Technologies for On-Site Positive Material Identification

White Paper

Typical application for PMI: Petrochemical plant

Positive Material Identification (PMI) programmes allow plants operators and maintenance professionals to test critical process components before and after they are put in service, so they do not have to just rely on the suppliers' certificates.

Chemical, petrochemical and power plants in particular have to use very specific materials that will withstand extreme conditions. Effects such as flow accelerated corrosion (FAC) or corrosion in the presence of chlorine might results in tension cracks. Another example is HTHA (High Temperature Hydrogene Attack), which leads to crack buildign in the material. This can be avoided by carrying out systematic monitoring of the materi-als over time using portable analytical test equipment on site, so shutdown of the plant can be avoided.

Until now there have been two established technologies for PMI inspection:

1. Handheld X-ray fluorescence spectrometers (HHXRF), such as the **X-MET** series, and
2. Mobile and portable optical emission spectrometers (OES) such as the **PMI-MASTER** Smart

Oxford Instruments recently launched the **m**Pulse, the world's first handheld metal analyser based on laser induced breakdown spectroscopy (LIBS). Although LIBS technology has been available for years, the **m**Pulse is the first commercially available handheld instrument. Now three technologies for PMI are available to users according to their specific application needs.

What are the advantages and limitations of these metal analysis technologies?

All three technologies may be used to analyse the elemental composition of metal samples and identify their specific alloy grade, but there are distinct differences in element selection and detection ranges.

OXFORD INSTRUMENTS

The Business of Science®

Technologies for On-Site Positive Material Identification

Handheld X-ray fluorescence (HHXRF)

HHXRF utilises an X-ray tube to induce a response from the atoms in the tested sample. It is a non-destructive technique, and is ideal for the analysis of finished surfaces. Standardless FP (fundamental parameters) analysis allows the testing of a wide range of elements and concentrations, and works well on flat surfaces, powders and granules. Empirical calibrations are derived by measuring a series of reference materials of a given type (e.g. copper alloys), and provide the best accuracy and precision, especially for light elements (Mg to S) and trace elements. HHXRF detects elements from element number 12 (magnesium) onwards. This means that elements such as carbon in carbon steel, nitrogen in duplex steel, or beryllium in copper cannot be analysed. Elements such as silicon, aluminium, and sulfur can be measured in less than 10 seconds at > 0.1% concentration. In spite of this limitation, HHXRF can be applied in many areas, for example the measurement of chromium, copper and nickel for FAC monitoring, the rapid alloy verification of incoming parts and in-service welds and components.

In fact, HHXRF is most prevalent because it is easy to use, works well for the vast majority of common alloys, and is truly portable. It can be used at locations that are difficult to access, it can measure in hard-to-reach corners and in small spaces.

X-MET*8000*

Handheld XRF

Portable optical emission spectroscopy (OES)

The **PMI-MASTER** Smart is a portable spark OES instrument which can analyse all important elements at low limits of detection, including carbon at levels of less than 0.03 % (300ppm), which characterises L-grades (304L, 316L). Nitrogen in duplex steels and low levels of titanium, aluminium and vanadium in low alloy steel can be detected.

However, there are limitations: compared to HHXRF the OES technique is more demanding. An electrical discharge (spark) needs to be generated under argon atmosphere to protect from oxidation. Furthermore visible burn spots are left on the surface. Spark OES metal analysers can only be used for the analysis of metals for which they have been calibrated, although it is fairly simple to create additional calibrations on modern OES analysers.

PMI-MASTER *Smart*

Portable optical emission spectrometer

Samples for spark OES need to be conductive, of a certain size and require sample preparation by grinding or milling. Powders and chippings cannot be detected.

The energy consumption of the spark generation is relatively high. Powerful batteries and Argon in compressed gas cylinders are required, limiting the operation of portable OES analysers. However, the portable PMI-MASTER Smart with its UVTouch probe enables to take the analyser to hard-to-reach spots in a safe manner.

Despite its limitations, the superb analytical performance of portable OES makes this technology a powerful tool for the analysis of metals, as its element range, accuracy and precision reach levels close to those of laboratory OES instruments with much lower detection limits than HHXRF and LIBS.

***m**Pulse*
Handheld LIBS

Flanges
Typical parts to be tested by PMI

Handheld laser induced breakdown spectroscopy (LIBS)

Compared to HHXRF and portable OES, LIBS is a relatively new technology for on-site metals analysis. The technique became possible due to powerful laser diodes. A laser pulse hits the sample surface causing a tiny amount of the sample to be ablated. The ablated mass further interacts with a trailing portion of the laser pulse to form a hot, highly energetic plasma that contains free electrons, excited atoms and ions. After each pulse, the plasma cools and characteristic light emissions can be observed as the energised ions return to their original atomic states.

In a typical test, the sample will be hit by thousands of pulses. The emitted light from the plasma is collected with a CCD for analysis. Each element in the periodic table is associated with unique emission lines, and the lines' intensities are proportional to the amount of the element: LIBS can be used to determine a sample's chemical composition rapidly. The laser burns are hardly visible and can be easily removed, making this a virtually non-destructive testing method.

The mPulse analyses samples in only one second. Due to size constraints, the wavelength range of the LIBS optics is limited

LIBS can typically detect the same elements as HHXRF, but is much quicker, especially when identifying aluminium alloys.

The mPulse can also determine elements that are not measurable by HHXRF, such as beryllium in copper. Because LIBS is mostly a surface analysis technique, samples have to be cleaned of contaminants (e.g. rust, dirt), and unlike OES, they can be conductive or non-conductive (as for HHXRF). The focussed laser beam with a diameter of less than 1 mm allows the analysis of very small pieces.

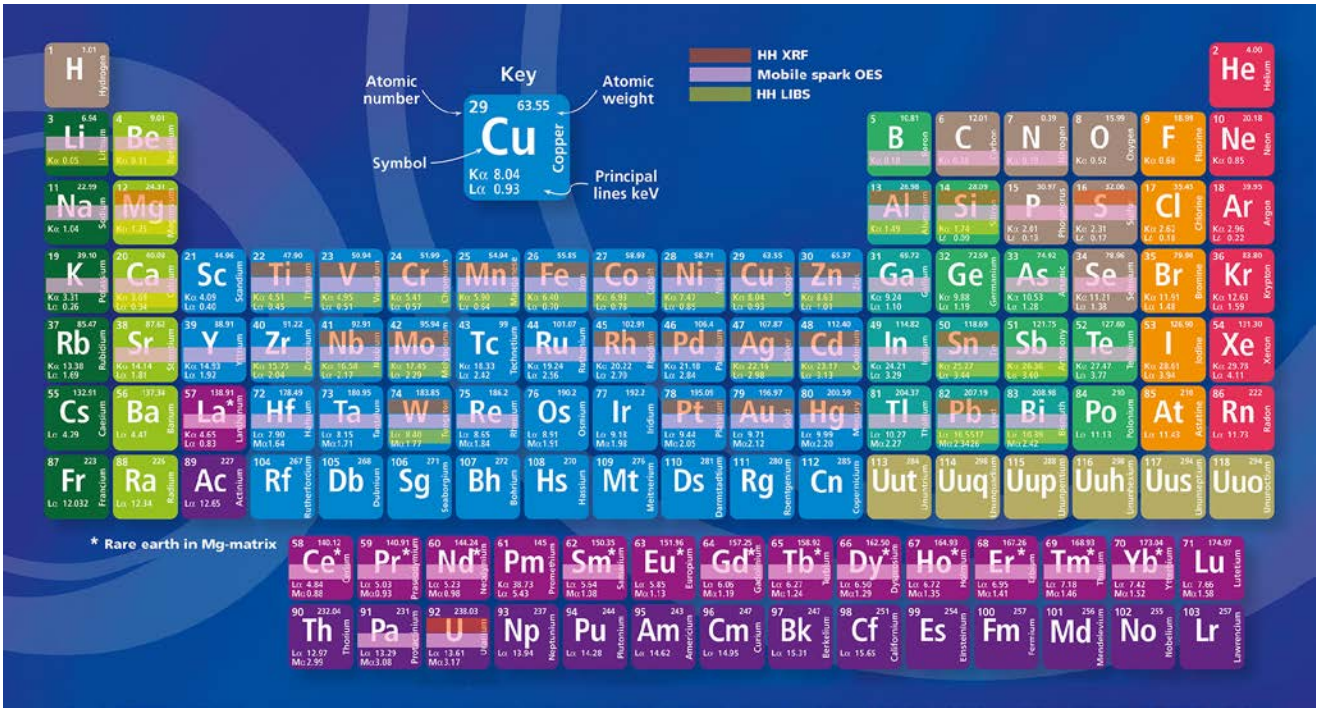
Conclusion

Portable OES analysers offer the best performance range for PMI inspection, but they are not as compact and easy to use as hand-held XRF or LIBS instruments. Their operation requires careful instructions and knowledge of sample preparation.

HHXRF and LIBS can be operated after a minimum amount of user training. Both types of handheld analysers are compact, lightweight and rugged, and can be used without additional accessories. Their limitation is mostly in the element range they can determine (for example, neither can separate L grade steel from non L grades).

The choice of technology depends heavily on the end-user requirements, i.e. elements of interest and the level of accuracy, precision and the limits of detection required, but also on ease-of-use, instrument size and weight.

For detailed product information and consultation about your specific application, please contact Oxford Instruments at industrial@oxinst.com



The periodic table displays which elements can be detected by which handheld / portable technology and thus facilitates the decision

Visit **www.oxford-instruments.com** for more information or contact **industrial@oxinst.com**

OXFORD INSTRUMENTS

The Business of Science®