SINGLE CRYSTAL X-RAY DIFFRACTION

Design and performance of X-ray microfocus sources

Technical Note 21

Overview

The first microfocus source for X-ray diffraction was described in 1998 by Arndt et al [1]. However, despite its impressive intensity, this first-generation microfocus source found limited acceptance due to relatively poor stability and short tube lifetimes.

The second-generation microfocus source, the IμS introduced by INCOATEC in 2006, replaced the inefficient metal total reflection optics used in the first-generation sources with modern Montel multilayer optics. This combination finally achieved a high brilliance, stable, long lifetime microfocus source. Because of these advantages the microfocus source has since become the standard source for laboratory crystallography [2].

The first IμS was based on microfocus tubes that were designed for Nondestructive Testing (NDT) rather than X-ray diffraction. However, these NDT tubes were found to be suboptimal for X-ray diffraction and, therefore, in 2014 Incoatec began to design and manufacture custom X-ray tubes optimized specifically for X-ray diffraction. This note describes the design principles and performance of the latest microfocus sources:

- the IμS 3.0 (2016) which features the first microfocus tube with an optimized take off angle which increases the source intensity by more than a factor of three compared to conventional NDT microfocus tubes,
- the IμS DIAMOND (2018) and the IμS DIAMOND II (2022) which feature unique hybrid diamond anode microfocus tubes providing up to 10 times higher X-ray intensity compared to conventional microfocus tubes.

Increasing brightness and intensity

What limits the brightness and intensity of an X-ray source, and how do the latest microfocus sources, the IμS 3.0 and the IμS DIAMOND II, achieve higher performance than conventional microfocus sources and even rotating anode generators? All laboratory sources are based on the principle of impact ionization of a material target. That is, an electron beam ionizes the K-shell electrons in a target, which, upon recombination, produces characteristic X-ray radiation.

Unfortunately, the energy efficiency of this process is relatively low, typically on the order of 1% [1].

This means that most of the incident electron beam energy is converted into heat. The removal of this waste heat limits the brightness and intensity of the source.

Brightness quantifies the number of X-ray photons emitted per unit area into a unit solid angle per unit time (units: X-rays·mm^2·mrad^{-2}·sec^{-1}). High brightness is necessary to produce an intense, highly collimated beam, such as required for single crystal X-ray crystallography. The brightness of an X-ray tube is proportional to the power density of the electron beam on the anode [1], that is

$$B = \eta p \qquad (1)$$

Where η is the X-ray conversion efficiency of the tube and p is the specific power density of the electron beam on the anode (units: W·mm^{-2}). Therefore, to maximize the brightness of an X-ray tube, one must increase the specific power density on the anode as high as possible. However, there is a limit since, as the power density increases so will the anode temperature and eventually the anode will melt. The maximum permissible power density in an X-ray tube (that is, the power density at which the anode starts to melt), P_m, is given by [3]:

$$P_m = \frac{2\kappa(T_m - T_0)}{r\sqrt{(\pi \ln 2)}} \qquad (2)$$

Where κ is the thermal conductivity of the anode, T_m is the melting point of the anode material and T_0 is the coolant temperature, and r is the radius of the electron beam. Eq. (2) therefore shows the design parameters available to increase the brightness of the tube:

- one may decrease the radius of the electron beam, r. This is of course the fundamental feature of all microfocus sources and indeed the reason why they are brighter than conventional tubes,
- one may increase the thermal conductivity of the anode, κ. In most cases, the thermal conductivity is a fixed property of the anode material. However, it is possible to increase the effective thermal conductivity by employing a hybrid anode as described below.

The intensity of the X-ray beam quantifies the number of X-rays that can be focussed onto the sample position per unit time (units: X-rays·mm^{-2}·sec^{-1}). The intensity is not a property of the X-ray tube alone but rather a property of the entire source, that is, the combination of the tube and optics. So, the same tube may produce different intensities when using different optics.

Practically, intensity is the single most important property of the source since the diffracted signal is directly proportional to the intensity. The relationship between brightness and intensity is given by Liouville's theorem

$$I = BR\delta^2 \qquad (3)$$

Where I is the intensity of the X-ray beam, B is the brightness of the tube, R is the reflectivity of the X-ray optics and δ is the capture or collection angle of the optics - also sometimes called the divergence angle.

It can be seen in Eq. (3) that to increase the source intensity for a given tube brightness, B, one may increase the collection angle of the optics, δ. This is the advantage of using Bragg multilayer optics as they allow relatively large capture angles and, as noted above, this was of course the principle innovation introduced in the IμS. The effective collection angle of the optics can also be increased by decreasing the take-off angle of the anode. This is the principle used in both the IμS 3.0 and IμS DIAMOND II as described below.

Optimizing take-off angle for higher intensity

It is well known that an elongated electron focus combined with a low anode take-off angle allows an X-ray tube to achieve a higher intensity [4]. The reason for this is that the X-ray emission from the anode is, to first order, Lambertian. Thus, viewing the anode at a low take-off angle produces a higher intensity beam, with the intensity increasing with the reciprocal of the tangent of the take-off angle. In practice, it is found that take-off angles on the order of 3-6 degrees are optimal for X-ray diffraction [4].

In essence, the projected area of, say, a 200 × 20 μm^2 focal spot viewed at an angle of 6 degrees appears as a 20 × 20 μm^2 spot but with 10 times the specific brightness. However, most commercially available microfocus sealed tubes were designed not for X-ray diffraction but rather for Nondestructive Testing (NDT). This type of tube is optimized to achieve a wide angle, uniform X-ray emission.

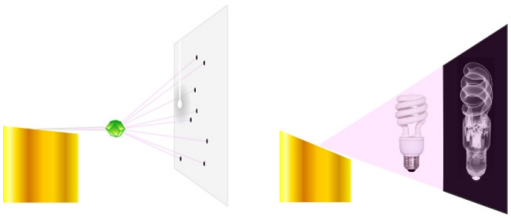

Fig. 1
A low take-off angle anode (left) produces a higher intensity X-ray beam and is thus optimal for crystallography.
In contrast, tubes designed for Nondestructive Testing (NDT) or radiography use a high take-off angle anode (right) to achieve a larger field of view.

Because of this a larger take-off angle is optimal. For example, all other things being equal, a tube with a take-off angle of 6 degrees will produce a beam with more than 3 times the intensity of a tube with a 16 degree take-off angle operated at the same power density.

The IμS 3.0 and IμS DIAMOND II tubes are the only microfocus tubes that were designed specifically for X-ray diffraction and thus employ an optimal take-off angle of 6 degrees. All other currently available microfocus sources on the market use high take-off angle (16 to 30 degrees) tubes designed for NDT.

Diamond hybrid anodes to increase tube brightness

As shown in Eq. (2) above, increasing the thermal conductivity of the anode will proportionally increase the allowable power density and thus increase the brightness.

The anode material is, of course, chosen based upon the desired wavelength of the characteristic radiation for a given application or experiment, typically copper (Cu), molybdenum (Mo), or silver (Ag). Thus, in a conventional tube the thermal conductivity is limited by the intrinsic conductivity of these metals.

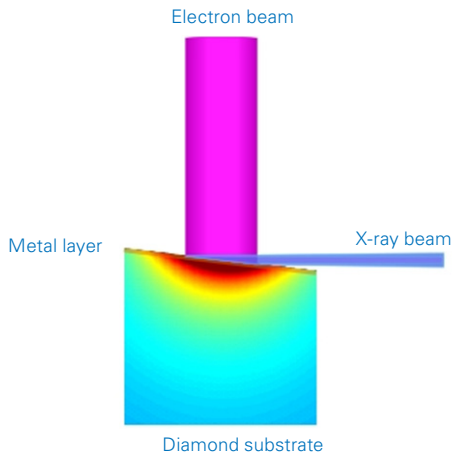

Fig. 2
Diamond has significantly higher thermal conductivity compared to common anode materials. Therefore, a hybrid anode consisting of a thin metal layer (copper, molybdenum, or silver) deposited onto a diamond substrate achieves a significantly higher effective thermal conductivity.

However, the IμS DIAMOND II extends this limit by using hybrid anodes which consist of a thin layer of metal deposited onto a diamond heat sink.

Diamond has a much higher heat conductivity than common anode materials and thus the metal-diamond hybrid anode has a significantly higher effective thermal conductivity [5]. In the IμS DIAMOND II this principle is extended even further by employing isotopically pure ^{12}C in the diamond substrates. Isotopically pure diamond has the hightest thermal conductivity of any known material, 50% higher than natural diamond.

The IμS DIAMOND II employs these novel diamond-metal hybrid tubes to achieve intensities up to 5-10 times higher than conventional microfocus tubes depending on the wavelength [6]. Indeed, the latest diamond anode tubes are able to achieve Mo intensities higher than typical rotating anodes running at more than an order of magnitude higher power (see Appendix below) with consequently much higher maintenance and operating costs.

Summary

The present generations of INCOATEC's IμS are the only microfocus sources on the market that employ microfocus tubes specifically designed for X-ray diffraction.

- The IμS 3.0 microfocus source employs the first microfocus tube with an optimal take-off angle for up to three times higher intensity compared to conventional microfocus sources that employ off-the-shelf tubes designed for Nondestructive Testing.
- The IμS DIAMOND II features a unique metal-diamond hybrid anode to optimize heat conduction and, thus, achieves the highest intensity ever achieved in a sealed tube, with up to 10 times the intensity of a conventional microfocus source.
- The intensity of the IμS DIAMOND II is directly comparable to the intensity from microfocus rotating anodes but with much lower maintenance and running costs. Both IμS sources employ unique active air cooling which achieves a very high beam stability together with extended tube lifetimes, typically over 5 years.

References

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Appendix

The table provides a comparison of the typical performance of microfocus sources (microfocus sealed tubes and microfocus rotating anodes). Values are for comparison only and are subject to change without notice.

Copper Sources	Power (W)	Cooling	Typical tube lifetime (years)	Beam size at focus (µm)	Max divergence (mrad)	Intensity (X-rays·mm^{-2}·sec^{-1})
Microfocus tube sources						
Conventional microfocus source*	50	water	3	-	10	2×10^{10}
Bruker IµS 3.0 Cu	55	air	5	100	13.5	7.6×10^{10}
Diamond anode microfocus sources						
Bruker IµS DIAMOND II Cu	50	air	5	80	13.5	1.2×10^{11}
Microfocus rotating anodes						
Typical rotating anode**	1200	water	1§	90	10	1.7×10^{11}

Molybdenum Sources	Power (W)	Cooling	Typical tube lifetime (years)	Beam size at focus (µm)	Max divergence (mrad)	Intensity (X-rays·mm^{-2}·sec^{-1})
Microfocus tube sources						
Conventional microfocus source*	50	water	3	-	4.5	1×10^{9}
Bruker IµS 3.0 Mo	70	air	5	100	5	3.3×10^{9}
Diamond anode microfocus sources						
Bruker IµS DIAMOND II Mo	80	air	5	80	5	10×10^{10}
Microfocus rotating anodes						
Typical rotating anode**	1200	water	1§	90	4.5	5.4×10^{9}

Silver Sources	Power (W)	Cooling	Typical tube lifetime (years)	Beam size at focus (µm)	Max divergence (mrad)	Intensity (X-rays·mm^{-2}·sec^{-1})
Microfocus tube sources						
Bruker IµS 3.0 Ag	70	air	5	100	4	1.5×10^{9}
Diamond anode microfocus sources						
Bruker IµS DIAMOND II Ag	50	air	5	80	4	3.8×10^{9}

* Microfocus source based on standard Non-Destructive Testing Tube (RTW).

** Microfocus rotating anode with 70 µm focus.

§ Refurbish (clean, polish) anode once per year.

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