Flotation and Infiltration of Artificial Alumina Rafts on the Surface of Molten Cryolite

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Abstract

Alumina powder injection into the electrolytic bath usually involves raft formation: the alumina spreads on the surface of the electrolyte while the bath temporarily freezes around the powder. It stays afloat on the surface until the infiltration of the alumina by the bath and disturbances (agitation, splashing of liquid) lead to the sinking of the raft. Compressed discs of secondary alumina were prepared and inserted on the surface of molten cryolitic bath as artificial rafts for certain periods of time, to observe the flotation of the disc, the form and thickness of the frozen bath layer and the infiltration of alumina by the bath. Several sets of tests were conducted. The apparent densities of the discs were calculated to determine the role of surface tension in the flotation of the discs.

Keywords: Alumina raft; artificial alumina raft; flotation of alumina raft; infiltration of alumina raft; frozen bath.

1. Introduction

Although it is well known in the industry that alumina powder could float on the molten bath, the phenomenon and its effects have not been thoroughly investigated yet. Research in this field mostly aims to determine and model the dissolution rate of alumina and its dominant driving mechanisms. The investigated parameters influencing the dissolution are mostly the properties of alumina and the electrolytic bath [1 - 4]. Relevant articles noted that alpha-alumina sinks and gamma-alumina floats [5]. Townsend [6] provided tables about the duration of flotation of several different types of alumina powders, differentiated between soft (snow-like, low mechanical strength) and hard (ice-like, high mechanical strength) crust, but did not find clear correlation between crusting tendencies and other, easily measurable properties of the powder. Walker [7] reported in his dissertation that the consistency of an alumina raft, or a “floating island” as he put it, is mushy after 15 seconds in the bath. Its structure was found to be composed of three different layers, namely the solidified electrolyte, alumina infiltrated by the electrolyte with changing composition and dry alumina. Also, pockets of infiltrated alumina in frozen bath were found in the layers. He attributed the sinking of agglomerates (sub-islands) to the breaking of the island caused by mechanical interferences, like the splashing of the bath or another alumina injection.

Even if the structure of compressed alumina discs differs slightly from that of injected, more loose alumina raft, it was chosen to eliminate the haphazardness of rafts created by regular
feeding method, to ensure that the raft could be taken out entirely and the samples shall be comparable to each other – and also to compare its behavior to the results of a mathematical model of the raft.

2. Experimental Setup

Smelter grade alumina was compressed into discs, placed on the surface of cryolitic bath. Then, after a certain period of time, they were recovered with tongs specially made for this job. The samples obtained this way have been weighed and broken or cut up along the diameter for observing the infiltration front with an ‘Innovation beyond Imagination’ USB Digital Microscope and for further analysis with a Scanning Electron Microscope (SEM JEOL JSM 6480 LV with EDS Oxford SiLi detector and Inca software).

2.1. Alumina discs

Secondary alumina discs (with 40 mm diameter and 8.6 mm thickness) were prepared using a Struers Labo-Press 3 machine. 12.5 mL powder was fed into the machine chamber, and exposed to 50 kN for 15 minutes which included a 3 minutes initial period where the powder was heated to 180 °C. The resulting bulk density of alumina powder was not a precisely defined value. It depended on the actual arrangement of the particles, and therefore the mass and thickness of the discs had some dispersion. The discs were very fragile, they crumbled easily and some powder got lost every time they were handled. For this reason, a separate batch of discs was prepared only for the statistical analysis of the properties of the discs.

2.2. Bath properties

The cryolitic bath was melted in a carbon crucible; the free surface of the bath was 7.6 cm x 7.6 cm square with rounded corners. The depth of the bath was 3.8 cm. The prepared bath contained 83 % cryolite, 11.5 % AlF₃ and 5.5 % CaF₂ but due to the presence of some impurities in the bath and the experimental setup prior to the insertion of discs, as well as due to the partial dissolution of the discs themselves, the concentration of alumina was not constant. The estimated alumina concentration for each series of insertions will be indicated in the next section; any change due to bath evaporation was neglected.

Occasionally a thermocouple was inserted in the bath and it showed approximately 10°C less than the thermocouple inserted in the bottom of the crucible. After insertion the temperature was decreasing due to the cold disc, but at some point it is inevitable and the small size was cost-effective and convenient for manipulations. To reduce the heat loss towards the open surface of the bath, the opening was covered with a quartz plate between the insertion and recovery of the discs.

2.3. Series of experiments

The details of the different experiments are presented in Table 1.

<table>
<thead>
<tr>
<th>Table 1. Parameters of experiments.</th>
<th>Initial alumina concentration of bath %</th>
<th>Temperature of crucible °C</th>
<th>Flotation times of discs (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>5,5</td>
<td>982</td>
<td>30, 60, 90</td>
</tr>
<tr>
<td>B</td>
<td>4,2</td>
<td>992</td>
<td>40, 90, 20, 70, 120, 60</td>
</tr>
</tbody>
</table>
It was concluded by calculations, density measurements and visual observation that the raft could stay afloat after its density exceeded the density of the bath by more than 20% and the surface tension played an important role in keeping it on the surface. Although the bath wets the alumina, the surface clung to the edge of the disc keeps it floating for up to four minutes. The effect of carbon particles and the significant amount of heat loss on the surface of the bath, resulting in the solidification of some of the bath around the edges of the discs, should be considered.

The sunken discs still contained some gas that was released soon after sinking.

5. Acknowledgements

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6. References