

## Design and development of position sensitive detectors for neutron scattering instruments at National Facility for Neutron Beam Research in India

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**Abstract.** Various neutron scattering instruments at Dhruva reactor, BARC, are equipped with indigenously developed neutron detectors. Range of detectors includes proportional counters, beam monitors and linear position sensitive detectors (PSD). One of the instruments is recently upgraded with multi-PSD system of high efficiency and high resolution PSDs arranged in stacking geometry. These efforts have resulted in improving the throughput of the instrument and reducing experiment time. Global scarcity of  $^3\text{He}$  has made essential to explore other options like  $\text{BF}_3$  gas and  $^{10}\text{B}$  coatings.

PSDs with coaxial geometry using  $\text{BF}_3$  gas and  $^{10}\text{B}$  coating (90% enriched) are fabricated and characterized successfully. These PSDs are used as the alternative to  $^3\text{He}$  PSD in equivalent geometry. Though efficiency of PSDs in similar dimensions is lower than that with  $^3\text{He}$ , these large numbers of PSDs can be arranged in multi-PSD system. The PSD design is optimized for reasonable efficiency. An array of 60  $\text{BF}_3$  filled PSDs (1 m long) is under development for the Time of Flight Instrument at Dhruva. Further improvement in efficiency can be obtained with novel designs with complex anode-cathode geometry. Various challenges arise for long term operation of PSDs with  $\text{BF}_3$  gas, in addition to complexity of data acquisition electronics. Study of gas aging with detector fabrication materials has been carried out. PSDs with  $^{10}\text{B}$  coating show advantage of non toxic nature but have low efficiency. Multiple  $^{10}\text{B}$  layers intercepting neutron beam are used to increase the efficiency. PSD designed with small anode-cathode spacing and array of multiwire grids placed between double sided  $^{10}\text{B}$  coated plates are being fabricated. Assembly is arranged in curvilinear geometry with zero parallax. Overview of these developments is presented.

### 1. Introduction

National Facility for Neutron Beam Research at Dhruva reactor consists of 12 beam lines, 8 in the reactor Hall and 4 in Guide Lab. The user community is from BARC, National laboratories and various Universities. The instruments are supported with indigenously developed detectors. Range of detectors consists of neutron beam monitor, neutron proportional counters and position sensitive detectors (PSD) (Figure 1). High efficiency high resolution  $^3\text{He}$  filled PSDs in stacking geometry are developed successfully. But recent scarcity of  $^3\text{He}$  globally has made it essential to find an alternative. It is essential to explore the use of other alternatives as  $\text{BF}_3$  and  $^{10}\text{B}$  coating, particularly for neutron scattering applications. No other currently available detector technology offers the stability, sensitivity and convenience of  $^3\text{He}$  filled detectors. Among various feasible alternatives,  $\text{BF}_3$  gas has most potential.  $\text{BF}_3$  filled PSDs were seldom used, but were not popular due to toxic and corrosive nature of  $\text{BF}_3$ . Handling the generation and distillation system and minimise impurity level is a challenge. Performance of  $\text{BF}_3$  filled position sensitive detectors (PSD) is evaluated and novel designs are being tried for gain in efficiency. Performance of  $^{10}\text{B}$  coating based PSD is also evaluated. Though efficiency of  $^{10}\text{B}$  coated detector in equivalent dimensions is lower than that with  $\text{BF}_3$ , advantage of  $^{10}\text{B}$  coated PSD is its non-toxic nature. The overview of these developments in detector instrumentation supporting the neutron scattering facility is presented.

### 2. Neutron detectors mounted at various neutron scattering instruments, Dhruva

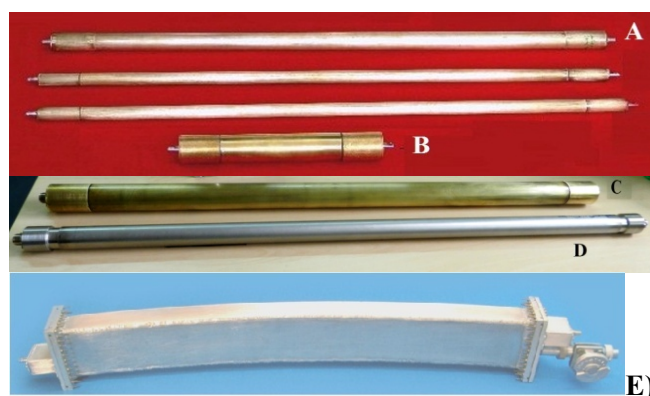
All spectrometers are equipped with in-house made  $\text{BF}_3$  filled beam monitor counters with 0.03 cps sensitivity. These monitors are used to normalise the scattering spectra for any fluctuations in neutron beam intensity with time. A small PSD (200 mm long and 50 mm dia) is mounted at Neutron Reflectometer. Charge division is carried out through resistive carbon coated quartz filament as anode.



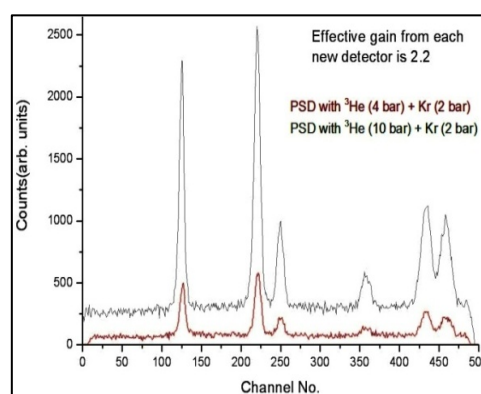
Small Angle Neutron Scattering instrument is equipped with a single linear PSD of 900 mm sensitive length and 5 mm position resolution. Powder Diffractometer is mounted with a multiPSD system with 5 PSDs [ $^3\text{He}$  (4 bar) + Kr (2 bar)] covering the  $2\theta$  range of  $3^\circ$  to  $140^\circ$ . Triple Axis Spectrometer, Single Crystal Diffractometer and Polarised Neutron Spectrometer are mounted with high efficiency counting detectors in end-on position with scanning mode.

## 2.1 Up gradation of Hi-Q Diffractometer using high efficiency PSDs

High Q Diffractometer was initially mounted with 5 PSDs [ $^3\text{He}$  (4 bar) + Kr (2 bar)] for data collection covering the  $2\theta$  range of  $2^\circ$  to  $140^\circ$  with some angular overlap. In order to improve the performance of instrument, high efficiency PSDs with various cathode diameters, fill pressures and additive gases were developed [1] and characterized. Design parameters and performance are optimized for high efficiency PSD [cathode diameter 37 mm and fill gas  $\text{He}^3$  (10 bar) + Kr (2 bar)]. The performance of these PSDs is tested at Hi Q Diffractometer. Figure 2 shows the comparison of diffraction data of an old PSD  $\text{He}^3$  (4 bar) and a new PSD  $\text{He}^3$  (10 bar) at High-Q spectrometer. Spectra indicate gain of 2.2 in counting statistics. Later this instrument was upgraded with 15 such PSDs, arranged in a pattern of 3 stacks with 5 PSDs in each stack. This setup with PSD in stacking geometry has improved the throughput of the instrument considerably.



**Figure 1** Picture of various linear 1-D PSDs developed for use at neutron spectrometers, at Dhruva A) High efficiency PSD for Hi Q Diffractometer B)  $\text{He}^3$  PSD for Neutron Reflectometer C)  $\text{B}^{10}$  coated PSD D)  $\text{BF}_3$  PSD for TOF instrument E) Curvilinear Multigrad PSD



**Figure 2** Comparison of Diffraction spectra recorded using various PSDs 1)  $\text{He}^3$  (4 bar) + Kr (2 bar) and 2)  $\text{He}^3$  (10 bar) + Kr (2 bar) with same geometry and angle covered.

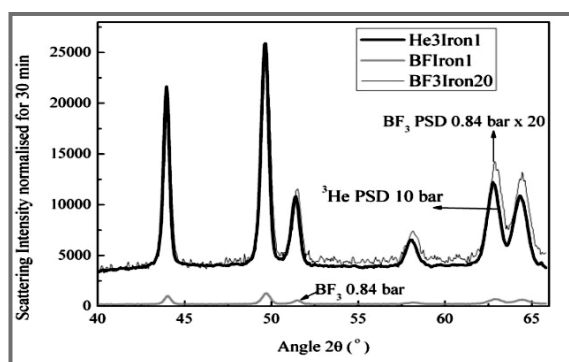
## 3. PSDs with $\text{BF}_3$ gas and $\text{B}^{10}$ coatings as $^3\text{He}$ alternatives

Further up gradations of instruments were hindered due to non-availability of  $^3\text{He}$  gas. Efforts are on to find suitable alternatives using  $\text{BF}_3$  gas and solid  $\text{B}^{10}$  layers. In-house facility for generation and distillation of  $\text{BF}_3$  gas and fabrication of detectors has facilitated various characterization of the gas behaviour in detector.

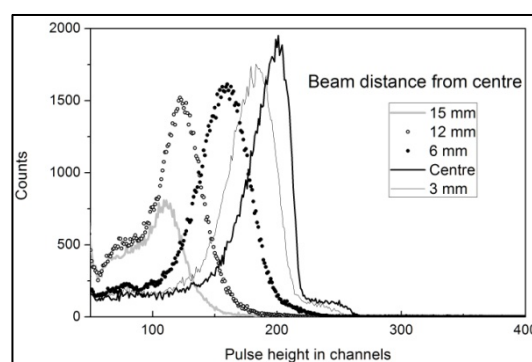
### 3.1. $\text{BF}_3$ based PSD

Generation of  $\text{BF}_3$  gas from  $\text{CaF}_2(\text{BF}_3)$  complex involves various processes, such as moisture extraction, gas generation by thermal decomposition of complex and distillation of gas at triple point temperature. Gas purification is carried out with repeated distillations. A PSD with 1 m long, 37 mm diameter cathode and 10  $\mu\text{m}$  anode wire is assembled and filled with  $\text{BF}_3$  gas at 0.8 bar pressure. Diffraction spectra of Fe is recorded with the PSD at Powder diffractometer using  $\lambda = 0.783 \text{ \AA}$  [2]. Comparison of spectra from  $\text{BF}_3$  PSD (0.8 bar) with  $^3\text{He}$  + Kr (10 + 2 bar) PSD is carried out with reference to peak intensity and peak width. Position resolution of the  $\text{BF}_3$  PSD is found to be  $\sim 7 \text{ mm}$ ,

and is acceptable for the present resolution of the Spectrometer. However, the efficiency of  $\text{BF}_3$  PSD is less than that of  $^3\text{He}$  PSD by a factor of 20 (Figure 3). Efforts are towards improving efficiency by high pressure gas filling and use of stacking geometry. Test on PSD filled with  $\text{BF}_3$  at 1.6 bar were also carried out and resulted in efficiency lower than  $^3\text{He}$  filled PSD by factor of 16, whereas, it resulted in broadening of peaks. Thus operating PSDs with high pressure in equivalent geometry do not help to gain efficiency. The nature of  $\text{BF}_3$  gas makes the avalanche formation very sensitive to small traces of impurities [3] like  $\text{SiF}_4$ ,  $\text{HF}$ , and  $\text{SF}_6$ . Pulse height distribution deteriorates with the pressure. Efforts are taken towards reducing impurity level by repeated distillations of  $\text{BF}_3$  gas. It has a limited effect.



**Figure 3.** Comparison of Diffraction spectra using  $^3\text{He}$  and  $\text{BF}_3$  filled PSDs mounted at Powder Diffractometer



**Figure 4** Effect of position of neutron interaction in drift region on pulse height distribution

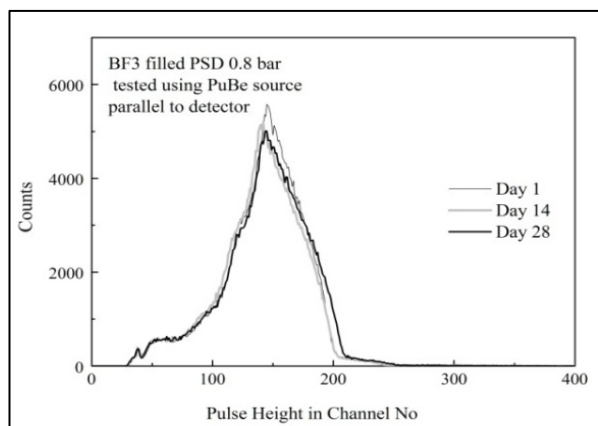
Effect of electronegative impurities in drift region is evaluated using a fine neutron beam parallel to anode. Beam is shifted along radial direction towards cathode. Effect of beam position on pulse height distribution using 2 mm point beam is shown in Figure 4. As the beam is shifted away from anode, drift length for all the primary charge clouds formed along the track of neutron is increased. Peak position of the pulse height spectrum is reduced and it is due to loss of primary electrons in the drift region. This loss of electrons is further amplified in the avalanche region resulting in lower charge collection. Recombination and electron attachment is dominant at large cathode radius and higher gas pressure. This effect can be reduced using higher drift field. Such behavior is not observed in  $\text{He}^3$  filled detectors.

### 3.2. PSDs for the proposed Time of Flight (TOF) Spectrometer at Dhruva

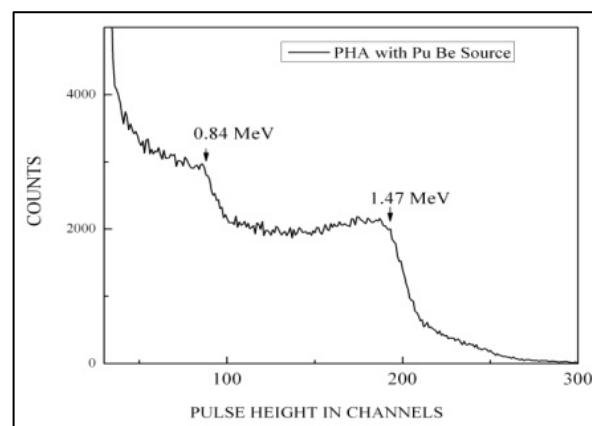
Various tests on effect of fill gas pressure, cathode drift region and anode dimensions on detector performance are carried out. These results are implemented in the PSD design. PSDs with higher gas pressure show deterioration in pulse shape, due to the increased impurity concentrations. They are well suited with lower drift region and high drift voltage. For similar gains, higher drift field is attained using thicker anode, whereas for PSDs based on charge division encoding, it is essential to use a resistive anode. Thus resistive anode of 25  $\mu\text{m}$  diameter is used.

The PSDs for detector bank of TOF spectrometer are optimised, considering the performance of  $\text{BF}_3$  detector. Dimensions of PSD is 38 mm diameter cathode, chosen to have higher beam height interception and gas depth in direction of neutron as compared to 25 mm dia cathode tube. Resistive anode used is 25  $\mu\text{m}$  NiCr and total anode resistance is  $\sim 3 \text{ k}\Omega$ . Drift field in the drift region at 1 cm from the anode is calculated for both the PSDs with anode bias needed for similar gas gains. Drift field increased from  $5.8 \times 10^3 \text{ V/cm}$  for 10  $\mu\text{m}$  anode to  $1.4 \times 10^4 \text{ V/cm}$  for 25  $\mu\text{m}$  anode. PSD in cylindrical geometry is mounted with two weldable, alumina ceramic feed thrus.  $\text{BF}_3$  gas does not show any aging with alumina ceramic. Inner wall of cathode is coated with activated charcoal. One of the PSDs filled at 1 bar  $\text{BF}_3$  gas, shows reasonable good pulse height distribution. It is tested for gas aging over few

weeks. Figure 5 shows the pulse height spectrum of the  $\text{BF}_3$  PSD recorded over a span of 28 days from gas filling. No aging or deviation in the performance over the holding time is noticed. Out gassing of PSDs for 15 days at  $110^\circ\text{C}$ , has been useful in maintaining the purity of gas. The multi-PSD system is designed to cover the detection area of  $2.5\text{ m}^2$  with  $2.5\text{ m}$  arc length ( $70^\circ$ ),  $1\text{ m}$  height ( $28^\circ$ ), consisting of an array of vertically arranged 60 PSDs ( $1\text{ m}$  long). PSD designs considering all these parameters have proved to be successful in replacing  $^3\text{He}$  PSDs. In spite of non availability of  $^3\text{He}$  gas, it has made possible the development of new TOF Spectrometer at neutron scattering facility.



**Figure 5** Pulse height spectra of  $\text{BF}_3$  filled PSD over the time of 28 days after gas filling.



**Figure 6** Pulse height distribution of a  $^{10}\text{B}$  coated PSD using Pu-Be neutron source

### 3.3. $\text{BF}_3$ filled PSD with complex geometry

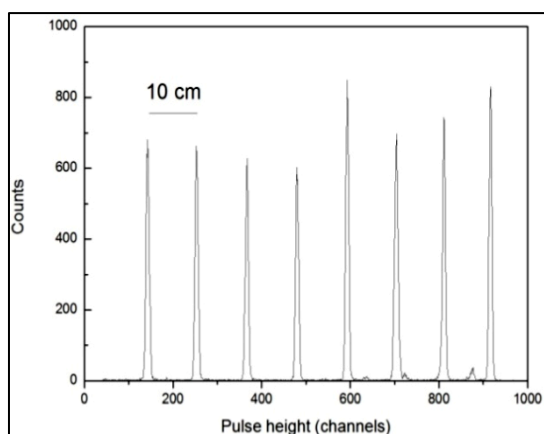
Considering these limitations on cathode diameter and fill gas pressure, gain in efficiency is attained using longer gas length using different geometry. It is possible with combination of multiple anode-cathode geometry. In such PSD, drift field and anode resistance can be designed independently. Complex geometry needs multiple wire supports and resistors for charge division. A multigrid PSD fabricated by ILL, Grenoble, was filled with  $\text{BF}_3$  gas at our facility and tested using neutron beam at Dhruva. Structure of the PSD, anode-cathode geometry is similar to the  $^{10}\text{B}$  coated Multigrid PSD [4] with 10 cells in direction of neutron beam. Gain in efficiency was recorded but gas aging within the PSD was noticed. Extensive out gassing of the PSD helped up to an extent.

In addition to complexity of data acquisition electronics, design and fabrication of a chamber and maintaining the gas purity over long time is a challenge. Restrictions are on use of insulators for wire assembly and surface mount resistors within the sensitive volume of PSD. Considering corrosive nature of  $\text{BF}_3$  gas, materials need to be chosen carefully to maintain good life of a PSD. Aging studies were carried out on various materials as Macor, PCB and Teflon. Teflon is found suitable with  $\text{BF}_3$ . Further design of multitube is included with Teflon supports for mounting anode wires. Though  $\text{BF}_3$  gas has limitations with impurity concentration, electro-negativity and aging with various materials, these efforts were helpful for design of new PSD.

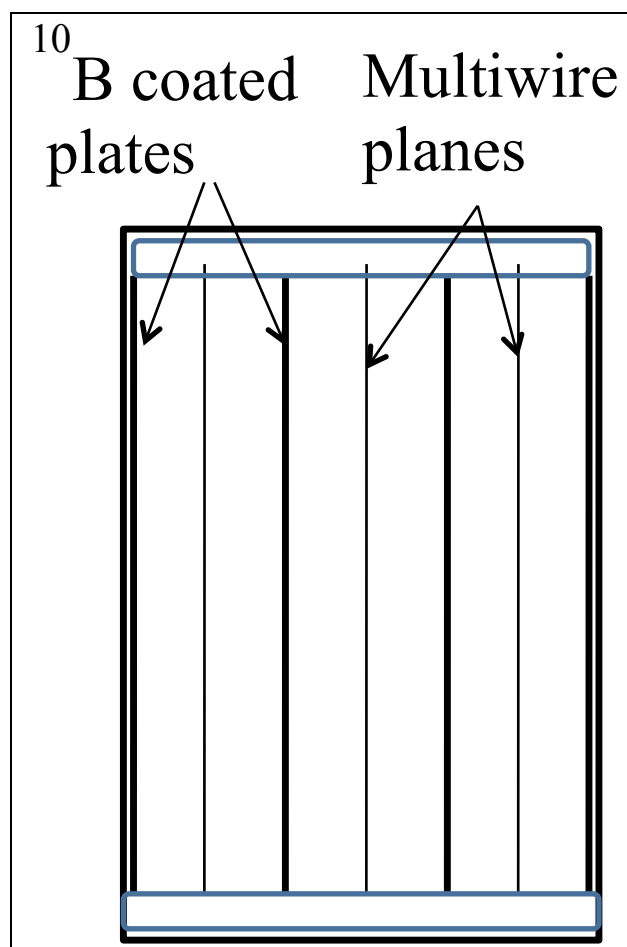
## 4. $^{10}\text{B}$ thin film based cylindrical PSD

$^{10}\text{B}$  coating is also a promising alternative to  $^3\text{He}$  with non toxic nature. Enriched  $^{10}\text{B}$  coating can be introduced on PSD cathode wall. Self absorption of charged particles,  $\text{Li}$  and  $\alpha$  within the coated layer, limits the neutron detection efficiency. Ionization resulting from emitted charged particles is recorded. Two prototype PSDs (PSD-1 and PSD-2) with  $^{10}\text{B}$  coating and coaxial cylindrical geometry are fabricated and characterized. Cathode dimensions are  $5\text{ cm}$  diameter and  $90\text{ cm}$  long and anode is  $25\text{ }\mu\text{m}$  diameter NiCr wire. Boron coating, 90%  $^{10}\text{B}$  enriched with coating thickness of  $1\text{ mg/cm}^2$  on inner lining of cathode, is obtained commercially with the painting and baking process. PSD-1 is filled with

gas Ar + CO<sub>2</sub> at 0.5 bar and evaluated for the pulse height, plateau characteristics and position resolution. Figure 6 shows the pulse height distribution of B<sup>10</sup> coated PSD. The position resolution was 10 mm. Thus PSD-2 is filled at 2 bar and position resolution is improved to 7 mm [4]. Figure 7 shows the position scan of 2 mm beam at 10 cm spacing, over the sensitive length of the PSD. Position resolution is acceptable for present instrument resolution. Uniformity of coating thickness was observed with the flooded neutrons and variation in intensity of pattern was within 2%. Neutron detection at each position in the spectrum is an integral contribution from all interaction with coating along circumference of cathode and projected on the anode. Sensitivity comparison of <sup>3</sup>He PSD (2 bar), BF<sub>3</sub> PSD (1 bar) and <sup>10</sup>B coated PSD was carried out using uniformly flooded neutrons. Sensitivity of the PSDs varies as 1:0.48:0.16 respectively at thermal neutron energy. Characterization of these PSDs was useful for further designs of large area PSD with multiple anode-cathode geometry. Advantage of non toxic nature of <sup>10</sup>B allows the choice of suitable proportional gas for fast drift velocity. Splitting of peaks is observed with inclined beam by 10°, as parallax is dominant at this cathode diameter. Parallax in He<sup>3</sup> PSD is observed as broadening of peaks at higher  $\theta$ , whereas, distinct position peaks are observed with <sup>10</sup>B coated PSD. This is due to that neutron interaction takes place at two points of cathode walls across neutron flight path and projection of these charge clouds on anode is recorded as position peaks. Further gain in efficiency can be obtained by introducing multiple <sup>10</sup>B coated layers along neutron path.



**Figure 7** Position scan of B<sup>10</sup> coated PSD-2 using a 2 mm fine collimated beam and 10 cm spacing.



**Figure 8** Schematic side view of a Teflon holder assembly of four <sup>10</sup>B coated plates and three multiwire grids.

#### 4.1. $^{10}\text{B}$ coated Multigrid Curvilinear PSD

Multiple  $^{10}\text{B}$  layers intercepting neutron beam and associated anodes for charge collection are essential to increase the neutron detection efficiency. A novel design with multiple  $^{10}\text{B}$  coated surfaces and curved design to avoid parallax is presented. An assembled curvilinear PSD is shown in Figure 1E and curved Teflon holder is used to support anode-cathode structure. Four  $^{10}\text{B}$  coated plates, with two single and two double sided coating, are arranged in curved casing. Six  $^{10}\text{B}$  coated surfaces are intercepted by the incident neutron beam. Height of coated plate is 6 cm and it adds to the height of neutron beam intercepted by detector. This can be further increased, considering the broadening at smaller angle. Each coated surface is facing a multiwire grid along the curvature of cathode plates. Mounting structure of anode grids and cathode plates is shown in Figure 8. Spacing between anode grid and cathode plate is 5.5 mm. Pitch of wire on anode grid is 5 mm. Position readout is carried out using charge division encoding method.

An arc length of 0.7 m (detector sensitive length) covers the angular range of  $23^\circ$ . The active gas thickness is 35 mm. Complete PCB and Teflon assembly is guided through a curved cathode casing. Three multiwire grids with resistive wires are connected in series. Central anode grid is mounted at radius 2000 mm and other two grids at  $\pm 12$  mm radii from central position. At constant sensitive length of 700 mm for all three grids, deviation in  $2\theta$  for other two grids is  $\pm 0.12^\circ \sim \pm 4$  mm when central anode wires on multiwire grids are aligned. This deviation in position can be normalized. Presently PSD with single grid is tested and position resolution obtained is 5 mm.

#### 5. Conclusions

The neutron scattering instruments at Dhruva are supported with indigenous development of neutron detectors and PSDs. Up gradation of Hi-Q Diffractometer using high efficiency and high resolution PSDs in stacking geometry was carried out with a considerable gain in throughput of Instrument. Efforts are initiated to find alternatives to  $^3\text{He}$ , using  $\text{BF}_3$  gas and  $\text{B}^{10}$  coatings. Prototype PSDs in coaxial geometry using these materials show encouraging results. In-house facility for generation and distillation of  $\text{BF}_3$  gas has facilitated characterization of gas behaviour in the detectors. PSDs for Time of Flight instrument are successfully designed and fabricated with acceptable efficiency and durability. Aging of  $\text{BF}_3$  gas due to various construction materials of complex geometry detector was noted. Results were useful for further designs of PSD with multiple anode-cathode structure. PSD with  $^{10}\text{B}$  coatings though show low efficiency, gain in efficiency is desired by introducing multiple  $^{10}\text{B}$  coated layers along neutron path. Design aspects of Curvilinear Multigrid PSD with zero parallax are mentioned. This novel design is expected to show 3 times gain in efficiency as compared to cylindrical PSD.

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