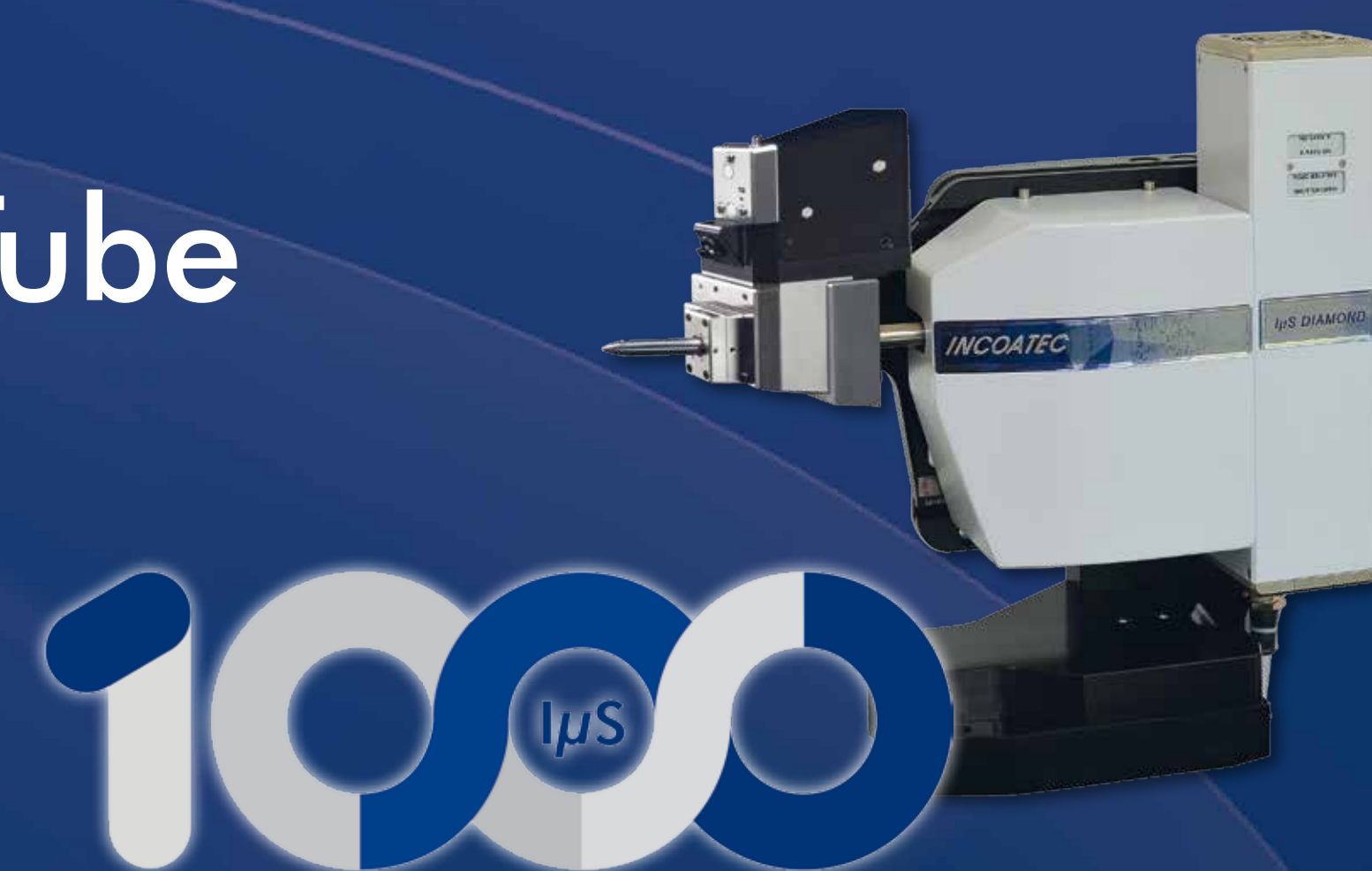


Shine Bright Like a Diamond: Microfocus X-ray Sealed Tube Sources with Diamond Hybrid Anode Technology

F. Hertlein¹, J. Graf¹, T. Stürzer², H. Ott², M. Benning³, P. Radcliffe¹, J. Schmidt-May¹, R. Durst², J. Wiesmann¹, C. Michaelsen¹

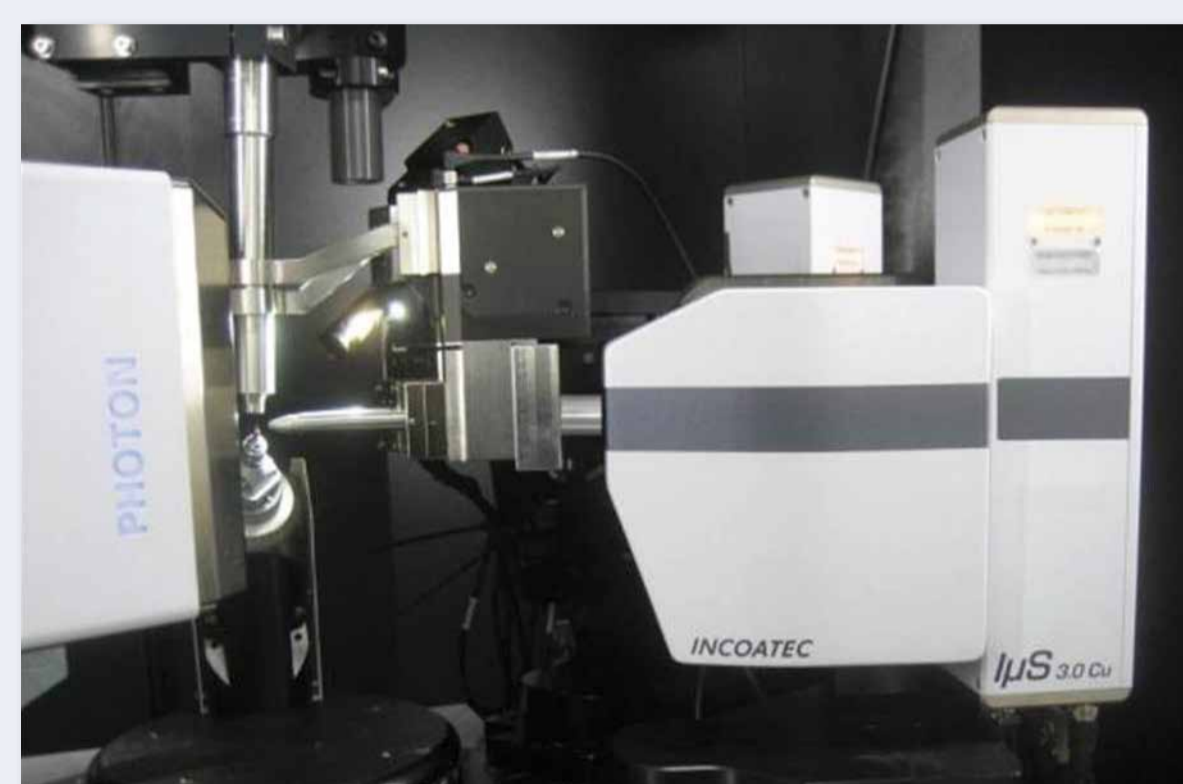
¹ Incoatec GmbH, Geesthacht, Germany; ² Bruker AXS GmbH, Karlsruhe, Germany; ³ Bruker AXS Inc., Madison (WI), USA



Introduction Modern low power microfocus X-ray sealed tube sources, such as the Incoatec Microfocus Source μ S, define the state-of-the-art for most in-house applications in X-ray diffraction [1,2]. Since its introduction in 2006, the Incoatec Microfocus Source μ S has become the market-leading microfocus sealed tube X-ray source for X-ray diffraction applications, such as single crystal diffraction or small angle scattering, with more than 1000 sources sold world-wide. Recently, we introduced a new class of microfocus sealed tube sources with a unique anode technology, the diamond hybrid anode, which uses industrial diamond as a heat sink. This μ S 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.

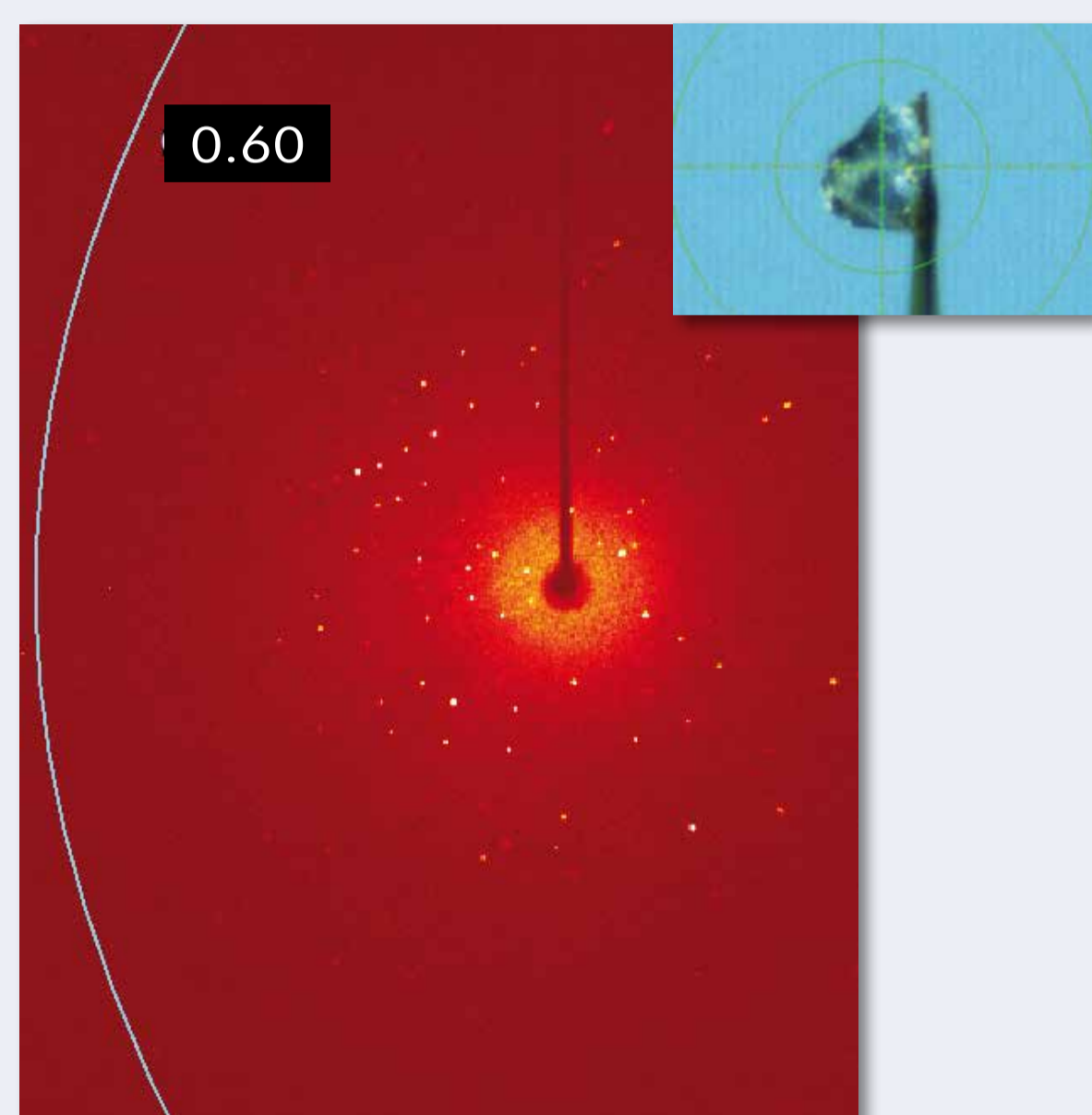
The μ S 3.0 – The first Microfocus Sealed Tube Designed for Crystallography

Throughout the past years, we have gradually improved the performance of our μ S 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 μ S. One important milestone was the launch of the μ S 3.0 at the AsCA conference in Kolkata in 2015. The μ S 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 4×10^{10} 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 μ S 3.0 the most user-friendly microfocus X-ray source ever.

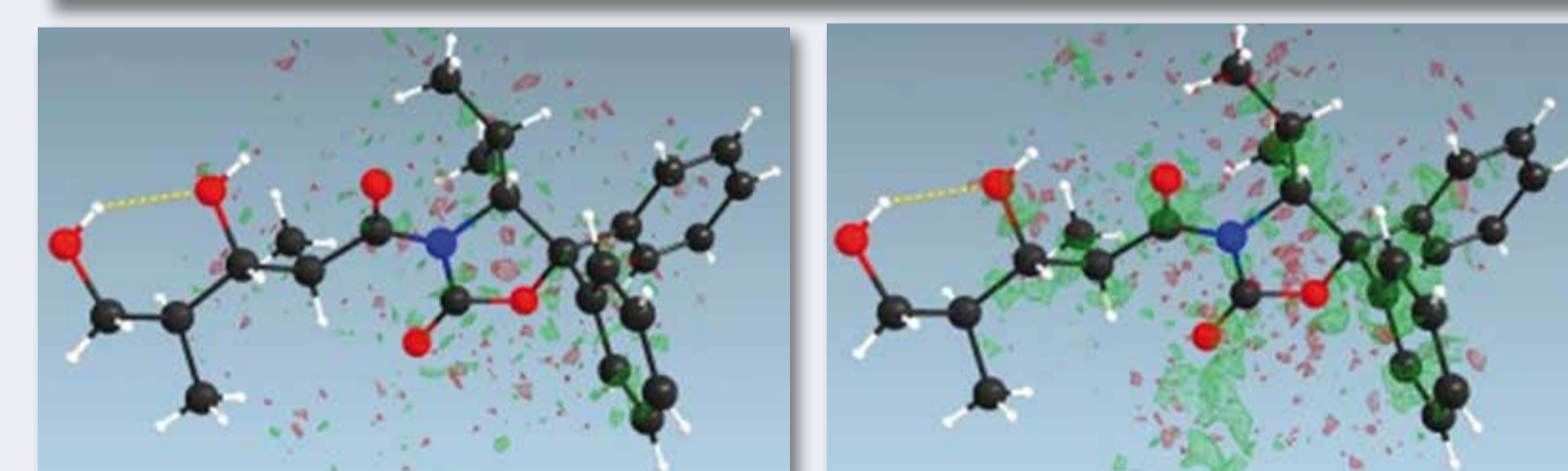


Absolute Structure Determination with the Mo- μ S 3.0
Data comparison on a medium well diffracting crystal of a typical pharmaceutical small molecule compound (0.09 x 0.10 x 0.12 mm³)

Source	Mo- μ S ^{High Brilliance}	Mo- μ S 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)
R1 (all), wR2 (all) [%]	7.17, 14.77	8.11, 18.43
Parsons Q	0.02(30)	0.01(20)
d(C-C) [Å]	1.387(4)	1.390(3)



Typical diffraction pattern recorded with a D8 VENTURE 2nd Gen. and Mo- μ S 3.0. The intensity gain of the μ S 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 electron density plots (both plotted for an iso-level of ± 0.25 e/Å³).



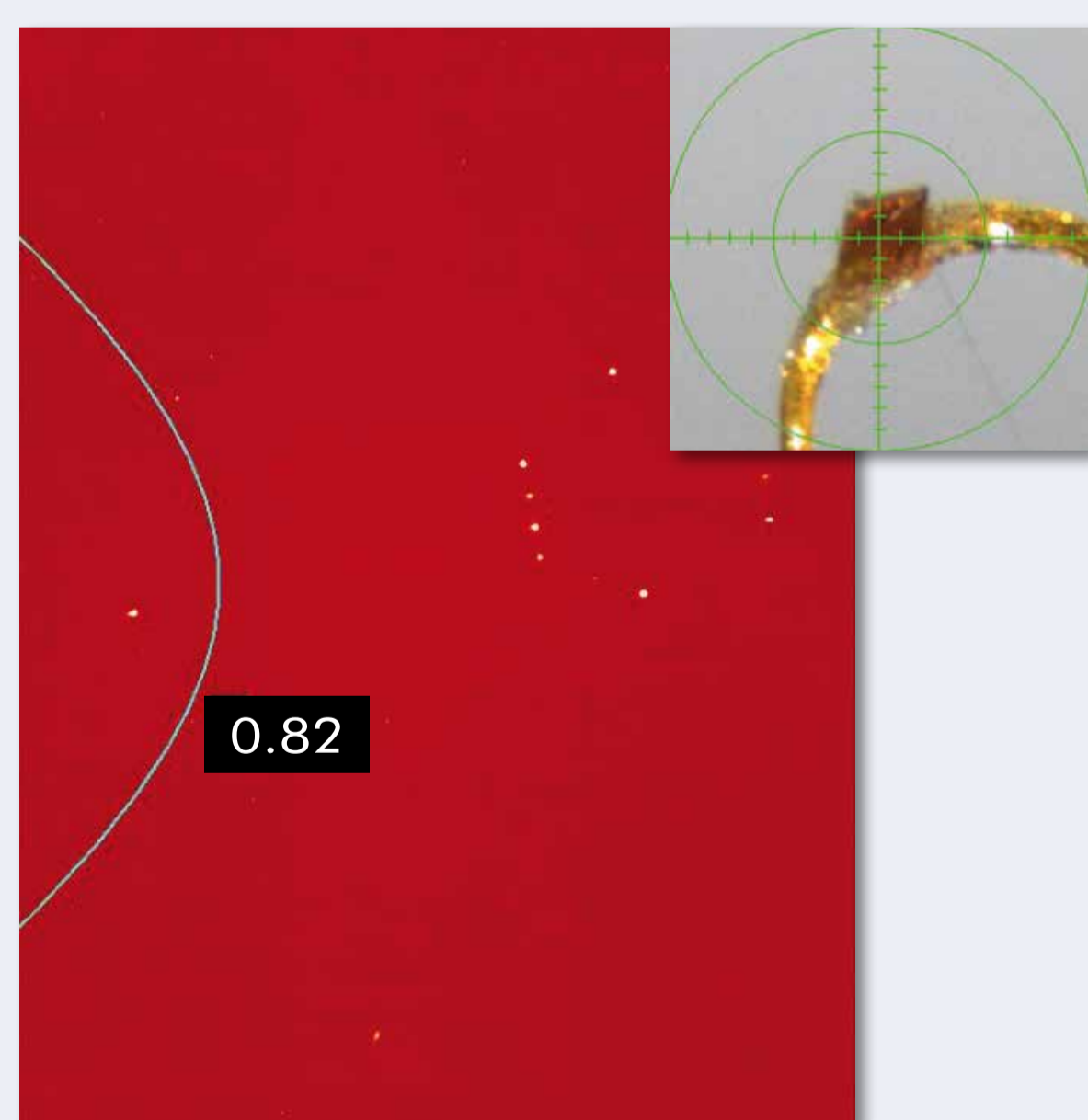
The Cu- μ S 3.0 with HELIOS EF Optics for Chemical Crystallography

One of the biggest challenges in chemical crystallography with in-house instrumentation is the structure determination on very small and weakly diffracting crystals. Therefore, we have developed the new HELIOS EF multilayer optics, which is designed to deliver an intensity in the range of 8×10^{10} phts/s/mm² in a focused beam with a divergence that matches the typical mosaicity of small and weakly diffracting samples. Although primarily designed for chemical crystallography, the HELIOS EF can also be used to study protein crystals with unit cell axes smaller than 100 Å. For unit cells with larger axes, one can either reduce the divergence of the HELIOS EF with appropriate pinhole collimators, or swap the optics with a HELIOS MX optics, which has a significantly lower divergence.

Performance Comparison on a Small Organic Crystal

Optics	HELIOS MX	HELIOS EF
Exposure time [s/0.5°]	2 / 4	2 / 4
Ratio of norm. <I>	1	2.14
<I/σ>	27.1	37.0
R(int) [%]	4.91	3.75
R1, wR2 [%]	3.27; 7.51	2.59; 6.65
Parsons Q	0.06(13)	0.02(8)
d(C-C) [Å]	1.526(5)	1.527(3)

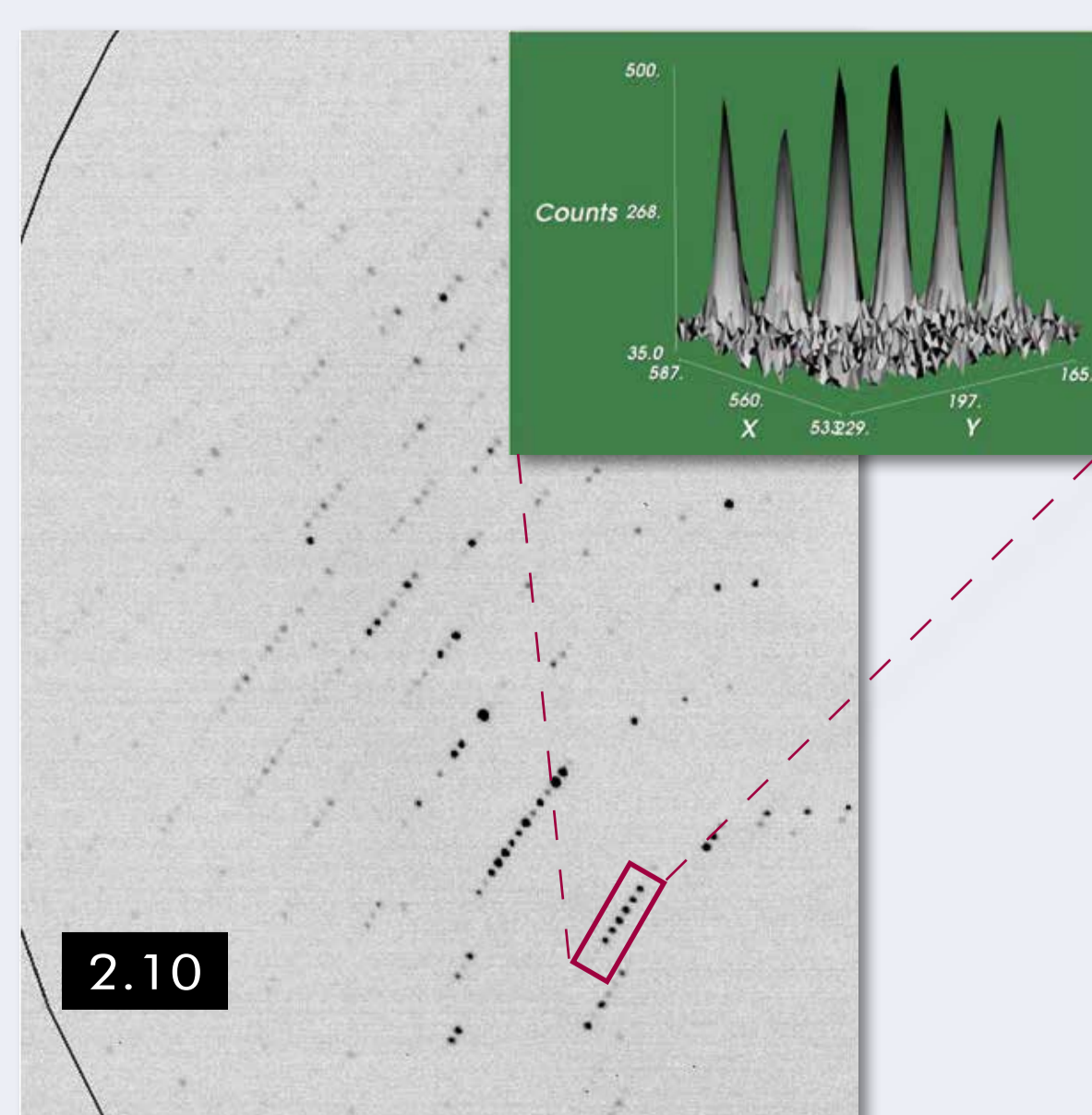
Data statistics on a small Vitamin C crystal (0.04 x 0.10 x 0.10 mm³), showing a 2-fold gain in the integrated intensity, and typical diffraction pattern recorded with the HELIOS EF optics (P2,2,2, a=8.4167(17) Å, b=13.761(3) Å, c=19.304(4) Å, Z=4).



Protein Crystallography: Resolving a 150 Å Unit Cell Axis

Optics	HELIOS MX	HELIOS EF
Exposure time [s/0.25°]	20	20
Resolution [Å]	2.10 (2.20 - 2.10)	2.10 (2.20 - 2.10)
Multiplicity	12.6 (3.9)	12.5 (3.4)
Ratio of norm. <I>	1	0.6
<I/σ>	39.8 (7.8)	34.8 (5.6)
R(int) [%]	4.76 (15.10)	5.42 (18.81)
R(p.i.m.) [%]	1.33 (7.78)	1.56 (10.90)

Data statistics of a Thaumatin crystal recorded with the HELIOS MX optics and with the HELIOS EF optics with a decreased divergence, and typical diffraction pattern recorded with the HELIOS EF optics, showing the resolved Bragg reflections along the 150 Å unit cell axis (P4,2,2, a=b=57.481(5) Å, c=149.63(1) Å, T=100 K).



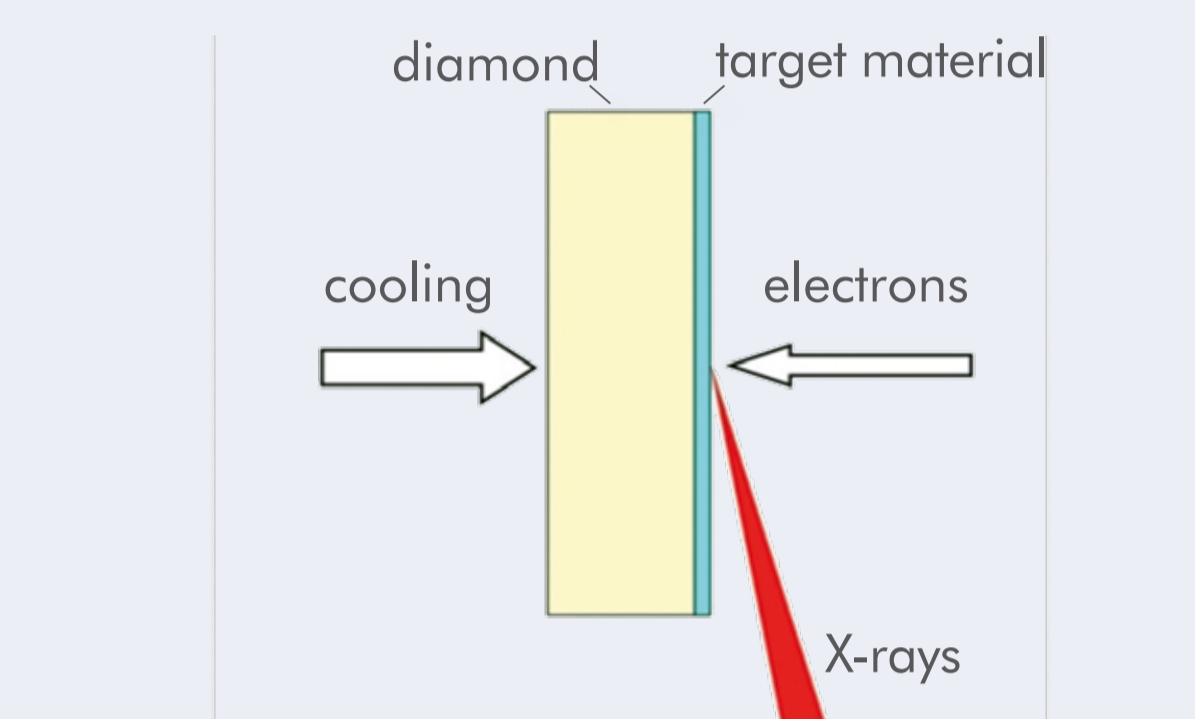
The μ S DIAMOND - The new Microfocus X-ray Tube with a Diamond Hybrid Anode

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 a heat sink. The μ S 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.

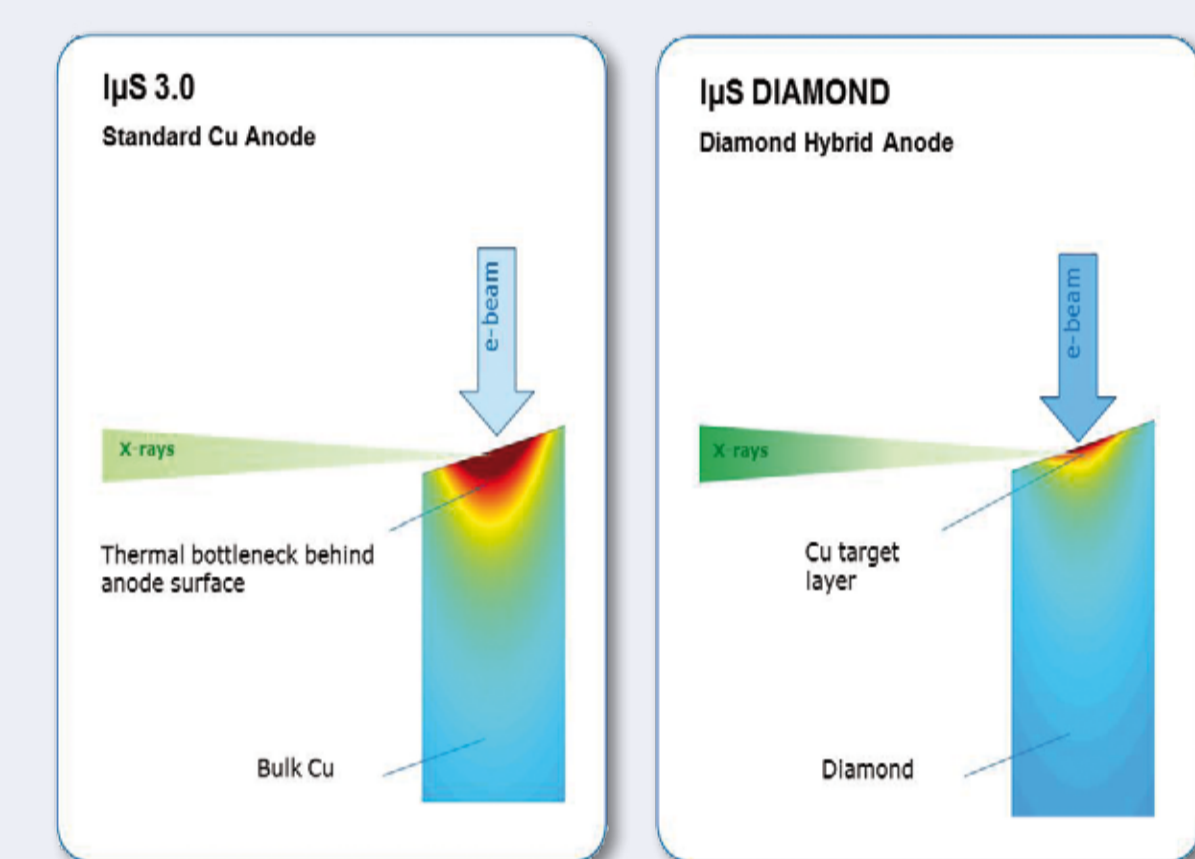
During the IUCr congress in Hyderabad, the μ S DIAMOND for protein crystallography was launched, featuring a focused beam of about 0.09 mm FWHM and an unprecedented intensity that is comparable to a low power microfocus rotating anode. It is now available for Cu-K α and Mo-K α radiation for applications such as single crystal diffraction.



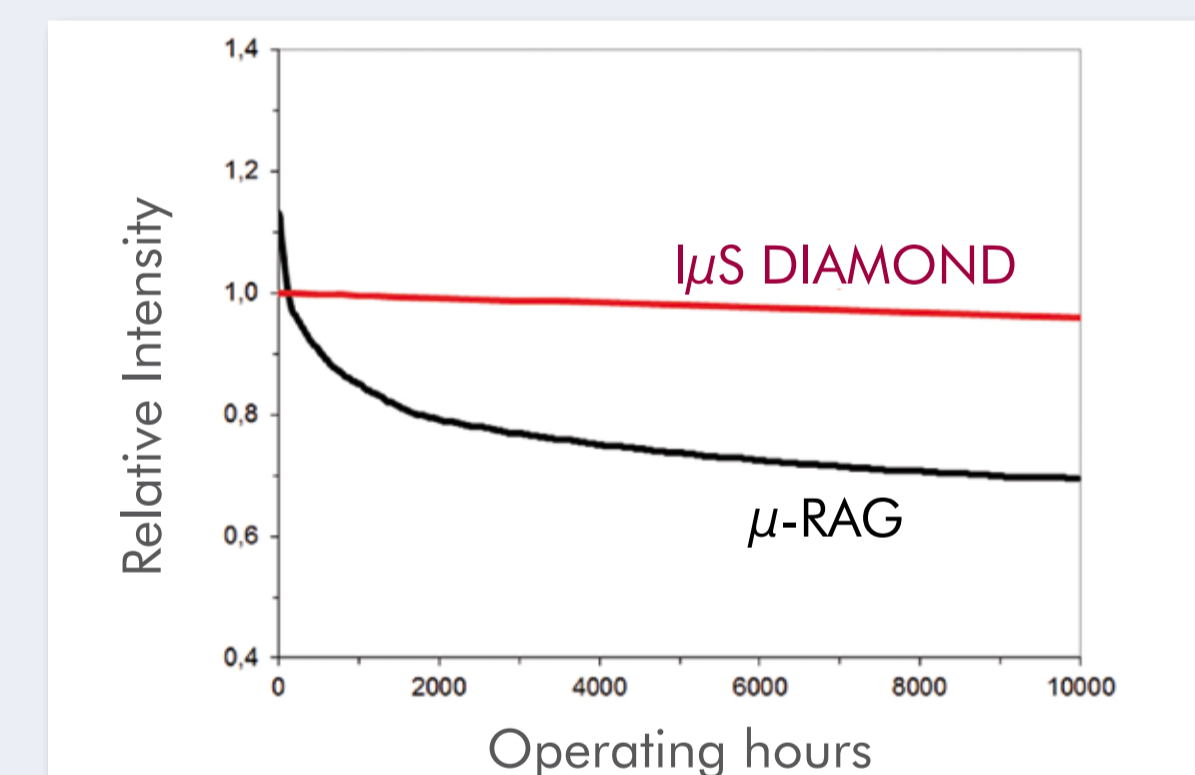
The diamond hybrid anode consists of an industrial diamond as the substrate that is coated with a layer of the target material (e.g. Cu, Mo, Ag), as shown in the illustration to the right. It takes advantage of the exceptional high thermal conductivity of diamond, which is about 5 times higher than that of copper and the highest known conductivity of all bulk materials [3]. The thin layer of the target material produces the X-rays while the underlying diamond substrate dissipates the heat load more efficiently than a conventional bulk copper anode. Consequently, the μ S DIAMOND can accept a higher power density in the focal spot on the anode without damaging the surface of the anode. A further 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 typically 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.



Principle of the diamond hybrid anode used in the air-cooled μ S 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 a μ S DIAMOND.

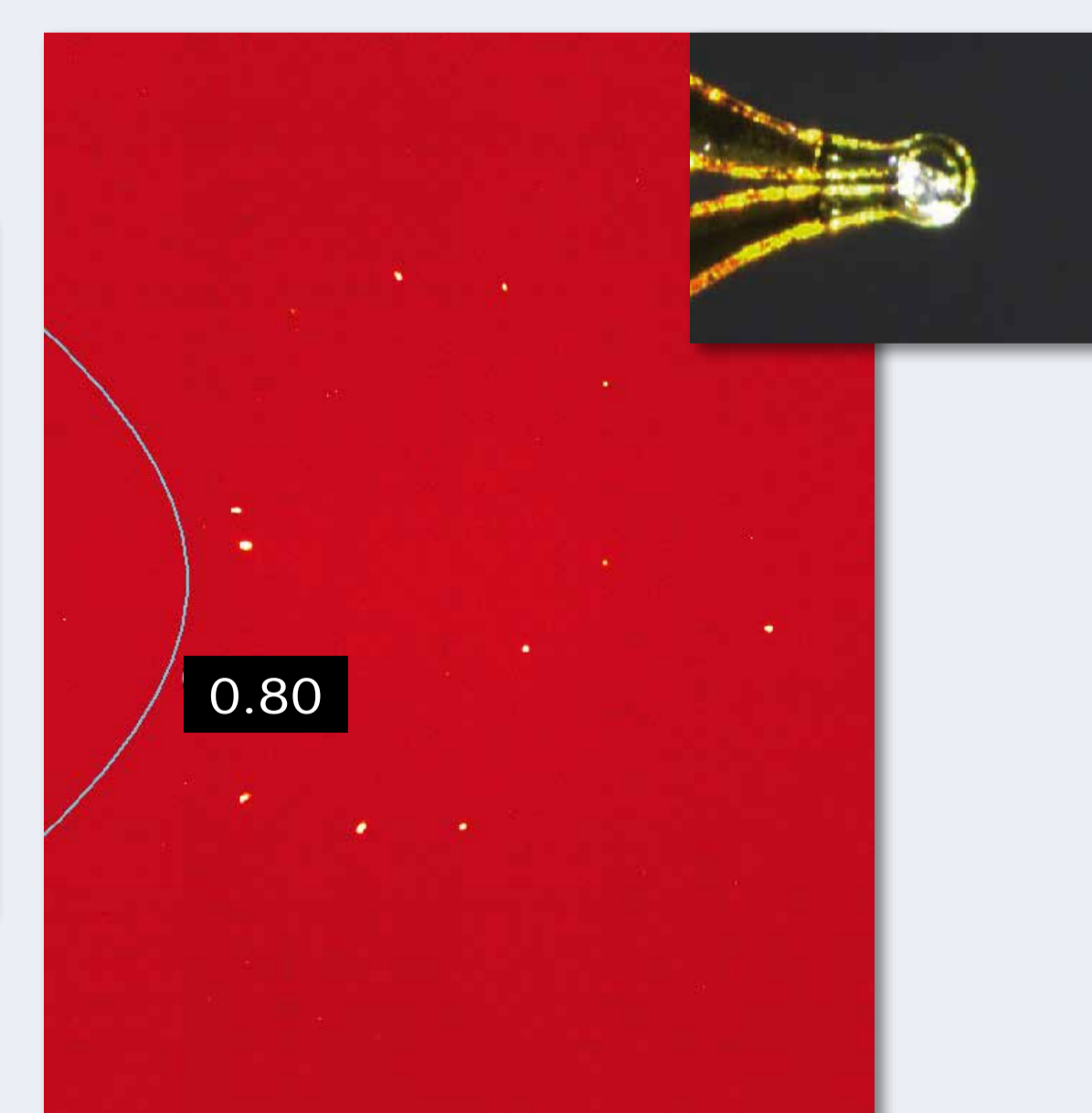
The balanced heat management in the μ S DIAMOND, however, assures that the intensity loss over time is only a few percent over 10,000 h of full power operation, which is significantly lower than in microfocus rotating anode sources. Therefore, the intensity of the μ S DIAMOND is about 20% higher than the average intensity output of a modern low power microfocus rotating anode. Thus, the μ S DIAMOND combines the performance of a low power microfocus rotating anode with the reliability, low maintenance, low cost of ownership and high uptime of a conventional microfocus sealed tube source with a bulk copper anode.

Small Molecule Crystallography with the μ S DIAMOND

Data comparison with a 1 kW microfocus rotating anode

Source	μ -RAG	μ S 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)
R1 (all), wR2 (all) [%]	2.83, 7.46	2.71, 7.43
Parsons Q	0.07(4)	0.05(4)
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 μ S 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 μ S, 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 μ S 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 μ S DIAMOND establishes a new class of X-ray sources, combining an intensity output comparable to 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

- [1] T. Schulz, K. Meindl, D. Leusser, D. Stern, J. Graf, C. Michaelsen, M. Ruf, G. M. Sheldrick, D. Stolke, *J. Appl. Cryst.*, 2009, 42, 885 – 891.
- [2] J. Wiesmann, J. Graf, C. Hoffmann, A. Hembd, C. Michaelsen, N. Yang, H. Cordes, B. He, U. Preckwinkel, K. Erlacher, *Part. Part. Syst. Charact.*, 2009, 26, 112–116.
- [3] A.L. Moore and L. Shi, *Materials Today*, 2014, 17, 163.
- [4] A. Mehranian, M. R. Ay, N. Riyahi Alam, H. Zaidi, *Med Phys.*, 2010, 37, 742-752.
- [5] R. Kákonyi, M. Erdélyi, and G. Szabó, *Med. Phys.*, 2010, 37, 5737–5745.