Fast crystal domain mapping using energy-dispersive micro-XRF

EXRS 2016, Post 89

Falk Reinhardt, Roald Tagle, Max Bügler, and Ulrich Waldschläger
Bruker Nano GmbH, Am Studio 2D,
12489 Berlin, Germany
Falk.Reinhardt@bruker.com

Introduction

In energy-dispersive XRF, Bragg diffraction peaks are often considered a bothersome artifact interfering with the fluorescence information. However, these Bragg peaks, as they are related to crystal orientation, provide additional information on the nature of the sample. Here, we describe how the M4 Tornado can be utilized to visualize crystal domains. This information is crucial to evaluate the quality of single crystals as well as the properties of polycrystalline materials. This principle can be used to identify sub-grain misorientation in single crystals as well as crystallite dimensions within multi-crystalline samples.

The method is non-preparative and non-destructive and can scan areas of up to 190 mm x 150 mm with 50 µm spatial resolution in less than 5 hours.

Instrument

The M4 Tornado is a spatially resolving X-ray fluorescence spectrometer which was developed for high speed 2D elemental analysis and visualization. The sample is excited with polychromatic X-rays (Rh-anode) and the radiation is detected with one or two SDDs.

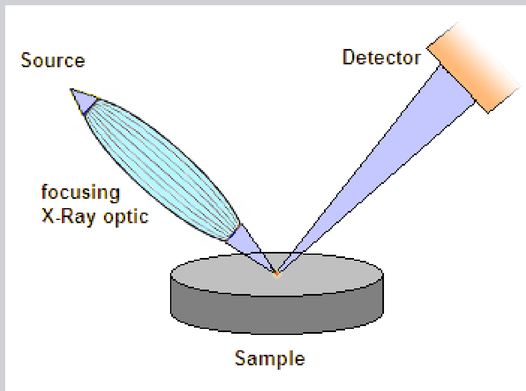

Fig. 1 Sketch of the setup

The polycapillary optic yields a divergence of the incident beam of ~50 mrad , the acceptance angle of the detector is ~ 14°. Thus, any crystalline sample has a high probability to produce detectable diffraction peaks.

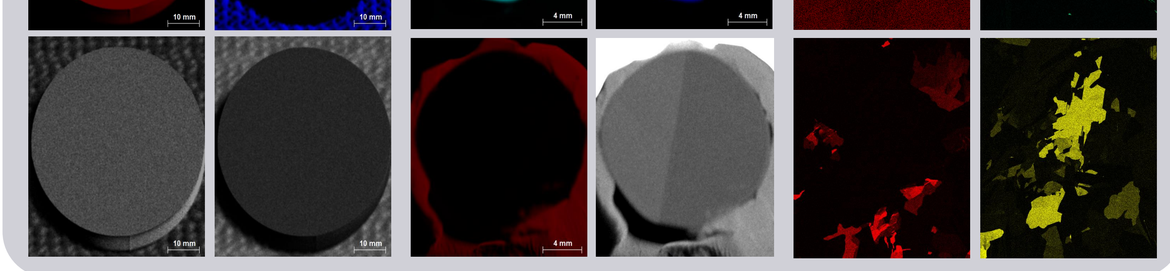

Fig. 3 Top row: Bragg diffraction peak distribution along the scanned sample surface. Middle row, sum spectra and selected energy regions (ROIs). Bottom part: element and Bragg peak intensity distributions.

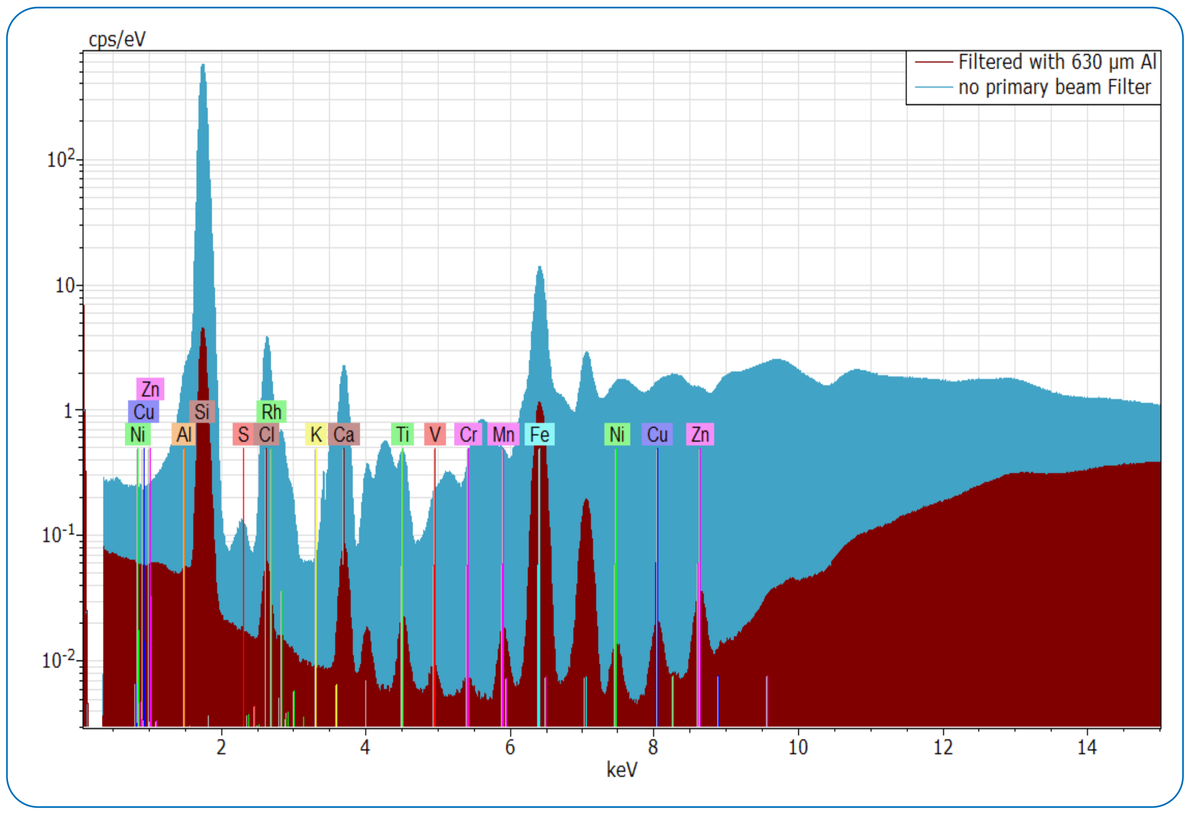

Fig. 2 Sum spectrum of a multi crystalline Si with impurities measured with (red) and without (cyan) primary beam filter. Trace and minor elements can only be quantified when diffraction is suppressed. The detected intensity is dramatically reduced.

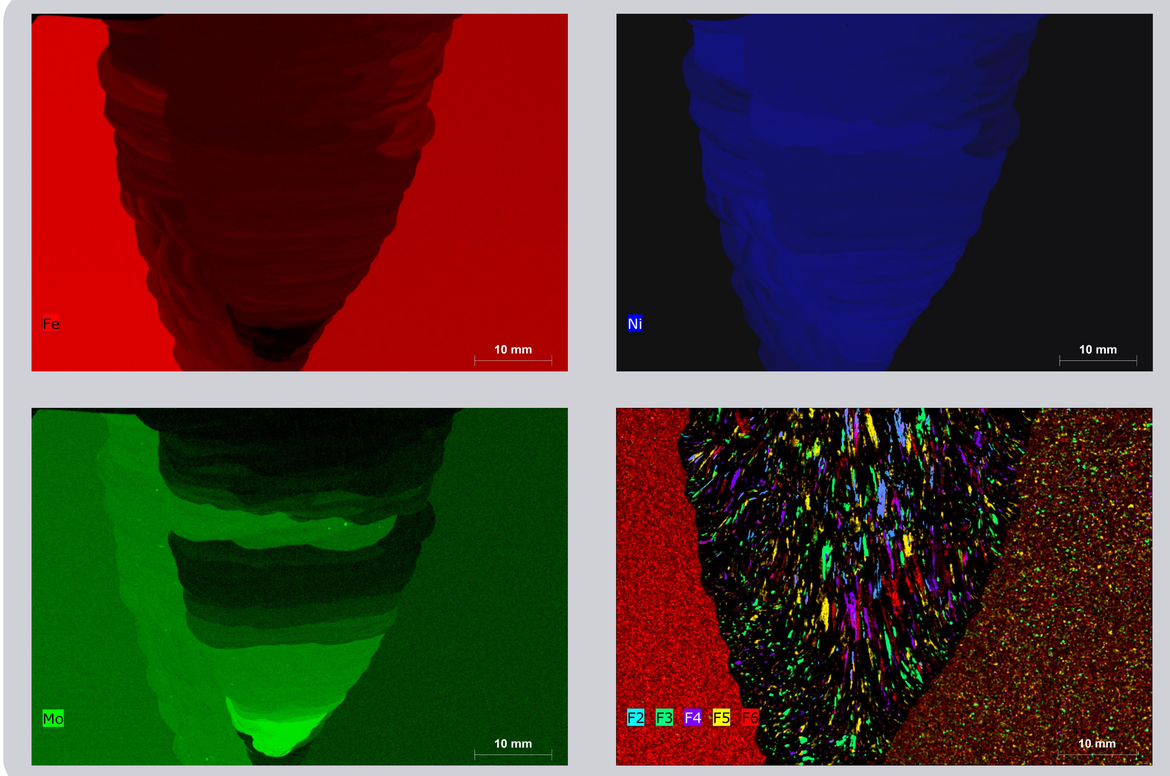

Fig. 4 Element map and diffraction pattern distribution of a large welding joint (70 mm x 50 mm). Two different steels were welded using a Ni rich welding consumable. The crystallization hints to the temperature gradient during and after the processing.

Diffraction in energy dispersive XRF

- Most industrial or geological samples are crystalline
- Diffraction peaks overlap with fluorescence lines and thus impede the analysis of minor and trace elements
- Usually by using filters the excitation spectrum is deprived of the energy ranges which are diffracted, this results in a “clean” spectrum
- This, in turn, reduces the excitation intensity and results in longer measurement times needed for a reliable analysis, especially for light elements

Summary

The M4 Tornado allows the fast measurement of the spatial distribution of Bragg peaks and the identification of crystal domains. In the field of crystal growth it can be indispensable to continuously check the structural quality of the grown bulk crystals, a task which is usually time consuming or challenging for large single crystals.

Conclusions

- EDXRF instruments are likely to detect diffraction peaks
- In contrast to XRD measurements the diffraction information is purely qualitative
- large sample areas can be analyzed with high spatial resolution and high speed

Technology