

AlZrC₂ synthesis

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Abstract

AlZrC₂ was synthesized using a direct solid-state method. Various types of carbon were used as starting materials together with aluminum zirconium carbide powders. The effect of different carbon on the formation of Al–Zr–C compounds formation were investigated. The atomic arrangement of carbon affects the Al–Zr–C compounds formation, i.e., crystalline carbon facilitates Zr₂Al₃C₅ formation while glassy carbon facilitates AlZrC₂ formation. High purity AlZrC₂ was prepared from the appropriate mol ratio of Al:ZrC:glassy carbon by heating in vacuum at 1600 °C for 1 h. However, a little amount of Zr₂Al₃C₅ still formed due to graphitization of glassy carbon at high temperature. Glassy carbon from sugar showed a lower degree of crystallization (graphitization) than glassy carbon from phenolic resin. Finally, the obtained two phase specimen was ground and heat treated again using pulse electric current sintering to produce single AlZrC₂.

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1. Introduction

Carbides are extensively used in various fields due to their excellent properties. For instance, SiC [1] is widely used as an engineering material because of its high-temperature fracture strength and creep resistance. The remarkable thermal stability and chemical inertness of B₄C make it well suited for applications requiring high stiffness or good wear resistance [2]. ZrC is an important structural material owing to its high strength and good corrosion resistance [3].

Several complex carbides have recently been synthesized and studied. For example, Al₈B₄C₇ [4] has remarkable antioxidant properties in carbon-containing refractory materials. Michalenko et al. found the AlZrC₂ phase in 1978 [5], but little has been reported about this material.

Hashimoto et al. [6] produced a composite material of AlZrC₂ and Al, and characterized its mechanical properties. They prepared the AlZrC₂ by heating a mixture of Al and ZrC, followed by leaching with HCl. After synthesizing, however, ZrC still remained in the sample.

We synthesized high purity AlZrC₂ using a direct solid-state method.

2. Experimental procedure

2.1. Starting materials

The starting materials we used were

- (1) Al, 99.9% pure; 10- μ m mean particle size (High Purity Chemicals Co., Ltd.).
- (2) ZrC, 95% pure (High Purity Chemicals Co., Ltd.).
- (3) Graphite, 99.7% pure; 5- μ m mean particle size (High Purity Chemicals Co., Ltd.).

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Table 1
Al–ZrC–C mixtures with graphite as source of carbon

Composition	Mol ratio of starting materials (Al:ZrC:C)	Mol ratio of elements (Al:Zr:C)
A	1:1:1	1:1:2
B	3.5:3.5:3	3.5:3.5:6.5
C	4:4:2	4:4:6
D	4.5:4.5:1	4.5:4.5:5.5
E	3.5:3:3.5	3.5:3:6.5
F	4:2:4	4:2:6
G	4.5:1:4.5	4.5:1:5.5

- (4) Amorphous carbon. In this work, we used three kinds of amorphous carbon, i.e.,
- Prepared by heating phenolic resin in a muffle furnace at 400 °C for 30 min and grinding it into small pieces, reheating it at 400 °C for 5 min, then grinding it into a fine powder.
 - Prepared by heating sugar in a porcelain crucible with Bunsen burner for 6 h and grinding it into small pieces, reheating it for 6 h, then grinding it into a fine powder.
 - Carbon black, as received from industry (specific surface area, 125 m²/g).

2.2. Sample preparation and heating

The starting materials were mixed in acetone at various mol ratios as shown in Tables 1 and 2 and Fig. 1. The mixtures were pressed into pellets of ~1.8 g, CIPed at 100 MPa, and heated under two different conditions:

- Ar atmosphere: The samples were placed in a refractory block lined with carbon paper and covered with powder having the same composition to prevent surface oxidation [6]. They were heated from room temperature to 1600 °C for 1 h at a heating rate 10 °C/min.
- Vacuum: The samples were pre-fired in Ar atmosphere as described above at 900 °C for 1 h to prevent Al vapor from attacking the carbon lining of the vacuum chamber. After pre-firing, the covered powder was removed. The samples were wrapped in carbon paper and placed in a carbon crucible before heating in vacuum at a heating rate

Table 2
Al–ZrC–C with amorphous carbon as source of carbon

Composition	Mol ratio of starting materials (Al:ZrC:C)
H (resin)	3:3:4
I (resin)	4:3:3
J (resin)	5.5:3:1.5
K (resin)	6:3:1
L (resin)	3:2.5:4.5
M (resin)	3.5:2.5:4
N (resin)	3:2:3
O (resin)	5:2.5:2.5
P (resin and carbon black)	35:28:37
Q (resin and sugar)	21:14:15

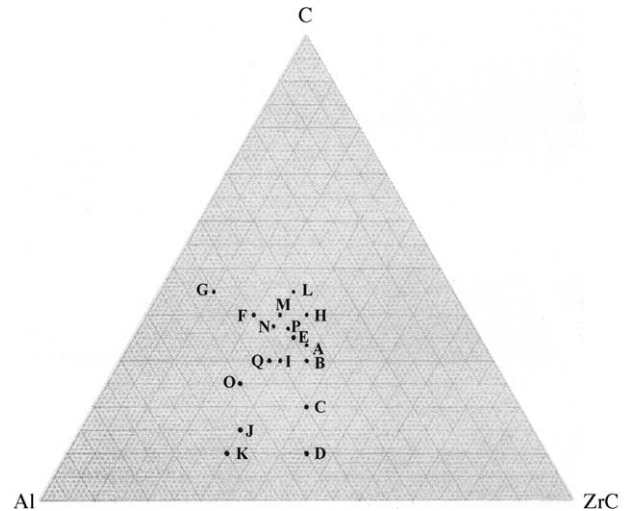


Fig. 1. Composition diagram of Al–ZrC–C mixtures.

10 °C/min from room temperature to the maximum temperature for 1 h.

2.3. Characterization

Samples were crushed and ground into fine powder. Phase change was characterized by XRD (Shimadzu XD-X1). Thermal analysis was carried out by DTA/TG (Rikaku Thermoplus TG8120) from room temperature to 1200 °C in air to check the exothermic reaction of the residual ZrC around 500–600 °C [7]. After synthesis, fresh crack samples were investigated by SEM (JEOL JSM-6360LV).

3. Results and discussion

3.1. Effect of composition on phase formation

Al–ZrC–graphite mixtures with various mol ratios were heated in Ar atmosphere. Phases formed within the Al–ZrC–graphite pellets after firing at 1600 °C for 1 h are shown in Fig. 2. Phase formation depends on the ratio of the starting materials. Fig. 2a shows the phase formation for an Al:ZrC mol ratio of 1 at varying amounts of graphite. Fig. 2b shows phase formation for an Al:graphite mol ratio of 1 at varying amounts of ZrC. In the middle zone of the composition diagram (Fig. 1), Zr₂Al₃C₅ formed after firing. This formation did not depend on the Al:ZrC:graphite ratio, as shown by the XRD patterns of the compositions A, B and E in Fig. 2a and b. The phase formation after firing was completely different away from this middle zone, i.e., closer to the edges of the composition diagram. At a constant mol ratio of Al to ZrC with lower graphite content (compositions C and D in Fig. 2a), no Zr₂Al₃C₅ was found. At a constant mol ratio of Al to graphite with lower ZrC content (compositions F and G in Fig. 2b), no AlZrC₂ was found

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