

# Development of a New On-Line Sensor for Steel Surface Contamination



Sarclad and CRM Group have developed a new on-line sensor for steel strip contamination. The system utilizes laser-induced breakdown spectroscopy to give a continuous and real-time assessment of the contamination levels post-cleaning on a typical steel strip processing line. Critically, this is the first system available that can distinguish between surface carbon and iron fines contamination with quantitative data. This will enable the highest product quality alongside optimized core cleaning section parameters to give the greatest process efficiency. This article describes the technology used, its advantages and performance in industrial trials, and addresses the practical considerations of implementing the technology in the commercial industrial environment.

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## Introduction

Strip processors, and in particular galvanizers, have long been looking for a way to measure the residual contamination of the substrate surface accurately and reliably at the exit of the cleaning section. This is indeed a key parameter for the quality of coated steel, especially in the automotive sector, where there should be no surface defects.

The contamination sources are various, but two main pollutants are generally mentioned: surface carbon from mill oil and iron fines. In the long term, carbon will turn into soot in the furnace, reducing its efficiency, and will eventually fall onto the rolls and the strip. Iron fines can create pickup defects on the rolls and increase the dross content in the zinc bath by combining with zinc and aluminum, leading to a drift in the bath composition. In addition, all these problems can result in aspect defects on the final product.

If residual contaminations are continuously monitored on-line, corrective actions can be planned before being forced to downgrade a whole production due to aspect defects. In addition, if the contamination source can be differentiated from the others, its cause can be

quickly identified and eliminated by making targeted corrections to the cleaning section.

CRM Group and Sarclad have therefore developed new on-line equipment able to differentiate between iron fines and surface carbon pollutions. This equipment uses a laser-induced breakdown spectroscopy (LIBS) method developed by CRM Group.<sup>1</sup> It can be placed at the exit of the cleaning section in galvanizing lines to measure the residual contamination levels. This article describes the work carried out to achieve this on-line monitoring with a LIBS-based sensor and the engineering developments to create the Sarclad Contamination Monitoring System, a device ready for installation and integration into any strip processing line.

## Discussion

### Introducing Laser-Induced Breakdown Spectroscopy

LIBS is a spectral analysis technique with many applications in geology, metallurgy and other physical sciences. Over the years, it has been implemented in several industrial environments with its robust design

and adequate protective equipment against the harsh external conditions (temperature, humidity, dirt, etc.).

In galvanizing lines, two major contamination sources to be monitored are the surface carbon coming from the mill's oil and the iron fines. Being able to differentiate both sources is highly valuable to galvanizers, as the causes, and subsequent mitigations, of an increase in pollution levels are not the same for surface carbon or iron fines.

### Laser-Induced Breakdown Spectroscopy Quick-Start Guide:

A short-pulsed laser beam with a very high energy density is delivered to the surface being analyzed through a focusing lens. That energy is sufficient to ionize matter and create a high-temperature plasma around the laser impact. Inside the plasma, electronic transitions occur continuously, powered by the laser beam energy. When dropping from an energized state back to a lower layer, an electron emits a photon at a specific wavelength given by the Planck-Einstein relation:

$$E = h\nu = hc/\lambda \quad (\text{Eq. 1})$$

where

$E$  = the photon's energy in Joules (J),

$h$  = the Planck constant in J's,

$\nu$  = the photon's frequency in  $s^{-1}$ ,

$c$  = the speed of light in m/s and

$\lambda$  = the photon's wavelength in m.

In an electronic transition,  $E$  is simply the energy difference between the energized state and the layer where the electron stops. The set of wavelengths emitted by a chemical element is called its emission spectrum and uniquely identifies it. The electromagnetic range covered by the lines in such a spectrum can extend from UV to visible and infrared.

Molecular vibrations and rotations can also occur in the plasma, due to thermal agitation. In this case, photons are emitted in molecular bands, i.e., groups of lines so closely spaced to each other that they all appear as a single band.

All these wavelengths come out of the plasma in all directions with the intensity being maximal at an angle perpendicular to the surface where the plasma was created. In the LIBS, this emitted light is then collected and directed by an appropriate combination of lenses

and mirrors to an optical fiber. The fiber transmits the light to a spectrometer and all the information it carries about the elements present in the plasma can be analyzed (Fig. 1).

### Utilization of LIBS in the Contamination Monitoring System:

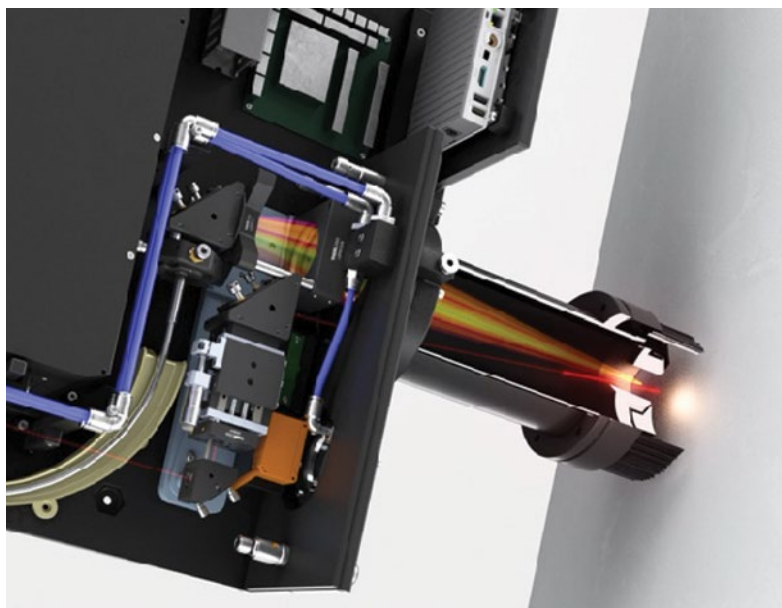
Since all wavelengths below 200 nm are absorbed by the oxygen in the air, the lines to be selected must lie in the visible range. In a LIBS spectrum, iron emits a lot of lines, while most carbon lines are found in the UV range. The remaining visible lines for carbon are either not sensitive enough or squeezed between two large iron lines.

To tackle this problem, the solution was to create a 100% nitrogen atmosphere around the plasma. The carbon and nitrogen thus recombine into cyanide radicals CN. These radicals emit a line in the visible range around 388 nm, which is sensitive enough and isolated from other lines. To make up for the random intensity variations between spectra related to plasma behavior but not to pollution variations, the surface carbon is monitored via the ratio of the CN line and one of the nitrogen lines. These principles are covered by U.S. patents.<sup>2,3</sup>

Likewise, the monitoring of iron fines contamination is achieved with a ratio of two iron lines. They were selected by a laboratory procedure developed by CRM Group and described in Reference 1. The chosen iron lines gave the best correlation with the iron fines levels on reference samples.

Figure 1

**Schematic of the laser-induced breakdown spectroscopy (LIBS) process, showing a pulsed laser beam (in red) generating a plasma on the surface and an associated spectral of light being collected for analysis.**



**Overview of the Demonstration Unit:** CRM Group and Sarclad developed an industrial demonstration sensor shown in Reference 2 and based on the LIBS technique described earlier. The objective of this equipment is to measure the residual contamination levels on the strip at the exit of the cleaning section in galvanizing lines. Both surface carbon and iron fines contaminations are considered.

The laser beam is focused through a set of diverging-converging lenses. Due to the combination of a low-backlash translation stage and a distance sensor, the focal point is always maintained at the surface of the strip, even if the thickness of the steel strip changes.

The casing of the sensor head has been designed to be water and dust tight. The whole system is kept at room temperature with a chilling and heating unit. The flow of nitrogen is adjusted by electrovalves and flow controllers.

Safety equipment such as mechanical interlocks and non-contact safety proximity switches ensure the system has a laser safety SIL rating of 3.

A spectra acquisition and analysis software has been developed by CRM Group. It outputs two monitoring signals: one for the surface carbon and one for the iron fines. It also controls the different components of the sensor head and continuously checks if all the conditions to safely operate the unit are met.

### Industrial Demonstration of the Contamination Monitoring System

The unit described above has been successfully installed and tested on three lines; the laboratory pilot line at CRM and two full-scale continuous strip galvanizing production plants. The data generated from these trials has been used to validate the measurement methodology,

Figure 2

LIBS demonstration unit with (1) the laser, (2) the laser controller and the spectrometer (below), (3) the laser beam focusing unit, (4) the light collecting unit and (5) the electronics box.

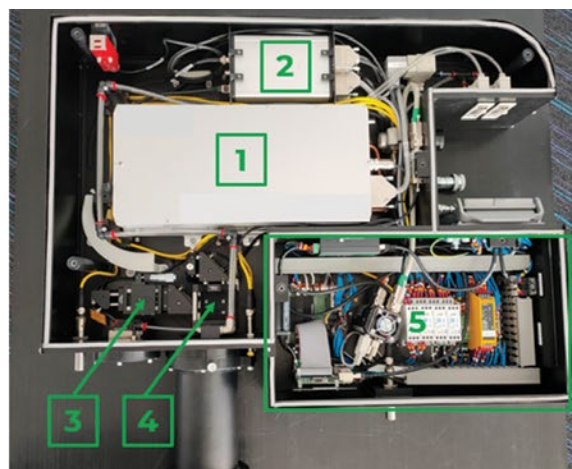
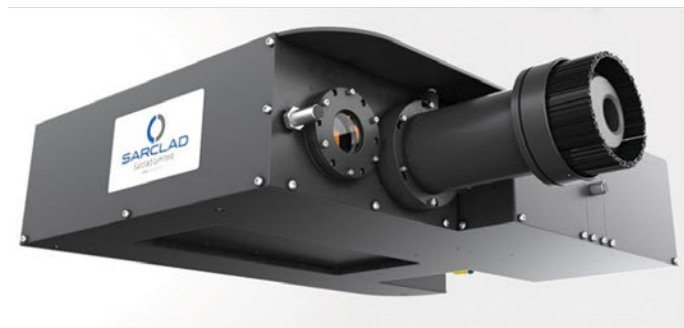


Figure 3

Correlations between LIBS ratios and laboratory tests.

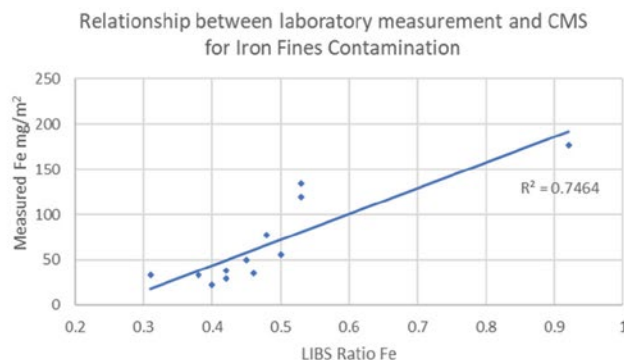
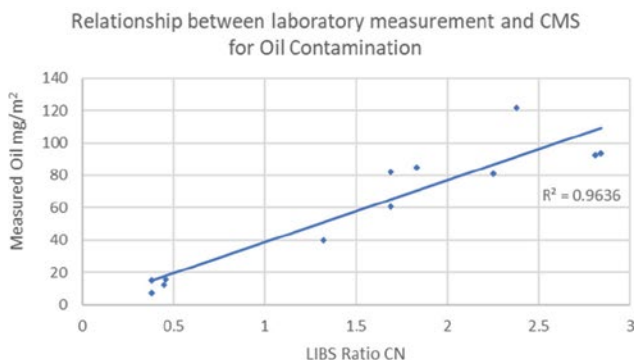
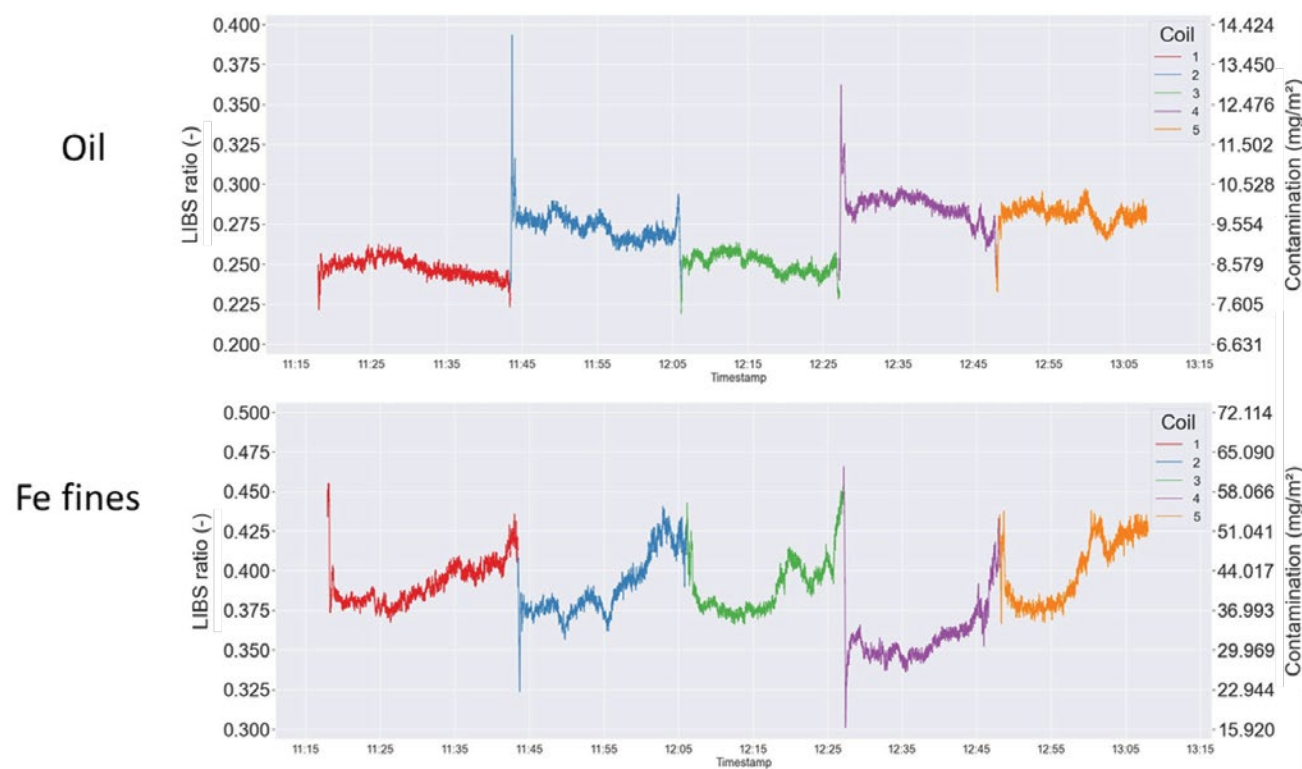


Figure 4

Example data traces of LIBS data for five consecutive coils, with calculated contamination levels included.



the results of which will be discussed here. Moreover, the trials have demonstrated the suitability of the measurement technology for the steel product and the robustness of the engineered solution of the measuring device in the industrial environment.

**Data Validation:** In order to determine the validity of the LIBS technology, it was necessary to undertake a comparison of measured LIBS ratios of wavelengths with verified contamination levels across the range of contamination levels of interest. To achieve this, a number of physical steel samples were taken from both industrial and laboratory steel coils. Adjacent samples were then subjected to both LIBS measurement by the demonstration unit and independent laboratory tests.

In the case of oil contamination, this was the total organic carbon (TOC) method and for iron fines the inductively coupled plasma (ICP) technique. Both techniques are recognized as reliable characterization methods for the given contamination species. The results achieved are given in Fig. 3.

Statistical analysis by linear regression shows an excellent correlation for both contamination species, demonstrating that the technique gives a reliable measure of contamination in the ranges of interest for cleaned steel strip in industrial processing lines.

By transposing these relationships to full coil data traces, it can be seen that the LIBS technique gives very high levels of measurement sensitivity.

Statistical analysis of the trace data shows a tight distribution of measurement variation, giving standard deviations of measurement for oil at 0.47 mg/m<sup>2</sup> and iron fines at 0.88 mg/m<sup>2</sup>. Table 1 shows the overall measurement performance, considering the measurement sensitivity to be defined by  $\pm 2$  standard deviations.

This high level of measurement sensitivity is further confirmed by Gossuin et al.,<sup>4</sup> in which the calculated variability of like-for-like measurements was shown to be ~5 times tighter for LIBS than the established laboratory tests.

Table 1

#### Overall Measurement Performance

Contamination species	Range mg/m <sup>2</sup>	Sensitivity mg/m <sup>2</sup>
Oil	0–120	$\pm 1$
Iron fines	0–200	$\pm 2$



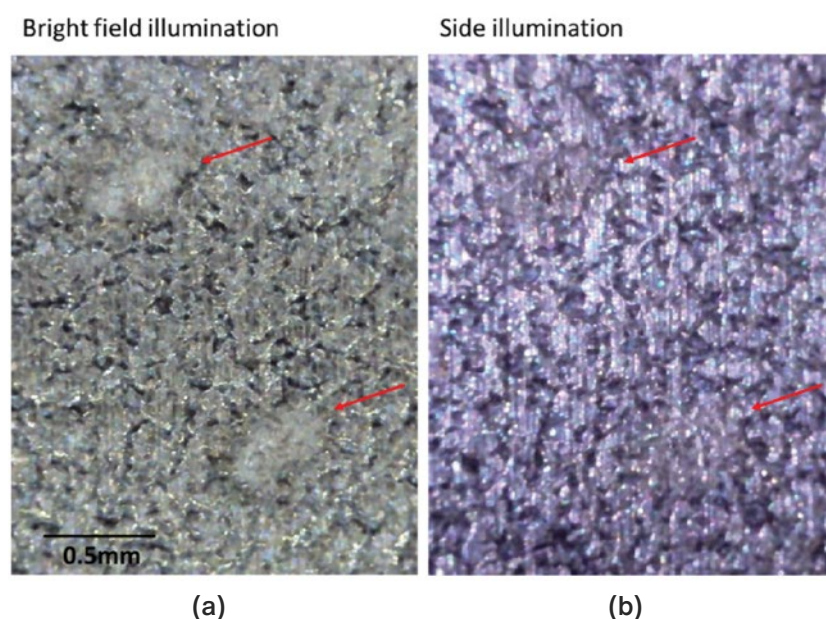
Figure 5

Photograph of blemishes on processed coil (industrial trial).



Figure 6

Optical microscopy of the laser blemish by bright field illumination (a) and side illumination (b).



**Suitability of Measurement Technology:** Surface quality is of paramount importance to many steel strip products; indeed, it is the very intent of this technology to facilitate improvements in such. LIBS technology is a noncontact process and so does not impart any mechanical damage to the product surface. However, it should be noted that the generation of the plasma by the LIBS process does impart a light blemish on the surface the strip (<0.5-mm diameter), as seen in Fig. 5.

Work has been undertaken to characterize this blemish to ensure that it is of no material concern to either the subsequent processing steps or final product quality of the coated steel strip. Optical microscopy of the blemish shows that there is no impact on the topography of the strip; this is particularly evident when the illumination is changed from bright field imaging to side illumination, a technique commonly used to highlight topographical features. This is shown in Fig. 6.

This is further demonstrated by characterization with a confocal microscope, which can be used to accurately measure the surface topography and derive a 3D map of the area affected by the plasma generation. Fig. 7 clearly shows that despite the sample area giving a visible difference by reflectance of light, no discernible change has been made to the strip surface texture.

Previous studies have been undertaken by CRM,<sup>5</sup> using scanning electron microscopy and utilizing backscattered electrons, similarly showing no topographical or chemical analysis change in this region. The studies also showed that wettability in the zinc coating process was unaffected, and bend tests were performed to show no deterioration in zinc adherence.

This minimal impact can be attributed to the carefully controlled process applied by the LIBS device. Care has been taken to ensure that an effective nitrogen shroud is present at the point of

laser impact. This is principally required to derive accurate independent measurement of oil contamination but has the additional benefit ensuring that oxidation reactions do not occur in the plasma. Moreover, the precise control of the conditions for plasma generation allows for a lower-energy laser pulse of typically ~1 W, reducing the potential for material damage.

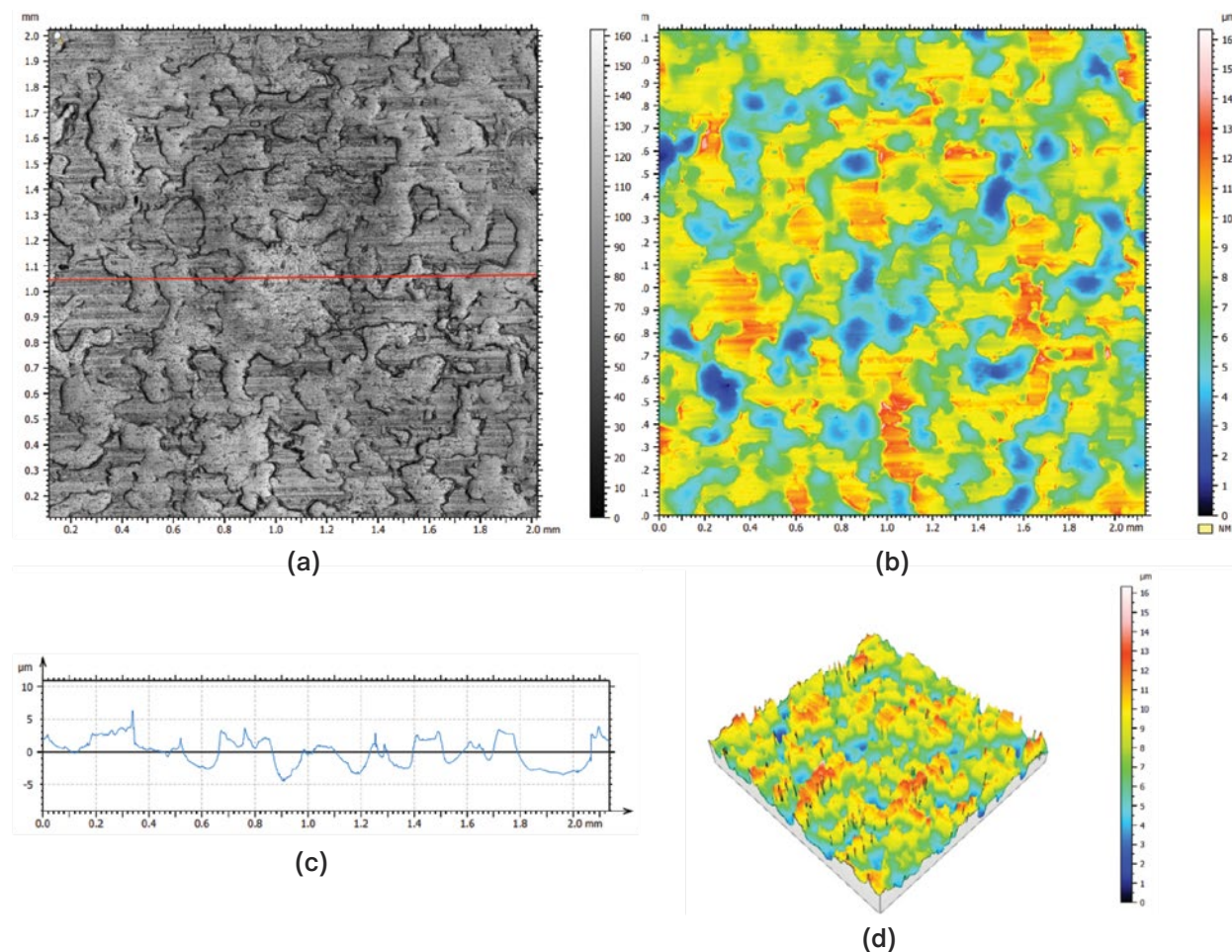
The nitrogen shroud also protects the system optics from damage due to buildup of process dirt from the industrial environment.

### Commercial Installation

Having established reliable data acquisition and demonstrated the suitability of the technology in the industrial environment, work has been undertaken at Sarclad (Rotherham, U.K.) to develop a robust commercial design for permanent installation at any typical cleaning

Figure 7

Confocal imaging of the steel strip surface at the area of plasma generation: Reflective intensity (a), topographical view of same area (b), line scan of topography of marked intersection (c), and 3D view (d).



line. The design review incorporated principles of design for manufacture, maintenance and use, and considers the operating environment and intent of the steel producer. The resultant product adheres to all relevant standards attains CE marking. The core areas of improvement are covered in the following.

**Automated Line Traverse:** A clear requirement for production use is the ability to provide measurements at any point across the width of the strip. To facilitate this, the unit is mounted on a motorized traverse rail which is controlled by an industrial programmable logic controller (PLC) capable of integration with the strip processing line process control to move to setpoints according to strip width and desired point of measurement. The PLC also facilitates system safety protocols and performance diagnostics.

Figure 8

Overview of the designed commercial solution.

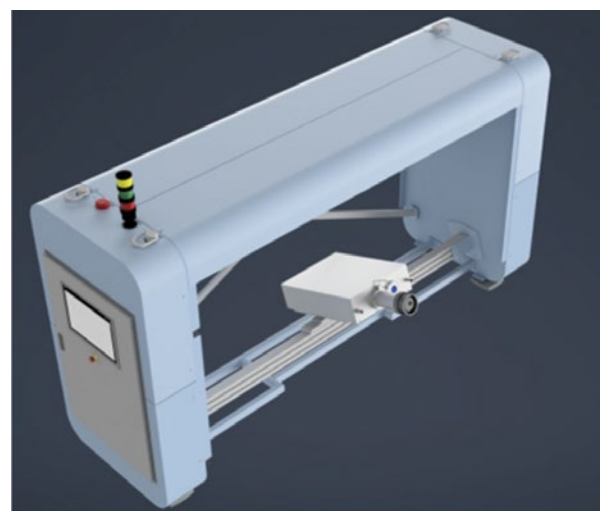
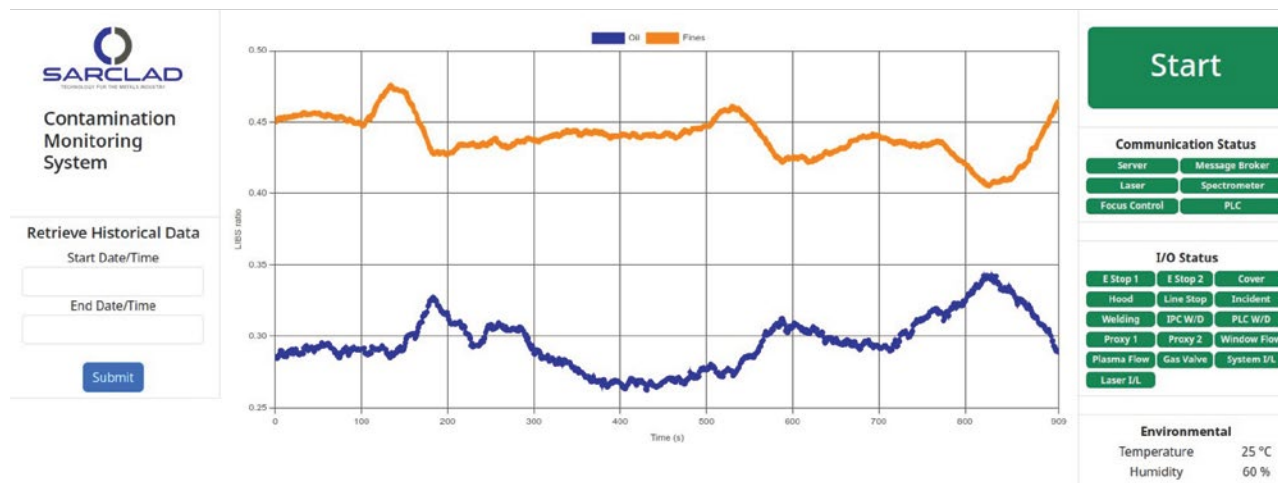


Figure 9

Software screenshot showing the live data view.



**Operator Interface, Data Analytics and Integration to Production Systems:** A web-based user interface has been developed to allow real-time access to the data by operators and control of core commands. The interface has been designed on a modular basis to cover all aspects of controls and communications utilizing Industrial Internet of Things methodology in a unified namespace to ensure seamless integration to plant systems and safe operation. The system is Industry 4.0 ready, utilizing data structures that are efficient for network traffic and are fully scalable.

The human-machine interface has an intuitive design, Fig. 9, and allows the operator to access real-time data, review historical data and export data for further analysis off-line, and perform system operational setup according to product mix/line operation. Communication to plant operating systems is via OPCUA.

**Simplified Design for Robust Operation and Maintenance:** The demonstration unit used in this study had certain design features that allowed flexibility of operation to optimize the technology in a research context. Elements of these flexibilities are not required in a permanent production unit. The principles of design for build and maintenance have been employed to arrive at a more robust design for the industrial environment.

**Laser Safety and System Security in the Operational Environment:** The installations in this study have fully complied with laser safety requirements by utilizing a combination of robust systems interlocks, shielding and exclusion zones. This is enhanced in the commercial product by designing in key shielding arrangements to the housing and site-specific working practices. Further, the integrated design facilitates armoring to protect the measuring head in the event of

a strip break, etc., with only the sacrificial laser beam shroud being exposed. The equipment is rated to IP53 to protect from dust and water, with the innovative laser beam shroud protecting the external optic. Site-specific measures will be incorporated to mitigate the effects of extreme temperatures, humidity and vibration.

## Conclusions

The application of LIBS technology, as developed by CRM, has been successfully demonstrated to reliably measure the contamination levels of oil and iron fines as independent and discrete species on the steel strip. Furthermore, this has been achieved in the industrial environment with an on-line sensor taking live measurement on the production line at full operating speed.

The reliability of the measurement has been demonstrated over a suitable range with very high levels of measurement sensitivity, as shown in Table 1.

Practical experience has shown that the minor blemish made by this noncontact measurement is not visible after coating. Further microscopy and laboratory tests have shown that the blemish makes no topographical change to the steel surface and no chemical or microstructural change occurring. Thus, the measurement process does not adversely affect either the galvanizing process or the end product quality.

A robust engineering solution has been developed to implement the technology in the industrial environment of a steel strip processing line. The unit has been exposed to the rigors of operation in such an environment and performed effectively. An appropriate software and control system has been developed to make this product ready for implementation and integration to any strip processing line.




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