STUDY OF THE RADIATION DAMAGE OF Nd-Fe-B PERMANENT MAGNETS

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SUMMARY

Nd-Fe-B permanent magnets are highly desirable for use in the insertion devices of synchrotron radiation sources due to their high remanence, or residual magnetic induction, and intrinsic coercivity. However, the radiation environment within high-energy electron storage rings necessitates the determination of the degree of radiation sensitivity as well as the mechanisms of radiation-induced demagnetization. A 0.5% change in the residual induction due to radiation damage cannot be tolerated in these devices. Sample Nd-Fe-B permanent magnets were irradiated at the Advanced Photon Source (APS) with bending magnet x-rays up to an absorbed dose of approximately 280 Mrad (1 Mrad = 10 kGy). Sample magnets were also irradiated with ^{60}Co γ-rays up to an absorbed dose of 700 Mrad at the National Institute of Standards and Technology's (NIST) standard gamma irradiation facility. Changes in the residual induction were found to be within the experimental uncertainties for both the x-ray and γ-ray irradiations. Sample Nd-Fe-B permanent magnets were then irradiated at Oak Ridge National Laboratory's (ORNL) Californium User Facility for Neutron Science with fast neutrons up to a total fast fluence of 1.61×10^{14} n/cm^2 and with thermal neutrons up to a total thermal fluence of 2.94×10^{12} n/cm^2. The fast-neutron irradiation with a ^{252}Cf spontaneous fission source revealed significant changes in residual induction of the sample magnets.

I. BACKGROUND

The Advanced Photon Source (APS), as well as other third-generation synchrotron light sources, uses permanent magnets in the insertion devices to produce x-rays for scientific research.[1,2] The APS specifically uses Nd-Fe-B permanent magnets in the insertion devices.[2] When placed in a high-energy storage ring, these permanent magnets are subjected to irradiation from synchrotron radiation, high-energy bremsstrahlung, and bremsstrahlung-produced neutrons. Previous investigations have exhibited varying degrees of degradation in the magnetization of these permanent magnets.[3,4,5,6,7]

The potential for irradiation of the Nd-Fe-B permanent magnets used in the APS insertion devices results from the proximity of the electron beam to the magnets themselves. During normal operation, the magnets will be as close as 3-5 mm from the electron beam. This proximity increases the potential of magnet irradiation from synchrotron radiation, high-energy bremsstrahlung, and photoneutrons produced by bremsstrahlung.

II. RESOURCES REQUIRED FOR IRRADIATION

A. Nd-Fe-B Sample Permanent Magnets

The specific Nd-Fe-B composition of the permanent magnet samples used in this investigation is a typical commercially available material known as N38H. The sample magnet dimensions were approximately 5 cm x 4.75 cm x 0.7 cm. The specific material and dimensions of the sample permanent magnets used in this particular study were primarily chosen based on similarity to the permanent magnets used in the APS insertion devices. All of the sample magnets were plated with a thin coating of nickel to alleviate chipping of the Nd-Fe-B magnetic material.

B. High-Dose Dosimetry

A high-dose dosimetry technique using radiachromic films was considered, tested, and calibrated at the APS through a collaboration with

NIST, for use during the x-ray irradiation. These radiachromic films were used to measure the absorbed irradiation doses received by the sample permanent magnets during the x-ray irradiation portion of the study. NIST used alanine dosimeters to determine the irradiation dose received by the sample permanent magnets during the γ-ray irradiations. To determine the neutron fluences obtained during the neutron irradiations at ORNL, an Indium foil activation technique was used.

C. Magnetic Measurement

Measurement of the degree of degradation in the magnetization of the Nd-Fe-B permanent magnets as a result of irradiation required a magnetic measurement system. This system was comprised of an eight-inch Helmholtz coil and a fluxmeter. Helmholtz coils were found to be ideal for the measurement of rare earth (samarium cobalt, neodymium) and hard ferrite permanent magnets. The Helmholtz coil measures the magnet sample as a single magnetic moment provided that the magnet sample's longest dimension is less than one third of the diameter of the coil.[8,9]

When the Helmholtz coil is used in conjunction with the fluxmeter, the intrinsic flux density B_{di} of a magnet sample can be obtained.[8] The intrinsic flux density B_{di} is defined as the magnetic moment per unit volume and is found by multiplying the initial reading from the coil by the Helmholtz coil constant and then dividing by the volume of the magnet sample. The particular fluxmeter used in this study automatically performed the calculation giving a final reading in units of B_{di}-Tesla. For a given magnet material and magnet geometry, the B_{di} value of a magnet is proportional to the residual induction B_r of the magnet material, which is also measured in units of Tesla. As a result, changes in the measured B_{di} value following irradiation will be seen in proportional changes to the actual residual induction B_r of the magnet sample.[10]

D. Temperature Measurement

Two aspects of the irradiation required temperature control and measurement, namely the radiachromic films and the sample magnets themselves. The main disadvantage of Nd-Fe-B permanent magnets is their low Curie temperature. This leads to increased sensitivity to thermal demagnetization and limits their maximum temperature range. The maximum use temperature for commercially available permanent magnets is typically less than 120°C.[11] In order to hinder temperature-induced demagnetization so as not to interfere with the radiation-induced demagnetization results, the temperature had to be kept below 60°C for the γ-ray irradiation and below 50°C for the x-ray irradiation.

All magnetic measurements were conducted at an ambient lab temperature of 20 ± 1°C. The magnetization of Nd-Fe-B permanent magnet material has a temperature coefficient of –0.15%/°C at room temperature.[9] Therefore, a room temperature variation of ± 1°C could account for a 0.15% reversible change in residual induction measurements. However, a 0.15% change falls within the measurement uncertainties associated with the fluxmeter, discussed previously. As a result, this study only considered the irreversible magnetization changes associated with temperature fluctuations.

III. X-RAY IRRADIATION OF THE SAMPLE MAGNETS

A. Measurement Approach

An APS beamline was used for the systematic x-ray irradiation of the Nd-Fe-B sample magnets. The sample magnets were placed in a specially designed holder inside the experimental hutch and irradiated with an x-ray spectrum with a peak energy of approximately 40-50 keV from the bending magnet source in Sector 9 of the storage ring. Seven sample magnets were irradiated to various absorbed doses, ranging from 30 Mrad to approximately 280 Mrad, during multiple trials.

The magnet irradiation configuration, placed inside the experimental hutch, consisted of a water-cooled magnet holder and a water-cooled aluminum spreader. The configuration was placed on a pedestal, which allowed for alignment of the sample magnet to the height of the beam window. A 1-cm-thick aluminum spreader was placed directly after the beam window in the experimental hutch, between the window and the magnet holder. The purpose of the aluminum spreader was to diffuse the dose received by the sample magnet, allowing a more uniform irradiation of the magnet. A PHOTON[12] calculation (Figure 1) was done to determine the bending magnet radiation spectrum that was used to irradiate the sample magnets at an APS beamline. PHOTON calculation showed that the 1-cm-thick spreader alleviated some of the heat load to the magnet by cutting the peak flux to the sample magnet from 10^{16} photons/s/keV to 10^{14} photons/s/keV.

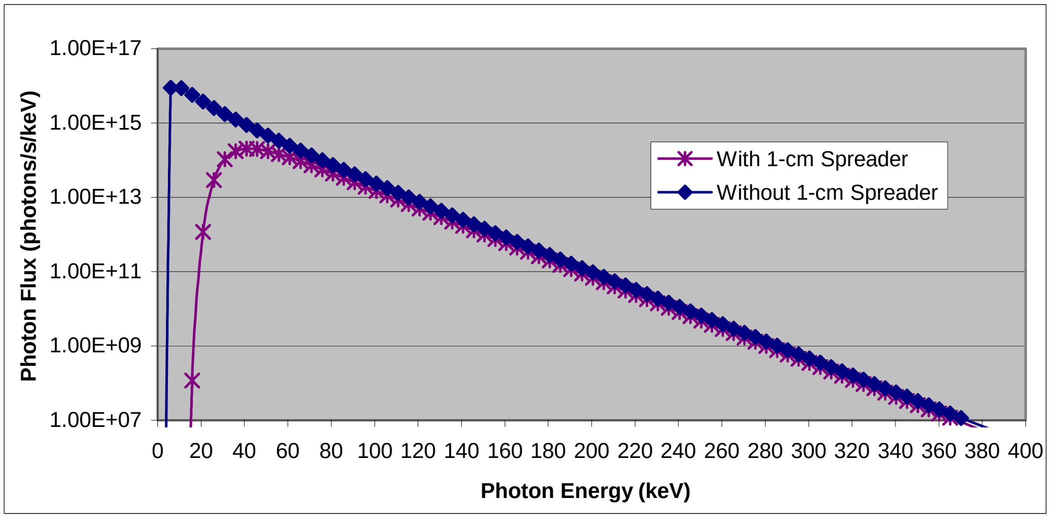

Figure 1: Calculated bending magnet irradiation spectrum for an APS beamline as a result of PHOTON simulation.

The magnet holder was placed directly after the aluminum spreader. The temperature during the irradiation was kept below 50ºC.

Along with temperature stabilization, a preliminary dose rate had to be determined prior to irradiation of the sample magnets. Radiachromic films were used to determine an approximate dose rate to the sample magnet, which was normalized, or based on the irradiation time and the beam current. Radiachromic films were placed on the top and the bottom of a sample permanent magnet that was then placed inside the magnet holder and irradiated for a specified period of time. The average normalized dose rate was determined to be approximately 0.15 Mrad/h/mA by taking an average of normalized dose rates measured using radiachromic films. This dose rate was used to determine the total absorbed dose to the magnet for extended periods of irradiation.

Seven sample magnets were irradiated with x-rays at an APS beamline to various absorbed doses ranging from approximately 30 Mrad to 280 Mrad. Two of the sample magnets were irradiated using a "stepwise" irradiation procedure of three, three-hour irradiations, resulting in a total irradiation time of nine hours. The other sample magnets were irradiated as one continuous irradiation. The absorbed doses to the sample magnets were determined using the 0.15-Mrad/h/mA normalized dose rate approximation, due to the response limit of radiachromic films at dose levels greater than 10 Mrad.

Following irradiation for the specified period of time, the magnet was removed and allowed to cool to ambient temperature in the lab. The radiachromic films were removed and read to determine an approximate absorbed dose to the sample magnet. Residual induction measurements were then taken for the sample magnet and the control magnets. The average of these "final" readings was compared to the "initial" readings taken prior to irradiation to determine if any measurable radiation-induced demagnetization had occurred. This process was then repeated for the next irradiation.

B. Results and Analysis

Residual induction measurements for each sample magnet prior to x-ray irradiation compared to measurements following irradiation revealed an average 0.2% decrease in the residual induction, which was found to be within the experimental uncertainties. Results shown in Figure 2 give the deterioration of the residual induction as a percent change for each irradiation step.

As stated previously, a 0.5% change in the magnetic field of insertion devices typically cannot be tolerated. Therefore, a 0.2% change would not be considered insignificant. The fact that the 0.2% change only occurred in the sample magnets and not in the control magnets also indicates a significant change. However, a 0.2% decrease in the residual induction is within the experimental uncertainties. Therefore, the observed changes cannot be solely attributed to radiation-induced demagnetization due to experimental

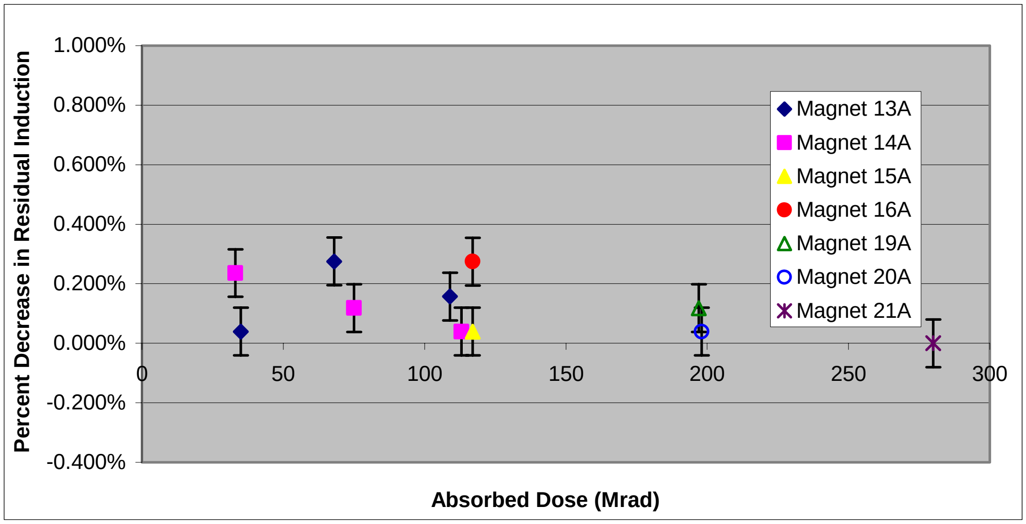

Figure 2: Percent decrease in residual induction of the irradiated sample magnets from APS bending magnet x-ray irradiation.

uncertainties, including precision limitations and reversible temperature effects during magnet measurements.

IV. γ-RAY IRRADIATION OF THE SAMPLE MAGNETS

A. Measurement Approach

The NIST standard gamma irradiation facility, a Cobalt-60 irradiation facility, was used to irradiate the Nd-Fe-B sample permanent magnets. Four sample magnets along with two control magnets were sent to NIST numerous times for a total of six irradiations.

For each of the first five irradiations, the four sample magnets were irradiated in absorbed dose increments of 100 Mrad. The magnets were irradiated in an absorbed dose increment of 200 Mrad for the sixth irradiation. The six γ-ray irradiations resulted in a total absorbed dose to the sample magnets of approximately 700 Mrad.

As stated previously, the temperature during the γ-ray irradiation was held constant within the irradiation facility, which helped to control any irreversible temperature-induced demagnetization of the sample magnets. The temperature was kept at approximately 30ºC with air blown down onto the samples. This was well below the 60ºC temperature requirement for the magnets. The high-dose dosimetry component of the irradiation was also satisfied by the NIST irradiation facility through the calibrated Gammacell. Calibrated radiachromic films, in pouches, were placed between the magnets for a three-hour irradiation to verify an absorbed dose rate of 708.6 krad/hr.

Prior to sending the magnets to NIST for irradiation, residual induction readings were taken at the APS for each magnet. Four sample magnets and two control magnets were read using the magnetic measurement system previously described. The average of five measurements served as initial readings to be compared to similar measurements taken at the APS following each irradiation.

B. Results and Analysis

Residual induction measurements for each magnet prior to γ-ray irradiation of the sample permanent magnets compared to measurements following irradiation revealed an average percent decrease in the residual induction of 0.1 - 0.2%, which was found to be within the experimental uncertainties. Results shown in Figure 3 give the deterioration of the residual induction as a percent change for each irradiation step as compared to the percent change observed in the unirradiated control magnets. Because high accuracy is required in the magnetic fields of insertion devices, changes of this magnitude may cause diminished performance. Typically a 0.5% change in the magnetic field of insertion devices cannot be tolerated. However, the 0.1 - 0.2%

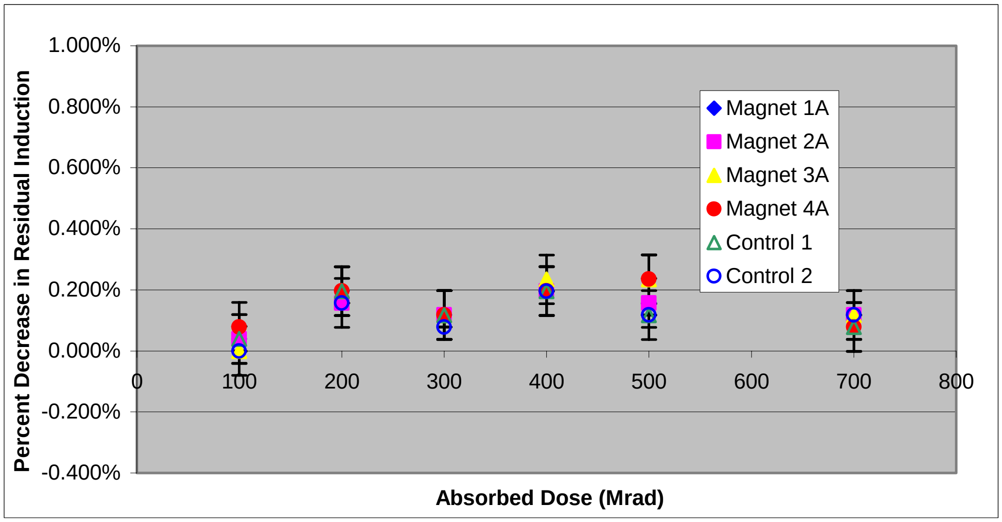

Figure 3: Percent decrease in residual induction of the irradiated sample magnets from ^{60}Co γ-ray irradiation at the NIST standard gamma irradiation facility.

changes observed in this case are within the experimental uncertainties previously discussed and could have been induced by reversible temperature demagnetization effects. Therefore, the changes in residual induction are not statistically significant and cannot be attributed to radiation-induced demagnetization of the sample magnets.

V. NEUTRON IRRADIATION OF THE SAMPLE MAGNETS

A. Measurement Approach

Neutron irradiation of the Nd-Fe-B permanent magnets was done in collaboration with Oak Ridge National Laboratory's (ORNL) Californium User Facility for Neutron Science. Sample permanent magnets were systematically irradiated with ^{252}Cf sources to incremental fluence levels in a fast-neutron spectrum, as well as a thermal-neutron spectrum. ORNL's Californium User Facility for Neutron Science was a user-friendly neutron irradiation facility that allowed for ideal irradiation configurations and experimental conditions. With a neutron emission rate of 2.31×10^6 neutrons/s/μg and a peak in the energy spectrum at 0.7 MeV (2.1 MeV average neutron energy), ^{252}Cf proved to be an ideal source for both fast-neutron and thermal-neutron irradiation of the sample magnets.

Irradiations were performed inside a hot cell. High-density concrete served as the hot cell's outer shielding, while a window of lead glass and water allowed for visual inspection of the irradiation configuration from outside the hot cell. The temperature during the irradiation was held constant within the hot cell due to the air-conditioned building and the large amount of thermal inertia within the cell. All temperature measurements within the hot cell were on the order of 25°C. This allowed for the control of any temperature-induced demagnetization of the sample magnets. An area was set up just outside of the hot cell's viewing window to check for activation of the sample magnets as they were removed from the cell. Another area was set up outside of the entrance to the hot cell for magnetic measurement of the sample and control magnets.

Fluence estimates were calculated prior to irradiation using the Monte Carlo N-Particle Transport Code System (MCNP)[13] and were based on source configuration, ^{252}Cf content, and total irradiation time. The calculated MCNP fluxes were compared to actual flux measurements that were done using activation detectors, in order to calibrate the MCNP results. Indium wires were irradiated in a fast-neutron spectrum, a thermal-neutron spectrum, and an intermediate-neutron spectrum. Wires were irradiated in the fast-neutron spectrum for approximately 24 hours using 46 mg of ^{252}Cf. In the thermal neutron spectrum Indium wires were irradiated for approximately six days using 49 mg of ^{252}Cf, while in the intermediate spectrum wires were irradiated for approximately 22 hours using 49 mg of ^{252}Cf. Following each irradiation, the wires were analyzed to determine the measured fast-neutron as well as the

Table 1: Flux calibration results for Indium wire activation measurement vs. MCNP simulation

Irradiation Spectrum	Thermal Flux Component		Fast Flux Component (E>0.1 MeV)	
	Measurement	MCNP Simulation	Measurement	MCNP Simulation
Fast-Neutron	4.8E+5 n/cm^2/s	1.1E+6 n/cm^2/s	4.9E+8 n/cm^2/s	4.3E+8 n/cm^2/s
Thermal-Neutron	1.0E+6 n/cm^2/s	3.0E+6 n/cm^2/s	7.3E+5 n/cm^2/s	4.3E+5 n/cm^2/s
Intermediate-Neutron	2.1E+7 n/cm^2/s	2.5E+7 n/cm^2/s	2.5E+7 n/cm^2/s	4.8E+6 n/cm^2/s

thermal-neutron flux component of the spectrum by analyzing ^{115m}In and ^{116}In activities. The measured fluxes were then compared to the fluxes that were calculated using MCNP (Table 1).

Prior to irradiation, residual induction readings were taken for each of the sample magnets along with the control magnets. The control magnets were set aside, away from any potential irradiation sources, while the sample magnets to be irradiated were placed inside the hot cell for irradiation. Four sample magnets along with two control magnets were used for the fast-neutron irradiation. Only two sample magnets and one control magnet were used in the subsequent thermal-neutron irradiation.

Following an irradiation, the sample magnets were removed from the hot cell via the transport chute, and checked for activation. Residual induction readings were taken for each of the sample magnets along with the control magnets. Ten readings were taken for each magnet. The average of these readings served as the final readings that were compared to the "initial" readings taken prior to irradiation. The comparisons were used to determine if the neutron irradiation had resulted in radiation-induced demagnetization of the sample magnets. Once all of the final readings were taken, the magnets were placed back inside the hot cell for the next irradiation in the cycle at a higher fluence level. The sample magnets were flipped over for every irradiation so that each face of the magnets faced the ^{252}Cf sources during every other irradiation. This allowed for a more uniform irradiation of the magnets.

The experimental procedure for the thermal-neutron irradiation was identical to the fast-neutron irradiation procedure. Approximately 22 cm of polyethylene moderation was placed between the ^{252}Cf source and the sample magnets for the thermal-neutron irradiation in order to thermalize the neutron spectrum. Polyethylene blocks were also placed around the entire thermal-neutron irradiation configuration to maximize the neutron flux.

The fast-neutron irradiation consisted of a total of eight irradiations. For each of the first six irradiations, four sample magnets were irradiated in two-decade total fluence increments ranging from approximately 10^6 n/cm^2 to 10^{14} n/cm^2. The final two irradiations were done using a second set of four sample magnets. This second set of magnets was irradiated to total fluences of approximately $2x10^{13}$ n/cm^2 and $4x10^{13}$ n/cm^2. The thermal-neutron irradiation consisted of a total of thirteen irradiations. The sample permanent magnets were irradiated in total fluence increments ranging from approximately 10^6 n/cm^2 to 10^{12} n/cm^2.

B. Results and Analysis

Residual induction measurements for each magnet prior to fast-neutron irradiation compared to measurements following irradiation revealed significant changes for fluence levels greater than $2x10^{13}$ n/cm^2. The results in Figure 4 give the deterioration of the residual induction as a percent change for each irradiation step as compared to the percent change observed in the unirradiated control magnets.

During irradiation of the first set of sample permanent magnets in the fast-neutron spectrum, significant changes were not evident until a fluence level of approximately $1.61x10^{14}$ n/cm^2. At this point, the differences between the initial residual induction measurements and the final measurements were well over 10% (see Figure 4), while the control magnets showed no signs of change. Due to the very large gap between the

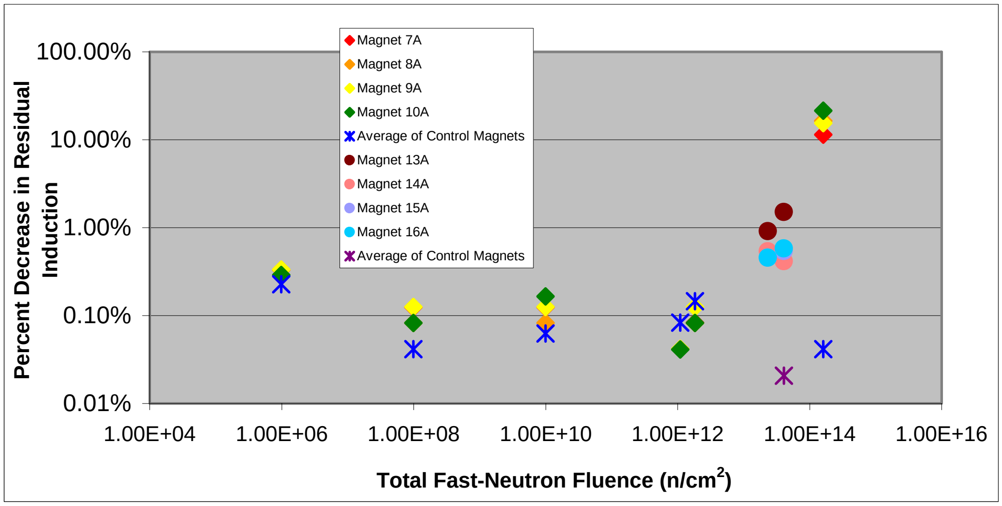

Figure 4: Percent decrease in residual induction of the irradiated sample magnets from fast-neutron irradiation at ORNL's Californium User Facility for Neutron Science.

1.61×10^{14} n/cm^2 point and the previous data point at 1.83×10^{12} n/cm^2 where statistically insignificant changes were indicated, it was determined that more points were needed. Because this particular set of magnets was already damaged at the 10^{14} fluence level, another set of magnets was irradiated twice, which resulted in data points at approximately 2.33×10^{13} n/cm^2 and 4.07×10^{13} n/cm^2. Both of these points indicated significant changes in the residual induction of the sample magnets. An average percent difference of 0.6% between the initial and final residual induction measurements was found at the 2.33×10^{13} n/cm^2 fluence level, and an average percent difference of 0.8% was found at the 4.07×10^{13} n/cm^2 fluence level.

A 0.5% change in the magnetic field of the insertion devices typically cannot be tolerated. The results of this study indicate that for fast-neutron fluences of 2.33×10^{13} n/cm^2, the 0.5% level of change is reached, and the percent change increases with rising fluence. From the present investigation we also know that annealing does not occur, as the damage does not cure itself over time. Residual induction measurements of the damaged magnets were taken two days after the damage occurred and again two and a half weeks after the damage occurred. The results of these measurements showed no change in the residual induction immediately following damage, after two days, and two and a half weeks later. The fact that annealing effects do not exist indicates that the damage is permanent and most likely related to either transmutation or permanent dislocation as a result of neutron interaction with the magnetic material.

At an irradiated total thermal-neutron fluence of 3.34×10^{12} n/cm^2 no measurable radiation-induced demagnetization of the sample permanent magnets was observed (Figure 5). Total thermal fluences greater than 3.34×10^{12} n/cm^2 could not be reached due to facility and time limitations.

VI. CONCLUSIONS

This study attempted to quantify the radiation-induced demagnetization of Nd-Fe-B permanent magnets from γ-rays, x-rays, and neutrons. Due to the many factors involved in the determination of radiation-induced demagnetization of Nd-Fe-B permanent magnets, along with great number of variations within each experimental procedure, it is difficult to make comparisons between investigations. The important point for each study is that a 0.5% change in the magnetic field of the insertion devices used in many synchrotron light sources typically cannot be tolerated. Therefore, even small variations of 0.1 - 0.2% are important as long as they can be determined statistically significant.

While only the fast-neutron irradiation results in this investigation could be determined statistically significant, this study extends beyond the scope of previous investigations. This investigation provided the first complete study of the radiation-

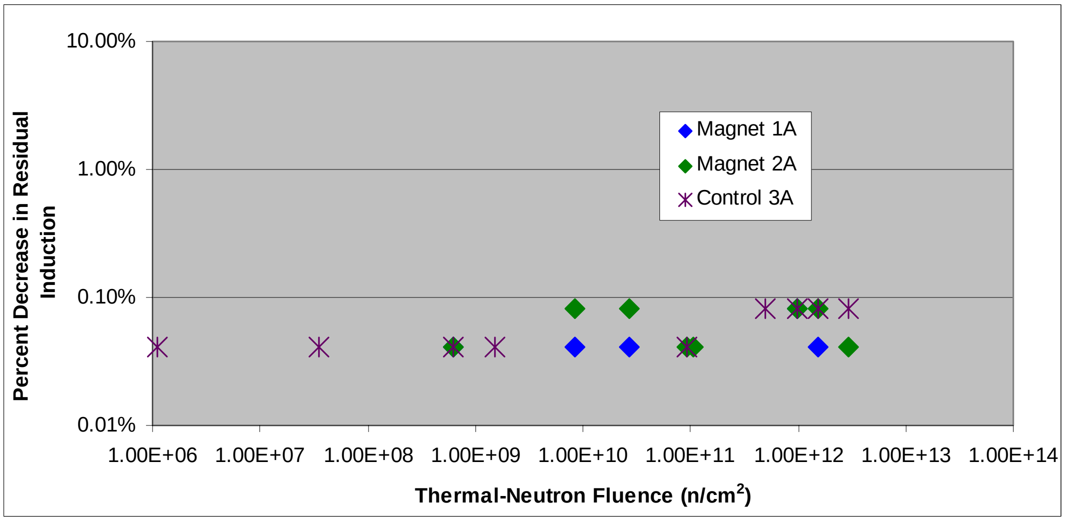

Figure 5: Percent decrease in residual induction of the irradiated sample magnets from thermal-neutron irradiation at ORNL's Californium User Facility for Neutron Science.

induced demagnetization of Nd-Fe-B (N38H) sample magnets from three types of radiation that the magnets are subjected to under normal operating conditions within synchrotron radiation sources. Specific fast-neutron irradiation as well as thermal-neutron irradiation was conducted. In the present investigation ^{60}Co irradiation went as far as 700 Mrad. The first investigation of the radiation-induced demagnetization of Nd-Fe-B sample magnets using a pure x-ray source was conducted. This work also considered temperature control during neutron irradiation as an important factor. The neutron irradiations were nonreactor-based irradiations, allowing for more controlled conditions of irradiation similar to the conditions that the sample magnets are subjected to in synchrotron radiation sources.

The results of this study, along with previous investigations, indicate varying degrees of degradation in the intensity of magnetization of Nd-Fe-B permanent magnets based on the irradiation source and the total absorbed dose to the magnets, to name a few of the variants. Results from previous investigations displayed a wide variation in the sensitivity to radiation, dependent on the magnet material and manufacturing process, which varies between magnet vendors. Among the different Nd-Fe-B compounds, it was also found in previous studies that those with higher remanence and lower coercivity were more susceptible to demagnetization by radiation.

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