High-Performance TiB<sub>ω</sub>/Ti Composite Prepared by Hot Extrusion

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Abstract: The low ductility of the titanium matrix composites (TMCs) at room temperature has become a significant obstacle for the engineering applications. Therefore, to achieve excellent mechanical properties in TiB<sub>ω</sub>/Ti composites, a processing route that hot extrusion on as-sintered TiB<sub>ω</sub>/Ti composites was proposed. Comparing with the microstructure of as-extruded TiB<sub>ω</sub>/Ti composite and as-sintered TiB<sub>ω</sub>/Ti composite, it revealed that the alignment of TiB whiskers in composite changed from randomness to along the extrusion direction with the hot extrusion completing. Moreover, according to the mechanical property test results, the mechanical property of as-extruded TiB<sub>ω</sub>/Ti composites enhanced at the same time (tensile strength of 789 MPa and plastic strain of 1.5% for as-sintered TiB<sub>ω</sub>/Ti composites; tensile strength of 940 MPa and plastic strain of 18.1% for as-extruded TiB<sub>ω</sub>/Ti composites). The obtained results provide a promising fabrication route for developing high-performance TiB<sub>ω</sub>/Ti composites suitable for industrial applications.

1. Introduction
Titanium and its alloys are widely used in aerospace, automotive, chemical, military, energy and other fields because of their low density, excellent corrosion resistance, and high specific strength[1-2]. However, the majority of titanium alloys exhibit rather low tribotechnical properties, which can be improved by adding reinforcements to titanium and its alloys to fabricate the titanium matrix composites[3]. Among kinds of reinforcing phase, TiB is regarded as the perfect reinforcement for its high hardness, high thermodynamic stability and approximate thermal expansion coefficient with the titanium matrix[4-5].

For an ideal reinforcing technique, in-situ reinforcements were formed during sintering or casting are promising methods. Li H et al [6] indicated that the titanium matrix composites prepared by in-situ techniques show excellent mechanical properties due to their uniform distributed reinforcement as well as the good combination with the matrix. However, the formation of Kirkendall’s pore mainly due to the huge difference in inter-diffusion rates between B and Ti atoms, which creates excessive vacancies near the in-situ TiB phase during sintering process[7], would greatly deteriorate the ductility of composites.

For eliminating the defects in as-sintered TiB<sub>ω</sub>/Ti composite, a method by carrying out the hot extrusion on as-sintered TiB<sub>ω</sub>/Ti composite was proposed in this study. Surprisingly, after hot extrusion, the relative density of the composites were also promoted to 99.99%, which basically
realized the nearly full density of the material; the mechanical property test results indicated that compared with as-sintered TiB\textsubscript{w}/Ti composite, as-extruded TiB\textsubscript{w}/Ti composite displays excellent mechanical properties of 940 MPa in tensile strength and 18.1\% in elongation, which is more suitable for engineering application.

2. Experiment
High purity TiH\textsubscript{2} powders (~325 mesh) and TiB\textsubscript{2} (~50 nm) powders were used for this study. Firstly, TiH\textsubscript{2} powders were mechanical blended with the 1.71 wt. \% TiB\textsubscript{2} powders (the corresponding volume fraction of in-situ TiB is 5 vol\%). Then, the mixture of powders were compacted into a cylinder shaped green with diameter of 30 mm with a pressure of 750 MPa and a holding time of 30 s at room temperature. Finally, the powder compacts were heated to 1250 °C with a heating rate of 10 °C/min, and then held for 4 hours in vacuum sintering furnace. After vacuum sintering, the as-sintered billet was pre-heated to 1050 °C and kept for 300 s under an argon gas atmosphere before hot extrusion. Then, the billet was extruded by a hydraulic press machine (Bala, China). the extrusion rate was set at 9:1. the final diameter of the extruded rod was 10 mm.

The extruded rod and the sintered rod were machined, the tensile samples exhibits a gauge section of 2 mm in width, 1.5 mm in thickness and 15 mm in length. The tensile mechanical property was test at a strain rate of 5×10^{-4} s\textsuperscript{-1} by a universal standard testing machine, respectively. The strain value was measured by an electronic extensometer. Each sample was tested more than three times to obtain an average result. The microstructure was observed by scanning electron microscope (SEM, FEI Nova Nano SEM 430, US). the samples were prepared using conventional techniques of grinding and mechanical polishing. Finally, the SEM samples were etched by Kroll’s solution (5 ml HF, 10 ml HNO\textsubscript{3}, 85 ml H\textsubscript{2}O) for 80 s. The relative densities of samples were measured by Archmedes’ principle.

3. Results and discussion

3.1. Relative density
The relative density results shows that the relative densities of the as-sintered TiB\textsubscript{w}/Ti composite and as-extruded TiB\textsubscript{w}/Ti composite are 98.78\% and 99.99\%, respectively. The result indicates that the hot extrusion tends to promote the pores in composites closure.

3.2. Microstructural characteristics
Fig. 1(a)-(b) depicts the SEM images of the as-sintered TiB reinforced Ti-based composites and as-extruded TiB reinforced Ti-based composites. For as-sintered TiB\textsubscript{w}/Ti composite, average aspect ratio of TiB whiskers is 20.36, which distributed uniformly randomly along the grain boundary and inside grain interior. Interestingly, the microstructure characteristics of as-sintered TiB\textsubscript{w}/Ti composite have changed after hot extrusion. Except the grain refinement, the TiB whiskers of as-extruded TiB\textsubscript{w}/Ti composite have aligned along the extrusion direction instead of distributing randomly. The main reason is that the as-extruded composite was subjected to multi-dimensional stress, which promote the rotation of TiB whiskers during the hot extrusion. In addition, TiB were broken into short segments during the extrusion, which led to a decreasing in their aspect ratio, the specific index is 8.80. The reason is that the ceramic phase TiB only twisted or broke during plastic deformation of the extrusion process[8].
3.3. Mechanical properties

Fig. 2 is the engineering stress-strain curves of as-sintered TiB\(_w\)/Ti composite and as-extruded TiB\(_w\)/Ti composite. Tab. 1 displays the mechanical performance indexes of as-sintered TiB\(_w\)/Ti composite and as-extruded TiB\(_w\)/Ti composite. The as-sintered TiB\(_w\)/Ti composite displayed tensile strength of 789 MPa and plastic strain of 1.5%. The as-extruded TiB\(_w\)/Ti composite displayed tensile strength of 940 MPa and plastic strain of 18.1%. It can be seen that the proposed preparation method could effectively enhance the mechanical properties of TiB\(_w\)/Ti composite.

![Fig. 1 SEM of (a) as-sintered TiB\(_w\)/Ti composite; (b) as-extruded TiB\(_w\)/Ti composite](image)

![Fig. 2 The engineering stress-strain curves of as-sintered TiB\(_w\)/Ti composite and as-extruded TiB\(_w\)/Ti composite](image)

| Material                  | Mechanical Properties |
|---------------------------|-----------------------|
|                           | Yield Strength (MPa)  |
|                           | Tensile Strength (MPa) |
|                           | Elongation (%)        |
| As-sintered TiB\(_w\)/Ti composite | 742                  | 789                  | 1.5 |
| As-extruded TiB\(_w\)/Ti composite | 834                  | 940                  | 18.1 |

General speaking, the increasing of ductility tends to accompany with the decrement on strength. Surprisingly, compared with the as-sintered TiB\(_w\)/Ti composite, the TiB\(_w\)/Ti composite fabricated by the method proposed realized synchronous improvement on strength and ductility. The increment on tensile plasticity in the present as-extruded TiB\(_w\)/Ti composite originates from the reduction of large amounts of porosity. The tensile strength increment in the present the as-extruded TiB\(_w\)/Ti composite originates from the grain refinement, the high dislocation density introduced by hot extrusion and the alignment of the TiB whisker along the extrusion direction[9]. The mechanical properties of TiB
reinforced titanium matrix composite fabricated by reported different methods was displayed in Tab. 2. The as-extruded TiB\textsubscript{w}/Ti composite shows a comparable and better combination of strength and ductility than other TMCs reported.

Tab. 2 Summary of mechanical properties indexes of TiB\textsubscript{w} reinforced titanium matrix composites prepared by different preparation method

| Matrix          | Reinforcement | Processing Route | Strength (MPa) | Elongation (%) | Ref. |
|-----------------|---------------|------------------|----------------|----------------|------|
| Ti-4.5Al-6.8Mo-1.5Fe | 5 vol% TiB    | HVC+PM           | 1038           | 2.19           | [10] |
| Ti60            | 3.4 vol% TiB  | RHP+HE           | 1377           | 5              | [9]  |
| Ti60            | 6.8 vol% TiB  | RHP+HE           | 1162           | 1.2            | [9]  |
| TA15            | 2 vol% TiB    | HP               | 1093           | 6.8            | [11] |
| TA15            | 2 vol% TiB    | HP+HR            | 1212           | 13             | [11] |
| TA15            | 5 vol% TiB    | HP               | 1134           | 2.1            | [11] |
| TA15            | 5 vol% TiB    | HP+HR            | 1274           | 3.2            | [11] |

3.4. Fractured morphology

Fig.3 Fractured morphology of (a) as-sintered TiB\textsubscript{w}/Ti composite; (b) as-extruded TiB\textsubscript{w}/Ti composite

Fig.3 displays the fractured morphology of the composite samples after the tensile test. The fractured surface of as-sintered TiB\textsubscript{w}/Ti composite is distributed with a large number of tearing edges and some river patterns, indicating that there were a large number of cracks that propagate along the cleavage plane when as-sintered TiB\textsubscript{w}/Ti composite underwent plastic deformation, which speed up the fracture process of the material. Conversely, the fractured surface of the as-extruded TiB\textsubscript{w}/Ti composite is distributed with a large number of dimples, which indicates that the as-extruded TiB\textsubscript{w}/Ti composite underwent a larger plastic strain during deformation, and exhibits extremely excellent tensile plasticity. Therefore, the preparation proposed could change the fracture mode of TiB\textsubscript{w}/Ti composite and increase ductility of TiB\textsubscript{w}/Ti composite.

4. Conclusion

An efficient process that pressureless sintering accompanied by hot extrusion was adopted to prepare high-performance TiB\textsubscript{w}/Ti composite. Microstructure and mechanical performance at room temperature for the as-extruded TiB\textsubscript{w}/Ti composite were investigated. The main conclusions are as follows:

1) Compared with as-sintered TiB\textsubscript{w}/Ti composite, as-extruded TiB\textsubscript{w}/Ti composite shows higher relative density, which is mainly due to the fact that the hot extrusion could promote pore closure.
After hot extrusion, the microstructure of TiB\textsubscript{w}/Ti composite has transformed, including the alignment of TiB whisker in TiB\textsubscript{w}/Ti composite transforming from randomness to along the extrusion direction and the grain refining.

Compared with as-sintered TiB\textsubscript{w}/Ti composite, the fractured surface of as-extruded TiB\textsubscript{w}/Ti composite displays ductile fracture instead of brittle fracture.

Compared with as-sintered TiB\textsubscript{w}/Ti composite, as-extruded TiB\textsubscript{w}/Ti composite exhibits excellent mechanical properties of 940 MPa in tensile strength and 18.1% in elongation.

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