Available online at www.sciencedirect.com
SCIENCE DIRECT
Trans. Nonferrous Met. Soc. China 17(2007) 1001−1004
Transactions of Nonferrous Metals Society of China
www.csu.edu.cn/ysxb/

Synthesis of Ti_2AlC by hot pressing and its mechanical and electrical properties

WANG Ping(王 苹)[1,2], MEI Bing-chu(梅炳初)[2], HONG Xiao-lin(洪小林)[2], ZHOU Wei-bing(周卫兵)[2]

1. School of Science, Wuhan University of Technology, Wuhan 430070, China;
2. State Key Laboratory of Advanced Technology for Materials Synthesis and Processing, Wuhan University of Technology, Wuhan 430070, China

Received 20 November 2006; accepted 10 May 2007

Abstract: Ti_2AlC bulk material was synthesized by hot pressing of mixture powders of TiC, Ti, Al and active carbon. The phase compositions of resultant product at different temperature were detected by X-ray diffractometer. The microstructures of the samples were observed by SEM. Finally, the mechanical properties and thermal properties of the sample at 1 400 ℃ were measured. The results show that high purity Ti_2AlC material with little Ti_3AlC_2 can be synthesized by hot pressing 0.5TiC/1.5Ti/1.0Al/0.5C at 1 400 ℃. Ti_2AlC exhibits high mechanical properties and metallic electrical properties. Its fracture toughness is 7.0 $MPa\cdot m^{1/2}$, its flexural strength is 384 MPa at room temperature, and its electrical conductivity is $2.56\times10^6\ \Omega^{-1}\cdot m^{-1}$ at room temperature.

Key words: hot pressing; Ti_2AlC; mechanical properties; electrical properties

1 Introduction

Layered ternary carbide Ti_2AlC has attracted increasing attention because it is a remarkable material that combines many of the merits of both metals and ceramics[1−12]. Like ceramics it has high mechanical properties, and like metals it is an excellent electrical conductor at room and high temperatures. Recently, the synthesis methods of Ti_2AlC developed its mechanical properties and electrical properties. According to previous literatures[13−14] TiC replacing a part of Ti and C elemental powder as a raw material can accelerate the reactions and was helpful for forming Ti_2AlC. In this study, Ti_2AlC bulk material with fantastic mechanical properties and electrical properties is synthesized by hot pressing 0.5TiC/1.5Ti/1.0Al/0.5C powder mixture. Active carbon rather than graphite is used as a raw material due to its high activity.

2 Experimental

Commercially available TiC (99.0% in purity, 11.8 μm), Ti (99.0% in purity, 10.6 μm), Al (99.8% in purity, 12.8 μm) and active carbon (99% in purity, 13.2 μm) (all from the General Research Institute of Nonferrous Metals, Beijing, China) were used as raw materials to synthesize Ti_2AlC. The mixture of 0.5TiC/1.5Ti/1.0Al/0.5C was firstly mixed in ethanol for 24 h, and was filled into graphite crucibles, then hot pressed at 1 300, 1 400, 1 450 and 1 500 ℃ for 60 min under a pressure of 30 MPa in flowing Ar atmosphere. After being hot pressed, the compacts were heated according to the following procedures: heating at a rate of 5 ℃/min to 300 ℃, then at 60 ℃/min to the final temperature. The soaking time was 60 min.

Phase identification of the material prepared at different temperatures was conducted via a X-ray diffractometer (D/MAX-RB, RIGAKU Corporation, Japan) after the graphite layer of the samples surface was removed. The microstructure evolution of the hot pressed material was investigated by scanning electron microscope (JSM-5610LV, JEOL Ltd., Japan).

The mechanical properties of Ti_2AlC were measured by using omnipotence ceramic material test machine (MTS-810 Ceramic Test System, USA). Moreover, the Vicker's hardness of Ti_2AlC was measured using Leitz Micro-hardness Tester (Leitz Wetzlar, Germany) at 0.3 mm/s and a load of 0.98 N.

The electrical conductivity of Ti_2AlC at room temperature and high temperature was measured using standard four-probe method (Thermo Electricity Test

Foundation item: Project(50572080) supported by the National Natural Science Foundation of China
Corresponding author: WANG Ping; Tel: +86-27-63178509; E-mail: wangping0904@whut.edu.cn

System, AEM-I, Japan) in Ar atmosphere.

3 Results and discussion

3.1 XRD patterns of Ti_2AlC

Fig.1 displays the XRD patterns of samples prepared by hot pressing 0.5TiC/1.5Ti/1.0Al/0.5C at different temperatures. It can be seen from Fig.1(a) that Ti_2AlC is the main crystal phase at 1 300 ℃, and the samples contains quite a large amount of TiC and Ti-Al intermetallics. The XRD result of the sample prepared at 1 400 ℃ (Fig.1(b)) shows the peaks of Ti_2AlC with little Ti_3AlC_2. At the same time, other peaks disappear. The XRD pattern of sample prepared at 1 450 ℃ (Fig.1(c)) is similar to that at 1 400 ℃. With further increasing the temperature, other new phase Ti_3AlC_2 is formed obviously (Fig.1(d)) although the main phase is also Ti_2AlC at 1 500 ℃. Fig.1 indicates that Ti_2AlC with little Ti_3AlC_2 can be synthesized at 1 400 ℃ and 1 450 ℃ by hot pressing TiC, Ti, Al and active carbon.

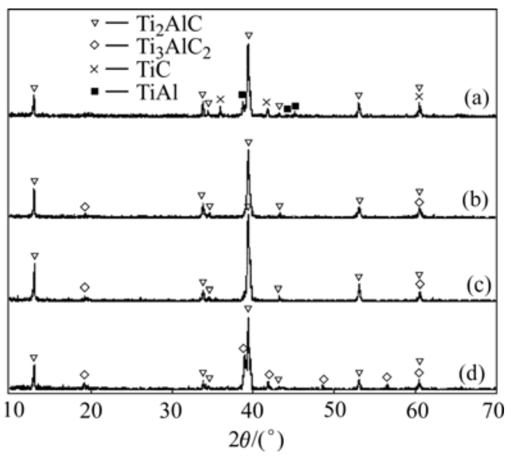

Fig.1 XRD patterns of samples synthesized by hot pressing 0.5TiC/1.5Ti/1.0Al/0.5C powder mixtures at 1 300 ℃ (a), 1 400 (b), 1 450 ℃ (c) and 1 500 ℃ (d)

3.2 Microstructure of Ti_2AlC

Fig.2 shows the microstructures of Ti_2AlC prepared by hot pressing at different temperatures. For the sample prepared at 1 300 ℃, a part of product has a tendency to form layered structure. But the majority is small crystal grains, and the size of them is between 2 and 8 μm. According to the XRD patterns of Fig.1(a), they are TiC and Ti-Al intermetallics. In the sample prepared at 1 400 ℃, the layered Ti_2AlC develops perfectly and layered characteristics become more obvious than that at 1300 ℃. The mean size of layered structure is 15 μm or so. As the synthesis temperature is elevated to 1 500 ℃, however, the edge of layered structure becomes unclear like dissolution and the size (below 10 μm) is smaller than that at 1 400 ℃.

The synthesis procedure of Ti_2AlC by hot pressing

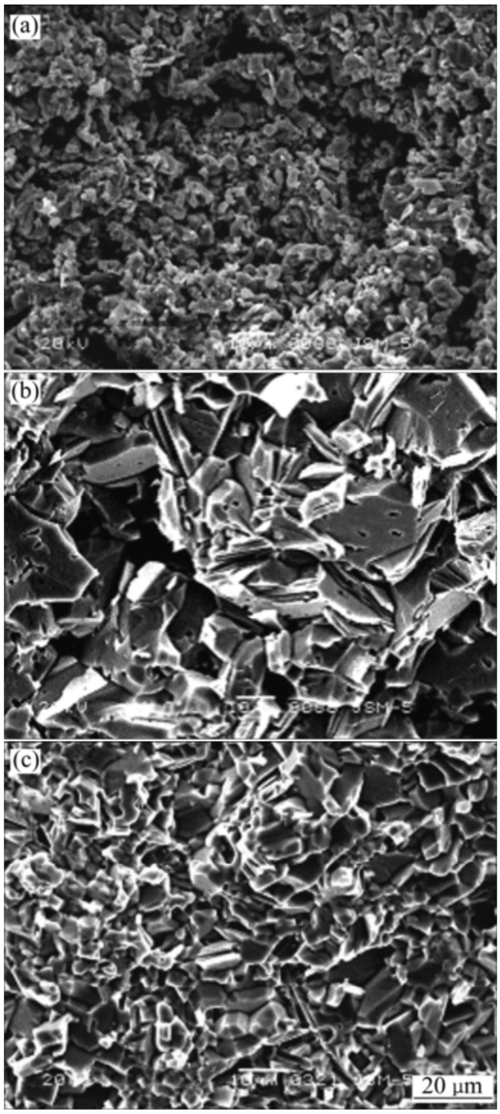

Fig.2 Microstructures of samples fabricated by hot pressing powder mixture at 1 300 ℃ (a), 1 400 ℃ (b) and 1 500 ℃ (c)

can be described as the following possible reactions:

$$Al(s) \rightarrow Al(l) \quad (1)$$

$$Al(l)+Ti(s) \rightarrow \text{Al-Ti intermetallics(s)} \quad (2)$$

$$Ti(s)+C(s) \rightarrow TiC(s) \quad (3)$$

$$TiC(s)+\text{Al-Ti intermetallics} \rightarrow Ti_2AlC(s) \quad (4)$$

$$2Ti_2AlC \rightarrow Ti_3AlC_2\ (s)+\text{Al-Ti intermetallics(s)} \quad (5)$$

According to previous literature[14], TiC is a key intermediate matter during the synthesis of Ti_2AlC. Ti and C must react into TiC to form Ti_2AlC with Al-Ti intermetallics if there is no TiC in raw materials (reaction (1)−(4)). However, because the formation of TiC from Ti and C is a strongly exothermic reaction[15], the temperature of green sample is much higher than that by hot pressing. Furthermore, the vapor pressure of Al in Ti-Al-C system is very high, so the loss of Al increases. If TiC is doped, the heat of reaction released between Ti and C decreases, so the temperature of green sample goes down. Due to the loss of Al decreasing, the synthesizing speed and the purity of Ti_2AlC are improved. But doping too much TiC cannot improve the synthesizing speed of Ti_2AlC when the amount of TiC reaches a value that is enough to form Ti_2AlC. As the temperature reaches about 1 500 ℃, a part of Ti_2AlC will react as reaction (5), and new phase Ti_3AlC_2 appears. Thus, doping TiC will help to improve the synthesizing speed of Ti_2AlC.

3.3 Mechanical properties of Ti_2AlC

As mentioned above the sample prepared at 1 400 ℃ is highly pure and dense Ti_2AlC. Its density is 4.10 g/cm^3 measured by Archimedes method, which is close to the theoretical density of 4.11 g/cm^3. Fig.3 shows the micrograph of fracture surfaces of Ti_2AlC. Rectangular bars for mechanical properties testing were cut from the hot pressed bulk by wire cutting, and the sizes of samples are listed in Table 1.

The test results of mechanical properties can be seen in Table 2. The fracture toughness of Ti_2AlC reaches 7.0 $MPa \cdot m^{1/2}$ and its flexural strength is 384 MPa. Moreover, its Vicker's hardness is between 4.2 GPa and 5.7 GPa.

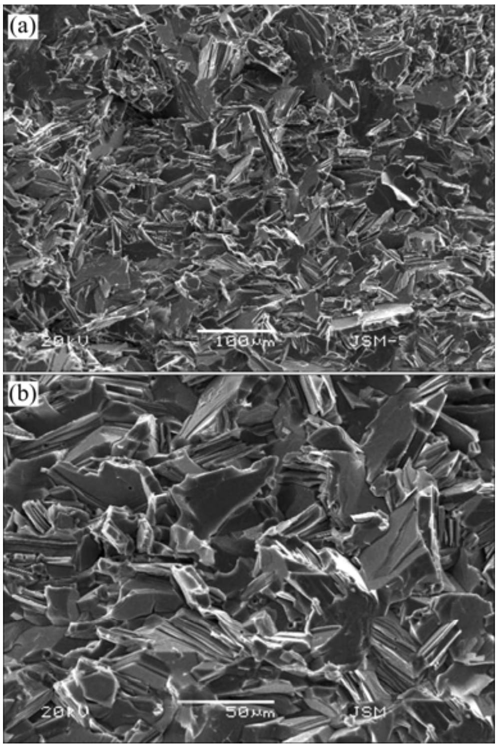

Fig.3 Fractographs of Ti_2AlC samples

Table 1 Size of samples for measurement of mechanical properties

Test of properties	Size/mm	Quantity of samples
Compressive strength	9.0×9.0×27.0	10
Three-point bending	3.0×4.0×36.0	10
Fracture toughness	2.5×5.0×25.0	10

Table 2 Mechanical properties of Ti_2AlC

Compressive strength/ MPa	Flexural strength/ MPa	Fracture toughness/ ($MPa \cdot m^{1/2}$)	Vicker's hardness/ GPa
670	384	7.0	4.2−5.7

3.4 Electrical properties of Ti_2AlC

Fig.4 shows the measured electrical resistivity of Ti_2AlC as a function of temperature. It is seen from the figure that, the electrical resistivity of Ti_2AlC increases linearly with increasing temperature, which is typical of metallic conductors. According to the fitting line of electrical resistivity in Fig.4, the temperature coefficient of electrical resistance of Ti_2AlC in the temperature range of 300−500 K is 2.3×10^{-3} K^{-1}. The electrical resistivity of Ti_2AlC is 0.39 $\mu\Omega\cdot m$ which is corresponded with electrical conductivity of 2.56×10^{6} $\Omega^{-1}\cdot m^{-1}$ at 300 K. They become 0.55 $\mu\Omega\cdot m$ and 1.83×10^{6} $\Omega^{-1}\cdot m^{-1}$ at 480 K respectively. This fact proves that Ti_2AlC exhibits a good electrical conductivity.

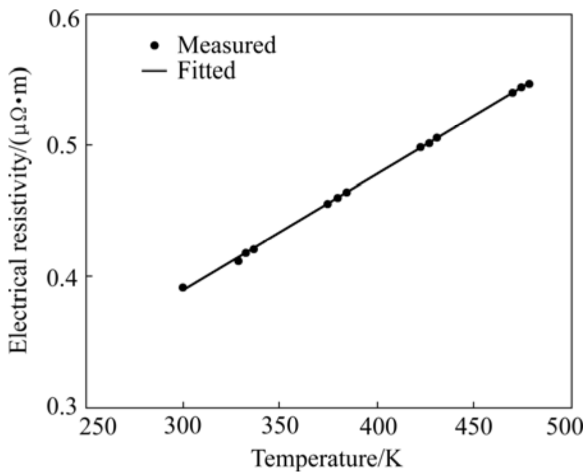

Fig.4 Temperature dependence of electrical resistivity of Ti_2AlC at 300−500 K

4 Conclusions

1) Bulk material of Ti_2AlC containing quite little Ti_3AlC_2 with density of 4.10 g/cm^3 is synthesized by hot pressing TiC, Al, Ti and active carbon at 1 400 ℃ for 60 min under a pressure of 30 MPa. Doping TiC will help to improve the synthesizing speed of Ti_2AlC.

2) The fracture toughness of Ti_2AlC synthesized at 1 400 ℃ is 7.0 $MPa\cdot m^{1/2}$, the flexural strength is 384 MPa at room temperature, and the compressive strength is 670 MPa. It is an excellent electrical conductor at 300− 500 K and the electrical conductivity is 2.56×10^{6} $\Omega^{-1}\cdot m^{-1}$ at room temperature.

References

[1] SCHUSTER J C, NOWOTNY H, VACCARO C. The ternary systems: Cr-Al-C, V-Al-C and Ti-Al-C and the behavior of the H-phases (M_2AlC) [J]. Journal of Solid State Chemistry, 1980, 32: 213−219.

[2] BARSOUM M W, BRODKIN D T, EL-RAGHY T. Layered machinable ceramics for high temperature applications [J]. Scripta Materialia, 1997, 36(5): 535−541.

[3] BARSOUM M W, EL-RAGHY T. A progress report on Ti_3SiC_2, Ti_3GeC_2, and the H-phase, M_2BX [J]. Journal of Materials Synthesis and Processing, 1997, 5(3): 197−216.

[4] RADOVIC M, BARSOUM M W, GANGULY A, ZHEN T, FINKEL P, KALIDINDI S R, LARA-CURZIO E. On the elastic properties and mechanical damping of Ti_3SiC_2, Ti_3GeC_2, $Ti_3Si_{0.5}Al_{0.5}C_2$ and Ti_2AlC in the 300−1 573 K temperature range [J]. Acta Mater, 2006, 54: 2757−2767.

[5] HETTINGER J D, LOFLAND S E, FINKEL P, MEEHAN T, PALMA J, HARRELL K, GUPTA S, GANGULY A, EL-RAGHY T, BARSOUM M W. Electrical transport, thermal transport, and elastic properties of M_2AlC (M=Ti, Cr, Nb and V) [J]. Physical Review B, 2005, 72:115−120.

[6] MEI Bing-chu, ZHOU Wei-bing, ZHU Jiao-qun, HONG Xiao-lin. Synthesis of high-purity Ti_2AlC by spark plasma sintering(SPS) of the elemental powders [J]. Journal of Materials Science, 2004, 39(4): 1471−1472.

[7] HONG Xiao-lin, MEI Bing-chu, ZHU Jiao-qun, ZHOU Wei-bing. Fabrication of Ti_2AlC by hot pressing of Ti, TiC, Al and active carbon powder mixtures [J]. Journal of Materials Science, 2004, 39(5): 1589−1592.

[8] BARSOUM M W, ALI M, EL-RAGHY T. Processing and characterization of Ti_2AlC, Ti_2AlN, and $Ti_2AlC_{0.5}N_{0.5}$ [J]. Metallurgical and Materials Transactions A, 2000, 31A(7): 1857−1865.

[9] BARSOUM M W, SALAMA I, EL-RAGHY T, GOLCZEWSKI J, PORTER W D, WANG H, SEIFERT H J, ALDINGER F. Thermal and electrical properties of Nb_2AlC, $(Ti, Nb)_2AlC$ and Ti_2AlC [J]. Metallurgical and Materials Transactions A, 2002, 33(9): 2775−2779.

[10] ZHOU Y C, SUN Z M. Electronic structure and bonding properties of layered machinable Ti_2AlC and Ti_2AlN ceramics [J]. Physical Review B, 2000, 61(19): 12570−12573.

[11] WANG X H, ZHOU Y C. Solid-liquid reaction synthesis and simultaneous densification of polycrystalline Ti_2AlC [J]. Zeitschrift Fur Metallkunde, 2002, 93(1): 66−71.

[12] ZHOU Y C, WANG X H. Deformation of polycrystalline Ti_2AlC under compression [J]. Materials Research Innovation, 2001, 5: 87−93.

[13] WANG Ping, MEI Bing-chu, HONG Xiao-lin, ZHU Jiao-qun, ZHOU Wei-bing. Effects of TiC addition on the synthesis of Ti_2AlC by hot pressing [J]. The Chinese Journal of Nonferrous Metals, 2005, 15(10):1550−1553. (in Chinese)

[14] TOMOSHIGE R, MATSUSHITA T. Production of titanium-aluminum-carbide ternary composites with dispersed fine TiC particles by combustion synthesis and their microstructure observation [J]. Journal of the Ceramic Society of Japan, 1996, 104(1206): 94−100.

[15] SUN Z M, AHUAJA R, LI S, SCHNEIDER J M. Structure and bulk modulus of M_2AlC (M=Ti, V, and Cr) [J]. Applied Physics Letters, 2003, 83(5): 899−901.

(Edited by YUAN Sai-qian)