Application note | AN41652

X-ray fluorescence

Geological and metal sample surface mapping with WDXRF spectrometer

Keywords
Elemental analysis, WDXRF spectrometer, XRF analysis, XRF elemental analysis, XRF instrument, XRF spectrometer, XRF spectrometry, XRF technique, XRF technology

ARL PERFORM'X Sequential X-Ray Fluorescence Spectrometer

Introduction

Wavelength dispersive XRF spectrometers are typically used for quantitative elemental analysis of many material types ranging from petrochemical, geochemical, metals, glass and ceramics, mining and cement. These samples are normally presented to the XRF instrument as uniform and homogenously prepared samples

With the introduction of the Thermo Scientific™ ARL™ PERFORM'X Spectrometer, a new dimension of sample analysis is now possible with sample surface mapping. The mapping capability enables heterogeneity, contamination, gradient, segregation and inclusion determination and analysis.

The ARL PERFORM'X mapping option can construct detailed composite maps of elemental distribution within a sample. The cartography control and overlay has a fine resolution of 0.1 mm steps providing superior analysis for process improvement and problem solving applications.

This ability bridges the gap between traditional bulk analysis and standard micro-analysis using microscopic techniques such as SEM.

Instrument

Thermo Scientific ARL PERFORM'X Series Spectrometer used in this analysis was a 4200 watt system. This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors, helium purge and our 5GN+ Rh X-ray tube for best performance from ultra-light to heaviest elements thanks to its 50 micron Be window. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month.

The ARL PERFORM'X offers the ultimate in performance and sample analysis safety. Its unique LoadSafe design includes a series of features that prevent any trouble during sample pumping and loading. Liquid cassette recognition prevents any liquid sample to be exposed to vacuum by mistake. Over exposure safety automatically ejects a liquid sample if X-ray exposure time is too long.

The Secutainer system protects the primary chamber by vacuum collecting any loose powders in a specially designed container, easily removed and cleaned by any operator. For spectral chamber protection, the ARL PERFORM'X uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. In the "LoadSafe Ultra" optional configuration, a special X-ray tube hood provides total protection against sample breakage or liquid cell rupture. The ARL PERFORM'X analyzer features mapping and small spot analysis allowing for 1.5 mm and 0.5 mm areas.

Mapping of geological samples

The ARL PERFORM'X mapping of this geological sample illustrates the ability to define and locate grain boundaries and crystalline structures that are not visible to the naked eye.

The formation of pseudotachylite is usually studied without reference to its chemistry, but in this example we can see a wealth of chemical information that bears upon the genesis of the material. Al, Si, K, Ca and Fe show large gradients while Na, Mg, Ti and Sr are more evenly distributed. Another example is shown in Figures 3 and 4 with a feldspar sample having a clear inhomogeneous structure. The ARL PERFORM'X identifies the element constituents in the selected section of the sample.

The interesting aspect of this analysis is in the differences of chemistry of the two feldspars, and how the plagioclase comes to mantle the earlier alkali feldspar. The chemistry gives a lot of basic information regarding this process.

Mapping of metallic samples

Mapping can be used for easy identification and quantification of contamination or metallic inclusions as shown in Figures 5 to to 8 of a copper rod inserted in a steel disc.

Standard-less analysis for mapping

The most useful development of XRF analytical programs has been the availability of "standard-less" packages. These packages allow for quantitative data to be obtained for completely unknown samples.

As in many real life situations, obtaining any or enough standards to create a calibration is not always possible. This is certainly the case when analyzing defects or unknown contamination. In such situations, we can offer the most comprehensive standard-less software on the market: Thermo Scientific UniQuant package.

It is a factory calibration based on 64 pure element standards that allows for concentration determination of unknown samples in any matrix by using complex mathematical algorithms for up to 79 elements. These algorithms correct for matrix effects as well as inter-elemental effects to provide a precise quantitative result.

Conclusion

The elemental mapping capability is a very interesting tool for all applications dealing with heterogeneity or concentration gradients in samples as well as when impurities or inclusions are found in given specimens. It is seen that analysis using mapping can easily be performed with the ARL PERFORM'X Sequential XRF Spectrometer. Operation is made easy through the state-of-the-art Thermo Scientific OXSAS Software that operates with Microsoft Windows 10 package.

Figure 1: Pseudotachylite sample with analyzed area.

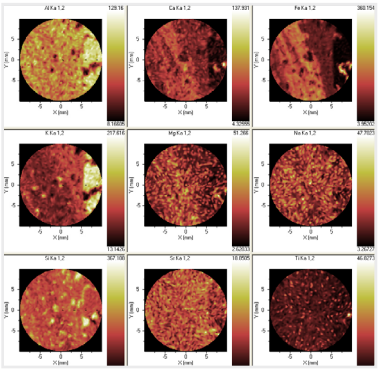

Figure 2: Distribution of nine elements in the sample (lighter color means higher presence).

Figure 3: Heterogeneous feldspar sample seen in the 30mm aperture sample holder.

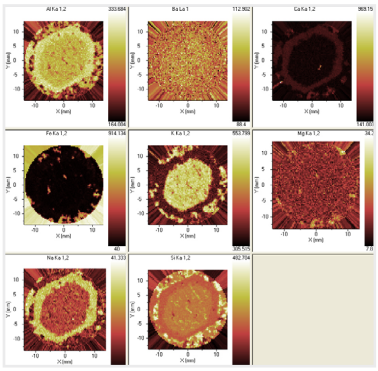

Figure 4: Distribution of eight elements in the sample.

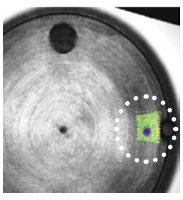

Figure 5: Selected area for mapping in green.

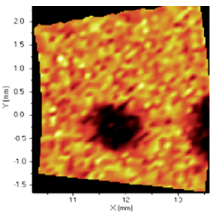

Figure 6: Distribution of Cr.

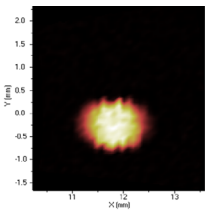

Figure 7: Distribution of Cu.

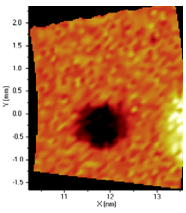

Figure 8: Distribution of Fe.

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