AppNotes MARCH 2016

Fast, Laser-based Cr Analysis for Flow Accelerated Corrosion (FAC) Applications

Laser-induced Breakdown Spectroscopy (LIBS) offers an alternative technique to both handheld XRF and spark OES for the analysis of a broad range of elements and alloys. With LIBS a plasma is created at the material surface as with spark optical emission spectroscopy (OES). Spectral lines from the various elements present are measured as the plasma cools. The wavelength of specific lines reveals the elements present, and the intensity of the light at a given wavelength is related to the concentration of each element.

SciAps is pleased to introduce the Z, a handheld analyzer utilizing laser induced breakdown spectroscopy (LIBS). There are three "must have's" for handheld LIBS when analyzing most alloys, especially ferrous, stainless and high temps. Those include a) a high energy pulsed laser, The Z uses 6 mJ/pulse at a 50 Hz rep rate, b) novel 50 Hz burst cleaning to eliminate sample grinding, and c) Opti-Purge[™] on-board argon purge for 10x or better precision. (Note the Z can also be equipped for ONLY airburn analysis for sites with compressed gas canister restrictions). The Z offers a number of advantages for FAC compared to traditional handheld XRF. Those advantages include faster Cr analysis at low concentrations (0.05%) – typically 3 seconds. The Z is laser based, and thus there is no ionizing radiation like X-ray. The elimination of X-rays greatly reduces the regulatory burden especially at nuclear fueled power plants. Finally the Z delivers the low atomic number performance (Li, Be, B, Mg, Al, Si) of mobile OES, while maintaining the portability of handheld XRF.



Opti-Purge™on-board argon purge





FAC Applications:

For FAC there's a need to quickly measure the chromium content in carbon steels. If the chromium concentration decreases below 0.1% (typical threshold value) the rate of corrosion in carbon steel flow systems increases rapidly. Handheld XRF has been used effectively for Cr analysis in carbon steels and in fact the SciAps X is one such analyzer. However LIBS offers several advantages. The Z requires about a 3 second test to measure 0.05% Cr. XRF typically requires 10 seconds or more to reach a suitable precision at the 0.1% concentration range in carbon steel. The Z is laser-based and is not subject to the many regulations required for x-ray. Many facilities, especially power plants, have very restrictive regulations concerning the use of handheld XRF. Table 1 shows 10x repeats for two Cr concentrations. One material is a carbon steel 1018 with Cr amount 0.111%. The second material is a 1117 carbon steel with Cr assay 0.076%. Repeatability data is shown in Table 1 including the standard deviation. Applying a confidence interval of 2 standard deviations to the 1018 alloy Cr result, the Cr is still measured reliably above a 0.1% threshold value. For the alloy 1117, a two sigma confidence band shows a maximum of 0.084% LIBS measurement for Cr. Thus Cr levels of concern below a typical 0.1% threshold are reliably measured as well.

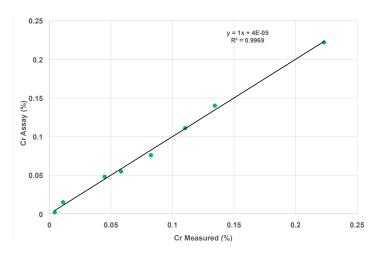
| Tal | ble | 27. |
|-----|-----|-----|
| | | |

| 1018 | Cr (%) | 1117 | Cr (%) |
|-----------|--------|-----------|--------|
| 1 | 0.109 | 1 | 0.078 |
| 2 | 0.113 | 2 | 0.077 |
| 3 | 0.115 | 3 | 0.081 |
| 4 | 0.120 | 4 | 0.077 |
| 5 | 0.119 | 5 | 0.079 |
| 6 | 0.104 | 6 | 0.079 |
| 7 | 0.112 | 7 | 0.076 |
| 8 | 0.116 | 8 | 0.079 |
| 9 | 0.121 | 9 | 0.078 |
| 10 | 0.113 | 10 | 0.074 |
| Average | 0.114 | Average | 0.078 |
| Std. Dev. | 0.0054 | Std. Dev. | 0.002 |
| RSD | 4.7% | RSD | 2.3% |

The Z makes a great alternative to XRF. It's a laser based system that can be operated under Class 1 conditions, thus eliminating all of the regulatory burden associated with X-ray. The Z also produces very good Cr results with a maximum 3 second test times. Results for several carbon steels comparing the handheld Z results versus certified assay are shown in Figure 1. The Cr limit of detection in 3 seconds is lower than 0.015%. The accuracy is also quite good as seen from the correlation. The slope is identically 1 to 4 significant figures, with virtually a zero offset.

Figure 1:

Measured Cr Versus Cr Assay for FAC Application



Summary:

The Z is the world's most advanced handheld LIBS analyzer. The Z possesses the three key requirements for successful, in-field analysis of a range of materials: a) burst cleaning to eliminate sample surface effects and grinding, b) argon purge (optional) for 10x or more precision compared to air-based analysis, and c) beam rastering. This combination provides proven performance on a range of materials – aluminum alloys, red metals, nickel and stainless and ferrous. The novel sample detection system allows the device to be operated under Class 1 conditions, thus eliminating the regulatory requirements of x-ray and class 3b LIBS devices.

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