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Stacking faults formation mechanism of *in situ* synthesized TiB whiskers

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In situ TiB whiskers have a hexagonal shape in the transverse section and grow along the $[0\,1\,0]_{TiB}$ direction. The crystallographic planes of the TiB whiskers in the transverse section are always (1 0 0), (1 0 1) and $(1\,0\,\bar{1})$. The stacking faults in TiB are typically with a stacking fault plane of $(1\,0\,0)_{TiB}$. The locations of boron atoms and the lattice mismatch energy between TiB and Ti matrix play key roles in the formation of stacking faults.

Keywords: *In situ*; TiB; Whisker; Stacking faults

TiB has been extensively used as a reinforcement for titanium metal matrix composites (MMCs) due to its excellent chemical stability and high stiffness [1,2]. In recent years, *in situ* reaction synthesis techniques have been used to fabricate Ti MMCs which have better properties, and these overcome all the disadvantages of conventional fabrication techniques [3–5]. The *in situ* reaction synthesis techniques enable fine and uniformly distributed TiB reinforcements to be produced, together with clean and well-bonded reinforcement-matrix interfaces [4–6]. The microstructures of *in situ* TiB whiskers made via various synthesis methods have been widely investigated by many researchers [1,5–10]. Different morphological types of TiB and stacking faults structure have been investigated and analyzed by Kooi et al. [6–8]. One interesting thing is that stacking faults have been observed in the *in situ* TiB crystals, whenever they were reaction-synthesized by laser cladding, common casting, rapid solidification or spark plasma sintering [6–11]. It was reported that the stacking faults formed in the $(1\,0\,0)_{TiB}$ plane and throughout the entire width of a fine TiB needle [6]. The *in situ* TiB nucleates and grows through the diffusion of boron atoms during the synthesis processing. However, the formation mechanism of stacking faults in *in situ* TiB still needs further investigation. Therefore, it is of significance to explore the growth mechanism of *in situ* TiB. In this paper, the formation mechanism of stacking faults is proposed and confirmed by high resolution electron microscopy (HREM) observation and crystal structural analysis.

Commercial Ti, Fe–65 wt.%Mo and TiB_2 powders were used as starting materials. The volume fraction of the *in situ* reaction-synthesized TiB whiskers in Ti matrix composite is 10%. The powder mixture was first ball-milled at a rotational speed of 500 rpm for 10 h in an argon atmosphere and then sintered in vacuum at 1000 °C for 5 min with a pressure of 20 MPa. Our previous studies showed that the *in situ* reaction between TiB_2 and Ti can occur and form *in situ* TiB whiskers when spark plasma sintering is carried out at 1000 °C for 5 min [4,10].

The samples for scanning electron microscopy (SEM) observations were prepared by standard metallographic methods. A deep etching process was performed on the polished samples with a solution of water, nitric acid and hydrofluoric acid in a proportion of 80:15:5. Morphology of *in situ* synthesized TiB whiskers was examined by a JSM-5600LV type SEM. Samples for transmission electron microscopy (TEM) and HREM observations were first polished to a thickness of 20 μm using SiC abrasive papers, and then punched into 3 mm diameter discs. The further thinning of the disc samples was performed by argon ion milling with an

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incident angle of 10° until perforation occurred. Microstructural observations were carried out using a Philips CM12 type TEM at 120 kV and a JEM-2010F type HREM at 200 kV, respectively.

The *in situ* TiB can be reaction-synthesized by spark plasma sintering at temperatures of 800–1200 °C as discussed in our previous paper [10]. Figure 1 shows the typical SEM morphology of deep-etched *in situ* synthesized TiB reinforced Ti matrix composite. The *in situ* TiB is whisker shaped and distributed uniformly in the titanium matrix. The transverse section of TiB whisker always exhibits a hexagonal shape as shown in Figure 2(a). Figure 2(b) shows the corresponding SAED pattern of TiB, the indexed result indicates that the crystallographic planes of the hexagonal transverse section are always (100), (101) and $(10\bar{1})$ planes. The *in situ* synthesized TiB whisker exhibits a growth orientation in the $[010]_{TiB}$ direction. Moreover, the planar stacking faults in $(100)_{TiB}$ plane are also observed in TiB whisker by TEM observations, as shown in Figure 2(a). These observations are in good agreement with previous studies [6–9,11]. Moreover, as shown in Figure 2(b), bright streaks of TiB are found in the SAED pattern. The streaking always occurs in the direction of $(100)_{TiB}$, which also denotes that the stacking faults are in the $(100)_{TiB}$ plane only.

Figure 3 shows the HREM image of perfect TiB crystal along the $[010]_{TiB}$ direction. The distances of two bright dots along $[100]_{TiB}$ and $[001]_{TiB}$ directions are

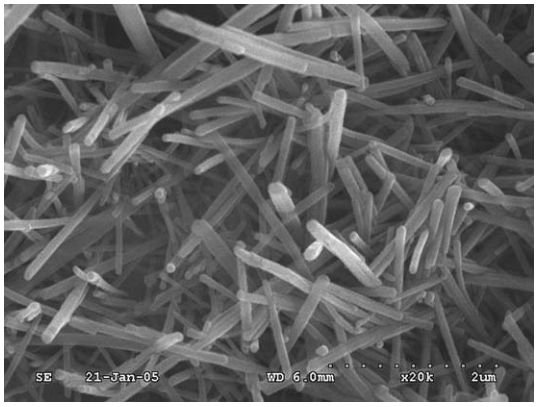

Figure 1. SEM morphology of the *in situ* synthesized TiB whiskers sintered at 1000 °C.

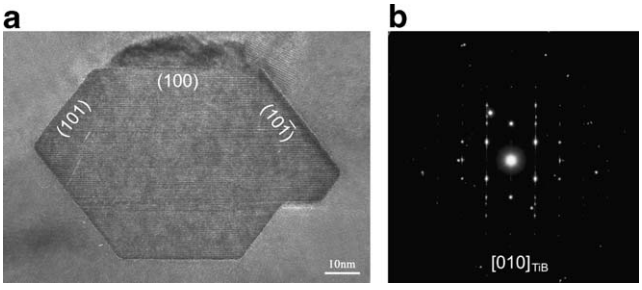

Figure 2. TEM image and SAED pattern of TiB whisker along $[010]_{TiB}$ direction: (a) TEM image showing the hexagonal cross section of TiB, (b) the SAED pattern along $[010]_{TiB}$ zone axis.

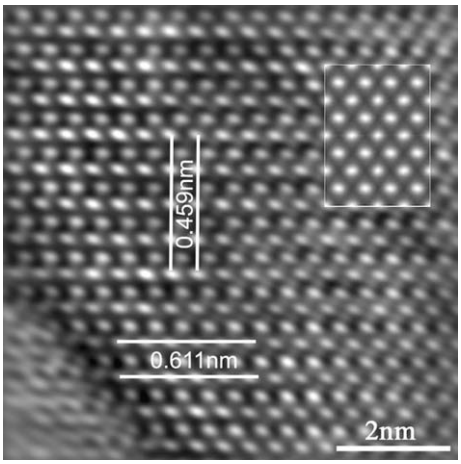

Figure 3. HREM and simulated image (in the rectangular frame) of perfect TiB along $[010]_{TiB}$ direction.

0.611 nm and 0.459 nm, respectively, which is in good agreement with the inter-planar distances of $(100)_{TiB}$ ($d_{(100)} = 0.612$ nm) and $(001)_{TiB}$ ($d_{(001)} = 0.456$ nm) planes. The simulated HREM image of perfect TiB with the B27 crystal structure is shown as an inset of Figure 3. The bright dots in the simulated image represent the Ti atoms in TiB crystal. Clearly, the arrangement of these dots agrees with those dots in the HREM image. So, in the HREM image, the bright dots represent the Ti atomic stacking sequence of $(010)_{TiB}$ plane in the TiB crystal structure. The characteristics of the stacking faults can be readily analyzed by comparing the change of Ti atoms in the HREM image. Figure 4 shows the HREM image of stacking faults in TiB, which shows the atomic arrangement in stacking period of Ti atoms along the $[100]_{TiB}$ direction. These stacking faults occur in 1–2 Ti atoms planes along the $[100]_{TiB}$ direction.

The TiB cell is the B27 structure with lattice parameters of $a = 0.612$ nm, $b = 0.306$ nm, and $c = 0.456$ nm. There are two Ti–B atomic layers ($b = 1/4$ layer and $b = 3/4$ layer) along the [010] axis of the B27 cell and each layer consists of two Ti and two B atoms. In the 1/4 layer of the TiB cell, two Ti atoms

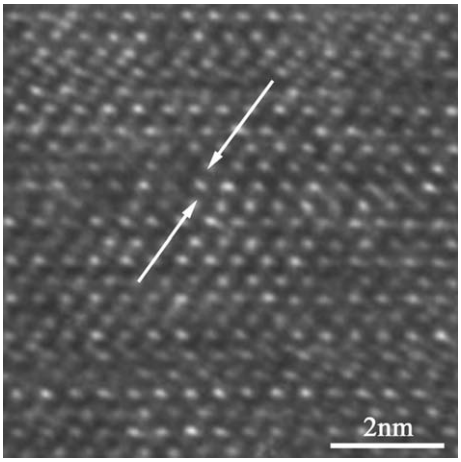

Figure 4. HREM image of stacking faults in TiB whisker along $[010]_{TiB}$ direction.