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

Jörg Wiesmann[1], Bernd Hasse[1], Frederik Vanmeert[2], Koen Jannsens[2]

[1] Incoatec GmbH, Max-Planck-Str. 2, 21502 Geesthacht, Germany; [2] Department of Chemistry, University of Antwerp, Prinsstraat 13, 2000 Antwerp, Belgium

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 sources most experiments were done at synchrotron sources, that are large and immovable. In this study a medieval book painting was investigated using XRF and XRD-techniques simultaneously by using the microfocus X-ray source IμS. With this low power air-cooled solution the X-rays can be used directly at the location of the object of interest.

Incoatec Microfocus Source IμS - The Source for Analytical X-ray Methods

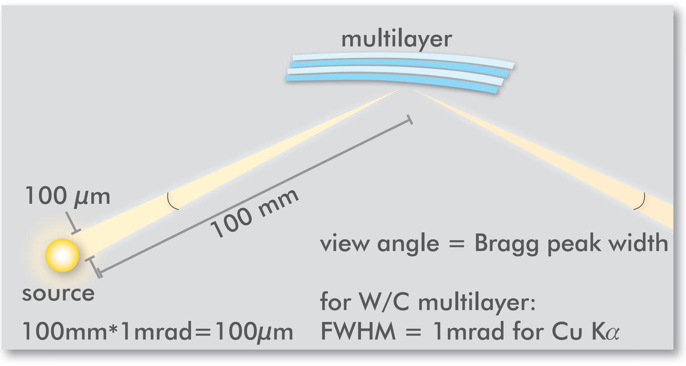
The IμS is an air-cooled high-brilliance X-ray source for diffractometry applications. The source is equipped with Incoatec´s new microfocus tubes IXT and with specially designed Montel optics that shape the X-ray beam in two dimensions and collects as much X-ray photons as possible. The optics form 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 downto 0.1 mm).

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

The first and second generation of the Incoatec Microfocus Source - the IμS (left) and IμS$^{High\ brilliance}$ (right)

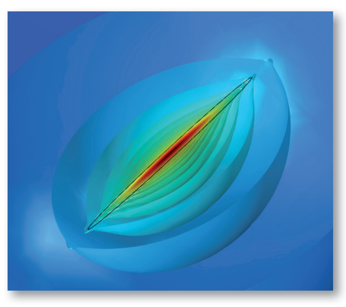
Montel Optics: Multilayer Optics for Perfect Beam Conditioning

Multilayers with thin films in the nanometer region are best suited for beam formation and monochromatization of X-rays. Applying Bragg's law 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 Kβ-radiation. To take account for the varying incident angles, the multilayer requires a lateral 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 beam 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.

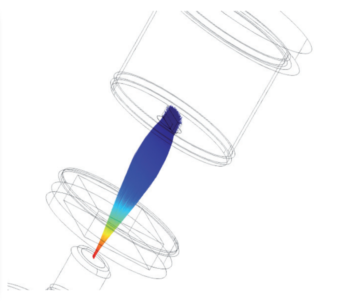


Incoatecs X-ray Tube IXT: High Brilliance with Low Maintenance

For some time Incoatec has been designing its own tubes. By changing cathode and anode parameters as well as geometries and take-off angles, an optimization of tubes for X-ray analytical applications could be achieved. The figures show simulations of critical tube parts: the anode, the cathode and the electron beam. IXT tubes for IμS provide a highly brilliant X-ray beam in a power range of 10-70 W. Its focal spot of the electron beam on the anode only has a diameter of 20-50 μm. Typical anode materials like Cu, Mo, Ag and Cr for XRD or Rh, W and Mo for XRF are available.



COMSOL simulation of the thermal load on the anode

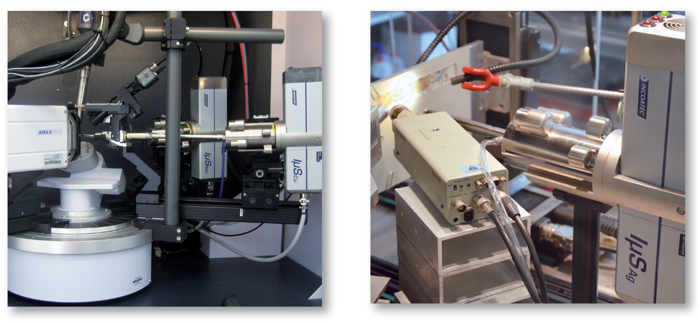


Ray-tracing of the electron beam, which is focused to the anode

As the brilliance cannot be increased by optics, therefore it is of utmost importance to achieve the highest possible brilliance within the tube. Due to the higher surface-to-volume ratio of the focal spot compared to typical water-cooled sealed tubes, IXT has an improved heat conductivity and thus allows significantly increased power densities. With values larger than 5 kW/mm² the performance is comparable to 5 kW-class rotating anodes.

Upgrading Existing Diffractometers with the IμS

With Incoatec´s established highly qualified customer and service support team we offer a unique opportunity of upgrading existing diffractometers with our high-performance IμS. Our long-standing experience is based on more than 80 projects worldwide in which we upgraded a wide variety of existing diffractometers or integrated the IμS in customized solutions.



Upgrade of an Bruker APEX II DUO (left) and a combined XRD/XRF setup (right) with IμS

Summary of IμS Technology

- Includes Incoatec´s X-ray Tube IXT - the first and only microfocus sealed tube optimized for crystallography, designed by Incoatec
- With state-of-the-art 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
- Improved user-friendliness
- Highest 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 analytics in the home-lab. More than 800 sources sold worldwide are proof of outstanding performance and reliability with best value for money. Get the experience of the IμS!

IμS for Scanning Microdiffraction - Investigation of Paintings

In recent years the interest in the non-destructive investigation of cultural heritage objects has risen strongly. Besides infrared and optical imaging and spectroscopic methods, X-ray methods like X-ray fluorescense, X-ray diffraction, X-ray radiography and X-ray imaging methods (e. g. tomography) are often used for the analysis of art objects [1].

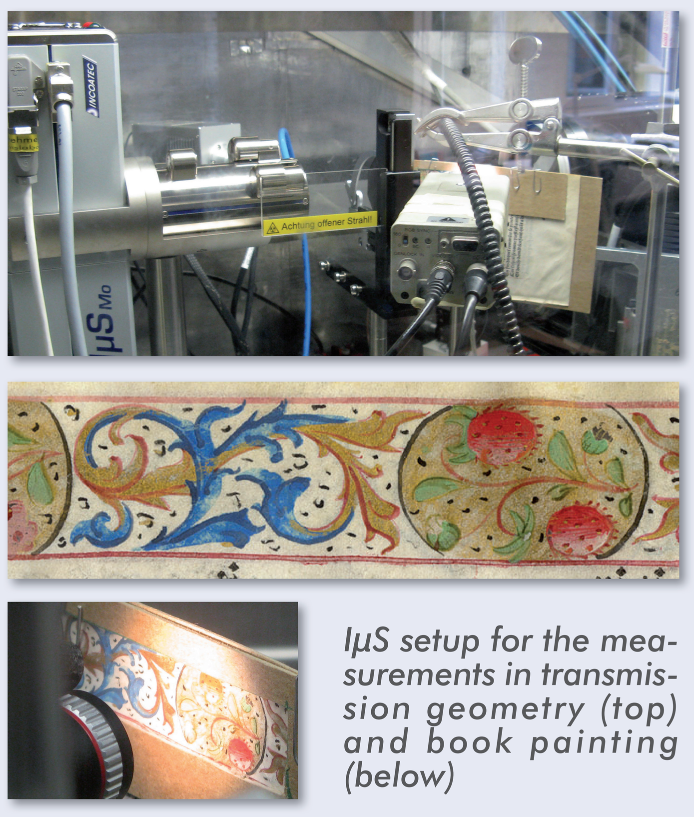
Using for example X-ray fluorescence (XRF) the scrolls of Qumran were investigated [2], fragments could be identified as belonging together or not [3]. Using XRF analysis of paintings generally provides information about the possible presence of elements on the surface of art-objects due to pollution (e.g. sulphur or chlorine), about the elements and pigments used by the artist, about previous restored areas detected by the presence of "modern" elements like titanium or zinc, and identification of fraudulent submission. In combination with X-ray diffraction (XRD) also the crystallographic composition of the used pigments could be characterised. A study about the alteration involved the oxidation of cadmium yellow (CdS) to $CdSO_4 \cdot 2H_2O$ under the influence of light, oxygen and moisture is an example of the use of this technique in the investigation of paintings [1]. Other X-ray methods are used as well. A painting by Rembrandt van Rijn was analysed at the synchrotron facility ESRF using an X-ray absorption imaging technique and with XRF reveailing an overpainting [4].

Frederik Vanmeert, University of Antwerp has examined De Heem´s painting 'Flowers and Insects', Royal Museum of Fine Arts Antwerp,with the Incoatec Microfocus Source IμS$^{High\ brilliance}$.

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-IμS 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.

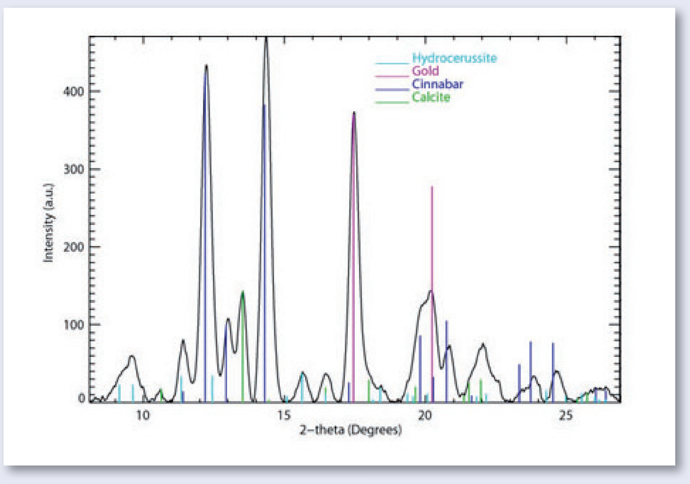
The focal position of the IμS was between sample and detector. A sample area of about 130 μm x 130 μm was illuminated with the X-ray beam. Using this setup, XRD frames were recorded within 30 seconds exposure time, the XRF measurement was done simultaneously.

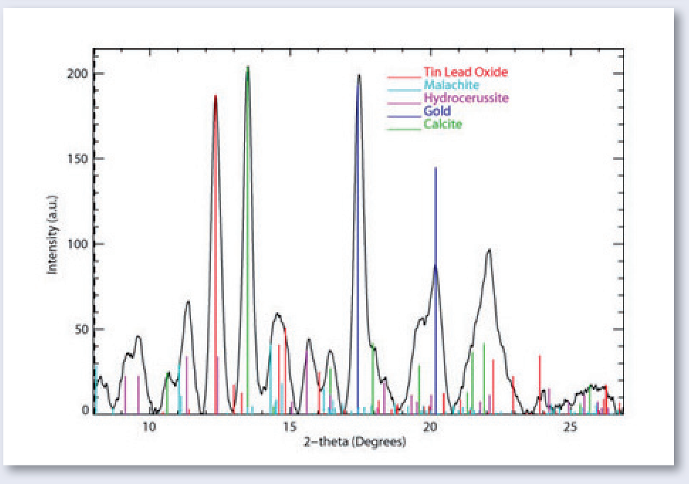


IμS setup for the measurements in transmission geometry (top) and book painting (below)

Results

Integrated XRD pattern for green and red regions are shown with peak positions of the used pigments. In an overnight-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 XRD-detector were just aligned 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

References

[1] K. Janssens et al., Photon-Based Techniques for Nondestructive Subsurface Analysis of Painted Cultural Heritage Artifacts. *Accounts of Chemical Research,* 2010, 43 (6), 814–825

[2] O. Hahn et al., Non-destructive investigation of the scroll material: „4Qcomposition Concerning Divine Providence" (4Q413). *Dead Sea Discoveries*, 2007, 14(3), 359-364

[3] I. Rabin et al., On the origin of the ink of the Thanksgiving scroll (1QHodayot). *Dead Sea Discoveries,* 2009, 16(1), 97-106

[4] http://www.esrf.eu/news/general/hidden-rembrandt/index_html; retrieved 20.08.2015

www.incoatec.de

INCOATEC
innovative coating technologies