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Original Research Paper

The effect of processing parameters in the carbothermal synthesis of titanium diboride powder

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ABSTRACT

The mechanism of the carbothermal method for synthesizing titanium diboride (TiB_2) powder has been studied. Mixtures of TiO_2, H_3BO_3 and carbon were heated in an argon atmosphere at 1000–1600 °C. The effect of the molar ratio and holding time on the phase evolution was studied by X-ray diffraction. The products were also characterized by scanning electron microscopy observations and particle size measurements.

For a composition with a molar ratio of TiO_2:H_3BO_3:C = 1:2.4:5 heated for 1 h, the simultaneous presence of TiC and TiB_2 phases at 1100 °C and the transformation of TiO_2 to Ti_2O_3 at 1200 °C and higher confirms that TiB_2 synthesis is based on a TiC formation mechanism, in which TiC may be formed from a reaction between TiO_2 or Ti_2O_3 and carbon. Then TiC may react with liquid B_2O_3 and/or gaseous B_2O_2 to form the TiB_2 phase. The reaction is completed at 1500 °C. Also by increasing the molar ratio of boric acid to 3, the impurities decreased considerably and pressing of the material had an obvious effect on decreasing the impurities, due to an increase of the surface contact of particles, which causes an effective inhibition of boron escape from the reaction chamber. Under these experimental conditions, a relatively narrow size distribution of TiB_2 particles was produced. When the reaction time increased to 1.5–2 h, grain growth of particles occurred. Therefore, a wider distribution of particle size was obtained.

1. Introduction

Titanium diboride (TiB_2) is an interesting material for its high melting point, high hardness, moderate density, high Young's modulus and low thermal expansion co-efficient [1–3]. It has been used in applications such as cutting tools, a wear resistance material, for metal melting crucibles and electrodes [3–5]. TiB_2 powder is prepared by a variety of methods such as the borothermic reduction of titania, fused-salt electrolysis, solution phase processing or carbothermal reduction [6–8].

Among the above mentioned processing techniques, the carbothermal reduction process is commercially used as the cheapest method because of inexpensive raw materials and it is a simple process. Also for each mole of TiB_2 produced, the process generates CO gas, which will release energy when burnt with oxygen [8]. Carlsson et al. [9] found a carbothermal reduction process for the synthesis of TiB_2, which has a vapour–liquid–solid growth mechanism. They found that TiB_2 was formed at the temperatures $\geqslant$1300 °C. Pei and Xiao [10] revealed that TiB_2 is produced by the reduction of TiC and B_2O_3 when the reaction temperature goes beyond 1367 °C.

The overall reaction of the carbothermal synthesis of TiB_2 powder is as follows [10]:

$$TiO_2 + H_3BO_3 + 5C = TiB_2 + 5CO\ (gas) + 3H_2O\ (gas) \quad (1)$$

Also the TiC phase may be formed from the reaction of TiO_2 and carbon as below [10]:

$$TiO_2 + 3C = TiC\ (s) + 2CO \quad (2)$$

B_2O_3 is formed from the decomposition of H_3BO_3. A significant loss of boron may be expected in the form of B_2O_3 and B_2O at the temperatures above 1127 °C [10,11].

The resulting TiC reacts with B_2O_3 (l) to form TiB_2 as follows [11]:

$$TiC\ (s) + B_2O_3\ (l) + 2C = TiB_2\ (s) + 3CO\ (g) \quad (3)$$

At a high temperature, liquid B_2O_3 reacts with carbon to form gaseous B_2O_2:

$$B_2O_3\ (l) + C = B_2O_2\ (g) + CO \quad (4)$$

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