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High tensile-strength and ductile titanium matrix composites strengthened by TiB nanowires

Liqing Huang^a, Lihua Wang^a, Ma Qian^{a,b,*}, Jin Zou^{a,c,**}^a School of Mechanical and Mining Engineering, The University of Queensland, Brisbane, QLD 4072, Australia^b Centre for Additive Manufacturing, School of Engineering, RMIT University, Melbourne, VIC 3000, Australia^c Centre for Microscopy and Microanalysis, The University of Queensland, Brisbane, QLD 4072, Australia

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ABSTRACT

A three-dimensional network-woven architecture made of TiB nanowires has been designed and realized in the matrix of a Ti6Al4V alloy. The architecturally nanostructured design was achieved by dispersing nanoparticles of B₄C or B onto the surfaces of spherical Ti6Al4V powder particles via mechanical mixing and subsequent consolidation by spark plasma sintering. The as-sintered nanostructured Ti6Al4V-TiB composites demonstrated excellent tensile strengths and ductility that are required for critical applications. The in situ formed TiB nanowires with aspect ratios up to 300 contributed to the high tensile strengths while the architectural design of the TiB nanowires ensured the good tensile ductility.

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Designing and developing metallic materials with both high tensile strengths and high tensile ductility is a constant pursuit of the materials research community [1]. Composite strengthening is an important mechanism of strengthening for metals. The Holy Grail for the design and fabrication of metal matrix composites (MMCs) is the achievement of both high tensile strengths and high tensile ductility, particularly by a cost-effective or low-cost fabrication approach. However, for titanium (Ti) MMCs, achieving high tensile strengths through the introduction of a large volume fraction of (e.g., >10%) ceramic reinforcements, which are often micrometre-sized, usually results in poor tensile ductility (e.g., <5% in most cases) [2–4]. This shortcoming has long hindered the further development of Ti MMCs for structural applications.

A potential solution to overcoming this strength-ductility paradox is the employment of nano-scaled high-aspect-ratio ceramics such as nanowires [5], nanotubes [6], or nanowhiskers [7]. Such reinforcements have shown a potent strengthening effect for metals due to their high strength as well as their high load-transfer efficiency from the matrix, both of which are related to their nano-diameters and high aspect ratios. Hence they offer the advantage of achieving significant strengthening with just a small presence, which can avoid the excessive introduction of brittle ceramic reinforcements. In addition, ceramic nanowires with high aspect ratios can be bent to a greater extent than

their other forms without abrupt fracture, or they can even exhibit limited plasticity, which is desired for the development of strong and ductile MMCs [8,9]. However, it has been significantly challenging to disperse nanoscaled reinforcements into a metal matrix by either mechanical or chemical means, both of which can lead to undesired contamination [6,10]. Recently, Lu [1] suggested that multiscale hierarchical structures may provide a promising option for improving the overall properties of metallic materials. Huang et al. [11] showed that a tailor-designed inhomogeneous distribution of reinforcements could mitigate the low tensile-ductility challenge in the fabrication of high-strength MMCs.

Owing to the increasingly demanding lightweight applications, it is desirable that the tensile strengths of Ti alloys can be further improved while maintaining sufficient (>6%) or good (>10%) tensile ductility [12]. Herein, we report an innovative development that has largely overcome this tensile-ductility challenge for the development of high-strength Ti MMCs. This is achieved via a unique nanostructured architectural design and its realization by a powder metallurgy process, i.e., spark plasma sintering (SPS). This new design together with the use of SPS ensures in situ formation of high-aspect-ratio TiB nanowires (TiB_{nws}) and their existence as a 3D network-woven structure in the Ti matrix, where TiB is chosen because it is most suitable for reinforcing Ti [13].

Spherical Ti6Al4V (in wt%) particles (40–70 μm, 0.15 wt%O and 0.17 wt%Fe) were mixed with nanoparticles of B₄C (<50 nm) or B (<100 nm) in a hardened steel jar under an argon atmosphere with a steel-ball-to-powder ratio of 2:1 at 150 rpm for 4 h. The purpose was to disperse the nanoparticles onto the surfaces of the Ti6Al4V particles. SPS was carried

* Corresponding author.

** Correspondence to: J. Zou, School of Mechanical and Mining Engineering, The University of Queensland, Brisbane, QLD 4072, Australia.

E-mail addresses: ma.qian@rmit.edu.au (M. Qian), j.zou@uq.edu.au (J. Zou).

out at temperatures between 1000 and 1100 °C for 8 min under 30 MPa (heating rate: 60 °C/min). Samples for characterisation were cut from as-sintered discs. As-polished samples were subjected to deep etching with a solution made of purified water, nitric acid, and hydrofluoric acid in a mole ratio of 85:10:4. Microstructures were characterized using SEM (JEOL JSM 7001F) and TEM (Philips Tecnai F20 FEG-TEM). The O and N contents were determined using a TCH-600H/N/O analyser. Rectangular cuboidal tensile samples with a gauge length of 8 mm and a cross-section of 2.2 mm × 1.5 mm were machined from as-sintered discs. Tensile testing was performed on an Instron-5584 at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ with an extensometer.

Fig. 1a shows the morphology and distribution of the TiB_{nws} observed in the as-sintered $\text{Ti6Al4V-5 vol\%}(\text{TiB} + \text{TiC})$ composite. A cell-like microstructure was formed with the cell size being similar to that of the Ti6Al4V powder, which suggests that the cell boundaries are the original particle-particle interfaces (with some B_4C nanoparticles). Fig. 1b is a SEM image taken from a typical cell and shows that each cell contains a high density of nanowires. A zoom-in SEM image (Fig. 1c) shows that the diameters of these nanowires range from <50 nm to ~300 nm and their lengths can be up to the entire cell size. A focused view of a typical cell boundary is shown Fig. 1d, in which a higher density of interlocked nanowires is observed, together with some fine particles (1–3 μm) scattered along the boundary, i.e., forming a second form of reinforcements in the matrix. Similar morphology and distribution of TiB nanowires were also observed in the Ti6Al4V composites reinforced with only TiB whiskers in this study.

To identify the nanowires observed in the Ti matrix, characterization by transmission electron microscopy (TEM) was performed. Fig. 2a is a TEM image taken near a cell boundary, which contains both nanowires and particles. Our selected area electron diffraction (SAED) investigation indicates that the particles are TiC , and an example is shown as inset. Fig. 2b is a zoom-in TEM image taken from the circled region in Fig. 2a. It shows that the nanowire has a hexagonal cross-section. The SAD pattern obtained from this region is shown in Fig. 2c, which

confirms that this region contains TiB (nanowire) and $\alpha\text{-Ti}$ (matrix). The nanowires are thus confirmed to be TiB_{nws} . The TiB_{nws} and TiC phases were formed through the reaction between the B_4C and the Ti matrix [14]. TEM characterization revealed that the TiB_{nws} observed grew along the [010] direction of TiB , and the orientation relationship between the TiB_{nws} and the $\alpha\text{-Ti}$ matrix is identified as: $(101)\text{TiB} // (0001)\alpha\text{-Ti}$ and $[010]\text{TiB} // [11\text{--}20]\alpha\text{-Ti}$, which is consistent with literature [15]. In fact, as shown in Fig. 1, each cell contains pockets of parallel TiB_{nws} , indicating that each pocket of parallel TiB_{nws} have grown along the same preferred direction. Since each spherical particle of Ti6Al4V was coated reasonably homogeneously with B_4C nanoparticles, as designed, a 3D network-woven structure of TiB_{nws} formed in the matrix of the composite as shown in Fig. 1.

Fig. 3a shows representative tensile strength-strain curves of the as-sintered $\text{Ti6Al4V-TiB}(\text{+TiC})$ MMCs where the detailed tensile property data are listed in Table 1. The as-sintered Ti6Al4V matrix alloy achieved tensile strength of $944 \pm 7 \text{ MPa}$ and strain-to-fracture of $13.0 \pm 0.8\%$. With 2.5 vol% ($\text{TiB}_{\text{nws}} + \text{TiC}$), the tensile strength was increased to $1184 \pm 10 \text{ MPa}$ while the strain-to-fracture reached beyond 10% (Fig. 3a). Further increasing the reinforcements to 5 vol% leads to the tensile strength being increased to $1267 \pm 14 \text{ MPa}$ while an average strain-to-fracture of $6.1 \pm 0.6\%$ was retained. These properties reached those of the Ti10V2Fe3Al (1240 MPa and 6.0%) in the forged and STA condition (STA: solution treated, water quenched and aged) [16], which is commonly used in aircraft landing gear systems [12]. Fig. 3b compares the tensile properties achieved in this study with those reported for Ti6Al4V -based MMCs (fabricated by powder metallurgy or casting, without thermo-mechanical processing) [13,14,17–25]. The distinct advantages as represented by the borderline shown in Fig. 3b highlight the critical importance of applying the nanostructured architectural design concept to fabricate strong and ductile Ti MMCs. Also included are the minimum tensile strength (1137 MPa) and elongation (10%) for mill-annealed Ti6Al4V in the STA condition for samples thinner than 12.7 mm [26]. Mill-annealed Ti6Al4V in the STA condition offers the

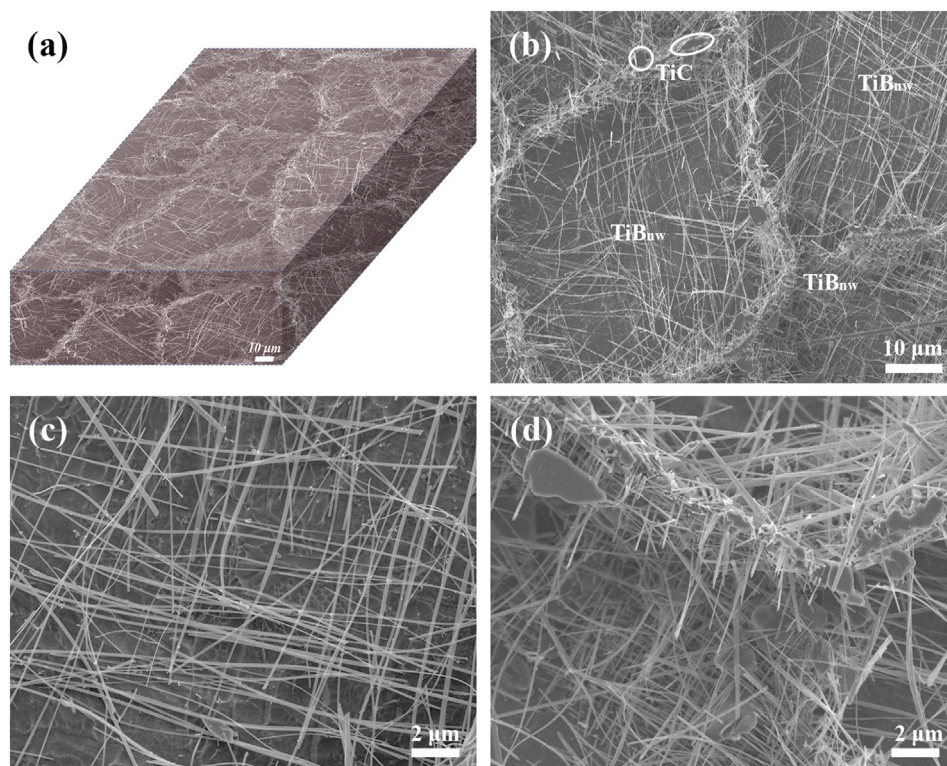


Fig. 1. SEM images showing the microstructures of a deep-etched as-sintered $\text{Ti6Al4V-5 vol\%}(\text{TiB} + \text{TiC})$ sample. (a) A pseudo-3D presentation of the as-sintered microstructure, (b) a focused view containing a typical cell, (c) nanowires inside a cell, and (d) a focused view of a cell boundary. TiB and TiC form during sintering through chemical reactions of $\text{Ti} + \text{B} \rightarrow \text{TiB}$ and $5\text{Ti} + \text{B}_4\text{C} \rightarrow 4\text{TiB} + \text{TiC}$.

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