Mechanochemical synthesis and characterization investigations of rare-earth borides and tungsten boride and tungsten silicide powders fabricated from low cost oxide powders

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Room temperature mechanochemical routes were carried out in the syntheses of nano-sized rare-earth boride (MB\textsubscript{6}, M = La, Sm, Ce) powders from M\textsubscript{2}O\textsubscript{3}–B\textsubscript{2}O\textsubscript{3}–Mg blends, tungsten boride powders from WO\textsubscript{3}–B\textsubscript{2}O\textsubscript{3}–Mg blends and tungsten silicides from WO\textsubscript{3}–SiO\textsubscript{2}–Mg powder blends. All synthesis reactions were driven by high-energy ball milling and were gradually examined in terms of milling duration and process control agent. Following the mechanochemical synthesis, unwanted MgO phase and Fe contamination worn off from the milling vial/balls were removed with HCl acid leaching under the effect of ultrasonics stirring. Pure rare-earth boride, tungsten boride and tungsten silicide powders were obtained after repeated centrifuging, repeated washing and drying. Subsequent annealing was performed in a tube furnace under Ar atmosphere inorder to reveal residual elements. Phase and microstructural characterizations of the milled, leached and annealed powders were performed using X-ray diffractometry (XRD), differential scanning calorimetry (DSC), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques. High-purity (> 99.99 \%) LaB\textsubscript{6}, CeB\textsubscript{6} and SmB\textsubscript{6} powders were successfully synthesized having average particle sizes of 80 nm, 86 nm and 81 nm, respectively.

Using stoichiometrically excess amounts of B\textsubscript{2}O\textsubscript{3}, pure W\textsubscript{2}B\textsubscript{5} powders with an average particle size of 226 nm and an average grain size of 55.3 nm were successfully synthesized. Likewise, TEM analysis revealed that pure W silicide nanoparticles with an average size of 97 nm were encapsulated by SiO\textsubscript{2} layers with an average thickness of 15 nm.