Unique Preparation of Hexaboride Nanocubes for Hydrogen Storage Applications

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The goal of this project was to design and characterize hexaboride ceramics with *unprecedented* and *tunable* hydrogen storage properties. Hexaborides have potential applications as multifunctional hydrogen storage materials due to their unique metal ion diffusion behavior. In this project, we successfully prepared ultra-fine powders of hexaborides, particularly lanthanum hexaboride via a combustion synthesis process. The combustion synthesis technique is amenable to the formation of oxides, as has been shown in countless studies. However, boride materials were thought to be unfeasible by this technique.

Combustion synthesis for producing hexaboride powders has a series of advantages. The process is highly efficient involving extremely fast heating rates, high temperatures, and short reaction times, while at the same time being easily scalable. In this type of synthesis, an exothermic reaction occurs between metal nitrates and a fuel. The type of fuel and the fuel-to-nitrate ratio are the most important controlling parameters for determining the reaction temperature reached during combustion.

During our synthesis, lanthanum nitrate and boron powders were mixed with carbohydrazide in an exothermic reaction. The process resulted in high-surface area powders of LaB_6. We have proven that the LaB_6 powders can be prepared in a few seconds taking advantage of this type of combustion reaction using a very low amount of fuel and rhombohedral boron powders. The addition of larger amounts of fuel or cubic boron results in the formation of a significant amount of undesirable oxide phases such as $LaBO_3$ and La_2O_3. Thus, the heat of reaction must be kept very low in order to promote the formation of the boride. According to x-ray diffraction results, the powders we have obtained contain about 3 mol% $LaBO_3$ impurities in the as-synthesized state. With an HCl and H_2SO_4 wash, these impurities have been removed effectively. Furthermore, the morphology of the powders from scanning electron microscopy is well-defined and in the range of 500 nm.

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