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Laser-Induced Breakdown Spectroscopy of Scaled Steel Samples taken from Continuous Casting Blooms

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Abstract

To analyse continuous casting steel blooms a removal of non-representative surface layers is required prior to the analysis. In this work, an optimized process is developed to ablate such layers and to analyse the bulk material underneath with laser-induced breakdown spectroscopy (LIBS). A high ablation rate is crucial since the time slot for an inline analysis is limited, e.g. to less than one minute. To get a deeper understanding of the material structure between bulk material and surface, samples are sawed out of steel blooms. The samples are analysed in lab scale experiments including LIBS measurements and cross-section polish methods. These studies show that the surface layers may consist both of oxides and metallic layers and typically have thicknesses from 200 µm to 600 µm each. The ablation behaviour of the oxide differs significantly from that of the metallic layers. An operation scheme for inline material identification is worked out to perform ablation and analysis with a single laser source. During the ablation phase and the subsequent measurement phase the laser source is operated with individually tailored parameters. A total penetration depth exceeding 1 mm in steel can be achieved within 20 seconds of ablation. Thereby the influence of non-representative surface layers on the following LIBS measurement can be suppressed to a large extend. For chromium, relative root mean square errors of predictions of less then 13 % were achieved on high alloy samples with up to 16 m.–% Cr and on low alloy samples with Cr contents below 2 m.–%.

Keywords: LIBS, inline measurement, laser-based scale ablation, steel analysis

1. Introduction

The application of LIBS for steel analysis is reviewed in several papers [1, 2, 3, 4]. Mostly, the analysis is carried out with a prior mechanical preparation step, e.g. grinding, of the steel surface. For industrial use, the analysis of the bulk material below a scale layer is a typical task. It is desirable to do this without a separate mechanical preparation. In [5] tailored laser pulse sequences have been used to ablate scale layers on production control samples taken from a steel melt by samplers. The background of this study is the analysis of continuous casting steel blooms which exhibit much thicker scale layers – up to 600 µm – than production control samples. The targeted duration of the scale layer ablation and the analysis is less than one minute because of the typical feeding rate of a hot rolling mill of one block per minute.

In this paper, we present a laser-based measurement procedure which uncovers the bulk material in a cleaning step. The analyte concentrations are determined after that in a second step. Both steps are carried out by laser irradiation. In the first part of the work, the ablation behaviours of steel and scale are compared using different laser parameters. Then the surface structure of samples sawed out of continuous casting steel blooms is examined by cross-section polish microscopy. Finally, the uncertainties and precisions of LIBS measurements during the ablation phase and a differently parametrized subsequent analysis phase of the bulk matrix are studied.

2. Experimental

The laser source used is a Nd:YAG-laser operating at $\lambda = 1064\,\text{nm}$. The oscillator is diode-pumped followed by a flash lamp-pumped amplifier. The laser source can be operated in different modes. In the double pulse-only mode (DPO), the repetition rate can be up to 120 Hz. Each cycle emits a double pulse (DP). This mode is known to be suited for LIBS measurements. On the contrary, the cleaning mode (CM) is optimized for a high ablation rate. The ablation is more effective, when the burst energy is split up into several weaker pulses [6, 7, 8], instead of one double or a single pulse of the same total burst energy. In the CM, the repetition rate is limited to 60 Hz, because in each cycle the flash lamp is ignited twice. During the first discharge many comparatively weak laser bursts are emitted. The next flash lamp discharge yields the double pulse. This double pulse is very similar to the pulses

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