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High resolution measurement of Bragg cut-off in beryllium†

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Abstract. Bragg cut-off for $(10\bar{1}0)$ plane of polycrystalline beryllium of various lengths at 300 and 116 K has been measured with an energy resolution of 5 μeV. The natural width of the cut-off is $12 \cdot 5 \pm 1 \cdot 5$ μeV, independent of temperature and length of beryllium and also of physical characteristics and certain metallurgical treatments of the powder. Such blocks of beryllium would be suitable for designing a ΔT-window spectrometer with resolution $\geqslant 20$ μeV. Bragg cut-offs corresponding to (0002) and $(10\bar{1}1)$ planes of beryllium have been separated for the first time. These can also be used for producing additional energy windows in a ΔT-window spectrometer, thus increasing its efficiency.

Keywords. Neutrons; beryllium; Bragg cut-off; high resolution crystal spectrometer.

1. Introduction

A successful implementation of the concept of a ΔT-window spectrometer proposed earlier (Goyal *et al* 1982, 1984) calls for a measurement of the natural width of the Bragg cut-off wavelength in beryllium at different temperatures. The information is also important, independently, since it gives an idea of the particle size and dispersion in lattice spacing due to metallurgical treatment of the sample. The best available measurement in literature is resolution limited (Dilg *et al* 1973) it being 20 μeV at ~ 5220 μeV giving $\Delta E/E = 4 \times 10^{-3}$ $(\Delta\lambda/\lambda = 2 \times 10^{-3})$. This does not give the natural width of the cut-off.

Measurements are reported here with a resolution $\Delta\lambda/\lambda \sim 5 \times 10^{-4}$ which, as will be shown, is far narrower than the natural width.

A neutron spectrometer specially set up for this is described in §2. Details of the sample preparation are described in § 3. The cut-off measurements on several pieces of beryllium at two different temperatures are reported in §4. A discussion and summary are provided in §§5 and 6 respectively.

2. Instrument

Figure 1 shows a schematic layout of the instrument installed at E-13 beam port of the CIRUS reactor. A well-collimated neutron beam from the reactor after passing through a single crystal of bismuth, used to attenuate γ-rays and fast neutrons, falls on the beryllium block whose transmission is to be measured. The transmitted beam is

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† Paper entitled 'ΔT-window spectrometer' will appear in the November issue of Pramana.

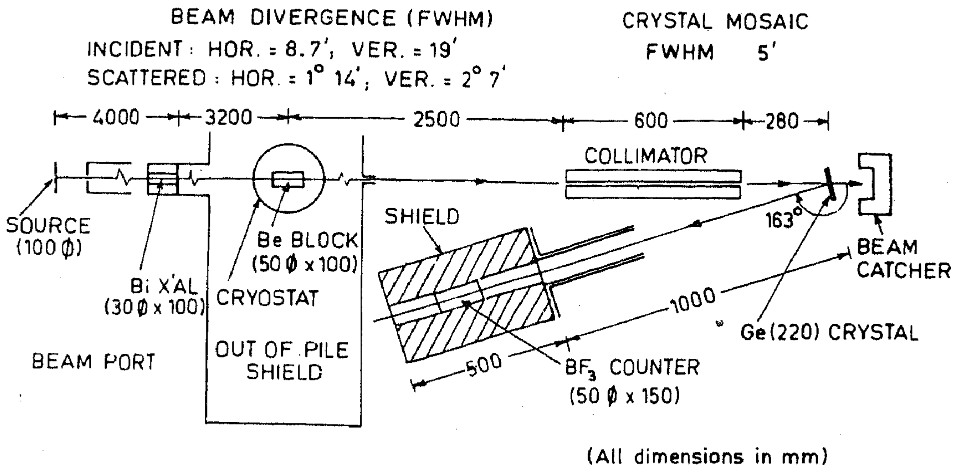

Figure 1. Schematic layout of the single crystal spectrometer used for high resolution Bragg cut-off measurements.

collimated (horizontal divergence α_0) and the energy analysed by (220) planes of a germanium single crystal whose mosaic spread (η) is ~ 5′ (FWHM). A BF$_3$ counter is used for detection. Energy analysis is performed by continuously varying the Bragg angle θ and the scattering angle 2θ in the ratio of 1 : 2. Each increment in 2θ corresponds to 7·5′.

The energy resolution ΔE_R of the spectrometer is given by

$$\Delta E_R = 2E \cot\theta \Delta\theta,$$

$$\Delta\theta = (\alpha_0^2 + \eta^2)^{1/2}.$$

For the energy $E \sim 5220\ \mu\text{eV}$, the Bragg angle for Ge(220) is ~ 81·5° giving ΔE_R(FWHM) ~ 5 μeV for $\alpha_0 = 8{\cdot}7'$ and $\eta = 5'$, and $\Delta E_R/E = 10^{-3}(\Delta\lambda/\lambda) = 5 \times 10^{-4}$). This is a much superior energy resolution than that used in earlier measurements (Egelstaff and Pease 1954; Dilg *et al* 1973) and is sufficient for our purpose.

3. Preparation of beryllium blocks

Beryllium blocks were prepared by vacuum hot pressing powder of −200 mesh size and purity of better than 98 % Be with ~ 1·8 % BeO, and machined to the desired dimensions at the Beryllium Pilot Plant, Vashi. The blocks were examined for blow holes and inclusions by x-ray radiography. The density of the blocks was better than 99 %. One block was also annealed for 1 hr at 800°C and furnace cooled and another, water quenched from 800°C. A block was also prepared from + 200 mesh size powder. The method adopted for making the blocks is expected to give good Bragg cut-off (Egelstaff and Pease 1954).

4. Observations

4.1 *Bragg cut-off measurements*

Bragg cut-off measurements (see also Thaper *et al* 1983) have been made on beryllium blocks having thickness of 50 mm, 100 mm and 150 mm at two different temperatures, *viz* 300 K and 116 K. High resolution data have been obtained for the $(10\bar{1}0)$ Bragg plane on 150 and 100 mm thick blocks using incident beam collimation of 8·7′. Bragg cut-offs corresponding to $(10\bar{1}0)$, (0002) and $(10\bar{1}1)$ planes have also been measured on a 50 mm long block using a poorer incident beam collimation of 20·8′. Direct beam has also been scanned with the same collimation of 20·8′. The energy calibration for these measurements is based on listed parameters of Be ($a = 2{\cdot}2654$ Å, $c = 3{\cdot}5807$ at 291 K) and Ge($a = 5{\cdot}6578$ Å at 298 K) (AIP Handbook 1972).

Figures 2 and 3 show the energy analysis of the cut-off scans of $(10\bar{1}0)$ plane for 150 and 100 mm thickness respectively at two different temperatures obtained with energy resolution, $\Delta E_R \simeq 5$ μeV. As the direct beam is flat in this energy region, the cut-off scans directly render counts proportional to the transmission. However, since the incident beam is not truly monochromatic but has a contribution from higher order neutrons transmission cannot be put in terms of cross-section. In these figures the net intensity below the Bragg cut-off energy is determined by inelastic scattering and absorption cross-sections while above this energy Bragg scattering also contributes to scattering out of the incident beam. Similar measurements were also made on three other blocks of Be *viz* 50 mm block of a quenched, an annealed and a sample made from plus 200 mesh powder.

It can be easily shown that the observed base width of the measured step is 1·15 times the full width at half maximum (FWHM) of the corresponding Bragg peak assuming the latter to have a Gaussian shape. Table 1 gives the observed base widths and corresponding Gaussian widths (uncorrected and corrected for instrumental resolution). It is seen that the measured widths of the cut-offs are much larger than the instrumental resolution of $\simeq 5$ μeV. Further, within the accuracy of the experiments the widths are insensitive to temperature or the length of the filter. The errors quoted in the table are due to the uncertainty in locating the cut-offs. Similar widths were found

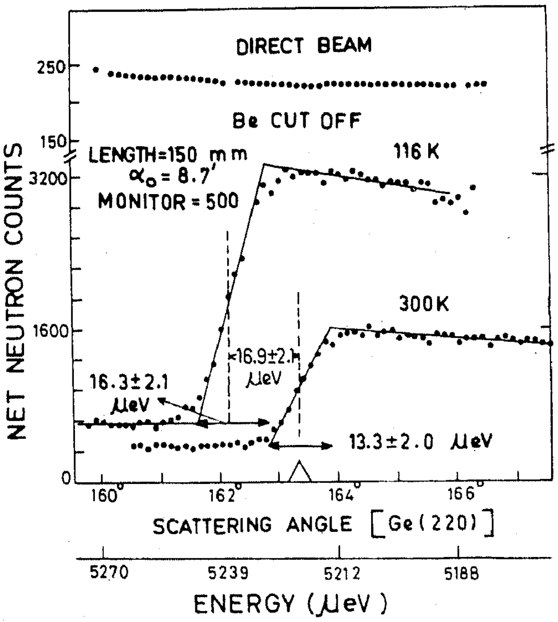

Figure 2. Energy analysis of **a.** direct beam, **b.** beam transmitted through 150 mm Be at 300 K and 116 K.

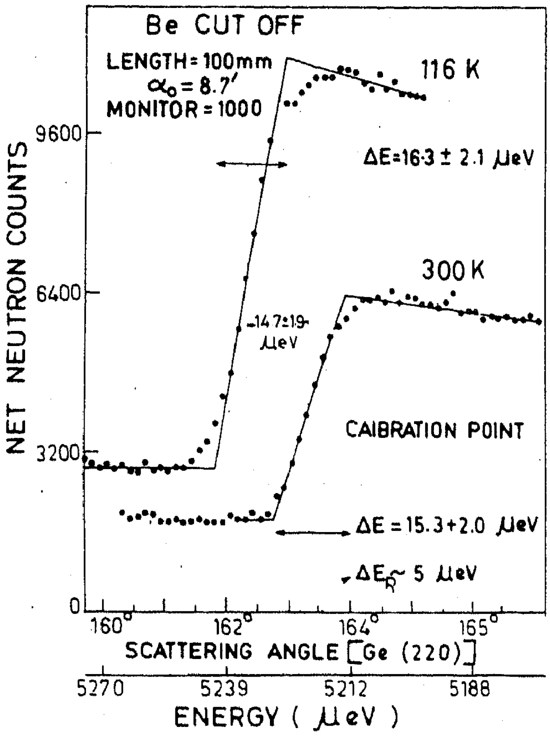

Figure 3. Energy analysis of beam transmitted through 100 mm Be at 300 K and 116 K.

Table 1. Observed and resolution-corrected widths.

Filter		Observed widths		
Length (mm)	Temp (K)	Base width (μeV)	Corresponding Gaussian widths (FWHM) μeV	Corrected Gaussian width (FWHM) μeV
100	300	15·3±2·0	13·3±1·7	12·4±1·7
	116	16·3±2·1	14·2±1·8	13·4±1·8
150	300	13·3±2·0	11·6±1·7	10·6±1·7
	116	16·3±2·1	14·2±1·8	13·4±1·8

for the other three blocks of beryllium; *viz* annealed, quenched and the one made from plus 200 mesh size powder.

Next we observe that the cut-off energy is shifted to a higher energy when the temperature of the block is lowered. The observed shift agrees with the value expected from the change in the lattice parameter of Be (AIP Handbook 1972) as can be seen from table 2. Within the accuracy of the experiment the observed shifts are insensitive to the length of the filter.

Because of the variation of the total cross-section with temperature both below and above the cut-off energy, the intensities are expected to change correspondingly. The observed ratios of intensities at 116 K and 300 K for energies below and above the Bragg cut-off, for the two lengths of the filter, are also given in table 2. The calculated ratio expected from the known cross-section data for Be (Hughes and Harvey 1955) is also shown in the table. The agreement between the two is satisfactory.

Table 2. Observed shifts and intensities.

			Intensity ratios			
Filter length (mm)	Shifts (μeV)		Observed		Calculated	
	Observed	Expected	Below	Above	Below	Above
100	$14{\cdot}7 \pm 1{\cdot}9$	16	1·6	1·7	1·5	1·7
150	$16{\cdot}9 \pm 2{\cdot}1$	16	2·0	1·7	2·1	1·9

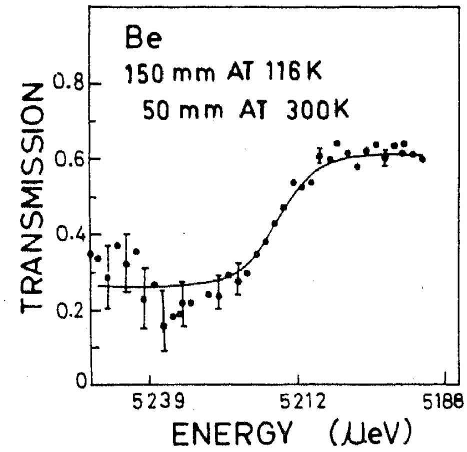

Figure 4. Transmission of 50 mm Be at 300 K as a function of energy as measured with beam transmitted through cooled Be (150 mm long at 116 K).

4.2 *Transmission near Bragg cut-off energy*

A measurement of the transmission corrected for higher order effects near the cut-off energy is of interest since it allows an estimate, though rough, of the reflectivity of polycrystalline beryllium; this quantity being of relevance in the design of the ΔT-window analyser. To eliminate the higher order neutrons the incident beam is first passed through a 150 mm long Be block at 116 K. This provides a 'clean' beam for energies less than $\sim$ 5236 μeV. This beam is now transmitted through a 50 mm block of Be at 300 K which allows neutron below 5220 μeV to pass through and reflects the neutrons between 5236 and 5220 μeV. Figure 4 shows the transmission of the second block of beryllium as a function of energy. The difference between the top and the bottom of the transmission curve can be used to roughly estimate the reflectivity which for 50 mm beryllium at 300 K comes to $32 \pm 10\%$ compared to an estimated value (Goyal *et al* 1984) of 30%.

4.3 *Bragg cut-offs from other planes*

Energy analysis of Bragg steps in transmission corresponding to $(10\bar{1}0)$, (0002) and $(10\bar{1}1)$ planes of Be has been made on a 50 mm long block with poorer incident beam collimation of 20·8′. Figure 5 shows the transmission (arbitrary scale) at 116 K plotted

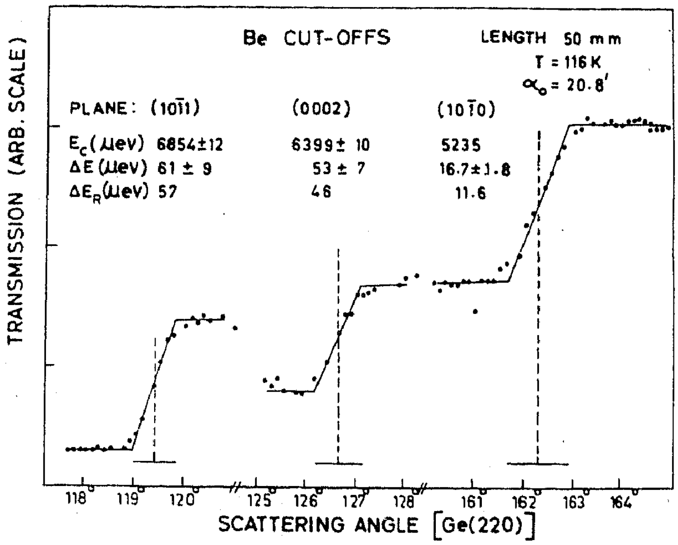

Figure 5. Energy analysis of beam transmitted through 50 mm Be at 116 K for the Bragg planes $(10\bar{1}0)$, (0002) and $(10\bar{1}1)$.

for the same incident beam intensity. Steps corresponding to the three Bragg planes $(10\bar{1}0)$, (0002) and $(10\bar{1}1)$ can be clearly seen. Using the cut-off energy for $(10\bar{1}0)$ planes as, 5235 μeV, the step energies, E_c, corresponding to (0002) and $(10\bar{1}1)$ are observed at 6399 ± 10 and 6854 ± 12 μeV respectively. The observed base widths, ΔE, for the three cut-offs are $16{\cdot}7 \pm 1{\cdot}8$, 53 ± 7 and 61 ± 9 μeV respectively. The estimated energy resolution values, ΔE_R, for the respective cut-off energies for beam collimation of 20·8′ are 11·6, 46 and 57 μeV. The separation between the cut-off planes of (0002) and $(10\bar{1}1)$ has been observed for the first time.

5. Discussion

The observed width of the Bragg cut-off from the $(10\bar{1}0)$ plane of beryllium is much larger than the instrumental resolution for all the blocks and at both the temperatures, as seen from tables 1 and 2 and figures 2 and 3. The widths could arise due to experimental reasons like multiple and thermal diffuse scattering, because of the metallurgical treatment given to the beryllium block like hot pressing, annealing or quenching or because of other physical reasons like the particle size of the starting material etc. It can be argued that the multiple scattering effect is negligible on the width. If the effect of thermal diffuse scattering was appreciable the width would show a narrowing at the lower temperature. This is not observed. Hence, the width has to be attributed as being primarily due to the metallurgical treatment of the sample itself. Since all the blocks (*i.e.* annealed, quenched and the one prepared from a larger particle size) show the same width it is further concluded that the width arises from the method of preparation itself and not from any later treatment or starting mesh size. If it is argued that the width arises solely from the size of the microcrystals then it is possible to evaluate an average microcrystal size to be ~ 1200 Å. (Thaper *et al* 1983). However, it cannot be ruled out that the width has a contribution from dispersion in the lattice spacing across grain boundaries.

In conclusion, 12·5 μeV seems to be the finest width (FWHM) obtainable with beryllium by giving the metallurgical treatments described in the text. The consequence of this for designing a ΔT-window spectrometer is that all the other resolution elements in the spectrometer should be comparable to this and the temperature difference between the two blocks of beryllium should be large enough to be compatible with this.

The measurements of the steps in transmission for (0002) and $(10\bar{1}1)$ are made with poorer resolution. While they are well separated in energy the poorer resolution used does not permit an evaluation of the natural width. However, if they were to be similar to that of $(10\bar{1}0)$ planes then it would be possible to use them also to produce additional energy windows.

6. Summary

Measurements of Bragg cut-off for beryllium of various lengths at 300 and 116 K have been made with an energy resolution of 5 μeV for the first time on a large-angle single crystal spectrometer designed for the purpose. The natural width of the cut-off of $(10\bar{1}0)$ Bragg plane is $12{\cdot}5 \pm 1{\cdot}5$ μeV, independent of temperature between 116 and 300 K. Such blocks of beryllium would be suitable for designing a ΔT-window spectrometer with energy resolution $\geqslant 20$ μeV.

Steps in transmission, separated by 455 μeV, have also been measured for (0002) and $(10\bar{1}1)$, for the first time. They can also be utilised for producing additional energy windows in a ΔT-window spectrometer, thus increasing its efficiency.

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