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Interfacial reactions between titanium and boron nitride nanotubes

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ABSTRACT

Titanium-coated boron nitride nanotubes prepared by sputtering method have been heated under different conditions to investigate reaction phases at their interface. It is found that TiB₂ nanocrystals firstly nucleate on the surface of nanotubes at 800 °C for 30 min. By the time 1 h, these nucleated TiB₂ crystals grow larger by consuming inner walls of nanotubes and tend to transform into TiB phase. With an exposure to higher temperatures and longer periods, needle-like TiB whiskers and plate-like TiB₂ remain in the composite.

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Titanium (Ti) is an attractive material because of its high strength to weight ratio compared to aluminium and steel [1,2]. It also possesses high corrosion resistance and good chemical inertness, a desirable feature for structural materials [3,4]. The recent adoption of TiB reinforced titanium matrix composites (TMC) widened the application of titanium composites in automotive industry [5–7]. Various kinds of reinforcements, such as WC, SiC, TiB and CNT, in the form of particles, whiskers, rods and nanotubes have been employed previously in titanium matrix composites [8–12]. Boron nitride nanotubes (BNNTs) have excellent physical, mechanical and chemical properties [13–16]. These attractive properties along with high fracture strain [17], high melting temperature of around 3000 °C, good thermal and chemical stability make them suitable candidates for reinforcements in structural composites [18–21]. BNNTs have been commonly applied to reinforce polymer matrix composites [22–28] and glass matrix composites [29,30]. However, BNNT reinforcement in the metal matrix composite is still in early stages, only a few investigations have been conducted recently for aluminium matrix composites [31–36], while no research has been studied titanium matrix till to date. Incorporation of BNNTs in aluminium matrix greatly improved the tensile and compressive strength by 150% and 250%, respectively [32,37]. It is known that the reactions between reinforcement and matrix material during thermo-mechanical processing of composites impose a significant effect on the final properties of the composite [38,39]. A thin reaction zone (<20 nm) may be beneficial as it enhances the bonding and good for mechanical properties [37]. But a thick reaction layer (>100 nm) at the interface has detrimental effect leading to catastrophic failure [40]. Previously the interfacial reaction

products between Ti and BN particles have been investigated [41–44] by sintering at the temperature range of 1000–1600 °C indicating formation of TiB, TiB₂ and TiN phases at their interfaces. But none of those explained the evolutionary mechanism of interfacial reaction. Therefore, finding the onset conditions for reaction, determination of reaction products and understanding their growth mechanism are of great interest in designing and preparation of BNNT reinforced Ti matrix composites. In this research, titanium-coated BNNTs were fabricated by sputtering process and their interfaces were investigated after different heat treatment conditions to evaluate the potentiality of BNNTs as reinforcement in titanium matrix composites.

Starting BNNTs were synthesized by ball milling and annealing method in a specially designed rotating furnace using ball milled boron powders [45–48]. As-synthesized BNNTs were first ultrasonicated in ethanol and dropped on the surface of a heated silicon wafer. Once the ethanol was evaporated, Ti film was deposited on to the BNNT-covered silicon wafer in vacuum by magnetron sputtering. Ti was deposited at room temperature for 600 s under 100 watt power and the Ar gas was introduced at a flow rate of 30 sccm with a gas pressure of 5 mTorr. The Ti-coated BNNT nanocomposites were then heat-treated in the temperature range of 500–1100 °C for different holding time under high purity Ar atmosphere. X-ray diffractometry (XRD) analyses of the heat treated nanocomposites were conducted using Cu-K α radiation source (0.154056 nm) in a 2 θ range of 10–110°. The morphology of the heat-treated nanocomposites were observed by a Zeiss Supra scanning electron microscope (SEM). Transmission electron microscopy (TEM) of the heat-treated nanocomposite samples were conducted using a JEOL 2100F operated at 200 kV.

The BNNTs synthesized by ball milling and annealing method have high density of nanotubes with a few microns in length and 30–100 nm in diameter (Fig. 1a). TEM image of the nanotubes at the inset

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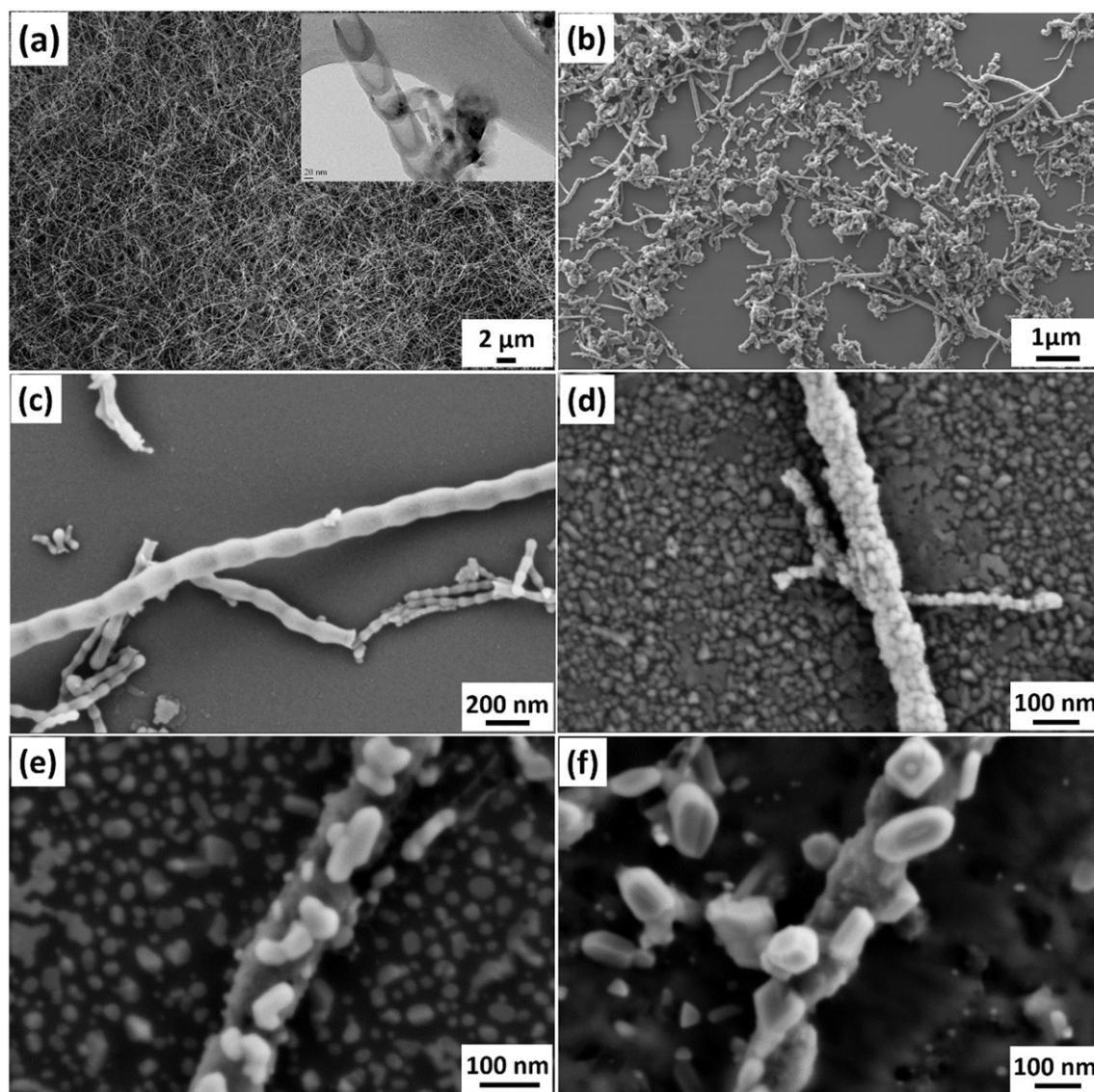


Fig. 1. SEM images of (a) as-synthesized BNNTs and (b) Ti coated BNNTs heat treated at (c) 800 °C for 10 min (d) 800 °C for 30 min (e) 800 °C for 1 h (f) 900 °C for 1 h.

shows bamboo-like structures featuring knots and joints stacked on each other. Fig. 1b shows SEM image of Ti-coated BNNTs without heat treatment. Due to thin coating of Ti, the hollow conical section inside the knots became invisible. The thickness of Ti coating was estimated ~10 nm and only top surfaces of the nanotubes were coated while underneath remains uncoated. The knots and joints of nanotubes are still clearly visible and no structural change of nanotube was observed after heat-treatment at 800 °C for 10 min (Fig. 1c), indicating that the nanotubes are stable at these high temperatures with titanium. However, a disturbance on the nanotube structure can be observed after they were heated at 800 °C for 30 min and 1 h (Fig. 1d and e). After 30 min of heating, some ultrafine particles were grown on the outside shell of the nanotubes at different locations and by the time of 1 h these particles are grown into bigger sizes. It can be assumed that the outer portion of nanotube which was in direct contact with Ti had been reacted to form these particles. This is more evident by observing the region of agglomerated nanotubes where unreacted BNNTs were seen underneath the reacted ones as Ti coating could not cover those nanotubes homogeneously. The formation of Ti grains at this temperature are also conspicuous on the substrate. The reaction was more dramatic for the sample heated at 900 °C for 1 h (Fig. 1f), where the reaction phase gradually consumed the inner section of nanotubes and grew into a plate-like

morphology mostly elongated and/or hexagonal prism like shape. Despite the formation of reaction products at different locations on nanotube surfaces, the rest of the bamboo structured nanotubes were still intact.

In order to identify the reaction phases, transmission electron microscopy (TEM) of the heated nanocomposites were conducted (Fig. 2a). After heat-treated at 800 °C for 30 min, nucleation of reaction phases took place at different locations on the surface of nanotubes and they appear as dark spots in the bright field image. However, most of the nucleated reaction phases were still in the premature state which had not grown up. A HRTEM image from such reaction product shows slightly elongated shape with 41 nm long and 23 nm wide (Fig. 2b). No lattice disturbance could be observed on the nanotube walls as a result of heat treatment. Lattice spacing (0.327 nm) measured from the HRTEM image (inset in Fig. 2b) of this nanocrystal corresponds (001) plane for a hexagonal TiB_2 crystal structure. The morphology, growth and distribution of reaction phases are more obvious in the TEM image of the nanocomposite samples heat-treated at 800 °C for 1 h (Fig. 2c). It shows that TiB_2 crystals were nucleated just on the surfaces of nanotube and then grown radially inside the nanotube by consuming the inner portion of nanotube. The arrow marks in the image represents nucleation of TiB_2 crystals on the reverse side of nanotube surface. A

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