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Microstructure and mechanical properties of in-situ synthesized TiB whiskers reinforced titanium matrix composites by high-velocity compaction

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ABSTRACT

High-velocity compaction (HVC) technology was applied to press titanium-based blended elemental powders containing boron sources of TiB_2 and LaB_6, and in-situ TiB whiskers reinforced titanium matrix composites (TMCs, TiB/Ti-4.5 Al-6.8 Mo-1.5 Fe) were successfully prepared after vacuum sintering at 1250 °C for 3 h. The effects of forming ability of the blended powders by HVC and TiB volume fraction (5–20%) on microstructure and mechanical properties of composites were studied. The results showed that the blended powders could be pressed by HVC into green compacts with relative density no less than 94%. While the hardness and brittleness of TMCs increased with increasing TiB content, the TMCs with 10% TiB had the maximum tensile strength of 1147 MPa due to the combined effects of a relatively high relative density, a sufficient reinforcement of TiB and a homogeneous microstructure for this composition. The properties of the matrix alloy were also studied.

1. Introduction

With rapid development of auto industry, the requirement for high performance powder metallurgy (PM) parts is steadily increasing. Density is one of the most important aspects to determine the mechanical properties of PM parts [1]. Nowadays, there have emerged several advanced methods, such as warm compaction (WC), double pressing double sintering (P2S2), powder forging (PF), high velocity compaction (HVC) and so on to produce parts with higher density, among which HVC is especially attractive from a cost-performance view [2]. The HVC process is similar to the conventional die compaction with regard to die filling, compacting and ejecting parts from die [3]. The unique feature of HVC lies in the densification process. A hydraulically operated rammer creates intensive shock waves, which transfer the compacting energy from the compaction tool to the powder [4]. It has been receiving more and more attention for the dual characteristics of high impact energy of dynamic compaction as well as high efficiency of conventional die compaction.

In the past decade, many metal powders have been pressed by HVC and the obtained green density is obviously higher than that by conventional forming method. For example, HVC could press iron-based powders and stainless steel powders into compacts with density in the range of 7.4–7.7 g/cm^3 [5]. Recent researches have demonstrated that HVC exhibits unique advantages in pressing those powders with high intrinsic work hardening rate, which are usually difficult to be compacted through conventional methods. For pure titanium powder compacted by HVC, compacts with green density of 93.5% by one strike and 98.5% by two strikes, respectively, were obtained [6]. And a much higher green density of 96.0% was achieved by one strike in another report [7]. In contrast, the green density could only reach about 85% by WC or cold isostatic pressing (CIP) method [8]. High green density makes it possible to prepare dense titanium materials through a simple sintering process. Unfortunately, current researches about HVC are mainly focused on the forming effects of ductile powders or the characterization of HVC itself. A whole process containing HVC as well as the following sintering step, especially in fabricating titanium-based alloys and composites, is seldom reported.

Particles- or whiskers-reinforced titanium matrix composites (TMCs) overcome the limitation of titanium alloys, which has higher specific strength, specific modulus and better wear resistance. In-situ formed TiB whiskers are currently recognized as one of the most compatible and effective reinforcements for TMCs. In the present work, HVC was adopted to press titanium-based blended powders containing element powders of Al, Mo, and Fe as well as boron sources of TiB_2 and LaB_6. The compacts were then sintered in vacuum to synthesize the in-situ TiB whiskers reinforced titanium matrix composites (TiB/Ti-4.5 Al-6.8 Mo-1.5 Fe). Compared with traditional Ti-6 Al-4 V, the matrix alloy (Ti-4.5 Al-6.8 Mo-1.5 Fe) is a low-cost titanium alloy in which cheaper Fe and Mo replace expensive V and the raw material costs are lowered by 30% [9]. The main purpose of adding LaB_6 is to capture oxygen in the titanium powder during sintering and hence to improve the ductility of TMCs [10]. The

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forming ability of mixtures was evaluated. And the microstructures and mechanical properties of four TMCs with TiB volume fraction of 5%, 10%, 15% and 20%, respectively, were investigated and compared with those of the matrix alloy.

2. Experimental

The raw powders used in this experiment were hydride–dehydride (HDH) Ti powder (<150 μm), alloying elemental powders Al, Mo and Fe (2–10 μm), boron sources of TiB_2 (<2.5 μm) and LaB_6 (<25 μm). The composition of the raw materials and the designed volume percentage of reinforcements are listed in Table 1.

Each powder was weighed, fully mixed and then pressed with HYP 35–7 high-velocity compaction machine. Since effects of stroke length, impact energy or velocity on green density had been widely reported [2,7], an optimized stroke length of 18 mm corresponding to impact energy of 1370 J or velocity of 4.50 m/s was adopted in this research. Then the compacts were sintered at 1250 °C for 3 h in a vacuum of 1×10^{-3} Pa.

The specimen density was determined by Archimedes method. The relative density (RD) was calculated based on the theoretical density which was calculated according to the rule of mixtures for powder compacts and the phase volume fraction for sintered composites respectively, as listed in Table 1. The phase and microstructure were examined through an X-ray diffraction (XRD) tester and a JXA-8100 electron probe microanalyzer (EPMA), respectively. The hardness of composites was characterized by a Vickers microhardness tester under a load of 2 kg for 15 s. Tensile tests at room temperature were done on an Instron 8802 testing machine with the crosshead moving rate of 0.5 mm/min and the testing samples were machined into dog-bone shape with a length of 32 mm and a gauge length of 10 mm.

3. Results and discussion

3.1. Thermodynamic analysis

For titanium-based green compacts containing boron sources of TiB_2 and LaB_6, the following two in-situ reactions might occur to synthesize TiB reinforced TMCs during the sintering process:

$$TiB_2 + Ti \rightarrow 2TiB \quad (1)$$

$$2LaB_6 + 12Ti + 3[O] \rightarrow La_2O_3 + 12TiB. \quad (2)$$

The Gibbs free energy of reaction (1) is negative, indicating that TiB_2 and Ti are thermodynamically unstable together and therefore react to form TiB, and this is widely reported [11,12]. That of reaction (2) was calculated using the thermodynamic data from Ref. [13], and the result is plotted in Fig. 1 together with that of reaction (1) for comparison. The Gibbs energy of reaction (2) increases slightly with increasing reaction temperature, but always keeps negative, which suggests that the reaction is thermodynamically feasible. Moreover, the Gibbs free energy of reaction (2) is far below than that of reaction (1). For example, the Gibbs free energy of reaction (2) is −3066 kJ/mol at 1250 °C and that of reaction (1) is −53 kJ/mol only. This suggests that reaction (2) is more prone to occur.

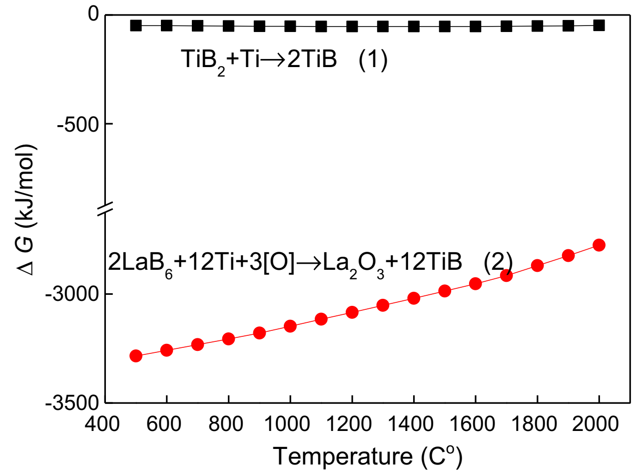

Fig. 1. The change of Gibbs free energy as a function of temperature for reactions (1) and (2).

3.2. Green and sintered density

Green density of blended powders pressed by HVC is shown in Fig. 2. It can be seen that the densities of four powders with boron sources are all lower than that of the matrix alloy. It should be attributed to the hindering effects caused by the added hard particles, i.e. TiB_2 and LaB_6 during the densification process. But all of the green densities are higher than 94% even if the TiB content in the sintered bodies reaches 20%. As the velocity of hammer striking upper punch is as high as about 4.50 m/s, and the striking happens in a short time, less than 20 ms, it is considered that heat exchange is seriously suppressed during the compaction and the process inherently induces adiabatic heating. It would make the metal particles softening or even melting at the contacting areas [3,14,15]. Therefore, the green density of titanium powder pressed by HVC is higher than that of both warm compacting (WC) and cold isostatic pressing (CIP). And the results illustrated in this experiment further indicate that HVC technology could press not only the pure titanium powder but also the titanium-based blended powders with a high content of hard particles into compacts with high green density.

The sintered density is also plotted in Fig. 2. As shown, the change trend of the sintered density is coincident with that of the green density. The matrix alloy has the highest sintered density, 99.63%. The densification of the composites should have been hindered by the nucleation and growth of TiB when TiB was in-situ synthesized during the sintering process. In general, the densification of composites is reduced with increasing reinforcement content [16,17]. In this research, however, the densities of the composites with 5–15% TiB are not reduced with increasing TiB content and all reach a relatively high value. For the

Table 1
Weight percentage of raw materials and volume fraction of reinforcements in composites.

Specimen	Reinforcement (vol.%)		Raw materials (wt.%)						Theoretical density (g/cm^3)	
	TiB	La_2O_3	Ti	Al	Mo	Fe	TiB_2	LaB_6	Green body	Sintered body
Matrix alloy	0	0	87.20	4.50	6.80	1.50	0	0	4.574	4.630
TMC1	5	0.5	85.04	4.25	6.43	1.42	1.98	0.88	4.573	4.633
TMC2	10	0.5	82.77	4.03	6.10	1.34	4.88	0.88	4.570	4.627
TMC3	15	0.5	80.50	3.81	5.76	1.27	7.78	0.88	4.567	4.621
TMC4	20	0.5	78.22	3.59	5.43	1.20	10.68	0.88	4.564	4.615