

Fabrication of Ti_2AlC - Ti_3AlC_2 - Ti_3SiC_2 composite by spark plasma sintering (SPS) method from elemental powders

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Recently, a new remarkable group of materials known as ternary-layered compounds or “machinable ceramics” has attracted interest of materials scientists. These ternary-layered ceramics are thermally and electrically conductive, relatively soft and readily machinable, they are relatively tough and not susceptible to thermal shock, and behave plastically at elevated temperatures. At the same time, these carbides are very refractory, oxidation resistant and maintain strength to temperatures that render the best superalloys available today unusable [1]. This unique combination of properties makes them likely candidates for structural applications at elevated temperatures, such as turbine blades and stators, heavy duty electric contacts, bearings, etc.

Ti_2AlC and Ti_3AlC_2 in Ti-Al-C system and Ti_3SiC_2 in Ti-Si-C system are typical compounds belonging to this family. However, it is difficult to fabricate single-phase bulk dense samples of Ti_2AlC , Ti_3AlC_2 or Ti_3SiC_2 because of their very narrow phase range in Ti-Al-C and Ti-Si-C phase diagrams. The resultant samples always contains, in most cases, TiC, as ancillary unwanted phase [2–4]. It was also found that small amount of Ti_2AlC always exists in Ti_3AlC_2 samples [5]. Furthermore, it has been proved that Al additive apparently enhances the synthesis of Ti_3SiC_2 [6]. When 0.3 mol Al was used to replace Si for preparing Ti_3SiC_2 , a new unknown phase in addition to Ti_3SiC_2 appeared in the resultant product [7]. This new unknown phase is probably Ti_3AlC_2 based on our analysis of the X-ray diffraction pattern. These imply that it is easier to synthesize composite containing these ternary carbides than to make single-phase carbides. Moreover, Ti_2AlC , Ti_3AlC_2 and Ti_3SiC_2 have similar properties and composite consisting of them may also have properties alike. Additionally, Spark plasma sintering (SPS) method is an innovative technique for rapid sintering materials with finer grains and better properties than those prepared by existing sintering methods [8].

The objective of this work was to fabricate Ti_2AlC - Ti_3AlC_2 - Ti_3SiC_2 composite in Ti-Al-Si-C quaternary system by SPS method from elemental powders.

All of the work was conducted using powder mixture of titanium (99.0% pure, 10.6 μ m), Al (99.8% pure, 12.8 μ m), Si (99.5% pure, 9.5 μ m) and carbon black (99%, 13.2 μ m) (all from Institute of Non-ferrous

Metals, Beijing, China). In brief, the mixture with a designed composition was firstly mixed for 24 hr, then placed in a graphite die, 20 mm in diameter, and finally sintered in a spark plasma sintering system (Mode SPS-1050). The powder mixture was heated at a rate of 80 °C/min until the requisite temperature was reached; the soaking time was 8 min.

In previous work, we endeavored in the fabrication of single phase Ti_2AlC , so the composition of the powder mixture in this work was designed as a basic composition of 2.0Ti/1.0Al/1.0C plus 0.2 mol Si and the final

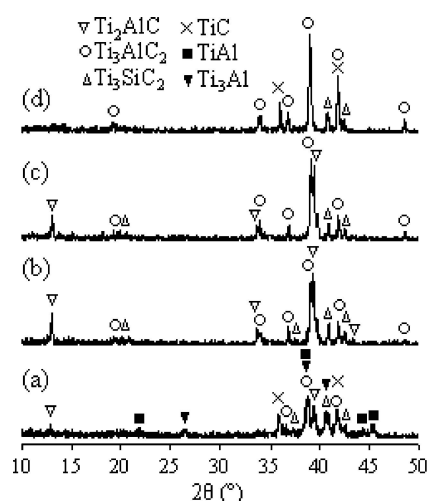


Figure 1 X-ray diffraction patterns of samples sintered at (a) 1000 °C, (b) 1100 °C, (c) 1200 °C, and (d) 1300 °C from 2.0Ti/1.0Al/0.2Si/1.0C powder mixture by SPS.

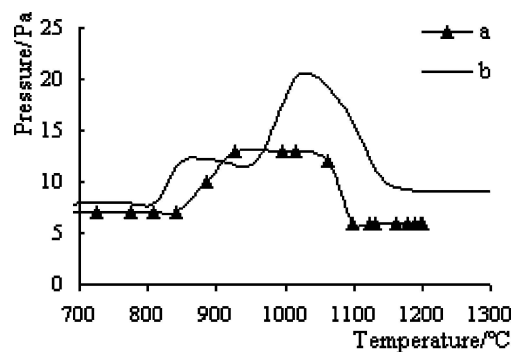


Figure 2 Temperature dependence of vacuum, (a) sample sintered at 1200 °C and (b) sample sintered at 1300 °C.

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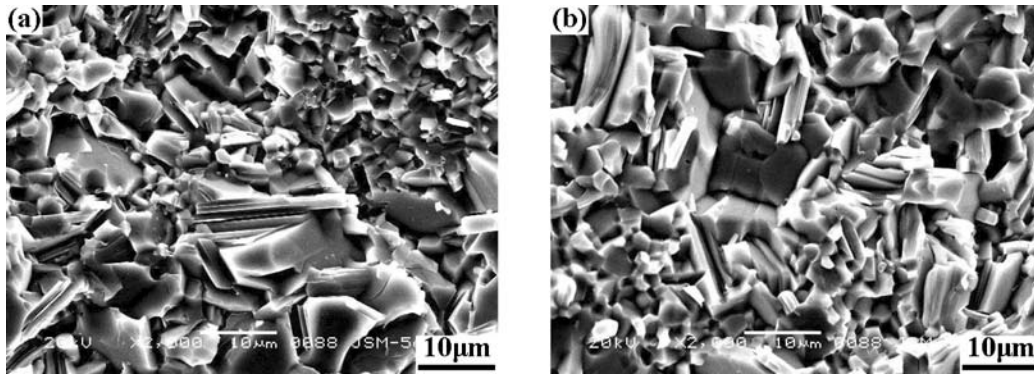


Figure 3 Microstructure of fracture surfaces of samples sintered at (a) 1100 °C, and (b) 1200 °C from 2.0Ti/1.0Al/0.2Si/1.0C powder mixture by SPS.

powder mixture was 2.0Ti/1.0Al/0.2Si/1.0C. Since Al and Si especially Al evaporate at high temperature, more Al + Si than required by the stoichiometric of Ti_2AlC was used to make up their loss.

Fig. 1 shows the phases presented after spark plasma sintering the 2.0Ti/1.0Al/0.2Si/1.0C powder mixture at different temperatures for 8 min, as determined by XRD. It is clear that when sintered at 1000 °C for 8 min, peaks of Ti_2AlC , Ti_3AlC_2 and Ti_3SiC_2 appeared. But there existed other unwanted phases: TiC and Ti-Al intermetallics such as TiAl and Ti_3Al . When the sintering temperature increased to 1100 or 1200 °C, only peaks of Ti_2AlC , Ti_3AlC_2 and Ti_3SiC_2 appeared in samples, all unnecessary phases disappeared. The apparent differences between these two samples are: Ti_2AlC content was more than that of Ti_3AlC_2 in sample sintered at 1100 °C and Ti_3AlC_2 content was more than that of Ti_2AlC in sample sintered at 1200 °C. While in sample sintered at 1300 °C, peaks of Ti_2AlC disappeared; peaks of TiC appeared again and Ti_3AlC_2 and Ti_3SiC_2 were other two major phases.

In Ti-Al-Si-C system, Al and Si are more volatile than other two elements. So, the appearance of TiC and the disappearance of Ti_2AlC in sample prepared at 1300 °C are possibly due to the rapid loss of Al and Si at high temperature. This is also verified by the temperature dependence of the vacuum pressure in the exuviated chamber of the SPS system, as shown in Fig. 2. In the spark plasma sintering process, the gas in the chamber is discharged out at a constant rate. As a result, the vacuum pressure maintains unchanged during the ordinary sintering process. However, a peak in the pressure may appear if a large quantity of gas is given out from a reaction in the sample. Pressure peaks appeared in both samples sintered at 1200 and 1300 °C. But for sample sintered at 1300 °C, pressure peak was more pronounced than that of sample sintered at 1200 °C, which indicates the evaporation of Al or Si is more serious in sample sintered at 1300 °C, leading to the formation of TiC and the disappearance of Ti_2AlC .

Shown in Fig. 3 are the scanning electron micrographs of the fracture surfaces of samples prepared at 1100 and 1200 °C. For sample prepared at 1100 °C, two kinds of grains appeared, a plate-like one with grain size of 16 μm and an equiaxed one with grain size of 5 μm. These two kinds of grains were distributed inhomogeneously. Sample synthesized at 1200 °C also had two kinds of grains. But these two kinds of grains had smaller difference in grain size in a range of 5–12 μm than that appeared in sample prepared at 1100 °C and distributed more homogeneously. There were no pores in these two samples, showing that samples were dense.

It is concluded from X-ray diffraction patterns and scanning electron micrographs that dense bulk composite only containing Ti_2AlC , Ti_3AlC_2 and Ti_3SiC_2 could be conveniently synthesized by SPS from mixed elemental powders with small amount of Si additive at 1100 and 1200 °C, a temperature lower than those used in HP and HIP methods for preparing single phase ternary carbides materials [3, 9, 10]. The whole process is completely finished in a few min.

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