Comparison of a conventional rotating anode generator with a Xenocs GeniX micro-beam generator

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Introduction

Conventional rotating anode X-ray generators (RAG's) like the Nonius FR591 and the Rigaku RU-H3R have been used for a long time as work horses for in-house crystallography. They are typically operated at 50 kV and 100 mA (i.e. 5 kW) with a focal cup of 0.3 x 3 mm. Usually, they are combined with confocal multi-layer optics like the Montel or Osmic mirrors. This type of generator requires a lot of routine maintenance like replacement of filaments and ferrofluidic seals. After some time of operation, the anode needs repolishing. These RAG's also require special electrical installations and a cooling water supply held at a certain temperature - and they occupy a lot of space.

The most severe limitation for modern crystallography, however, might be, that the beam produced by those generators is considerably larger than the crystals being irradiated. In fact, the multi-layer mirrors can use only a small fraction of the primary beam and even with that small fraction, the divergence is relatively large. That all translates into a comparatively low "brilliance" of the beam.

This circumstance pushed the development of small focus/high brilliance rotating anode generators like the Microstar™ (Bruker-Nonius) or the Micromax-007™ and FR-E™ (Rigaku) and optimized multi-layer optics. While being very powerful, those combinations of generators and optics are very expensive and, in terms of electrical and as well as cooling water installations, they have similar requirements as conventional RAG's.

A crystallographer's dream would certainly be a high-brilliance RAG with the price tag and the running costs of a sealed-tube generator. In the real world, the best approach to this dream might be a modern micro-focus generator like the Xenocs GeniX Cu High Flux™ (Figure 1). In combination with a mar345dtb image plate detector and goniometer system, this becomes a powerful yet very affordable tool for all the needs of in-house data collection.

In this study, we have used a mar345dtb detector system (Figure 2) to compare the performance of a GeniX Cu High Flux generator operated at 50kV/1mA (50 W) with a conventional rotating anode generator (RAG) operated at 40kV/50mA (2000 W) with a 0.2 x 2 mm focal cup. As test case, we used 2 frozen lysozyme crystals of different size to assess the differences in "brilliance" of the beam.

Figure 1: GeniX Cu High Flux micro-beam generator with single reflection multi-layer optics

Experiment

X-ray Detector System

The mar345 image plate detector (Figure 2) is a very low noise image plate detector that delivers superb data quality. The scan times are as short as 34 seconds for a 180 mm diameter scan and only 66 sec for a 345 mm diameter scan. The pixel size is either 100µ or 150µ.
The "***desktop beamline***" (***dtb***) is a very powerful single axis goniometer system. A unique feature is the capability of automatically finding and optimizing the X-ray beam. It is also very helpful for characterizing the X-ray beam.

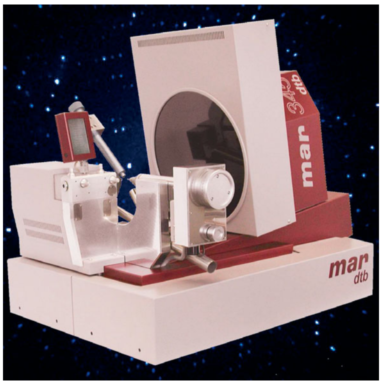


Figure 2: mar345 image plate detector mounted on a "desktop beamline"

GeniX Cu High Flux X-ray Generator

The GeniX is a high brilliance micro-beam generator (Figure 1) that is operated at 50 Watts (50 kV, 1 mA). The generator is coupled to a single reflection multi-layer mirror that delivers a beam with physical dimensions in the focal spot of approx. 230 µm x 230 µm FWHM and a divergence of < 5 mrad (Figure 3). Despite the low power, the efficient coupling of the micro-beam generator with the Xenocs optic provides a high intensity beam with performance equivalent to that of traditional rotating anode generators, but with the benefit of lower facilities and maintenance requirements. In fact, the electrical requirements of the GeniX generator are such that the unit can be run from a standard single phase wallsocket. There is no need for a cooling water supply, since the included water/air-cooler is en entirely closed system.

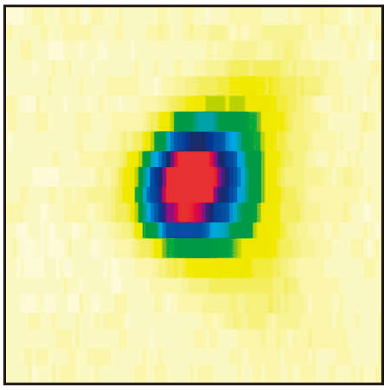


Figure 3: primary beam of the GeniX generator at focal distance. The shown area is 1 x 1 mm (as scanned by the dtb).

Rotating Anode Generator

In this study, we used a conventional rotating anode generator that has been operated at 2000 Watts (40 kV, 50 mA) with a focal cup of 0.2 x 2 mm. With this type of focal cup, the maximum power is 3000 Watts. A more common configuration is a 0.3 x 3 mm focal cup that can run at 5 to 6 kW, but for combination with multi-layer mirrors, the smaller focus works better. The generator was equipped with an Osmic "blue" multi-layer mirror. The resulting beam had physical dimensions in the focal spot of approx. 350 µm x 350 µm FWHM (Figure 4).

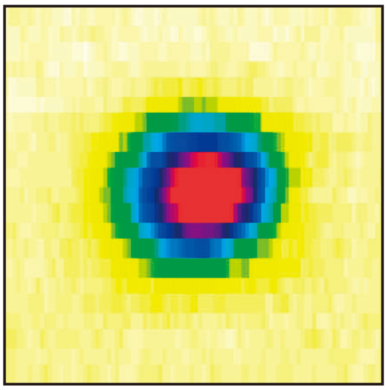
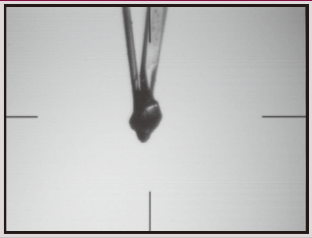
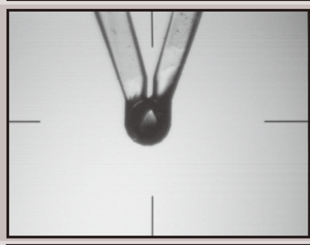
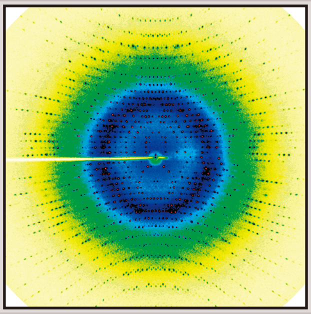
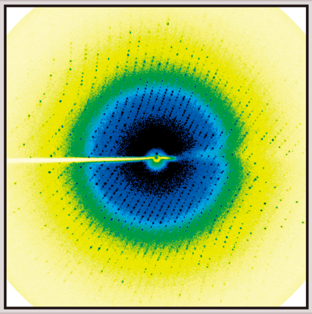


Figure 4: primary beam of the RAG at focal distance. The shown area is 1 x 1 mm (as scanned by the dtb).

Data collection and processing

The data for all crystals were collected first on the RAG, then on the GeniX generator. The crystals were kept on the same mar345dtb detector system and have not been taken down when switching generators. Thus, exactly the same orientation and the same angular range could be kept. Data were processed using the automar program package (i.e. programs *marProcess* for data reduction, *marPost* for data merging and *marScale* for scaling).

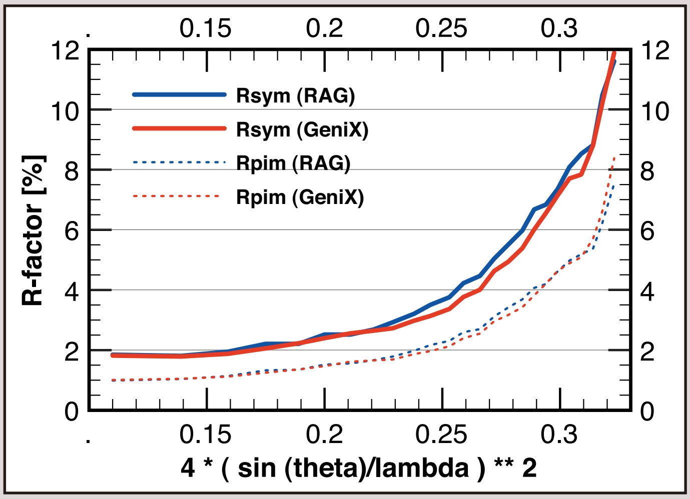
Crystal	lysozyme1	lysozyme2
Crystal at $\varphi=0^0$		
Crystal at $\varphi=90^0$		
Diffraction pattern		
Space group	$P\,4_3\,2_1\,2$	
Unit cell axes	a=79.1 b=79.1 c=37.9 Ang.	
High resolution limit	1.55 Ang.	1.7 Ang.
Mosaicity	0.2^0	0.3^0
Size of crystal	400µm x 300µm x 200µm	120µm x 100µm x 80 µm
Distance crystal-detector	100 mm	100 mm
Exposure time per image	120 sec	240 sec
Total no. of images	90	90
Total data collection time	4 hrs 57 min	9 hrs 30 min
Delta-φ per image	1.0^0	1.0^0
Completeness	99.4 %	99.9 %
Multiplicity	6.60	6.58
Rsym RAG / GeniX	2.7 / 2.5 %	6.9 / 6.0 %
<Intensity> RAG / GeniX	7879 / 5858	1290 / 1547
I / σ RAG / GeniX	11.0 / 11.2	4.9 / 5.6

Data comparison

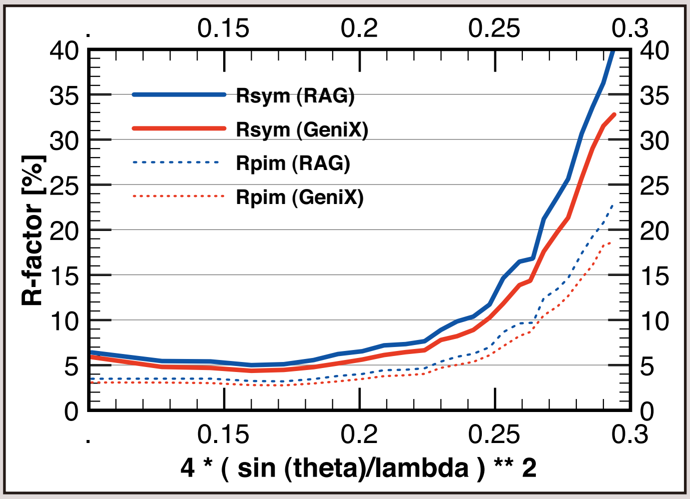
In the plots given below, the RAG data are drawn in blue, the ones from the GeniX generator are drawn in red, respectively. In the intensity plot, the I/σ values (dotted lines) scale to the y-axis on the right hand side of the plot.

lysozyme1	lysozyme2

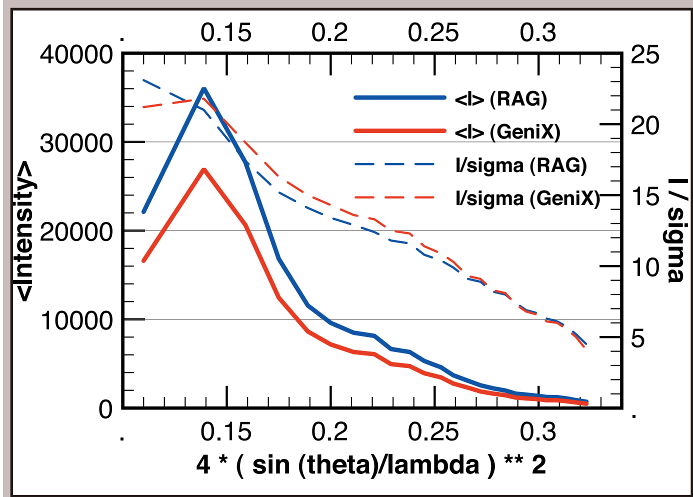
R-factors vs. resolution



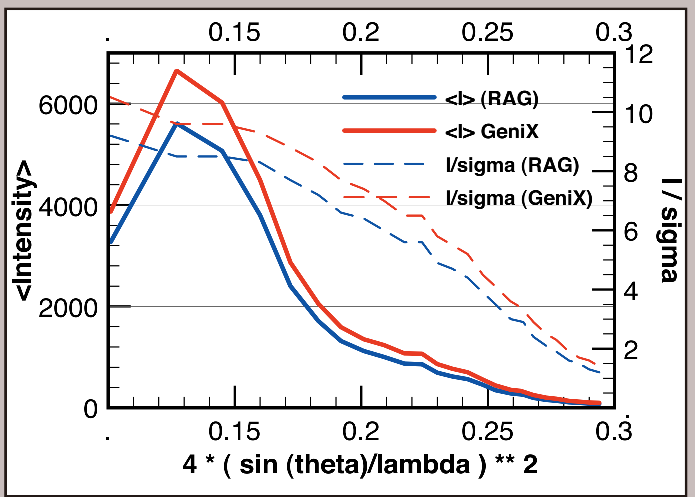
R-factors vs. resolution



Intensities vs. resolution



Intensities vs. resolution



Conclusion

For a large crystal (lysozyme1) the RAG data yield larger net intensity values, yet slightly smaller I/σ-values than the GeniX generator, while the R-factor curves are virtually identical. For a small crystal (lysozyme2) the increased brilliance of the GeniX generator become more obvious. The GeniX generators outperforms the rotating anode generator in all aspects, even by larger net intensities. We have to keep in mind, that the RAG has been operated only at 2 kW instead of the nominal power of 3.0 kW (with the 0.2 x 2 mm focal cup), but from the results we conclude that the GeniX generator compares approximately to a conventional RAG with multi-layer optics running at full power.

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