Effects of Temperature on Mechanical Properties and Element Diffusion of Ti3SiC2/Al2O3 Composites

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Abstract. Ti3SiC2/Al2O3 composites were prepared by vacuum hot pressing sintering at different temperature (1400 °C, 1450 °C and 1500 °C) under 30 MPa for 1.0 h, the effects of temperature on element diffusion and mechanical properties of Ti3SiC2/Al2O3 composites were investigated. Temperature has a strong influence on the mechanical properties and interfacial element diffusion of materials that higher temperature is beneficial to the densification of Ti3SiC2/Al2O3 composites to enhance the mechanical properties of it. The diffusion of Ti and Si occurs mainly at higher temperature accompanied by some decomposition of Ti3SiC2.

1. Introduction

Ceramics composites have attracted a lot of interests for excellent comprehensive performance of mechanical, electrical and chemical stability in engineering structure, biological medicine, aerospace machinery and other fields [1-4]. Alumina was used as matrix for high hardness and strength in many composite systems to get excellent mechanical properties. Ti3SiC2 has superb toughness like metal as a machinable ceramic which belongs to a larger system of cermet known as M_{n+1}AX_{n} (MAX) phases, where the M is an transition metal, A is mostly IIIA and IVA element, X is C or N and n=1, 2, 3 [5-6]. The physicochemical compatibility complementarity of mechanical properties between Al2O3 and Ti3SiC2 has been fully affirmed that great prospects for development of Ti3SiC2/Al2O3 composites can be realized in high technology fields such as thermal protection systems and other major engineering equipment core components [7-9].

In most studies, Ti3SiC2 was used as matrix, hard phase was introduced into it to improve the comprehensive mechanical properties. Al2O3 is seldom used as matrix in the composites composed of Ti3SiC2 and Al2O3 [10-13]. In our previous research, the superiority of mechanical properties and feasibility of preparation process of Ti3SiC2/Al2O3 composites have been determined by orthogonal test [6]. It was proved that good mechanical properties of Ti3SiC2/Al2O3 composites could be obtained by using high volume fraction Al2O3 (60.0 vol.%) as matrix and compounding with 40.0 vol.% Ti3SiC2, at the same time, the influence of temperature on the morphology and properties was obvious under orthogonal analysis.

In this work, we further focus on the effect of temperature on element diffusion and mechanical properties of Ti3SiC2/Al2O3 composites. Homogeneous Ti3SiC2/Al2O3 composites were prepared at different temperature to study on the microstructure and mechanical properties of it. The method of powder coated tablets was also used to observe the element distribution at interfaces in the heterogeneous Ti3SiC2/Al2O3 composites.
2. Experimental Procedure

2.1. Preparation of Homogeneous Samples
Ti3SiC2 (>98% pure, average particle size: 2 μm, Shanghai ST-Nano Science and Technology, China) and Al2O3 (99.9% pure, average particle size: 1 μm, Shanghai ST-Nano Science and Technology, China) were used as raw material to prepare Ti3SiC2/Al2O3 composites by powder metallurgy at different temperature (1400°C, 1450°C and 1500°C). The specific experimental process is as follows: firstly, Ti3SiC2 and Al2O3 were mixed in a planetary ball mill (XQM-2, China) for 4h, the weight ratio of powders-Al2O3 ball-anhydrous ethanol was 1:2:1. Then the slurry was dried at 50°C for 12h to dry, sieved by a 300 mesh sifter and placed in graphite mold with inner diameter of 45 mm. Then the sintering process was performed in vacuum hot pressing furnace (VVPgr-80-2300, China) with heating rates of 10°C/min (0-1200°C) and 5°C/min (≥1200°C) for 1h under 30 MPa.

2.2. Preparation of Heterogeneous Samples
In order to observe the diffusion behavior of elements between Ti3SiC2 and Al2O3 more intuitively, 15 g Al2O3 powder was poured into the graphite mold with a inner diameter of 45 mm and spread out, then the Ti3SiC2 plate (Ti3SiC2 powder pressed) was placed above the powder followed by another 15 g Al2O3 powder spread out. Finally, a pressure head is added and the mold was put into the furnace under the same sintering process (1400 °C, 1450 °C and 1500 °C) as the preparation of homogeneous materials. The sintered sample profile is shown in Figure 1, the apparent interface between the two phases was observed after incision.

![Figure 1. The profile of homogeneous samples and heterogeneous samples.](image)

2.3. Mechanical Testing and Microstructure Characterization
After grinding and polishing, the sintered body was cut into strips with a size of 36 mm×4 mm×3 mm for mechanical properties testing by electromechanical universal testing machine (CMT5504, MTSSYSTEMS, China), bending strength was tested more than three times and averaged. Vickers hardness tester (HV-1000IS, China) was used to measure the micro-hardness for a load of 9.8 N applied for 15 s after polishing (more than ten measurements).

The density of Ti3SiC2/Al2O3 composites was recorded using a density balance via the Archimedes method at room temperature. X-ray diffractometer (XRD, D8 ADVANCE, Bruker) was used to judge the phase composition analysis. The microstructure and elements diffusion were performed by scanning electron microscopy (SEM, JSM-7610F, United States) equipped with energy dispersive spectroscopy (EDS).

3. Results and Discussion

3.1. Phase Analysis
Figure 2 shows the XRD analysis of Ti3SiC2/Al2O3 composites at different temperature, it is obvious that the brittle diffraction peak of Ti3SiC2 and Al2O3 can be detected in all of the samples, the diffraction peaks of the two phases are sharp and clear, which shows a good crystallization state of Ti3SiC2/Al2O3 composites. A weak characteristic diffraction peak with low intensity at 41.48° is
confirmed as TiC, according to the test results of raw materials, the appearance of TiC is due to impurities in raw materials Ti₃SiC₂. When the temperature rises to 1500 °C, a new diffraction peak that did not appear at other temperatures is detected at 35.72° of TiC, the decomposition of Ti₃SiC₂ can be inferred at high temperatures. It is shown that the preparation of Ti₃SiC₂/Al₂O₃ composites does not require excessive temperature, if the temperature is further increased, the composition of the material may be greatly destroyed.

![Figure 2. The XRD patterns. a: raw materials Ti₃SiC₂, b: Ti₃SiC₂/Al₂O₃ composites at different temperature.](image)

3.2. **Morphology Analysis**

The microstructures of homogeneity Ti₃SiC₂/Al₂O₃ composites are shown in Figure 3, when sintering temperature is 1400 °C, it can be seen from the SEM images that the sample has relatively poor density for considerable pores, apparent grain boundaries and gaps (as shown in Figure 3a and 3b). Meanwhile, the growth of tabular grains with the continually increase of temperature results in the non-compact bonding of Ti₃SiC₂ and Al₂O₃ directly due to morphological differences. The further rise of temperature is beneficial to the densification that the two phases are closely bound without obvious gaps (as shown in Figure 3c and 3d). Accordingly, the density of the material enhanced with the temperature under the same pressure, when the sintering temperature is 1500°C, the relative density of the Ti₃SiC₂/Al₂O₃ composites increases to 99.1 % (as shown in Figure 4).

![Figure 3. The SEM images of Ti₃SiC₂/Al₂O₃ composites. a-b: 1400 °C, c-d: 1500 °C](image)
To further determine the effect of temperature on the combination of Ti$_3$SiC$_2$ and Al$_2$O$_3$, after sintering Ti$_3$SiC$_2$ plate into Al$_2$O$_3$ powder, the EDS was used for elemental analysis of heterogeneous composites at the Ti$_3$SiC$_2$/Al$_2$O$_3$ interfaces in cross section which were sintered at different temperatures (1400°C, 1450 °C, 1500 °C) for 0.5 h are shown in Figure 5. In sintering process, Ti element and Si element diffuses into alumina matrix, the diffusion depth moves continuously to the left (i.e. alumina) along the interface of Ti$_3$SiC$_2$ and Al$_2$O$_3$ with the increase of temperature. Temperature significantly increased the diffusion efficiency of elements that the thickness of diffusion layer raise from 120 μm to 183 μm. At this time, temperature promotes the diffusion of elements and sintering of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites. It can also be seen from macroscopic photographs of cameras that there is an increasing grey diffusion layer.

![Figure 4](image)

**Figure 4.** The relative density of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites at different temperature.

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![Figure 5](image)

**Figure 5.** EDS analysis for the interfaces of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites sintered at different temperature for 1.0 h.a: 1400 °C, b: 1450 °C and c: 1500 °C.

### 3.3. Mechanical Properties

The flexural strength and Vickers hardness of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites are shown in Figure 6. There is an increasing trend in both strength and hardness that the increase of temperature is beneficial to the improvement of mechanical properties. Firstly, the strength and hardness of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites show a strong correlation with density, the improvement of mechanical properties were primarily attributable to the dense microstructure. According to semi-empirical formula: $\sigma = \sigma_0 \exp(-bp)$,
where $\sigma$ and $p$ are strength and porosity, respectively, the existence of voids and gaps can significantly inhibit the development of mechanical properties of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites, dense structures resist more stress to maintain the serviceability of materials.

**Figure 6.** The bending strength and micro-hardness of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites at different temperature.

**Figure 7.** The SEM images of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites at 1500 °C.

Compared with hardness, the flexural strength of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites is not show significant enhancement in the later stage, which can be attributed to the phenomenon that the decomposition of Ti$_3$SiC$_2$ makes the local uniform of microstructure and phase distribution, molten Si is bound to exist with the generation of TiC, but the short holding time and melting temperature cannot make the Si uniform distribution. Thus, local structural defects and fragile interfaces produce, these defects are few and have little impact, so the trend of performance improvement is still obvious to some extent. In addition. There are abundant layered structures and essential columnar structures of Ti$_3$SiC$_2$ (Figure 7a), which can prolong and deflect the crack path and inhibit the crack growth in a local range, so the strength of Ti$_3$SiC$_2$/Al$_2$O$_3$ composites could be improved with the development of lamellar Ti$_3$SiC$_2$. Meanwhile, Al$_2$O$_3$ raw material still keeps fine grain size after sintering, and no abnormal growth occurs(Figure 7b), fine grain strengthening is beneficial to the improvement of material strength[14].

**4. Conclusions**

Ti$_3$SiC$_2$/Al$_2$O$_3$ composites were successfully fabricated via vacuum hot pressing sintering at different temperature and the same pressure and holding time to study on the effect of temperature on the mechanical and micro-structure. Based on the experimental results, the following conclusions were
obtained:

1. The higher the temperature, the better the mechanical properties, increasing temperature is beneficial to sintering by forming liquid phase.

2. The increase of flexural strength is not obvious at the later stage for the decomposition of Ti$_3$SiC$_2$ at high temperature for intrinsic defect

3. Ti$_3$SiC$_2$ to Al$_2$O$_3$ diffusion is the main phenomenon at the interface of two phases for the active elements Ti and Si in Ti$_3$SiC$_2$.

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6. References

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