

MICROFOCUSING X-RAY EQUIPMENT FOR THE LAB DIFFRACTOMETER

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ABSTRACT

Results of a new stationary microfocusing sealed tube X-ray source are presented. Measurements with the new low-maintenance, high-brilliance microfocus source I μ S equipped with different two-dimensional beam shaping multilayer optics are shown. The comparison of I μ S with typical sealed tube fine focus systems shows data of outstanding quality in diffractometry applications using a two-dimensional detector. A huge improvement in intensity and resolution by a factor of about 16 was observed. The I μ S delivers very promising results especially for measurements of powders in transmission geometry. The focusing on the detector enables better crystallite statistics and better resolution. For some applications there are even intensity gain factors in the range of 100 achievable. For small angle scattering a factor of 5 in comparison to a typical sealed tube instrument was observed when using an I μ S with optics for a parallel beam.

INTRODUCTION

X-ray diffraction (XRD) is a very widespread non-destructive technique for analyzing a wide range of materials throughout industry and research institutions. The main hardware components of laboratory X-ray diffractometers include an X-ray source, optics for monochromatization and shaping of the X-ray beam, a sample stage usually on a goniometer, and an appropriate detector. In this contribution, we will present a new set-up for the beam delivering system, comprising of the source plus the optics. The Incoatec Microfocus Source I μ STM has a small spot of below 50 μ m and a very high power load on the anode. The small size of the source is ideal for the use of two-dimensional shaping X-ray optics. Laterally graded multilayer mirrors collimate the flux emitted from the source focus within a large solid angle and reflect a monochromatic beam onto the sample.

Parallel beam geometries are always needed for measurements which cannot accept a large divergence. Typical applications for parallel-beam geometry are X-ray reflectometry, small angle scattering, and high-resolution measurements of lattice constants. Focusing geometries are used when analyzing a small amount of, for example, powder or very small and weakly scattering samples where every photon is needed. The outer dimensions of the crystals or small amount of powder samples typically range from 10 to 300 μ m. Ideally, the X-ray beam is shaped to match the dimensions of the single crystal. If the beam is too small, only a small and—in the case of a sample rotation—alternating part of the crystal is exposed by the beam and contributes to the diffractogram. If the beam is larger than the crystal, the unused portion of the beam contributes only to the background and reduces the data quality.

The following section “Incoatec Microfocus Source I μ S” presents the common solutions for the X-ray generation in home-lab instruments. The second part will highlight the design, the production, and the characteristics of the two-dimensional shaping multilayer optics. We will conclude this section with a summary of the technical details of the I μ S. The last section “Applications” will show selected examples of the gain in performance with the new I μ S in comparison to conventional sealed tube configurations.

THE PHYSICS

Typical X-ray sources in lab instruments produce a broad spectrum of photon energies. The source spectrum consists of characteristic emission lines which are defined by the anode material, and of the continuous spectrum of the Bremsstrahlung, which is dependent on the generator settings. In diffractometry only a small range of the characteristic radiation is usually used and the beam needs to be monochromatized. The most widespread photon energies are CuK α at 8.04 keV and MoK α at 17.45 keV. The beam of an X-ray source is characterized by the flux density Φ (counts/s/mm²) and the convergence angle Ω (mrad). The brilliance of an X-ray source is $B = \Phi \div (\Omega_x \times \Omega_y)$, with x and y as the two axes perpendicular to the propagation direction of the X-ray beam. For a symmetrical spot the brilliance is

$$B = \Phi \div \Omega^2 \text{ [counts/s/mm}^2\text{/mrad}^2\text{]} \quad (1)$$

which is flux density divided by the convergence/divergence. The Liouville theorem declares that B is a constant of the source and can not be increased without increasing the power density on the anode. If an optical element reflects the beam, the brilliance would only remain constant in the ideal case, that is, when the reflectivity R of the optics is 100%. In reality, the brilliance is reduced by the true reflectivity R of the optics that is usually $\leq 90\%$.

Therefore, the source is an important contributor to the performance quality of an X-ray instrument. The source should offer the best possible brilliance. The beam shaping optics should, then, perform without significant loss to preserve the source brilliance. As stated in the introduction, the beam cross-section should match the sample diameter. The best way to achieve this is to focus the beam onto the sample. Otherwise, the beam needs to be shaped by using slits or pinholes which diminish the total flux on the sample.

For high performance optics, the main requirements are best possible reflectivity and precise beam shaping to satisfy the requirements of the experiment on the spatial resolution and on the total flux density. Furthermore, most modern X-ray analytical methods need a monochromatic beam. Synthetic multilayer mirrors are well established as excellent beam-shaping devices with a good spectral purity [1,2]. Their high reflectivity and broad rocking curve make them the ideal optics for conserving the source brilliance [3]. These mirrors consist of multilayer films which reflect X-rays by the effect of Bragg diffraction. The modified Bragg equation for multilayers is

$$\lambda = 2d \sin \theta \times \left(1 - \frac{\delta}{\sin^2 \theta} \right) \quad (2)$$

with wavelength λ [nm], lattice constant d [nm], angle of incidence θ , and the dispersion δ .

INCOATEC MICROFOCUS SOURCE I μ S

Generally, there exist three different types of X-ray sources for home-lab diffractometry: sealed tubes (ST), microfocus low-power tubes (μ S), and rotating anode generators (RAG). In comparison, the water-cooled sealed tubes are inexpensive and need little maintenance. The most brilliant sources with the best performance, but also with the highest price and significant maintenance are the latest generations of rotating-anode generators. They offer the highest brilliances. The performance of the newest microfocusing rotating anode generators, which still run at about 2.7 kW, is comparable with synchrotrons of the 2nd generation.

For I μ S we decided to incorporate the newest type of microfocus low power sources, which offer a small source spot in the range of below 50 μ m. The X-rays are produced very effectively with a high-power load in that small spot. This low-power tube needs a generator with only 30 W continuous maximum power. The complete source is, therefore, operated air-cooled by a small fan and does not require an additional cooling medium. Figure 1 shows a picture of the I μ S system.

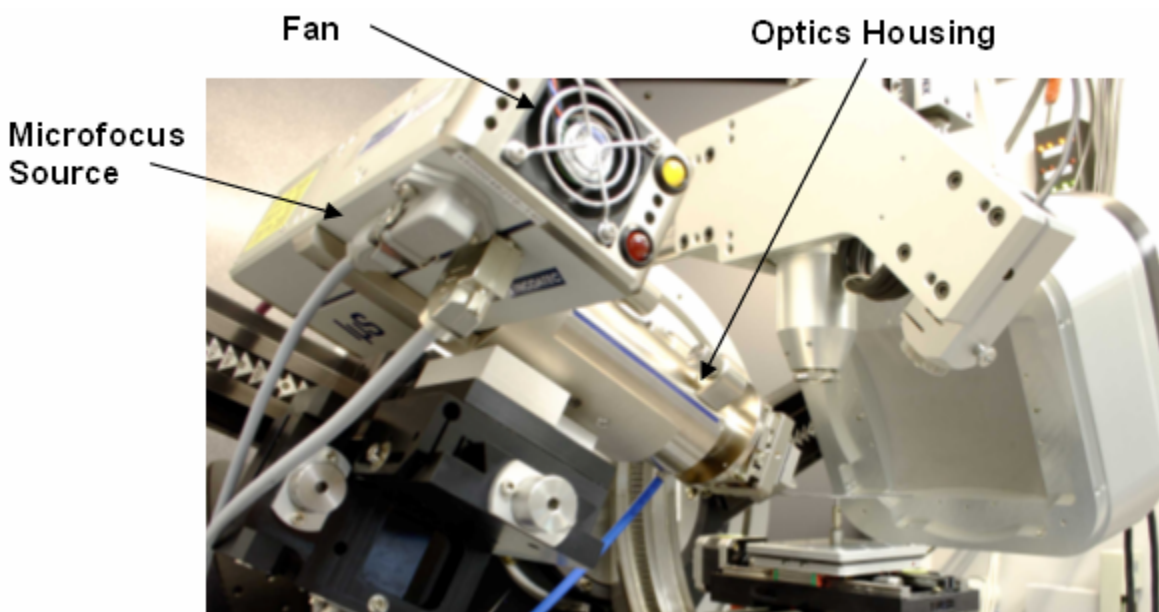


Figure 1. Incoatec Microfocus Source I μ S integrated in an XRD system with a 2D detector (Bruker D8 GADDS).

The beam profile of the source is shaped in two dimensions by the newest type of Montel mirrors [4–6], the so-called Quazar optics [7]. Using ray tracing calculations, they are optimized with respect to the source profile and to the application of the users. The optics are mounted in a housing which is evacuated to prevent radiation damage of the multilayer and to minimize air absorption.

Basically, three types of Quazar Optics are offered for the following purposes: (a) focusing-beam optics which deliver as much flux as possible in a small spot with a moderate divergence, (b) parallel-beam optics with low divergence with a larger beam size, and (c) hybride optics that focus in one dimension and that render a parallel beam perpendicular to it.

Type (a) is called SCD and is mainly used for single crystal diffraction. Type (b) is called LD and offers a beam with low divergence, for applications like small angle scattering. These low divergence models are available with different divergences from 0.35 to 1 mrad. Other models can be made upon request. Since brilliance is not a function of the optics, Eq. (1) can be used to calculate the flux density differences between the models with different divergences. It can be seen that the flux densities of the LD models are typically 1-2 orders of magnitude lower than the flux densities of the SCD models. The integral flux is then a function of the cross section of the beam. Since most low-divergence applications such as microdiffraction do not require large beam cross sections, the flux of the LD models is usually significantly smaller than the flux of the SCD models.

Table I. Technical details for different types of $I\mu S$.

	Flux (counts/s)	Focal Spot Diameter (μm)	Divergence (mrad)	Distance between source and image focus (mm)
SCD Cu	$> 3 \times 10^8$	250	5	600
SCD Mo	$> 1 \times 10^7$	100	5	400
LD Cu			0.35 to 1	

The main technical details are summarized in Table I. $I\mu S$ is currently available for Cu and Mo radiation. The high flux and the stable beam allow for high performance measurements for SCD or SAXS.

APPLICATIONS

Selected results that compare $I\mu S$ to conventional sources used for typical XRD applications with a two-dimensional detector are presented. All measurements were performed with a VÅNTEC-2000 detector.

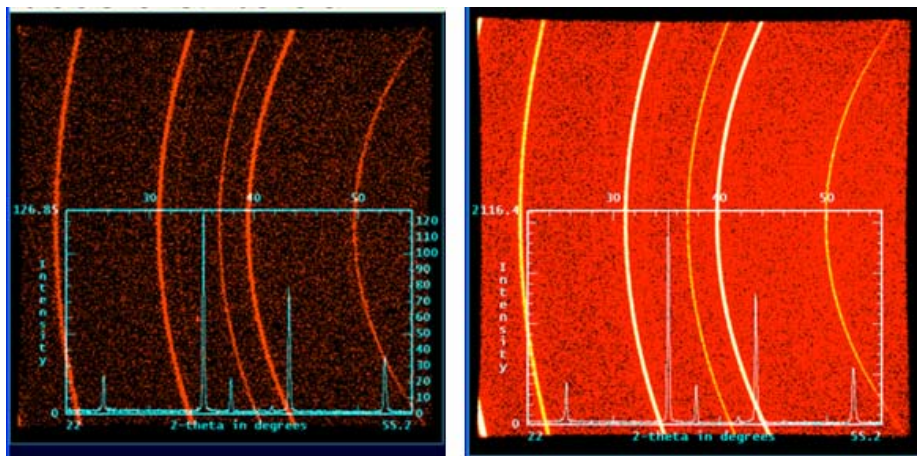


Figure 2. Signal of corundum with a sealed tube set-up (left) and an $I\mu S$ set-up (right). Measurement time was 100 s each.

Figure 2 shows the signal of a corundum standard sample on a state-of-the-art two-dimensional X-ray detector (VÅNTEC-2000) combined with a sealed tube (ST) with a Göbel Mirror for a parallel beam (left), and with the $I\mu S$ with a Hybride Quazar Optics (right). This hybride optics is focusing in one direction and delivers a parallel beam in the other direction. The detection time was in both cases 100 s, the geometry was the same, and in both cases a 0.3 mm collimator was

used. The total counts were left 78000 and right 1235000. Thus the I μ S enables a 16 times higher intensity. The resolution was about 0.19° in both cases. To obtain the same high intensity as the I μ S, but with a ST, a collimator of > 0.8 mm is needed. However, the use of a larger collimator is accompanied by a loss of resolution.

Figure 3 shows a second example of the I μ S performance. The typical quartz powder was measured and the “five fingers” of the quartz signal are well resolved. For sealed tube systems, this is only possible with point detectors. Here it is demonstrated for the first time with a system containing a two-dimensional detector and a microfocus source.

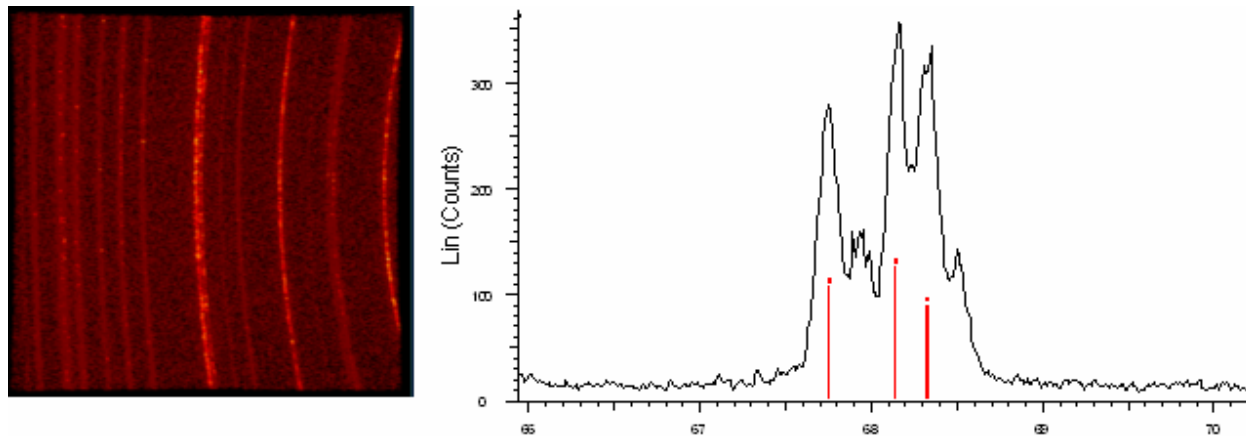


Figure 3. Signal of quartz powder with an I μ S: diffraction pattern (left) and line-scan in the range of the “Five-Finger”-peak (right). The measurement was made with a Bruker D8 GADDS and 600 s collection time.

I μ S was also used for measurements in transmission. Here, a focusing Quazar optics was used, which focused the beam into the detector. Figure 4 shows a comparison of a ST set-up (left) with I μ S (right). Both systems were comparable with a 0.3 mm collimator. For the ST the detection time was 120 s; for I μ S it was 15 s—8 times lower.

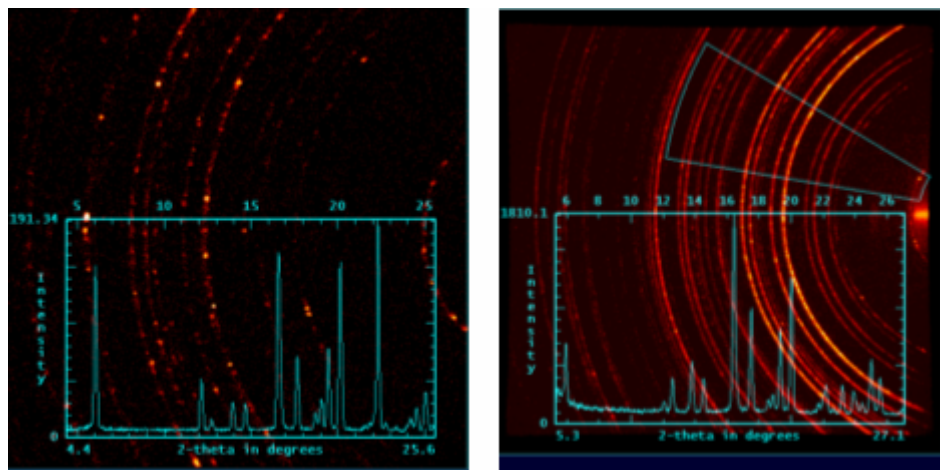


Figure 4. Signal of Ibuprofen with a sealed-tube (left) and an I μ S (right), both with a Bruker D8 GADDS and 120 s (left) and 15 s (right) collection time.

With the I μ S a larger sample area is irradiated. Together with the higher flux density of the system, much better crystallite statistics and intensities were achieved. If enough samples are present, the intensity gain of the I μ S in comparison to the ST can be 100 times or more.

Finally, tests of the small angle scattering module were performed with the low-divergence Quazar mirror providing a divergence of about 1 mrad. The scattering patterns were recorded with a Bruker AXS NanoSTAR using a three-pinhole system (750, 400, and 1000 μm). The HiSTAR detector was placed 1050 mm behind the sample stage. Figure 5 shows a typical scattering plot. In the three-pinhole SAXS set-up, $\text{I}\mu\text{S}$ -SAXS-LD shows a five-fold intensity gain over the conventional home-lab set-up with a 1.4 kW fine focus sealed tube and cross-coupled Göbel mirrors. This gain in performance is the optimum achievable value as predicted by ray tracing calculations.

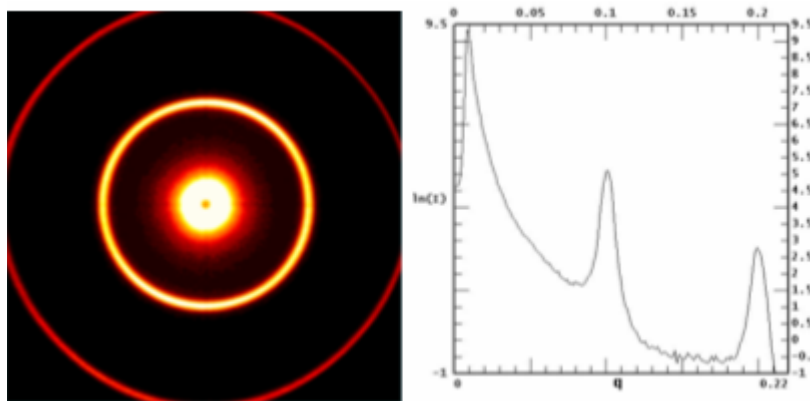


Figure 5. Typical scattering pattern together with the horizontal integration of a silver behenate standard sample.

SUMMARY

The new Incoatec Microfocus Source $\text{I}\mu\text{S}$ incorporates an optimized combination of an extremely bright and very durable stationary 30 W microfocus source and the newest type of Montel optics, the Quazar multilayer optics. $\text{I}\mu\text{S}$ has all the advantages of a sealed tube system, and a performance exceeding combinations of traditional rotating anodes with multilayer optics. With $\text{I}\mu\text{S}$ we have collected data of outstanding quality in applications like phase identification, μ -diffraction, screening, and small-angle scattering. The applications demonstrate that we achieve much better quality in XRD applications with a two-dimensional detector.

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