Investigation of illuminated manuscripts by microdiffraction using an aircooled X-ray microfocus source

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In recent years the interest in the non-destructive investigation of cultural heritage objects has risen strongly. X-ray methods are often used for the analysis of paintings and books. Due to the need of high brilliance for the used X-ray synchrotron sources, that are large and immovable, it is desirable to have microfocus X-ray sources. Such X-ray sources can also be used for X-ray fluorescence analysis, which is an important tool in archaeology.

For some time Incoatec has been designing its own X-ray tubes [1]. Other X-ray methods are used as well. A painting by Rembrandt van Rijn was analysed at the Royal Museum of Fine Arts, Antwerp, using the Incoatec Microfocus Source IµS. The IµS is an air-cooled high-brilliance X-ray source for diffractometry applications. The source is designed around a newly developed microfocus X-ray tube with specially designed Montel optics that shape the X-ray beam in two dimensions and collect as much X-ray photons as possible. The optics form either a highly collimated beam (size of 0.5 to 2 mm) with a low divergence (below 0.5 mrad) or a focusing beam with higher divergence (up to 10 mrad) and very small focal spots at the sample (diameters down to 0.1 mm).

The Cu-Kβ with collimating optics can be used for small angle scattering methods, also in grazing incidence for surface investigations, and standard X-ray diffraction studies. With focusing optics experiments can be carried out in transmission geometry, especially in powder diffraction applications and for cryocrytography.

The Mo- and Ag-Kβ highly absorbing and radiation damage sensitive can be investigated. Consequently, these sources are often used for chemical cryotrification and become more and more interesting for investigation of soft matter samples or for XRD measurements during the growth of nanosized materials.

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Montel Optics: Multilayer Optics for Perfect Beam Conditioning

Multilayers with thin films in the nanometer region are best suited for beam formation and monochromatization of X-rays. Applying Bragg’s low X-rays are collected in a solid angle of approximately 1 mrad e.g. at W/C multilayers and are redirected with a reflectivity larger than 80 % while simultaneously suppressing Kα-radiation. To take account for the varying incidence angles, the multilayer requires a gradual gradient of the layer thickness. It is possible to focus the incident beam with an elliptically shaped substrate. A collimation is achieved with a parabolically shaped substrate. A 2-dim shaping of the beams is possible by combining and fixing two multilayer mirrors side-by-side in an L-shape. This assembly is called Montel optics. In this geometry the beams are doubly reflected and thus the monochromatization effect is squared. The graphic illustrates that the multilayer optics are an ideal combination for X-ray sources with a focal spot diameter well below 100 µm on the anode.

Experimental Set-up

A medieval book painting was investigated using XRF and XRD-techniques simultaneously. While with XRF the elemental composition of the used pigments are analysed, with XRD crystallographic information could be revealed. Using both methods, the chemical composition of the pigments could be analysed. Here a Mo-Kβ source with focusing optics was used. The sample was tilted by 45° relative to the beam direction. The XRD patterns were recorded with a SMART 1000 CCD-detector (Bruker AXS) in transmission geometry, XRF signals were measured with an energy dispersive detector arranged in 90° to the beam direction.

The focal position of the IµS was between sample and detector. A sample area of about 130 µm x 130 µm was illuminated with the X-ray beam. Using this setup, XRD frames were recorded within 30 seconds exposure time, the XRF measurement was done simultaneously.

The XµS setup for the measurements in transmission geometry (top) and backscattering (below).

Results

Integrated XRD pattern for green and red regions are shown with peak positions of the used pigments. In an overview-scan (approx. 18 hours) an area of several square millimeter and a resolution of 150 µm could be investigated. This study shows, that it is possible to obtain high quality results even with a quite simple setup. The IµS, the sample and the XRF-detector were just placed on an optical bench while the XRF-detector was just placed on a shelf in a suitable position.

Diffraction pattern of green (top) and red (below) colored regions.

Summary of IµS Technology

- Suitable for Incoatec’s X-ray Tube IXT - the first and only microfocus sealed tube optimized for cryotrification, designed by Incoatec
- High performance Incoatec Montel multilayer optics for 2D focusing or collimating
- Available for different energies - Cu, Mo, Ag, Cr and Co radiation
- Superb performance comparable to first generation microfocus rotating anodes
- Long lifetime without maintenance, and low cost of ownership
- Improves user-friendliness
- High safety standards and fully compliant with Machinery Directive 2006/42/EC
- Easily adaptable to all common goniometers
- Makes experiments possible which a few years ago could only be performed at synchrotrons

During the last 10 years since its launch, the IµS is regarded as the superior source for X-ray analysis in the field. The IµS sources sold worldwide are proof of outstanding performance and reliability with best value for money. Get the experience of the IµS!

References


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