Announcing the Final Examination of Zhilin Xie for the degree of Doctor of Philosophy

Time & Location: October 27, 2014 at 10:30 AM in ENG 288
Title: Rhenium, Osmium and Iridium Diborides by Mechanochemistry: Synthesis, Structure, Thermal Stability and Mechanical Properties

Borides are implemented in a range of industrial applications due to their unique mechanical, electrical, thermal and catalytic properties. In particular, transition metal diborides are of special interest. In the recent years, borides of rhenium (Re), osmium (Os) and iridium (Ir) have been studied as for their ultra-hardness and superior stiffness. In this dissertation, a mechanochemical method is introduced to produce rhenium diboride (ReB2) powder, a novel hexagonal osmium diboride (h-OsB2), and iridium borides' powders. Densification by Spark Plasma Sintering, thermal stability and mechanical properties of h-OsB2 were also studied.

ReB2 was recently reported to exhibit high hardness and low compressibility, which both are strong functions of its stoichiometry, namely Re to B ratio. Most of the techniques used for ReB2 synthesis reported 1:2.5 Re to B ratio because of the loss of the B during high temperature synthesis. However, as a result of B excess, the amorphous boron, located along the grain boundaries of polycrystalline ReB2, would degrade the ReB2 properties. Therefore, techniques which could allow synthesizing the stoichiometric ReB2 preferably at room temperature are in high demand. ReB2 powder was synthesized at low temperature using mechanochemical route by milling elemental crystalline Re and amorphous B powders in the SPEX 8000 high energy ball mill for 80 hours. The formation of boron and perrhenic acids are also reported after ReB2 powder was exposed to the moist air environment for a twelve month period of time.

Hexagonal osmium diboride (h-OsB2), a theoretically predicted high-pressure phase, has been synthesized for the first time by a mechanochemical method, i.e., high energy ball milling. X-ray diffraction (XRD) indicated the formation of h-OsB2 after 2.5 hours of milling, and the reaction reaches equilibrium after 18 hours of milling. The lattice parameters of the h-OsB2 are a=2.916Å and c=7.376 Å, with a P63/mmc space group. Transmission electron microscopy confirmed the appearance of the h-OsB2 phase. The thermal stability of h-OsB2 powder was studied by heating under argon up to 876 °C and cooling in vacuo down to -225 °C. The oxidation mechanism of h-OsB2 has also been proposed. The hexagonal phase partially converted to the orthorhombic phase (20 wt.%) after spark plasma sintering of h-OsB2 at 1500°C and 50MPa for 5 minutes. Hardness and Young's modulus of the h-OsB2 were measured to be 31 ± 9 GPa and 574 ± 112 GPa, respectively by nanoindentation method.

Prior to this research a number of compounds have been prepared in Ir-B system with lower than 2 boron stoichiometry, and no IrB2 phases have been synthesized experimentally. In this dissertation, three new iridium boride phases, ReB2-type IrB2, AlB2-type IrB2 and IrB have been synthesized with a similar mechanochemical method. The formation of these three phases has been confirmed by both X-ray diffraction (XRD) and transmission electron microscope (TEM) after 30 hours of ball milling and 48 hours of annealing. The IrB2 phases have hexagonal crystal structures and the new IrB phase has an orthorhombic crystal structure. The segregation of iridium from iridium borides' lattices has also been studied by high resolution TEM.

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