

## X-ray fluorescence micro-analysis for environmental science

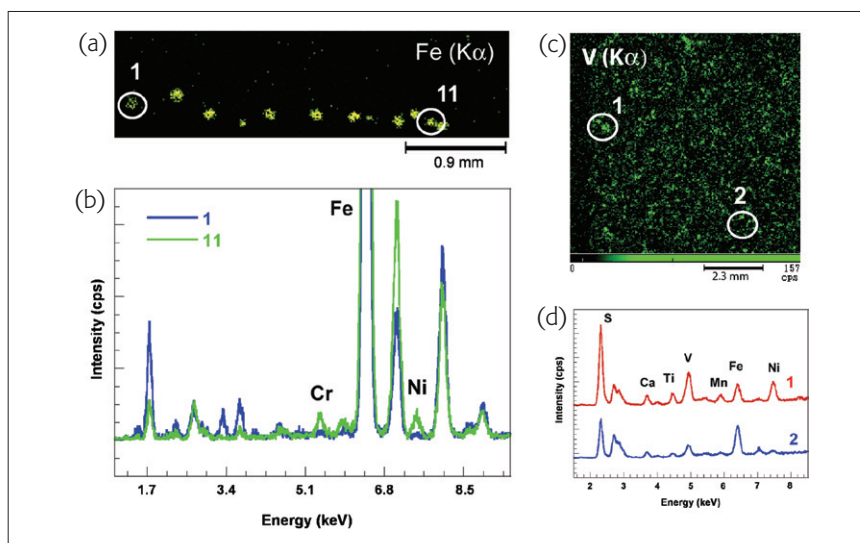
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## Introduction

X-ray fluorescence (XRF) is a well-established technique for fast quantitative and qualitative elemental analysis. One very important area which is seeing increased interest is environmental science. XRF is already providing useful information for scientists working in this field, whether it be for quantitative analysis of contaminants in soils, identification of radioactive elements (e.g. uranium), composition analysis of rocks and minerals, characterisation of materials for recycling etc.

In recent years, developments in x-ray optics and the ability to create high intensity microscopic x-ray beams has led to a new generation of laboratory energy dispersive XRF instruments capable of analysing discrete particles as small as 10 µm, and scanning surfaces to create fast high-resolution elemental images. With mono-capillary optics collimated beams are possible, with diameters ranging from a millimetre down to a few micrometres, providing % and sub-% concentration for the elements from sodium upwards. This removes the need for traditional sample preparation, and even large samples can be analysed whole, while individual features can be interrogated to produce a detailed characterisation of elemental composition. In the case of environmental sciences for which the studied samples are generally disordered, heterogeneous and/or fragile, non-destructive X-ray fluorescence micro-analysis is an ideal technique.

Industry has quickly grasped this technology with confidence as a method of ensuring compliance to the new RoHS (Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment) directive. Micro-XRF allows even minute electronic components and circuitry to be individually analysed in a matter of seconds or



**Figure 1.** (a) elemental image (Fe  $K\alpha$ ) of micrometeorites, acquired with 10 µm beam, (b) spectra obtained from micrometeorites 1 and 11, (c) elemental image (V  $K\alpha$ ) of petrochemical dust, and (d) spectra obtained from high and low vanadium concentration particles.

minutes, with sensitivity well within the limits defined in the directives. As a fast, routine screening tool, the technique is unrivalled.

However, beyond this very active embrace of micro-XRF there are numerous other areas where the same technology is helping researchers to study and protect our environment.

## Terrestrial and extra-terrestrial dust particles

Perhaps the most obvious application of micro-XRF is for the analysis of individual microscopic particles, where more traditional techniques such as bulk/macro XRF or ICP are not applicable. Figure 1(a) shows an elemental image acquired over a number of micrometeorite particles all of which are less than 100 µm in diameter. The earth is constantly bombarded by such extra-terrestrial dust, which is believed to be a fundamental building block in the formation of planets, and

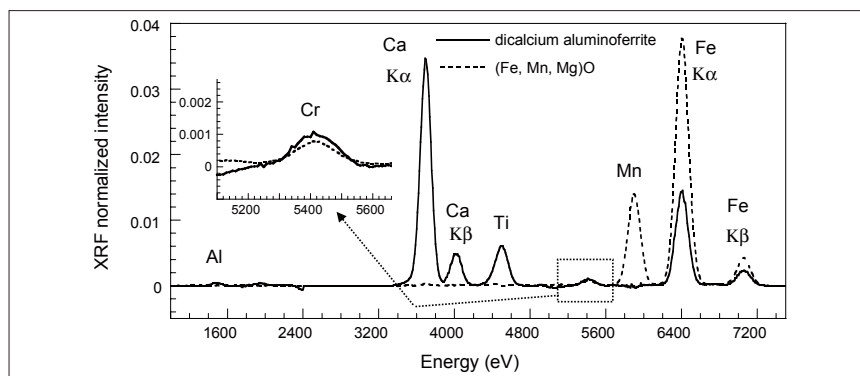
a pathway for the introduction of vital mineral materials to the young planet Earth. Such samples are nevertheless rare and require non-destructive analysis. Moreover the composition of micrometeorites is generally altered at the surface (to a depth of a few microns) depending on the time spent within the terrestrial atmosphere. Therefore surface-only techniques like SEM-EDX are unable to provide chemical analysis of the near-surface to bulk regions—the deeper penetration of XRF analysis thus provides additional information. Spot analysis of two of these grains [Figure 1(b)] clearly shows that their material composition can be identified and distinguished with analysis times of just 60 s.

Figure 1(c) shows far more numerous atmospheric particles caused by petrochemical industrial processes—these contain vanadium, a toxic pollutant which can cause various harmful effects in humans, particular in the lungs, eyes,

nose and throat. Quantitative analysis of these particles collected on filters identifies two main forms, with significant differences in V, S, Fe and Ni concentrations. Understanding particle composition provides useful information on their toxicity, but such analysis is impossible with non-spatially resolved techniques.

### Recycling waste: Reuse of steel slag

Basic Oxygen Furnace (BOF) steel slag is obtained during the transformation process of pig iron to steel in LD converters and is attractive as a construction material due to its excellent technical material properties. This by-product is mainly composed of Fe, Ca, Si but also contains trace elements potentially harmful to the environment like Cr. Therefore the decision to reuse it should be based on solid knowledge of their environmental impacts particularly from the durability aspect. A SIMPLe-to-use Interactive Self-modeling Mixture Analysis (SIMPLISMA) of the micro-XRF data [chemometric analysis of micro-XRF (multipoint mode)] clearly indicates two different Cr locations in BOF slag: a solid solution phase (Fe, Mn, Mg)O and a dicalcium aluminoferrite phase (Brownmillerite). Both of these phases remain unaltered when BOF slags are in contact with water,



**Figure 2.** Resolved spectra obtained with SIMPLISMA procedure.

which clearly explains the low level of Cr release during leaching tests.

### Conclusions

The ability to analyse individual microscopic particles for composition and create detailed elemental images with compact benchtop micro-XRF analysers is being widely embraced by scientists in varied application areas, including forensics, pharmaceuticals, geology, materials and electronics. For environmental scientists, the move from older bulk XRF techniques to the latest microscopic techniques can provide a new dimension in their research, and provide insight to the earth and the universe, and the impact mankind is having.

### Acknowledgements

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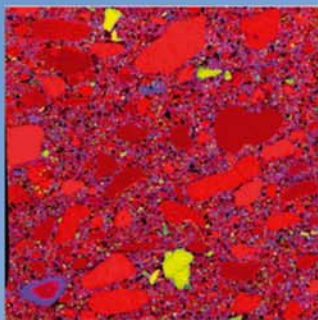
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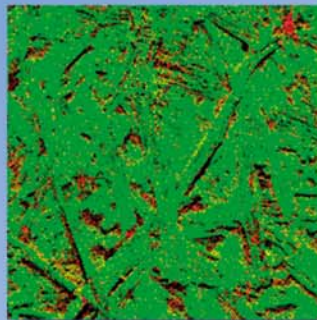
## Geology

Rock sections  
Grain boundaries  
Particle composition  
Sediment cores  
Phase analysis



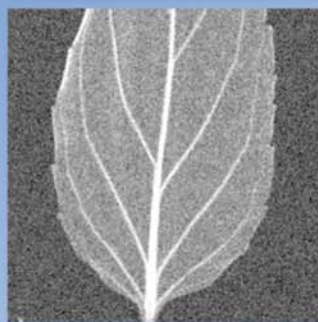
## Materials

Alloys and metals  
Semiconductors  
Layer thickness  
Concrete and cement  
Engine wear particles  
Fillers and coatings



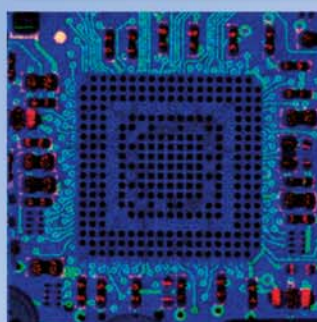
## Biology / Medicine

Tissue  
Metabolic accretions  
Leaves  
Bone  
Contamination  
Heavy metals  
Drug pathways  
Disease diagnosis



## Electronics

Resin embedded components  
Solder defects  
WEEE/RoHS  
Troubleshooting  
Plating thickness  
Contaminants



## Forensics

Fingerprints,  
Glasses,  
Gun shot residues,  
Gems  
Inks  
Pigments



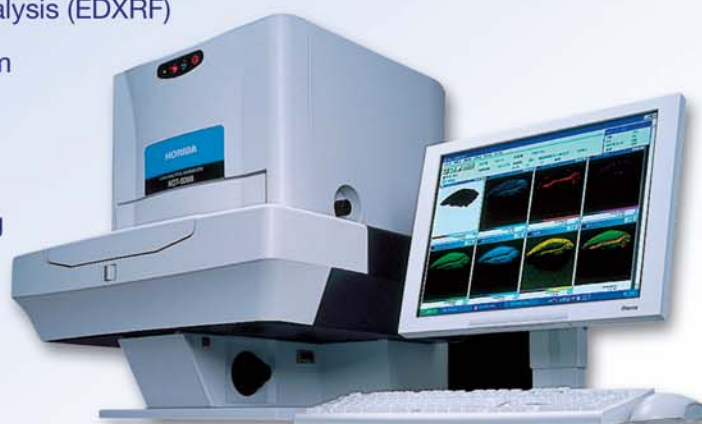
## Museums

Inks and pigments  
Papers  
Gemstones  
Inclusions  
Internal structure analysis  
Corrosion  
Metals / alloys



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