



Laser-induced breakdown spectroscopy and multivariate statistics for the rapid identification of oxide inclusions in steel products



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ABSTRACT

Laser induced breakdown spectroscopy (LIBS) scanning measurements can generally be used to detect the presence of non-metallic inclusions in steel samples. However, the inexistence of appropriate standards to calibrate the LIBS instrument signal means that its application is limited to identifying simple diatomic inclusions and inclusions that are chemically fully distinct from one another. Oxide inclusions in steel products have varied and complex chemical content, with an approximate size of interest of 1 μm . Several oxide inclusions types have chemical elements in common, but it is the concentration of these elements that makes an inclusion type have little or, on the contrary, deleterious impact on the final steel product quality. During the LIBS measurement of such inclusions, the spectroscopic signal is influenced not only by the inclusions' chemical concentrations but also by their varying size and associated laser ablation matrix effects. To address the complexity of calibrating the LIBS instrument signal for identifying such inclusion species, a new approach was developed where a calibration dataset was created, combining the elemental concentrations of typical oxide inclusions with the associated LIBS signal, in order to define a multivariate discriminant function capable of identifying oxide inclusions from LIBS data obtained from the measurement of unknown samples.

The new method was applied to a variety of steel product samples. Inclusions populations consisting of mixtures of several complex oxides, with overlapping chemical content and size ranging typically from 1 to 5 μm , were identified and correlated well with validation data. The ability to identify complex inclusion types from LIBS data could open the way to new applications as, for a given sample area, the LIBS measurement is performed in a fraction of the time required by scanning electron microscopy, which is the conventional technique used for inclusion characterisation in steel. Applications that would require speed for high throughput can benefit from this development where the LIBS measurement of inclusions can be used as a first stage rapid screening method for quality purposes. On the other hand, applications requiring inclusions data over large sample areas for investigation related to steel cleanliness could also benefit from such development.

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1. Introduction

Non-metallic oxide inclusions are a residual product of the steel-making process. For a given steel grade, a specific de-oxidising agent is added (such as Si or Al) to the liquid steel to capture the dissolved free oxygen. The large majority of the oxide particles formed, also called non-metallic inclusions, are then extracted out of the liquid steel by floatation, interaction and transfer into the slag during steel-making. However, the removal of inclusions is not 100% efficient and the process must be controlled to guarantee that only the least harmful oxide inclusions are formed, or left, in the final steel product.

Non-metallic inclusions fall into two groups, indigenous and exogenous [1]. Indigenous inclusions result from reactions in the melt. They form an inclusion population that is typical of the steel grade manufactured and the resulting inclusion population tends to be

consistent and controlled. Exogenous inclusions are generated by the accidental incorporation of slag, refractories or other unwanted materials with which the molten steel comes into contact. They share elements in common with indigenous inclusions. They can result in larger sized particles, and have a sporadic occurrence. In general terms, the presence of oxide inclusions remains acceptable whilst their chemical content, size and numbers remain within defined boundaries for a given steel grade. The term “steel cleanliness” is used to describe this. When non-metallic oxide inclusions fall outside of the cleanliness specifications, they are likely to become detrimental to the steel products' properties, as they act as points of weakness in the steel material from which failure will stem.

How detrimental an oxide inclusion is depends on the steel grade considered and the intended use of the steel product. In general terms, for cleanliness-critical thin gauge or thin diameter products, large and non-deformable oxide inclusions are the ones that will induce failure and their presence in the final product must be avoided at all costs. The chemical make-up of oxide inclusions has a strong influence [2,3]

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and is a good indicator of their harmfulness as, depending on their chemistry, inclusions can be deformed by elongation or fracture into smaller particles, reducing the deleterious impact on the steel properties. However, inclusions with chemistries that make them undeformable create points of weakness during shaping or cold drawing of the steel product and are likely to lead to early failure during the product transformation or during its service lifetime. For a given steel grade, a type of inclusion population is expected and deviation from this indicates an unstable process and can highlight quality issues.

For the routine assessment in production of steel products' cleanness levels, conventional methods of characterisation are used, such as optical microscopy for checking the number and size range of non-metallic inclusions according to standard procedures, as well as scanning electron microscopy coupled with energy dispersive X-ray analysis (SEM-EDX) for characterising inclusions chemical content. Although automated systems for the optical microscopy method are commercially available, human intervention is still required to discriminate between inclusions and artefacts, in addition to which, no detailed chemical information is available. In turn, SEM-EDX characterisation of inclusions can be fully automated and provide information on chemistry, size and number, but the time-intensive nature of the measurement means that only small areas of the steel samples can be characterised, limiting high-throughput or how representative of the actual inclusion population the measurement might be.

The high speed scanning measurement that LIBS can perform could make the technique appropriate for the rapid screening of steel products' cleanness levels in order to flag up deviations from the normal inclusion population for a given steel grade and the presence of unacceptable inclusion types. Ideally, the technique would provide chemical information to identify the inclusions types, as well as information on sizes and numbers. The inclusions number is a parameter that should be easily extracted from LIBS mapping data. On the other hand, inclusions chemical identification and size assessment are more complex tasks and this paper addresses the former by describing the development of a method for the chemical identification of inclusions from the data obtained by LIBS scanning measurements. The case of cleanness-critical Si-killed products was considered, where a typical oxide inclusion population is expected, including different oxides with overlapping chemistry and inclusions sizes ranging approximately between 1 and 5 μm . The ability to detect and identify the typical inclusion species of such a steel grade, as well as the potential presence of exogenous and detrimental inclusion species, from LIBS data, is described in this paper.

In the literature, we find that the direct observation of coinciding LIBS intensity peaks of two or more chemical elements was used to identify simple diatomic inclusions in steel, such as TiN, or for the qualitative identification of more complex inclusions that have distinct elemental compositions [4–7]. Previous work also included the identification of large inclusions, as found in solidified samples of liquid steel [8], where univariate calibration was used to quantify the inclusions' chemical content to distinguish them apart. In this case, the inclusion sizes were larger than the laser beam diameter, insuring that the ablation took place on oxide inclusion material only, minimising the variation of matrix effects during LIBS measurements and removing the effect of inclusions size on the LIBS signal. Such approach is not applicable to the identification of the small inclusions typically found in rolled steel products, where inclusion sizes are significantly smaller than the laser beam size. Finally, principal component analysis (PCA) was applied to the entire LIBS intensity dataset obtained from the ablation of a steel sample containing three types of inclusions that were chemically fully distinct [9]. This illustrated the ability of PCA to separate data related to the steel matrix and to the three individual inclusion types based on raw LIBS qualitative data.

To successfully identify small oxide inclusions that share elements in common, a distinction based on qualitative data is not sufficient. A means to identify the inclusion types based on their oxide components'

concentrations is required to label them reliably. Given the LIBS signal obtained from the ablation of such inclusions is influenced not only by its chemical concentrations, but also its size and associated matrix effect, multivariate statistics were used to resolve this multi-factorial problem. To start with, a calibration data set was built by SEM-EDX quantification of the inclusions content in specific steel samples, followed by laser ablation of the characterised samples surfaces. The SEM-EDX concentration data were combined with the LIBS intensities, on a particle to particle basis, and the resulting data set was submitted to cluster analysis, firstly, to check whether the LIBS data appeared to contain relevant and sufficient information to distinguish the inclusion types from one another, secondly, to provide the basis for calibrating a multivariate discriminant function that enables the prediction of inclusion types from the LIBS data obtained from the measurement of unknown steel samples. The new inclusion identification method is described step-by-step in this paper.

2. Experimental

SEM-EDX, being the conventional technique for characterising non-metallic inclusions chemistry, was used to enable the development and the validation of the LIBS method. For this reason, all steel samples were characterised by SEM-EDX prior to LIBS measurement to build a training model and also to cross-check the results obtained on test samples.

2.1. Instrument

An FEI Quanta 600 Field Emission Gun Scanning Electron Microscope coupled with an EDAX Energy Dispersive Analyser was used for the characterisation of non-metallic inclusions in the steel samples for training and validation purposes. The inclusions were detected using the backscattered electron signal, providing the coordinates, number and size of the inclusions. X-ray detection was used to measure the chemical content of each inclusion.

A high spectral resolution LIBS instrument, with a micro-scale scanning facility and rapid signal acquisition was used for this development work. The instrument's hardware was originally built by Fraunhofer ILT [10,11] and its characteristics are summarised in Table 1.

2.2. Samples

All samples used in this study were taken from production rolled products that consisted of rods with diameters ranging from 5.5 to 15 mm. The cross-sections of the samples were mounted into resin to facilitate their manipulation during sample preparation and measurements by SEM-EDX and LIBS.

Rod samples from different steel grades were used to offer a wide range of inclusion types with varying chemical concentrations. They

Table 1
LIBS instrument hardware description.

Laser	Water-cooled Nd:YLF diode-pumped solid state laser Wavelength 1047 nm Maximum power of 3.3 W at 1 kHz Pulse length of 9 ns
Spectrometer	Paschen–Runge type with a 0.75 m Rowland Circle 45 photomultiplier detectors Spectral range 130 to 671 nm Spectral Resolution 15 to 20 pm
Sample chamber	Ar-filled Pressure control (15 mbar to atmospheric)
Scanning stages	Direct Drive stage Position accuracy 1 μm Translation range 100 mm \times 100 mm
Signal electronics	Multi-channel integrator electronics MCI-P Maximum measuring frequency 1.2 kHz Delay time controllable Integration time controllable

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