D4 ENDEAVOR

ANALYSIS OF SAMPLES CONTAINING FE WITH THE SOL-X DETECTOR

Introduction

Fluorescence radiation has an impairing effect on the quality of XRD measurement results if samples containing e.g. Fe or Mn are investigated using Cu-radiation. This is due to the significantly higher background and hence significantly lower peak-to-background ratio. Figure 1, for example, shows a measurement on a composite of Quartz and Hematite.

A diffracted beam monochromator is commonly used to suppress the fluorescence. The application of the monochromator drastically improves the peak-to-background ratio of the measurement (Fig. 2), but at the expense of a substantial loss of peak intensity. This eventually results in longer measurement times.

Switching to another X-ray wavelength is virtually the only alternative. Therefore, in cases where the loss of intensity can not be accepted from an analytical point of view, it is self-evident to have a diffracted beam set-up offering optimum peak intensities and a peak-to-background ratio.

This wish can now be fulfilled by the Sol-X energy dispersive detector a new innovative XRD solution from Bruker AXS. It meets the requirements for a large X-ray active area as well as for an energy resolution to separate CuKβ radiation or FeKα fluorescence from CuKα simultaneously (Fig. 3). The users main benefits are a reduction of the measurement time by a factor of about 3 and no extra effort when dealing with X-ray wavelength appropriate diffracted beam monochromators.

The Sol-X gives higher flexibility over diffracted beam monochromators, because it can be easily adjusted to the most commonly used wavelengths like CrKα, CoKα, MoKα and CuKβ. With an active detector window area of 4 x 15 mm^2 the entire beam of a long fine focus X-ray tube contributes to the XRD experiment, which results in higher count rates and, finally, smaller measurement times or higher quality of the data.

Instrumentation

- D4 ENDEAVOR
- 2.2 kW Cu long fine focus X-ray tube
- rotating sample stage
- 0.5° divergence slit system
- 0.5° anti-scatter slit system
- 4° Soller slits (primary and secondary)
- 0.1 mm receiving slit

Options:

- Ni-Filter, scintillation counter
- Graphite secondary monochromator, scintillation counter
- Sol-X, energy dispersive detector

Measurement parameters:

Scan Mode:	locked coupled
Tube power:	40 kV, 35 mA
Measurement range:	18° to 56° (2Theta)
Step size:	0.02 ° (2Theta)
Step time:	1 s

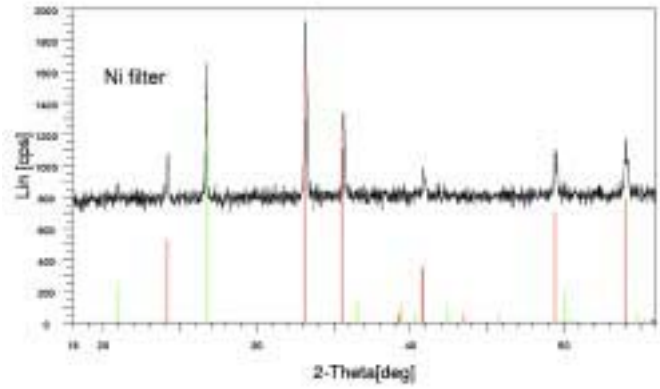

Figure 1: Measurement of a Hematite/Quartz mixture with Ni-Filter – high intensity, poor peak to background ratio. The green lines indicate the Quartz ICDD pattern, and the red lines indicate the Hematite pattern.

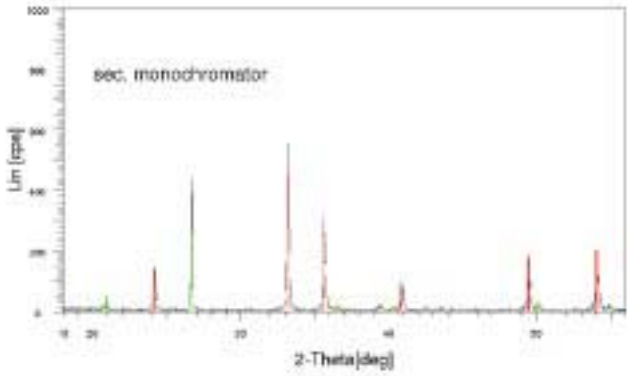

Figure 2: Measurement of a Hematite/Quartz mixture with secondary monochromator – low intensity, good peak to background ratio

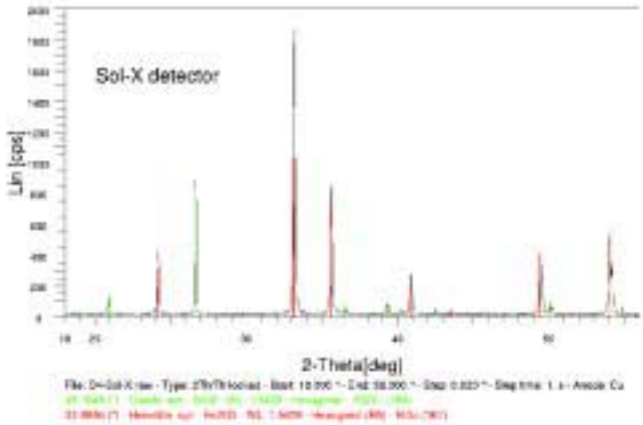

Figure 3: Measurement of a Hematite/Quartz mixture with Sol-X solid state detector – high intensity, superior peak to background ratio

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