HIGH-PERFORMANCE XRPD WITH A NEW CHROMIUM MICRO FOCUS SOURCE

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The instrumentation for x-ray powder diffraction was dramatically improved by the introduction of linear and 2-dimensional detectors to record the diffracted patterns and the introduction of multilayer mirrors as monochromatizing beam concentrators on the primary beam side. The major effort was to improve the efficiency of XRPD and simultaneously the data quality.

Until recently a missing mosaic stone for this goal was a high-brilliance microfocus source in combination with a 2D focussing multilayer optics. For Cu–Kα radiation an ideally tuned source was introduced by Incoatec (IμS, Montel-type “Quazar”optics) at the ECM 2006. A new modification of this system was now developed for Cr radiation. With a tube power of only 13.5 Watt the photon flux density of monochromatic Cr-Kα radiation into the focal spot (330 μm) at the sample is about as high as from a 1.5 kW Cr standard x-ray tube. Of course, the micro-focus source is air cooled.

The detectors used were linear PSPC (Braun PSM50) and a new 2D-detector (Bruker VÅNTEC 2000). Especially the combination of the area detector with this extraordinary source inspires new ideas for the instrumentation of XRPD with respect to mobile or even hand-held systems.

The importance of Cr-radiation for applications in XRPD results from the following properties:
1) The photon energy below the absorption edge of elements from the iron- and RE-groups avoids the excitation of fluorescence background and increases the diffraction signal (applications: identification of retained austenite, hardening phases, surface coatings)
2) Stress analysis in steel lives from the high intensity and angular position at 156°, of the ferrite (210)-peak with Cr-Kα radiation. Here, a high local resolution is required to identify critical spots in welding seams or pre-strained motor parts like tooth wheels
3) The long wavelength makes Cr-radiation especially useful for large unit cells.

Our test measurements were selected samples from the real world of present analytical tasks. The well known applications are related with important mechanical engineering subjects for the automotive --, construction- and machine tool industries, but also technologies for catalysis and luminescent materials.