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Laser-based diagnostics for the steel industry

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Abstract: The properties and performance of steel products depend essentially on their chemical composition and their microstructure (phase fraction, grain size, texture...). Two laser-based technologies that are being developed by the Industrial Material Institute of the National Research Council of Canada permit on-line and in-process measurement of these key parameters.
First, we outline Laser-Induced Breakdown Spectroscopy (LIBS), which is based on the production of a plasma by a high-power pulsed laser and the analysis of the emitted light with a spectrometer to find out the elements present in the material. This technology which we could foresee being used in ladle metallurgy has been demonstrated so far for real time determination of composition in galvanization baths and is being commercialized.
The other technology is laser-ultrasonics, which consists in the generation of ultrasound with a laser and the detection of the ultrasonic waves with a second laser coupled to an optical interferometer. This technology is now used for measuring the wall thickness, eccentricity and austenite grain size of seamless tubes. It has been licensed and is now commercially available. Also, laser-ultrasonics has measured on-line texture and thickness of steel strips. When combined with a Gleeble thermo-mechanical simulator, many other applications to steel processing have been demonstrated such as the monitoring of grain growth, phase transformations and recrystallization. Examples of successful monitoring of metallurgical transformations include austenite decomposition in TRIP steels and recrystallization of IF steels. These applications could ultimately be implemented on-line for process control.

Key words: Laser Induced Breakdown Spectroscopy, Laser Induced Plasma Spectroscopy, chemical composition, galvanization, laser-ultrasonics, laser-ultrasound, microstructure, grain size, retained austenite, texture, on-line sensing

1 Introduction

Productivity in the steel industry, like in many other industries, can be improved by sensors that can be used on-line or during processing. Such sensors can provide key information allowing to fine tune the process or to reject out-of-specifications or flawed products. Since in the steel industry, most of the processes are at elevated temperature or involve fast moving products or occur in a harsh environment, sensing at distance with lasers is advantageous and in some cases the only feasible technique. In this paper, we are presenting two laser-based technologies that are being developed by the Industrial Material Institute of the National Research Council of Canada. They are designed for on-line and in-process measurement of chemical composition (Laser induced Breakdown Spectroscopy) and for the determination of thickness and m icrostructural properties (Laser-Ultrasonics). These techniques have been demonstrated in laboratory, in on-line trials and for some applications have been commercially available and used in industry for a few years.

2 Laser Induced Breakdown Spectroscopy

Laser-induced breakdown spectroscopy (LIBS) is an all-optical technique for the continuous elemental analysis of liquids, solids, and gasses [1]. LIBS analysis is carried out by focusing a high-power pulsed laser beam on the material, thereby vaporizing and ionizing a small volume of the target (typically 10^{-8}–10^{-6} cm^3) to produce a plasma. Optical emission from this plasma, which is representative of the ablated material, is analyzed with a spectrograph to obtain qualitative and quantitative information about its atomic composition. The technique operates at a distance (one meter and more) and is therefore very suitable for on-line and *in-situ* analysis in hostile environments, in particular for the analysis of the composition of molten metals. The approach that we use for performing a measurement truly representative of the bulk of the molten metal bath and not from its surface where contamination occurs is depicted in Figure 1. A tube or lance is immersed in the molten metal. Inert gas (argon) is flown down the tube and produces bubbles. The laser is fired inside the bubble, thus insuring a surface with composition truly representative of the melt.
The technique has many industrial applications to hydro and pyro metallurgy and has been applied in particular to the monitoring of the composition of the zinc bath for hot dip galvanization of steel [2]. For this purpose, a spectral window centered on 305 nm that includes spectral emission lines from zinc, aluminum and iron is chosen.

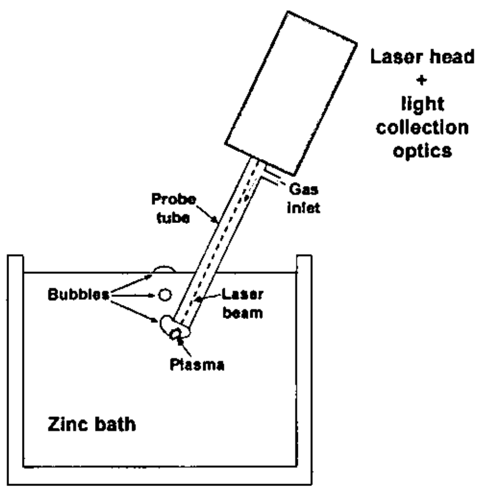

Figure 1 Schematics of the approach used for composition measurement by LIBS in molten metals

The technique was tested on-site during the transition from galvannealing to galvanizing bath compositions. As seen in Figure 2, the composition information provided in real time by LIBS agrees very well with laboratory measurements made from time to time following bath sampling. LIBS has the distinct advantage of responding more rapidly to any change of composition, as shown in the figure by the spikes in Al or Fe composition that follow the introduction in the bath of an ingot with 4.5%Al.
The application to galvanization having been very successful, a commercial prototype is being developed now specifically for this application. Other applications in steel making, such composition measurement in a ladle are considered for future development.

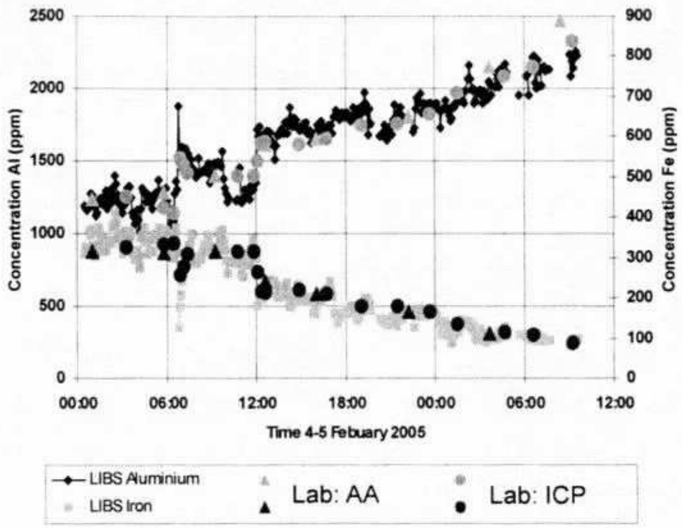

Figure 2 Al and Fe contents of the molten zinc bath as measured by LIBS. Dark and light triangles or circles correspond to laboratory measurements on samples taken from the bath by Atomic Absorption Spectroscopy or Inductively Coupled Plasma Spectroscopy

3 Laser-ultrasonics

The technique uses two lasers, one for the generation of ultrasound and another one coupled to an optical interferometer for detection [3]. The generation laser pulse heats the material's surface and vaporizes a small volume (some nm of material) that, by a recoil effect and plasma pressure, generates an ultrasonic wave that propagates through the material. Simultaneously the detection laser illuminates the sample surface and the reflected (or scattered) light, phase-modulated by the surface motion caused by acoustic disturbances, is demodulated by the interferometer.

3.1 Laboratory experimentation

At IMI/CNRC, a laser-ultrasonic system coupled to a Gleeble™ 3500 has been exploited for many years and has been a powerful tool for monitoring microstructure transformations. Recent developments have made this system compact and robust and ready for commercialization. We present below several results that have been obtained with the system.

3.1.1 Results on Grain Size/Growth

It is well known that ultrasonic attenuation depends upon grain size: the coarser the grains are, the larger is the attenuation. Laser-ultrasonics coupled to a furnace can monitor grain size evolution *in-situ* and in real time, in particular austenite grain size, which determines to a significant extent the mechanical properties of steels. Following calibration grain growth can be monitored as shown, by example, in Figure 3. This figure shows the grain growth for a low carbon steel heated at 5 °C/s to 1100 °C and held at this temperature for about 500 seconds while performing laser-ultrasonic measurements. After proper cooling, the prior austenitic grain size was measured metallographically and was found to be very close to that determined by laser-ultrasound at the end of the thermal cycle as shown in Figure 3.

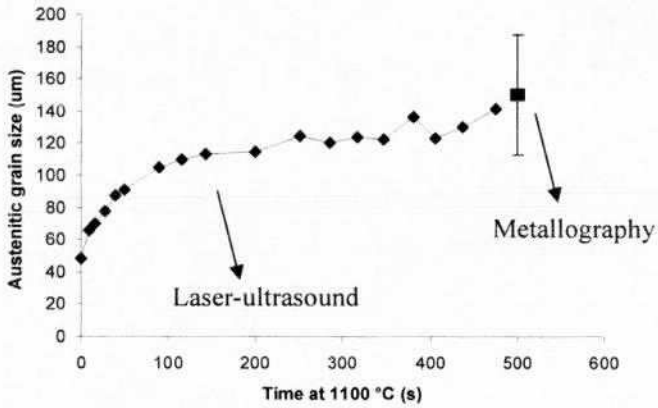

Figure 3. Real-time laser-ultrasonic measurement of austenitic grain sizes at 1100 °C for an A36 steel. The grain size measured by metallography after proper cooling is also shown.

3.1.2 Results on Phase Transformation

The sensitivity of ultrasonic velocity to phase fraction is due to the different elastic constants and/or densities of the different phase components. A classical case of phase transformation is the austenite/ferrite transformation in steels. Figure 4 shows the velocity during austenite decomposition of low carbon steel. The sample was submitted to a thermal cycle simulating that of a heat-affected zone during welding at a peak temperature of 1350°C followed by cooling from 800 and 500 °C in 30 seconds. One advantage of using the laser ultrasonic technique instead of the classic dilatometry is the potentiality to measure an absolute phase fraction. This advantage can be particularly useful for retained austenite fraction determination in TRIP or other multiphase steels. Laser-ultrasonics could be also applied to monitoring and quantifying austenite decomposition in a production line.

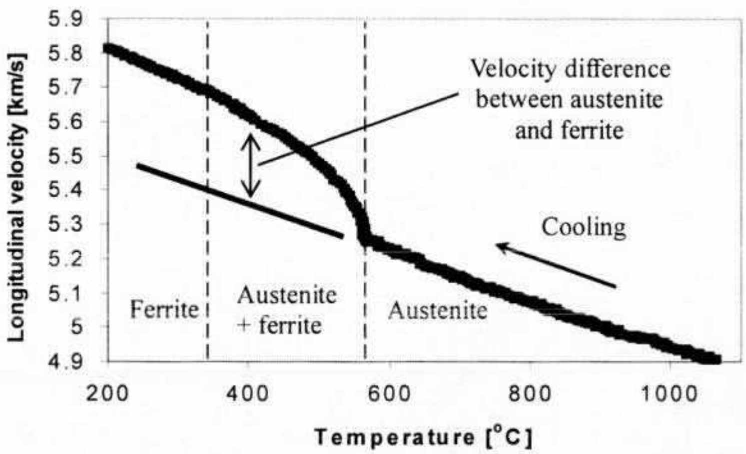

Figure 4. Longitudinal ultrasonic velocity variation during rapid cooling of low carbon steel.

3.1.3 Results on Recrystallization

The recrystallization of cold deformed materials results very often in a texture change and, accordingly, the elastic constants also change. A single velocity measurement can be sufficient to determine the recrystallized fraction, as shown in Figure 6 for the recrystallization of an interstitial free (IF) steel. A strong gamma fiber texture is expected to be developed in the final stages of recrystallization and the velocities changes can be directly correlated with this texture component.

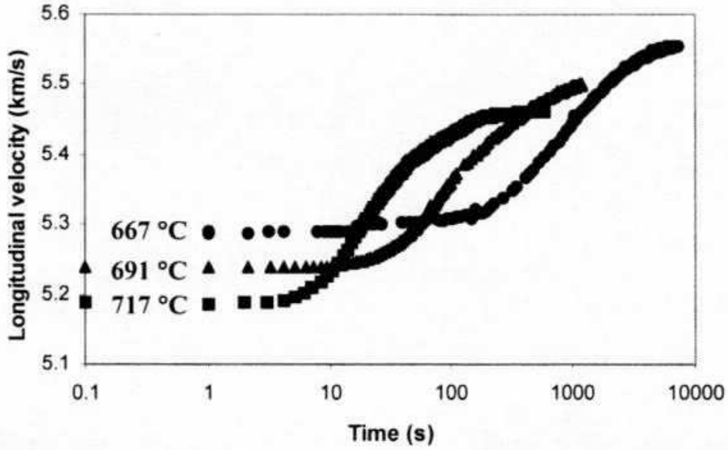

Figure 5. Longitudinal velocity variation during recrystallization of IF steel at various isothermal annealing temperatures.

3.2 On-line implementation

Laser-ultrasonics has been developed for measuring on-line the wall thickness and eccentricity of seamless tubes [4]. The system implanted on a tube mill line of the Timken Company has been in use for about 3 years and has measured more than a million tubes. Thickness is derived from the measurement of the time interval between the echoes reverberating within the tube wall and from a calibrated value of the acoustic velocity. Figure 6 shows a view of the sensor head installed above the line on top of a passing tube. This head is linked by optical fibers to the lasers and the interferometer located in a remote and environment-controlled cabin. By using the same ultrasonic data but analyzing ultrasonic attenuation instead of velocity, austenitic grain size can be measured on-line as shown in Figure 7.

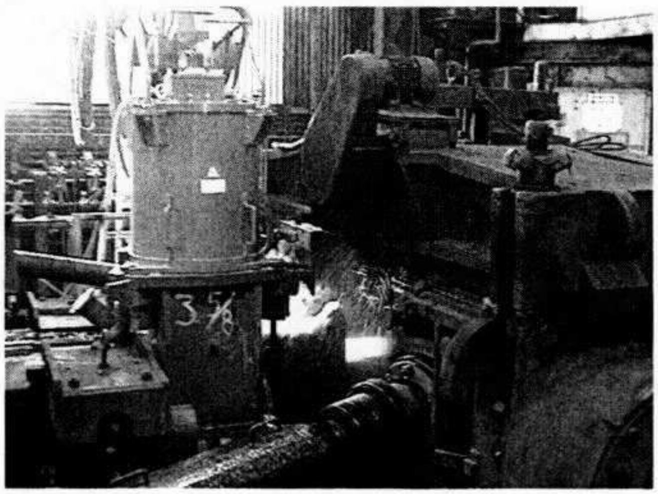

Figure 6 View of the inspection head located on-line measuring a tube being processed.

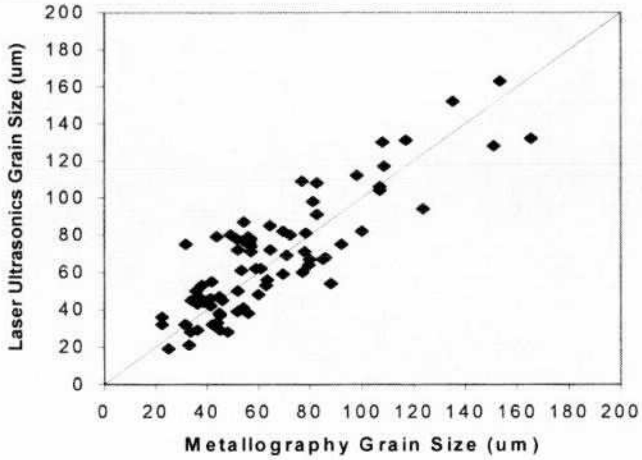

Figure 7 Grain sizes measured on-line by the laser ultrasonic system as a function of those obtained by metallography on the same tubes after proper quenching.

4. Conclusion

We have presented two laser-based technologies that allow on-line and in-process sensing. These technologies provide key sensing parameters for steel production and manufacturing, i.e. chemical composition, microstructure and wall thickness. Both technologies offer many interesting perspectives beyond those reported here. LIBS, which has many applications to hydro and pyro metallurgy, has been demonstrated in a galvanization plant and is now being commercialized for this application. It could be extended to ladle metallurgy also. Laser-ultrasonics is now commercially available for measuring wall thickness, eccentricity and grain size of seamless tubes. When coupled to a thermo-mechanical simulator, this technology allows monitoring metallurgical parameters like grain size, phase fraction and recrystallized fraction. For this purpose a compact and cost-effective laser-ultrasonic system has been developed and is being made commercially available. The monitoring tasks implemented in the laboratory can be further transferred to the production line for real time monitoring, which is a significant advantage to traditional techniques, such as metallographic examination or dilatometry that do not have this capability.

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