

Demagnetization of $\text{Nd}_2\text{Fe}_{14}\text{B}$, $\text{Pr}_2\text{Fe}_{14}\text{B}$ and $\text{Sm}_2\text{Co}_{17}$ permanent magnets in spallation irradiation fields

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Abstract

Prompted by the need for radiation-resistant permanent magnets for the insertion devices (IDs) of high-brilliance next-generation synchrotrons such as the NSLS II, the demagnetization of $\text{Nd}_2\text{Fe}_{14}\text{B}$ and $\text{Pr}_2\text{Fe}_{14}\text{B}$ was studied after exposure to a mixed irradiating field. Degradation and damage of the permanent magnetic material by components of electromagnetic showers induced in magnets by intense high-energy electron beams will alter the magnetic field structure of the IDs. Plate-like $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets were irradiated to 1.8 GRad dose and were evaluated against $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets irradiated to lower dose of 20 MRad. In addition, annular $\text{Sm}_2\text{Co}_{17}$ and $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets integrated within a ferrofluidic feedthrough rotary seal were also irradiated to dose levels of 2 GRad for $\text{Sm}_2\text{Co}_{17}$ and 20 MRad for $\text{Nd}_2\text{Fe}_{14}\text{B}$.

Post-irradiation measurements of the magnetic intensity revealed that severe demagnetization exceeding 85% occurs in $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets after only 50 MRad dose and over 87% for $\text{Pr}_2\text{Fe}_{14}\text{B}$ after 10 MRad dose. The annular-shaped $\text{Sm}_2\text{Co}_{17}$ magnets of the ferrofluidic feedthroughs were almost insensitive to irradiation up to a dose of 2 GRad. Annular shape $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets also showed resistance to demagnetization, a direct consequence of the annular shape which is characterized by the removal of the stronger demagnetizing field present at the center of a disk-like magnet. The sensitivity of boron-based permanent magnets to the neutron energy (thermal vs. fast) was also assessed via specifically designed experiments and discussed.

Keywords: Permanent magnets, irradiation-induced demagnetization, radiation damage

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1. Introduction

The understanding of radiation-induced demagnetization of rare earth permanent magnets has seen renewed interest driven by the need for radiation-resistant magnets in a variety of applications including insertion devices (ID) for next generation synchrotron light sources and Free Electron Lasers (FEL) as well as rotary ferrofluidic feedthroughs (FFFT) in high radiation environments of future projectile fragment separator facilities such as the Facility for Rare Isotope Beams (FRIB), to name a few. The harsh radiation environment in these applications is composed of a broad energy spectrum of photons, X-rays, electrons and neutrons. High brilliance in the next generation light sources such as the NSLS II is obtained from the high magnetic fields in the insertion devices. Permanent magnets are used in these devices to produce x-rays for scientific research. The beam lifetime is limited to 3 hours by single Coulomb scattering in the bunch (Touschek effect). This effect occurs everywhere around the storage ring circumference leading to unavoidable beam loss in the adjacent low-aperture insertion devices. This raises the issue of degradation and damage of the permanent magnetic material of the various IDs caused by direct and scattered radiation. Direct electron beam irradiation by miss-steered or lost electron beam on the magnet would result in an electromagnetic shower consisting of photons, electrons, positrons, and neutrons. FFFT devices rely on the magnetic field provided by their permanent magnets for rotary motion through a vacuum boundary in the high-radiation field of the target system of facilities such as FRIB. Radiation-induced demagnetization from the mixed spectrum of fragments, neutrons, electrons and gamma rays will inevitably result in performance degradation of the FFFT.

Radiation damage in all materials is a function of the irradiating species type and their energy, the dose rate as well as the total absorbed dose, the irradiation temperature, the impurity content of the material and the irradiation history. Regarding the irradiating species a damage hierarchy expressed as $p > n > e > \gamma$ in the mixed field is believed to be in place with high-energy protons more damaging than low-energy counterparts. Over the years radiation damage studies of permanent magnets have been performed under a variety of radiation fields, such as electron, proton, gamma, neutron and mixed electron-photon (bremsstrahlung). The studies have shown that charged particles and high-energy neutrons are effective at causing serious radiation damage in permanent magnets resulting in significant loss of magnetic field intensity. While findings from radiation damage studies are often difficult to interpret as they are influenced by factors such as irradiation temperature, sample geometry, grade and magnet composition, general trends have been established. These delineate the effects of irradiating species (gammas vs. charged particles for example) or permanent magnet type (i.e. Nd-Fe-B vs. Sm-Co) or composition ($\text{Sm}_2\text{Co}_{17}$ vs. SmCo_5). The resistance to demagnetization by irradiation and thus the choice of the type of a permanent magnet depends on a number of parameters. For example, the larger the coercivity (ability of a material to resist demagnetization by an external magnetic field) is, the higher the radiation resistance. Sm-Co magnets have been found to be more radiation resistant than Nd-Fe-B counterparts which could be attributed to their higher Curie temperature and superior thermal properties. However, Sm-Co magnets have lower energy products than Nd-Fe-B magnets and are significantly more expensive.

In [1] the effects of x-rays, photons, and neutrons, delineated thermal and fast, on $\text{Nd}_2\text{Fe}_{14}\text{B}$ N38H-grade magnets used in the Advanced Photon Source (APS) insertion devices were studied. Photon irradiation from ^{60}Co source to 700 MRad (100 MRad=1 MGy) was reached in [1]. In addition the demagnetizing effects of fast neutron fluence of 1.61×10^{14} n/cm² and thermal neutron fluence of 2.94×10^{12} n/cm² were evaluated. A change of only 0.108% was observed for 700 MRad photon irradiation and 16.172% for the fast neutron irradiation. No change was observed for the lower fluence thermal neutron. In [2] the effects of electron-beams and photon irradiation on the magnetic flux of Nd-Fe-B and Sm-Co permanent magnets were studied. Similarly as in [1] ^{60}Co photon irradiation of Nd-Fe-B permanent magnets to an absorbed dose of 280 MRad showed magnetic field intensity reduction below 0.5%. Nd-Fe-B magnets, however, irradiated to an electron dose of 260 MRad exhibited magnetic flux loss of ~9% with an increase in flux

loss for higher doses. $\text{Nd}_2\text{Fe}_{14}\text{B}$ and $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets considered for cryogenic undulators were irradiated with 2.5 GeV electrons [3] and were found that the resistance to radiation increased substantially at low temperatures. The partial demagnetization of the IDs at the ESRF (European Synchrotron Radiation Facility), from miss-steering of the electron beam onto the vacuum chamber wall of the device resulted in severe degradation of magnetic properties [4]. For estimated absorbed dose in the range of 5.1-6.7 MRad the resulted peak magnetic field loss in the device was approximately 8%.

Other studies [5, 6] of gamma-irradiated $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets showed a total flux loss of 1.5% at 50 MRad. For charged particle irradiation (106 MeV ^2H ions) and absorbed dose of only 2.6 MRad total loss of flux was >80%. Demagnetization effects of protons on Sm-Co and Nd-Fe-B permanent magnets were studied in [7] using 500 MeV proton beams with the irradiation temperatures kept below 120°C. Sm-Co magnets exhibited a significant demagnetization when irradiated by the protons at dose levels in the range of 1-10 GRad. Nd-Fe-B magnets during the same proton irradiation experiments proved to be extremely sensitive to ionizing radiation losing essentially all of their magnetization at a dose of 70 MRad and over 50% at 4 MRad. Y. Ito [8] investigated radiation damage of Nd-Fe-B magnets from 200 MeV protons to low doses (up to 20 MRad) and found that the magnetic flux of Nd-Fe-B magnets decreased drastically as the proton dose increased. It was found [8] that following low-dose irradiation/demagnetization the demagnetized magnets can be fully magnetized again to the same intensity as before the irradiation.

In [9] 20 MeV protons were used to irradiate Nd-Fe-B magnets at different temperatures and orientations with respect to irradiating field. The study reported that at low temperature magnets are insensitive to radiation whereas at room temperature 100 MRad dose was sufficient to reduce the magnetization by 40%. Also revealed in [9] is the strong magnet shape effect on its demagnetization behavior. Specifically, their experimental data confirmed previously observed behavior [5-6], that when the thickness of the sample along the magnetization axis is large, the demagnetization is smaller. In [10] SmCo_5 magnets were irradiated with 20 MeV protons at high temperatures (300 – 800 K) and exhibited high sensitivity to irradiation. The magnetic flux loss measured was also matched with a theoretical model based on local heating induced by the incoming particles. Cost and Brown [11-12] have exposed Sm-Co and Nd-Fe-B permanent to fluences reaching 1.2×10^{16} neutrons/cm² while considering various varying parameters such as irradiation temperature, length-to-diameter (L/D) ratio in magnet dimensions and magnet vendors. They quantified that the decay rates for Nd-Fe-B are faster than for Sm-Co magnets. They also reported that grain size may affect radiation-induced decay. For Nd-Fe-B they observed that the initial loss of remanence is extremely sensitive to temperature (twice as fast at 426 K than at 350 K, 10% for a fluence of 10^{15} n/cm²). Post-irradiation re-magnetization resulted in full recovery of remanence and in 20% increase in coercivity.

Permanent magnets have been irradiated [13] in a reactor at neutron fluxes up to 8×10^{19} neutrons/cm² ($0 < E < 7$ MeV) and $\text{Sm}_2\text{Co}_{17}$ magnets were found to be 10 times less sensitive to neutron irradiation than SmCo_5 . Neutron induced demagnetization on permanent magnets have been also studied by Chen and coworkers [14] and Samin [15] where the effects of thermal and fast neutrons were delineated. It was found in [15] that the combination of fast and thermal neutrons has a stronger de-magnetizing effect than fast neutrons alone. Studies on Nd-Fe-B magnets of insertion devices [16] and the radiation resistance of Sm-Co based magnets [17] have also been studied. Studies on permanent magnet demagnetization of Advanced Photon Source (APS) undulators were conducted [18] with demagnetization found to be confined to a limited volume at the surface close to the electron beam. Results of the study also suggested that a small volume at the surface to have acquired a weak magnetization in the opposite direction. Important aspect of the study is the performance of predictive simulations of both the radiation environment [19] and of the magnetization profiles [18] which closely reproduced the magnetic measurements

The present study, in an effort to reproduce realistic particle accelerator conditions where the demagnetization field is a mixed field rather than a field where the effects of individual irradiation species is delineated, has focused on the demagnetizing effects induced by the irradiating particle cocktail by

conducting a series of challenging experiments in an actual accelerator setting. To the author's knowledge this represents the first such study where a mixed irradiation field has been used to assess the vulnerability of the studied magnet family.

The effect of neutron moderation (ratio of fast to thermal neutrons) on the demagnetization of $\text{Nd}_2\text{Fe}_{14}\text{B}$ was assessed and experimentally confirmed at an order of magnitude higher fluence from what has been reported to-date [15] the more pronounced demagnetization effect of thermal neutrons compared to fast neutrons due to the thermal neutron capture reaction of boron.

The demagnetization behavior of $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets, under consideration in synchrotron insertion devices due to their higher Curie temperature to replace $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets, has only been assessed to-date [3] to electron fluence of $\sim 2 \times 10^{15}$ indicating higher resistance than its $\text{Nd}_2\text{Fe}_{14}\text{B}$ counterpart. This study addresses the effects of a mixed irradiation field to at least an order of magnitude higher in irradiating fluence while also evaluating (a) the magnet L/W shape factor and (b) the manufacturer and grade.

Finally, the present study seeks to deduce the first experimental results of the impact that the annular magnet shape has on the demagnetization resistance to irradiation. Confirmation of the increased resistance to demagnetization stemming from the removal of the inherent demagnetizing forces present in the magnet's central section (low permeance part) could have significant technological implications, particularly in insertion devices of high brilliance synchrotron light sources.

Presented in the subsequent sections, following the pre-irradiation characterization of the various types and shapes of permanent magnets used in the study as well as the description of the experimental details, the effect of high fluence, mixed irradiation fields on the different magnets and configurations are presented in detail. The mixed irradiation field utilized to assess the demagnetization was produced by the spallation of 118 MeV protons of flux $\sim 6.0 \times 10^{14}$ p/s on an array of isotope producing targets at the Brookhaven Linear Isotope Producer (BLIP) and consisted of thermal and fast neutrons, secondary protons, photons and electrons. In the present study fluence levels in excess of an order of magnitude higher than fluences achieved in studies conducted previously while studying the effects of individual irradiating species and reported/referenced herein, were reached. These correspond to absorbed doses up to 2 GRad for the $\text{Nd}_2\text{Fe}_{14}\text{B}$, $\text{Pr}_2\text{Fe}_{14}\text{B}$ and $\text{Sm}_2\text{Co}_{27}$ magnets studied and reported.

Significant findings of the undertaken study that are reported in the subsequent sections include (a) experimental confirmation of the increased demagnetizing effect of thermal neutrons as compared to fast neutrons in boron containing magnet compounds due to thermal neutron capture reaction, (b) demagnetization susceptibility of $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets characterized by higher Curie temperature when subjected to a mixed irradiation, as compared to $\text{Nd}_2\text{Fe}_{14}\text{B}$ counterparts that exhibit lower Curie temperature, and contrary to the findings of previous studies [3] reporting higher resistance to demagnetization of $\text{Pr}_2\text{Fe}_{14}\text{B}$ subjected to electrons at a lower fluence, and (c) experimental evidence of significantly enhanced demagnetization resistance of annular shaped $\text{Nd}_2\text{Fe}_{14}\text{B}$ and $\text{Sm}_2\text{Co}_{27}$ magnets.

2. Experimental

2.1 Permanent magnet samples

Block $\text{Nd}_2\text{Fe}_{14}\text{B}$ Magnets

Five commercially available $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets received from APS (produced at Dexter Corporation, Magnetic Technologies) with dimensions of 50 mm x 47.5 mm x 7 mm were used in the study. These

magnets were of N38H grade and they were of the exact type used extensively in the IDs of APS. Grade N38H is characterized by coercivity and has higher resistance to demagnetization than some other Nd-Fe-B grades. All five magnets were plated with a thin coating of nickel to reduce corrosion of the magnetic material. The Curie temperature of this N38H grade is $\sim 320^{\circ}\text{C}$. Each magnet had a unique identification namely, 3A, 6A, 11A, 12A and 20A.

The magnetic field intensity for each of the five $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets was measured with a Hall probe supported on a 2-mm thick non-magnetic plate which was introduced to facilitate remote measurements on the magnets residing in the hot cell following irradiation. This procedure was adopted so the pre-irradiation and post-irradiation measurement technique and parameters are identical. The five (5) block $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets exhibited very similar magnetic field values with an average of ~ 0.15 Tesla at the geometric center and ~ 0.22 Tesla at the edges.

Block $\text{Pr}_2\text{Fe}_{14}\text{B}$ Magnets

Two pairs of $\text{Pr}_2\text{Fe}_{14}\text{B}$ (Praseodymium-Iron-Boron) magnets were used in the demagnetization study. Specifically, two 53 CR grade (Sumitomo Special Metal Co., Ltd.) magnet blocks of size 50 mm x 30 mm x 5 mm and two 47 CR (Hitachi) magnets of size 40 mm x 20 mm x 4 mm were used. The Curie temperature of these $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets is $\sim 370^{\circ}\text{C}$, a desired parameter that makes them an attractive alternative to Nd-Fe-B magnets. Pr-Fe-B magnets have been developed for cryogenic devices [3]. Their remanence and coercivity can be increased by lowering the temperature below 77 K which gives them an advantage over Nd-Fe-B magnets. Demagnetization at low temperatures (145 K) from electron irradiation was shown to be remarkably smaller than at 300K [3].

Regarding their demagnetization resistance to radiation, it is anticipated to be driven by coercivity of the materials. The fact that praseodymium (Pr) material has lower coercivity than neodymium (Nd) or Sm-Co may result in an overall reduction of the resistance. The objective of the present study is to quantify the radiation resistance of $\text{Pr}_2\text{Fe}_{14}\text{B}$ to elevated fluences of mixed irradiation fields and compare it to their $\text{Nd}_2\text{Fe}_{14}\text{B}$ counterparts.



Figure 1: (a) $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnet vacuum capsule, and (b) contained pair of magnets encapsulated. Image at right following irradiation with magnets located within the hot cell.

For the irradiation phase of the experiment the two grades were paired up within a special stainless steel vacuum capsule (see Figure 1). The capsule windows were made of 0.03048 mm-thick SS304 steel (designated as #2 in Figure 1). The pair of $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets was placed within a specially designed cavity with a 230 μm -thick non-magnetic SS304 plate separating the two magnets. Vacuum atmosphere was achieved in the two capsules containing the magnet pairs by welding the assembly within a vacuum

chamber. The magnetization of these magnets are, like their $\text{Nd}_2\text{Fe}_{14}\text{B}$ counterparts, is along the small dimension (through thickness). Shown in Figure 1b is a pair of $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets following irradiation and after they were retrieved from the special vacuum capsule inside the hot cell of the Isotope Processing Facility.

Magnetic field measurements were performed for the two pairs of $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets (53 CR and 47 CR) prior to their irradiation. Measurements were made with the probe on the 2-mm thick non-magnetic support in an effort to make direct comparison with measurements following irradiation that required remote handling and the need for the probe support. Slightly stronger magnetic fields were recorded for the smaller size (47 CR, Hitachi 40 mm x 20 mm x 4 mm) magnets. Specifically, the two 47 CR magnets registered very similar magnetic field strengths of 0.135 Tesla at the geometric center of the magnet and 0.17 Tesla at the edge. The two larger magnets (53 CR, Sumitomo 50 mm x 30 mm x 5 mm) registered magnetic field strengths of 0.115 Tesla at the geometric center and 0.155 Tesla at the edges.

Annular $\text{Sm}_2\text{Co}_{17}$ and $\text{Nd}_2\text{Fe}_{14}\text{B}$ Magnets

Stemming from their high Curie temperature, their crystalline anisotropy and their exhibited highest resistance to radiation-induced demagnetization amongst Sm-Co compounds [7, 11, 14], $\text{Sm}_2\text{Co}_{17}$ magnets are the preferred choice in a number of applications, particularly those associated with high temperatures. Sm-Co magnets also exhibit high resistance to oxidation and corrosion thus requiring no surface treatment, in contrast to Nd-Fe-B counterparts; they are more brittle and come at a higher cost.

Annular shaped $\text{Sm}_2\text{Co}_{17}$ and $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets are utilized in ferrofluidic feedthrough (FFFT) rotary seals (schematic cross section shown in Figure 2) where they provide the necessary magnetic field for the function of the ferrofluid. A ferrofluid feedthrough rotary seal enables a dynamic, hermetic seal for rotating mechanisms. The ferrofluid resides in the small gaps between the shaft and pole magnet (see Figure 2) thus creating a set of liquid O-rings around the rotating shaft and enabling the separation of atmosphere and vacuum. The annular permanent magnet resides inside the created vacuum cavity and never comes into contact with the ferrofluid. The magnetic field increases the apparent viscosity of the ferrofluid to the point of becoming a viscoelastic liquid. The ferrofluid itself is a magnetically stable colloidal suspension, typically made of iron oxide nanoparticles coated with a surfactant, such as oleic acid, and suspended in an organic liquid carrier, such as hydrocarbon, ester, fluorocarbon, etc. The composition of a typical ferrofluid is ~5% magnetic solids, ~10% surfactant and 85% liquid carrier, by volume. Surfactant acts as a stabilizing agent by preventing the attractive forces, i.e. van der Waals and magnetic forces, of materials even when a high intensity external magnetic field is applied. Ferrofluidic feedthrough rotary seals containing either Nd-Fe-B or Sm-Co permanent magnets have been considered for use in the target and beam dump systems of the Facility for Rare Isotope Beams (FRIB).

Two ferrofluidic feedthroughs containing annular $\text{Sm}_2\text{Co}_{17}$ magnets and one containing $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnet were irradiated using the mixed field generated downstream of the isotope target array at the BLIP beamline endstation as shown in Figure 5. The annular $\text{Sm}_2\text{Co}_{17}$ magnets, magnetized as shown in the schematic of Figure 2 were irradiated to doses of 200 MRad and 2 GRad respectively.

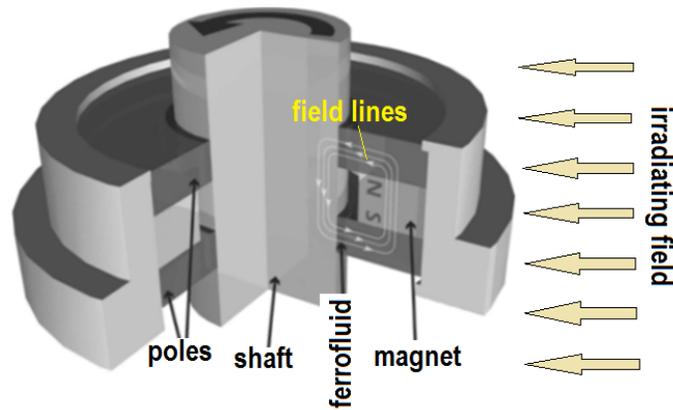


Figure 2: Schematic of FFFT containing annular $\text{Sm}_2\text{Co}_{17}$ or $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets

The ferrofluidic feedthrough unit containing the $\text{Nd}_2\text{Fe}_{14}\text{B}$ annular magnet was also irradiated in a similar orientation with respect to the irradiating flux to a lower dose (20 MRad).

As noted above, the ferrofluid which plays the role of the O-ring at the interface between the shaft and the poles (shown in Figure 2) does not come into contact with the annular $\text{Sm}_2\text{Co}_{17}$ or $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets to have any influence during irradiation.

Magnetic field measurements were performed prior to irradiation for the three FFFT units in a similar manner as the block $\text{Nd}_2\text{Fe}_{14}\text{B}$ and $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets (i.e. probe attached to a nonmagnetic support plate that enables remote scanning within the hot cell). The magnetic field measurements were made over the housing of the FFFT unit (outer surface) and revealed (a) sensitivity with the radial distance resulting from the use of the 2-mm thick non-magnetic probe support and (b) slightly stronger field for the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnet. Specifically, the peak magnetic field measured with the probe in contact with the outer surface of the FFFT housing for the $\text{Sm}_2\text{Co}_{17}$ was 40.3 mT (0.0403 Tesla) and with the 2-mm probe support 26.2 mT. For the FFFT containing the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnet the peak field measured with the probe on the 2-mm support was 28.0 mT.

2.2 Irradiation Experiments and Species

Irradiations of the three types of permanent magnets were performed at the BLIP target station used for medical isotope production material irradiation. The mixed field produced downstream of the isotope targets (Figure 3a) by the 118 MeV proton beam of flux $\sim 6.0 \times 10^{14} \text{ s}^{-1}$ is used for irradiating the magnets. The 118 MeV protons are stopped by the array isotope production targets made of rubidium chloride and gallium and through spallation produce a broad spectrum of thermal and fast neutrons, secondary protons, electrons and gamma rays. Positions A and B shown in Figure 3a were utilized for the irradiation of the five $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets. Specifically, four magnets were irradiated at Position A and one (3A) at position B. Neutron moderation due to the water volume at Position B is expected to change the ratio of fast to thermal neutrons. The irradiation of a magnet at Position B aimed to explore such effect. Figure 3b depicts the MARS15 [21] calculated spatial distribution of the neutron flux normalized to 10^{12} protons/sec. 90-95 μA of beam current corresponding to $5.62\text{-}5.95 \times 10^{14}$ p/s was irradiating the flux on isotope targets. The nickel-plated magnets were in direct contact with the BLIP de-ionized cooling water flowing past the two magnet surfaces. Thermal analysis based on energy deposition on the magnet from the spallation field and forced convection heat transfer in the channels was performed using the LS-DYNA code [24]. The temperature of the magnet during irradiation was estimated to be $\sim 70^\circ\text{C}$.

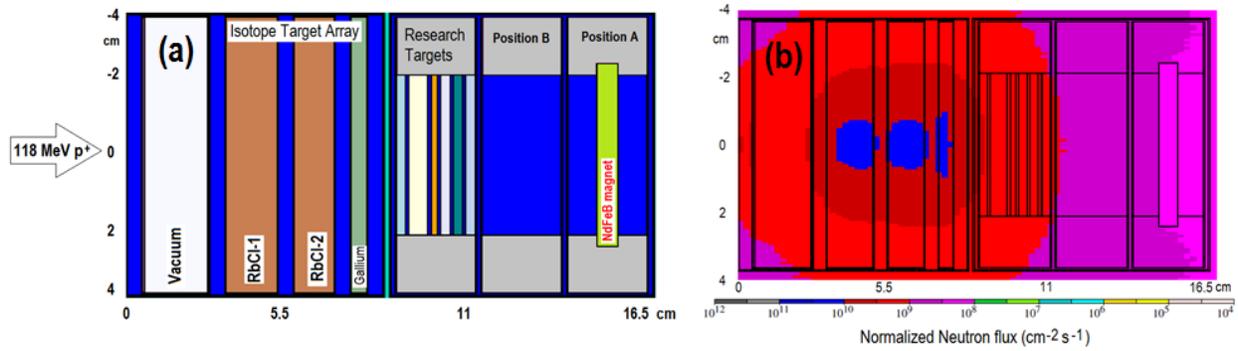


Figure 3: (a) Schematic of $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnet irradiation set-up as in the MARS15 model, (b) MARS15-calculated neutron flux profile

Depicted in Figure 4 are the neutron and gamma spectra at two locations within the spallation field downstream of the isotope targets. The estimated neutron flux at Position A where four of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets were irradiated was $\sim 1.483 \times 10^{11} \text{ n/cm}^2\text{-s}$ and mean energy $\langle E_n \rangle = 9 \text{ MeV}$. The gamma flux is $2.5 \times 10^{11} \text{ cm}^{-2}\text{-s}^{-1}$ while the secondary proton (recoils) flux and electron flux are ~ 4 and 3 orders of magnitude lower respectively. The neutron fluence for the absorbed dose of 1,800 MRad and 50 MRad were estimated to be $\sim 4.38 \times 10^{17} \text{ cm}^{-2}$ and $1.29 \times 10^{16} \text{ cm}^{-2}$ respectively. Table 1 lists the primary proton beam current and the estimated absorption dose received by the five magnets. As shown in the table the absorbed dose ranged between 50 MRad and 1.8 GRad.

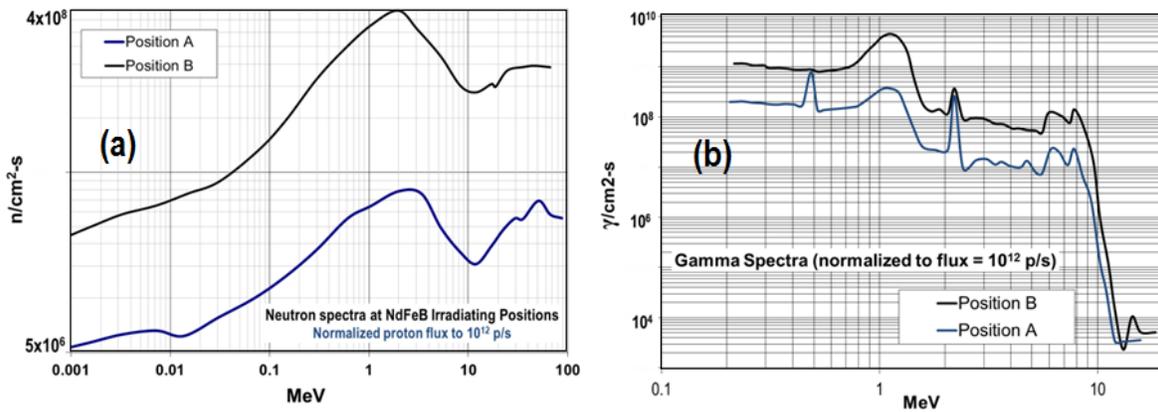


Figure 4: Neutron (a) and gamma (b) energy spectra at locations A and B in irradiating spallation field

Table 1 lists the total 118 MeV proton beam current on the RbCl and Ga spallation targets located upstream of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets being irradiated (see Figure 3) and the estimated dose received by each magnet due to the generated mixed field at the magnet location.

Table 1: Integrated proton beam current of irradiation experiments of $\text{Nd}_2\text{Fe}_{14}\text{B}$ block magnets and corresponding magnet absorbed dose

Magnet	Current	Dose
Magnet 11A	78,000 $\mu\text{A-hrs}$	1.8 GRad
Magnet 12A	45,000 $\mu\text{A-hrs}$	1.0 GRad
Magnet 3A	50,000 $\mu\text{A-hrs}$	1.2 GRad
Magnet 20A	11,000 $\mu\text{A-hrs}$	240 MRad
Magnet 6A	2,300 $\mu\text{A-hrs}$	50 MRad

Subsequent to the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets the two capsules containing pairs of 53 CR and 47 CR $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets were irradiated at Position A of the spallation field (see Fig. 3a) and for two primary proton beam current levels, 920 μA -hours and 447 μA -hours. These levels correspond to 20 MRad and 10 MRad absorbed doses respectively. The neutron flux during irradiation was $\sim 1.56 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$ and the mean neutron energy $\langle E_n \rangle = 9 \text{ MeV}$. The corresponding neutron fluences were estimated to be $0.52 \times 10^{16} \text{ n/cm}^2$ and $0.255 \times 10^{16} \text{ n/cm}^2$ respectively. Similarly, numerical analyses based on LS-DYNA code [24] were employed to estimate temperatures during steady-state irradiation. These accounted for contacts between the magnets and the non-magnetic separating plates as well as contact with inner faces of the capsule upstream and downstream windows. Conservative estimates that accounted for uncertainties regarding the heat transfer across contacting surfaces the temperature of the magnets irradiated under vacuum was estimated to be between 80-100°C, a value much lower than the 370°C Curie temperature of these $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets. It was assessed [9, 10] that irradiation temperature plays a significant role in the demagnetization process. Measured and calculated temperature dependence [9, 10] showed that while there was nearly no loss at $\sim 20 \text{ K}$ irradiation temperature for proton-irradiated Nd-Fe-B magnets, only 70% of the magnetic field remained for a 300 K irradiation temperature. Sm-Co permanent magnets on the other hand showed sensitivity to demagnetization at temperatures $> 300 \text{ K}$ reaching 50% of the original value at 850 K under proton irradiation. The temperature contribution alone on demagnetization of the $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets studied and reported here was not measured. These will be quantified during a follow-up phase where more precise irradiation temperatures will be established.

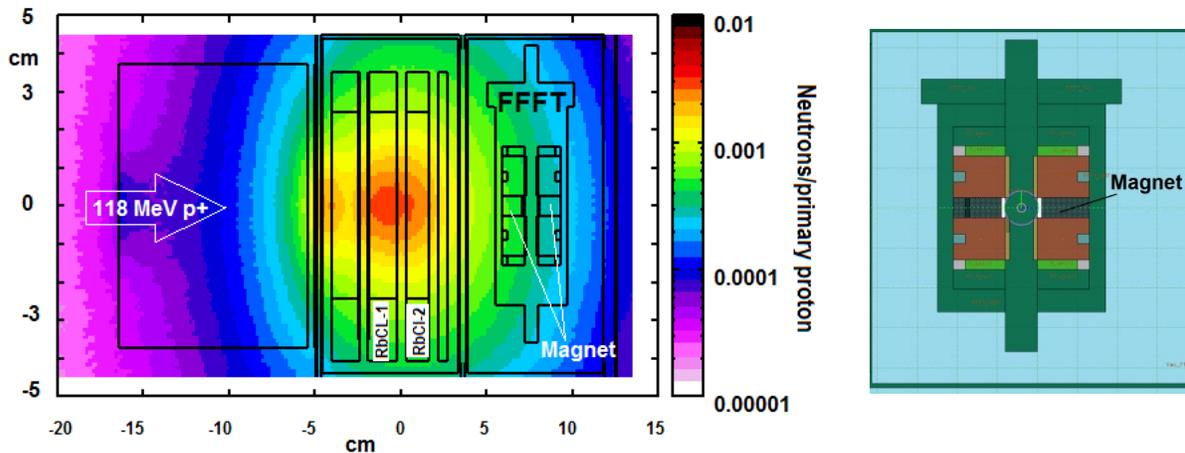
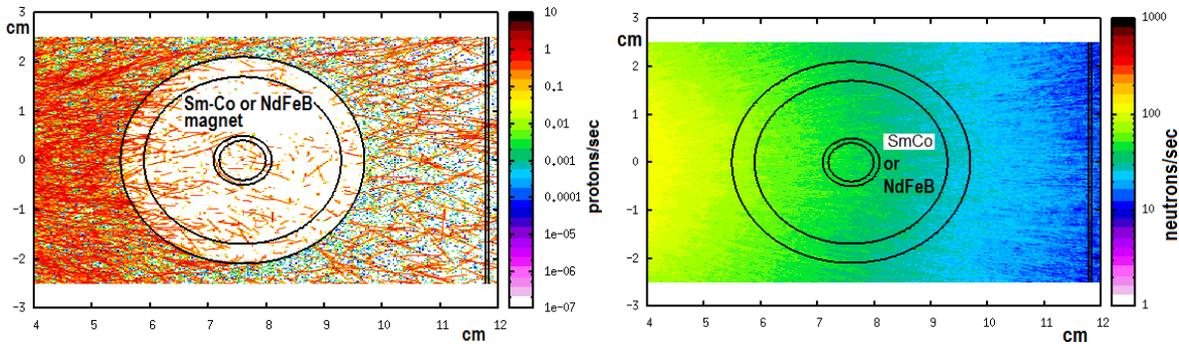


Figure 5: Schematic of ferrofluidic feedthrough irradiation set up (left) and numerically modelled cross-sectional details (right)



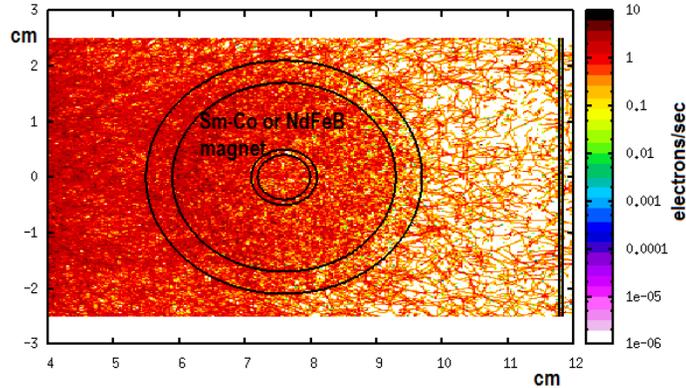


Figure 6: FLUKA-calculated profiles of irradiating species of $\text{Sm}_2\text{Co}_{17}$ and $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets in FFFT. Depicted is the dominance of the neutron contribution to the possible effects (note the non-uniformity in irradiation)

Calculated with FLUKA code [22, 23] neutron distribution for the irradiation of the three FFFT units containing the annular $\text{Sm}_2\text{Co}_{17}$ or $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets is shown in Figure 5a. The cross-section of the feedthrough with the orientation of the annular permanent magnets is shown in Figure 5b. The non-uniformity of the mixed field irradiating the magnet is depicted in Figure 6 for secondary protons, neutrons and electrons. The irradiation durations were selected such that 2 GRad and 200 MRad absorbed doses will be seen by the feedthrough units with Sm-Co type magnets and 20 MRad for the Nd-Fe-B-type. Important to note is the orientation of the magnetization in the annular magnets relative to the irradiating field (see Figure 2). While the block-type $\text{Nd}_2\text{Fe}_{14}\text{B}$ and $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets had the magnetization aligned with the field (more-less), in the FFFT case the irradiating field is normal to the magnetization.

2.3 Measuring Techniques

Magnetic flux prior and post irradiation of the block Nd-Fe-B and Pr-Fe-B magnets and the FFFT units were measured using a hall probe and a fluxmeter [model DTM-141 Teslameter]. The block magnets were mapped by moving the probe ~ 2 mm above the surfaces (positive and negative) and normal to the magnetization direction. The FFFT magnetic field intensity was measured along the shaft and across the contained magnet ~ 2 mm from the stainless steel protective shell. The magnetic flux intensity of the unirradiated FFFT units was also measured/verified using a LakeShore 410 Gaussmeter. Following irradiation, the block magnets and the FFFT units were measured at the hot cell laboratory of the Isotope Extraction Facility and in particular the HEPA filtered fume hood using an identical probe and system to that used to baseline them prior to irradiation (DTM-141 Teslameter). Several measurements were performed for the unirradiated magnets and average values were used for demagnetization assessments. A high sensitivity ORTEC Ge detector was used to extract photon spectra from irradiated Nd-Fe-B magnets and FFFTs. The photon spectra of the Nd-Fe-B magnets were used to study possible effects of the composition of neutron flux (Position A vs. Position B) on demagnetization.

3. Experimental Results and Post-irradiation Magnet Evaluation

3.1 Rectangular $\text{Nd}_2\text{Fe}_{14}\text{B}$ Magnets

Values of the magnetic intensity/profile of the five block, or plate-like, $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets measured shortly after completion of each irradiation are shown in Figure 7. The measurements revealed that demagnetization in excess of 97% has occurred at the geometrical center of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets and in excess of 82% near the edges at absorbed dose levels as low as 50 MRad. Similar reduction was measured for both the positive and the negative surfaces. Important to note in Figure 7 is the demagnetization of magnet 3A which received

1.2 GRad dose while irradiated at Position B (see Figure 3a). During irradiation of magnet 3A the neutron moderation induced by the volume of cooling water in front of the magnets being irradiated was removed which changed the fast-to-thermal neutron ratio in favor of fast neutrons. As seen in Figure 7, for both the geometric center and the edges of the magnet the demagnetization is affected indicating the sensitivity of the magnetic material and in particular the boron content to the neutron energy. $\text{Nd}_2\text{Fe}_{14}\text{B}$ is 26.7% neodymium, 72.3% iron and only 1% boron by atomic weight. While boron is only 1% it is considered to be a dominant demagnetization factor due to the very large neutron capture cross section in its stable isotopes (3.8 kbarn in ^{10}B compared to 36.6 b in ^{59}Co). The role of boron in the demagnetization observed will be discussed further in a subsequent section.

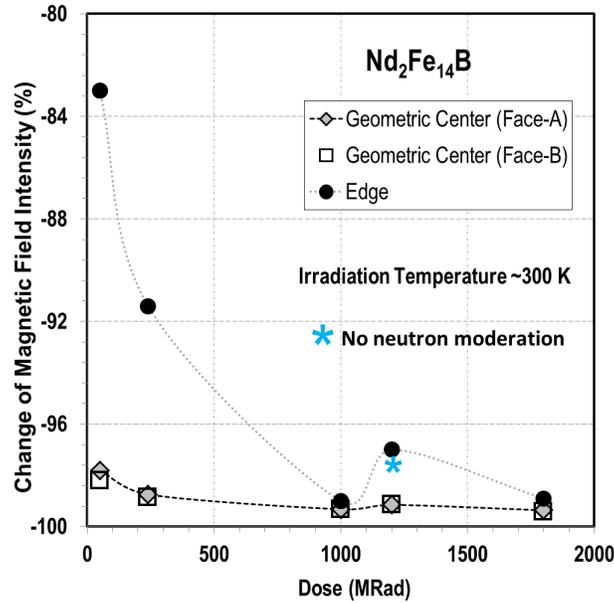


Figure 7: Demagnetization of $\text{Nd}_2\text{Fe}_{14}\text{B}$ block magnets in a mixed irradiating field.

Cited past studies [2, 3, 4] have shown that demagnetization attributed to electron and gamma fluences only accounts for a few percent and therefore the dramatic loss observed in the present study is safely assessed to be the result of neutron irradiation. Figure 8 depicts the variation of the magnetic field from the geometric center to the edges of the magnet and the demagnetization measured following 1.8 Grad absorbed dose (maximum dose of the magnet array). A similar magnetic flux intensity distribution was reported [5]. Following a 4-year cool-down period magnetic fields were re-measured using the same measuring procedure and probe. No significant recovery or change in the magnetic field intensity measured promptly after irradiation was observed.

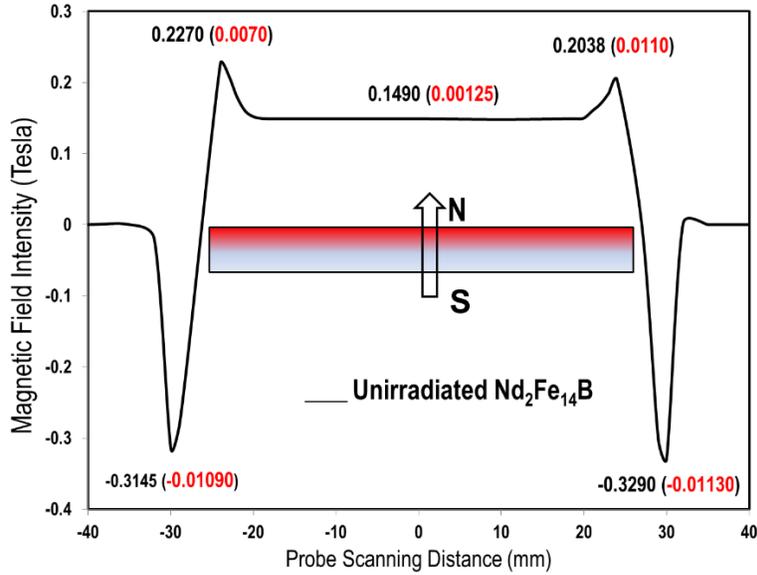


Figure 8: Magnetic field intensity distribution across the length of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnet pre- and post-irradiation to 1.8 GRad absorbed dose (magnet 11A). Values in parentheses are post-irradiation measurements.

3.2 Rectangular $\text{Pr}_2\text{Fe}_{14}\text{B}$ Magnets

Results obtained by scanning the pairs of plate-shaped $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets shown in figure 1b following irradiations with 920- and 447 μA -hours of primary proton beam (20 MRad and 10 MRad respectively) revealed dramatic flux losses. Specifically the smaller size samples lost $\sim 96\%$ of the pre-irradiation flux after 10 MRad and $\sim 98\%$ after 20 MRad. The larger magnets (53 CR) lost $\sim 89\%$ of the flux after 10 MRad and $\sim 93\%$ after 20 MRad. The field loss in these magnets was based on the flux intensity measured at the geometric center and the average of several measurements. Similarly with the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets the pre-irradiation flux increased near the edges of the block (i.e. from ~ 0.136 T at the center to ~ 0.175 T near the edges).

Based on the post-irradiation results it is evident that the Pr-Fe-B magnets are more sensitive to radiation than the Nd-Fe-B counterparts. While the irradiation temperature of the $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets was higher due to the experimental configuration during irradiation, but still well below the Curie temperature, the irradiation temperature alone does not explain the flux loss measured under similar irradiating field. As noted in section 2.1 the difference stems from the demagnetization mechanism which is driven by the fact that Pr material has lower coercivity than its Nd counterpart.

3.3 Annular $\text{Sm}_2\text{Co}_{17}$ and $\text{Nd}_2\text{Fe}_{14}\text{B}$ Magnets

Three (3) ferrofluidic rotary feedthrough units (FFFT) containing annular $\text{Sm}_2\text{Co}_{17}$ and $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets were irradiated to dose levels of 2 GRad and 200 MRad for the two $\text{Sm}_2\text{Co}_{17}$ type FFFT and 20 MRad for the $\text{Nd}_2\text{Fe}_{14}\text{B}$ type FFFT [20]. These values represent peak doses occurring at the upstream face of the annular magnet where the irradiating field is more intense (as shown in Figures 6) resulting from the orientation of the magnet relative to the impinging field. The flux and fluence of the different irradiating species for the 2 GRad FFFT with the $\text{Sm}_2\text{Co}_{17}$ magnet were estimated to be as follows:

- Proton flux 1.2×10^8 p/cm²-s and proton peak fluence 1.14×10^{14} p/cm²
- Neutron flux 8.6×10^{10} n/cm²-s and peak neutron fluence 8.17×10^{16} neutrons/cm²
- γ flux 1.31×10^{11} g/cm²-s with peak γ fluence 1.24×10^{17} /cm²

- Electron flux $2.85 \times 10^9 \text{ e/cm}^2\text{-s}$ and electron fluence $2.7 \times 10^{15} \text{ e/cm}^2$

Mapping of the magnetic field intensity was conducted by moving the Hall probe along the outer shell of the FFFT at a distance of $\sim 2 \text{ mm}$ above the surface. Essentially the probe travelled parallel to the magnetization field of the annular magnet (see Figure 2). Figure 9 depicts pre- and post-irradiation flux intensity for the annular magnets tested. The findings confirm previously observed results [11], regarding the resistance of Sm-Co-type magnets to demagnetization by irradiation. A small flux loss is experienced in the Sm-Co magnets even at the 2 GRad dose. It should be stressed that measurements of the magnet fluxes is not ideal given that magnet is encased within the FFFT stainless steel shell and therefore the changes recorded are not exact and should be only used for reference. Based on results of the current study on block Nd-Fe-B magnets and of past studies [16] greater loss of flux would have been expected for 20 MRad absorbed dose. The observed insensitivity of the annular Nd-Fe-B magnet is assessed to be the result of the particular shape and the magnetization direction (parallel to the thin direction or parallel to the axis). As explained in more detail in the summary section, the low permeance part of the magnet where inherent demagnetizing forces are higher (central section) is removed in an annular-shaped magnet rendering more radiation tolerant.

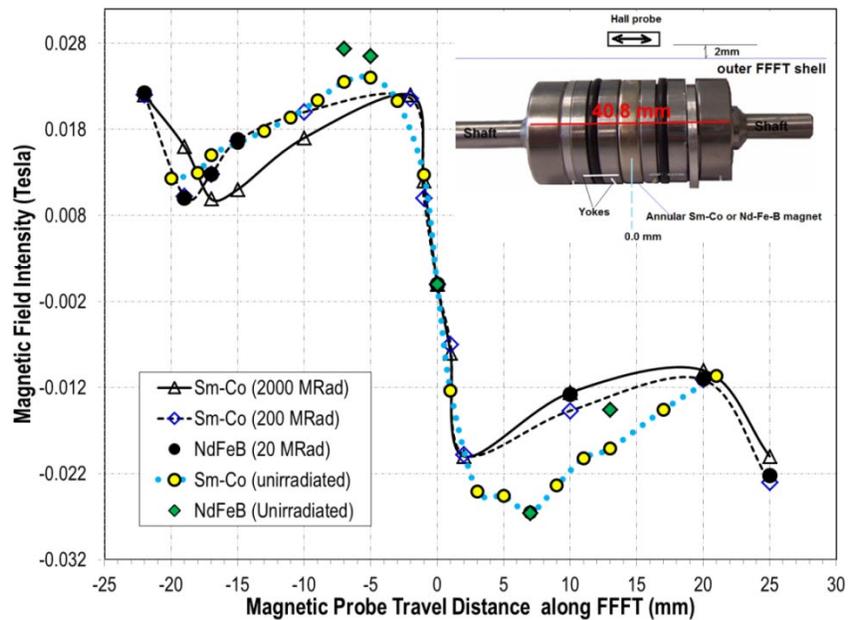


Figure 9: Measured magnetic field intensity of irradiated FFFTs

4. Discussion and Conclusion

Sensitivity of $\text{Nd}_2\text{Fe}_{14}\text{B}$ to neutron energy and Boron effect: In order to qualify the sensitivity of the plate-like Nd-Fe-B magnets to the neutron energy (i.e. the ratio of fast to thermal neutrons) one of the magnets was irradiated to 1.2 GRad at Position B. As shown in Figure 4a which depicts calculated neutron spectra at the two locations, the effect of the volume of water in the path of the irradiating flux in front of the magnet is noticeable. An increase in the thermal part of the spectrum is noted as a result of fast neutron moderation. This resulted into a noticeable effect in the demagnetization sensitivity. In particular, the presence of faster neutrons in the irradiation field (Position B) resulted in reducing the flux loss as clearly depicted in Figure 7. This is in agreement with findings in [15] where it was verified that the magnetic flux decayed faster for the fast plus thermal neutrons than fast neutrons alone. To further explore potential differences in the nuclear response of the magnets exposed to these two differing fields, photon spectra

were collected using a high-sensitivity ORTEC Ge detector. While similar photon spectra were obtained for the magnets irradiated at Position A, differences are noted in the spectra of the irradiated Nd-Fe-B magnet at Position B exposed to more fast neutrons. Figure 10 compares the spectra of the Nd-Fe-B magnet irradiated to 1.8 GRad (magnet 11A) at Position A with the spectra of the Nd-Fe-B magnet irradiated to 1.2 GRad (magnet 3A) at Position B. It should be noted that, while there is 100% agreement in the major photopeaks, distinct differences in the form of new peaks provide some evidence of potential influence the irradiating field at positions A and B may have in the demagnetization response.

The weak differences and change of the demagnetization trend observed in Figure 7 for magnets 11A and 3A may stem from their position in the mixed field during irradiation and be affected by the thermal to fast neutron ratio. Irradiations, however, to lower doses and thus lower demagnetizations, which in turn will amplify these potential differences and confirm the neutron energy effects, need to be conducted in new studies. The severe loss of flux in Nd-Fe-B and Pr-Fe-B magnets as compared to the Sm-Co counterparts is attributed to the boron content. The effect of the *thermal* neutron irradiation comes from the $^{10}\text{B}(n,\alpha)^7\text{Li}$ capture reaction on the 19.8% isotope and the ensuing cascade defects in the Nd-Fe-B or Pr-Fe-B materials of the energetic charged particles created. Large lattice temperatures result locally exceeding the Curie temperatures of these boron containing magnet materials, leading to nucleation of the magnetized grains.

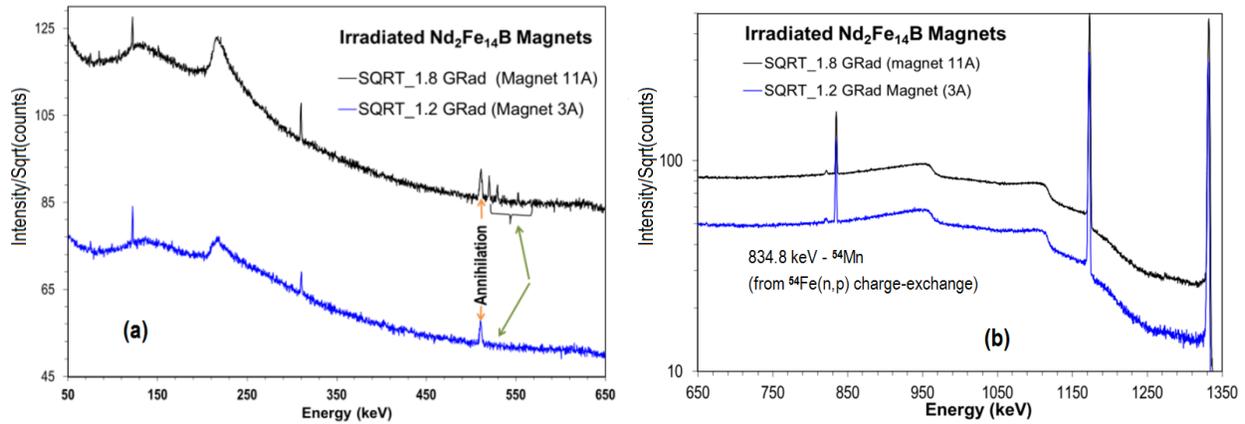


Figure 10: Photon spectra of magnets irradiated in two positions of the mixed field. (a) 50-650 keV and (b) 650-1350 keV energy ranges

Temperature Dependence: It has been shown in several studies [9-12] that radiation temperature plays a significant role in the demagnetization process. This has been established for Nd-Fe-B and Sm-Co magnets which exhibit temperature sensitivities over different regimes (≥ 20 K for Nd-Fe-B and ≥ 300 K for Sm-Co). The effect of irradiation temperature on Pr-Fe-B magnets has yet to be established in detail through measurements. In the experiments conducted, Pr-Fe-B magnets were irradiated at an estimated temperature of 80-100°C which is assumed to have contributed to the dramatic magnetic field loss measured for these magnets. Additional experiments with varying irradiation temperature are envisioned for the Pr-Fe-B magnets. In all conducted experiments where Nd-Fe-B, Pr-Fe-B and Sm-Co type magnets were exposed to neutron-dominated mixed field, the temperature remained below the respective Curie temperatures.

Shape Dependence: The current study confirmed, on the basis of the demagnetization behavior of Nd₂Fe₁₄B magnets, that shape plays a dominant role. Plate-like Nd₂Fe₁₄B magnets exhibited high sensitivity to irradiation while annular-shaped Nd₂Fe₁₄B magnets have shown insensitivity. The shape effect may be explained as follows: a plate like magnet magnetized parallel to the thickness or thin dimension will have a field with a “hole” in the middle as shown in Figure 8. The thinner the magnet the more pronounced the hole. As the magnet gets thicker the field profile will be showing a peak in the middle instead of a hole [5].

This effect is caused by the demagnetization forces that “want” to minimize the overall magnet moment. For two identical magnets with the same remanence, but different coercivity, the one with the highest H_c will have a shallower hole. In an annular or ring magnet magnetized parallel to the thin dimension, a large part of the demagnetization associated with the low permeance part has been removed thus rendering the permanent magnet more radiation tolerant.

The differences observed in the demagnetization of plate-like $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnets (53 CR grade vs. 47 CR grade) are not dramatic or beyond experimental measurement errors to attribute it to the L/W shape factor. It is assessed that the small demagnetization variation is the result of the $\text{Pr}_2\text{Fe}_{14}\text{B}$ magnet grade.

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