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Synthesis and characterization of Cr_2AlC ceramics prepared by spark plasma sintering

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Abstract

The investigation of bulk Cr_2AlC ceramic fabricated by Spark Plasma Sintering (SPS) from coarse powders (CAC10) and fine powders (NCAC10) in the temperature range of 1100–1400 °C was carried out. The XRD results indicate that Cr_2AlC, as major phase, always appears with minor and trace amount of Cr_7C_3 and Cr_2Al respectively in both NCAC10 and CAC10 samples and the amounts of later two phases decrease with increase in temperature. However, the Cr_2AlC phase content in NCAC10 is higher than that of CAC10 sintered at the same temperature. The micrographs of back-scattered SEM show that grains with smaller size and pores with fewer amounts appear in SPSed NCAC10 in comparison to that of CAC10. As consequence, the higher hardness (5.6 GPa) of NCAC10 than that (3.9 GPa) of CAC10 was obtained. The patterns of XRD, microstructure and hardness of samples HPed at 1400 °C for the same composition were also presented for comparison.

Keywords: Cr_2AlC compound; Spark Plasma Sintering (SPS); X-ray diffraction (XRD); Back-scattered electron image; Mechanical properties

1. Introduction

The ternary carbides, such as Ti_3SiC_2 [1,2], Ti_3AlC_2 [3] and Ti_2AlC [4], have aroused researchers' more and more interest because of their excellent mechanical, thermal, electrical and chemical properties, in which the carbides belong to $M_{n+1}AX_n$ system (where $n=1, 2, 3$, M is an early transition metal, A is an IIIA or IVA element, and X is C or N, abbreviated as MAX) and the same hexagonal group of $P6_3/mmc$.

As a member of MAX system, the lattice parameters of Cr_2AlC and its phase relationship in the system of Cr–Al–C had been identified in 1980s [5]. Recently, theoretical calculation on Cr_2AlC were carried out by Sun et al., [6], where the results show that Cr_2AlC possesses the highest theoretical bulk modulus among M_2AlC (M=Ti, V, Cr, Nb and Ta) that is resulted from its greatest M–C bond energy. Later, the results that bulk Cr_2AlC showed an excellent oxidation resistance at 1200 °C comparing with that of Ti_3SiC_2 was reported [7], further revealing that Cr_2AlC could be a promising material. In addition, Cr_2AlC ceramics were studied in our previous work, in which the electrical and thermal properties as well as phase formation sequence of Cr_2AlC were described in Refs. [8,9] respectively.

Spark Plasma Sintering (SPS), as a new sintering technique, has been employed by Zhang et al., [10] and Gao et al., [11] on the fabrication of Ti_3SiC_2 and Mei et al., [12] on TiAl/Ti_2AlC composites as well as Wang et al., [13] on Ti_3SiC_2/Al_2O_3 composites. It is noted that SPS technique can fabricate dense samples at low temperature with short time, whereas application of SPS on preparation of bulk Cr_2AlC has not been available in literature yet.

The purpose of this paper concerns the synthesis and densification behavior as well as the microstructure observation of Cr_2AlC samples sintered by SPS technique from coarse and fine starting powders. On the other hand, the phase assembly, microstructure and hardness of Cr_2AlC samples prepared by hot-pressing will also be presented for comparison.

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2. Experimental procedure

There are two kinds of raw powders used as starting materials: 1) chromium (~32 μm, 99.95%), aluminum (~130 μm, 99.95%) and graphite (~5 μm, 99%) powders were used as the coarse starting materials, namely CAC10; 2) chromium with small particle size (~3 μm), aluminum (~3 μm, 99%) and the same graphite powders as that used in CAC10 were used as the fine starting materials, named as NCAC10. Fine chromium was obtained by milling chromium powder mentioned above for 8 h using Si_3N_4 ball as milling media in planet milling machine.

The powders were weighed according to the composition Cr:Al:C=2:1.1:1 and milled in absolute alcohol for 24 h using Si_3N_4 ball as milling media. Dried powders were pre-pressed as pellets and sintered in SPS equipment (FCT-Systeme, Gewerbepark 11, 96528 Rauenstein, Germany) at a designed temperature for 5 min under 50 MPa with a heating rate of 200 °C/min. Samples hot-pressed at 1400 °C for 1 h under 20 MPa, named as HPed samples, were prepared for comparison with SPSed ones.

Density of the sintered samples was measured by Archimedes principle. Phase assemblages were determined by X-ray diffraction (XRD) with CuKα radiation at 40 kV and 100 mA (D/max 2550 V, Japan). The Vickers hardness was determined by indentation using a Vickers diamond indenter and a load of 20 N for 10 s (Akashi). Microstructure observation by SEM with back-scattered electron image was performed via an electron probe microanalyzer (JEOL JXA-8100F, Japan) on the polished surface of samples.

3. Results and discussion

3.1. XRD analysis of SPSed Cr_2AlC samples

The XRD patterns of samples CAC10 SPSed ranging from 1250 °C to 1400 °C are illustrated as Fig. 1(a)–(d), where the XRD pattern of sample HPed at 1400 °C is also shown in Fig. 1 for comparison. For the SPSed samples, Cr_2AlC appears as a major phase, together with small amount of Cr_7C_3 and Cr_2Al. It is found that the amount of Cr_7C_3 and Cr_2Al are comparable in the sample SPSed at 1250 °C. When the temperature increases from 1300 °C to 1400 °C, Cr_2Al content decreases quickly while the amount of Cr_7C_3 goes down slowly. It is noted that the phase assembly and their content of CAC10 SPSed at 1400 °C are very similar to those of HPed, as shown in Fig. 1(d) and (e) respectively, except for the existence of trace amount of Cr_2Al in SPSed one.

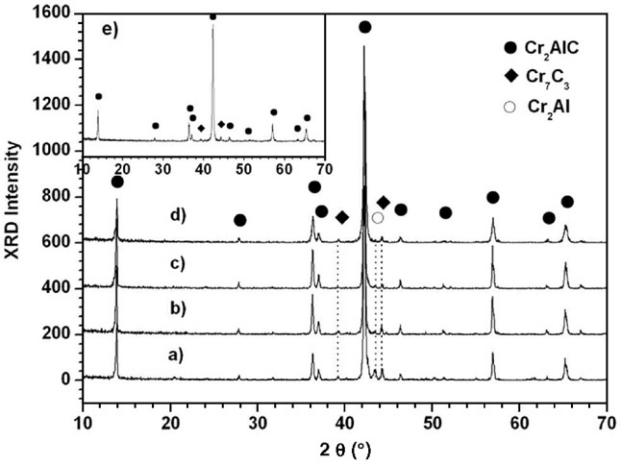

Fig. 1. XRD patterns of samples CAC10 SPSed at (a) 1250 °C, (b) 1300 °C, (c) 1350 °C, (d) 1400 °C and (e) XRD pattern of sample CAC10 HPed at 1400 °C.

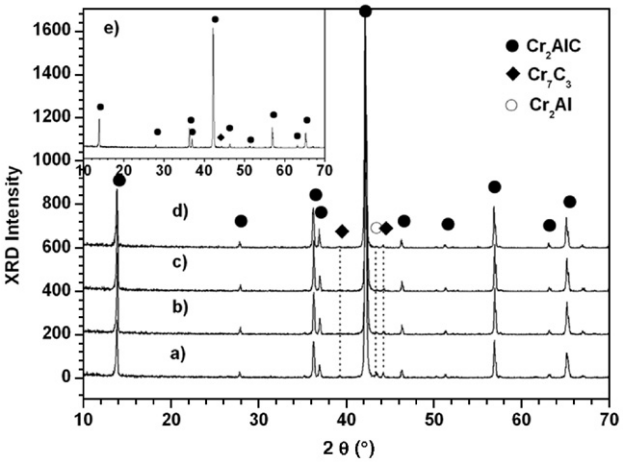

Fig. 2. XRD patterns of samples NCAC10 SPSed at (a) 1100 °C, (b) 1200 °C, (c) 1300 °C, (d) 1400 °C and (e) XRD pattern of sample NCAC10 HPed at 1400 °C.

The XRD patterns of samples NCAC10 SPSed in the temperature range of 1100–1400 °C are shown in Fig. 2(a)–(d). Besides, the XRD pattern of sample NCAC10 HPed at 1400 °C is illustrated as Fig. 2(e) for comparison. It is noticed, that there also exist three phases in samples NCAC10, just like that in samples CAC10, i.e. Cr_2AlC, as major phase, with minor amount of Cr_7C_3 and trace amount of Cr_2Al, and the amount of later two phases are declined as the increment of temperature from 1100 °C to 1400 °C. However, it is difficult to find trace amount of Cr_2Al in HPed NCAC10 as the case of CAC10. It should be pointed out that the amount of Cr_2AlC in NCAC10 sample SPSed at 1400 °C could reach as high as 99 wt.%, while it is 97 wt.% in SPSed CAC10 for the same temperature.

The fact that XRD results of sample NCAC10 SPSed in the temperature range of 1200–1400 °C contain close phase assembly and phase content, suggests that the reactions are almost finished at 1200 °C and

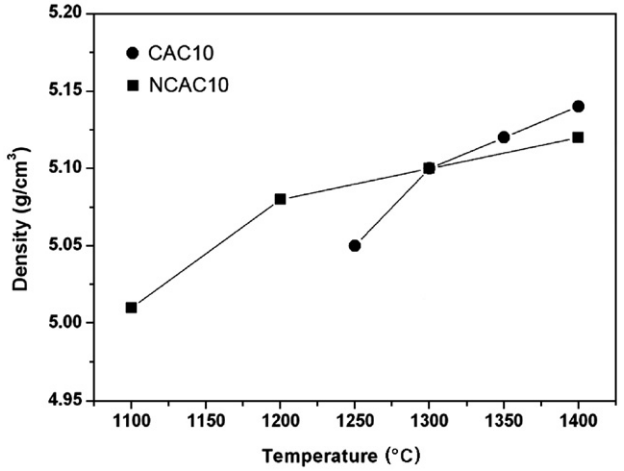

Fig. 3. The variation of bulk densities of SPSed samples *vs* sintering temperature.