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Non-equilibrium wetting and capture of boron nitride nanotubes in molten aluminum during plasma spray

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ABSTRACT

Boron Nitride Nanotube (BNNT) is integrated into Aluminum (Al) matrix by plasma spray, involving nonequilibrium solidification. A wet chemistry approach is adopted to obtain Al-BNNT powder feedstock, which is plasma sprayed to form Al-BNNT composite. BNNTs survive the harsh and reactive conditions in plasma and are uniformly distributed in Al matrix. Trace amounts of AlN and AlB₂ formed due to Al-BNNT reactions act as covalently bonded interfacial anchors. Rapid solidification enables efficient capture of BNNT in the molten Al due to viscous drag. BNNT improves the stiffness and hardness of the composite, acting as an effective mechanical reinforcement.

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Boron nitride nanotube (BNNT) has recently emerged as a promising nanomaterial for reinforcing lightweight metals to obtain enhanced strength and stiffness [1–9]. A structural analog of carbon nanotube (CNT), BNNT is characterized by elastic modulus and tensile strength exceeding 1 TPa and 60 GPa, respectively [10,11]. However, in striking contrast to CNT, BNNT can withstand elevated temperatures (700–1000 °C) without structural degradation or oxidation [12–14]. This makes them attractive for developing metal matrix composites (MMC), as most of the metallurgical operations involve elevated temperatures [15–18]. These desirable characteristics of BNNT have been leveraged for developing Aluminum and Titanium-based composites by physical vapor deposition [1,16,17], solid-state sintering [2–8] and solidification routes [9,15]. BNNT addition has been reported to enhance the tensile strength [2,5–7,9], elastic modulus [2,7], compressive strength [3,4] and hardness of the metals [2,5,8], evidencing its suitability as a reinforcing candidate. Solidification is particularly important processing route for the large-scale fabrication of MMC. Equilibrium solidification has been demonstrated as a useful technique for integrating BNNT in Al [15]. Our group reported minimal chemical reactions between Al and BNNT during equilibrium solidification, which results in favorable interfacial wetting of nanotube by molten metal [15].

Due to scalability and rapid processing, plasma spray is one of the most widely accepted solidification routes for developing metallic coatings (surface engineering) and near net shape structures (additive manufacturing) [19–21]. In plasma spray, a high voltage discharge between the anode and the cathode ionizes the gas and accelerates the metal particles to deposit them on a substrate [22]. The layer-by-layer

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deposition of powder particles generates a 3D structure, with splats as its building block [21]. However, some challenges are required to be addressed for plasma spraying of Al-BNNT composite. Firstly, the nanotubes tend to agglomerate and form clusters due to strong van der Waals interactions [23]. Nanotube clusters are stress concentrators and can act as crack nuclei [24]. Therefore, de-agglomeration and uniform dispersion of BNNT in metal powder is critical before plasma spray. Secondly, the rapid heating and cooling involved in plasma spray mean the solidification process is non-equilibrium [25]. The phenomena of wetting and capture of BNNT by the moving solidification front are not known for non-equilibrium rapid solidification. A better understanding of the chemical reactions and physical interactions between Al and BNNT needs to be developed. In this article, a wet chemistry route is proposed to disperse BNNT in Al. Microstructure investigations are performed to study non-equilibrium wetting and capture of BNNT by Al. Finally, the localized nanomechanical properties of the composite are evaluated to assess the reinforcement due to BNNT.

The boron nitride nanotubes used in this study were fabricated by pressurized vapor/condenser method (BNNT P1 Beta, BNNT, LLC., Newport News, Virginia) [26]. These nanotubes were received as clustered balls. Fig. 1a shows as-received BNNT fibrous ball and the SEM image of the long (up to 200 μ m), fine (~10 nm) and densely clustered nanotubes. Due to the very large surface area of the nanotubes, these agglomerates are held together by strong inter-particle forces. For effective reinforcement, it is essential to have a uniform distribution of nanotubes in the metal matrix. A wet chemistry approach employing ionic surfactant was adopted to break nanotube clusters and disperse them [27]. The nanotube clusters and Sodium Dodecylbenzenesulfonate (NaDDBS, Sigma Aldrich, St. Louis, Missouri) surfactant were added to de-ionized water in a beaker. The mixture was bath sonicated for 12 h. Sonication



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Fig. 1. (a) Wet chemistry pathway for dispersion of BNNT in water (electron micrographs before and after dispersion are shown), and (b) SEM micrograph of Al-BNNT composite powder.

assists in breaking the nanotube agglomerates and NaDDBS aids in dispersing the nanotubes in water, such that they do not re-agglomerate [28]. A BNNT concentration of 10 mg/mL and BNNT: NaDDBS ratio of 1:10 were used for obtaining the dispersion. Transmission electron microscopy (TEM) of the aqueous solution showed separated nanotubes, with no agglomeration (Fig. 1a). Ionic surfactant molecule, due to the presence of both polar and apolar groups, adsorbs at the interface of BNNT and water and reduces the surface tension [29]. While the hydrophobic hydrocarbon tail wraps around the nanotube, the hydrophilic polar head protrudes into the water. Hence, the water molecules adhere to the otherwise non-wetting nanotubes. Fig. 1a schematically shows the mechanism of surfactant action. Spherical Al powder (H30, Valimet Inc., USA), with 90% particles having a diameter <58 µm, was added and thoroughly mixed in the aqueous BNNT dispersion using magnetic stirrer. The Al-BNNT mixture was then heated to evaporate the water. Scanning electron microscopy of the composite powder mixture revealed that long nanotubes tend to wrap around the metallic particle surface, without clustering (Fig. 1b). This approach facilitates deagglomeration of nanotubes and preparation of a uniform mixture of Al and BNNT.

The homogeneous Al-BNNT powder mixture was plasma sprayed using an SG 100 gun (Praxair Surface Technologies, Indianapolis, Indiana) on a 6061 Al substrate. The obtained composite coating was ~300 µm in thickness and had 1 vol% of BNNT. The plasma sprayed deposit was sectioned, and the cross-section was metallographically polished. SEM micrograph of the polished cross-section revealed swirly features, which are Al splats (Fig. 2a). Fig. 2b shows the solidified microstructure, comprising of fused metal particles, some degree of porosity and tubular features. The nanotubes in the microstructure act as crack bridges during fracturing of the composite. The high magnification SEM micrograph shows the nanotube is well-embedded and wrapped by Al post-solidification (Fig. 2c). X-ray diffraction (XRD) of the coating detected h-BN, AlN and AlB₂ phases (shown in the inset of Fig. 2d). This suggests the integration of h-BN phase (associated with BNNT) in Al during plasma spray, along with minimal interfacial reactions. It should be noted that there was no prominent peak associated with Al₂O₃



Fig. 2. (a) SEM micrograph of the polished cross-section of plasma sprayed coating, showing swirly splats as the fundamental microstructural building blocks, (b) SEM micrograph showing long nanotubes acting as crack bridges in the microstructure, (c) high magnification SEM micrograph showing a nanotube coated and embedded by Al post-solidification, attesting excellent wetting and capture of BNNT, and (d) X-ray diffraction pattern for Al-BNNT coating.

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