

Different Synthetic Routes of Group Six (VIB) Nano-Metal Borides

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Abstract

For the first time, the synthetic comparison between the solid and liquid phases of metal borides was subjected to empirical investigation. The magnesiothermic reaction of CrO_3 , B_2O_3 , and Mg yielded the only Cr_2O_3 . Similarly, the direct reaction of CrO_3 or CrCl_3 with elemental boron yielded chromium oxides. On the other hand, direct reaction of pure Cr metal with elemental boron in 1:1, 1:2 and 1:3 ratios, showed that only 1:3 ratio, produced a mixture of Cr_2B , Cr_2O_3 , and CrO_3 (minimal oxides). Going down group six, (VIB) MoO_3 and WO_3 , reacted directly with elemental boron in a muffle furnace at 500, 700 and 1000°C for 2, 2 and 15 hours respectively, yielding black beautiful nanocrystals of Mo_2B_4 , MoB , WB_4 , and W_2B_5 . The liquid phase reaction of molybdenum pentachloride, MoCl_5 , with sodium borohydride, NaBH_4 (1:5 ratio), produced black nanocrystals and nanorods of MoB and MoB_4 . Reacting chromium trichloride with sodium borohydride, NaBH_4 (1:3 ratio), in solution, produced pure black nanocrystals and nanorods of Cr_2B , while the reaction of chromium dichloride with sodium borohydride, NaBH_4 (1:2 ratio), in solution, produced a mixture of Cr_2B and Cr_2O_3 .

Keywords: Nanocompounds; Metal Borides; Nanowires; Nanocrystals

Introduction

A great number of transition metal borides were synthesized. That is due to their special characters including high hardness, high melting points, high-temperature strength, corrosion resistance, chemical stability, wear resistance and electrical properties [1-6]. Several chromium borides are known by their wide range of atomic ratios (Cr_3B_3 , CrB , Cr_3B_4 , Cr_2B_3 and CrB_2 [7-9]). Two among these, CrB and CrB_2 , are more important since they are closer to the properties mentioned above. Synthetic preparation of chromium boride is a very difficult process, because binary side products are always there including chromium oxides. Several synthetic methods have been tried like combustion synthesis [8,9], thermal evaporation process [10], and pulsed magnetron sputtering [11].

On the other hand, molybdenum and tungsten borides have similar properties to those of chromium borides, except solubility of the oxides in water where Molybdenum is sparingly soluble while tungsten is insoluble. Molybdenum forms MoB , Mo_2B , MoB_2 , Mo_2B_5 and MoB_4 which synthesized through different methods, such as mechanochemical [12], electrochemical [13,14], and hydrothermal methods [15], self-propagating high-temperature synthesis, with highly exothermic reaction [16,17]. Tungsten forms W_2B , W_2B_3 , WB , WB_4 , and WB_{12} , which are prepared by many different methods such as chemical vapor deposition, solid-state reaction, ionic melts, self-propagating high-temperature synthesis, arc plasma melting and mechanochemical method [18-21].

Even though, as mention above, several synthetic methods were applied to produce borides, almost none of these studies proved effective in the formation of single phase borides, rather a mixture of boride phases was formed [22,23]. In this study, two different methods were carried out in the synthetic processing of chromium, molybdenum and tungsten borides, namely, solid states and wet reaction.

Experimentation/Empirical Analysis

Reagents

Unless otherwise specified, reagent grade chemicals were employed. The precursor materials were Chromium trioxide (CrO_3 , BDH), Molybdenum (VI) Oxide (MoO_3 , Alfa Aesar), and Tungsten Oxide (WO_3 , Alfa Aesar). Boron Powder (Amorphous) (B, Loba Chemie),

Boron (III) Oxide (B_2O_3 , Alfa Aesar). Magnesium Powder (BDH). Sodium Borohydride (NaBH_4 , BDH), Chromium(III) Chloride (Anhydrous) (CrCl_3 , Fluka AG, Chem), Chromium(III) Chloride Hexahydrate ($\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$, BDF), and Hydrochloric acid (HCl, Sigma-Aldrich, fuming >37%). Chromium Powder (Cr, Alfa Aesar).

The Powder X-ray diffraction (XRD) measurement was carried out with a Bruker D8 Advance diffractometer ($\text{CuK}\alpha$ $\lambda=1.54$ Å; Ni filter; 40 KV, 40 mA; Divergence slit: 1 mm, LynxEye one-dimensional detector, Detector slit: 8 mm). Scanning Electron Microscope and Transmission Electron Microscope (SEM and TEM) were performed by employing a microscope of model Titan 80-300 CT from FEI Company (Hillsboro, OR), which was equipped with a field emission gun (FEG) and a charged-Couple Devices (CCD) camera of $4\text{k} \times 4\text{k}$ pixels. Furthermore, the analyses of all samples were performed by operating the microscope at 300 kV accelerating voltage in order to take full advantage of the highest spatial resolution possible from this microscope. An aperture of 100 microns was also inserted into the back-focal plane (BPF) of the objective lens, to improve the image contrast. The Magnetization measurement was carried out in an ever-cool Squid-Vibrating Sample Magnetometer (SVSM), from the manufacturer Quantum Design, USA.

Synthesis

Solid phase

Preparation of metal borides using boron oxide and magnesium

Chromium boride



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