



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 (TiB₂) powder has been studied. Mixtures of TiO₂, H₃BO₃ 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 TiO₂:H₃BO₃:C = 1:2.4:5 heated for 1 h, the simultaneous presence of TiC and TiB₂ phases at 1100 °C and the transformation of TiO₂ to Ti₂O₃ at 1200 °C and higher confirms that TiB₂ synthesis is based on a TiC formation mechanism, in which TiC may be formed from a reaction between TiO₂ or Ti₂O₃ and carbon. Then TiC may react with liquid B₂O₃ and/or gaseous B₂O₂ to form the TiB₂ 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 TiB₂ 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.

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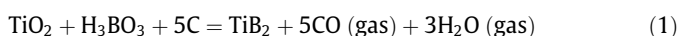
1. Introduction

Titanium diboride (TiB₂) 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]. TiB₂ 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 TiB₂ 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 TiB₂, which has a vapour–liquid–solid growth mechanism. They found that TiB₂ was formed at the temperatures

≥ 1300 °C. Pei and Xiao [10] revealed that TiB₂ is produced by the reduction of TiC and B₂O₃ when the reaction temperature goes beyond 1367 °C.

The overall reaction of the carbothermal synthesis of TiB₂ powder is as follows [10]:

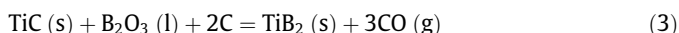


Also the TiC phase may be formed from the reaction of TiO₂ and carbon as below [10]:



B₂O₃ is formed from the decomposition of H₃BO₃. A significant loss of boron may be expected in the form of B₂O₃ and B₂O at the temperatures above 1127 °C [10,11].

The resulting TiC reacts with B₂O₃ (l) to form TiB₂ as follows [11]:



At a high temperature, liquid B₂O₃ reacts with carbon to form gaseous B₂O₂:



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