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# Echidna—the new high-resolution powder diffractometer being built at OPAL

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**Abstract**

A new powder diffractometer aiming for high angular, and thus high reciprocal space, resolution is being constructed within the Neutron Beam Instrumentation Project at the upcoming Australian Neutron Source OPAL, near Sydney. The neutron flux at the sample can be expected to be up to $10^7\,\mathrm{n/cm^2/s}$. With an array of 128 position sensitive detectors, each equipped with a 30 cm high Söller collimator of 5 arc min acceptance this instrument will have one of the highest performances of its kind. In addition to classical applications in powder diffraction, the quasi two-dimensional detector will be used for rapid texture measurements, where high separation of peaks is necessary. Even single crystal reciprocal space mapping is envisaged. The article compiles an overview of the design, status of the project and potential research activities.

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## 1. Introduction

Neutron and X-ray powder diffraction techniques are both very well established in modern condensed matter research. It is therefore not surprising that powder diffractometers are amongst the first instruments to be constructed at the new reactor OPAL at ANSTO [1,2] and the Australian Synchrotron [3] being built in Australia. While the beam characteristics from modern synchrotron sources can reach a competitive penetration power [4] and allow for unrivaled resolution [5], rapid time-dependent studies or the investigation of individual grain correlations [6], neutron powder diffraction bears the following advantages:

- The absorption coefficient and the coherent scattering depend on the isotope rather than the atomic number, which can be a tremendous advantage, i.e. for the refinement of light elements in heavy metal oxides, such as perovskites, zeolites, relaxor ferroelectrics to mention but a few.
- In contrast to X-rays, the nuclear form factor for neutron diffraction is constant and therefore exclusively allows to measure at high momentum transfers.
- The neutron has a magnetic moment and is a unique probe for magnetic structure determination.
- Neutron beams are naturally large and penetrating and give a good powder average over many grains. This holds in particular where the sample cannot be spun such as in texture measurements.
- Beam geometries of neutron vs high energy X-rays [4] can be advantageous for neutrons [7]. Large scattering angles between 90° and 180° can be easily obtained. Together with the weak absorption this allows for an isotropic extension of the gauge volume. This advantage can be applied in simpler volume correction for classical powder diffraction and plays an essential role in strain and texture measurements.

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## 2. The new instrument

ECHIDNA, Fig. 1, will be the high-resolution powder diffractometer within ANSTO's initial eight strategic instruments of the Neutron Beam Instrumentation Project, which is a sub-project of the OPAL reactor project [2]. With 20 MW thermal power and an unperturbed thermal flux density of $4\times10^{14}\,\mathrm{n/cm^2/s}$ OPAL feeds a $30\times5\,\mathrm{cm}^2$ wide thermal guide TG1 coated with $m=3$ vertical and $m=2.5$ horizontal supermirror. On TG1, at 49 m from the source, is the pyrolytic graphite or Ge monochromator for WOMBAT, the complementary high-intensity powder diffractometer [8]. The transmitted beam then continues in a guide of equal cross section and $m=2$ supermirror to the ECHIDNA monochromator at 58 m from the source. The far distance from the reactor still delivers >90% of the flux at the reactor face but a much better signal-to-background ratio by a factor of 2–5. This beam is vertically focused down to the sample position which resides on a heavy goniometer. Scattering angles between 80° and 155° can be chosen for the monochromator, allowing a wide range of wavelength, resolution and intensity options. Optionally, 5′ or 10′ primary and 10′ secondary collimators can be inserted in front and after the monochromator, respectively. The radiation scattered from the sample is collected in a set of 128 units consisting of a 5′ collimator in front of a vertically oriented linear position sensitive $^3$He detector tube of 30 cm height. The mean scattering plane is horizontal and the detector units are aligned in an arc of 158.75° around the sample. The detector design is very similar to, and shares many common characteristics of, the recently refurbished D2B powder diffractometer at the ILL [9].

Intensities $>10^6\,\mathrm{n/cm^2/s}$ at high resolution are expected at the sample position. The resolution for ECHIDNA has been estimated by the Cagliotti formalism and the expected FWHMs are plotted in Fig. 2 assuming a Ge 115 monochromator with 0.15° mosaic spread and a take-off angle of 155°. Different cases have been considered as shown in the legend. 60′ is a good estimate for the uncollimated beam and the integrated intensity values as a fraction of the fully uncollimated setup are given in the parentheses. The instrument has been designed for ultimate resolution, which is best with a value of $\Delta d/d=\Delta G/G=4.3\times10^{-4}$ FWHM, i.e. changes in lattice spacings can be measured down to the $10^{-5}$ level and thus reaches the region where it is limited by grain size effects.

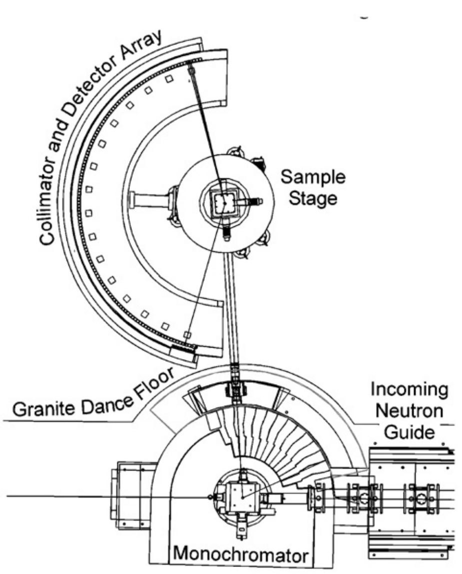

Fig. 1. Layout of the instrument.

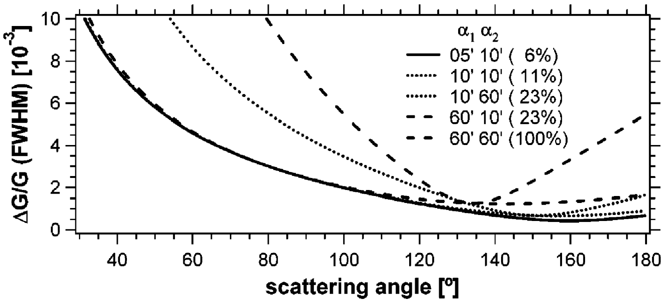

Fig. 2. Resolution of the scattering vector G for different collimations.

Two monochromator assemblies are being constructed for ECHIDNA. The first consists of 23 slabs of plastically deformed, stapled and bonded Germanium wafers [10], each stack 8 mm thick, 12 mm high and about 70 mm broad, tiled vertically on a sagittal focusing device that covers the whole height of the guide. One speciality is the three-dimensional alignment of the crystal slabs with a (1 1 0) direction pointing vertical and a surface normal of (3 3 5). This allows for the selection of any hhl reflection by simple rotation of the crystallographic angle as an offset around the vertical axis. Fig. 3 shows as an example the beam geometries for the (5 5 1) reflection. This design allows for an almost continuous wavelength choice in the range between 0.8 Å and 3.3 Å. The second monochromator assembly is a fixed-curvature Ge-511 device that served the former H1A powder diffractometer at Brookhaven National Laboratory (BNL) [10].

The data acquisition is quasi-2D, i.e. the vertical resolution is given by the detector electronics and will be re-sampled into 32 or 64 channels after correction of position and intensity while the horizontal scattering angle is scanned by a few degree in order to overlap the pattern in one or two detectors. Routines for on-line data presentation are foreseen.

The sample area is large and versatile, allowing for 500–1000 kg load on top of the goniometer. A quasi 4-circle mode will be implemented within the limits of the cross tilt and 2D horizontal translations are foreseen. Optionally, Eulerian cradles, sample changers or sample environments as cryostats, magnets, furnaces, can also be mounted.

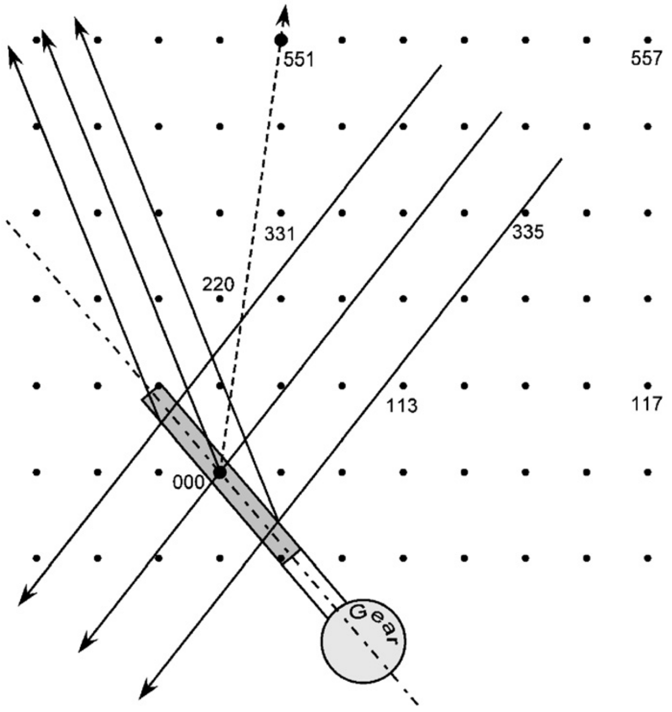

Fig. 3. Beam geometries for the asymmetric 5 5 1 reflection of the monochromator sketched for $2\theta = 120^\circ$. The overlayed reciprocal lattice indicates the direction of the scattering vector which bisects the angle between the incoming and the reflected beam. The [3 3 5] orientation is the surface normal of the gray-shaded crystal slab.

## 3. Applications

The main applications for ECHIDNA will, of course, be in the classical neutron powder diffraction area's of refining cell parameters and materials structures [11]. Perovskites are a good example of this, where the refinement of oxygen atoms in a heavy metal environment needs the benefits of the neutron cross section and the magnetic probe [12]. Here, the line shape and resolution function play a fundamental role in the refinement process and the user can choose the region of interest of the 2D detector scan. The curvature of the Debye–Scherrer cones broadens the resolution off of the horizontal plane and may lead to asymmetric line profiles. Therefore, the peak position should be evaluated from the central region while intensity information may be taken from the entire Debye–Scherrer ring section.

Texture measurements and evaluation routines will also be developed for the new instrument. The large number of detectors and the second dimension will cover a large solid angle in orientation space and thus be very competitive for the acquisition time. High resolution will be needed in industry-related multi-phase systems, such as intermetallic titanium–aluminium compounds or geologically relevant rock samples [13,14]. Textures of minority phases with low intensity may have to be separated from the resolution wings of the majority peaks.

Similarly, the detector covers simultaneously a large area in reciprocal space which makes its use interesting for single crystal reciprocal space mapping, such as investigated in Ref. [15]. Entire reciprocal planes can be scanned in relatively short times allowing whole superstructures to be obtained along with short range order and diffuse scattering. The high angular resolution makes ECHIDNA distinct from our Quasi-Laue-Diffractometer KOALA of the VIVALDI-type [16] and allows for the determination of peak widths and rocking curves.

We also intend to test the instruments performance in some non-traditional areas, such as diffraction from floating crystallites in liquids which potentially leads into investigations of crystallization processes upon cooling and again, makes the instrument valuable for multi-phase intermetallic systems which occur in industry.

## 4. Conclusion

The high-resolution powder diffractometer ECHIDNA will be a state of the art instrument employing the newest neutron scattering technologies. It will become operational in late 2006 and be open for user service early in 2007.

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