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

B. Shahbahrami a,⁎, F. Golestani Fard b, A. Sedghi c

a Faculty of Engineering, Islamic Azad University Saveh Branch, Saveh, Iran
b Faculty of Materials Science and Engineering, Iran University of Science and Technology, Tehran, Iran
c Faculty of Engineering, International Imam Khomeini University, Qazvin, Iran

Available online 15 March 2011

Article info

Article history:
Received 23 October 2010
Received in revised form 27 February 2011
Accepted 1 March 2011
Available online 15 March 2011

Keywords:
Titanium diboride powder
Carbothermal synthesis
Mechanism
Non-oxide powder

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 confirm 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.

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 coefficient [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]:

\[
\text{TiO}_2 + 3\text{C} = \text{TiC} + 2\text{CO} \quad (2)
\]

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

\[
\text{TiO}_2 + 5\text{C} = \text{TiB}_2 + 5\text{CO} \quad (1)
\]

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]:

\[
\text{TiC} + \text{B}_2\text{O}_3 (l) + 2\text{C} = \text{TiB}_2 + 3\text{CO} \quad (3)
\]

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

\[
\text{B}_2\text{O}_3 (l) + \text{C} = \text{B}_2\text{O}_2 (g) + \text{CO} \quad (4)
\]