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Synthesis of Nano Titanium diboride Powders by Carbothermal Reduction

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1. Introduction

Titanium diboride (TiB_2) has attracted great interest for thermal, mechanical, electrical and chemical properties. As a result, TiB_2 has broad applications in cutting tool composites, wear resistant parts and electrodes. Normally, TiB_2 is produced by reducing titanium oxide with either boron oxide and carbon, boron and carbon, boron carbide or an alkali metal and boron oxide. These processes typically require long reaction periods, during which the TiB_2 particles are sintered.

The present study describes the production of ultrafine sized TiB_2 particles by carbothermal reduction, using precursors prepared from TiO_2, B_2O_3 and carbon resin.

2. Experimental procedure

Ultrafine TiB_2 powders were synthesized by carbothermal reduction. TiO_2, B_2O_3 powders and carbon resin were used as starting materials. The average size of TiO_2 powder was 40~50nm. TiO_2, B_2O_3 and carbon resin were mixed at prescribed ratios in acetone, and planetary milling for 2h in engineering plastic jar. After milling, the slurry was dried in a rotary evaporator at 70℃ and granulated using 100-mesh sieve. The mixture was put into graphite crucible and the TiB_2 powders were synthesized in a graphite vacuum furnace at 1250~1500℃ in vacuum and flowing argon gas. The synthesizing reaction can be expressed with the following equation:

$$TiO_2(s) + B_2O_3(s) + 5C(s) \rightarrow TiB_2(s) + 5CO(g)$$

X-ray diffraction (XRD) was performed to identify the phase of producted particles. The average size was measured by scanning electron microscopy (SEM). The structure of the particles was determined by analyzing the high-resolution transmission electron microscopy (HRTEM).

3. Results and discussion

The precursor was prepared at a molar composition of TiO_2:B_2O_3:C=1:2:5. TiB_2 was formed at temperature of 1250℃. Phases in the powder produced at 1250℃ were TiB_2 and TiC which were determined by XRD. Only TiB_2 phase was found in the temperature range 1300~150

0℃. But the surface of particles was covered with oxide amorphous layer which was determined by XPS and HRTEM. The average particle size was ~1㎛ calcination at 1300℃ for 10min. As the spex milling for 30h in a engineering plastic jar with Si_3N_4 ball, the average particle size was decreased to 100~200㎚.