs and the reaction between CNTs and Al. XRD analyses were performed using a Rigaku D/Max-2500 X-ray diffractometer with Cu Kα (λ= 0.15406 nm) radiation. The dislocation density of samples was analyzed using [3]

$$\rho = (3\sqrt{2\pi}\langle\varepsilon^2\rangle^{1/2})/(Db)$$

Where  $D$  is the equivalent average grain size,  $\langle \varepsilon^2 \rangle^{1/2}$  is the micro strain,  $\rho$  is the dislocation density,  $b$  is Burgers vector of Al, which is 0.286nm [4]. The as-fabricated composite was sectioned into tensile specimens along the rolling direction (RD), and tensile tests were carried out at a constant strain rate of  $1 \times 10^{-4} \text{ s}^{-1}$  at room T.

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