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Neutron Transmission of Single-Crystal Sapphire Filters

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Abstract

A simple additive formula is given that permits the calculation of the nuclear capture, thermal diffuse and Bragg scattering cross-sections as a function of sapphire temperature and crystal parameters. We have developed a computer program that allows calculations of the thermal neutron transmission for the sapphire rhombohedral structure and its equivalent trigonal structure. The calculated total cross-section values and effective attenuation coefficient for mono-crystalline sapphire at different temperatures are compared with measured values. Overall agreement is indicated between the formula fits and experimental data. We discuss the use of sapphire single-crystal as a thermal neutron filter in terms of the optimum crystal thickness, mosaic spread, temperature, cutting plane and tuning for efficient transmission of thermal-reactor neutrons.

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Key words: Neutron transmission, Sapphire, Neutron filters.

1. Introduction

The use of large, perfect single-crystals of various materials as filters for thermal neutron beams has long been known [1]. Several materials such as quartz (SiO_2) [2], bismuth [3], silicon [4], lead [5] and sapphire (Al_2O_3) [6-9] have been suggested as most successful filter materials. At high neutron energies, greater than about 1eV, the total neutron cross section σ_t of each of the above mentioned materials is in the range of a few barns, but at lower thermal energies, less than 0.1eV, where much of the coherent Bragg scattering is disallowed the effective cross-section for single-crystal specimens is much reduced. That is also due to the decrease of the thermal diffuse (TDS) or inelastic scattering cross-section with the decrease of neutron energy. "Freund [10] has reviewed a variety of single-crystal filter materials and calculated the total neutron cross-sections from transmission measurements. He uses a simple model to determine the single-phonon, multiple-phonon and absorption cross-sections as functions of the neutron wavelength and fits the data to a general formula with two adjustable parameters. There is good agreement between the calculated neutron cross-sections and the experimentally determined cross-sections for several materials, e.g. single-crystal silicon at both 300 and 77 K. There are, however, substantial differences between his results for sapphire and the experimental results of Nieman et al. [6], especially at neutron energies >100 meV. Freund [10] suggests that improvements in the quality of sapphire single-crystals will reduce these differences and thereby decrease the problems associated with elastic scattering of the higher-energy thermal neutrons. Born et al. [11] have examined the intensity transmitted by a single-crystal of Al_2O_3 over a range of orientation angles within ±2° and have observed no significant changes. They have found that a 90 mm-long sapphire-crystal filter has a transmission of about 0.8 for wavelengths in the range 0.12-0.24 nm and 0.07 for epithermal neutrons. These transmission results are in good agreement with those obtained by Nieman et al. [6], who suggest that the effective attenuation coefficient can be minimized by fine-tuning the crystal orientation. Born et al. [11] have found that there is no need to tune the crystals for

every wavelength. They find that the beam intensity transmitted by their crystals does not depend on the crystal orientation." reported by Mildner et al.[7] .

The wavelength dependence of the transmission probability of a beam of neutrons through super optical quality single-crystal sapphire at room temperature, has been measured by Mildner et al. [7]. using the time-of-flight (TOF) spectrometer installed on C3 beam line at the Intense Pulsed Neutron Source (IPNS) at Argonne Nationai Laboratory. The measurements extend over the neutron wavelength range 0.05-1.2 nm. Sharp dips in the transmission caused by Bragg reflection are not found. The data below the broad dip around 0.2 nm in the cross-section have been fitted to a function that accounts for multi-phonon scattering. Measurements performed on single-crystals with increased lattice distortion (or mosaic spread) showed an increase in the cross-section at long wavelengths. Mildner et al. [7] showed that cross-section data measured for sapphire single-crystal along the c-axis can be fitted to a function given by Freund [10] that accounts for multi-phonon scattering, and showed that this can be further generalized for an anharmonic effects. Their data have been fitted to a function with four parameters. However later measurements Mildner and Lamaze [8] show lower grade crystals perform equally well as optimum grade crystals- contrary- to previous results. Mildner and Lamaze [8] using TOF spectrometer installed on C1 beam line at IPNS have shown that the thermal and cold neutron transmission through high-grade single-crystal sapphire does not degrade after irradiation within a beam port in a fluence of $2x10^{17}$ cm^{-2} fast neutrons and $2x10^{9}$ R gammas from a reactor. They have found that these irradiated crystals have neutron transmission properties similar to those of virgin crystals.

Recently, Mook and Hamilton [9] have measured the neutron transmission through high quality sapphire single-crystal at different temperatures. Their total cross-sections were measured at the Oak Ridge Linear Electron Accelerator (ORELA). They report that the neutron transmission through crystals along the a-axis is considerably better than along the c-axis, when it used as a thermal neutron filter.

The present work concerns a detailed study of the attenuation of thermal neutron beams through a large sapphire single-crystal. The optimum orientation, transmission direction, mosaic spread and the effect of tuning for the efficient transmission of thermal reactor neutrons are also given.

2. Theoretical Treatments

The total cross-section determining the attenuation of neutrons by a crystalline solid is given by

$$\sigma = \sigma_{abs} + \sigma_{tds} + \sigma_{Bragg} \qquad (1)$$

where σ_{abs} the absorption cross-section due to nuclear capture processes, σ_{tds} is the thermal diffuse scattering (*TDS*) or inelastic scattering cross-section and σ_{Bragg} corresponds to elastic or Bragg scattering. The first contribution σ_{abs} for sapphire is simply proportional to the neutron wavelength λ and energy in the range $10^{-4} < E < 10$ eV. Thus, σ_{abs} can be written as $\sigma_{abs} = C_1 E^{-1/2}$ with C_1 a constant which can be calculated from values provided by Sears [12].

As shown by Freund [10], the second contribution σ_{tds} can be split in two parts, σ_{mph} (multiple phonon) and σ_{sph} (single phonon), depending on neutron energy

$$\sigma_{tds} = [A/(A+1)]^2 \sigma_{bat}\left[1 - e^{-WC_2E}\right] + E^{-1/2}\left[C_1 + \frac{\theta_D^{1/2}\sigma_{bat}}{36A}\begin{cases} R.....X \le 6 \\ 3.3X^{-7/2}...X > 6 \end{cases}\right] \qquad (2)$$

where e^{-W} is the Debye-Waller factor [10], C_2 is a constant which is dependent on the scattering material and given by equation $C_2 = 4.27\exp[A/61]$ Freund [10] , $X = \theta_D / T$ (T is the sample temperature), σ_{bat} is the sum of coherent and incoherent scattering cross-sections

of the bound atom), A in case of compounds is the average atomic mass number, and the series R is given by $R=\sum_{n=0}^{22} B_n X^{n-1}/\left[n!(n+5/2)\right]$, with B_n being the Bernoulli numbers [13].

The single phonon scattering cross-section, concerns the energy range $E<<k_B\theta_D$, where k_B is Boltzmann's constant and θ_D is the Debye temperature characteristic of the material. It is determined by phonon annihilation processes. The second part of *TDS* is predominant in the range $E\geq k_BT$ where also down scattering and multi-phonon processes occur.

However, using the static incoherent approximation, Cassels [14] has estimated the short-wavelength elastic cross-section, which is extinct for perfect single crystals. Hence the multi-phonon scattering cross-section in the range $E>>k_B\theta$, given by the first term of Eq.(2), can be replaced by:

$$\sigma_{mph}=\sigma_{free}\left\{1-\left(\lambda^2/2W\right)\left[1-\exp\left(-2W/\lambda^2\right)\right]\right. \tag{3}$$

The contribution of Bragg scattering σ_{Bragg} to the total attenuation arises from coherent elastic scattering due to reflections from different (hkl) planes. In the case of mono-crystalline material, the Bragg scattering cross-section is given by:

$$\sigma_{Bragg}=\frac{1}{Nt_0}\ln\left(\frac{1}{\prod_{hkl}\left(1-P_{hkl}^{0}\right)}\right) \tag{4}$$

where N is the atom number density , t_0 is the thickness of the crystal in the beam direction , and P^{θ}_{hkl} is the reflecting power of the (hkl) plane inclined by an angle θ_{hkl} to the incident beam direction.

As shown by Naguib and Adib [15], the reflecting power P^{θ}_{hkl} for an imperfect single crystal depends upon its mosaic spread, the direction cosine of the incident beam γ_0 relative to the inward normal to the crystal surface cutting along the (h_c k_c l_c) plane, the direction cosine of the diffracted beam γ_{hkl} and the inclination of the (hkl) plane to the crystal surface α_{hkl}. For simplicity, P^{θ}_{hkl} for sapphire - rhombohedral structure was deduced from its equivalent trigonal structure with unit cell parameter a_o=0.4759 nm and c_o = 1.299 nm. Usually sapphire single-crystals are cut along either the c-axis i.e. along (001) plane or the a-axis (100).

Therefore, following Naguib and Adib [16], if the angle between the neutron beam direction and the direction of the cutting plane is ψ, then the direction cosine of the diffracted beam γ_{hkl} for cutting along (001) plane can be expressed as:

$$\gamma_{hkl} = d_{hkl}\left\{\frac{l}{c_0}\cos\Psi + \left[\frac{2}{\sqrt{3}a_0}\left(\frac{h}{2}+k\right)\right]\sin\Psi\right\} \tag{5}$$

and the inclination α_{hkl} angle can be described by:

$$\cos\alpha_{hkl} = d_{hkl}/c_0$$

While in case of the cutting plane (100)

$$\gamma_{hkl} = d_{hkl}\left\{\sqrt{\frac{4}{3a_0^2}\left(h+\frac{k}{2}\right)}\cos\Psi + \left[\frac{2}{3a_0c_0}\left(h+\frac{k}{2}-1\right)\right]\sin\Psi\right\} \tag{6}$$

and

$$\cos\alpha_{hkl} = \sqrt{\frac{4}{3a_0^2}} d_{hkl}\left(h + \frac{k}{2}\right),$$

where

$$d_{hkl} = \frac{1}{\sqrt{\frac{4}{3a_0^2}\left(h^2 + k^2 + hk\right) + \frac{l^2}{c_0^2}}} \tag{7}$$

A computer code SAP (Sapphire) has been developed in order to calculate the total cross-section and transmission of neutrons of energy range from 10^{-4} to 10 eV incident on sapphire mono-crystals. The SAP code is an adapted version of the ISCANF [15] and ISCANF- I [16] codes. The adapted version can additionally provide the following calculations:

1. The σ_{TDS} term using both Freund's [10] and Cassels's [14] formula each in its given energy range .

2. The reflecting power P_{hkl}^{0} due to reflections from (hkl) planes and their higher orders having non-zero crystallographic Q-value of a trigonal structure which is equivalent to the sapphire rhombohederal structure with unit cell having 4 Al atoms and 10 Oxygen atoms .

3. For comparison of experimental neutron transmission data with the calculated values, the program takes into consideration the effects of both neutron wavelength resolution and incident neutron beam divergence.

3. Results and Discussions

The formula given by Eq.(1) was calculated and compared with the experimental results for mono-crystalline sapphire. The main physical parameters required in the calculation are listed in Table (1), where the crystallographic specifications of α-Al_2O_3 (sapphire) are taken from Ref.[17, 18].

Table (1): Physical parameters of sapphire

Average at. Wt.	20.4
Density	3.965 gm/cm^3
Crystal structure	Rhombohedral (n=2), or Trigonal (n=6)
Lattice parameters	Rhombohedral: a_o=0.5128nm=b=c; and α=55.28°=β=γ
	Trigonal: a_o=0.4759nm and c_o = 1.299nm
Atomic positions	Al: w, w, w; W, W, W; 1/2+w, 1/2+w, 1/2+w;
(2 atoms/unit cell)	1/2-w, 1/2-w, 1/2-w; and w=0.352
	O: u, 1/2-u, 1/4; 1/2-u, 1/4, u; 1/4, u, 1/2-u; $\bar{u}$, 1/2+u, -1/4;
	1/2+u, -1/4, $\bar{u}$; -1/4, $\bar{u}$, 1/2+u; and u = 0.556
Coherent scattering length	$b_{Aluminum}$= 3.449 fm, b_{Oxygen}= 5.805 fm
Absorption cross-section (σ_a)	0.231 barn at λ=0.1798 nm
Bound atom scattering cross-section (σ_{bat})	15.7 barn
Debye temperature θ_D	1040 K
Melting point	2040°C

The total cross-section data reported by Nieman et al. [6] at both room and liquid N_2 temperatures, are displayed in Fig.(1) as closed and opened squares, respectively. The filter transmission measurements were made with the aid of two triple-axis crystal spectrometers, at facilities E-13 and L-3 located at the NRX and NRU reactors respectively, at Chalk River [6].

The calculated values, using the SAP code are displayed as solid lines, assuming that a perfect mono-crystalline sapphire and the values of C_2=4.6 Å^{-2}eV^{-1} and θ_D=1040 K given by Freund [10], are used. The calculated values are in reasonable agreement with the measured ones, for neutron energies less than 0.05 eV and with greater disagreement observed for higher energies. As shown in Fig.(1), the inclusion of only the multi-phonon term given by

Cassels [14] at $E >> k_B\theta_D$ improves significantly the agreement at $E > 0.05$ eV. Therefore, the calculations presented below were carried out using the formula given by Freund [10] for $E \leq 0.1$ eV, and the multiphonon Cassels formula for $E > 0.1$ eV. Moreover, the calculation of neutron cross-section take into account the Bragg scattering term given in Eq.(4).

We have also calculated the neutron transmission as a function of wavelength, for different thicknesses of 27, 54 and 81 mm of super optical quality sapphire single-crystals with [001] axis parallel to the incoming neutron beam at room temperature. The calculation was carried out within steps of λ=0.001 nm in the wavelength band from 0.001 nm up to 1.0 nm . The wavelength spread $\Delta\lambda$ was selected to be 0.004 nm, which is equal to that reported by Mildner et al [7] .The result of these calculations are displayed in Fig.(2) along with the experimental data reported by Mildner et al. [7]. The agreement obtained between the calculations and the experimental data supports the application of additive cross-section formulae and the SAP code.

Fig.(3) shows the neutron transmission reported by Mook and Hamilton [9] for a sapphire crystal for the a-axis direction over a large energy range. Also displayed as solid lines, are the calculated values assuming an 8 cm thick sapphire single crystal cut along (100) plane (i.e. along the a-axis) and with a FWHM mosaic spread of 1min of arc (FWHM = 2.354 standard deviation of mosaic spread). The agreement is reasonable within the range of fluctuations of the experimental values. However, a slight disagreement is obtained for, the c-axis neutron transmission of sapphire at different temperatures as a function of neutron energy.

From these data Mook and Hamilton have provided plots of the temperature dependence of the transmission for the two directions. These are displayed in Fig.(4), where the points are the mean values deduced from the measured neutron transmission between

10 and 50 meV shown in Fig.(3), at each temperature. Also displayed as solid lines, are the calculated mean values for crystal mosaic spreads of 1' and 5' respectively . As may be observed, the agreement is reasonable for the a-axis data when the FWHM of the mosaic spread of the sapphire single crystal is between 1' and 5' of arc. For the c-axis data the experimental transmission values decrease more rapidly with increasing temperature than both of the calculated values and a-axis data. Such discrepancy is difficult to explain, it may be due to some impurities or defects in the used crystals. Moreover the multiphonon scattering cross-section calculated using the static incoherent approximation, Cassels [14] and even inclusion of an anharmonic term, Mildner et al [7] can not explain such temperature dependence of the cross-section as a function of crystal orientation at the same crystal thickness.

The (hkl) planes, their corresponding wavelength λ and structure factors F_{hkl} for the Bragg reflections expected for sapphire single crystal when a collimated white neutron beam is incident in the [001] direction with angle of inclination θ_{hkl} were calculated and listed in Table 2. Also for comparison the corresponding values calculated by Mildner et al [7] are listed . The agreement between them is evidence.

Table2: Calculated positions of *hkl* reflections along c-axis

Hkl	M	λ_{hkl}-nm		θ_{hkl}-Rad		$\left\|F_{hkl}^2\right\|^2.10^{30}\ m^2$
		Present	Mildner[7]	Present	Mildner[7]	
006	2	0.4330	0.4331	0.498π	0.5π	570.8
104	6	0.4007	0.4006	0.2875π	0.288π	64.31
012	6	0.3729	0.3727	0180π	0.180π	51.13
018	6	0.2811	0.2811	0.380π	0.381π	81.30
116	12	0.2369	0.2368	0.265π	0.265π	706.4
1,0,10	6	0.2363	0.2363	0.403π	0.403π	284.8
0,0,12	2	0.2165	0.2165	0.498π	0.5π	1263.0
119	12	0.2110	0.2110	0.327π	0.326π	785.6
113	12	0.2009	0.2009	0.160π	0.160π	730.4
208	6	0.2004	0.2003	0.287π	0.288π	0.185
024	6	0.1864	0.1863	0.180π	0.180π	284.0
0,2,10	6	0.1859	0.1859	0.321π	0.320π	54.18
1,1,12	12	0.1794	0.1794	0.364π	0.364π	187.6
1,1,14	6	0.1766	0.1766	0.429π	0.430π	71.0
128	12	0.1556	---------	0.243π	-------	7.1

Similar calculations where carried out and listed in Table 3, when the neutron beam is incident in the [100]. Where the multiplicity factor M is the number of [hkl] planes having the same λ_{hkl} and p^{θ}_{hkl}. Therefore the resulting neutron transmission from them $T_{Bragg} = (1 - p^{\theta}_{hkl})^{M}$. From Tables 2 & 3,it is apparent that the number of reflecting planes in [001] direction in the wavelength band from 0.15 nm up to 0.45 nm is less than that of [100] direction , however their multiplicity factors are almost greater than the later.

Table3: Calculated positions of *hkl* reflections along a-axis

hkl	*M*	λ_{hkl}-nm	θ_{hkl}Rad	$F^2_{hkl}.10^{30}\ m^2$	*hkl*	*M*	λ_{hkl}-nm	θ_{hkl}Rad	$F^2_{hkl}.10^{30}\ m^2$
$\bar{1}02$	2	0.5875	1.005	57.05	324	4	0.1600	1.078	409.0
110	4	0.4120	1.047	7.94	$\bar{1}14$	4	0.1579	0.3146	56.44
202	2	0.3745	1.264	4.30	208	2	0.1579	0.6674	0.174
104	2	0.3158	0.667	63.3	226	8	0.1583	0.8618	597.8
113	8	0.3167	0.8618	825.2	$\bar{1}14$	4	0.1579	0.3146	56.16
012	4	0.2935	0.4359	55.15	$13\bar{2}$	4	0.1532	0.7517	120.9
$\bar{2}04$	2	0.2935	1.005	303.1	$32\bar{5}$	4	0.1532	1.040	103.0
211	4	0.2905	1.217	79.05	416	8	0.1506	1.136	490.5
$21\bar{2}$	4	0.2785	1.165	1.46	$23\bar{1}$	4	0.1510	0.9287	102.5
214	4	0.2395	1.020	195.3	318	4	0.1487	0.917	154.9
$12\bar{1}$	4	0.2320	0.8489	76.97	024	4	0.1469	0.4359	263.7
122	4	0.2230	0.8558	1.42	$\bar{4}08$	2	0.1469	1.005	367.2
$31\bar{1}$	4	0.2200	1.313	36.25	511	4	0.1458	1.405	288.6
$\bar{3}15$	4	0.21625	0.9449	79.07	$23\bar{4}$	4	0.1400	0.8803	390.9
312	4	0.216	1.273	131.6	$42\bar{4}$	4	0.1392	0.7129	131.6
220	4	0.20575	1.047	2.99	030	4	0.1374	0.5236	1696
$31\bar{4}$	4	0.1975	1.157	12.6	330	4	0.1375	1.047	1305
306	4	0.1960	1.005	402.3	425	4	0.1352	1.132	155
223	8	0.1916	0.9885	628.10	$51\bar{5}$	4	0.1353	1.253	279.9
$\bar{3}24$	4	0.1915	0.7499	187.9	4,0,10	2	0.1262	0.9003	665
116	8	0.1867	0.6222	675.3	523	8	0.1240	1.253	269.8
404	2	0.1879	1.264	11.92	229	8	0.1225	0.7319	536.1
$\bar{2}22$	4	0.1870	0.4968	3.94	336	8	0.1211	0.9456	182.5
410	4	0.1765	1.381	0.206	143	8	0.1129	0.6958	438.3
217	4	0.1727	0.809	72.45	119	8	0.1112	0.4658	620.4
125	4	0.1732	0.7054	72.45	3,0,12	4	0.1058	0.6674	812.6
321	4	0.1726	1.156	106.8	7,0,10	2	0.0977	1.145	1054
502	2	0.1622	1.445	44.57	6,0,12	4	1.0097	1.005	437.9

Consequently, T_{Bragg} depends not only on the mosaic spread value of the sapphire single crystal but also on its orientation with respect to the incident neutron beam direction.

To decrease the fluctuations in the transmission, which are due to Bragg reflections, an optimum choice of the crystal mosaic spread is essential. Neutron transmission in the a-axis direction through 8 cm thick sapphire crystal, cooled to the liquid N_2 temperature for different values of mosaic spread were calculated and displayed in Fig.(5). For comparison, Fig.(6) shows similar calculations for the transmission in the c-axis direction for a 8 cm thick sapphire crystal under the same conditions .One can observe, that the Bragg dips in the neutron transmission through high-quality sapphire single crystal (FWHM) ≈ 1') are drastically reduced.. Such crystals are expensive to produce. " The γ-ray diffraction measurements carried out by Born et al [11] indicate the crystalline perfection and homogeneity of the high-quality sapphire crystals that are now available. The mosaic spreads (5-15") are very much lower than typical angular divergences for thermal-neutron beam tubes". Mildner et al [7]. It is obvious that the neutron transmission through 8 cm sapphire single crystal with mosaic spread of 5' along the a-axis is less disturbed by parasitic Bragg reflections than along the c-axis. Since the strong reflections from (119) and (113) planes disturb the neutron transmission along the c-axis $E \cong 0.02$ eV . As can be also observed parasitic Bragg reflections can limit the use of sapphire as a thermal neutron filter for mosaic spreads > 5'.

Therefore high quality sapphire single crystals along the a-axis is a better choice than that along the c-axis. Such conclusion is in agreement with the experimental results reported by Mook et al [9].

To find the optimum sapphire thickness, the neutron transmission through different crystal thickness, were calculated. Fig. 7a shows the result of calculation through a sapphire (100) crystal having mosaic spread of 5' while Fig.7b shows the situation through sapphire (001). It would appear that a 7.5 cm thick sapphire (100) is sufficient for removing neutrons with energies > 1 eV (T_n < 8%) while providing high transmission (T_n > 85%) for neutron energies < 0.02 eV . The fluctuations in the transmission curve which are due to Bragg reflections are less than 5%. These fluctuations can be smoothed by increasing the incident neutron beam divergence.

Fig. 8 shows that 7 cm thick sapphire (100) crystal can be successfully used to transmit a thermal reactor flux having a Maxwellian distribution with neutron gas temperature close to 300 K , while significantly rejecting the accompanying slowing down flux (dE/E) ;with neutron energy E > 1 eV. Fig.8 also shows that there is a small increase ≈ 5% at neutron energies < 0.02 eV in the neutron transmission through the cooled sapphire crystal at LN_2 temperature. Such small improvement as reported by Mildner et al [7] & [8] for many applications, this may be insufficient to warrant the expense and inconvenience of cryogenics.

Nieman et al [6] reported that low quality sapphire can be considerably more efficient thermal neutron filter than either silicon or quartz. For full realization of sapphire's potential., the user must prepared to fine tune the crystal orientation to minimize the attenuation coefficient for each energy of interest.

To demonstrate the effect of tuning on the neutron transmission through sapphire crystal , the calculation were carried out for sapphire single crystal at room temperature (having a mosaic spread of 1°) as a function of neutron energy for different angles ψ (for ψ = 0 , ±5° and ±10°) between the neutron beam direction and that of the major axis. The result of the calculations are displayed in Figs.(9) and (10) for the a-axis and c-axis directions, respectively. Such calculation shows that one may use a low quality sapphire single-crystal as a sufficient thermal neutron filter. Moreover, it is obvious that the tuning is sufficient within ±10° and that the transmission along the c-axis is preferable to the a-axis direction, since it is less disturbed by huge number of parasitic Bragg reflections.

4. Conclusion

We have calculated the total neutron cross-section and effective attenuation coefficient for sapphire single crystals with rhombohedral structure with an accuracy sufficient for determining the neutron transmission characteristics. Calculation shows that 7.5 cm thick sapphire single crystal, cut along a-axis and with a FWHM on a mosaic spread of 5 min of arc, is a good thermal neutron filter. A crystal cut along c-axis with a mosaic spread of

about 1° is also an efficient thermal neutron filter, when it is fine-tuned to minimize the attenuation for each neutron energy of interest.

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