

Fabrication of high purity dense Ti_2AlC – Ti_3AlC_2 composite by spark plasma sintering method

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Ti_2AlC and Ti_3AlC_2 are both layered ceramics belonging to a series that has a formula of $M_{n+1}AX_n$, in which M is a transition element, A is III and IV group element and X represents C or N. Although they were discovered several decades ago [1], it was only recently that material scientists began to focus on the synthesis of such materials. Ti_2AlC and Ti_3AlC_2 combined the properties of ceramics and metals. To date we have known that Ti_2AlC and Ti_3AlC_2 are both featured with two kinds of properties: they are easily machinable with handsaw without lubrication and self-lubricant. In addition, they have high flexural strength; they can resist high temperature oxidation and are low density. Much of the work focuses on the fabrication of high purity Ti_2AlC and Ti_3AlC_2 . Fabrication method includes hot pressing and hot isostatic pressing [2, 3]. However, the fabrication of these two materials with high purity is difficult since TiC always resides in the final materials. In addition, the authors know from their previous work that Ti_2AlC and Ti_3AlC_2 always transforms to each other. So it is more difficult to synthesis pure single phase Ti_2AlC or Ti_3AlC_2 . Thus, we think maybe Ti_2AlC – Ti_3AlC_2 composites can be prepared without impurity such as TiC.

The purpose of the paper is to report the fabrication of pure Ti_2AlC – Ti_3AlC_2 composites using spark plasma sintering method.

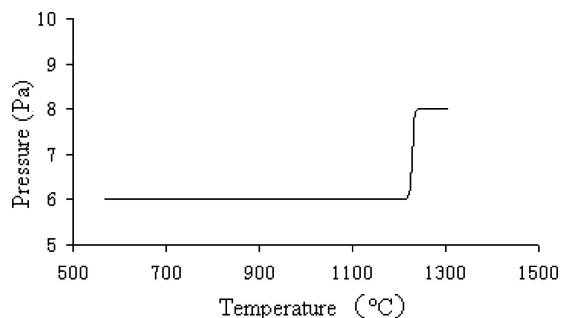


Figure 2 Vacuum vs. temperature curve of sample sintered at 1300 °C.

TiC, aluminum and titanium were used as raw materials. They were ball-mixed for 24 hr in a plastic pot. The composition was TiC:Al:Ti = 1:1:1 by atomic ratio. The mixture was put in a graphite die with a diameter of 16 mm. The graphite die was then put in the Spark Plasma Sintering (SPS) furnace. The furnace was evacuated to a pressure less than 6 Pa. The mixture was then sintered at different temperatures: 1100, 1200 and 1300 °C for 8 min with a pressure of 30 MPa. The sintering power was 70 W, the input and output impulse ratio was 12:2, the samples were heated at a rate of 80 °C per minute. The temperature was measured with IR high temperature thermometer.

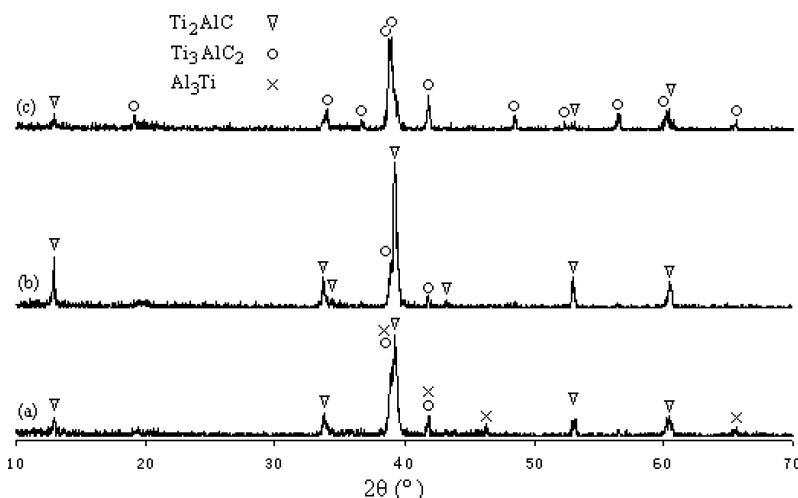


Figure 1 XRD patterns of samples sintered at (a) 1100 °C; (b) 1200 °C and (c) 1300 °C by spark plasma sintering method using powder mixture of 1 mol Ti + 1 mol TiC + 1 mol Al.

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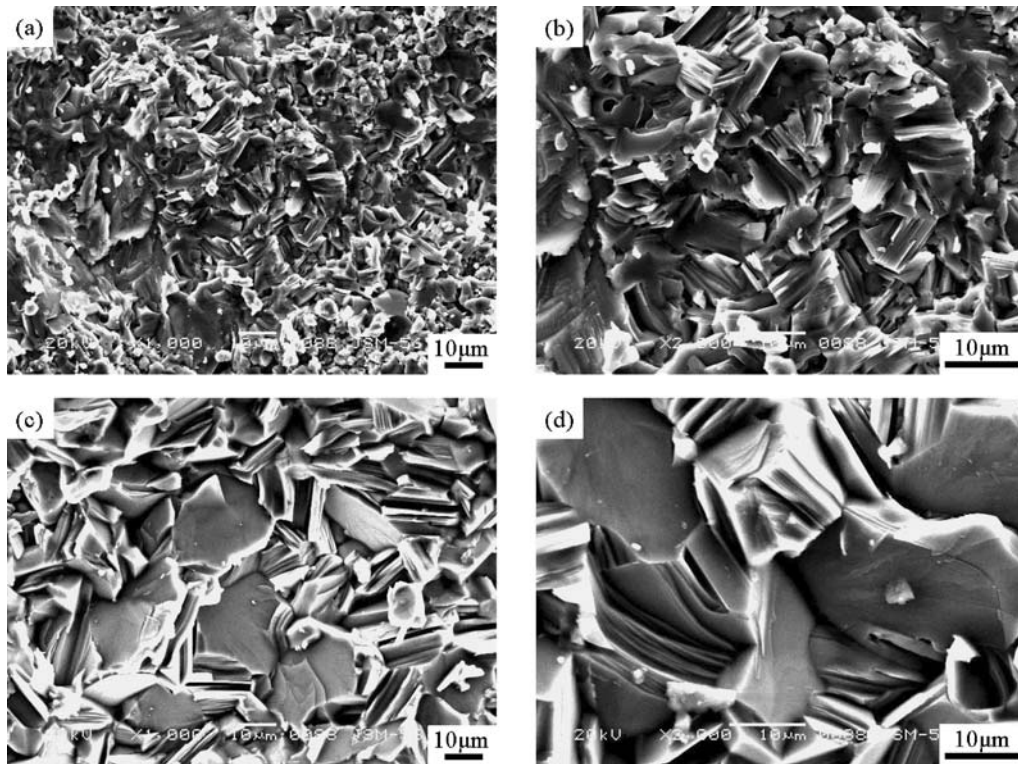


Figure 3 SEM of fracture surfaces of samples sintered at 1100 °C ((a) and (b)) and 1200 °C ((c) and (d)).

Phase composition of the samples sintered at different temperatures was investigated with XRD (Model D/MAX-RB, RIGAKU Corporation, Japan). The microstructure of the samples was analyzed with SEM (Model JSM-5610LV, JEOL Ltd., Japan).

Fig. 1 is the XRD patterns of samples sintered at different temperatures by SPS method. It can be seen clearly that Ti_2AlC has occurred and was the main crystal phase in the samples when the powder mixture was sintered at 1100 °C (curve (a) in Fig. 1). Also there existed peaks of alloy Al_3Ti and Ti_3AlC_2 . When sintered at 1200 °C, the sample was made up of Ti_2AlC and Ti_3AlC_2 , while alloy Al_3Ti disappeared (curve (b) in Fig. 1). Ti_2AlC was still the main crystal phase in the sample and its content increased, while the content of Ti_3AlC_2 almost remained unchanged. When sintered at 1300 °C, the sample was also composed of Ti_2AlC and Ti_3AlC_2 , but the main phase was Ti_3AlC_2 instead of Ti_2AlC (curve (c) in Fig. 1).

The above results revealed that Ti_2AlC would transform to Ti_3AlC_2 when temperature reaches 1300 °C. We think there are two possible processes: (1) $Ti_2AlC + TiC \rightarrow Ti_3AlC_2$ and (2) $3Ti_2AlC + C \rightarrow 2Ti_3AlC_2 + Al$.

Since no TiC existed in the sample before transformation (see curves (a) and (b) in Fig. 1), process (1) could not occur. Compared with element titanium and carbon, Al is more volatile. The saturated vapor tension of pure Al at 1100, 1200 and 1300 °C is 0.22, 1.38 and 6.57 Pa, respectively [4]. Thus, the evaporation speed of Al will increase greatly when temperature increases from 1100 to 1300 °C. In fact in the sintering curve of the sample sintered at 1300 °C the pressure increased sharply at about 1235 °C (see Fig. 2), this confirms that a kind of gas evaporated from the sample. In this case it can only be aluminum.

So when sintering temperature increased to 1300 °C, Ti_2AlC transformed to Ti_3AlC_2 through process (2). Thus $Ti_2AlC-Ti_3AlC_2$ composites can be prepared in which Ti_2AlC is the main phase. If temperature increases to 1300 °C, the sample fabricated is $Ti_3AlC_2-Ti_2AlC$ in which Ti_3AlC_2 is the main phase.

Fig. 3 shows the microstructure of samples sintered at 1100 and 1200 °C. It can be clearly seen from Fig. 3 that sample sintered at 1100 °C is mainly made up of typical lamellar crystal including small content of white fine grain (Fig. 3a and b). According to XRD result we think it is Al_3Ti . When sintered at 1200 °C, sample is completely composed of lamellar crystal, white fine grain viz. Al_3Ti disappeared (Fig. 3c and d). No pore can be observed in the fracture surface of sample sintered at 1200 °C, this indicates that the sample is dense. Also, the crystal size in samples sintered at 1200 °C is larger than that in samples sintered at 1100 °C.

In summary, pure and dense $Ti_2AlC-Ti_3AlC_2$ composite can be easily prepared using TiC , Al and carbon sintered at 1200 °C by SPS method. When temperature is raised, Ti_2AlC will transform to Ti_3AlC_2 because of the evaporation of Al.

References

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