THE NEW SINGLE CRYSTAL DIFFRACTOMETER SC3

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ABSTRACT

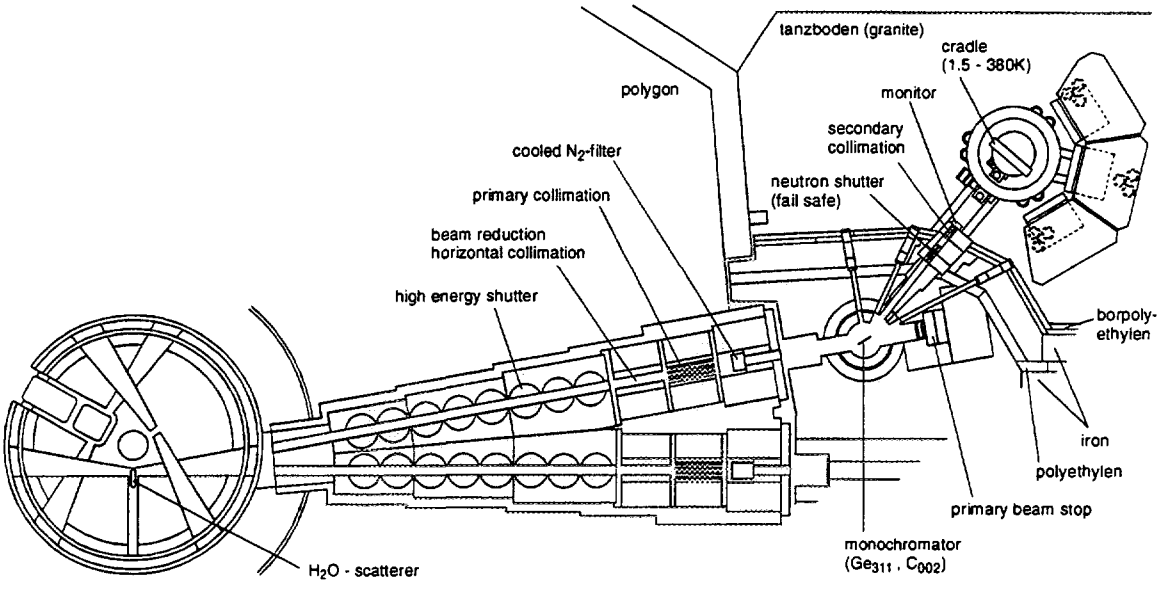
Single crystal diffraction is a powerful method for the determination of precise structure parameters, superlattices, stress. Neutron single crystal diffraction gives additionally to X-rays information on magnetic structures, both commensurate and incommensurate, hydrogen positions, hydrogen bonding behavior and accurate bondlengths, e.g. important in cuprates. The method is therefore especially powerful if combined with X-ray diffraction results. The new instrument at SINQ has been designed for inorganic materials and is positioned at a thermal beam tube, pointing on an water scatterer. This scatterer is presently operating with H_2O at ambient temperature, but a change to another medium at different temperature is possible. The instrument will be equipped with three area detectors, moving at fixed difference in 2Θ. Each detector may be individually moved around a vertical circle (tilting angle γ), allowing to use not only 4-circle geometry in the temperature range from 1.5 to 380K, but also any equipment from a dilution refrigerator (7 mK) to a heavy magnet. A high temperature furnace for 4-circle geometry is foreseen as a future option.

1. Introduction

An ideal four circle diffractometer can be defined by three points: Source, sample position and the detector, defining the diffraction plane normally to be horizontal for neutron diffraction. Four angles are used to position the sample: The angle ω, which is vertical rotation axis, the angle χ defined as the rotation around a perpendicular axis and the angle ϕ, perpendicular to the rotation axis of χ. The last angle used for four circle geometry is the detector angle 2Θ. As this geometry is over determining the system, a fifth pseudo-angle ψ may be defined as the rotation angle around the scattering vector τ. ψ is defined to be zero if τ is measured in bisection mode. Therefore we can measure a reflection *hkl* at any desired ψ, which allows us to avoid shadowed regions, e.g. caused by the massive mechanics of the cradle and allows to perform Renninger-scans [1] to determine anisotropic extinction and absorption. However, if the cradle cannot support the weight of the sample environment or the equipment is not supposed to be tilted such as a very low temperature dilution cryostat, our spectrometer allows out-of-plane measurements by tilting the detectors (angle γ) individually out of plane instead of tilting τ into the plane by the help of the cradle.

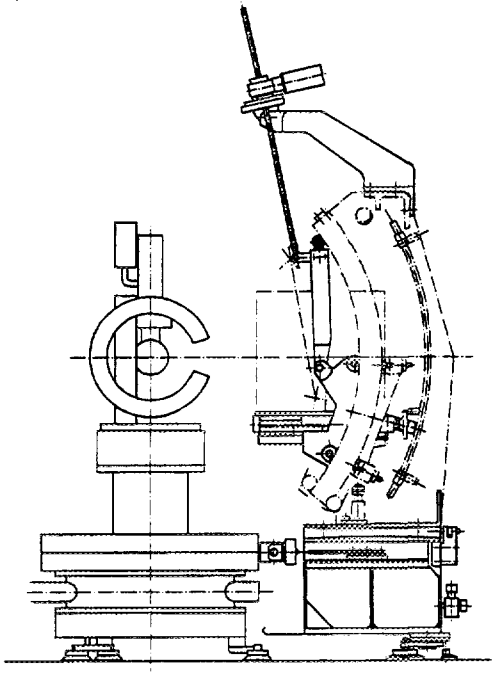
The SC3 gives all the possibilities mentioned above. In addition it is equipped with three area detectors allowing the simultaneous measurement of 10 to 20 reflections, depending on both crystal symmetry and lattice spacing. This is particularly important for medium thermal neutron flux as the case of the continuos neutron source SINQ.

Fig. 1:
Geometry of the four-circle diffractometer showing the situation at beam port 41 pointing onto the H_20 scatterer presently installed in a T-shaped beam tube.



2. Why neutron single crystal diffraction?

Fig. 2:
Vertical cut through one of the detector systems.



As pointed out by the contribution of P. Fischer et al [2] on neutron powder diffraction, neutrons are a versatile tool especially due to the fact, that the neutron is a neutral particle, not interacting strongly with the sample. This avoids surface scattering, giving real information on the whole crystal. Another important fact is, that the scattering potential is located at the very small nucleus, which causes the form factor to be practically constant as a function of $\sin(\Theta)/\lambda$. This is expressed by the following two functions (e.g. [3]):

$$V(\mathbf{r}) = \frac{2\pi\hbar^2}{m} b \cdot \delta(\mathbf{r} - \mathbf{R})$$

$$\langle \mathbf{k}'|V|\mathbf{k}\rangle = \frac{m}{2\pi \cdot \hbar^2} \frac{2\pi \cdot \hbar^2}{m} b \int d\mathbf{r} \cdot \exp(-i\mathbf{k}'\mathbf{r}) \delta(\mathbf{r}) \exp(i\mathbf{k}\mathbf{r}) = b = const,$$

where **k** is the wave vector of the incoming beam, **k'** the outgoing wave vector, **R** the locations of the nucleus, V the scattering potential and b the nuclear scattering amplitude. Therefore, b shows no dependence on the momentum transfer. The absolute value of **k** and **k'** is identical as we are assuming elastic scattering. This is in contrast to X-rays, where the scattering occurs at the electron shells, forcing a fall-off of this value at high scattering angles as the scattering potential may not be described anymore as a δ-function located at the nucleus as done for neutrons. Neutrons yield therefore more accurate information on thermal motions. In addition the neutron has a magnetic moment. As neutron diffraction can be done on our instrument down to 7 mK, this opens a wide field of applications. The advantage of single crystal diffraction to powder diffraction is obvious: Reflections are separated in reciprocal space, allowing a much better and less model-based interpretation of the data.

3. Instrument Layout

The SC3 is built as a good resolution single crystal diffraction instrument, covering the most often asked area between D9 and D19 at ILL. The operating mode is close to D19, the resolution between D9 and D19.

3.1 Specialties of the SC3

The SC3 is equipped with three area detectors of extended size. This allows the simultaneous measurement of 10 to 20 reflections. The number strongly depends on symmetry (lower symmetry = more simultaneous reflections) and size of the unit cell (small unit cell = more simultaneous reflections). Therefore the longer the expected measurement time, the more the instrument compensates by simultaneous measurement of reflections on the detectors. This is essential, as SINQ yields a thermal flux of a medium size reactor, and SINQ is not equipped with a hot source. However, the resolution will limit this compensation to the point where reflections start to overlap.

The second specialty is the tilting possibility, which allows to install almost any equipment on the instrument. However, by tilting the detectors one is leaving the horizontal scattering plane and therefore looses resolution [4]. The instrument has flexible supports for the detectors, allowing to follow expected future development of detectors.

3.2 The monochromator

A focusing composite germanium monochromator (figure 5) using the reflection 311 presently under completion [5] will be installed as the primary monochromator. Anisotropic mosaic (c.f. figure 4) is used to gain additional intensity without loosing resolution. Firstly, we will use a take-off angle $2\Theta_M$ of 40.2° yielding a wavelength of λ=1.15 Å. The second monochromator mounted on the lift system is pyrolitic graphite C_{002}, giving access to λ=2.3 Å at high intensity/low resolution, ideal for many magnetic structure problems and also alignment of the crystals. The germanium Ge_{311} monochromator has a with of 55 mm and a height of 115 mm.

Fig. 4:
Ge_{311}-wafer package No.3, consisting of 24 wafers after 6 bending cycles [6] all flattened with a ceramic plate and glued togehether using tin (measured at T13C, ILL Grenoble, M.Böhm. The figures show the highly anisotropic mosaic useful for diffraction.

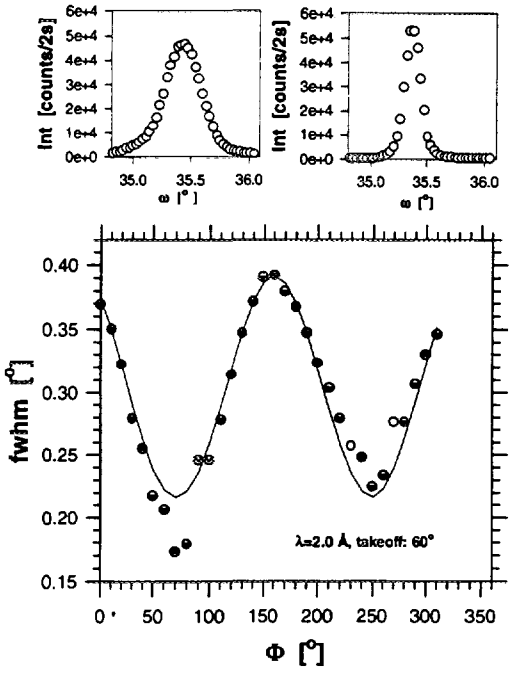
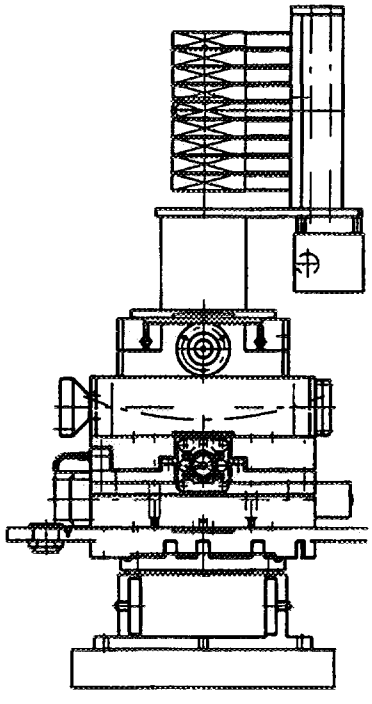
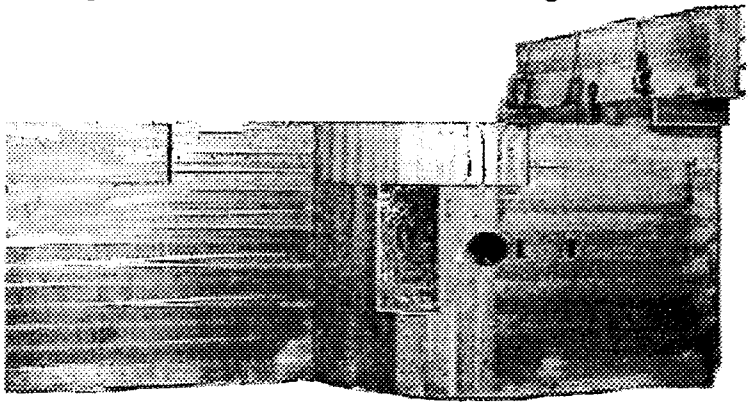


Fig. 5:
Mechanical layout of the focusing monochromator including the tilting, translation and rotation axis.



Possible monochromator take-off-angles are 17.5°, 38.4°±4.0°, 54.6° and 90°. This allows a limited adaptation to future developments of monochromators. A full flexibility has been abandoned due to a lack of experience for shielding high energy neutrons and as shielding calculations are limited in the accuracy. The massive inner part of the shielding is shown in figure 6. It is surrounded by polyethylene, additional steel and borpolyethylen as shown in figure 1.

Fig. 6:
Inner part of the monochromator shielding.



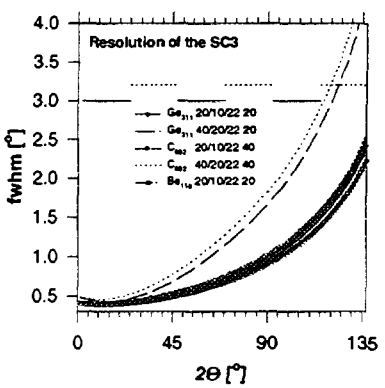
3.3 The detector

We are presently mounting a two dimensional He^3-gas detector [7] based on an extended microstrip plate, similar to the original design of Oed et al. [6]. The detector is using charge division with 2 readouts on front and back side and a energy readout on the front side. This electronic could be replaced in the future by a single-'wire'-readout system. Microstrip detectors are relatively simple in as the stripes are manufactured by photographic methods. Compared to present scintillation plates, gas detectors allow a single event read-out, allowing a better peak-to-background separation.

Table 1:
Parameters of the He^3 based microstrip detector system used for the SC3.

detector size	$203/203mm^2$ (8 by 8 ")	
glass type	Schott S 8900, 0.5 mm thick	
substrate	chromium twice	200nm, sputtered
active area	176/186	mm
anode thickness (front)	12	
number of anodes	190	µm
cathode thickness	500	µm
delay line thickness	40	µm
distance anode/anode	1000	µm
resolution	1.5-2	mm
R anode	≈14	kΩ
R cathode (energy)	40	µm
cathode width (back)	960	
number of cathodes	172	µm
gap	40	µm
resolution	2-2.5	mm
R cathode	≈13	kΩ
^{3}Helium (detection)	4.5	bar
CF_4 (stopping gas)	1.5	pixels
readout:	256x256	max.
Voltage:	1.3-1.5	kV

Fig. 7:
Resolution of the SC3 using different monochromators and different collimations. Beryllium is presently not available. D1-D3 and D1'-D3' show two positions of the detectors covering the full 2Θ-range.



4. Applications

The use of the SC3 is the determination of nuclear and magnetic structures of single and quasi crystals of size >5 mm^3 with unit cells in the range of ≈15-20 Å (depending the symmetry), investigations of structural and magnetic phase transitions, oxygen positions, hydrogen bonding and ordering, quasi-crystals, texture, pole figure-measurements, magnetic and nuclear superstructures, determination of unknown magnetic modulations. The temperature range available is 7 mK to 450 K in the beginning and could be extended up to 2000K. Above 1.5K we can use the Eulerian cradle, below 1.5K and with 'strange' auxiliary equipment, we have to use the (from the point of resolution) less favorable tilting mode.

5. Acknowledgments

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6. References

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