HORIBA Scientific

Application Note

Material Science

XGT

XGT13

Micro-XRF analysis for metal debris and particle analysis

The characterisation of microscopic particles remains an important task for analysts, whether in forensic science (eg, glass fragments, paint flakes, gun shot residues), environmental science (eg, soil, atmospheric pollutants, cosmic dust), geology (eg, rock particles for mining exploration) and engineering (engine wear debris).

The bench top XGT-5000 x-ray fluorescence (XRF) micro-analyser is ideally suited for just such analysis, for it offers fast, non-destructive elemental characterisation with a unique high spatial resolution (10 µm). The analysis is both qualitative and quantitative, so it can quickly identify specific elements present or provide compositional information with ppm/percent accuracy (eg, specific alloy chemistry)

Metal paticle analysis

Analysis of engine wear debris is an important example where micro-XRF is particularly well suited. Jet engines in regular use experience continuous wear on the various parts of the engine (eg, gearbox, bearings) which results in small metal fragments entering the lubrication oil channels. These can be collected using carefully placed magnets, and are then analysed. Since different metals and alloys are used in the different engine components, by characterising the metallic composition of the fragments it is possible to identify the origin of the wear particles. This provides an engineering team with valuable information concerning the overall engine health.
Similar analyses are used in troubleshooting roles in many manufacturing plants, as a method of providing very fast, detailed composition analysis of small metal fragments.

Making the analysis

The XGT-5000 offers high intensity x-ray beams with 100 µm and 10 µm diameters, thus allowing a wide range of particle sizes to be efficiently analysed. The particles can be simply deposited on the sample stage, or affixed to double sided tape (which is elementally clean for XRF purposes). Low and high magnification cameras provide clear images of the particles, and it is a simple matter to locate a specific particle for analysis (see Figure 1).

Figure 3: High resolution optical image of metal fragments. The red circle indicates the 100 µm analysis spot.

Alternatively, it is possible to scan an area automatically, to build up a detailed elemental image, covering a very large number of particles in one analysis. Thus it is possible to very quickly locate specific particles of interest, which can then be reanalysed in detail if necessary. Once the data has been acquired, a spectral searching module can be used to compare the spectrum with previously saved spectra (from known components), to provide fast and simple identification.

Results

In this example, metal particles trapped in a filter were transferred to a thin polymer sheet and analysed with the XGT-5000.

JOBIN YVON Technology

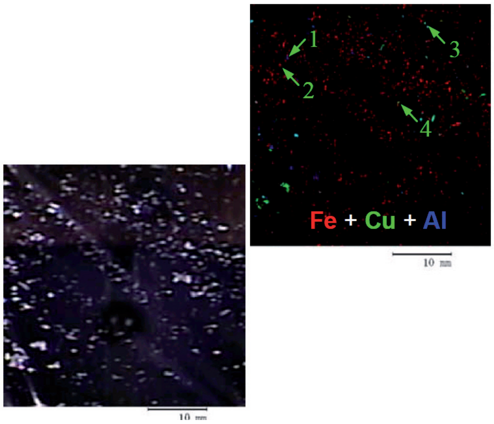


Figure 2: Optical image (left) and element image (right) of microscopic metal particles

Figure 2 illustrates the low resolution optical image taken over a 10 cm x 10 cm area, with hundreds of particles visible, ranging from a few mm, down to a few 10 s of microns in size. A composite element image is also shown, illustrating the majority of the particles to be mainly composed of iron, whilst a smaller of number of copper and/or aluminium fragments are also present. Four particles were chosen for more detailed analysis (labelled in green).

Spot analyses on these representative particles are shown in Figure 3. Particles 1 and 3 are dominated by aluminium, and quantitative analysis indicates these are high purity aluminium (>99%) and aluminium-copper alloy (including other low concentration metals such as lead). Particle 2 is a steel, with >99.9% iron and trace amounts of chromium and manganese. Particle 4 is identified as a tin bronze (copper-lead-tin alloy) – the spectrum is dominated by copper (>80%) but with significant lead and tin content too.

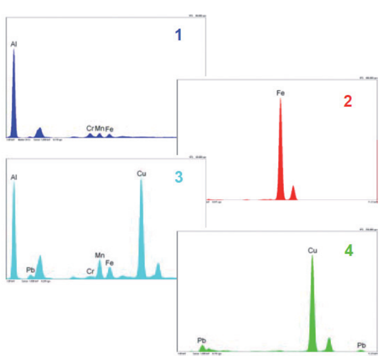


Figure 3: XRF spectra acquired from particles indicated in Figure 2.

Finally, in order to demonstrate the high resolution imaging capability of the XGT-5000, a small area of the original sample was chosen for analysis with the 10 µm beam. Although many of the particles are relatively large (50-100 µm), smaller fragments are also apparent (<30 µm). The excellent beam properties possible with the unique 10 µm x-ray beam on the XGT-5000 allow the shapes of the individual particles to be very accurately imaged. This can provide further indications on the origin of a particle, and the manner in which it was formed.

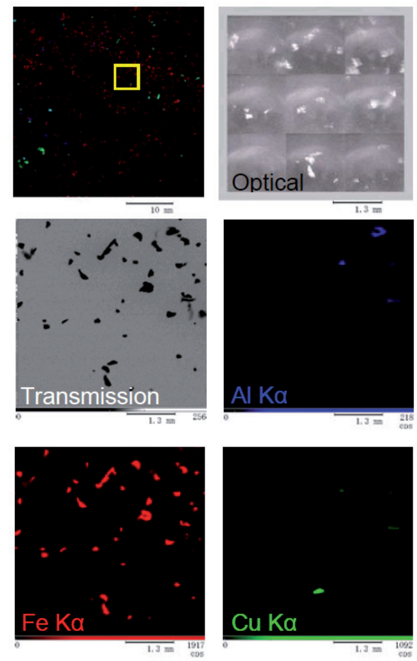
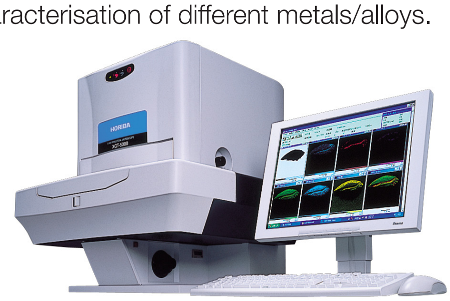


Figure 4: Optical and element images acquired over small area of particles with 10 µm beam diameter. The mapping area relative to the original large area scan is marked by the yellow box in the top left image.

Summary

Fast analysis of minute particles is an important requirement in many varied applications. As illustrated here, the XGT-5000 micro-XRF system is ideally suited for such work, with x-ray beams of 10 µm and 100 µm diameters allowing discrete analysis of the particles. In addition, the automated XY scanning function enables large numbers of particles to be imaged, allowing the easy location of areas of interest. The quantitative nature of XRF provides specifical compositional characterisation of different metals/alloys.



XGT-5000

info.sci@horiba.com
www.horiba.com/scientific

HORIBA Scientific

USA: +1 732 494 8660 **France:** +33 (0)1 64 54 13 00 **Germany:** +49 (0)89 4623 17-0
UK: +44 (0)20 8204 8142 **Italy:** +39 0 2 5760 3050 **Japan:** +81 (0)3 38618231
Spain: +34 91 490 23 34 **China:** +86 (0)10 8567 9966 **Brazil :** +55 11 5545 1540
Other Countries: +33 (0)1 64 54 13 00