FAST SPINNER FOR POWDER DIFFRACTION AT THE ESRF

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Abstract

A new generation of fast capillary spinners has been developed at ESRF, for powder diffraction experiments. Two beam lines are using these kinds of spinner, ID11 and ID31.

To improve the statistical powder averaging during the measurement, we maximise the number of powder grains taking part. For this we increase the number of revolutions of the capillary samples, with special fast spinners.

The detector of ID11 allows a data set to be acquired in as little as 5ms to 10ms. To realise one revolution of the sample per acquisition the frequency of the spinner must be 100Hz to 200 Hz. Our fast spinner is able to reach a frequency of 600 Hz. The speed generally used for scanning the detector arm of ID31 is 0.5 to 10 deg/min with a final step of 0.002 deg. Thus frequencies of up to 80 Hz are needed for the fast spinner used at ID31.

For the ID31 beam line we have manufactured two special spinners, an orbital spinner and a spinner with a translation of the sample perpendicular to X beam, useful for samples that are susceptible to radiation damage. The spinners also allow samples to be changed easily, with minimum time lost in aligning the sample on the axis of the diffractometer.

The goal of this paper is to show the design and the characteristics of these fast spinners.

1 Introduction

The performance of the ESRF beamlines adapted for powder diffraction experiments necessitates high-speed spinning of the sample. Sufficiently high speeds cannot be obtained with a conventional device, like the goniometric heads generally used for this type of application.

2 Design

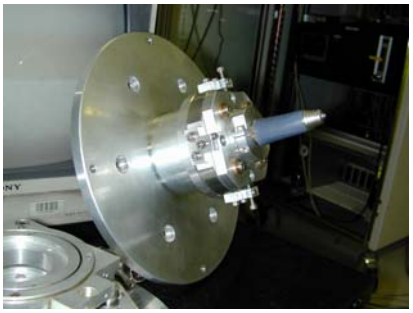
In 2000 we built a fast spinner allowing rotation speeds of up to 200 Hz (12000 rpm). This spinner is based around a commercial dentist's drill, (an ULTIMAT 400 manufactured by NSK, Japan). At the heart of this device is a brushless motor capable of frequencies of up to 600 Hz. The device is delivered with a dedicated electronic control box (picture 1).

The device is mounted on the φ axis of the Kappa diffractometer of the ID11 beamline and a special brass holder allows positioning of the sample in the X-ray beam. (picture 2).

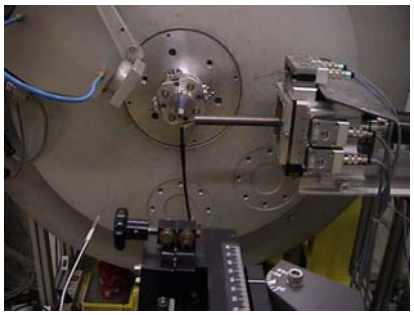
The capillary sample is glued with wax into the cylindrical brass holder, 3 mm in diameter, containing a precision-drilled hole along its axis. An exact correspondance between the external diameter of the capillary and the diameter of the hole ensures the capillary is centred in the holder. A series of holders with holes of 0.4 mm – 1.5 mm has been manufactured, allowing the full range of capillaries commonly used in powder measurements to be mounted. The brass holder rod is locked into the ULTIMAT 400 by means of the device's standard

chuck, designed to take dentistry tools. The same spinner has been used at the ID31 powder diffraction beamline since 2002 (picture 3).
The eccentricity of the rotation axis given by the supplier is less than 9 μm. The ease with which holders can be removed from or inserted into the chuck means that sample changes and positioning of the sample in the X-ray beam takes only a few seconds to perform.

Picture 1: Ultimat with electronic control box



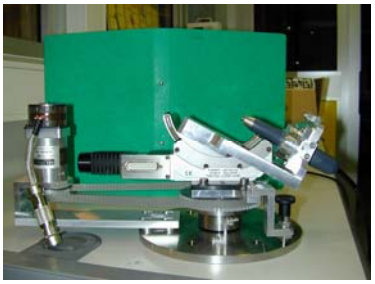
Picture 2: Fast Spinner for ID11



Picture 3: Spinner at ID 31

2.1 Gandolfi style orbital spinner

A Gandolfi-style spinner using an ULTIMAT 500 has been designed for the ID31 beamline. The main axis of the spinner is vertical with an oscillation of ±45˚ about the diffractometer axis (perpendicular to X ray beam), and an oscillation of –30˚ to +45˚ about the X-ray beam. (picture 4)



Picture 4: Gandolfi-style Orbita Spinner for ID31

2.2 Spinner with Translation

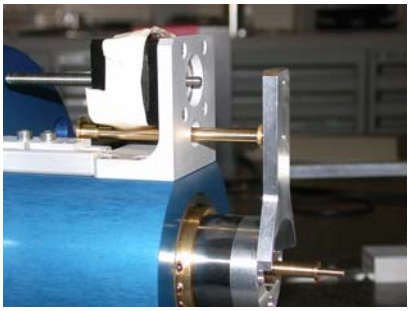
For samples that are susceptible to radiation damage under the very intense incident X-ray beam, (such as certain organic and organometallic substances, as well as proteins), we have designed a special spinner with an additional axial translation, (picture 5, 6, 7).

A stepper motor drives the rotation of the axis of the spinner at up to 166 Hz (10000 rpm). The axial translation is made by a linear actuator “HSI" with a resolution of 3.048 μm per motor step. The range of the translation is 37.5 mm. Working with a 2-mm-wide incident X-ray beam, as many as 18 diffraction patterns can be collected from a full capillary before all the sample is damaged. This greatly improves the efficiency of measurements made on radiation-sensitive samples. The spinner can be combined with a Cryostream nitrogen cooler/heater (80 K – 500 K), mounted co-axially with the sample. The capillary can actually pass safely up inside the nozzle of the cooler. This spinner has been in service at the ID31 beamline since March 2004. The eccentricity of the spinner assembly is less than 4 μm.
Two kinds of sample holder are available for this device.

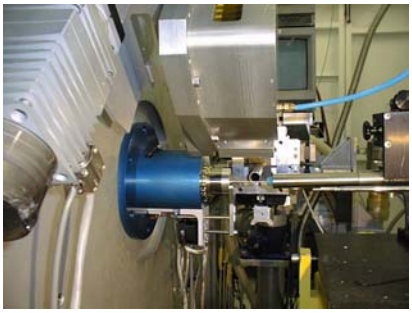
For one spinner we use a SCHAUBLIN grip capable of receiving cylindrical holders from 1mm up to 5mm in diameter. This allows a great variety of sample holders to be accommodated, such as ceramic tubes used to support platinum capillaries used in measurements at very high temperatures.

For a second spinner we have designed a magnetic self-centring coupling for the capillary support (picture 8).

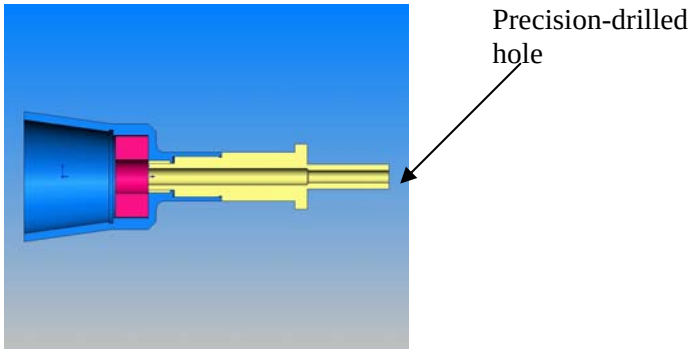
The aim of this device is to use an automatic sample loading system (ASLS) to load and unload the samples. The ASLS is under manufacturing and will be tested at ID31 during the second part of 2004. A cassette of 50 samples will give a large autonomy without user intervention inside the experimental hutch.



Pictures 5, 6: Spinner with axial translation for ID31



Picture 7: Spinner with axial translation mounted at ID31 beamline



Picture 8: Magnetic Fixation for the Capillary Holder

3 CONCLUSION

Since 2000 fast spinners have been used at ESRF for power diffraction experiments.
The different applications of these spinners, (fast spinners, orbital spinners, spinners with axial translation), the ease for loading and positioning the sample, and the overall performances of these devices offer major advantages for the users.