Current Status of Home-Lab Microfocus X-ray Sources for Crystallography

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10 years IµS — Celebrate the power

Introduction

Modern low power microfocus X-ray sources, such as the Incoatec Microfocus Soure IµS or the METALJET X-ray source, define the state-of-the-art for most in-house applications in X-ray diffraction. These sources deliver a brightness beyond that of comparable traditional X-ray sources at power settings below 1 kW. They are usually combined with multilayer X-ray mirrors which are excellent X-ray optical devices for beam shaping and preserving the brightness of the source. Here, we will be reviewing the latest developments for mircorfocus X-ray sources

Incoatec Microfocus Source IµS

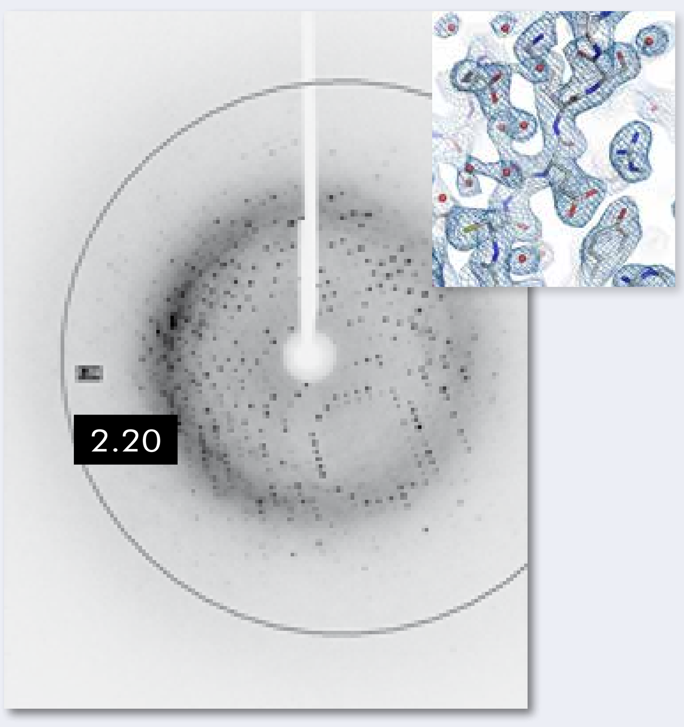
Since its introduction in 2006, the Incoatec Microfocus Source IµS has become the gold standard for low power low maintanance home-lab X-ray sources. The IµS combines a low power microfocus X-ray sealed tube with dedicated Montel multilayer mirrors and delivers intensities beyond those of traditional rotating anode sources. With more than 800 sources sold world-wide, the IµS is the market-leading microfocus source for X-ray diffraction applications, such as single crystal diffraction on small molecule and protein crystals as well as small angle scattering.

- Low power microfocus sealed tube
- Air-cooled
- Operated typically below 100 W
- Power load ~ 5 kW/mm²
- Montel multilayer mirrors for focused or collimated beam applications
- Available for Cu-Kα, Mo-Kα, Ag-Kα
- Single port source

Protein Crystallography with the Cu-IµS 3.0

215° data collection on a thin crystal of human NEIL1 (Endonuclease VIII-like protein)

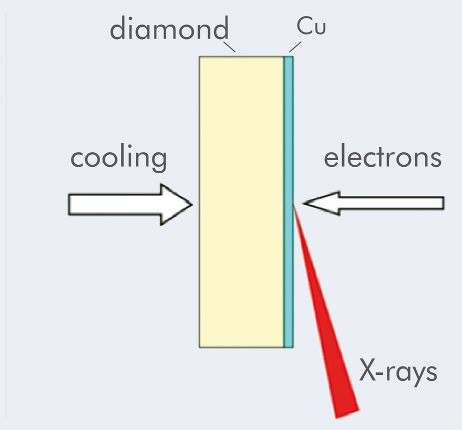
Crystal Size [mm³]	0.15 x 0.12 x 0.02
Exposure time [s/0.5°]	100
Total time [h]	12
Resolution [Å]	2.25 (2.32 - 2.25)
Completeness [%]	99.6 (99.7)
Multiplicity	5.1 (3.5)
$<I/\sigma(I)>$	13.2 (2.6)
R_{merge}	0.0707 (0.3234)
$CC_{1/2}$ at High res [%]	89
R; R_{free}	0.196; 0.250



Data statistics (above, left), typical diffraction pattern (above, right) and section of the electron density map (right) of the human NEIL1 protein after structure solution by molecular replacement and refinement with REFMAC.

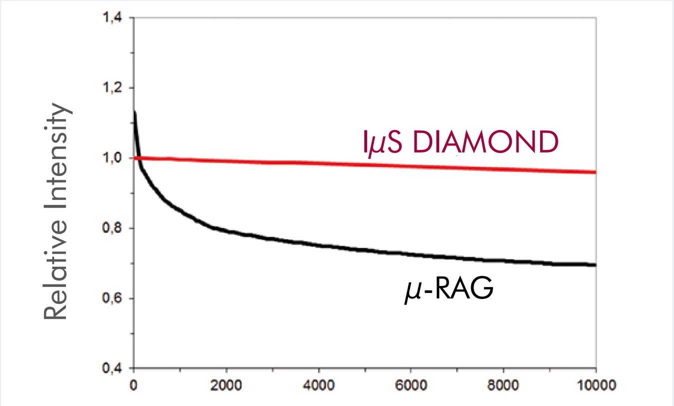
The IµS DIAMOND - The new Micorofocus X-ray Tube with a Diamond Hybrid Anode

The latest development of Incoatec's X-ray tube factory is a new microfocus sealed tube with a unique anode technology, the diamond hybrid anode. It comprises a bulk industrial diamond that is coated with a layer of the target material (e.g. Cu). The diamond hybrid anode takes advantage of the higher thermal conductivity of diamond, compared to bulk Cu anodes. Thus, the heat dissipation in a diamond hybrid anode is more efficient. Consequently, the IµS DIAMOND can accept a higher power density in the focal spot on the anode without damaging the surface of the target layer, resulting in a superb intensity.



Principle of the diamond hybrid anode used in the air-cooled IµS DIAMOND.

The heat management in the IµS DIAMOND assures that the intensity loss over time is only a few percent over 10,000 h of full power operation, which is significantly lower than in microfocus rotating anode sources. Typically, microfocus rotating anodes lose about 20-30% of their initial intensity within the first 1000 h of operation time. This is due to an increasing surface roughening caused by thermal stress in the focal spot on the rotating target. As a consequence, the intensity of the IµS DIAMOND is about 20% higher than the average output of a modern microfocus rotating anode.

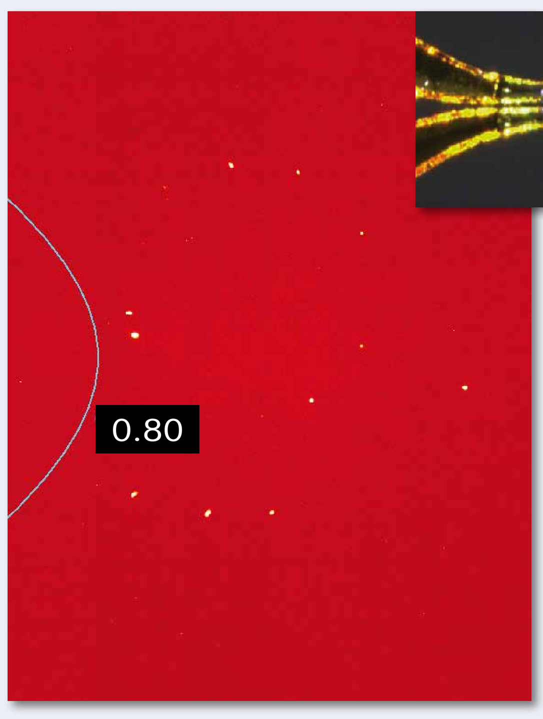


Comparison of the decay in the relative intensity over time for a low power microfocus rotating anode and for an IµS DIAMOND.

The IµS DIAMOND establishes a new class of X-ray sources, combining an intensity output that exceeds the intensity of a microfocus rotating anode with all the comfort of a standard IµS with a bulk Cu anode.

Source	µ-RAG	IµS DIAMOND
Exposure time [s/°]	20	20
$<I>_{norm}$	50425	64479
Resolution [Å]	0.80 (0.90 - 0.80)	0.80 (0.90 - 0.80)
Multiplicity	6.6 (5.0)	6.6 (5.0)
$<I/\sigma>$	49.7 (38.8)	55.2 (41.3)
$R1$ (all), $wR2$ (all) [%]	2.83, 7.46	2.71, 7.43
d(C-C) [Å]	1.518(2)	1.519(2)

Data statistics for a small crystal (0.10 x 0.04 x 0.04 mm³) of an organic compound collected with a µ-RAG (after ~1500 h of full power operation) and with an IµS DIAMOND.



METALJET X-ray Source

The brilliance of conventional X-ray sources using a solid metal target is limited by the maximum power load that can be applied without melting the anode material. The METALJET X-ray source technology has overcome this limitation, as it uses molten alloys containing Gallium and Indium as a high-speed liquid metal-jet target instead of fast spinning solid metal targets. Therefore, power loads of more than 100 kW/mm² in a spot size of 20 µm or smaller can be applied, which are an order of magnitude larger than those of modern microfocus rotating anodes.

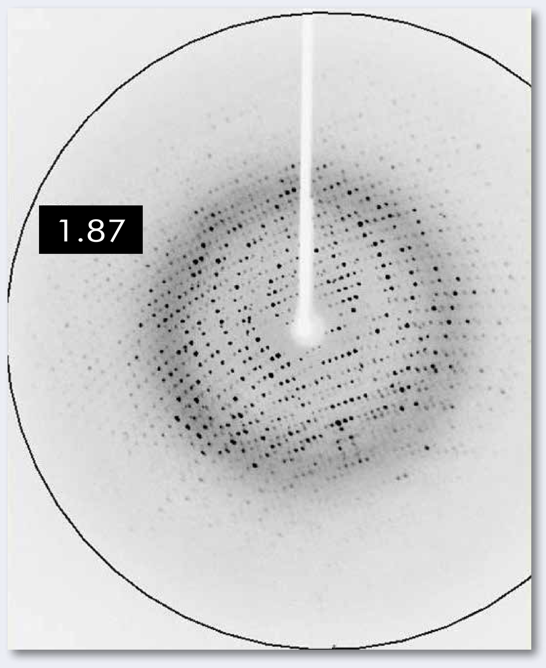
- High-speed liquid metal-jet target
- Air-cooled
- Operated typically at 200 W
- Power load > 100 kW/mm²
- Montel multilayer mirrors for focused or collimated beam applications
- Available for Ga-Kα (1.34 Å), In-Kα (0.51 Å)
- Dual port source

For preserving the extreme brightness of the source, high quality multilayer mirrors are needed, since a smaller source requires greater precision in the manufacturing of the multilayers. The HELIOS MX mirrors for Ga-Kα (9.3 keV) and In-Kα (24.2 keV) are synchrotron-class optics tailor-made for the METALJET source for single crystal diffraction applications. They deliver a small and intense X-ray beam that is much brighter than what is currently achieved with microfocus rotating anode sources. As the energy of the Ga-Kα line is close to the Cu-Kα line, the METALJET using a Gallium rich alloy as target is the source of choice for ultimate performance in the home lab for applications such as protein and pharmaceutical crystallography, as well as small angle scattering.

Fast Data Collection for High Throughput Screening

100° Data Set from a Protein Complex within 4 min

Crystal Size [mm³]	0.10 x 0.80 x 0.05
Exposure time [s/°]	2
Resolution [Å]	1.95 (2.05 - 1.95)
Completeness (%)	97.5 (92.2)
Multiplicity	3.68 (2.34)
$<I/\sigma(I)>$	10.75 (2.77)
R_{merge} (%)	6.58 (23.91)
R_{sigma} (%)	5.99 (25.36)
R_{pim} (%)	3.58 (17.95)
CC(½) at 2.00 Å	86 %

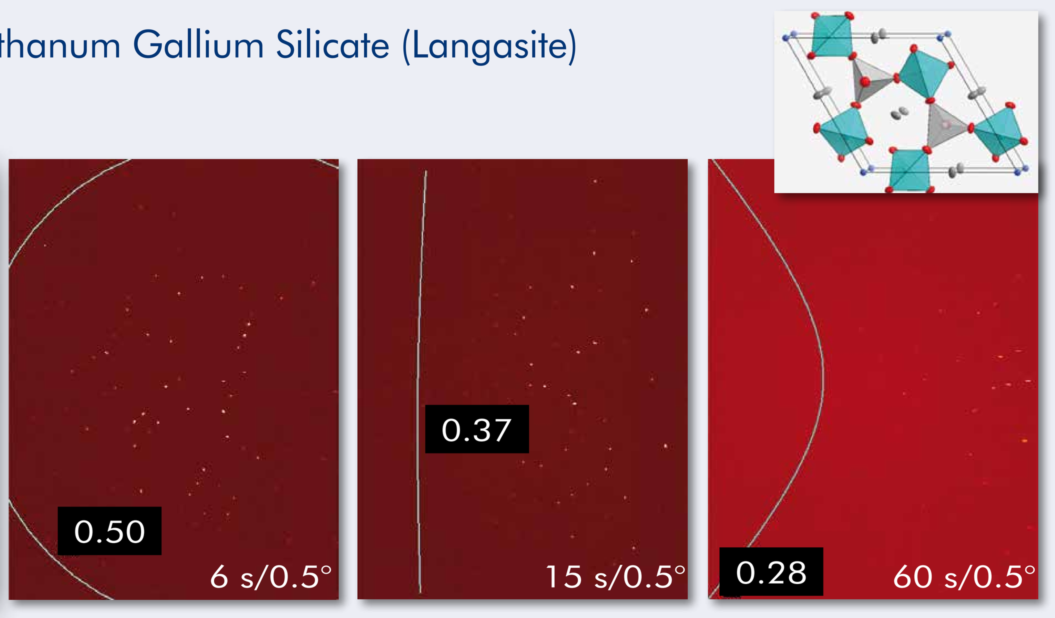


Data statistics and typical diffraction pattern of a protein complex, recorded with a D8 VENTURE 2nd Gen. and the METALJET X-ray source using a Ga rich alloy. The good data quality allowed for a structure solution by molecular replacement.

By using an Indium rich target, the yield from the In-Kα line, which is close to the Ag-Kα line, can be maximized, giving highest performance for structure determination on absorbing materials and for high-pressure experiments using DAC's. The very short wavelength minimizes the absorption and leads to a compression of the q-space. Compared to Ag-Kα, about 50% more unique data are accessible.

Data from a Highly Absorbing Lanthanum Gallium Silicate (Langasite) Collected with the In METALJET

Source	In METALJET
Total time [d]	2.7
Exposure time [s/0.5°]	6 / 15 / 60
Resolution [Å]	0.28 (0.38 - 0.28)
Completeness (%)	99.9 (99.9)
Multiplicity	37.1 (26.7)
$<I/\sigma>$	38.2 (21.6)
R_{int} (%)	7.93 (16.77)
$R1$, $wR2$ (%)	2.78, 7.44



Data statistics and typical diffraction patterns of a small Langasite crystal at low, medium and high resolution, collected with a D8 VENTURE 2nd Gen. and the METALJET X-ray source using an In rich alloy.

Crystal of a Small Molecule Compound in a DAC

Due to the very short wavelength of In-Kα, the reciprocal space is compressed and, therefore, the number of reflections that are accessible in the high pressure experiment is increased by a factor of about 2.5 when compared to Mo-Kα. The increased resolution and number of unique data facilitate structure solution and refinement of high-pressure phases.

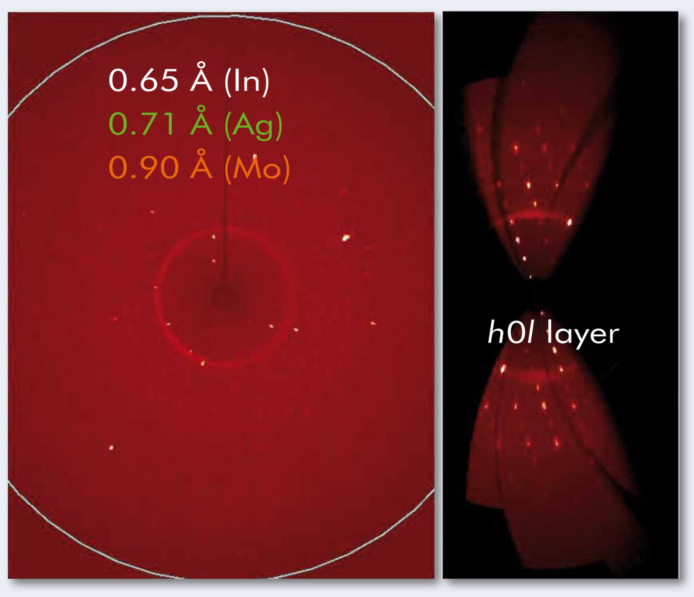


Illustration of the compression of the q-space for different energies

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