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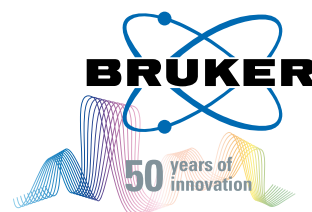
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Advances in μ -XRF

Analysis of elemental distribution in three dimensions



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The macroscopic properties of technical materials are often determined by their elemental composition. Because most technical materials are inhomogeneous, analytical methods are required that allow elemental analysis of these inhomogeneities – not only as a point by point measurement but also as a complete 2D-distribution. Micro X-ray fluorescence (μ -XRF) has recently been established for this purpose. The availability of X-ray optics that capture a large solid angle of tube radiation and concentrate it to small sample areas down in the range of 25 μm makes position sensitive elemental analysis possible with μ -XRF.

Compared to other position sensitive elemental analysis methods μ -XRF provides easy sample handling, which in turn enables quick analysis results. Another advantage of μ -XRF is its high sensitivity for low concentrations with detection limits in the low ppm-range. The method also has a high penetration depth delivering representative analyses of bulk materials and permitting the examination of multiple layer systems.

Conditions for Distribution Analysis

In contrast to other analysis methods, where an electron beam, e.g. EDS, or a proton beam is deflected in order to analyze different sample areas, the sample stage has to be moved for μ -XRF analysis. Therefore, not only the spot size of the analyzed area, but also the step size of the stage determines the spatial resolution. Furthermore the efficiency depends on the speed of the stage and the measured intensity. In order to achieve a sufficient contrast between pixels, it is also necessary to measure at high count rates. This requires both, high excitation intensity, and a detector and data processing system with high count rate capability. In particular if all the accumulated data needs to be saved for further evaluation.

Bruker Nano's new M4 Tornado meets all of these requirements. X-ray tubes with high brightness in combination with efficient poly-capillary optics permit very high excitation intensity for small spot sizes. Fluorescence intensities of more than 300 kcps are measured with the latest generation of silicon drift detectors (SDD). The TurboSpeed stage can move with a speed of up to 200 mm/s enabling

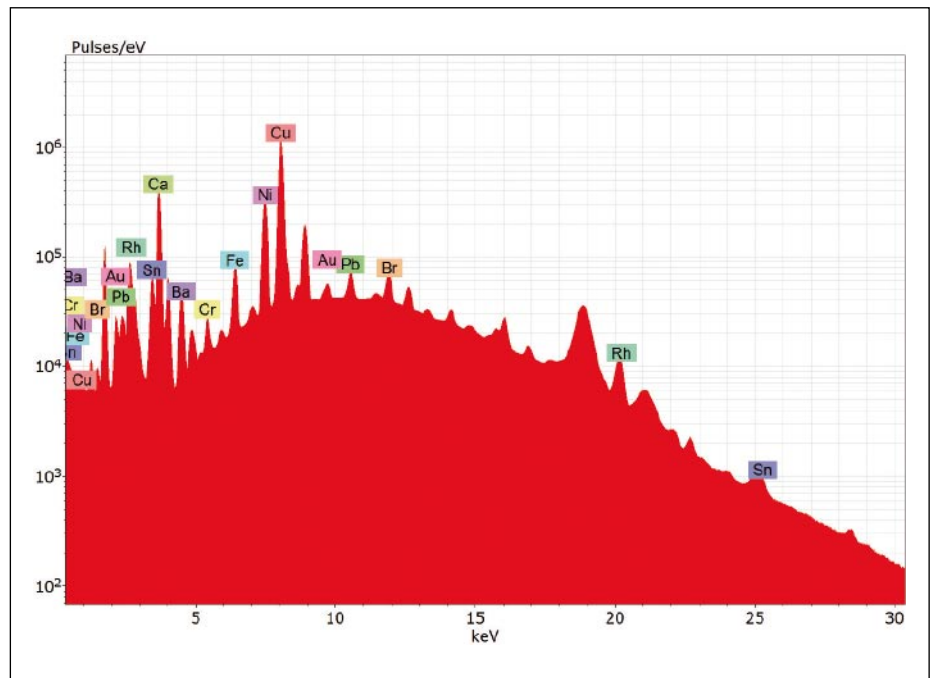


Fig. 1: Sum spectrum of the PCB in a digital watch

a step size of 5 μm and a minimum measurement time of 2.5 ms per pixel. Approximately 1000 counts can be accumulated as spectra and saved together with the pixel coordinates in this short period of time for reference or analysis purposes later on. Bruker Nano combines this position tagged spectrometry function with intensive pre-processing to reduce redundant data in the spectra and compress the amount of data that has to be saved.

In this way large data amounts can be collected and prepared for further intensive evaluation. Various results can be extracted from the saved database. Examples are spectra of single points or of areas with similar composition, in order to improve the statistics for further quantification and distribution analysis in the form of line scans or element mappings. Further options are the calculation of the distribution of any other energy range in order to derive more information from the sample or using chemometric methods for data evaluation and further compression.

Application Example – Measurement on a Printed Circuit Board

The examination of a printed circuit board (PCB) in a digital watch demon-

strates the analysis options described above. The measurement parameters were: analyzed area: 32 x 35 mm with a step size of 80 μm , resulting in 400 x 440 pixels, measurement time per pixel 15 ms, total measurement time below one hour.

Figure 1 shows the sum spectrum of all pixels. This can be calculated by accumulating the spectra of all pixels. The sum spectrum shows a wide variety of different elements as expected in a highly complex and inhomogeneous sample like a PCB. Additionally the “MaximumPixel-Spectrum” can be calculated. For this every channel of the spectra of all pixels is searched for its highest content. A new spectrum is generated from this evaluation showing the highest content of all pixels for every channel. This permits the identification of elements in the analyzed area that are concentrated only in single points and would be covered by spectral background in the sum spectrum, because their concentration averaged over the complete sample is very small.

Distribution analysis provides another interesting data presentation option. Because complete spectra are saved for every pixel, distribution images can also be determined for all energies. This is possible for the fluorescent energy of elements, but can be also carried out for the

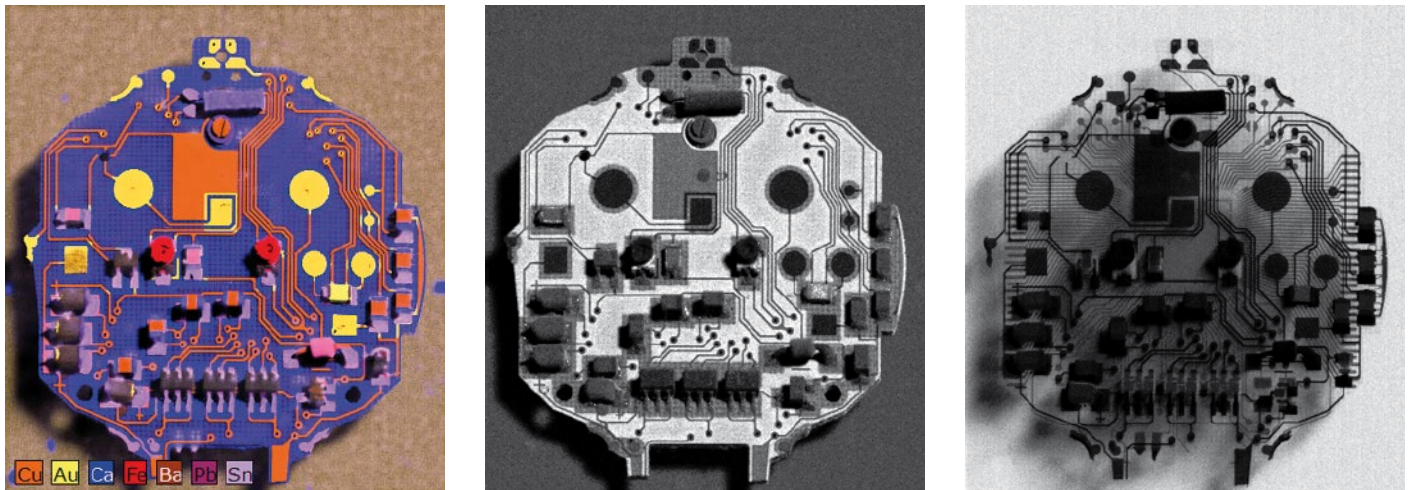


Fig. 2: Distribution of several elements (a) as well as of Rh-L- (b) and Rh-K-scattering (c)

Rh-L and Rh-K scatter lines. The element lines supply the distribution of the corresponding element. The depth of this information depends on the energy of the element's fluorescence line, as well as the absorption in the matrix i.e. the element's atomic number.

The primary radiation of the M4 Tornado's rhodium also penetrates into the sample and is scattered inside. How it is scattered depends on the material distribution in the sample. In particular the high-energy Rh-K radiation is elastic and in turn inelastic when scattered. The intensity of inelastic scattering depends on the average atomic number i.e. it is larger for light elements and smaller for heavy elements. This rule can be referred to when interpreting figure 2 showing the elemental distribution for several main elements and the distribution of scattered Rh-L and Rh-K radiation in the PCB sample.

Rh-L radiation has a low energy (2.6 keV) and cannot penetrate deeply into the sample. The scattering in this case is a surface effect and the image

shows the distribution on the surface (fig. 2b). The scattering intensity is determined by elements on the surface of the sample and also by topological effects. Rh-K radiation on the contrary has a high energy (22.2 keV) and penetrates deeply into the sample. The distribution of scattered intensity depends on the scattering material – high for light materials and low for heavy materials, as well as on the absorption of radiation inside the sample due to heavy elements (fig. 2c). In the discussed sample, the Cu traces on the backside of the PCB can be visualized through the distribution analysis of the Rh-K radiation. These Cu traces cannot be identified in the elemental distribution (fig. 2a) because the energy of the Cu radiation is too low to penetrate the complete PCB from the bottom. Therefore figure 2a only shows the elemental distribution on the surface of the sample, which includes Cu traces. In this case it is also possible to determine other elements located inside the sample – for example the grid of SiO₂-fibres used to stabilize the PCB material can be de-

tected in areas not covered by electronic components.

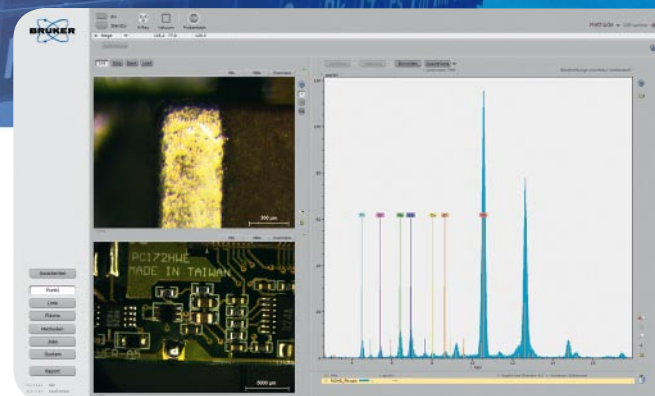
Conclusion

Micro X-ray fluorescence is a very powerful tool for elemental distribution analysis, both on the surface and within the sample. This requires not only an instrument with high performance regarding excitation intensity, step size and measurement speed, but also sophisticated data acquisition. The accumulation of data streams with an extremely high amount of data needs to be accomplished. Additionally, various options for data evaluation are required ensuring that all available data is utilized for a complete characterization of the analyzed material.

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A new definition of μ -XRF



M4 TORNADO



25 μ m
SpotSize

X-Ray optics for
smallest spot sizes



Turbo
Speed
Stage

Distribution analysis
"on the fly"



XFlash®
Technology

XFlash® detector
technology



M-Quant

Reliable standardless
quantification of bulk material



EasyLoad
Chamber

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with EasyLoad function

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