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Synthesis and Characterization of Fe, Ru and Os Metal Borides

Omar Ali Al-Zain* and Al-Masoudi Reem Soliyman

Chemistry Department, Faculty of Science, King Abdulaziz University, Jiddah, Saudi Arabia

Abstract

Direct reaction of Fe_2O_3 at 500, 700 and 1000°C for two, two and 15 hours, respectively, with elemental boron in a muffle furnace yielding black beautiful nanorods of FeB crystals with high yield of 72% (as proved by xrd, tem and Magnetic hysteresis). Going down the group of the transition metals $RuCl_3$ and $OsCl_3$ reacted directly with elemental boron in a muffle furnace at 500, 700 and 1000°C for two, two and 15 hours, respectively yielding nanorods crystals (as proved by xrd, tem and Magnetic hysteresis) of RuB_2, Ru_2B_3 (yield was 68%) and OsB_2 (yield was 56%).

Keywords: Nanocomponds; Metal borides; Nanowires; Nanocrystals

Introduction

Boron is used for over 200 inorganic compounds, especially metal borides. Metal borides, which are formed by transition metals or alkaline earth metals, are indispensable materials to develop electronic or nano-ordered devices, because they are mechanically hard materials with high melting points and good corrosion resistance [1].

Metal borides stay mostly uncommon for chemists, in spite of a specific set of very good properties as semiconductors and catalysts, but also with bulk boride noncompliant conductive ceramics, hard magnets, superconductors, and hard materials that already supplied the worldwide industrial market with phosphide. Fundamental property aspects of bulk metal borides and metal phosphides are also the focus of many researchers: conduction, luminescence, field emission [2]. Thermoelectric [3] and magnetic [4] properties, ultra-hardness [5] and behaviors as diffusion barriers. Iron boride is attractive because of the magnetic properties and catalytic properties [6]. The development of iron boride change coatings on ferrous substrates is alluring for an extensive variety of applications [7,8].

Iron boride transformation coatings can be synthesized in numerous ways, including molten salt boron izing, gas boron izing, with and without electrolysis [9,10]. In the Ru-B system, RuB, RuB_2, Ru_7B_3 and Ru_2B_3 were acquired experimentally [11-13]. Recently, OsB_2 was prepared at ambient conditions [14] and the consolidation of boron into the osmium lattice gives rise to a significant enhancement of the hardness. The blend of discernible mechanical properties and the low-cost prepare condition recommends that Ru and Os borides might be good candidates for hard materials. Addition of more boron reinforces the mechanical properties of metal borides.

In an effort to find a better synthetic rout to produce nanocrystals of metal borides. We are reporting here the synthesis and characterization of Fe, Ru and Os Metal Borides following our previous publications in this journal [15,16].

Experimentation/Empirical Analysis

Reagents

Unless otherwise specified, reagent grade chemicals were employed. The precursor materials were Iron Oxide (Fe_2O_3, Sigma-Aldrich), Ruthenium (III) Chloride ($RuCl_3$, 36-40% Ru ACROS Organic), Osmium (III) Chloride ($OsCl_3$, ACROS Organic). Boron Powder (Amorphous) (B, Loba Chemie). Hydrochloric acid (HCl, Sigma-Aldrich, fuming >37%).

The Powder X-ray diffraction (XRD) measurement was carried out with a Bruker D8 Advance diffractometer (CuKα λ=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 4k x 4k 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

Preparation of metal borides using metal oxide and boron powder

Iron boride: 7.9844 g of Iron (III) Oxide (50 mmol) and 10.811 g Amorphous Boron Powder (250 mmol), were mixed in a porcelain crucible and placed in a muffle furnace. Temperature gradually raised starting 500°C for two hours then 700°C for two hours and finally 1000°C for 15 hours. After cooling to the room temperature in a muffle furnace, the mixture was leached with 1 N HCl at 100°C for three hour under magnetic stirring. Solution was filtered and washed with distilled water and ethanol. The precipitate was then treated with 1 N NH_4Cl at 100°C for three hour under magnetic stirring. Solution was filtered and washed with distilled water and ethanol and dried in a vacuum oven at 150°C for 3 hours. The yield is 72%.

$$Fe_2O_3 + 4\ B \xrightarrow[500\text{-}1000\,^{\circ}C]{\Delta} 2\ FeB + B_2O_3$$

***Corresponding author:** Omar Ali Al-Zain, Chemistry Department, Faculty of Science, King Abdulaziz University, Jiddah, Saudi Arabia, Tel: 966555600872; E-mail: alzain2005@yahoo.com

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Preparation of metal borides using metal chlorides and boron powder

***Ruthenium boride*:** 0.6223 g of Ruthenium (III) Chloride (1 mmol) and 0.8649 g Amorphous Boron Powder (10 mmol), were mixed in a porcelain crucible and placed in a muffle furnace. Temperature gradually raised starting 500°C for two hours then 700°C for two hours and finally 1000°C for 15 hours. After cooling to the room temperature in a muffle furnace, the mixture was leached with 1 N HCl at 100°C for three hour under magnetic stirring. Solution was filtered and washed with distilled water and ethanol. The precipitate was then treated with 1 N NH_4Cl at 100°C for three hour under magnetic stirring. Solution was filtered and washed with distilled water and ethanol and dried in a vacuum oven at 150°C for 3 hours. The yield is 68%.

$$3\ RuCl_3 + 8\ B \xrightarrow[500\text{-}1000^{\circ}C]{\Delta} RuB_2 + Ru_2B_3 + 3\ BCl_3$$

Osmium boride

0.5932 g of Osmium (III) Chloride (2 mmol) and 0.6487 g Amorphous Boron Powder (20 mmol), were mixed in a porcelain crucible and placed in a muffle furnace. Temperature gradually raised starting 500°C for two hours then 700°C for two hours and finally 1000°C for 15 hours. After cooling to the room temperature in a muffle furnace, the mixture was leached with 1 N HCl at 100°C for three hour under magnetic stirring. Solution was filtered and washed with distilled water and ethanol. The precipitate was then treated with 1 N NH_4Cl at 100°C for three hour under magnetic stirring. Solution was filtered and washed with distilled water and ethanol and dried in a vacuum oven at 150°C for 3 hours. The yield is 56%.

$$OsCl_3 + 3\ B \xrightarrow[500\text{-}1000^{\circ}C]{\Delta} OsB_2 + BCl_3$$

Results and Discussion

Two different preparation methods in the solid phase were applied, metal oxide and metal chloride mixed with elemental boron in muffle furnace.

Iron oxide, Fe_2O_3, and elemental boron were reacted in a muffle furnace at 1000°C for 15 hours under argon atmosphere. Leaching, filtering and drying the product yielding beautiful black crystals of FeB as it could be seen in XRD (Figure 1a). TEM (Figure 1b) clearly shows nanocrystals and nanorods. Magnetic hysteresis of FeB reflects superparamagnetic properties [17] which indicates the nanosystem (Figure 1c).

Going down group eight (VIIIB) Ruthenium chloride, $RuCl_3$, reacts with elemental boron in muffle furnace at 1000°C for 15 hours under a blanket of argon. Leaching, filtering and drying the product yielding beautiful black nanorods crystal mixture of RuB_2 and Ru_2B_3 as it could be seen in XRD (Figure 2a). TEM (Figure 2b) clearly shows nano and nanorod crystals. Magnetic hysteresis of the product of RuB_2 and Ru_2B_3 reflects superparamagnetic properties (Figure 2c).

Similarly Osmium chloride, $OsCl_3$, reacted with elemental boron in muffle furnace at 1000°C for 15 hours under a blanket of argon. Leaching, filtering and drying the product yielding beautiful black nanorod crystals of OsB_2 as it could be seen in XRD pattern (Figure 3a). SEM (Figure 3b) shows a homogenous cluster of crystals. TEM (Figure 3c) shows nano crystals and nanorod. Magnetic hysteresis of the, OsB_2, product indicates superparamagnetic properties (Figure 3d).

Conclusion

Direct reaction of iron oxide and ruthenium and osmium chlorides with elemental boron produced a nanocrystals and nanorods of metal borides, Solid phase reaction of group eight (XIII) proved that first, second and third rows transition metals work very well. Very encouraging results where the yields were 72, 68 and 56% for iron, ruthenium and osmium respectively. High purity was obvious from XRD patterns. TEM and magnetic hysteresis superparamagnetism clearly prove nanocrystals and nanorods.

In previous work of group V (V, Nb and Ta) [15] and group XI (Cr, Mo and W) [16] solid phase worked fine with second and third rows of transition metals, while in case of group XIII (Fe, Ru and Os) solid phase worked fine with first, second and third rows of transition metals

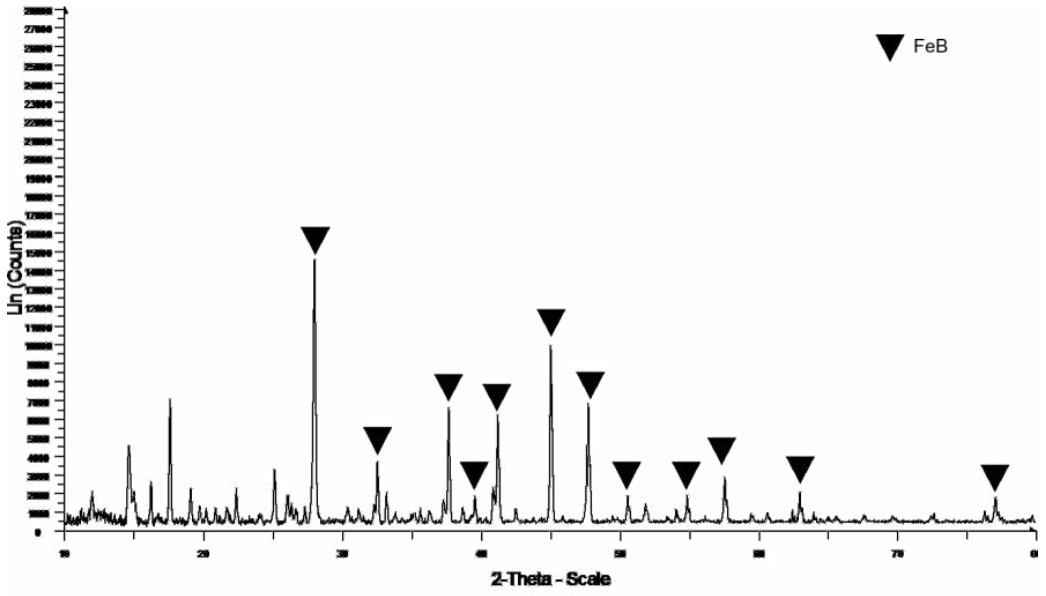

Figure 1a: XRD spectra of FeB produced by direct reaction of Fe_2O_3 and B.

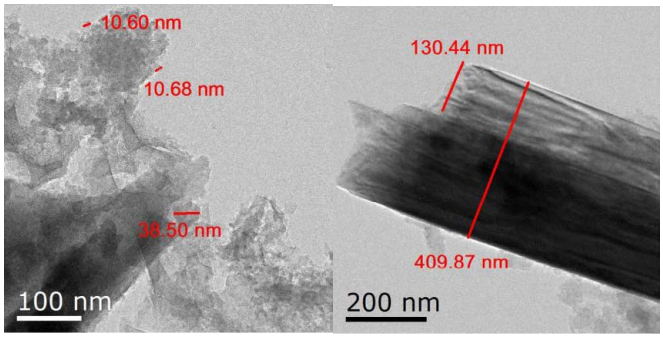

Figure 1b: TEM image of beautiful nanorods crystals FeB.

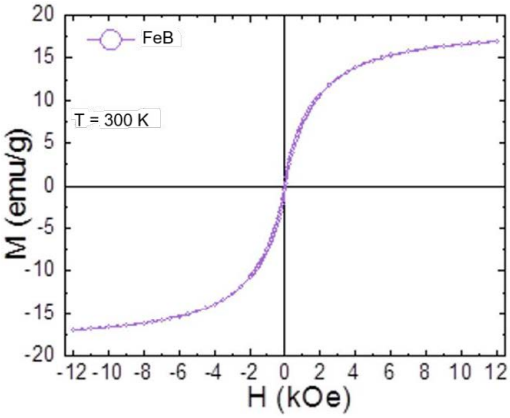

Figure 1c: Magnetic hysteresis superparamagnetism of FeB.

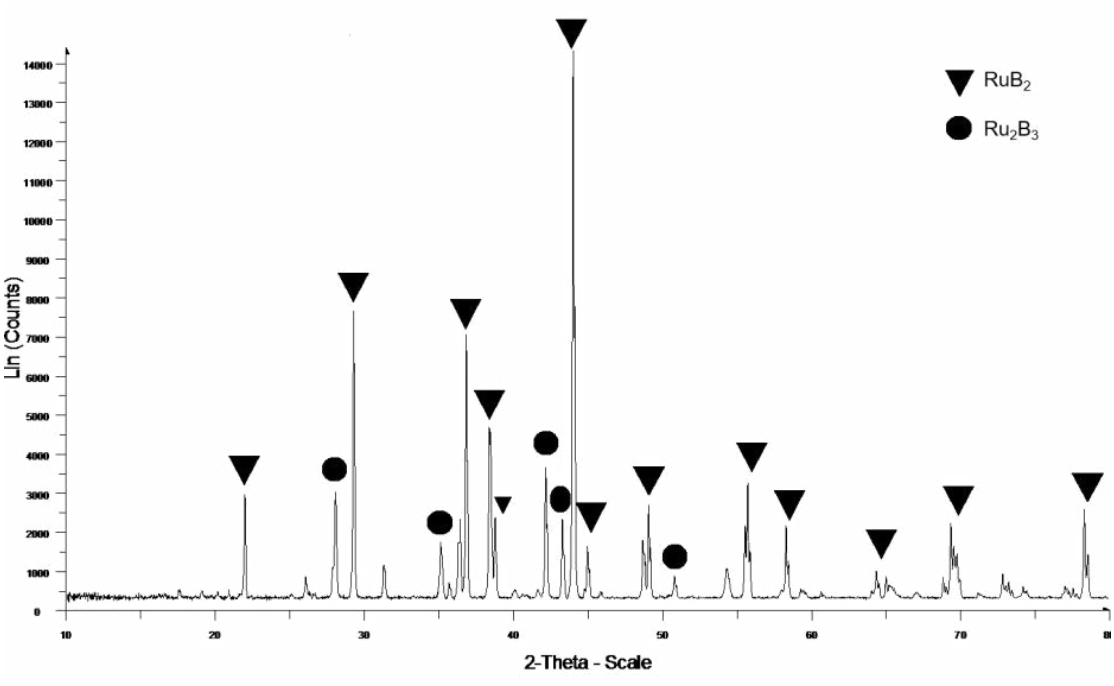

Figure 2a: XRD spectra of black crystals of RuB_2 and Ru_2B_3 produced by direct reaction of $RuCl_3$ and B.

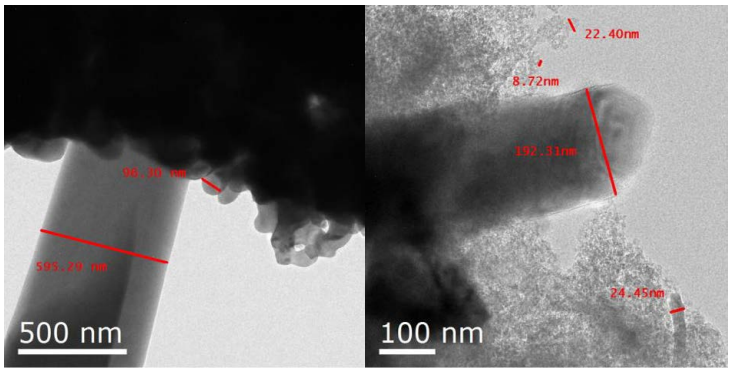

Figure 2b: TEM image of nanorods crystals of RuB_2 and Ru_2B_3.

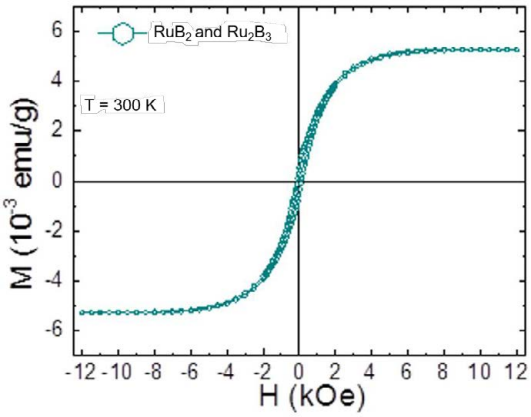

Figure 2c: Magnetic hysteresis shows superparamagnetism of RuB_2 and Ru_2B_3 nanorods.

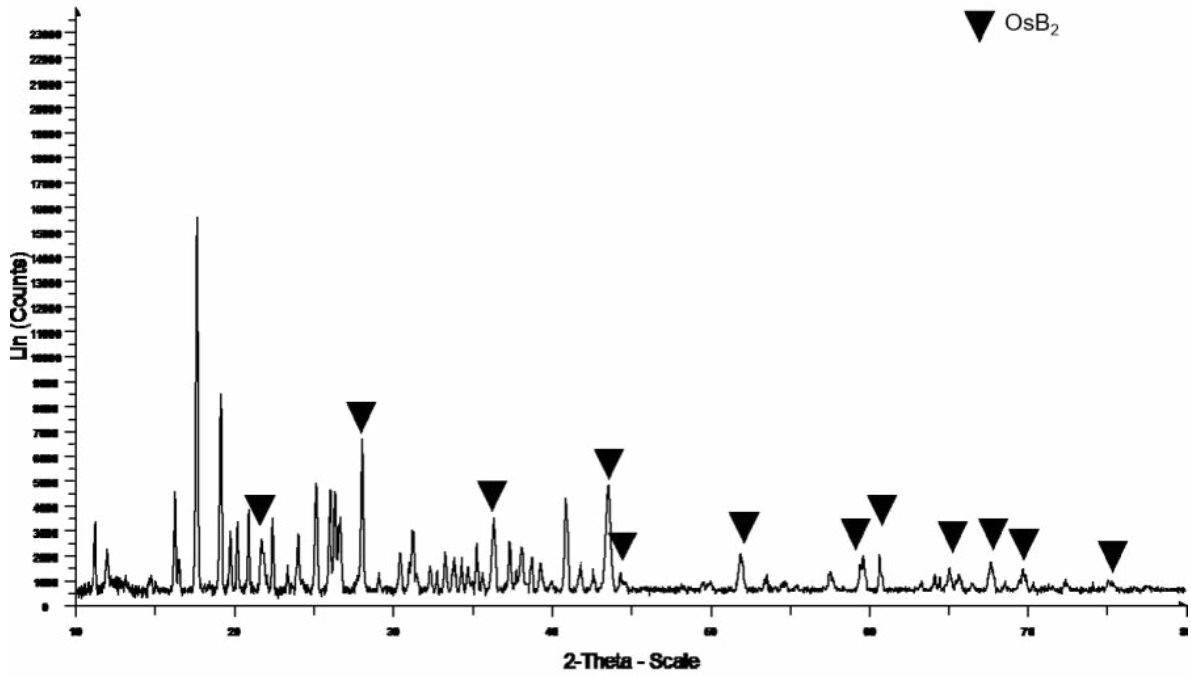

Figure 3a: XRD pattern of OsB_2 yielded by reaction of $OsCl_3$ with B.

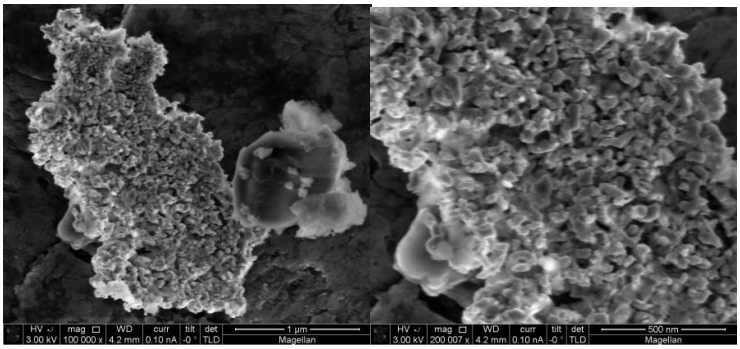

Figure 3b: SEM image of crystals of OsB_2.

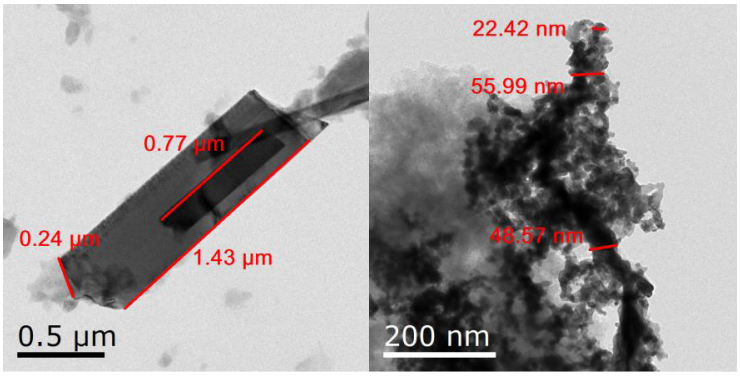

Figure 3c: TEM image of nanorod crystals of OsB_2.

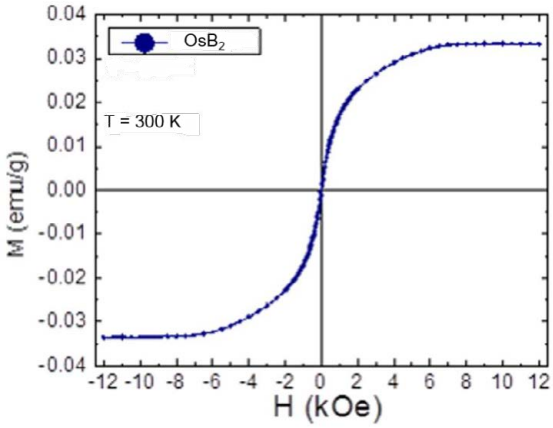

Figure 3d: Magnetic hysteresis superparamagnetism of OsB_2.

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