Shine Bright Like a Diamond: Microfocus X-ray Sources with Diamond Hybrid Anode Technology

J. Graf[1], T. Stürzer[2], H. Ott[2], M. Benning[3], P. Radcliffe[1], J. Schmidt-May[1], R. Durst[2], J. Wiesmann[1], C. Michaelsen[1]

[1] Incoatec GmbH, Geesthacht, Germany; [2] Bruker AXS GmbH, Karlsruhe, Germany, [3] Bruker AXS Inc., Madison (WI), USA

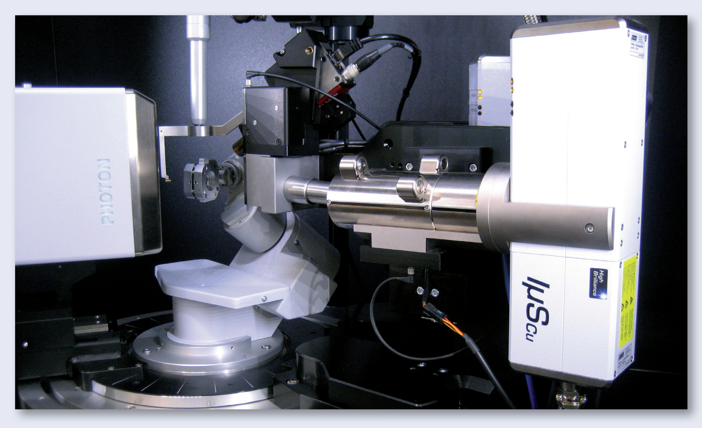
Introduction

Modern low power microfocus X-ray sealed tube sources, such as the Incoatec Microfocus Soure IµS, define the state-of-the-art for most in-house applications in X-ray diffraction. These sources are usually combined with multilayer X-ray mirrors as beam shaping devices and deliver a brightness beyond that of comparable traditional X-ray sources, at power settings below 1 kW [1,2]. During the IUCr congress in 2017, we introduced a new class of microfocus sealed tube sources with a unique anode technology, the diamond hybrid anode, which uses industrial diamond as heat sink. This IµS DIAMOND combines the performance of a modern 1 kW microfocus rotating anode with all the comfort of a conventional microfocus sealed tube source. Here, we will be reviewing the latest developments for microfocus sealed tube 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 850 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.

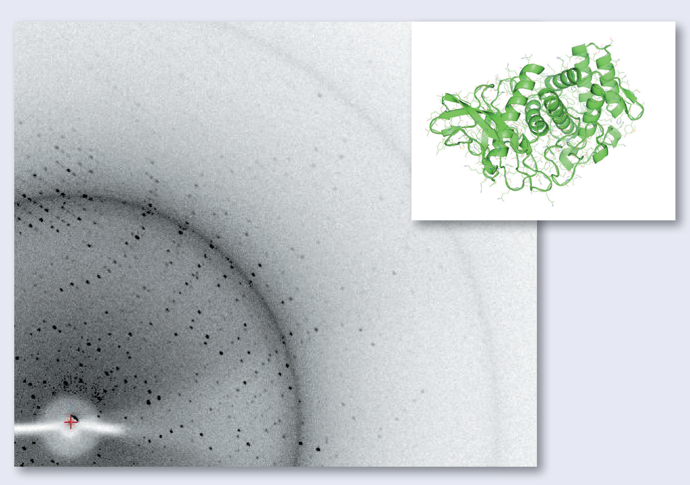
- Low power microfocus sealed tube
- Air-cooled
- Operated typically below 100 W
- Power load ~ 5 kW/mm²
- Montel multilayer mirrors for focused or collimated beam applications
- Available for Cu-Kα, Mo-Kα, Ag-Kα (others on request)



Protein Screening with the Cu-IµS

Data comparison with a traditional 4 kW rotating anode generator on a small crystal of Thermolysin

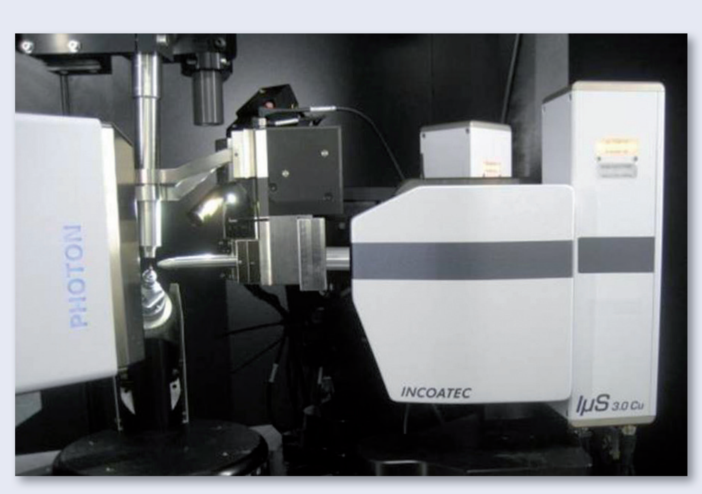
Source	30 W Cu-IµS	4 kW RAG
Exposure time [min/0.5°]	10	10
Resolution [Å]	2.10 (2.14 - 2.10)	2.10 (2.14 - 2.10)
Completeness [%]	98.5 (97.1)	99.9 (100)
Multiplicity	4.4 (4.6)	6.7 (6.6)
<I/σ>	20.1 (4.8)	20.2 (4.4)
R (syml) [%]	7.1 (32.9)	9.7 (45.1)



Data comparison for a small crystal (0.20 x 0.06 x 0.04 mm³) of Thermolysin collected with a 30 W Cu-IµS (left) and a traditional 4 kW RAG combined with 1st generation multilayer mirrors (right). Both data sets were recorded with an image plate detector.

The IµS 3.0 – The first Microfocus Sealed Tube Designed for Crystallography

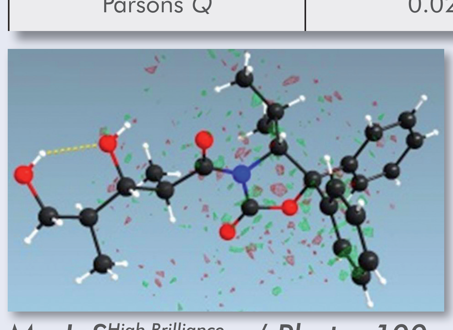
Over the past years, we have gradually improved the performance of our IµS by optimizing critical parameters in the X-ray tube, such as take-off angle and electron beam focusing. Further, the X-ray optical design of our multilayer mirrors was improved to match the properties of the source to the requirements of the various applications of the IµS.
One important milestone was the launch of the IµS 3.0 at the AsCA conference in Kolkata in 2015. The IµS 3.0 is the first microfocus sealed tube source that is fully optimized for X-ray diffraction applications. It combines an X-ray source with a superb intensity in the range of 4x10¹⁰ phts/s/mm² with a new beam path design and a high-precision mounting concept (quick-lock concept) allowing for a true downstream alignment and swappable optics. This makes the IµS 3.0 the most user-friendly microfocus X-ray source ever.



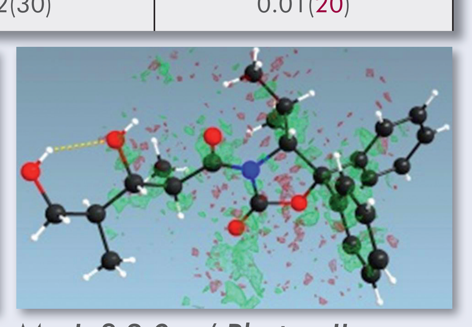
Absolute Structure Determination with the Mo-IµS 3.0

Data comparison on a medium well diffracting crystal of a typical pharmaceutical small molecule compound (0.12 x 0.10 x 0.09 mm³)

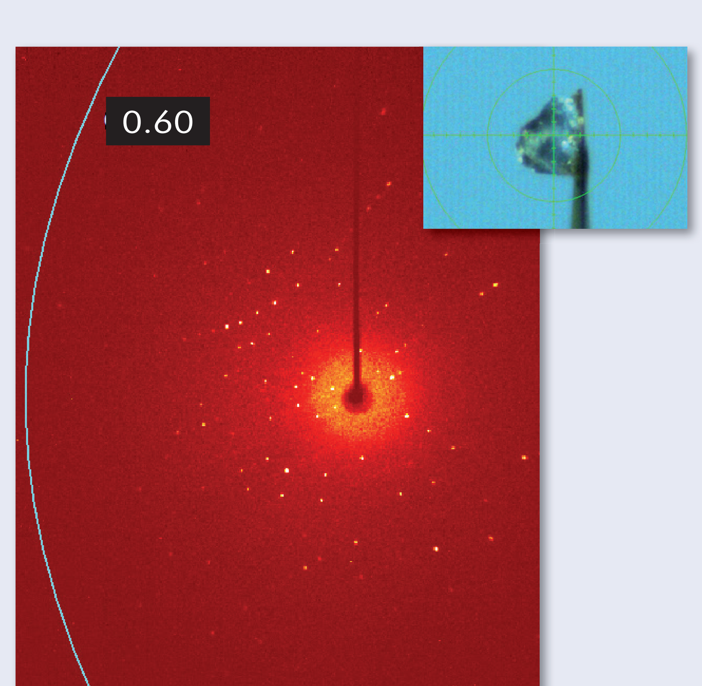
Source	Mo-IµS$^{High\ Brilliance}$	Mo-IµS 3.0
Detector	Photon 100	Photon II
Exposure time [s/0.3°]	35; 15 h	35; 15 h
Max. Resolution [Å]	0.64	< 0.56
Resolution [Å]	0.80 (0.90 - 0.80)	0.80 (0.90 - 0.80)
Multiplicity	12.7 (9.1)	11.7 (9.3)
<I/σ>	41.6 (9.7)	55.2 (18.8)
*R*1 (all), *wR*2 (all) [%]	7.17, 14.77	8.11, 18.43
d(C-C) [Å]	1.387(4)	1.390(3)
Parsons *Q*	0.02(30)	0.01(20)



Mo-IµS$^{High\ Brilliance}$ w/ Photon100



Mo-IµS 3.0 w/ Photon II

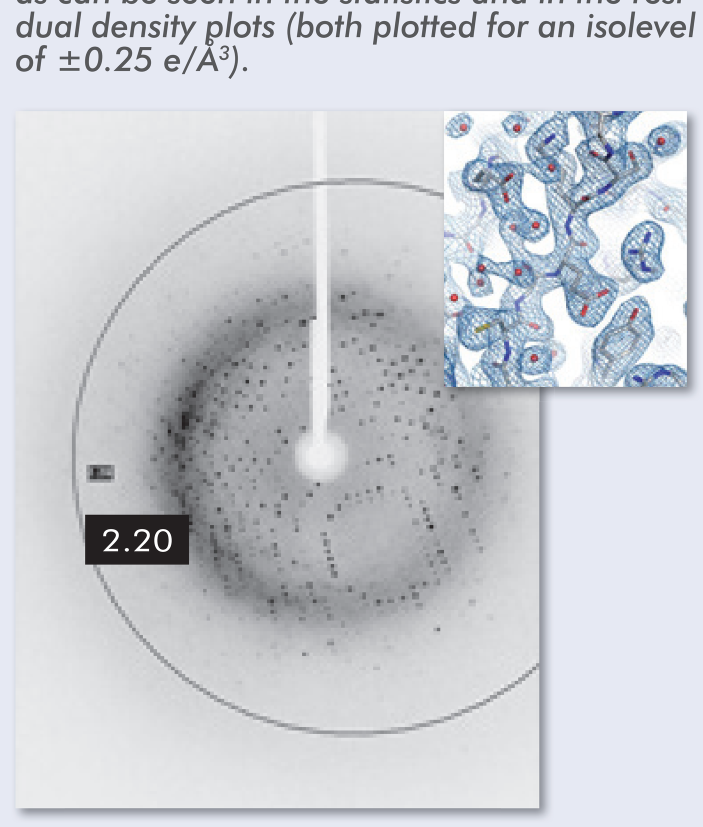


Typical diffraction pattern recorded with a D8 VENTURE 2nd Gen. and Mo-IµS 3.0. The intensity gain of the IµS 3.0 and the superb performance of the PHOTON II detector result in a significant improvement of the data quality, as can be seen in the statistics and in the residual density plots (both plotted for an isolevel of ±0.25 e/Å³).

Protein Crystallography with the Cu-IµS 3.0

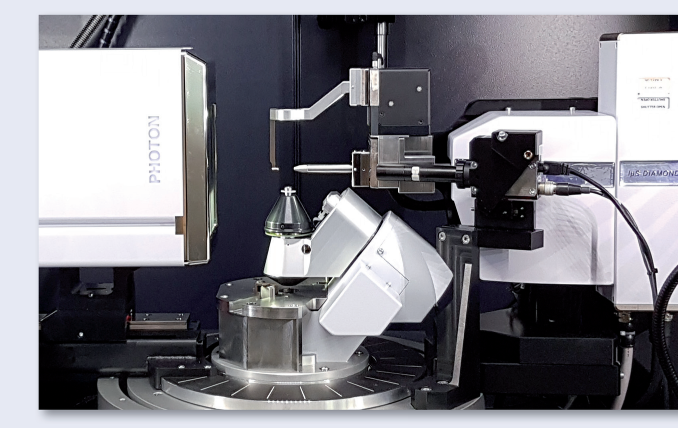
215° data collection on a thin crystal of human NEIL1 (Endonuclease VIII-like protein)

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/σ(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



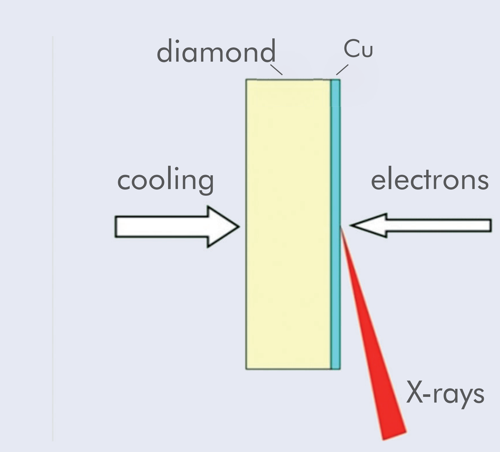
Data statistics (above, left), typical diffraction pattern (above, right) and section of the electron density map (right) of the human NEIL1 protein after structure solution by molecular replacement and refinement with REFMAC.

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

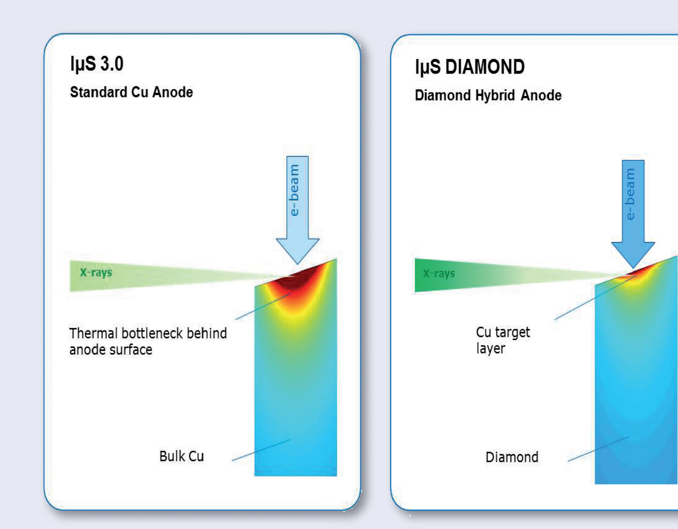


The latest development of Incoatec's X-ray tube factory is a new microfocus sealed tube with a unique anode technology, the diamond hybrid anode, using an industrial diamond substrate as heat sink. The IµS DIAMOND combines this new anode technology with the latest generation of Montel multilayer optics to form the most intense microfocus X-ray sealed tube source for in-house diffraction applications.
During the 24th IUCr congress in Hyderabad, the Cu-IµS DIAMOND for protein crystallography was launched, featuring an unprecedented intensity that is comparable to a low power microfocus rotating anode in a focused beam of about 0.09 mm FWHM.

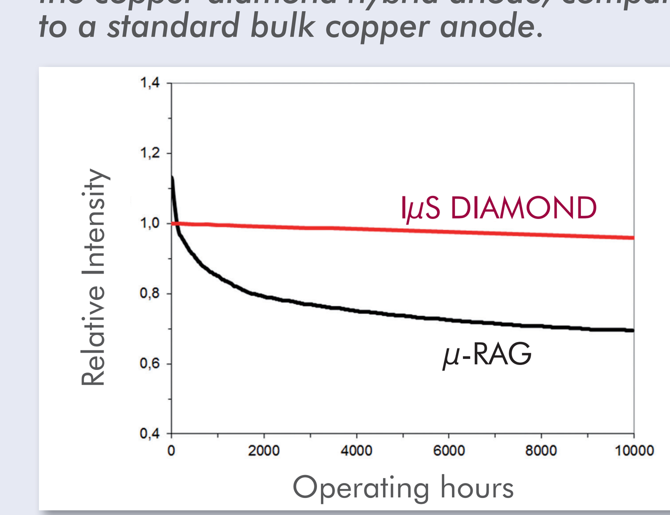
The diamond hybrid anode comprises an industrial diamond as substrate that is coated with a layer of the target material (e.g. Cu), as shown in the illustration to the right. It takes advantage of the exceptional high thermal conductivity of diamond, which is about 5 times higher than that of copper and the highest known conductivity of all bulk materials [3]. The thin copper layer produces the X-rays while the underlying diamond substrate dissipates the heat load more efficiently than a conventional bulk copper anode. 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.
Another advantage of the diamond hybrid anode is that it is much more stable and long-lived than conventional rotating anodes. In a conventional rotating anode the anode typical rotates at about 10,000 rpm. This means the surface is rapidly and repeatedly heated and cooled, millions of times per day. This repeated sequence of thermal expansion and contraction causes the surface of the anode to develop microcracks due to metal fatigue [4,5]. The surface roughening then leads to a gradual reduction in the X-ray output from the anode, typically by 30-40% per year.
The balanced heat management in the IµS DIAMOND, however, 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. Therefore, the intensity of the IµS DIAMOND is about 20% higher than the average intensity output of a modern microfocus rotating anode. The IµS DIAMOND therefore combines the performance of a low power microfocus rotating anode but with the reliability, low maintenance, low cost of ownership and high uptime of a conventional microfocus sealed tube source with a bulk copper anode.



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



Simulation of the improved heat dissipation in the copper-diamond hybrid anode, compared to a standard bulk copper anode.



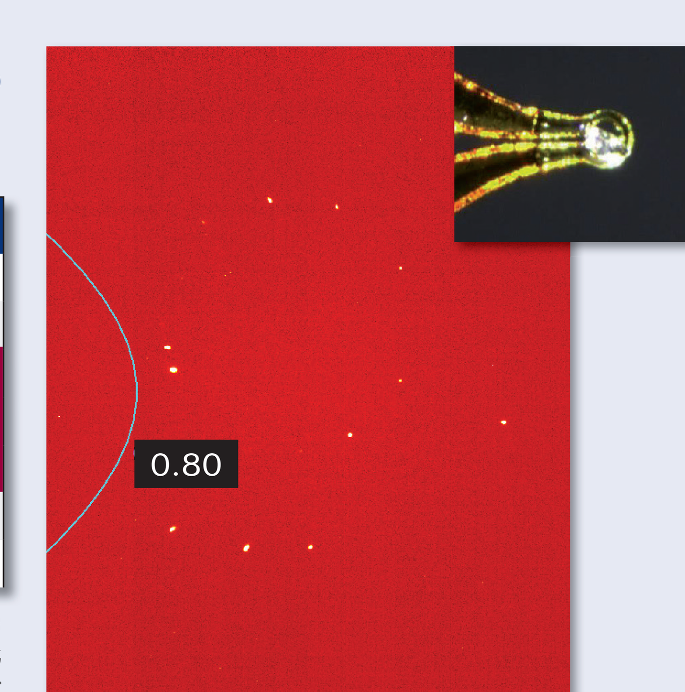
Comparison of the decay in the intensity over time for a low power microfocus rotating anode and for an IµS DIAMOND.

Small Molecule Crystallography with the IµS DIAMOND

Data comparison with a 1 kW microfocus rotating 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/σ>	49.7 (38.8)	55.2 (41.3)
*R*1 (all), *wR*2 (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 1 kW µ-RAG (after ~1500 h of full power operation) and with an IµS DIAMOND.



Conclusion

The quest for ever brighter laboratory sources has continued to increase the X-ray intensity available in the home laboratory. Over the past 10 years, microfocus X-ray sealed tube sources, such as the IµS, have revolutionized laboratory analytical X-ray instrumentation and made the conventional X-ray sources such as fine focus sealed tubes and rotating anodes obsolete for many applications. Most recently the IµS DIAMOND features a novel diamond hybrid anode which achieves a performance previously only attainable with state-of-the-art low power microfocus rotating anodes. The IµS DIAMOND establishes a new class of X-ray sources, combining an intensity output comparable or even exceeding the intensity of a modern low power microfocus rotating anode with all the comfort and lifetime of a standard microfocus sealed tube source with a bulk anode.

References

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