In-Situ Monitoring of Additions to Liquid Slag using Laser-Induced Breakdown Spectroscopy (LIBS)

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ABSTRACT

Laser-induced breakdown spectroscopy (LIBS) was applied for the *in-situ* monitoring of CuO and ZnO additions to liquid slag. A synthetic starting slag, with a SiO_2-FeO_x matrix and lacking Cu and Zn, was melted at a temperature of 1300 °C. Over time CuO and ZnO powders were added to the melt and the evolution of the Cu- and Zn-signal was monitored from start to finish. Overall, an increase in the Cu- and Zn-signal can be observed with each addition. The increase in viscosity due to the addition of room-temperature oxidic powders, causes scattering in the signal.

INTRODUCTION

Laser-induced breakdown spectroscopy (LIBS) is an analytical technique which uses a pulsed laser to initiate plasma formation at the surface of materials. The resulting plasma light is characteristic for the elemental composition of the material. A pulsed laser is focused on the sample and heats it up locally. The material is evaporated and eventually a plasma is formed. The light of the plasma is transported to the spectrometer using lenses, mirrors and/or fiber optic cables. The spectrally resolved signal is subsequently used to characterize the sample. Owing to its versatile setup and other advantages, LIBS can be used for *in situ* applications (Figure 1). In this study, CuO and ZnO powders were added to a SiO_2-FeO_x based melt, mimicking a fayalitic slag. The additions were monitored *in situ* using LIBS.

METHODS AND MATERIALS

A synthetic SiO_2-FeO_x based slag (510.0 g) was introduced to a Ni-crucible and placed in a furnace at a temperature of 1300 °C. Figure 1 shows a schematic overview of the used LIBS setup. When the furnace reached a temperature of 1300 °C, a total of 25 LIBS spectra were accumulated every minute. In between LIBS measurements, CuO was added to the melt in four steps, until a total amount of 5.3 g was supplemented. This procedure was repeated with ZnO powder, with a total addition of 30.4 g.

RESULTS

Overall, the signal increases with the addition of CuO and ZnO to the melt (Figures 2 and 3, respectively).

1. There is an initial peak directly after each addition: the powder is added at the surface of the melt, i.e. the same location where the LIBS data is collected.
2. The signal decreases over time until the next addition: the CuO and ZnO powder dissolves in the melt, resulting in a lower signal at the surface.
3. Scattering of the Zn signal due to an increase in viscosity: the addition of ZnO powder to the melt causes a temporary drop in temperature and the formation of stable phases due to the introduction of oxygen. As a result, the melt becomes more viscous, preventing a good mixing and the creation of a heterogeneous material.

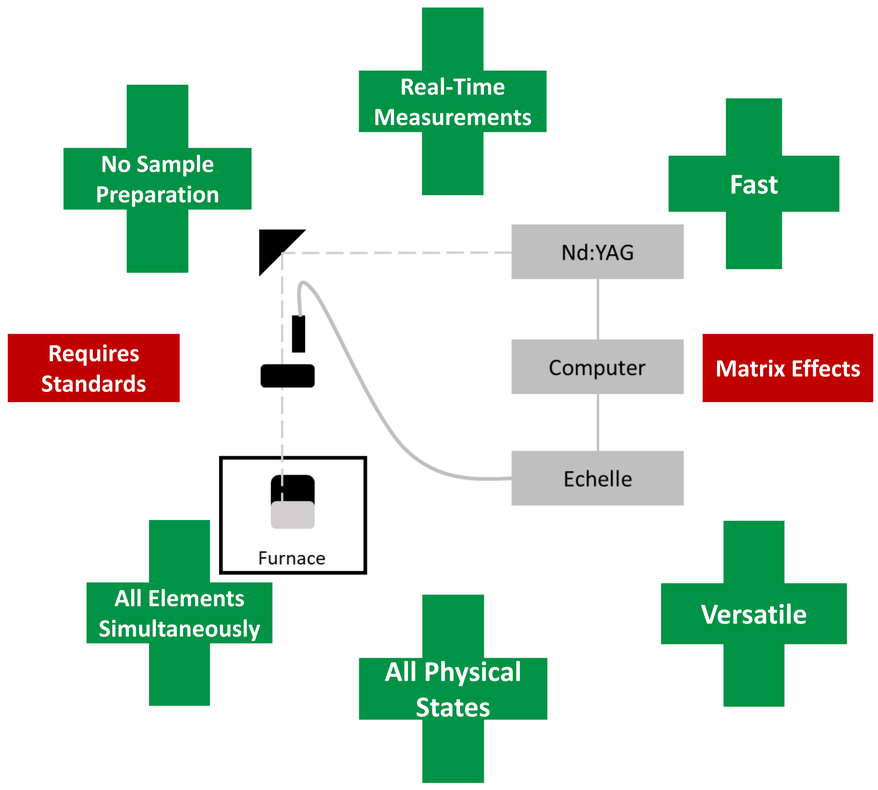

Figure 1: Schematic overview of the LIBS setup and the advantages and disadvantages of the LIBS technique

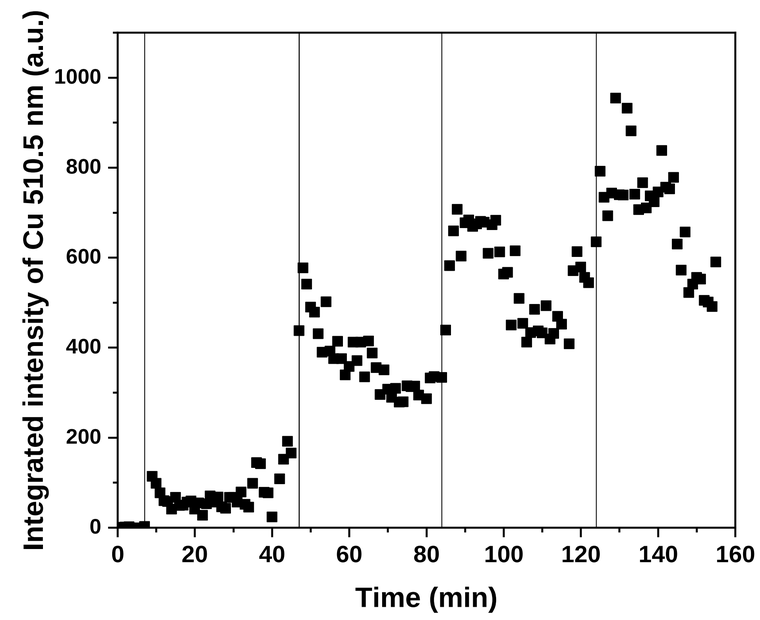

Figure 2: Signal increase of Cu 510.5 nm with the addition of CuO. The vertical lines indicate an addition of CuO to the molten slag

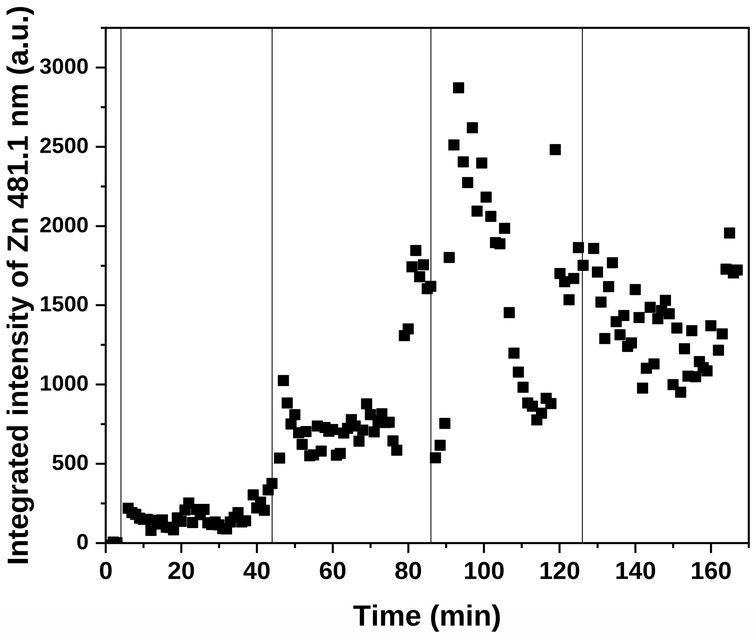

Figure 3: Signal increase of Zn 481.1 nm with the addition of ZnO. The vertical lines indicate an addition of ZnO to the molten slag

CONCLUSIONS

- ✓ Successful monitoring of CuO and ZnO additions to a SiO_2-FeO_x rich slag
- ✓ The addition of oxidic powders results in an increase in viscosity, which limits the mixing of the melt and introduces heterogeneity to the melt, resulting in a more scattered signal
- ✓ Additional methods are needed to obtain quantitative results

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