Production of Nickel Boron Alloys via Self-Propagating High Temperature Synthesis

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Abstract

Nickel boron alloys are generally preferred as master alloys and brazing filler materials. Especially in the production of high-performance complex alloys, using master alloys has particularly important role. In terms of final product properties, master alloy composition and purity effects many critical material behaviors. Within the scope of this study production of nickel boron master alloy (NiB) which includes 15 wt.% boron alloys were investigated. Instead of its industrial production methods, carbothermic and aluminothermic processes in DC electric arc furnace (EAF), self propagating high tempreature synthesis (SHS) were used. The SHS method provides the possibility of production in a very short time without requiring external energy except trigger energy to start the reaction. During the experiments, amounts of initial powder ratio calculated using with FactSage 7.1 thermochemistry simulation program. Thermochemical evaluations were made to estimate the adiabatic temperatures and possible product compositions in the final product by using FactSage 7.1 thermochemical software. The SHS process was performed in alumina crucibles which were pre-heated in an oven. Under normal gravity and air environment, oxide powders of metals ( NiO, B₂O₃ ) and Al as a metallic reductant were used. While increasing B₂O₃ ratio the results were close to commercial NiB composition and best boron ratio in NiB were obtained from stoichiometric %115 B₂O₃ experiment. This alloy includes 11.47 wt.% B, 77.55 wt.% Ni and was successfully produced with 80% Ni, 58% B recovery.

1. Introduction

Self-propagating High-temperature Synthesis (SHS) is a type of reaction synthesis that bases upon the heat generation between reactants by giving trigger energy to obtain products by virtue of self-sustaining reaction. SHS, eliminates the main drawback of traditional production processes: high energy capacity and low productivity. Due to the fact that it requires a minimum amount of equipment, a punch and die or ceramic crucible, reactant powders, mixing equipment and an ignition source, this method is enormously cost-saving among other processes. Different types of carbides, borides, nitrides, silicides, oxides, hydrides, and complex intermetallics can be synthesized via SHS. Carbothermic and aluminothermic reduction of boron oxide (B₂O₃) and boric acid (H₃BO₃) is mainly used to produce nickel boron alloys (NiB) with a B content ranging from 15-20% in a D.C. arc furnace. Charcoal or wood chips and other carbonaceous materials used as reductants for the production of NiB obviously present a competitive price advantage over common metallic reductants such as Al, Mg, Ca and Si. Notwithstanding the low Al content of the final alloys produced by the carbothermic process, inevitably carbon contamination in final product is exist.

2. Experimental Procedure

In the experimental set NiO and B₂O₃ powders are used as nickel and boron source, Al powders are used as a reductant. All reactants properties were given at Table 1.

<table>
<thead>
<tr>
<th>Component</th>
<th>Purity, %</th>
<th>Grain Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiO / Ni</td>
<td>99.03 / 77.87</td>
<td>&lt; 45 μm</td>
</tr>
<tr>
<td>B₂O₃</td>
<td>99.50</td>
<td>&lt; 150 μm</td>
</tr>
<tr>
<td>Al</td>
<td>94.00*</td>
<td>&lt; 45 μm</td>
</tr>
</tbody>
</table>

*0.028% Fe, 0.29% Cr, 0.56% Zn, 0.45% Mn, 4.65% Mg, 0.12% Si.

Thermodynamic calculations for many reaction systems, adiabatic temperatures were investigated before performing any experiments. Possible phases and required initial mixture amounts calculated with using FactSage 7.1 thermochemical simulation program. The starting materials were nickel oxide (NiO), boron oxide (B₂O₃) and aluminum (Al) powders. Before the reaction, the alumina crucibles undergo pre-heating at 500°C for 20 minutes. First experimental setup reductant amount was increased from 95% to 120% stoichiometry with 5% interval. Metal recovery and SHS yield were calculated. Adiabatic temperature ($T_{ad}$) is a very important parameter to predict the self-sustainability of the reaction. If the $T_{ad}$ of reaction is higher than 2073 K, the reaction is exothermic and self-sustainable. The $T_{ad}$ of the formation of NiB (given as Eq. 3) was calculated as 2673.3 K by using FactSage 7.1.

$$3\text{NiO} + 2\text{Al} \rightarrow 3\text{Ni} + \text{Al}_2\text{O}_3$$  \hspace{1cm} (1)

$$\text{B}_2\text{O}_3 + 2\text{Al} \rightarrow 2\text{B} + \text{Al}_2\text{O}_3$$ \hspace{1cm} (2)

$$6\text{NiO} + 3\text{B}_2\text{O}_3 + 10\text{Al} \rightarrow 6\text{NiB} + 5\text{Al}_2\text{O}_3$$ \hspace{1cm} (3)
Figure 1. Adiabatic temperature with increasing Al.

Figure 2. Possible phases depending on increasing aluminum.

Those ingredients of various proportions were mixed for 15 minutes in turbula mixer and then transferred into an alumina crucible. In second stage; electrical current was integrated into the system via tungsten wire which is inserted into the charge. Hereby combustion was occurred and reaction took place within the pot. After the experiment, SHS product was kept waiting for cooling than metal and slag samples were took for characterization process.

3. Results and Discussion

NiB containing SHS products were analyzed by chemical analysis and atomic absorption spectrometry (AAS, Perkin Elmer Analyst 800) techniques.

Chemical analysis of SHS products metal and slag phase related experiments were presented in Table 2.

According to Table 2. Boron content of the mixture decreases with increasing Al amount.

Table 2. Chemical Analysis of SHS Products.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>%Ni</th>
<th>%B</th>
<th>%Al</th>
<th>%Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>95Al</td>
<td>83.97</td>
<td>9.00</td>
<td>5.81</td>
<td>0.33</td>
</tr>
<tr>
<td>100Al</td>
<td>80.00</td>
<td>9.46</td>
<td>9.71</td>
<td>0.33</td>
</tr>
<tr>
<td>105Al</td>
<td>79.38</td>
<td>8.75</td>
<td>10.63</td>
<td>0.28</td>
</tr>
<tr>
<td>110Al</td>
<td>79.28</td>
<td>8.12</td>
<td>11.55</td>
<td>0.22</td>
</tr>
<tr>
<td>115Al</td>
<td>76.97</td>
<td>7.95</td>
<td>13.40</td>
<td>0.33</td>
</tr>
<tr>
<td>120Al</td>
<td>72.64</td>
<td>7.01</td>
<td>17.67</td>
<td>0.51</td>
</tr>
</tbody>
</table>

After the chemical analysis of the SHS products metallic distribution ratios of elements in the metal, slag and gasify were investigated. Figure 2. shows metal (Ni,B,Al) distribution among metal, slag and gas phase of the experiments.

Figure 3 presents that highest Ni recovery was observed at 95% stoichiometric mixture. On the other hand the highest boron recovery was observed at 100% stoichiometric mixture.

4. Conclusion

In conclusion the parameters which affect NiB production via SHS method were investigated. Increasing the amount of reductant leads to increment in Al content in SHS product. In the future study we are going to do brazing paste from nickel boron powders and Al dross will use as reductant.

References


