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Highly textured Ti_2AlN ceramic prepared via thermal explosion followed by edge-free spark plasma sintering

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ABSTRACT

Highly textured Ti_2AlN ceramic was successfully fabricated by edge-free spark plasma sintering (EFSPS) of Ti_2AlN discs synthesized by thermal explosion (TE). The orientation degree of as-sintered Ti_2AlN ceramic was evaluated by X-ray diffraction and electron backscattered diffraction. It was found that the preferred orientation of Ti_2AlN grains paralleled to the SPS-loading direction was along the *c*-axis and the Lotgering orientation factor on the textured top surface was as high as $f_{(00l)} = 0.80$. Due to the highly textured microstructure, the obtained Ti_2AlN ceramic showed anisotropic mechanical and physical properties. The results highlight the advantages of EFSPS combined with TE technique.

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Textured ceramics in which the grain orientation is consistent, have been attracted great attention due to the unique physical and mechanical properties [1–2]. For MAX phases (M is the transition metal, A is A group element and X is C or N), owning to the hexagonal crystal structure and anisotropic properties along the *c*- and *a*- or *b*- axis [3], it is expected that textured MAX phases ceramics may show superior physical and mechanical properties as compared to their randomly oriented form. However, over the past decade, most works have focused on the bulk MAX phases without texture since it is difficult to prepare textured ceramics by conventional sintering methods. Considering the excellent physical and mechanical properties of bulk MAX phases [4–5], there is a great motivation to fabricate textured MAX phases which may be beneficial not only to understand the intrinsic anisotropic characteristics but also to broaden their practical applications.

Although more than seventy MAX phases have been explored, only a few textured MAX phases have been achieved. For instance, Hu et al. [6–7] fabricated highly textured Nb_4AlC_3 and Ti_3SiC_2 ceramics by strong magnetic field alignment method and found that both strength and toughness have been improved. Zhang et al. [8] also prepared highly textured Ti_3AlC_2 ceramic with high anisotropic physical and mechanical properties by the same technique. Nevertheless, the secondary oxide phase was unavoidable in their work. Edge-free spark plasma sintering (EFSPS) is another effective way to prepare textured MAX phases. Recently, Lapauw et al. [9] successfully fabricated textured Ti_3SiC_2, Ti_3AlC_2 and Ti_2AlC ceramics by EFSPS. However, the orientation factors of these textured ceramics are as low as 0.5. In addition, Duan et al. [10] prepared textured Cr_2AlC ceramic by SPS of fine Cr_2AlC powders but the orientation factor is still low. Therefore, preparing highly textured MAX phase ceramic with high purity still remains a challenge.

Ti_2AlN is a typical MAX phase and has been widely investigated [11–13]. In recent work, Liu et al. [14] found preferred growth behavior along the *c*-axis during microwave sintering. However, highly textured Ti_2AlN ceramic has never been reported. In our previous work, Ti_2AlN ceramic with small particle size and high reactivity has been synthesized rapidly by thermal explosion (TE) [15]. The purpose of present work is to fabricate highly textured Ti_2AlN ceramic combining TE and EFSPS technique. The anisotropic microstructure and properties are also investigated.

The schematic representation for the preparation of highly textured Ti_2AlN ceramic is given in Fig. 1. Firstly, porous Ti_2AlN ceramic was synthesized by the thermal explosion of Ti/Al/TiN powder mixture (Fig.1(a)). The detailed process has been given elsewhere [15]. Briefly, the Ti, Al, and TiN powders with a molar ratio of 1.1:1.1:1 were mixed, dried and cold-pressed into cylindrical disc with a diameter of 12 mm. Then the disc was directly put into an air furnace which had been preheated to 700 °C. After holding for 2 min, the reacted disc was directly taken out and cooled down freely to room temperature. Secondly, the as-synthesized Ti_2AlN ceramic was grinded by sandpaper to remove the oxidized surface and then was put into a graphite mold with an inner diameter of 16 mm for EFSPS (Fig. 1(b)). The furnace temperature was

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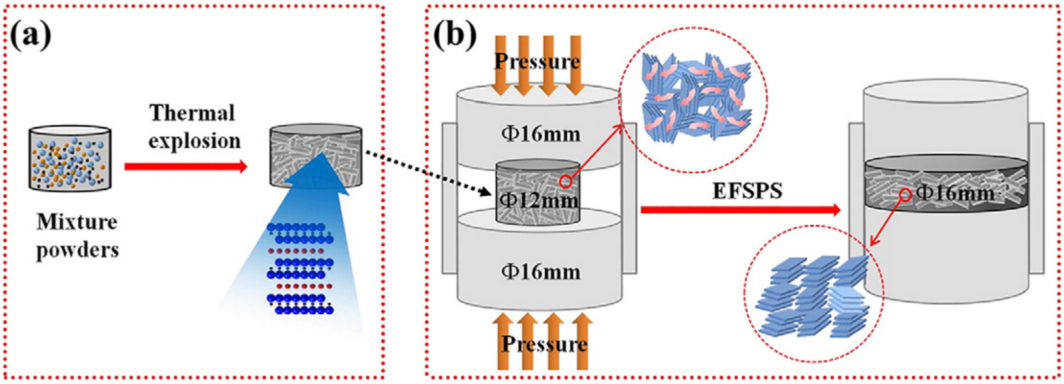

Fig. 1. Schematic representation for the preparation of highly textured Ti_2AlN ceramic. (a) thermal explosion, (b) EFSPS.

increased to 1100 °C with a heating rate of 100 °C min^{-1}. When the as-synthesized Ti_2AlN ceramic started to shrink at 950 °C, the uniaxial pressure of 50 MPa was slowly applied within 1 min. After kept for 5 min at 1100 °C in a vacuum atmosphere, the textured Ti_2AlN ceramic was obtained.

The textured top surface (TTS) and textured cross surface (TCS), as well as the top surface of bulk Ti_2AlN before texturing were examined by X-ray diffraction (XRD) with CuKα radiation over a 2θ range of 10–70° at a step size of 0.02°. The relative contents of different phases were calculated by X'Pert HighScore Plus. The Lotgering orientation factor (f_L) was determined as follows: $f_L = (P - P_0) / (1 - P_0)$. For *c*-axis orientation, P and P_0 were obtained from the ratio of $\sum(00l)$ / $\sum(hkl)$. Here, P was from the textured Ti_2AlN sample and P_0 was from the XRD date (JCPDS card No. 65-3496). $\sum(00l)$ and $\sum(hkl)$ were the sums of peak intensities corresponding to the (00*l*) and (*hkl*) planes, respectively. Furthermore, the grain orientation maps were constructed by electron backscattered diffraction (EBSD, JSM-6700F). Before EBSD, the TTS and TCS were polished by a vibratory polisher to eliminate the surface stress. The density of samples after TE and EFSPS was measured by the Archimedes' method. The Vickers hardness on polished TTS and TCS were determined by a hardness tester at the loads of 9.8–98 N with a fixed contact time of 15 s and the indentation area was observed by scanning electron microscopy (SEM, S4800). The Young's modulus was evaluated by an ultrasonic equipment (UMS-100, TECLAB (CHINA) Ltd). The electrical conductivity was measured using the Seebeck & Electric Resistivity Unit (LSR-3, LINSEIS, Germany) and the thermal conductivity was measured by a Xenon flash apparatus (LFA 447 Nanoflash, NETZSCH, Germany).

Fig. 2 shows the XRD patterns of Ti_2AlN ceramic after thermal explosion and EFSPS. It is obvious that Ti_2AlN has been synthesized as a major phase with small amounts of TiN as secondary phase in the samples after thermal explosion (Fig. 2(a)). Other phase except Ti_2AlN and TiN is not observed after EFSPS (Fig. 2(b) and Fig. 2(c)), indicating that the decomposition of Ti_2AlN has not been occurred during EFSPS. As shown in XRD pattern of TTS (perpendicular to the *c*-axis, Fig.2(c)), the diffraction intensity of {00*l*} planes become prominent while the peak intensity of others is inapparent. But on the TCS (parallel to the *c*-axis, Fig. 2(b)), the characteristic peak intensity of {00*l*} planes has almost completely disappeared. This indicates that the Ti_2AlN grains show a highly orientation along *c*-axis after EFSPS. In order to describe the texture degree, the f_L was calculated. The results showed that $f_{(00l)}$ reached up to 0.80 for {00*l*} planes while $f_{(hk0)}$ was only 0.33 for {*hk*0} planes. The difference between $f_{(00l)}$ and $f_{(hk0)}$ further confirmed that the orientation of Ti_2AlN grains was along *c*-axis. It should be noted that the relative peak intensity of TiN also changed after EFSPS although the intensity is weak. As enlarged at the 2θ of 34–45° in Fig. 2, the diffraction peaks corresponding to the (111) and (200) planes of TiN phase are present on the TE bulk and TCS. However, the peak of (200) plane is disappeared and only the peak of (111) plane can be seen on TTS. This indicates that the orientation of residual TiN phase has also been occurred since the orientation relationships between Ti_2AlN and TiN are (0001)Ti_2AlN// (111)TiN at the TiN/Ti_2AlN interface [12].

In order to reveal the orientation distribution of Ti_2AlN grains, EBSD was conducted on both TTS and TCS. As shown in Fig. 3(a) and Fig. 3(b), different color indicates different grain orientation on TTS and TCS. It is apparent that on TTS, the exposed crystal planes are almost (000*l*) planes. While on TCS, almost no (000*l*) planes are exposed. Instead, $(01\bar{1}0)$ and $(\bar{1}2\bar{1}0)$ planes are the main exposed crystal planes. Fig. 3(c) and Fig. 3(d) gives the inverse pole figure according to Fig. 3(a) and Fig. 3(b). From inverse pole figure it can be deduced that the orientation of Ti_2AlN grains is along the *c*-axis which is perpendicular to the top surface. Furthermore, it can also be revealed that the orientation difference for almost Ti_2AlN grains along *c*-axis is less than 10°. The results from EBSD analysis are well agreement with the orientation factor calculated from XRD data.

The orientation factor of textured Ti_2AlN is far higher than other textured MAX phases prepared by EFSPS. The main reason may be attributed to the mechanical oriented deformation. Since the Ti—Al bonding is weaker than Ti—N bonding in Ti_2AlN lattice, the Ti_2N atom layers in Ti_2AlN grains tend to slip along Al atom layers when a high shear stress is applied. Although the uniaxial force is used during EFSPS, the grains in Ti_2AlN bulk actually suffer a complicated stress in the view of microscale. For the grains in which the *c*-axis is not paralleled to the direction of external force, the shear stress along Al atom layers becomes prominent. Therefore, when slipping motion is activated at high temperature,

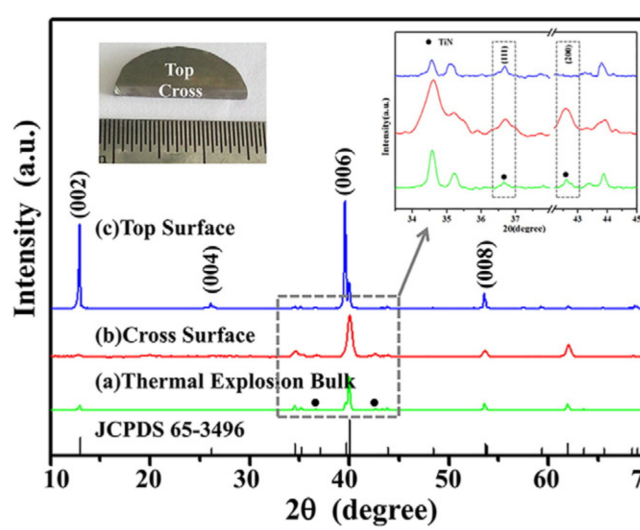

Fig. 2. XRD patterns of Ti_2AlN ceramics. (a)Ti_2AlN bulk after thermal explosion. (b) TCS and (c) TTS of Ti_2AlN after EFSPS.

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