

# Investigation on Synthesis and Antioxidant Properties of Calcium Hexaborid

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## 1- Introduction

Calcium hexaboride has high melting point (2372 K), high hardness (27 GPa), low density (2.45 g/cm<sup>3</sup>), high Young's modulus (379 GPa) and low thermal expansion coefficient. This material has capability for being used in composite and as an antioxidant in oxide-graphite refractories. Calcium hexaboride can be synthesized by different processes such as hydrothermal, solid state and combustion synthesis. CaB<sub>6</sub> as an antioxidant in MgO-C refractories can reduce the oxidation of graphite. In this study synthesis and application of CaB<sub>6</sub> as an antioxidant are investigated.

## 2- Experimental

Boric acid and calcium hydroxide in autoclave at 200°C and pressure of 2 bar for 3h were mixed until hydration of calcium hydrated borate (CaB<sub>6</sub>O<sub>10</sub>. nH<sub>2</sub>O) obtain. After calcination of the samples at 400°C for 2h CaB<sub>6</sub>O<sub>10</sub> was achieved and then it was mixed with reduction agents such as Mg, Al, or Si and then pressed as a pellet. The pellets were calcined at 650-900°C in carbon bed. After calcination, CaB<sub>6</sub> and different oxides such as MgO, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> were established. Then samples milled and then leached to reduce oxide impurities. Different origin synthesis CaB<sub>6</sub> and Al powders were added to MgO, graphite and novolak resin and mixed. Different batches were pressed in the form of 40 mm diameter samples with 800 kgf/cm<sup>2</sup> pressure. Then, oxidation resistance of samples at 800-1400°C for 4h in air atmosphere were measured.

## 3- Results and Discussion

The results of combustion synthesis of CaB<sub>6</sub> from mixing of CaB<sub>6</sub>O<sub>10</sub> and Mg at carbon bed after calcination at different temperatures (Fig. 1) show that calcium and magnesium boride are presented as minor phases. After leaching according to Fig. 2C high purity

calcium hexaboride with high purity by this method was achieved.

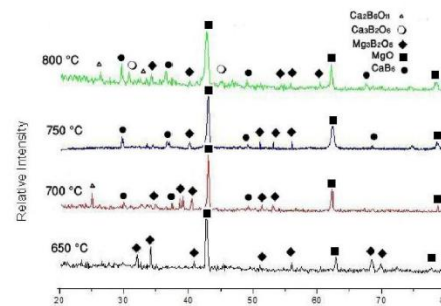


Fig. 1 XRD results of pellets containing CaB<sub>6</sub>O<sub>10</sub>+Mg after heat treatment at different temperatures in carbon bed

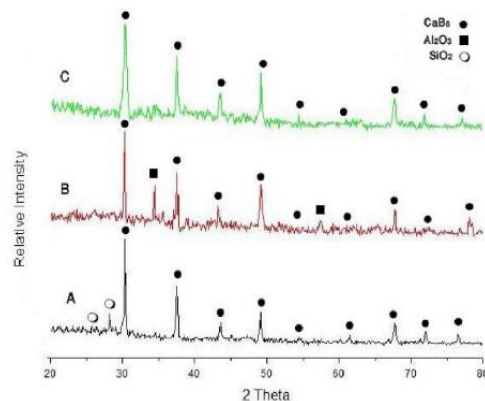


Fig. 2 XRD results of various synthesized CaB<sub>6</sub> after leaching a) with silicon; b) with aluminum; c) with magnesium

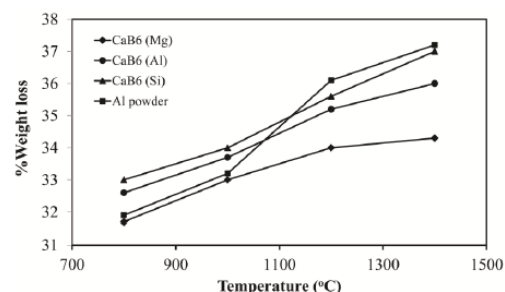


Fig. 3 Weight loss of samples containing CaB<sub>6</sub> or Al at different temperatures in oxidation atmosphere

Fig. 3 shows the weight loss of MgO-C refractories containing different kinds of CaB<sub>6</sub> that is synthesized with reduction agent of Mg, Al, and Si. The results show that the CaB<sub>6</sub> obtain from mixing of CaB<sub>6</sub>O<sub>10</sub> and Mg as reduction agent has better oxidation resistance capability in comparison with Al.

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#### **4- Conclusions**

1. Combustion synthesis of  $\text{CaB}_6$  was carried out by mixing  $\text{CaB}_6\text{O}_{10}$  with Mg and then leaching can produce purer  $\text{CaB}_6$  compared to synthesis from mixing  $\text{CaB}_6\text{O}_{10}$  with Al and Si.
2. MgO-C containing  $\text{CaB}_6$  has better oxidation resistance capability in comparison to normal refractories with Al as an antioxidant.