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In situ laser-induced breakdown spectroscopy measurements of chemical compositions in stainless steels during tungsten inert gas welding☆

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ABSTRACT

A laser-induced breakdown spectroscopy (LIBS) system was combined with a bead-on-plate Tungsten Inert Gas (TIG) welding process for the in situ measurement of chemical compositions in austenitic stainless steels during welding. Monitoring the weld pool's chemical composition allows governing the weld pool solidification behavior, and thus enables the reduction of susceptibility to weld defects. Conventional inspection methods for weld seams (e.g. ultrasonic inspection) cannot be performed during the welding process. The analysis system also allows in situ study of the correlation between the occurrence of weld defects and changes in the chemical composition in the weld pool or in the two-phase region where solid and liquid phase coexist. First experiments showed that both the shielding Ar gas and the welding arc plasma have a significant effect on the selected Cr II, Ni II and Mn II characteristic emissions, namely an artificial increase of intensity values via unspecific emission in the spectra. In situ investigations showed that this artificial intensity increase reached a maximum in presence of weld plume. Moreover, an explicit decay has been observed with the termination of the welding plume due to infrared radiation during sample cooling. Furthermore, LIBS can be used after welding to map element distribution. For austenitic stainless steels, Mn accumulations on both sides of the weld could be detected between the heat affected zone (HAZ) and the base material.

1. Introduction

Currently used inspection methods for weld seams measure, for the most part, post factum and thus cannot prevent weld defects. An in situ analysis of the weld pool's chemical composition could potentially enable control of the welding process by regulating the respective welding parameters in order to prevent possible weld defects [1]. Here, the most critical cracking phenomena are hot cracking/tearing or solidification cracking [2,3]. The chemical composition of the weld pool is, from a metallurgical point of view, the most critical parameter for cracking [4]. Hence, a crucial prerequisite of component safety is a hot crack assessment whilst producing and processing of metallic materials [5]. Therefore, an in situ LIBS-based analysis system allows not only the study of the correlation between the occurrence of weld defects and the chemical composition of the weld pool [6], but could also enable better control of the welding process.

Laser-induced break down spectroscopy (LIBS) is a technique based on the measurement and analysis of element-specific light. The light is emitted from the material's surface, where samples are excited by a pulsed laser beam. Energy is locally absorbed and material starts to evaporate. A plasma is generated in this vapor, resulting in the excitation of the material constituents. As the plasma cools, electrons emit characteristic radiation. The emitted light is detected and analyzed by a spectrometer [7]. The measurements can be quantified by using appropriate calibration standards and are subsequently converted into mass fractions of each element present.

Tungsten Inert Gas (TIG) welding is a joining process of metals using the heat from an electric arc established between a non-consumable tungsten electrode and the weld material. Typically, an inert gas such as argon or helium is used as shielding to protect the weld pool from oxidation. The main welding process parameters are the welding current,

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welding speed and the shielding gas flow rate [8]. This welding technique is relatively easy to handle, requires minimal space and allows for automated welding with very high reproducibility.

There is published work in the literature about on-line weld defect monitoring using optical inspection methods by measuring light emissions directly from the welding plasma. For instance, Mills [9] discussed adaptation of emission spectroscopy methods for welding arc diagnostics. Mirapeix et al. [10,11] proposed a real-time arc welding quality monitoring system based on temperature measurements. They calculated electron temperatures in the welding plasma from various Ar emission lines during TIG welding and found a relationship between the temperatures of the electrons in the plasma and weld defects. Garcia-Allende et al. [12] have used a similar spectra-processing technique designed for applications in online detection and classification of arc welding defects. Huber et al. [13] presented spectroscopic investigations during laser beam welding processes to gain insight into metal vapor behavior. Gott et al. [14] used a combination of high-speed digital imaging and spatially-resolved spectroscopy on a submerged arc welding process. They were able to observe changes in the chemical composition during welding.

Currently, LIBS has a vast application area in a variety of research fields, such as continuous steel casting monitoring in the steel industry [15,16], scrap sorting tasks [17,18], explosives detection [19–21], mining and mineral resource exploration [22,23], geology and environmental science [24–27], archeology [28,29], biomedical applications on organic tissues [30,31] and space exploration [32–35]. However, the complexity of the emission spectrum of high-alloyed steels makes it difficult to interpret the plasma emissions [36]. Advancements of both spectrometer and detector technologies allow a rapid and continuous data acquisition in a wide spectral range via LIBS measurements (~ 200–1000 nm) [37,38]. At present, echelle spectrometers provide sufficient spectral resolution and a wide and accessible spectral wavelength range [17]. Moreover, ICCD (Intensified Charge Coupled Device) detectors coupled with echelle spectrometers enable the detection of very weak signals in the presence of intensive background radiation [37], e.g. as in welding plasmas.

In this work, we present a LIBS-based, on-line chemical composition monitoring system coupled with a TIG arc welding system. We present first results of in situ measurements of selected line emissions gained during welding of austenitic stainless steels.

2. Materials and methods

2.1. Materials

EN grade 1.4404 austenitic high-alloyed stainless steel was used for the welding experiments. This material has excellent weldability. Therefore, the material is suitable to repeat experiments under constant conditions. EN grade 1.4404 stainless steel has a vast application field such as in heat exchangers, medical implants, chemical containers, jet engine parts, and parts exposed to marine atmospheres. The concentrations of the main alloying elements of the used sample material are shown in Table 1.

Samples with the dimensions 320 mm × 35 mm × 3 mm (length × width × thickness) were manufactured by Outokumpu. Prior to welding, the samples were cleaned with acetone in order to remove any residual greases or oils from the surface (e.g. resultant oils from handling and/or machining).

Table 1
Main alloying element concentrations of grade 1.4404 stainless steel according to manufacturer's data.

	C (wt%)	Cr (wt%)	Ni (wt%)	Mn (wt%)	Si (wt%)	Fe (wt%)
Manufacturer data sheet	0.014	17.100	11.200	0.780	0.350	balance

2.2. Experimental set-up

The experiments were conducted using a TIG arc welding system composed of a Castolin Eutectic (Frankfurt, Germany) CastoTIG 1611 DC power supply and a Castolin TIG torch using 1.6 mm tungsten electrode. The electrode tip was extended approximately 3 mm out from the ceramic nozzle (diameter 6 mm). The distance from the electrode to the base material was set to 3 mm.

The LIBS system employed a 1064 nm Nd:YAG laser at 25 Hz with maximum output energy of 200 mJ (Quantel CFR 200, Newbury, UK), which is connected via a LIBS electronic controller (LTB Berlin, Germany) to an echelle spectrometer Aryelle 200 (LTB Berlin) with ICCD[1] detector iStar (Andor, Belfast, Northern Ireland). Fig. 1. exhibits a sketch of the experimental setup.

The pulsed laser was focused on the sample 10 mm behind the tungsten electrode (see Fig. 2). The laser beam was focused on the sample with 1065 nm optimized telescopic lenses, and mirror collection optics focused the signal onto a 400 μm glass fiber for transmission to the spectrometer. The spectrometer has a spectral range of 190 to 569 nm and a resolving power of approximately 8000. The laser frequency was 25 Hz. Each spectral measurement accumulated 15 laser shots set with a 1.0 μs measurement delay and 1.0 μs gate width. The ICCD gain was set to 1700 – after several trials, this relatively high gain was necessary in order to differentiate the laser-induced plasma signal from the background caused by the weld plume. The gate width had to be kept relatively short in order to limit oversaturation of the detector from the intense broadband weld-plasma emission. Spectra were recorded approximately every 0.9 s.

A linear motorized stage was used to move the samples for the welding process. Apart from the stage-mounted sample, the remainder of the experimental setup remained static. The samples were moved with constant speed of 20 cm/min. Bead-on-plate welding was performed at 120 A with 12 l/min shielding Ar gas flow. Three seams were conducted with the same process parameters (weld seam (1), (2), (3)). The linear stage and the LIBS system were controlled from an automatized computer interface. The welding process was started manually via a self-built remote interface for the TIG arc-welding system.

The experiments were performed in the following order: 1.) the LIBS system was started; 2.) after a 3 s delay, Ar flow was initiated; 3) after another 3 s delay, the arc was ignited and 70 mm movement of the stage began; 4.) end of stage movement was reached and the weld arc terminated – Ar flow continued; 5) finally, the LIBS system was stopped. Data treatment and calculation were completed with the self-written control and classification software module for the spectrometer “Sophi” (LTB Berlin).

3. Results and discussion

3.1. Effect of the shielding gas and welding plasma on the LIBS signal

The first measurements showed a significant increase in recorded relative intensity values with the start of the shielding argon gas flow, and even further artificial increase when the welding arc was initiated. This artificial increase was caused by the welding plasma. Moreover, it is difficult to distinguish welding plasma intensity values from the laser-induced plasma values.

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[1] The selection of detectors in any LIBS-experiment is crucial. Although CCD-type detectors have a lower detection limits (i.e. higher sensitivity) and a better signal-to-noise ratio, an ICCD detector was selected. The latter provides a higher gate-width flexibility and enables detection of weak signals in the presence of high background radiation [37,39].