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Advanced Application Development: Using the Acidity Index to improve Borate Fusion

Abstract

Based on the acidity index (AI) we set up an application for very different samples using only two different lithium borate fluxes. In the first step, we assessed the sample composition of the powdered material by XRF and calculated the material-specific AI. Then, based on this specific AI, we determined the optimum flux composition. In all samples, the subsequent fusion process with the individually determined flux composition resulted in high-quality fused beads. This shows that the AI is a valuable tool in target-oriented application development for borate fusion.

Key words

• **Borate Fusion** • **Acidity Index** • **HAG-HF** • **Choice of Flux** • **XRF**

Introduction

In the production of glass beads, varying sample properties usually entail the use of different fluxes. A non-optimum flux composition might impede the fusion process and result in undissolved particles, cracked beads or crystallization. In these cases, the underlying cause is the poor dissolving power of the used flux in combination with the specific sample material. Therefore, it is inefficient to increase the fusion temperature or duration. Instead, choosing a flux composition that is specifically adjusted to the sample properties usually ensures complete dissolution of all mineral phases and improves homogeneity of the bead. This again has a beneficial effect on analytical precision and accuracy.

In this application note, we introduce the so-called acidity index (AI) and explain how it can be applied for calculation of the optimum flux composition.

Methods

For this test series, we used eight different sample materials with varying material properties including geological raw materials, slag, glass and mineral wools. Initially, the sample material was ground using an automatic HP-MA pulverizing mill with a tungsten carbide grinding vessel. Subsequently, the powdered samples were analyzed using a PANalytical Epsilon 3XL EDXRF-spectrometer. Figure 1 shows the composition of all eight samples. There are vast differences in composition between the samples ranging from high silica concentration to high magnesia and calcium concentration.

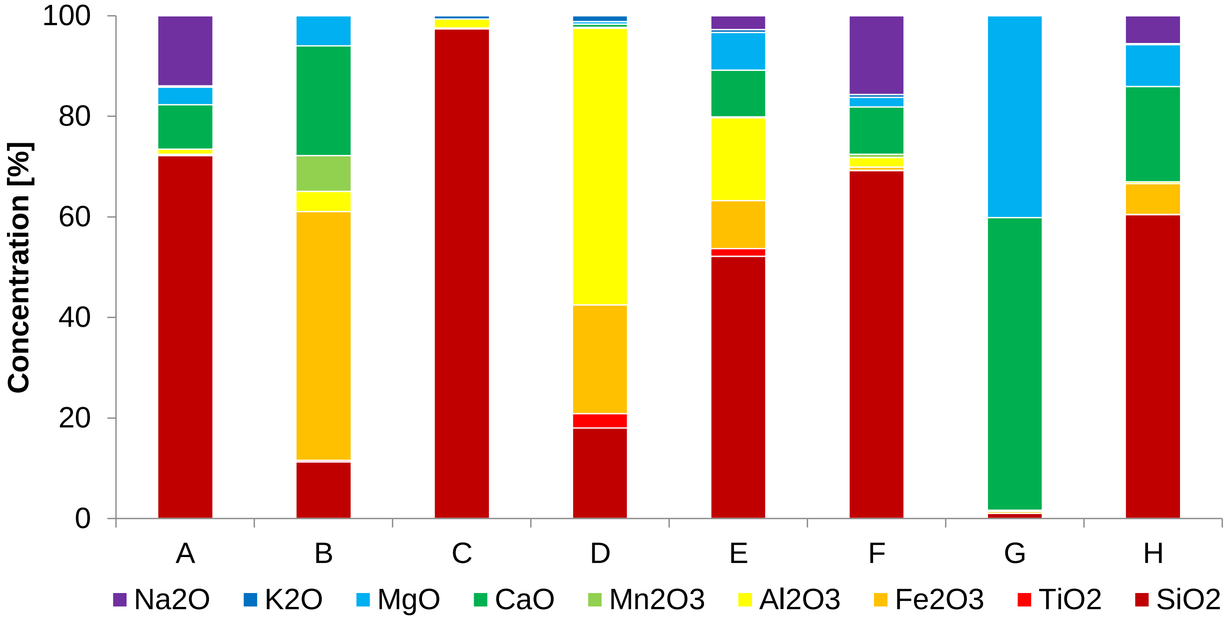

Figure 1: *Composition of all eight samples analyzed. The samples are composed very differently ranging from almost 100 % SiO_2 to almost 100 % MgO + CaO. The components are sorted by acidity, red is more acidic, purple is more alkaline.*

Calculation of the acidity index (AI):

The overall acidity index of the mixture between sample and flux was determined by the following formula:

$$\mathrm{AI_{tot}} = \frac{n \times \frac{\#O}{\#Metal}}{n+m} + \frac{m \times \mathrm{AI_{Flux}}}{n+m}$$

where n is the amount of sample, m is the amount of flux in the final bead and $\frac{\#O}{\#Metal}$ is the amount of oxygen atoms divided by the number of metal atoms of the sample. The AI of the flux is calculated in the same way with $\frac{\#O}{\#Metal}$, resulting in an AI for lithium tetraborate (LiT) of $1.1\overline{7}$, and for lithium metaborate (LiM) of 1.00.

In a first step, we calculated the AI of the sample based on the XRF analysis of the powdered sample. Subsequently, the optimum flux composition for each sample was computed. The following constraints were specified:

1. The target AI value for the bead was set between 1.13 and 1.15 providing the highest probability for stable beads.

2. LiM can only be employed in the maximum ratio of 12:22 because higher LiM concentrations significantly increase crystallization probability of the bead.

3. Flux to sample ratio is fixed to 10:1.

4. The amount of sample is 1 g and the amount of flux is 10 g.

Results

Figure 2 shows the calculated optimum flux composition for each sample based on a ratio of 1 g sample and 10 g flux. Based on the calculated LiT:LiM ratio we aligned each sample to one of four commercially available ready-to-use mixtures (12:22, 50:50, 67:33, 100% LiT). Five samples showed almost the same LiT:LiM ratio, while the other samples plotted within LiT, 67:33 or even very close to 12:22 flux.

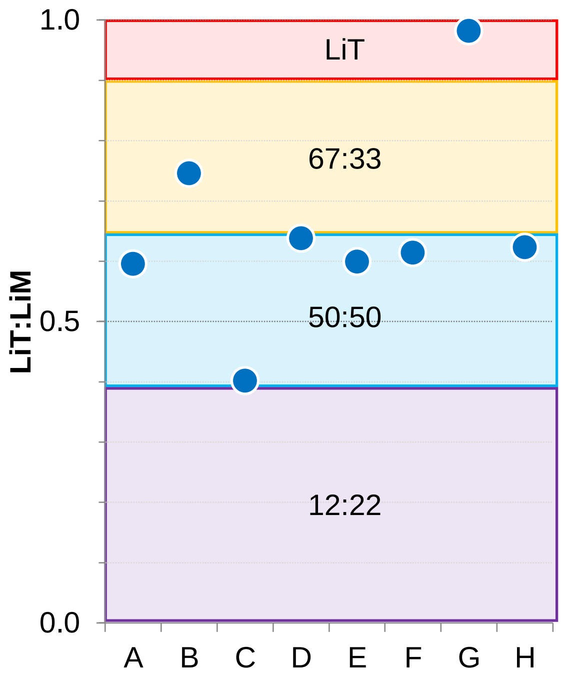

Figure 2: *Calculated optimum flux composition. The colored fields mark the approximate area of flux use. Most of the samples plot within the 50:50 field.*

After calculation of the optimal flux composition, fusion tests with the material were performed using $Pt_{95}Au_5$ crucibles in the Bead One HF induction fusion system. Duration of the fusion was set to 20 min at 1050 °C including 12 min of rocking. For a 39 mm bead, 1 ± 0.0001 g sample and 10 ± 0.0003 g flux were filled into a crucible. In general, no additives other than flux were used for fusion. Only for sample B, an oxidizer was added in order to ensure complete oxidation of reducing phases and prevent potential damage to the platinum ware.

All beads of this study were successfully prepared without any problems. Visual inspection revealed no crystallization, cracks or other anomalies.

Discussion

With the calculated AI, six samples plotted inside the 50:50, one inside the 67:33 and one in the LiT field. In order to comply with the constraint of only two fluxes to be used, sample G was shifted to the 67:33 flux. Subsequently, a feasibility study was performed in which each material was successfully fused.

If only one single flux type is available, it might also be possible to use the 50:50 flux for all samples. Another preparation method could apply the 67:33 flux for all materials except for sample C where the 12:22 flux is mandatory due to its high Si content. A further approach for enhancing the fusion method could be more effective pulverizing of the material as reduced particle size will increase solubility due to a higher specific surface.

The calculation of the AI can be a very powerful tool to choose the optimal flux composition for unknown samples. It reduces the time required for development of fusion applications. Furthermore, the optimal flux lowers the fusion duration time and increases the sample throughput. Moreover, precision and accuracy are improved due to the complete dissolution of the sample within the melt avoiding any mineralogical effects.

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