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Rapid synthesis of TiC/Ti_3SiC_2 composites by laser melting

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ABSTRACT

TiC/Ti_3SiC_2 composites were successfully synthesized by laser melting (LM) technique using Ti–Si–TiC with a molar ratio of 1:1.2:2 as starting powders in the holding time range of 15–60 s. Phase content and microstructure of the synthesized samples were analyzed by X-ray diffraction (XRD) and scanning electron microscopy (SEM) and X-ray energy dispersive spectrometer (EDS). The synthesis was extremely fast because the migration of solute mainly depended on the convection mixing of molten pool caused by intense laser beam which was far faster than the solid state diffusion by traditional methods. The apparent density and hardness of the composites are higher than Ti_3SiC_2 because of the existence of TiC phase.

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Introduction

Ternary layered compound (MAX phases) with a general formula of $M_{n+1}AX_n$ (n = 1, 2, 3, M is an early transition metal, A is an A group element, and X is C and/or N) has been attracting more attention owing to the unique combination merits of both metals and ceramics [1]. Ti_3SiC_2 is one of the most extensively studied MAX phases [2–5]. It is formed by exothermic chemical reaction between elemental powders during thermal processing. With metal-like properties, it exhibits high thermal and electrical conductivity, excellent thermal shock resistance and damage tolerance, and it is machinable. At the same time, it has properties of ceramics like good mechanical properties and oxidation resistance at elevated temperature, high elastic modulus and thermal stability at high temperature [6–10]. In addition, in contrast to the normal brittle ceramics, Ti_3SiC_2 exhibits some abnormal room-temperature compressive plasticity. Due to the above excellent properties, the applications of this material are very potential. It can be used as a high-temperature structural material, and also to process abrasion-resistant components and rotating parts, and so on.

Unlike traditional binary carbides, however, the ternary layered carbide Ti_3SiC_2 is relatively soft (Vickers hardness of 4 GPa), and has lower wear resistance and thermal stability (1700 °C). In order to conquer these obstacles, hard materials such as SiC [11], Al_2O_3 [12] and TiB_2 [13] had been incorporated into Ti_3SiC_2. As for reinforcing the phase in composites, TiC has many desirable properties, for example high hardness (Vickers hardness of 28 GPa) and good thermal stability. Additionally, it is a suitable reinforcing phase for the Ti_3SiC_2 matrix because their thermal expansion coefficients match very well (7.7×10^{-6} °C^{-1} for TiC and 9.1×10^{-6} °C^{-1} for Ti_3SiC_2, respectively) [14]. In fact, TiC phase usually coexists and shows special orientation relationship with Ti_3SiC_2 phase during the synthesis process of bulk Ti_3SiC_2 due to narrow stable zone of Ti_3SiC_2 phase in such a system [5,15]. Therefore, it is expected to be a good candidate material to improve the wear and/or mechanical properties of Ti_3SiC_2.

Many reports have been concentrated on the synthesis of TiC/Ti_3SiC_2 composites using different techniques such as hot isostatic pressing (HIP) [14], pulse discharge sintering (PDS) or spark plasma sintering (SPS) [16–18], metallic alloying (MA) [19] and hot pressing (HP) [20]. However, they usually require long processing time and high pressure, and there are very few researches that referred to rapid synthesis of TiC/Ti_3SiC_2 composites until now. As is known to all, it is difficult to rapid synthesize TiC/Ti_3SiC_2 composites because of a very slow diffusion rate of solute at solid state. Therefore, it is necessary to shorten the reactive time and elevate the synthesis efficiency to fabricate TiC/Ti_3SiC_2 composites through special methods. Laser melting, with its advantage of high density of energy, excellent power stability, high beam flexibility and so on, has attracted more attentions in recent years [21–24]. The main purpose of this paper is to exhibit the possibility of rapid synthesis of TiC/Ti_3SiC_2 composites by laser melting for the first time.

Experimental methods

The starting powder materials were: 99.7% purity titanium with a mean particle size of 150 μm, 99.5% purity silicon with an average size of 75 μm, and 99.5% purity titanium carbide with a mean size of 2.6 μm. Titanium, silicon, and titanium carbide powder mixtures with a molar ratio of 1:1.2:2 were used to synthesize TiC/Ti_3SiC_2 composite

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samples. The powder mixtures were first mixed in an agate mortar for 10 min, and then dried in an electric oven at 100 °C for 2 h.

The self-developed water-cooled copper-mold laser melting furnace is schematically illustrated in Fig. 1. The laser melting experiments were carried out on a 5 kW CO_2 laser materials processing system equipped with four-axis computer numerical controlled (CNC) work table. The laser melting parameters were: laser power separately 3.5 kW, 4.5 kW and 5 kW, beam diameter 13 mm, and melting time 15 s, 30 s, 45 s and 60 s, respectively.

Phase constituents of the samples were determined by means of Rigaku D/max 2200 pc automatic X-ray diffraction (XRD) with Cu target K_α radiation at 40 kV and 40 mA, using a continuous scan mode at 4°/min. The observations on the original surfaces, polished surfaces and fracture surfaces were performed using Camscan 3400 scanning electron microscope (SEM). Chemical compositions of the phase constituents were analyzed by Oxford energy dispersive spectrometer (EDS). The polished surfaces were etched 10 s using a mixed acid of HF and HNO_3 in a volume ratio of $HF:HNO_3:H_2O = 1:6:7$, and the fracture surfaces were produced artificially.

The density of synthesized samples was measured according to Archimedes principle. The Vickers hardness was measured using FM-800 Vickers hardness tester with a testing load of 500 g and a dwelling time of 10 s. At least 12 indentations were made on each sample. Quantitative determination of porosity was performed using a computer-aided SEM image analysis system.

Results and discussion

XRD results

The appearance of the laser melting TiC/Ti_3SiC_2 composites is shown in Fig. 2. The size of the synthesized samples is approximately Φ13 mm × 2 mm. The shape of the samples is sound and free from cracks. It indicates that there is no thermal explosion during laser melting the powder mixtures of Ti/Si/TiC.

Fig. 3 shows a series of results of XRD analyses for the laser melting TiC/Ti_3SiC_2 composite samples synthesized at laser power 3.5 kW. It can be seen that a large amount of Ti_3SiC_2 has been already synthesized by laser melting. Ti_3SiC_2 was found to be the main crystalline phase and TiC was presented as a minor phase at melting time 15 s. The presence

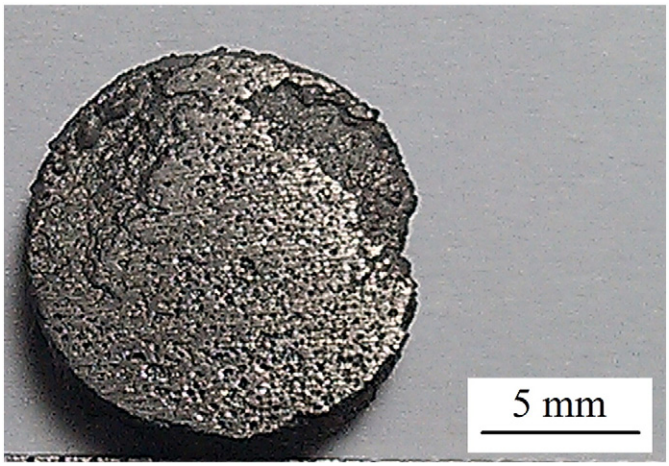

Fig. 2. Appearance of the laser melting TiC/Ti_3SiC_2 composites.

of a large amount of TiC indicated that it was not consumed completely to form Ti_3SiC_2. As the melting time was increased to 30 s, the content of TiC phase was decreased while the intensity of Ti_3SiC_2 peaks was getting stronger. However, the relative intensity of TiC peaks was abruptly increased while the content of Ti_3SiC_2 was decreased and became relatively of low value at melting time 45 s. Further increase of melting time to 60 s resulted in more increase of content of the TiC phase with decrease of Ti_3SiC_2, which might be due to the partial decomposition of synthesized Ti_3SiC_2.

The results of XRD analyses for the laser melting TiC/Ti_3SiC_2 composite samples synthesized at laser power 4.5 kW are shown in Fig. 4. For the specimen at melting time 15 s, the value of the main

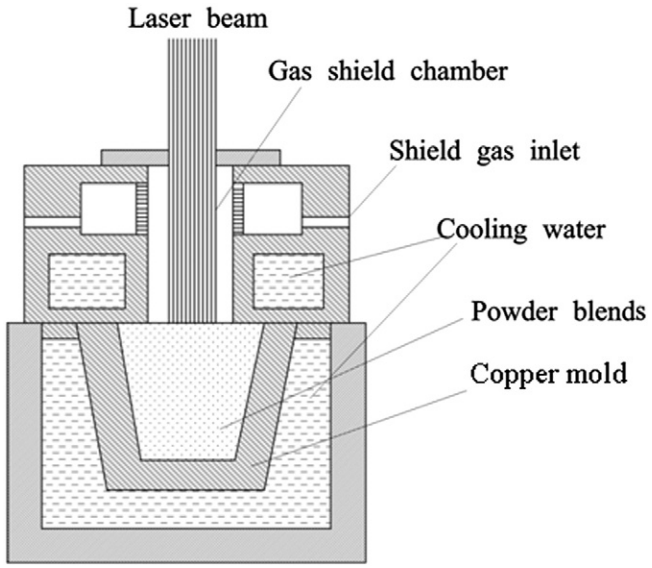

Fig. 1. Schematic of the water-cooled copper-mold laser melting furnace.

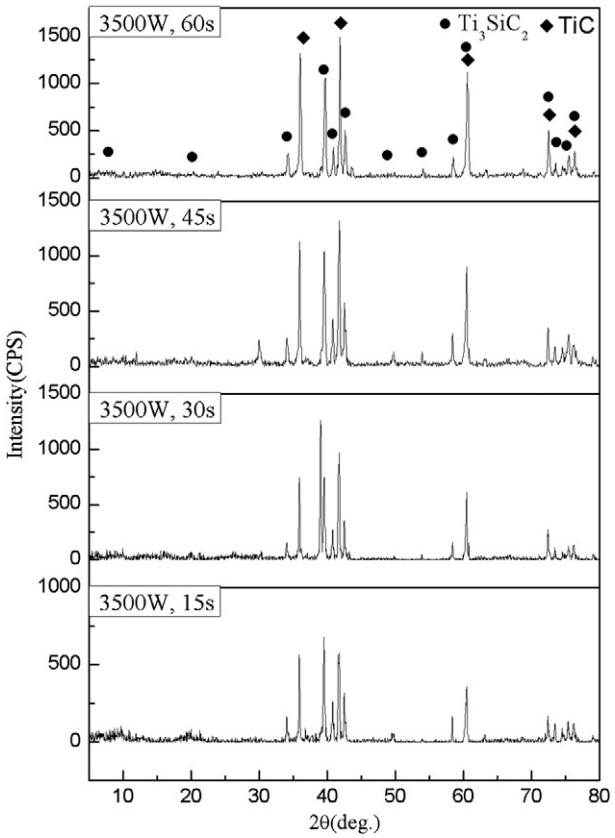

Fig. 3. X-ray diffraction patterns of laser melting TiC/Ti_3SiC_2 composites at 3.5 kW.

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