



**Radiation Induced Defects in High-Z Shielded
Ytterbium Doped Fibers**

THESIS

Diana Cruz, Major, USA
AFIT-ENP-MS-18-M-074

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DOPED FIBERS

THESIS

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Degree of Master of Science in Applied Physics

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Major, USA

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Abstract

Studies of amorphous silica fibers bombarded with neutral particles indicate that both photons and neutrons have the same mechanism for creating defects, however rate of creation and affected wavelengths for each type is unclear [38] [39]. Some studies indicate that photon radiation induced attenuation (RIA) is higher than neutron RIA, while others indicate the reverse due to secondary effects by neutrons. Additionally, it appears that the two neutral particle radiation sources produce defects at significantly different rates despite using the same dose rate [39]. In 2015, Borman irradiated Ytterbium-Doped Fibers (YDF) using both neutral particles and identified possible wavelength dependent defects by Gaussian deconvolution. It is difficult to positively attribute defects to one or the other when both are introduced in the sample concurrently. In this work, this issue is further explored in the current experiment by placing lead shielding at various thicknesses in the line of radiation from a nuclear reactor to the YDF, which is then exposed to neutrons and photons from a nuclear reactor source. Reducing photon fluence via various thicknesses of high-Z shielding materials, while maintaining the same neutron fluence, provides for a comparison of defect rates in YDF.

In comparing neutron dominant and γ dominant radiation sources for a similar total dose, the peak between 2-3 eV in the 6/125 YDF shifts approximately 0.4 eV (neutron radiation dominant dataset is left of the γ radiation dominant dataset), with a smaller defect peak deconvoluted around 3.2 eV in the neutron dominant radiation source results. The 20/400 YDF deconvolves to more Gaussian peaks, with a 0.1 eV shift for the 1-1.5 eV peak and a 0.15 eV shift for the 2-2.5 eV peak (leftward). Noticeably, the γ radiation dominant source data shows a peak around

1.6 eV significantly more pronounced than in the neutron radiation dominant source dataset.

Acknowledgements

Dedico este documento a mi sobrina, cuyo nacimiento me inspiró a continuar el camino hacia la iluminación. Espero que algún día leas esta dedicación y realices el impacto que tuviste al llegar a este mundo. Pro Deo et patria.

Diana Cruz

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List of Abbreviations

Abbreviation	Page
YDF	Ytterbium-Doped Fibers iv
EMP	Electromagnetic Pulse 1
SSL	Solid State Lasers 2
GL	Gas Lasers 2
LPL	Liquid-Phase Lasers 2
REDF	Rare Earth Doped Fiber 2
YDFL	Ytterbium Doped Fiber Lasers 2
RE	Rare Earth 2
LMA	Large Mode Area 3
RIA	radiation induced attenuation 4
RIE	radiation induced emission 4
RE	Rare Earth 6
DND	Direct Nanoparticle Deposition 12
OCVD	Outside Chemical Vapor Deposition 12
MCVD	Modified Chemical Vapor Deposition 12
EM	electromagnetic radiation 13
PE	photoelectric effect 13
CP	Compton scattering 13
PP	pair production 13
OSURR	Ohio State University Research Reactor 21
OD	outer diameter 24
UPS	Uninterruptible Power Supply 28

Abbreviation		Page
CCS	Compact CCD Spectrometers	29
XRF	X-ray Fluorescence Spectroscopy	32
SEM-EDX	Scanning Electron Microscope-Energy Deposition X-ray Spectroscopy	32
NAA	Neutron Activation Analysis	32

RADIATION INDUCED DEFECTS IN HIGH-Z SHIELDED YTTERBIUM DOPED FIBERS

I. Introduction

Our continued resolve to remain the prepotent nuclear power [91] in the world requires us to consider both the status of our nuclear inventory and the nuclear capabilities of other countries. Foreign nuclear capability drives the modification of our arsenal to ensure weapon survivability in the most extreme scenarios envisioned [8]. To do so, we must study nuclear weapons effects, especially in cutting edge technology. Several new technologies such as laser-based directed-energy weapons [88] require the use of fiber optics and associated equipment, and although we cannot test this equipment using a nuclear weapon, we can approximate the effects of a nuclear weapon by exposing fiber optics to other radiation sources, such as a nuclear reactor. Additionally, should we desire to monitor nuclear radiation in a reactor or, perhaps, an underground or surface nuclear test, updating our monitoring systems to include radiation sensitive materials, such as doped fibers, may be a means for detecting radioactivity just above background radiation.

1.1 Motivation

Researchers have known the benefits of replacing electrical cables with fiber optic cables for several decades [9]. A fiber optic cable does not suffer effects from electromagnetic radiation, ensuring that Electromagnetic Pulse (EMP) effects are negligible to non-charged carriers. In contrast, conventional cables may retain functional damage or circuit upset due to EMP [8]. This functional damage in conventional cables

may also facilitate the destruction of transmitters and receivers, as the increased voltage is not contained solely in the cable. Fiber optic cables may also transmit in standard telecommunication windows easily, ensuring wide bandwidth while also typically constructed an order of magnitude smaller than electrical cables [9]. Along with this advantage, the use of fiber optics to communicate over large distances with high bandwidth is a significant advantage over electronic communications materials [37].

Fiber optics also have an advantage as components of modern systems such as lasers. As of 2015, Solid State Lasers (SSL), Gas Lasers (GL), and Liquid-Phase Lasers (LPL) (dye) still dominate the type of gain medium and pumping scheme of lasers. One significant drawback of SSL is thermal management; improper management results in optical effects and possible fracture of the gain medium. Fiber optic lasers, an atypical subset of SSL, have the advantage of emission characteristics that are insensitive to temperature, and limited power emission may be avoided by using multiple fiber amplifiers in high power applications [88]. GL in general self-eliminate as portable weapon systems as they are large and require logistical support (e.g. disposal of chemicals after use and maintenance), not to mention the possibility of destruction on board, which may result in the platforms complete destruction. One particular class of optical fiber, the Rare Earth Doped Fiber (REDF), has advantages as a pumping mechanism in lasers.

Ytterbium Doped Fiber Lasers (YDFL) gained popularity in the early 2000s and in 2010 were considered the newest technology in the field of Rare Earth (RE) doped fiber lasers [66]. Not only does a fiber have a wide bandwidth, typical fibers cover the US military standard of 100/400. As a medium, YDFL in particular have a broad gain bandwidth from 975-1180 nm (optimal between 1060 nm-1100 nm), a broad absorption band (900-980 nm), a long lifetime (1 ms), and a high doping

concentration due to its 3-level structure [88]. The geometry of the fiber is also important in transmission. A significant milestone will be reached when researchers identify how to control nonlinear phenomena in lasers. One way to mitigate the effects of nonlinear phenomena is to use a Large Mode Area (LMA) fiber structure and a shorter fiber; research utilizing fibers of one meter or less with a LMA core is well-placed.

One particular area of interest for this subset of fiber is the effect of neutral radiation on YDF. Many studies include irradiation by photons or mixed irradiation by photons and neutrons, yet few focus on the role neutrons play in creating defects in YDF. Of concern is the idea that photon irradiation could mask particle-specific defect rates and affect total dose and dose rates to arrive at complete attenuation of the fiber. As Borman noted, both γ and neutron radiation produce defects in YDF, and the focus now is to experimentally determine the results of irradiating YDF with a constant neutron fluence while simultaneously modifying the fluence of γ radiation using high-Z shielding [6].

1.2 Research Objective

The objective of this research is to determine neutron radiation effects on YDF, and subsequently compare this data to γ radiation and mixed γ and neutron radiation [6]. Research questions supporting to this objective are:

1. Can we determine which defects present in YDF exposed to radiation are due to neutron emission?
2. Can we determine at what wavelengths YDF is most susceptible to radiation within the gain bandwidth?
3. Is there a minimum amount of neutron fluence necessary for defect generation?

If so, what defects appear and which type is the most significant contributor to radiation induced attenuation?

The objective and subsequent questions require a three-phase approach:

1. Determination of equipment necessary to optimize neutron radiation while maintaining sufficient transmission intensities for measurement.
2. Provide data for existing models and compare current data to previous experimental results. Begin monochromatic source use during radiation.
3. Modify existing models to fit experimental data and begin determination of neutron specific defects.

1.3 Problem Statement

Fiber resiliency is tested using measurements including radiation induced attenuation (RIA), radiation induced emission (RIE), and indicated changes in index of refraction (compaction) [21]. These three macroscopic measurements combined identify whether a fiber undergoes microscopic (defect generation) and/or macroscopic changes (change in output intensity). To further investigate defect generation, absolute characterization of the system should be determined. The fluence of the neutrons and charged particles must be such that defect generation, and thus change in light transmission intensity, is above the residual noise floor. Additionally, γ radiation must be significantly attenuated to be able to attribute defects to neutrons. In doing so, one may further illuminate defect generation and predict defect generation caused by sources of primarily neutron fluences that may not be easily achievable in a laboratory setting, but are a potential hazard to future defense related systems in exo- and endo-atmospheric applications.

1.4 Thesis Outline

This thesis is composed of five chapters, with Chapters 2 and 3 providing background with respect to nuclear physics, to include unit conventions, mechanisms of action, the relation between nuclear reactions and defect creation in fiber, and optical fiber characteristics. Chapter 3 focuses on the preparation of the fiber. Chapter 4 provides results and analysis of the data acquired while Chapter 5 includes a detailed discussion of the peak shifts observed between γ radiation and neutron radiation dominant sources as well as features unique to γ radiation dominant datasets. An explanation of the relative role of RIA between fibers is discussed as well. Additionally, a method of identifying these peaks by named defects is provided in the future work section.

II. Theory and Background

This chapter begins with an explanation of the primary instrument of experimentation, the optical fiber. Features of fiber, including a general explanation of the propagation of light, issues with propagation, and various accessories that reduce attenuation, are discussed, with a more detailed explanation in Appendix A. It is also important to understand which doping elements are responsible for increased performance over undoped, pristine fibers and the co-dopants necessary for Rare Earth (RE) doping. The effects of radiation (both γ and neutron radiation) on each of these elements and compounds follows with a separation by the typical total dose and dose rate of an event.

2.1 Behavior of Light

Werner Heisenberg first published in 1927 the Uncertainty Principle—despite learning that the transition from one discrete energy level to another has a definite value, the reality of the Uncertainty Principle requires scientists to consider each discrete energy level as having a minimum amount of uncertainty related to it, or measure the energy level for an infinite amount of time. Only one of these is practical. The fact that we cannot measure an energy level for an infinite amount of time has direct consequences in fiber. Even for “monochromatic” light, there exists a dispersion angle, since the uncertainty in energy is carried through to wavelength via the de Broglie relation and results in imperfect coherence. As light propagates in space, this imperfect coherence disperses the energy of the wave. This will come into play when the absorption properties of the experimental fiber are discussed.

For broad light sources, such as a white light source, there is no semblance of coherence and dispersion occurs at greater angles. In fiber, this dispersion is coun-

teracted by the periodic “refocusing” of light within the fiber. The propagation of light in fiber may be modeled by “zig-zag” analysis [95]. Zig-zag analysis evaluates light using geometrical optics. With the view of light as rays, we can trace a ray’s entrance into the fiber using Snell’s Law

$$n_0 \sin \theta_a = n_1 \sin \theta_{conf}, \quad (1)$$

where n_0 is the index of refraction of the cladding surrounding the core, n_1 is the index of refraction of the core, θ_a is the acceptance angle, and θ_{conf} is the confinement angle. As long as the ray enters within the acceptance angle, it will meet the minimum criteria for confinement. The critical angle, which is usually related to total internal refraction, is related to the confinement angle by

$$\theta_{crit} = 90^\circ + \theta_{conf}. \quad (2)$$

It is total internal refraction that allows light rays to hit the boundary between n_0 and n_1 and be reflected [37]. These angles require that the index of refraction of the core be larger than the index of refraction of the cladding that surrounds the core in a fiber. This difference need only be about 1% to be successful [37]. Numerical aperture (N.A.), which is used to describe fiber and is critical to reducing attenuation when coupling fiber, is given as

$$N.A. = \sqrt{n_0^2 - n_1^2}. \quad (3)$$

This is simple to apply for a fiber whose core is the only element that propagates light. Double clad (DC) fibers, which are used to propagate light in a larger total area, makes this calculation more complicated, as light is traveling at different speeds and at different angles per mode within the core and inner cladding of a DC fiber. Having said this, we may assume that the majority of light is passing through the center of the fiber while the “tail ends” contain a small percentage of the light transmitted [95]. This is a macroscopic view of light propagation, which does not consider microscopic properties. What happens when light is propagated through a nonperiodic insulator?

2.2 Characteristics of Glass

Although man first manufactured glass over 4000 years ago, significant advances in the study of glass at the microscopic level did not begin until x-ray and neutron diffraction spectroscopy and subsequent methods (NMR, Raman.etc) were developed in the first half of the 20th century [67]. Unlike crystals, glass has no periodicity, symmetry, or long range order, and therefore cannot be thoroughly investigated or modeled using crystal structure techniques that depend on these factors [61]. The fundamental difference between crystal lattices and non-crystalline solids such as glass has to do with the freedom each atom has to bond with other atoms. Both crystals and glass (AX_4) have ordered structural units, however the structural units in a crystal must bond with the same torsion and bond angles, thus giving a uniform lattice distribution. In vitreous glass, both bond and torsion angles have a range of acceptable degrees for connectivity—the interconnection of adjacent structural units is the first molecular structure to significantly contribute to disorder [61]. This is a second factor that affects the absorption features seen in glasses such as SiO_2 . The microscopic structure of glass directly influences spectroscopic methods used in the experiment. Another factor that influences transmission is the homogeneity of the glass.

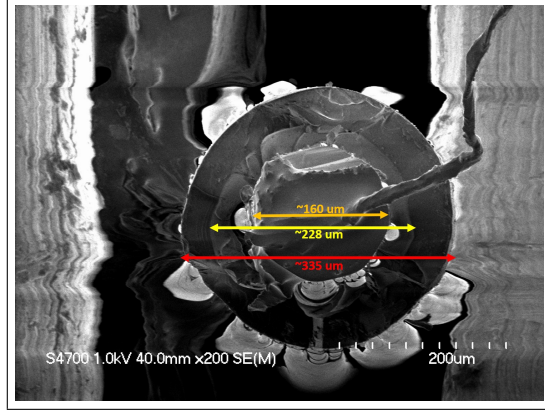
2.3 General Characteristics of Fiber

Various references [64] [37] describe optical fibers as “light pipes”—glass tubes used to guide light from a light source to a light detector [11]. Since the mid 1800s, scientists manipulated common household items that were transparent, such as salt, in an effort to create light pipes; over the years, they concluded that glass and plastic were best qualified for manipulation at various temperatures and humidities. The creation of fiber requires heating to the point of melting the glass and then extracting

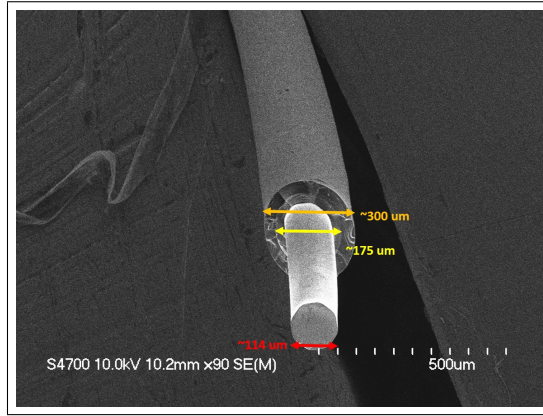
thin, uniform strands. Light propagation in glass depends on its purity—the addition of other elements besides the host glass may add to either the scattering or absorption of light at certain desired wavelengths. Propagation over long distances was not possible early on as the impurities in manufacture caused too much absorption in the fiber, thus ending in significant attenuation of the signal over short distances [37]. Now the purity of a typical silica fiber is in the range of one impurity per billion. Once the issue of homogeneity was addressed, manufacturers began to tinker with geometries optimized for specific operations. The geometry of the experimental fiber is addressed in Appendix A; the appendix includes equations used to find figures of merit for the fibers in this study, as well as any experimental procedures that added to transmission losses unrelated to radiation damage. The chief cause of transmission losses is discussed next.

Fiber Losses.

Coupled fibers were mismatched due to core differences or N.A.; this mismatch may be due to equipment limitations, such as needing a certain N.A. for the spectrometer used despite the width of the experimental fiber (systematic), or stripping one fiber too narrowly or another too widely (see Figure 1). Cleaving at an angle may give fibers different reflection losses according to the initial acceptance angles of both fibers. The angle of dispersion may change if the end is not flat and parallel to the axis line of the receiving fiber. The nearest to flat we can get using a mechanical cleaver is difficult to ascertain, however images generated by a scanning electron microscope in a flat surface to within 0.001 radians. As discussed before, the homogeneity is important. Purposeful doping of the host matrix may be completed to facilitate light transmission for certain wavelengths. This is the case with the dopants found in the experimental fiber.



(a) 160 μ m Inner Cladding



(b) 114 μ m Inner Cladding

Figure 1. Fiber cross sections under SEM magnification that show the 2 claddings and the coating. The left was cleaved with scissors while the right was cleaved with a mechanical cleaver. Unlike Figure 1(a), Figure 1(b) has partial stripping of the inner cladding, allowing for less light through and potential leakage due to damage.

Dopants in Fiber.

Because the mechanism of propagation in fiber requires different indices of refraction between the fiber core and cladding, with the core at a higher index of refraction than the cladding, dopants may either be added to the core, cladding, or both. In the case where the cladding is doped, a higher index of refraction is attained with dopants such as Germanium—an element that raises the index of refraction of the silica core without contributing significant absorption issues. Fluorine is a typical

dopant as it reduces the index of refraction of the cladding. In the case where the amplifying properties of the fiber need increasing, RE are used [37].

RE Dopants and Co-dopants.

Since the early 1960s when researchers discovered that optical fiber could be used to lower the requisite pump power for gain when using glass amplifiers and lasers, scientists continue to optimize the pumping efficiency of fibers, including doping fibers with REs. For the purposes of lasers and amplifiers, doping with REs typically refers to the lanthanides ($Z=57$ to $Z=71$), not the actinides ($Z=89$ to $Z=103$). As the lanthanide contraction results in an inverse relationship between proton number and atomic radius, ionization energy increases. This results in many lanthanides having trivalent ionization, which glass-based optical devices use due to insensitivity of trivalent ions to the host makeup. RE ions also lead to efficient emission, which is favorable for pumping mechanisms [13].

Although adding dopants increases the efficiency of laser amplifiers, the effect becomes deleterious as concentration increases because ion-ion interaction rates rise as concentration increases. Negative effects vary by RE elements, but include cross-relaxation (an ion gives its excitation energy to a neighboring ion) and cooperative up-conversion (two excited ions interact with one going to the ground state while the other is further excited). For example, Yb^{3+} , unlike certain bands of Er^{3+} , uses a three-level transfer mechanism instead of up-conversion, allowing for higher concentration prior to quenching. Comparing the quantum efficiency of an ion to the level of concentration is a figure of merit for determining the best RE dopant. Typical concentrations for REs are below $1 \times 10^{19}/\text{cm}^3$ to avoid concentration quenching, although this also depends on the co-dopants and the type of glass used as the host. For example, in amorphous silica hosts, microscopic clustering occurs with only small

amounts of dopant because of the need for the dopant to share non-bridging oxygen. [13]

Another factor that determines clustering phenomena is the addition of co-dopants. Ample literature suggests that the addition of Al to RE doping results in “solvation shell” which allows REs to readily incorporate into the host matrix [13]. Al further allows higher emission and absorption cross-sections and low background losses. However, the level of co-dopants, especially using a silica host, determines the level of color defect generation, and thus the level of photodarkening. In the case of photodarkening, the level of Al is not as important as the level of boron or phosphorus, for example in [52, 80]. Therefore, an ideal mix of Yb/P/Al/Si allows for the highest cross sections without significant photodarkening and background losses. Several methods of deposition are available for inserting dopants into a host matrix. However, one method provides a significant advantage in homogeneity for both the host matrix and dopants.

Direct Nanoparticle Deposition in Fiber.

Direct Nanoparticle Deposition (DND) is important because DND deposition of fiber is supposed to be more homogeneous than Outside Chemical Vapor Deposition (OCVD) or Modified Chemical Vapor Deposition (MCVD), which are the two most popular methods of forming doped fiber. As of this writing, most research papers in the literature review, dating from the 1970s to the 2000s, use either OCVD or MCVD. MCVD limits the number of deposition layers in a core to anywhere from 2 to 10 layers, thus limiting the distribution of dopants in the core. When co-dopants are involved, the solution-doping technique typically used in MCVD encourages clustering in the fiber. In DND, the amount of layers deposited is in the hundreds instead of tens, and the simultaneous addition of REs with modifier ions such as aluminum

and phosphorus reduces clustering; side-by-side images of MCVD and DND show a smoother, and thus, more homogeneous doping cross section in the DND produced fiber. Because DND directly reduces the amount of photodarkening and clustering in the fiber, we must consider this advantage over MCVD/OCVD produced fiber experiments. Now that the inherent characteristics of the fiber are illuminated, we may address the changes caused by interactions between our pristine fiber and neutral particles. [49]

2.4 Radiation Exposure

An understanding of radiation interactions with matter precedes discussion on defect analysis in fibers attenuated by radiation exposure. Each of the three sources of radiation considered here (i.e. irradiation by natural sources, by nuclear reactor, and by a nuclear detonation) carries a distinct span of neutral particle energies and rates of generation—two quantities that, apart from activity, $\mathcal{A}$, are necessary to understand the consequences of radiation exposure. These interactions are further divided by particle and target of interaction (i.e. fiber or surrounding materials). Because the source used in this experiment produces neutrons and photons at high fluxes, the focus is on the possible reactions that result from neutral particle radiation.

Radiation Interactions with Matter.

The interaction of electromagnetic radiation (EM) with matter is divided into three processes: photoelectric effect (PE), Compton scattering (CP), and pair production (PP). These three processes have characteristic ranges of interaction based on the number of protons (Z) of the target material and the energy of EM radiation (in MeV). The total linear attenuation coefficient, μ , is a useful metric for determining the attenuation of EM radiation in matter. It is dependent on the Z of the material,

its density, and thickness. The intensity (I) of the attenuated EM radiation may be determined using

$$I = I_0 \cdot e^{-\mu t}, \quad (4)$$

where I_0 is the initial intensity and t is the thickness of the material. Although the density does not appear in the equation, it is useful to divide the individual components of μ by the density to inhibit the expression of the effects electrons have in the material with respect to the three modes of interaction [50].

As seen in [50], PE dominates at low Z and low energies, while PP dominates at high Z and high energy, with CP dominating between 0.5 to 5 MeV regardless of Z . Since Z number varies in this experiment from 1 to 83 and energies vary from about 0.01 to 10 MeV, the dominance regions are dependent on the element. However, since the primary determinant of laser amplification is Yb, the γ interaction is predominantly CP (below 5 MeV) and PP (above 5 MeV). Additionally, due to the relatively low Z of Al and P, PE and CP dominate γ interactions. Figures 3 and 4 contain mass attenuation coefficient versus energy plots for all relevant elements in this experiment.

Radiation Induced Attenuation.

RIA is attenuation attributed to an increase in absorption features in the fiber. Wavelength, dose, and dose rate determine the extent of RIA, as seen in

$$RIA(\lambda, I) = \frac{-10}{L} \cdot \log_{10} \left(\frac{I(\lambda)}{I_0(\lambda)} \right). \quad (5)$$

' I ' is the intensity of the fiber at some point during or after irradiation and ' I_0 ' represents the initial intensity, λ is the wavelength, and ' L ' is the length of the fiber irradiated. This equation can be modified to

$$RIA(\lambda, I) = \frac{-10}{L - \ell} \cdot \log_{10} \left(\frac{I(\lambda)}{I_0(\lambda)} \right), \quad (6)$$

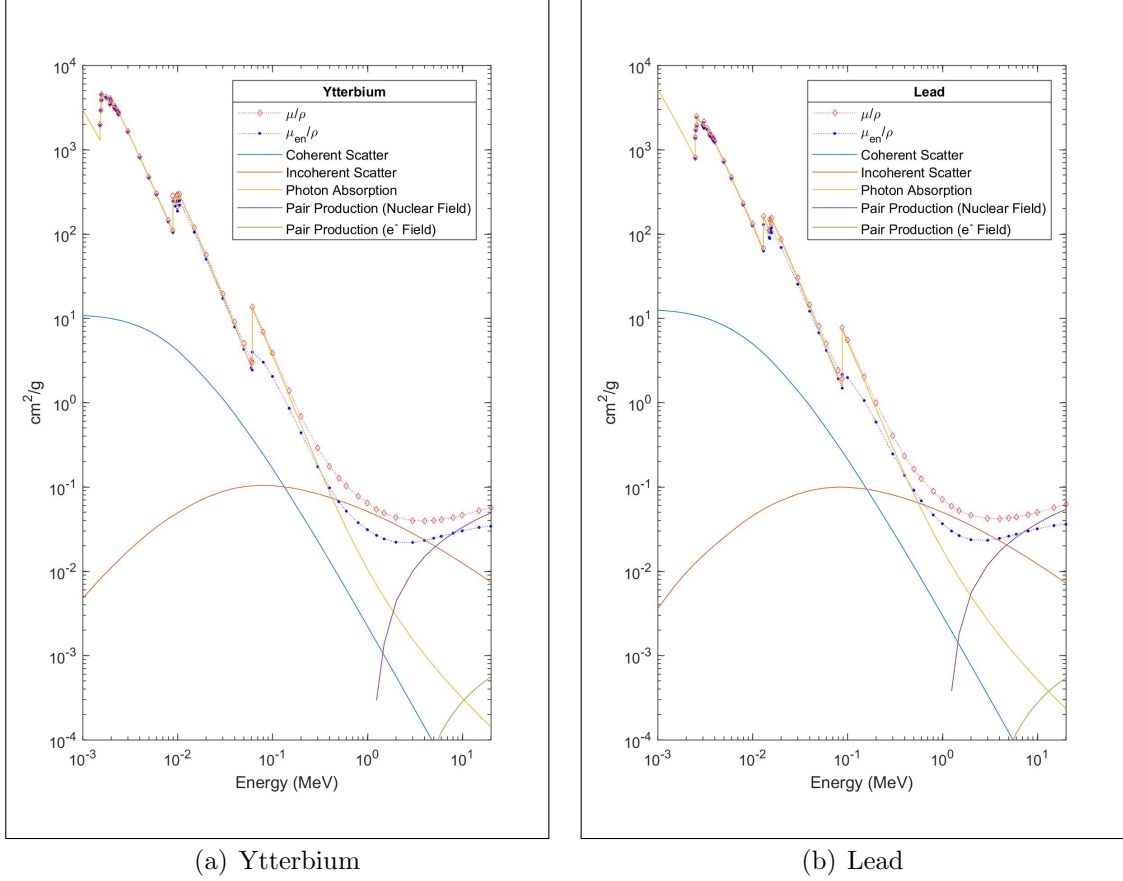


Figure 2. Like Yb in Figure 2(a), Pb shows a small CP energy span due to its high- Z . The 2 mass attenuation charts demonstrate the much smaller energy span for CP dominance than in Figures 3 or 4, where inelastic scattering predominates.

where ℓ is the length of unirradiated fiber, to account for patch or passive cables used in this experiment that were not irradiated. However, because a portion of both patch and passive cable was unshielded, we use Equation (16) since the length of unirradiated fiber after each run is unknown. It is important to note that RIA is a result of performing a logarithmic calculation on the ratio of intensity before and after a certain dose. Therefore, if the change in intensity of the fiber is small, large, nonrepresentative results will follow. In this experiment, γ and neutron radiation produce both ionizing and nonionizing radiation within and around the fiber. Photon energies in both the X-ray range and the γ -ray range are seen either as primary or

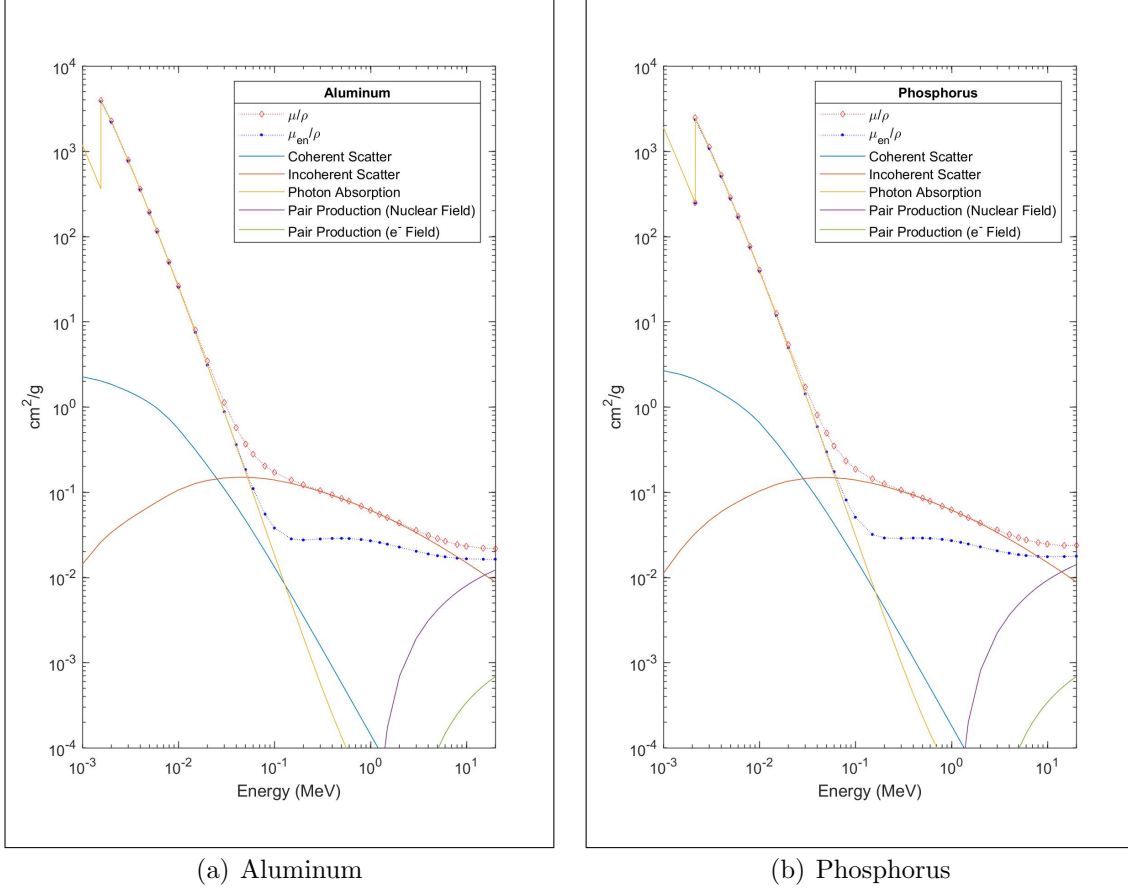


Figure 3. The 2 mass attenuation charts above shows the the primary EM interaction mechanism for the modifier elements that ensure Yb homogeneity in the host matrix. Note the difference in CP dependence between Yb (Figure 2(a)) and its co-dopants.

secondary products. The energy ranges of the neutrons produced as primary or secondary products range from thermal to fast neutrons. At the microscopic level, RIA may change the index of refraction of the fiber, the density of the fiber (related to the index of refraction), and the absorption features of the fiber [21]. Photon radiation of RE doped fibers produces significantly higher RIA than in non-doped fibers [38] [93].

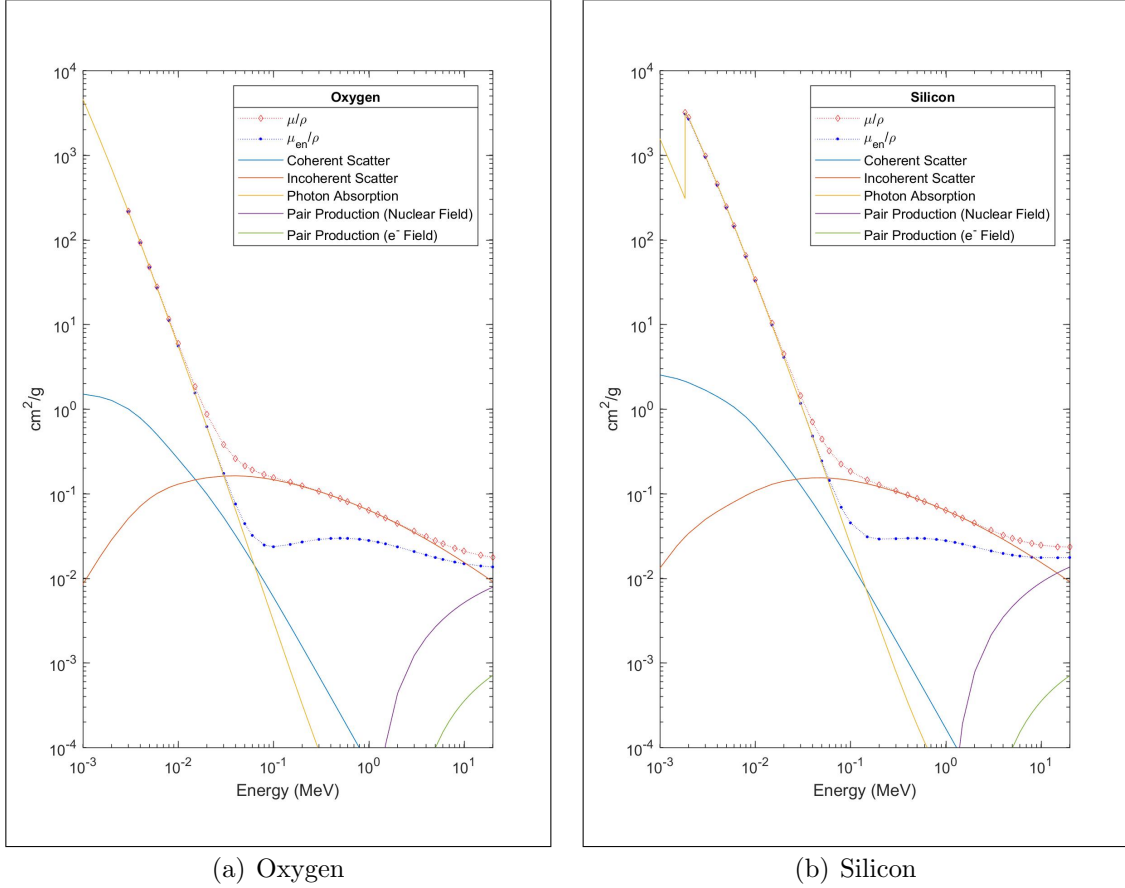


Figure 4. The 2 mass attenuation coefficients above represent the host glass. Based on the results, we may determine the relative amount of low energy photons in the unknown photon spectrum of the OSURR.

Sources of Radiation.

Background radiation, present in cosmic rays and naturally occurring radioactive isotopes, typically make up about 0.1-0.2 rem /year [50]. We receive this radiation at typical temperatures close to 300 K with counts for neutron energies between 10 keV and 1 MeV below 1 neutron per minute at an altitude below 1000 feet. For thermal neutrons, the count is higher, however the thermal neutrons do not have sufficient energy to induce significant defect centers, so they are not considered. In a nuclear reactor, neutrons and photons are emitted with keV-MeV levels of energy and at

fluxes at least a dozen orders of magnitude above that of cosmic rays, with typical temperatures in the hundreds to thousands of degrees kelvin. Finally, in a nuclear weapon, we see a huge leap in temperature to millions of degrees and even higher neutron energies and fluxes [8, 22]. These three sources vary greatly in energy and flux. However, a good estimate of the results of radiation by neutrons and photons may be made using a nuclear reactor at a high enough flux and time to model exposure of a fiber within the prompt radius (2 miles) of a 20 kT nuclear detonation.

Decay Rates, Dose, and Dose Rates.

The rate (R) at which a reaction produces radioactive species is given as

$$R = N_0 \cdot \sigma \cdot I, \quad (7)$$

where N_0 represents the initial amount of target atoms (cm^{-2}), σ represents the cross section (given as an “area”, however this is not a physical area, but rather indicative of probability of interaction) in barns, and I is the flux of particles incident on the target (in $\text{s}^{-1} \cdot \text{cm}^{-2}$). Once N_0 atoms are irradiated, three primary methods of decay are observed: α decay, β decay, and γ decay. The rate of decay is dependent on the rate of production of the parent population and the rate of decay of each method of decay for each daughter, typically designated by branching ratios of total decay. Decay products are important as the amount of a material may be determined through inductive processes. Henschel states that γ radiation induces losses up to 2.5 times higher than neutrons (14 MeV) for the same dose rate [39]. Borman [6] and Morana [57]) state that secondary ionization effects are negligible as the γ dose provided is significantly higher than the neutron dose, however in the case of this experiment, the γ dose is attenuated up to about a $\frac{1}{3}$ of the neutron dose—another factor to keep in mind when comparing features between γ radiation and neutron radiation dominant datasets.

Fiber Defects Specific to Irradiation.

The consequences of γ and neutron radiation in fiber are dependent on fiber elemental content, the temperature of irradiation, and the nature of the radiation. Defect dependence on total dose and dose rate is separately discussed below. The centers present within typical glass fibers are described by horizontal lines to represent complete bonds and dots to represent unpaired electrons in Table 1. Any double or triple horizontal lines below represent single covalent bonds between Si and O.

Table 1. Listed are the most prevalent defects created in SiO_2 . These defects do not take into consideration the doping of Y_2O_3 , Al_2O_3 , or P_2O_3 . However, similar defects may be created by replacing the Si dependence with the dopant (e.g. Al).

Chemical Structure	Name
$\equiv\text{Si}\cdot$	E'
$\equiv\text{Si-O}\cdot$	NBOHC
$\equiv\text{Si-O-O-Si}\equiv$	POL
$\equiv\text{Si-O-O}\cdot$	POR
$\equiv\text{Si-Si}\equiv$	Neutral O Vacancy
$=\text{Si-Si}\cdot$	Two-fold Coordinated Si

Modeling Defects in Fiber.

After accounting for both the fiber properties and radiation effects of this experiment, we must make sense of the data. Prominent publications in this field typically model defects using Gaussian bands added together to mimic the experimental results for radiation damage to fibers [21]. Gaussian deconvolution is a good way to ascertain defect centers. A Gaussian may be used to approximate a defect center because of several factors. As discussed earlier, a defect center is created by impurities in the host matrix. This means that there is a characteristic band gap energy for each of

these features [74]. These band gaps may be bridged when incoming radiation (either neutron or γ in this case) deposits energy in the fiber such that electrons are promoted to the conduction band. Other bonds as shown in Table 1 may also be formed. The Uncertainty Principle ensures that this band gap energy is not truly discrete (natural broadening). Additionally, the fact that the matrix is a glass means that the angles and bond lengths vary for each structural unit, also broadening the energy required to release an electron or form a bond not present in large amount in a pristine fiber. Most importantly, PE and CP produce electrons of varying energies, with some more probable than others [50]. None of these factors are Gaussian in nature, and their combination may result in an asymmetric response curve. However, for the purposes of this experiment, we are interested in finding where a peak may be located, and whether the distribution is Lorentzian, Maxwell-Boltzmann, or other, the peak is roughly Gaussian in nature. We do not take into consideration interconversion of defect centers or temperature effects during irradiation.

Should the material be characterized, experiments such as those found in [24], where techniques such as Electron Spin Resonance (ESR) were used to positively associate a Gaussian band with a defect center, may be used in conjunction with a deconvolved Gaussian to determine the identity of the defect center. Therefore, a complete characterization uses both a theoretical model and experimental data.

2.5 Background

Two recent studies by [73] and [6], respectively, analyzed the effects of γ and mixed γ and neutron radiation on YDF of various diameters.

Bormans study, completed in 2015, using both pure γ and mixed neutron and γ radiation doses on YDFs indicates that core/inner cladding diameters of 6/125, 10/125, and 20/400 μm YDFs have wavelength-dependent absorption features, with

the doping concentration affecting the amount of attenuation in the fibers. The setup for this study is similar to Borman's 2015 work and includes the use of the same research reactor, a hollow aluminum 7 inch dry tube, various fibers, and a light source and spectrometer. Specifically, the same core diameter fibers, passive and patched cables, and similarly doped active fibers are used. Diagrams are available from [6] for a general setup. Modifications to this setup are contained in Chapter 3. Figure 5 from [6] shows that at a high enough dose, thermal neutrons contribute little to the RIA, while Figures 6(a) and 6(b) show the effects on the defect magnitude with and without cadmium included.

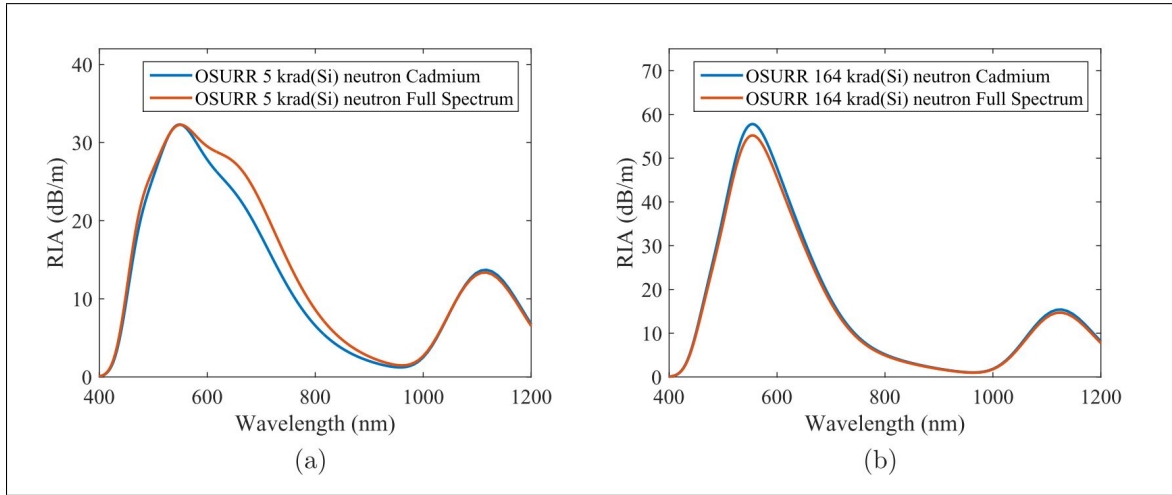


Figure 5. The above figure from [6] shows the low dependence on thermal neutrons for RIA. Hence, the experiment was modified to not include a cadmium shield, as the cadmium shield actually caused activity within the experimental setup to increase.

All fibers show a defect around 975 nm, but the 10 μm fiber had the largest absorption feature at this wavelength. At 1064 nm and 475 nm, the 20 μm had the largest attenuation. Additional absorptions are seen at 539 and 620 nm. The radiation from the Ohio State University Research Reactor (OSURR) specifically contributes to defects from 550-975 nm. In comparison to a pure γ source, this is indicative that neutrons contribute to defects below 600 nm [6].

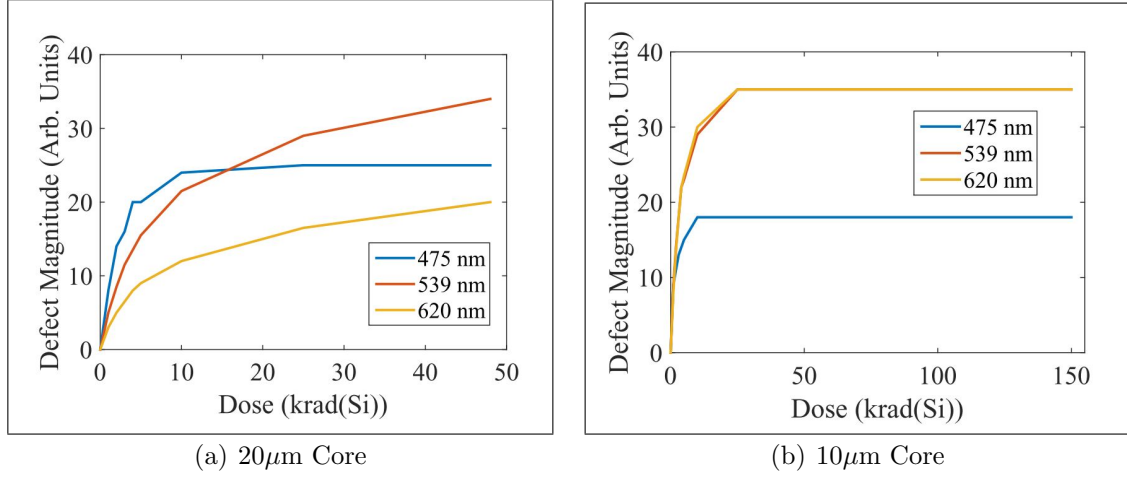


Figure 6. 6(a) is a fiber with cadmium shielding while 6(b) is a fiber without cadmium shielding. Although we see a marked difference in defect magnitude, the rate of defect creation clearly shows that most of the defects are created within 25 krad(Si). Thus, the time that the reactor was held at full power for this experiment was significantly shorter than in [6], where these figures are taken from.

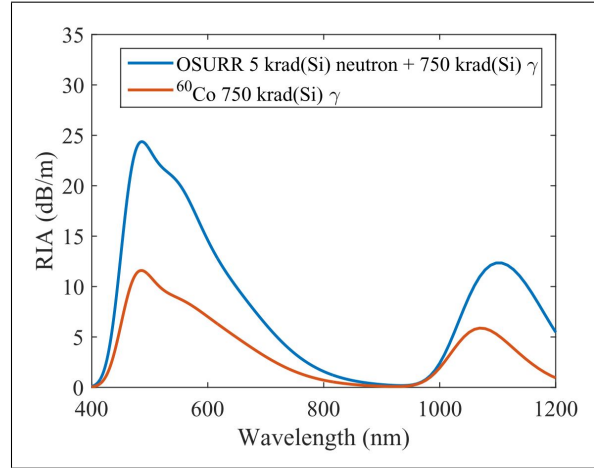


Figure 7. A direct comparison of holding the photon flux the same while removing the neutron flux is shown above. This plot, taken from [6], shows the difference in attenuation between mixed(i.e. photon and neutron) and photon radiation. It is the objective of this research to determine if neutrons are the sole cause of the difference between the two lines.

III. Experimental Equipment, Design, and Analysis

3.1 Chapter Overview

This chapter begins with a description of equipment used followed by the procedure behind the collection of experimental data including *in situ* measurements, statistical techniques, and modal analysis methods. A list of equipment completes this chapter for ease of reference.

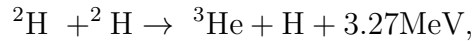
3.2 Equipment

This section includes characteristics of equipment and operational limits as well as experimental setup at both the AFIT site and the OSURR site.

Radiation Sources.

The DD-109 (Adelphi Technology Inc.[4]), pictured in Figure 8, is a Deuterium-Deuterium (D-D or ^2H - ^2H) neutron generator. Pressurized $^2\text{H}_2$ is fed (01) into a microwave chamber which ionizes the gas, turning it into a plasma. This plasma is then accelerated (02) through an aperture which limits the radius of the $^2\text{H}_2$ beam being formed. The beam is focused to intersect with a cone-shaped ^2H target (03).

Using beam-target fusion,



occurs which produces 2.45 MeV neutrons (0.8 MeV ^3He) emitted in roughly 4π sr, as the beam has width and does not contact the target at a point [8]. The output is estimated to be 1×10^9 neutrons/s, which for a SM fiber of length 30 cm by 3×10^{-4} cm radius placed parallel and 15.5 cm from the center of the source, yields about 6×10^3 neutrons/s. For a 20 μm core at the same distance and length, the neutron

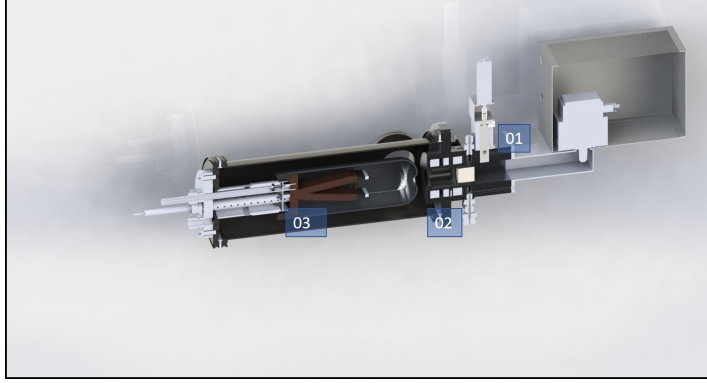


Figure 8. Because of the wide dispersion of neutrons in the DD109M, the flux is relatively low compared to fluxes in literature focused on radiation induced defects in fiber. The advantage of a neutron generator is that the interference of photons is less. Additionally, weight restrictions do not play a significant role when using high-Z shielding. (Adapted from *Adelphi Technology* (2017))

flux is about 4×10^4 neutrons/s. Shielding materials may be easily placed around a sample as the neutron generator sits horizontally laid out on a typical lab table. [4]

The OSURR is a fission reactor operating with 19.5% enriched U_3Si_2 at a maximum power of 500 kW, although the maximum operating limit is 450 kW (90%). Located within a pool of H_2O about 20 feet deep (for neutron moderation and photon shielding), an experiment may be placed within about 10 cm of the fuel rods using an aluminum tube constructed of 6061 T6 Al, measuring approximately 20.5 feet tall, 6.75 inches ID, and 7.0 inches outer diameter (OD), allowing for substantial neutron and γ flux within the tube at heights relatively level with the fuel rods.

This flux attenuates as in Figure 9, with the maximum neutron flux in the 7 inch dry tube calculated to be 1.5×10^{12} n/cm²/s, with 73% as thermal neutrons [78]. Equivalent neutron dose rate in rad-Si/hr is 6.5×10^4 and in 1.0 Mev equivalent neutron flux is 2.0×10^{11} (Si) n/cm²/s. All calculations are taken for a detector 12 inches from the bottom of the aluminum tube adjacent to the reactor. The maximum γ radiation exposure rate is calculated to be 8.7×10^6 R/hr or 7.6×10^6 rad-Si/hr at 450 kW [78].

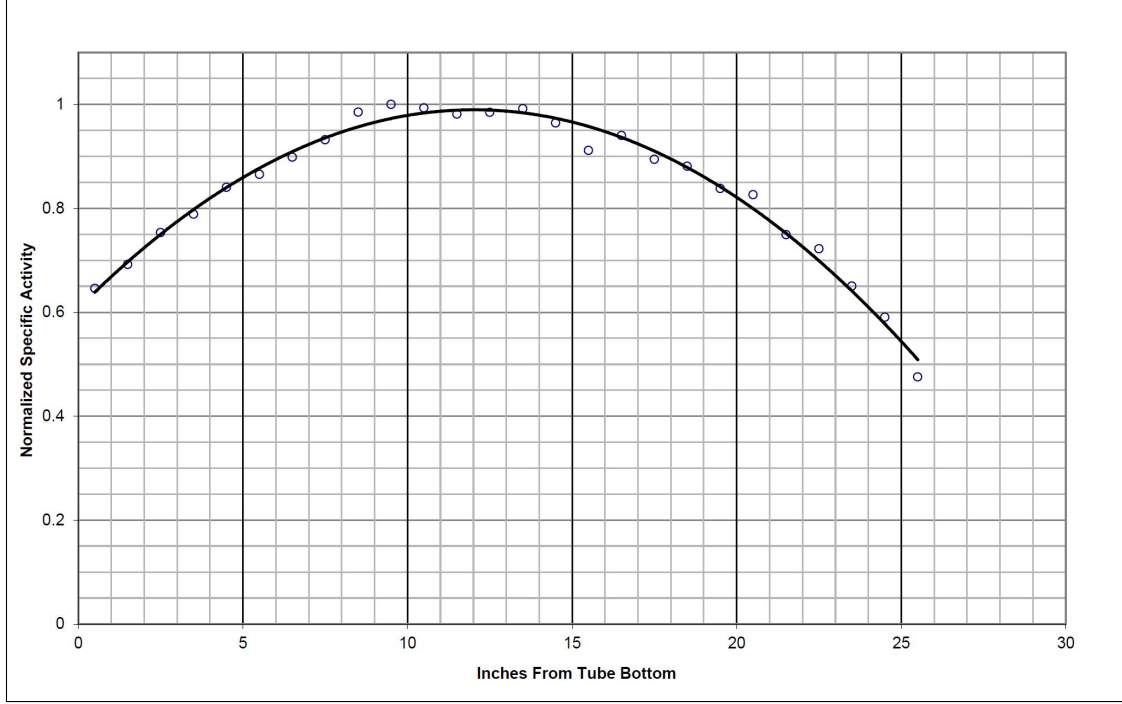


Figure 9. The r^2 dropoff in activity was used to determine the radius of the coiled fiber prior to experimentation. Additionally, the peak determined the height of the experimental rig lowered into the 7-inch dry tube. (Adapted from *NRL* (2013))

Experimental Sample Container.

The Small component/Package Uniform Drum-Device (SPUD-D), featured in Figure 10 was built in collaboration with the AFIT fabrication shop to facilitate experiments concerning neutron and photon radiation damage of electrical devices and optics at the OSURR. Two models of SPUD-D were built—one with OD 6.6 inches and the other with OD 5.75 inches. Both can house up to 5.5 inches of shielding unilaterally or 2.5 inches of shielding bilaterally. The first model includes nested plates of bismuth (Bi)/titanium (Ti) as well as a 1/16 inch thick cadmium lining, ensuring thermal neutrons are attenuated prior to hitting experiment devices and shielding plates. The second model comes with lead (Pb) plates fitted to a 5.5 inch diameter. The two plates closest to the reactor are each 2 inches thick and 7 inches tall, while the mounting lead plate, furthest from the reactor and the plate used to attached

the fibers, is 0.5 inches thick. Notably, the thin plate has a significant amount of antimony present, suggesting a density lower than that of the other two plates. A lower density will also result in lower attenuation per unit thickness. Both models mark the 12 inch point where the maximum fluxes are found and use three legs to contact the ground, eliminating rotation of the sample once contact with the steel plates found at the bottom of the aluminum tube is made. All outer components of the SPUD-Ds are made of 6061 T6 Al.

Effects of Shielding on Photons and Neutrons.

Recall that some of the photon radiation is attenuated on the first plate since lower energy level photons may be attenuated more easily than higher level photons with a thinner plate. So the total amount of rads does not necessarily decrease as with Beer's law. We should see a significant decrease after the first 2-inch Pb shield if it is the case that many of the photons are lower energy. This hardened spectrum will then decay to a soft spectrum as it further scatters through the high-Z shielding. An experimental method using dosimeters is used to make a guess as to the photon spectrum of the nuclear reactor. Because the high-Z shielding chosen has a low interaction cross-sections with neutrons, no significant attenuation of neutrons should occur, both in their amount or their energy, due to lead. [5]

Light Sources.

The SLS201L(/M) Thorlabs stabilized Tungsten light source was used to provide a broad light source for the YDFs. This light source generates output from 300 to 2600 nm and a peak at 1000 nm for a color temperature of 2796 K. It provides constant parameters of intensity with minimal noise, leaving outside power fluctuations as the only varying parameter for the light source. Figure 11 shows the response curve for

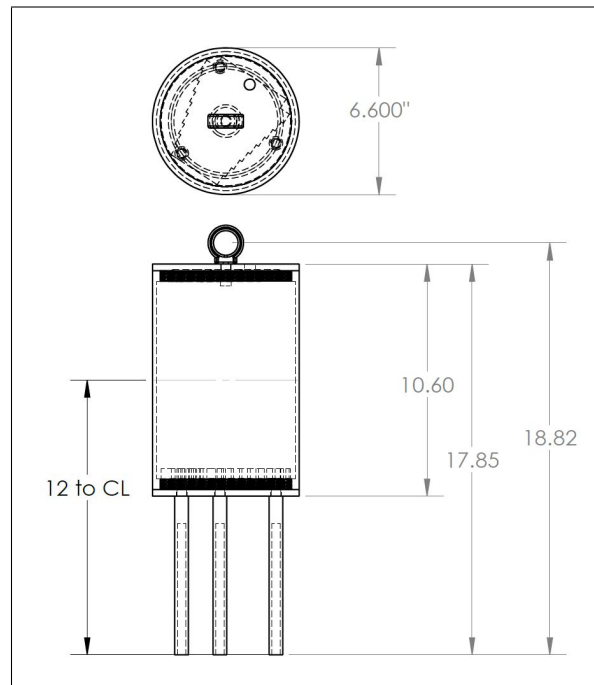


Figure 10. Rig used to house both the high-Z shielding and the coiled fiber samples within the 7-inch dry tube. The “12 to CL” indicates 12 inches to centerline and is the critical height to ensure the fiber is receiving the estimated neutron and photon flux (estimation completed by OSURR staff). This is important as attenuation of the flux follows an r^2 drop off. (Adapted from *Brian Crabtree and Chris Harkless (2017)*)

the SLS201L. With the exception of the peaks within the 800-1250 nm range and the right tail of the lights response curve, the SLS201L maintains a typical black body spectrum at the color temperature. The response curves varied slightly between the two light sources, however to avoid inconsistencies due to the light source, the same light source was used for each type of fiber. The lamp was operational for at least 30 minutes prior to each run to ensure temperature stability. [84]

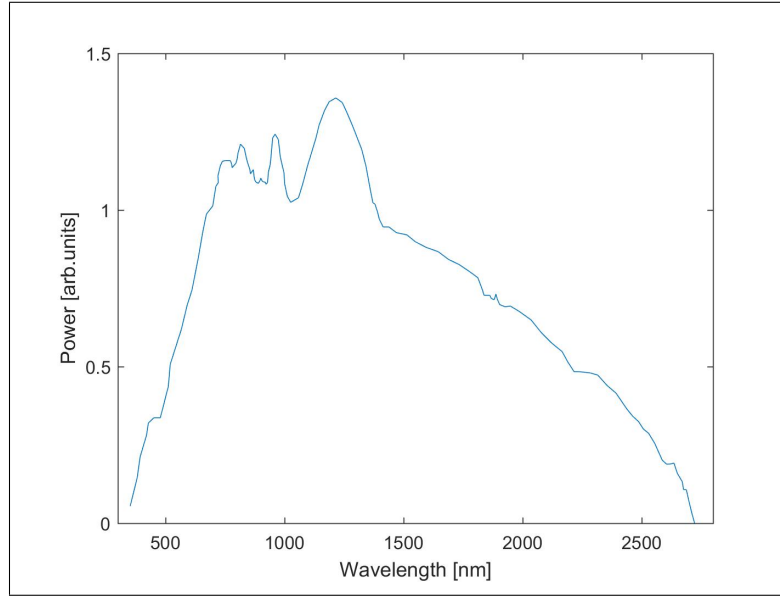


Figure 11. The response curve of the Tungsten Halogen Lamp indicates that less than 0.3 power [arb. units] is provided to fibers with spans well below 500 nm. This response must be subtracted from the fiber signal to ensure absolute characterization. (Adapted from *Thorlabs SLS201 Manual [Revised]* (2017))

Uninterruptible Power Supply.

The SU2200XL_a is an Uninterruptible Power Supply (UPS) with a $\pm 2\%$ variance in voltage (V) and frequency (Hz) used to maintain power to the light sources, spectrometers, and laptop housing the operating software. Because the native power supply in both the neutron generator and the nuclear reactor buildings fluctuated enough to cause significant changes in the spectrometer measured intensity, the UPS was used to maintain a frequency close to 60 Hz and a voltage close to 120 V. The

highest observed deviation while using the UPS for voltage was ± 0.1 V while the highest deviation for frequency was ± 0.1 Hz. It appears that this UPS is more susceptible to voltage over frequency oscillations. Actual power fluctuations in the neutron generator were not controllable. Noticeable fluctuations in power and sudden current drops were present during measurement of irradiation effects by the neutron generator. [89]

Measurement Sensors.

This section includes the various lab equipment used to determine figures of merit for the research. It is broadly separated into two focus areas: optical and elemental properties of materials and nuclear reactions between sources and materials.

Fiber Coupled CCS175/200.

Two Compact CCD Spectrometers (CCS) Thorlabs Czerny-Turner spectrometers were used during *in situ* data collection for both the neutron generator and the OS-URR radiation sources (CCS 175 and CCS 200). Both spectrometers have S/N ratios of $\leq 2000:1$, with a 3648 pixel CCD line array sensitivity of 160 V/(lx·s). For the CCS 175, the wavelength range is 500-1000 nm and a resolution of 6 pixels/nm. For the CCS200 the wavelength range is 200-1000 nm and a resolution of 4 pixels/nm. Intensity is set from 0.000 to 1.000, with integration times differentiating the efficiency of transmitting light for each tested fiber. Should the integration time result in values above 1.000, the spectrometers record these values as “1.0” (saturation). Each fiber was initially scaled to between 0.90-1.0 in keeping with the recommendations of the CCS manual, but also to check the integration time (how each signal was scaled). Most samples had integration times in the tens of milliseconds, however one had an integration time of 650 milliseconds (the 6.35 cm shielded 20/400 YDF), therefore it is

known that the inherent fiber intensity differed prior to irradiation. All fibers had an integration time below the sampling rate. Both spectrometers are factory calibrated in regards to wavelength, with this calibration as the default source as opposed to a user calibration which may be more accurate than the default calibration. Note that the recorded data measurements do not account for grating efficiency, grating density, or blaze angle—thus using the same fiber on both spectrometers results in different values for the wavelength region where they overlap (500-1000 nm). A source with well-known spectral features is used for the factory calibration, with interpolation between recorded pixels assumed. Note that intensity values given below -0.10 are artifacts of the spectrometer itself. [86]

Active Fibers.

The Thorlabs YB1200-6/125DC cladding-pumped SM fiber features a core of $6.0 \pm 0.8 \mu\text{m}$ MFD (N.A. of 0.15 ± 0.01), and an octagonal inner cladding of $125 \pm 2 \mu\text{m}$ (N.A. is ≥ 0.48). The fibers cladding-pumped slope efficiency is 77%. The lengths of fiber used for experiments were 0.3 and 1.0 meters with FC/PC terminators. The microstripper used to prepare the fiber for use, Mechanical MS-FOK-1 kit, limited the stripping diameter to 114-160 μm (purple blade for stripping up to 125-135 μm), allowing for partial stripping of the inner cladding at times depending on the force used to clamp the microstripper. As seen in Figure 1, the variance in preparation mechanisms directly affects fiber size, thus affecting weight of fiber, number of modes available, and light propagation. The ideal pristine fiber maintains an intact inner cladding and core. Thus, without direct measurement for each fiber using, for example, an SEM-EDX, we cannot guarantee that a mechanical stripping method did not affect the inner cladding.

Passive Fibers and Accessories.

The Thorlabs YB1200-20/400DC cladding-pumped MM fiber features a core of $20 \pm 2\mu\text{m}$ MFD (N.A. of 0.07 ± 0.005) and an octagonal inner cladding of $400 \pm 15\mu\text{m}$ (N.A. is >0.46). This fiber’s cladding-pumped slope efficiency is 75%. The primary fibers used to connect active fibers to the light source and detector were the Thorlabs FG050LGA, with a $50\mu\text{m} \pm 2\%$ core and $125\mu\text{m} \pm 1\mu\text{m}$ cladding for the 6/125 μm SM fiber and the FG105LCA $105\mu\text{m} \pm 2\%$ core and $125\mu\text{m} \pm 1\mu\text{m}$ cladding for the 20/400 μm MM fiber. Both fibers have a wavelength operating range of 400-2400 nm and a N.A. of 0.22 ± 0.02 , which matches the CCS175 connection (N.A. = 0.22 and 50 μm diameter), but not the CCS200 (N.A. = 0.22 and a 200 μm diameter) calibration specifications.

3.3 Methodology

This section describes the procedures used to record measurements using both radiation sources. Although there is a “typical” setup, on-the-spot changes are described as needed since the OSURR is a limited resource and adjustments were required at times to maintain the testing schedule. Any results produced with alternate setups are indicated throughout the analysis and results chapter. In both setups, the light sources, spectrometers, and computers were attached to the UPS system. Pb was used as the high-Z shielding material for both radiation sources as well, although the size of the individual Pb plates varied from 2 inches (the two plates closest to the reactor) to 0.5 inches (the mounting plate within SPUD-D). The two thick plates had a high lead content, however the mounting plate had significant amounts of antimony present. To determine the elemental makeup of the samples and equipment being irradiated, several methods of characterization were used.

3.4 Material Characterization

Three methods were used to determine the doping concentration within the two fibers and the elemental analysis of shielding materials. The three methods are X-ray Fluorescence Spectroscopy (XRF), Scanning Electron Microscope-Energy Deposition X-ray Spectroscopy (SEM-EDX), and Neutron Activation Analysis (NAA). Attempts were made with all three methods to determine the doping concentration of the fiber, and NAA and XRF were used to determine the makeup of shielding materials.

XRF.

XRF was conducted to ensure that the appropriate mass attenuation coefficients were used to calculate the amount of attenuation of γ rays and to ensure that no significant neutron capture occurred due to any of the materials used in the experiment. Recall that the first SPUD-D had shielding advertised as Bi. Upon XRF analysis, we discovered that the shielding, in fact, had a significant amount of Sn (about 30%). This changed the mass attenuation coefficient from $7.214 \times 10^{-2} \text{ cm}^2/\text{g}$ to $6.203 \times 10^{-2} \text{ cm}^2/\text{g}$ and the density of the material from 9.78 g/cm^3 to 9.02 g/cm^3 . It is also important to note that of all the materials used, Sn has the lowest melting point (around 450° F), and depending on how it is distributed within the plate, the melting point could lower to about 170° F . A preliminary calculation using Beer's Law, with an initial dose rate of 7621 krad(Si)/hr , a linear attenuation coefficient for lead (using a γ ray energy of 1 MeV) of 0.8054 cm^{-1} , and a thickness of 12 cm indicates that dose would be reduced by about 3 orders of magnitude. Cadmium was used to line the first SPUD-D rig due to the presence of Bi (thermal neutrons would cause a higher activation in Bi and thus lead to longer wait times before the rig could be raised from the reactor floor). The sample of cadmium used to line SPUD-D was one piece, however particular elemental makeup was unknown prior to the XRF. XRF analysis

indicates that at least 10% of the Cd lining is made of palladium, although why Pd is used is still being explored. It is possible that the XRF misidentified palladium peaks within the analysis chamber, causing palladium to appear. After the first SPUD-D was damaged, the second SPUD-D was made without cadmium.

The second SPUD-D rig used lead in lieu of bismuth/tin (Bi/Sn) as a shielding material due to the fusion of Bi/Sn during flux wire tests. With the exception of the mounting plate, the shielding is made of a high percentage of lead. The mounting plate has antimony (Sb) present, although the percent amount is unknown.

Several attempts were made to mount a 6/125 YDF sample for analysis with the XRF. However, the smallest beam width available to this particular XRF is 10 μm . However, the probing voltage is limited to 30 keV, ensuring that the K shell of some elements in the fiber cannot be probed adequately. Figure 12 shows the results of probing the fiber. Another limitation of the XRF is the fact that probing below ^{11}Na is not possible, eliminating the identification and percentage of O, F, and H in the fiber. However, identifying the content of Yb, P, Al, and Si and knowing the typical bonds formed between O and these elements may lead to a rough estimate of O content. Unfortunately, as Figure 12 shows, no amounts of the latter group of elements was found. Additionally, because the XRF itself contains rhenium (Rh) for the operation of the X-ray gun, Rh may be eliminated from the spectrum, leaving Si, S, Cl, and Ti. The presence of Cl in the sample was surprising considering initial conversations with the manufacturer indicated that F1 was the additional dopant in the inner cladding and no mention of Cl was made. Because of these limitations, and the recent upgrade of the SEM with a new EDX system, SEM-EDX was tried next.

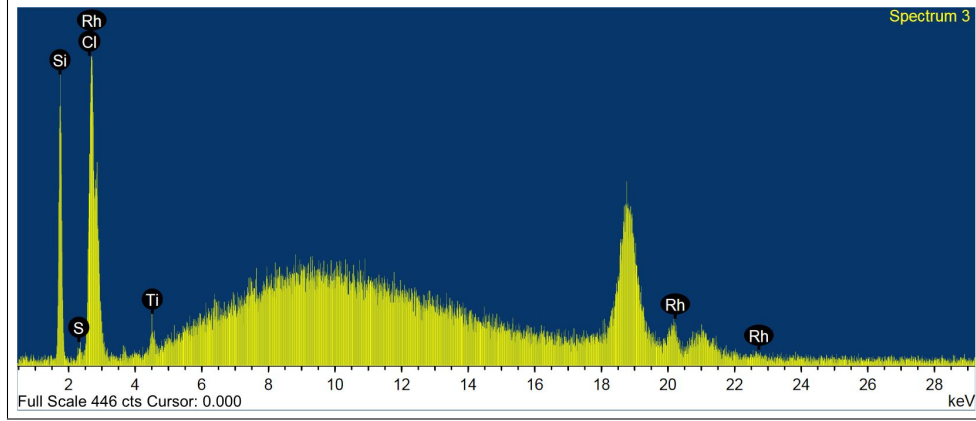


Figure 12. Quantitatively, the highest atomic percentage for this spectrum was ^{14}Si at 65.9% and ^{17}Cl at 28.7%. Titanium is present as the Al mount was covered in Ti film to ensure it did not interfere with the Al concentration in the fiber. The spectrum failed to identify the key RE dopant and the co-dopant P.

SEM-EDX.

The SEM-EDX was used to probe fiber content and obtain images of the cleaving performed by the Fujitsama cleaver. Both the 20/400 YDF and the 6/125 YDF, along with a known sample of 40/900 YDF at 2.6% concentration were measured. Although the fiber was cleaved, it was not polished, and therefore the accuracy of these measurements is limited to about $\pm 5\%$. The working distance for each sample was between 7-10 mm. Using a probing voltage of 20 keV, we expected to see K and L lines for P, Al, and Si elements and L and M lines for Yb. However, nothing but the host silica was detected. After review, and accounting for the overvoltage (factor of 1.5-3 for the given energy band) necessary in practice to adequately energize the material with the electron beam, we calculated that a minimum energy to excite the M1 X-ray line was under 20 keV, but the L3 transition exceeded this energy by about 6 keV using an overvoltage factor of 3. Although this energy was not sufficient to excite the L band of Yb, it caused the fiber to quickly accumulate surface charge. At this point it was only necessary to obtain a rough image of the core, however, even when the probe beam was aligned within the core, no positive identification of Al,

P, or Yb was made. A second attempt was made after tilting the mounting point towards the EDX to ensure that the probe and sample were well-aligned, however this yielded similar, ambiguous results.

NAA.

NAA was conducted by inserting three samples of YDF into the Rabbit Facility of the OSURR. This facility produced a total neutron flux of $3.2 \times 10^{12} \text{ n/cm}^2 \cdot \text{s}$, which produced a neutron dose of $4.0 \times 10^4 \text{ rad-Si}$ in the 15 minutes of irradiation. Gamma ray spectroscopy was completed 22 hours after irradiation with an in-house OSURR gamma spectrometer by NRL staff. The energy limit for this spectrometer was calibrated to 2 MeV, therefore higher energy γ s such as sodium's 2754.05 keV were not positively confirmed. However, as the weight mean uncertainty values show, close energy levels for the given elements did not interfere with a positive identification of the primary dopants. Only $^{141}\text{Cerium}$, with an uncertainty in the same order of magnitude as the mean activity, had this issue. Although the fiber has a natural cylindrical geometry, in order to avoid pulverizing a future radioactive source, the fiber was clipped to sample sizes of approximately 1 inch (about 17 mg) and a point geometry was used instead of cylindrical geometry. The clippings were made to fit within sample bottles used in the Rabbit.

The reference fiber is manufactured by INO, and the company was contacted to get the concentration of co-dopants within the core and inner cladding. The results of NAA are listed in Table 2. From Table 2 we can see that the ratio of Ytterbium in the two unknown fibers to the INO are 1/6 and 1/4, respectively. An estimation of concentration for the RE and the co-dopant aluminum may be made, although this would be a rough estimate as the fibers could not be mechanically stripped to the core diameter of each. Instead, both the size of the inner cladding and core were taken

into account and the fibers were stripped to their smallest diameter possible. One blade could not be used on all three fibers due to breaking below 125 μm . To further complicate the process, the INO fiber was not only doped with aluminum and P in the core, but also the inner cladding, giving the doping area of aluminum and P a larger size, thus leading to larger activity. Although we have confirmed the presence of aluminum and Yb in each fiber, their respective doping concentrations remain elusive, as the inability to mechanically strip to core sizes below 80 μm disallows an accurate measure of core weight. Additionally, because phosphorus has no (n,γ) reactions, it does not appear in this analysis, therefore it is unknown whether P makes up a higher or lower concentration than aluminum within the core. From the manufacturer of the experimental fibers, both YDF 6/125 and YDF 20/400 had claddings doped with Fl, however no Fl was detected via NAA. The percentage of Al_2O_3 and P_2O_3 in the core and cladding may be confirmed by INO in the future, and a request for the recorded counts of γ s for each sample should allow a better determination of the elements present in the sample by determining the yield γ s/decay and using the weighted activities per elements to back calculate from the total mass of each element present in the fiber [Bevelacqua]. Another consideration in this calculation must be the neutron cross section used to calculate the mass, as the neutrons produced by the reactor are not monoenergetic. This both effects the cross section value (σ) and the flux reacting with the sample (ϕ) in the equation

$$\mathcal{A} = 0.0602 \cdot \frac{m}{A} \cdot \sigma \cdot \phi \cdot (1 - e^{-\lambda t}), \quad (8)$$

where m is the mass, $\mathcal{A}$ is the activity, A is the atomic weight, λ is the decay constant of the given element, and t is time of irradiation.

Bromine and lead mixed with antimony were both used as shielding materials during the OSURR irradiation portion of the experiment. For lead, bromine, and antimony, given a mass of 1 μg (simple contamination), all produce decay products

Table 2. The table below lists the weighted mean activity of each identified element. This analysis was used to determine the relative doping concentration of the RE and its co-dopants.

Fiber Type Nuclide Names	Core Diameter μm	Inner Cladding Diameter μm	Dead Time (%)	ID Conf.	Weight Mean Activity ($\mu\text{Ci/ea}$)	Weight Mean Activity Uncertainty (1σ)
INO Fiber	40	90	0.20			
NA-24				0.988	4.0186387E-03	1.0075606E-04
BR-82x				0.414	2.2114960E-04	1.2403206E-05
YB-169				0.443	1.4693268E-03	2.7370077E-05
YB-175				1.000	9.8463334E-02	5.1336754E-03
6/125 YDF	6	125	0.06			
NA-24				0.988	1.2384877E-03	4.9994549E-05
SB-122				0.898	9.1041191E-05	8.3092418E-06
YB-175				0.729	3.3446429E-03	2.4436566E-04
20/400 YDF	20	400	0.06			
NA-24				0.986	1.8151339E-04	2.1218628E-05
TE-125m				0.963	3.8086160E-03	5.7929900E-04
CE-141				0.958	3.9800366E-06	6.5158270E-06
YB175				0.863	6.9194585E-03	1.2530775E-04

that are isotopes (n,act) of the parent (given an irradiation time of 15 minutes and an acquisition time after irradiation at 22 hours). Yb has a (n,act) as well as a β decay, with all products as isotopes except the β decay (Lu176 in the unidentified peaks portion of the report).

Unlike the above potential contaminants, the presence of ^{141}Ce in the 20/400 YDF (named nuclide) is unexpected. The reaction Pr-141 (n,p) Ce-141 has a half life of 32.5 days and, per the NIST activation calculator ref NIST website, at least $0.01 \mu\text{g}$ of Pr must be present to have any significant activation at 22 hours. As mentioned before though, the uncertainty is greater than the given activity for this element, despite an ID confidence close of 0.958 indicating the presence of ^{141}Ce may be a simple software limitation. Conversely, the software may simply indicate that accurate activity may not be possible, but the presence of ^{141}Ce is confirmed. This remains to be investigated in conjunction with the OSURR staff. The presence

of ^{75}Ge , a common dopant used to raise the index of refraction, in both samples (unknown peaks) was also surprising at first. However, on closer examination, ^{75}Ge has energy peaks close to those of ^{169}Yb , and more ^{169}Yb peaks (63.28 keV and 130.44 keV) besides the 198.02 keV close to Ge were identified, indicating that this unknown element is probably ^{169}Yb . ^{169}Yb was confirmed with an ID confidence of 0.443 in the INO manufactured fiber.

3.5 Neutron Generator Setup

The setup started with preparation of the fiber. Scissor cut YB1200 20/400DC spooled fiber was cut to between 28-31 cm, stripped, cleaved, and terminated with Thorlabs BFT1s and SMA905 MM Connectors ($\varnothing 410\ \mu\text{m}$). Once fiber preparation was completed, the DD109 startup sequence began (fibers were outside the neutron generator room). A Bonner sphere was placed as shown in Figure 13 as one of the indicators that the neutron generator was working properly. A 5V switch was connected to the outside of the generator room to control turning the neutron generator accelerator on and off. After initial setup of the neutron generator was complete, the 5V power supply was turned on, effectively reducing the current to zero and shutting off neutron production. The prepped fiber's BFT1s were connected to FG105LCA patch cables (25 m length). These in turn were connected to a SLS201L and the CCS175. Once the timed sequence on the SPLICCO software for the CCS175 was set (samples taken every second) and the signal was set to between 0.9 and 1.0 intensity (by varying integration time), the 5V power supply was shut off, allowing current to flow and produce neutrons. Readings were taken from 30 minutes to 6 hours.

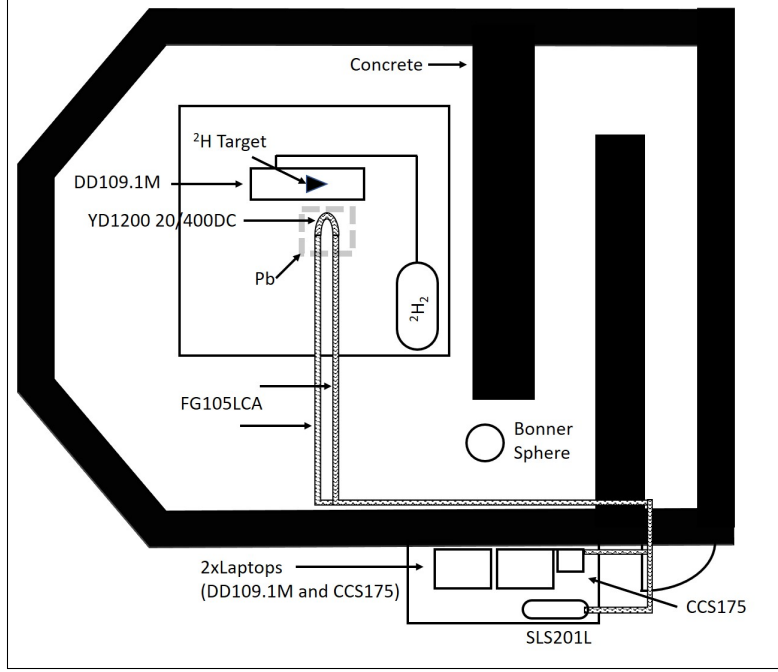


Figure 13. The figure above shows the large working area for high-Z shielding materials. The thick concrete walls ensured that neutron radiation did not affect the light sources or spectrometers. Unfortunately, due to a low originating neutron flux, the cross section of interaction between the neutron flux and a fiber with micron order of magnitude radii was small.

Preliminary Results from the Neutron Generator.

Per the manufacturer's website, the neutron generator produces approximately 2×10^9 n/s. At a direct line distance of about 0.2 m from the source, the fibers are bombarded with approximately 3.8×10^8 n/m²/s, yielding fluences at 30 min, 3 hours, and 5 hours of 6.94×10^8 n/cm², 4.16×10^8 n/cm², and 6.94×10^9 n/cm², respectively. These fluences were at least 1-2 orders of magnitude below literature values used to cause defects in amorphous silica and doped amorphous silica [21] [57] [35]. The results show that no discernible defects were detected, as seen in Figures 14,15, and 16. The first plot shows the changes in intensity over all wavelengths, while the subsequent side by side plots show changes at specific wavelengths.

This information allows us to better understand the minimum neutron flux necessary to produce defects. Although the total dose would increase over time, the

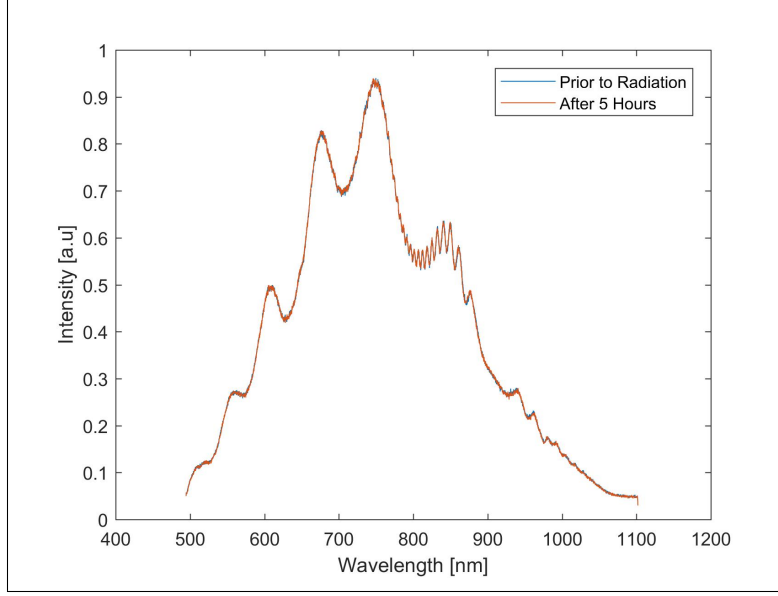


Figure 14. Although it appears as one plot, there are actually two. The first is the initial intensity plot prior to radiation, and the second is the plot after 5 hours of radiation. Note that the response from the light source is not subtracted.

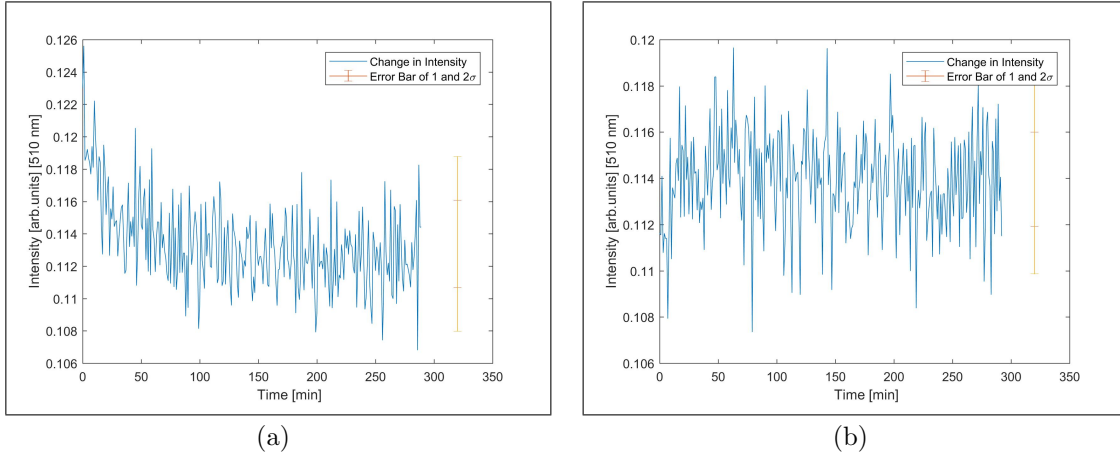


Figure 15. As can be seen above, the Figure 15(a) was generated with no radiation over 5 hours while the Figure 15(b) was subjected to radiation for 5 hours (510 nm). No noticeable differences in each and no trend showing an increase in attenuation or emission were observed for this arbitrary wavelength. Other random wavelengths at different signal strengths were checked with similar results.

dependence on dose rate over total dose is evident here. The OSURR produces a flux 3 orders of magnitude greater than the neutron generator, albeit at similar energy levels, with the majority of neutrons having thermal neutron energy levels (< 0.25

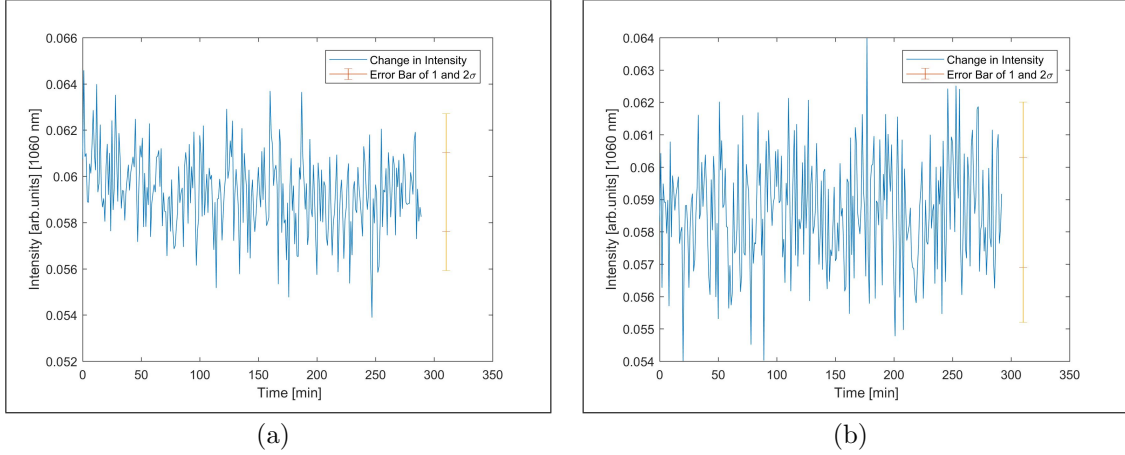


Figure 16. As can be seen above, Figure 16(a) was generated with no radiation over 5 hours while the Figure 16(b) subjected to radiation for 5 hours (1060 nm). No noticeable differences in each and no trend showing an increase in attenuation or emission was observed.

eV).

3.6 OSURR Setup

The OSURR setup also began with fiber preparation. Unlike the scissor cut fibers of the DD109M setup, all active fibers used with the OSURR were precut (0.3 and 1 m for the 6/125 μm fiber and 0.3 m for the 20/400 μm fiber) and permanently terminated by the manufacturer. If passive matched fiber was used, it was prepared as above with the exception that a 550 cord shell jacket was fashioned for the bare fiber to protect it from damage. After sufficient time to allow activation to decay to safe levels, irradiated passive/patch fiber was cut and disposed of and a new end was stripped, cleaved, terminated, and measured with the CCS. If patch cable was used, the jacket and plastic tubing were removed and the coating stripped. All non-permanent terminations were completed using either $\varnothing 410 \mu\text{m}$ (MM) or $\varnothing 125 \mu\text{m}$ (SM) connectors. Prior to termination of the ends, the fiber was fed through a hole on the lid of SPUD-D. Once this was completed, the active fiber was coiled to no

larger than a $\varnothing 2.54$ cm circle to allow uniform radiation of as much fiber as possible. The fiber was held in place on the shielding plate by painters tape and the ends were connected to either patch or passive matched cable. Once all plates were in position within SPUD-D, the cylindrical body was rotated to align with marked grooves on the outside of SPUD-D indicating that the face of the plates were normal to the primary direction of radiation from the fuel rod case. The mounted fiber plate was farthest from the radiation source.

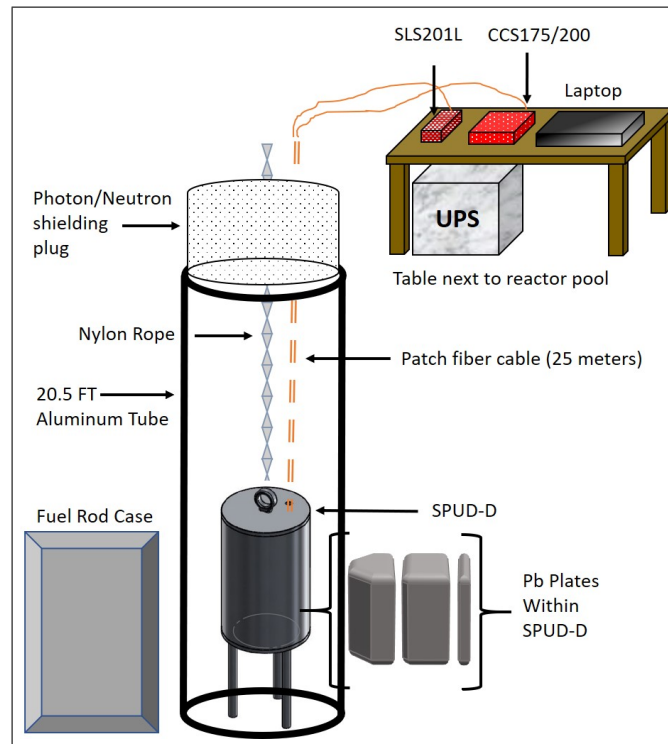


Figure 17. SPUD-D was situated such that the centerline of flux from the fuel rod case interacted with SPUD-D at exactly 12 inches from the floor. Note that passive/patch cables were not shielded outside of SPUD-D; the weight added to shield the patch cables was prohibitive as the rig would have to be manually lowered into position.

SPUD-D was then connected to a 0.35 ton-rated nylon rope and lowered to the bottom of the aluminum tube. As anticipated, SPUD-D rotated as it was lowered and once the aluminum tube was placed immediately next to the reactor, the tube itself was rotated to ensure proper alignment. Direction was checked using a LED flashlight

and binoculars. The distance to the lid was checked using a LASER rangefinder. This was completed to ensure proper sitting of the SPUD-D on the 7-inch dry-tube floor. The plug was lowered using an overhead crane and the fiber cable and rope were fed through a cut, single helical groove on the edge of the plug. The fiber was then taped to the concrete surrounding the pool and initial readings were taken after all parts ceased movement. For the OSURR, both fast (minimum of 1 pass every second) and timed (1 second) CCS settings were used to record data beginning at the time when the power of the reactor surpassed 1%. Typical run times ranged from 30 minutes to an hour including the startup sequence to get the OSURR from 1% to 90%. Once the fibers reached RIA saturation or the intensity reading held relatively stable, they were allowed to recover for 10-15 minutes prior to increasing reactor power to 90% a second time, after which the reactor was shut down. This was completed to test the annealing times of each fiber after varied max power periods. The time at 90% varied by run from 5-45 minutes.

To establish redundancy in measurement with previous experiments at the OSURR, a pyramid-shaped rig made of two aluminum bands was utilized. All of the elements in Figure 17 were the same with the exception of SPUD-D. Radiation times were established using the saturation point as above, generally taking about 25 minutes from startup to shutdown. Recovery times were measured through about 15 minutes.

For the measurement of neutron fluence, the flux wire method was used. The 5 wire dosimeters used were cobalt, copper, iron, nickel, and titanium. Neutron energy levels from 1×10^{-2} to 1×10^4 eV and about 1×10^6 to 1×10^7 eV may be detected with these elements via (n,X) reactions. SAND II software is used to determine counts. Note that because no “initial guesses” are fed into the software for energy ranges from 1×10^4 eV and about 1×10^6 (the lower range of “fast neutrons”), the

software will interpolate these counts.

For photon measurements, two each Landauer InLight[®] nanoDot[™] dosimeters were mounted at the shielding wall, between the nearest and second nearest plates to the reactor, and to the mounting point of the fiber to establish γ attenuation significant enough to allow for the majority of the RIA produced as a consequence of neutron fluence.

IV. Results and Analysis

4.1 OSURR

The OSURR facility was used not only to irradiate samples, but also to provide information on neutron total dose and dose rate, γ total dose and dose rate, and conduct Neutron Activation Analysis (NAA) of the fiber. NAA, in conjunction with two methods of spectroscopy at the AFIT, were used to determine the presence of dopants and their respective concentrations.¹

The OSURR staff conducted a flux wire experiment, as described in Chapter 2, to determine the neutron flux within SPUD-D, however unforeseen fusion in the shielding materials due to thermal radiation caused the analysis of results to be halted. The neutron flux was then determined using the original measurement by OSURR staff in 2013 [77]. This substitution is acceptable as the neutron capture cross sections of Al, the elements SPUD-D is made of, and Pb, the shielding material, are low enough that the neutron flux is not significantly affected. Figure 18 shows the neutron spectrum based on the decay of the flux wires. Using the fluxes given in the chart with thermal and fast neutron cross sections may facilitate material characterization as well as detailed information on defect creation.

InLight® nanoDotTM Dosimeters were positioned in three places, two each, to measure the attenuation of γ radiation with Pb shielding. The first was at the shield line (no Pb shielding), the second was behind the first Pb brick (max width of 5.08 cm), and the third was behind both the mounting shield (1.27 cm) as well as the middle Pb brick (6.35 cm) for a total Pb shielding of normal incident γ radiation of

¹It is important to note the the clocks for the linear, logarithmic, and actual GPS time are not synced within the reactor software. Thus, a lag of approximately 42 seconds is sufficient enough that when plots are created without accounting for this time discrepancy, it appears as if the fibers are gaining in intensity transmission even when the reactor is at full power. Once the times were synced across the three independent systems, fiber behavior followed existing literature.

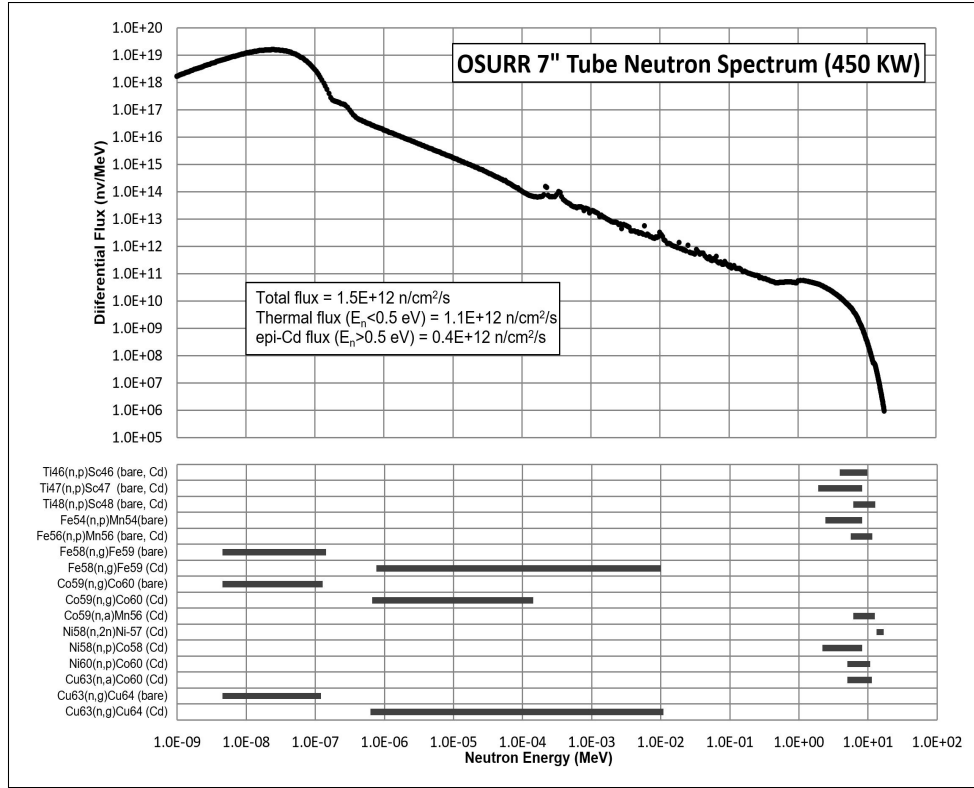


Figure 18. Note the two ranges where elemental decay limitations exist. The SAND II system was used to determine both regions from 1E-7 MeV to 1E-6 MeV and 1E-2 MeV to 1.0 MeV. SAND II predictions for the OSURR were separately verified by [83] using MCNP. Courtesy OSURR Staff [[77]]

11.43 cm. As the first was wedged between a Pb brick and the Al surface of SPUD-D and the second was wedged between two Pb bricks, hammering of the surface was conducted to create an indentation for housing the two nanoDots. Although the nanoDot is classified as a Radiologic Quality Assurance Instrument, and may not be used for primary dosimetry readings, its sensitivity and range (0.93 and 5keV-20MeV) are sufficient enough for a characterization of attenuation due to γ radiation. The accuracy of the nanoDot is $\pm 10\%$ and $\pm 5\%$ for the photon energy range of 5 MeV-20 MeV while the precision is $\pm 5\%$. The nanoDots were calibrated using a ^{137}Cs source. Unfortunately, the total dose operating range peaks at greater than 100 Gray, effectively forcing the OSURR staff to maintain the power of the nuclear reactor at 1 kW, or 0.2%. In order to avoid saturation, the run time for this procedure was 20

minutes. Table 3 displays the results of the experiment without correction for linear, but not proportional relationships.

Table 3. The conversion factor used between rads and Roentgen was 0.877 as the ratio of the mass attenuation coefficients for silica and air is close to 1 for the energy range of γ s radiation incident on the nanoDots. This table is used to extrapolate what the dose/dose rate due to γ s radiation is at 450 kW.* Exceeds analytical capabilities. Dose provided is an estimate.

	nanoDot Serial #	Amount of Shielding (cm)	Dose at 1 kW for 20 min (rads)	Dose at 1 kW for 60 min (rads)	Dose (R/hr)	Dose at 450 kW (R/hr)
	DN08631799E	11.43	861.75	2585.30	2947.84	1326529.16
	DB081154310	11.43	903.00	2709.00	3088.90	1390021.90
	DN08752475O	5.08	1,010.23	3030.68	3455.74	1555082.24
	DN08754315U	5.08	1,048.55	3145.64	3586.81	1614066.60
*	DB08315094U	0.00	14,677.20	44031.59	50207.05	22593173.83
*	DN08630947K	0.00	12,653.46	37960.38	43284.36	19477959.78

Interestingly, the attenuation after γ path lengths of 5 cm decreases dramatically. The attenuation after γ path lengths from 5 cm to 11 cm is significantly less than from 0 to 5 cm. This may be because the radiation spectrum is initially hardened, then softened as it scatters within the rest of the lead shielding until it exits the mounting plate. During initial calculations, the mass attenuation coefficient for Pb was obtained for γ energies of 1 MeV. As the γ spectrum for the nuclear reactor is unknown, the ratio of lower energy to higher energy photons was unclear. It could be that there is a significant lower energy γ spectrum, however this does not take into account the softening of the spectrum after the initial low energy γ rays are absorbed. This attenuation reading does not take into account the $1/r$ ratio of dispersion for a cylindrical source, although the total width of SPUD-D normal to the reactor core is only 12.7 cm, and decrease due to r is not pronounced.

Upon review of the extrapolated doses at 450 kW, it was apparent that the method was overestimating the dose by at least factor of 2. This ratio was determined using the expected dose rate of 8.7×10^6 R/hr measured experimentally at 450 kW at that

position. The OSURR staff have experimental γ exposure rates for power at 0 kW, 3kW, 10 kW, 30 kW, and 100 kW (0.00%, 0.60%, 2.00%, 6.00%, and 20.00% power, respectively). It appears that although all of the graphs of exposure rate are linear, they are not proportional (i.e. the slope of a 5 kW exposure rate is not 5 times greater than a 1 kW exposure rate). Therefore, the multiplication of dose rate at 1 kW by 450 to give the dose at 450 kW is incorrect.

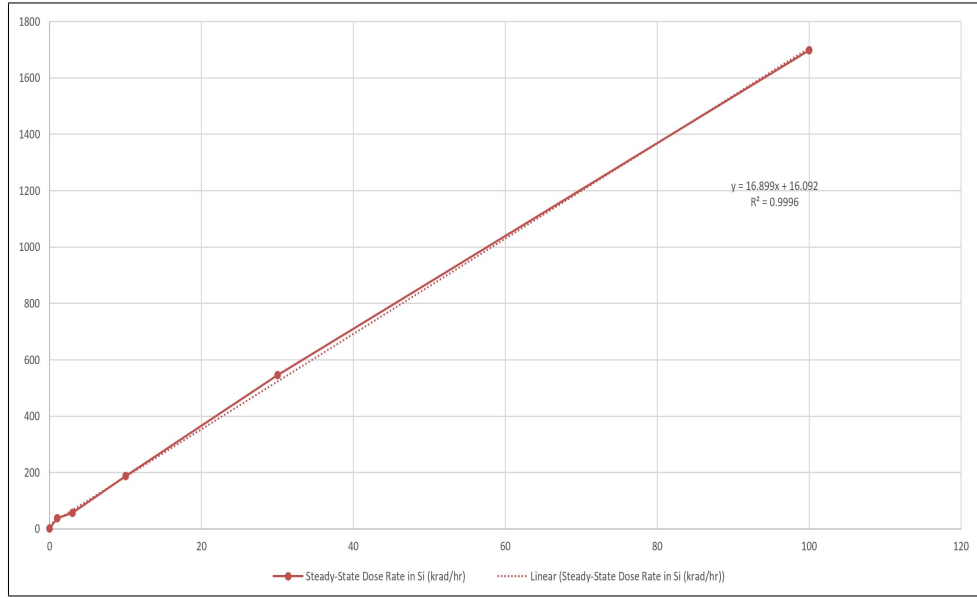


Figure 19. The fit does not cross at [0,0] indicating that even when the reactor is at ‘0%’, there is still a dose present. Although the point at 0.2% (power during which nanoDots were embedded in the lead shielding), an R^2 value close to 1.0 indicates a good prediction of full power. This matches well with [6] and [79].

Although the neutron dose is calculated to be 65.815 krad(Si)/hr from [6], the photon dose will be at at least an order of magnitude different and dependent on the amount of shielding present in front of the sample. To ensure an accurate dose rate for γ radiation, an analysis of the 4 slopes at 4 powers was conducted to attempt an extrapolation of the γ radiation at 450 kW. A linear fit of the data provided by NRL resulted in an $R^2 = 0.9996$, ensuring that the predicted value for the surface dose rate at zero shielding is accurate. From there, and using the given data from the nanoDots, an exponential fit was applied to the two datasets provided by the

nanoDots, with an initial $R^2 = 0.7379$. A second attempt, this time using MATLAB in place of Excel, yielded a much better fit with $R^2 = 0.9946$. Using this exponential equation with the initial intensity (dose rate) set to the extrapolated value of 7621.00 krad(Si)/hr (within 94.48% agreement with [6]). The approximate dose rate was found to be 7621.00 krad(Si)/hr at 0.0 cm lead shielding, 292.50 krad(Si)/hr at 6.35 cm of shielding, and 21.60 krad(Si)/hr at 11.43 cm of shielding. Since nanoDots were not used immediately in front of the mount plate, the dose rate was interpolated at 1.27 cm to be 3970.5 krad(Si)/hr. Adding both the neutron and photon radiation dose rates for each shielding scenario results in an estimated dose rate as seen in 4. [47]

Table 4. This table shows the percentage of neutron dose that makes up the total dose received from the nuclear reactor. Because the neutron dose is unaffected by the shielding material, it stays the same as the γ radiation dose is attenuated in the shielding.

Pb Shielding (cm)	Total Dose Rate krad(Si)/hr	Percent n ⁰ Radiation (%)
0.00	7686.80	0.86
1.27	4036.30	1.63
6.35	358.32	18.37
11.43	87.42	75.29

4.2 6/125 YDF Irradiation

The 6/125 YDF fibers were irradiated four times with time intervals as seen in Table 5. FP represents the time the reactor reached full power or ended an iteration at full power while the 5% simply indicates the points at which the reactor reached 25 kW, the minimum point at which the fiber is considered to begin irradiation, chosen arbitrarily.

The wavelengths were chosen to correspond to peaks for defects including, but not limited to, ALOHCs (1) and NBOHCs (5) found in Borman’s 2015 work [6] through Gaussian deconvolution. Standard features of the fiber itself such as the absorp-

Table 5. This table summarizes the the level of shielding and the amount of radiation received at 90% reactor power and the annealing time (taken as the time the reactor stays at or below 1% power). For all graphs in this thesis, the first power up to 90% was used to identify radiation defects and their growth rates, while the last power down is used to describe annealing in the fiber.

Reactor Power (%)	No Shielding	11.43 cm Pb	6.35 cm Pb (Recorded)	6.35 cm Pb *	1.27 cm Pb	Average
1st 5%	11:16:40	12:15:30	12:49:40	12:51:20	15:17:10	
Last 90% 1FP	11:25:20	12:25:00	13:01:50	13:09:05	15:29:10	
2nd 5%	11:46:30	12:40:00	13:17:30	13:24:45	15:40:20	
Last 90% 2 FP	12:03:00	12:49:30	13:21:20	13:28:35	15:47:20	
3rd 5%	12:04:00	12:50:30	13:22:15	13:29:30	15:48:20	0:01:40
1 FP Time at 90%	0:08:40	0:09:30	0:12:10	0:10:45	0:12:00	0:10:35
2 FP Time at 90%	0:16:30	0:09:30	0:03:50	0:03:50	0:07:00	0:09:12
Total Time at 90%	0:25:10	0:19:00	0:16:00	0:21:35	0:19:00	0:19:47
1st Anneal Time (at 1%)	0:21:10	0:15:00	0:15:40	0:15:40	0:11:10	0:15:45
Data Sets start time	11:16:41	12:14:10	12:42:25	12:42:25	15:12:36	

tion/emission cross section peak at 976 nm are included to monitor changes at these critical wavelengths. This wavelength was also included because it gives an indication of the relative amount of doping in the samples of fibers. Borman states that of the two fibers, the 6/125 YDF has the higher doping concentration by 2.2 times over the 20/400 YDF [6]. Because the fibers are similar in function and are comparable despite the manufacturer, this ratio is assumed for the fibers in this experiment, pending material characterization. Additionally, defects center energies found in [18] are used to further interpret Gaussian deconvolution. Interstitial defects such as O₂ interstitials could not be evaluated due to the wavelength limitation around 1000 nm.

Gaps in the data exist because of the limitations on data counts for the CCS200 and experimenter limitations. As the software can only sample 1000 points at a time, regardless of integration time, there are periodic gaps of about 30 seconds.

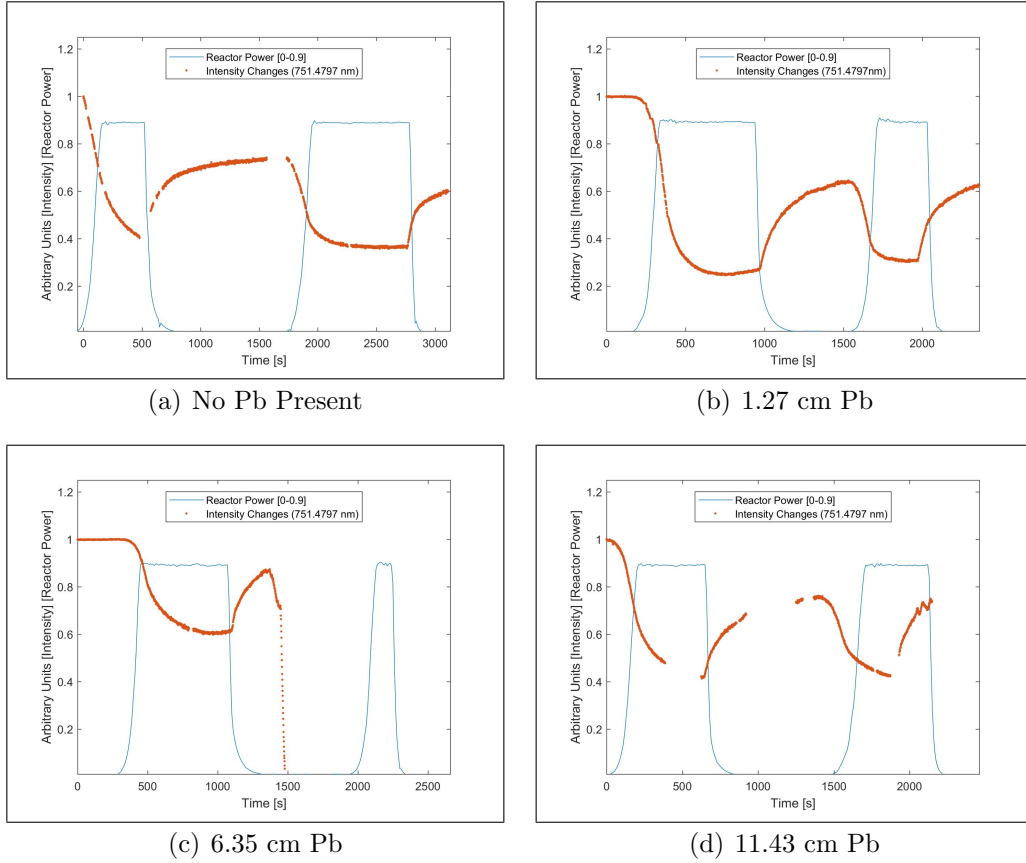


Figure 20. These figures show a typical dataset for a fiber at a specific wavelength along with the corresponding changes in reactor power in time. Note the similarity of response between (a) and (d), indicating a possible rotation of SPUD-D prior to irradiation resulting in no γ radiation attenuation by the 11.43 Pb shield. Subfigure (c) is also interesting in that typical breaks in the fiber instantaneously drop the intensity to 0, whereas (c) gradually drops to 0 and stays there for not only the shown wavelength, but all wavelengths. This could indicate creeping in the fiber, which could either be a factor of temperature, mechanical stress, or both.

Additionally, unidentified glitches in the software also resulted in periodic skipping of iterations, sometimes by three or four seconds. The accumulation of these skips were accounted for as well. All wavelengths were normalized to the pristine condition for intensity calculations and graphs, regardless of RIA or RIE contributions later during nuclear reactor irradiation. Thus, what appears to be RIE is shown with intensity values above 1.0 in the graphs. However, intensities were not normalized for RIA as the RIA equation accounts for the relative change in intensity.

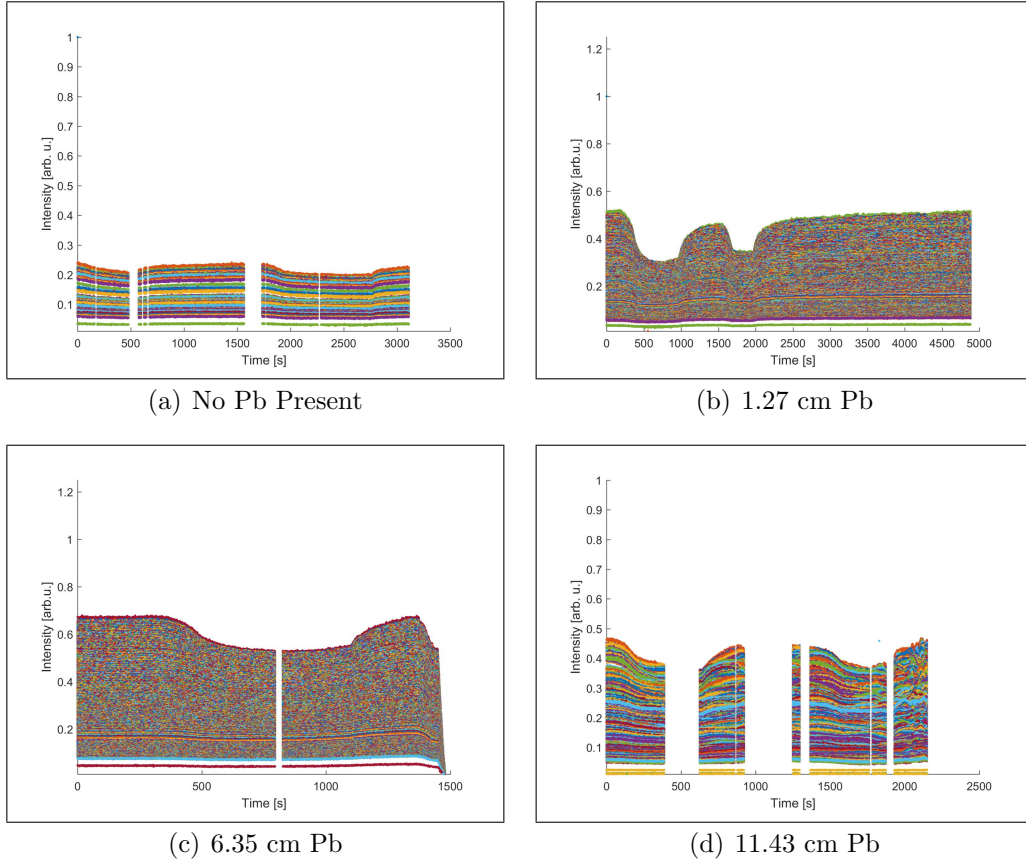


Figure 21. The four figures demonstrate many plotted wavelengths with no readily discernible correlation (e.g. 500 nm and below measurements had RIE characteristics) between wavelengths and the presence of RIE. These graphs point out that there is no coordination between initial intensity, wavelength, and shielding status for what may be RIE.

Figure 21 is not intended to demonstrate the changes in intensity by wavelength, but rather to display the density of wavelengths that appear to have RIE after 5 minutes of irradiation. If we consider that (a) and (d) had no effective attenuation of γ radiation because (a) had no shielding and (d) was improperly positioned during irradiation, these graphs imply that even a small amount of lead shielding leads to what appears to be RIE in a significant number of wavelengths (between 1/3 to a 1/4 of over 3000 recorded wavelengths) and lesser so in unshielded samples. Originally, the thought was that the intensity must be close to 0 at many wavelengths and therefore

a small uptick may result in what appears to be RIE but is actually simple oscillations within the CCS200s' resolution limits. Of the defect wavelengths mentioned above, 920 nm, 975 nm, and the cutoff wavelength at 1010 nm all showed RIE. It is known that RIE may be masked by the weight of RIA, and once RIA is removed RIE effects show [21]. Therefore, these wavelengths could potentially have RIE even from initial radiation above a reactor power of 25 kW. However, minute differences in intensity may also cause artifacts within the sample, as nothing in the literature indicates that the dose rates used cause RIE. Typically, the difference between the initial and peak intensity after irradiation is small, leaving only a span a few hundred nm wide that shows significant RIA, as in Figures 23 and 30. The idea that this may be fluorescence seems doubtful as well because fluorescent states are not long-lived; it is unknown whether a phosphorescent state was excited at this time, however no literature suggests this except in the case of pulse photon radiation [21].

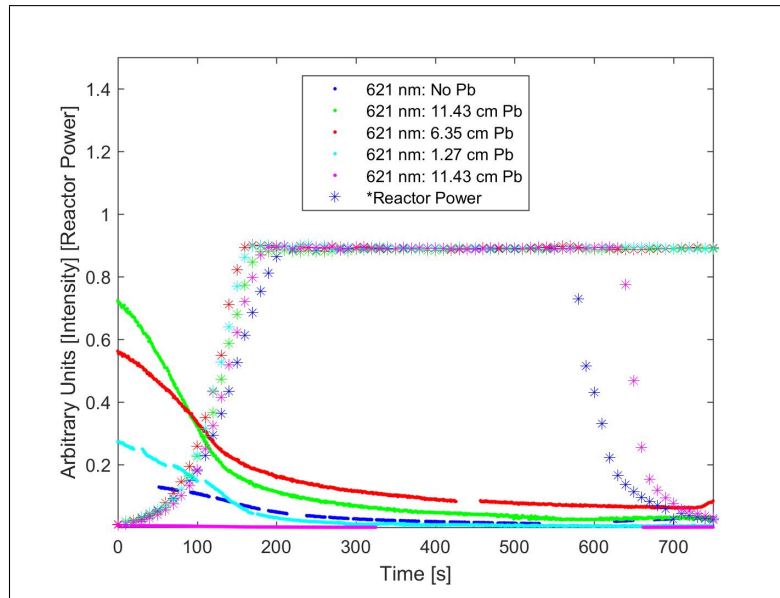


Figure 22. The 621 nm wavelength was chosen as one of several that represent a potential defect center peak. Although the data shows a descent as reactor power increases (asterisk plots), it is clear the the 11.43 cm data set drops faster than the 6.35 data set. This graph is not normalized so that similar integration times for each sample are shown.

Figure 22 represents the data used in RIA analysis using intensity. Because none of the fibers had the same integration time, it may indicate that the efficiency of some fibers was not as good as others. Since the same light source was used for each type of fiber (i.e. the same tungsten lamp was used for all 6/125 YDF), as well as the same patch cable, it appears that the integration time may be a good indicator of transmission efficiency.²

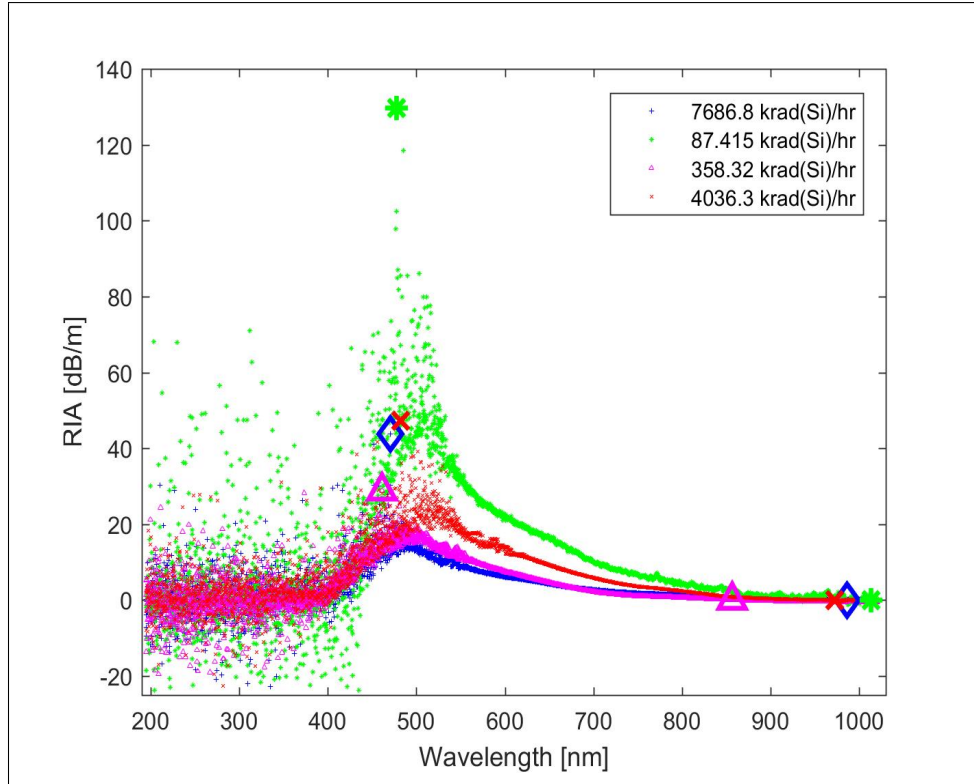


Figure 23. The RIA calculated for the CCS 200 wavelength 830 seconds after the OSURR surpassed 1%. The initial intensity below 475 nm was very small, resulting in largely varying RIA calculations below this wavelength. Note the consistent clustering for all dose rates indicating the minimum and maximum RIA for each dose rate.

Once data was collected (in units of intensity), the initial and final readings for each of the four instances of varying shielding were taken. Figures 23, 24, and 25

²Note that the integration times sometimes varied as much as two orders of magnitude, even in a setup that used the exact same accessories and equipment. All fibers were swiped clean prior to use, however intrinsic differences in the irradiated fiber cannot be discounted as the fibers were not materially characterized. These intrinsic differences may be attributed to slight differences in concentration, deposition, or losses created when the SMA or FC/PC ends were spliced to the fiber.

indicate a general clustering for both the minimum and maximum RIA shown. The large variance in RIA values below certain wavelengths may be due to the optimum ranges for the given fibers, such as the SM 6/125 YDF. The cutoff wavelength for this fiber is calculated using Equation (5) and is well above the CCS scanning limits. However, the MFD of the 6/125 YDF is such that more than one mode may propagate through, as the limit for D to ensure only one mode propagates is given by Equation (4) to be between 0.3 and 2 μ for a wavelength ranges from 200-1200 nm. [18] states that when a fiber is transmitting light outside of its optimal SM wavelength range, these changes in modes result in ‘sudden’ changes in RIA. A low signal due to the range limitation on the lower end of the spectrum (due to the tungsten lamp, whose intensity is low at 500 nm and below) may also account for these RIA anomalies at the scanning limits.

Figure 25 demonstrates an anomaly isolated to the highest shielded intensity readings. An oscillatory, aperiodic data set appears as early as 775 nm and continues through 1000 nm, but only for the 11.43 cm data set. Negative intensity values from the CCS indicate artifacts, however in this case it appears to be an emission center around 975 nm, consistent in all four data sets. This is difficult to ascertain though as the RIE, though present, is also small (less than a dB/m).

Gaussian deconvolution using the unshielded, irradiated fiber data and the 11.43 cm shielded, irradiated fiber data was completed to compare the effects of γ radiation versus neutron radiation. Unfortunately, because of the large variance at both ends of the spectrum, it was difficult to deconvolve defect centers outside of the 550-1000 nm (1.1-3 eV) band, thus leaving the wavelength-dependent effects of neutron radiation below approximately 500 nm inconclusive. We can speak to the magnitude of these defects though. As seen in Figure 23, the 11.43 cm shielded run had a peak RIA three times as high as the lower shielded run. A better comparison is made between

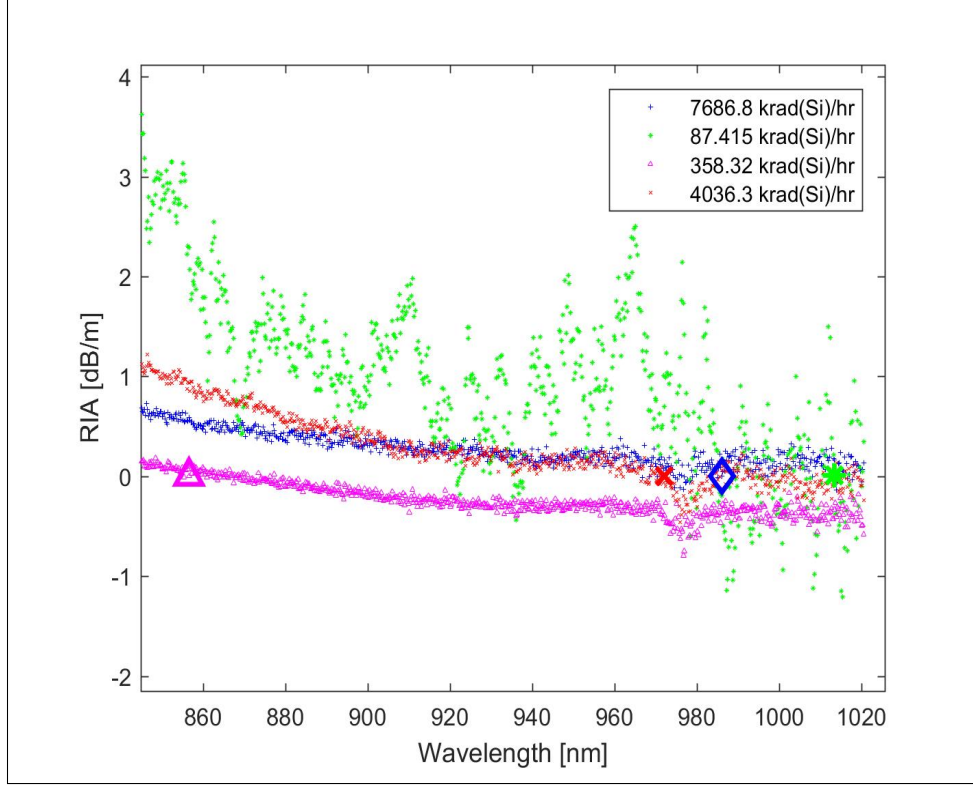


Figure 24. The RIA calculated for the CCS 200 wavelength 830 seconds after the OSURR surpassed 1%. A cutout of Figure 23 shows that only the most heavily shielded fiber had significant variation in RIA between 900-1000 nm, a typically stable RIA region as significant RIA in a Y/P/Al fiber is attributed to the codopants. This region also includes the emission wavelength of Yb for the $^2F_{5/2} \rightarrow ^2F_{7/2}$ transition, which may be seen in the small dip near 975 nm. [18].

a dose rate of 358.32 krad(Si)/hr and 87.42 krad(Si)/hr, as these two are within an order of magnitude of each other and represent a 80% predominant γ radiation dose rate and a 75% predominant neutron radiation dose rate, respectively. A shift in the minimum RIA of approximately 20 nm between these two data points is shown in Figure 25. RIA values above 30 dB/m, shown in Figure 26, are concentrated around 2 eV and 6 eV, however the latter is well within the artifact zone due to the limits of the CCS 200. Table 6 details the values for the defect centers, their widths, and amplitudes for the 11.43 cm and 6.35 cm shielded datasets where the total dose is similar.

RIA is significantly larger for 11.43 shielded fibers, despite the three order of

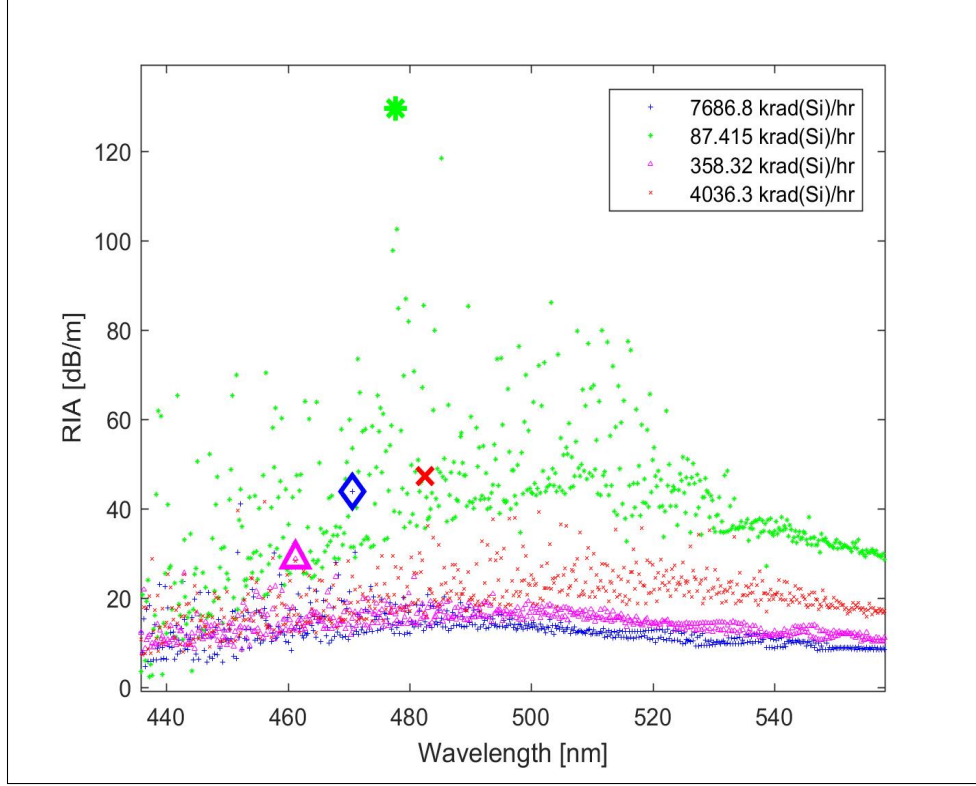


Figure 25. The RIA calculated for the CCS 200 wavelength 830 seconds after the OSURR surpassed 1%. A cutout of Figure 23 shows that the maximum RIA value for each dose rate clusters around the 450-500 nm region, consistent perhaps with neutron induced POHC defects (if P is present). Note the order of magnitude increase in RIA when the majority (75%) of the radiation is from neutrons.

magnitude difference in dose rate. Despite this, a cluster of high RIA near the defined portion of the spectrum indicates that pure a pure radiation dose rate may increase the RIA drastically. It is known that, up to the same total dose, γ radiation causes losses 2.5 times faster than neutrons, however care must be taken to consider the effects of neutron-induced secondary ionizations in the fiber, as these are more prominent in the absence of a significant γ radiation dose rate [38, 57].

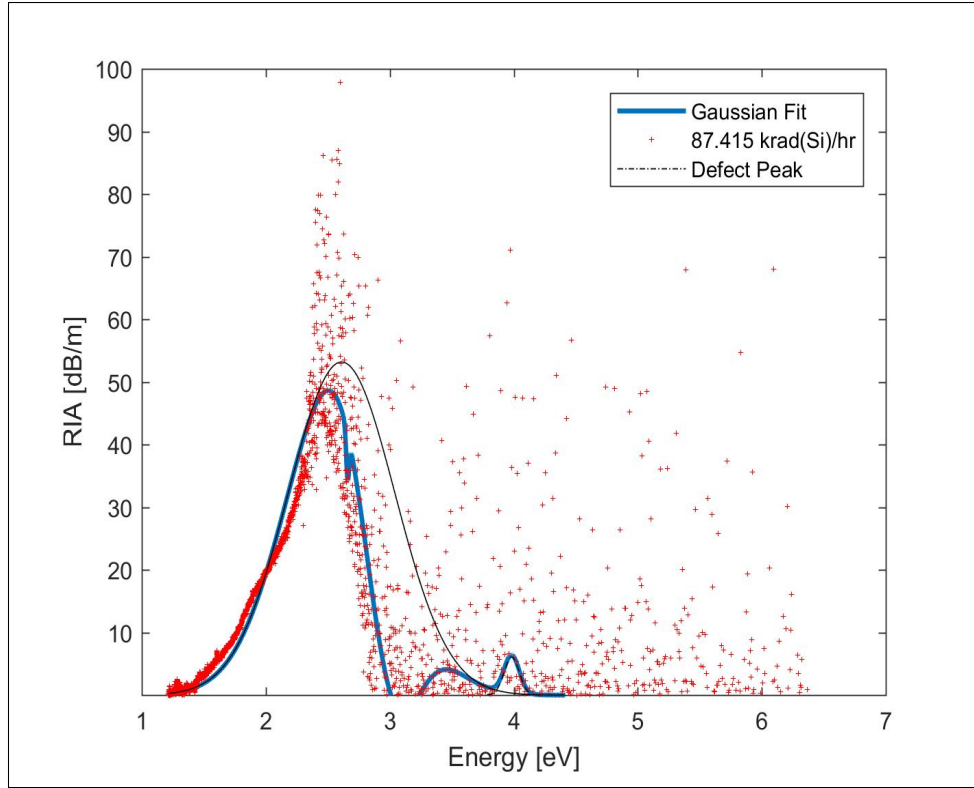


Figure 26. There is a the large variance in RIA above approximately 3 eV in this plot of RIA versus energy for a fully shielded fiber. The width of the defect peak close to 2.61 eV is more narrow, yet nearly three time higher. The corresponding wavelength, about 475 nm, corresponds to the behavior seen in Figures 23 and 25, again supporting the hypothesis that neutrons increase the defect magnitude and rate below 500 nm, as stated in [6].

Table 6. This table shows the percentage of neutron dose that makes up the total dose received from the nuclear reactor and the corresponding Gaussian peaks from deconvolution of the total Gaussian. The overall pattern and number of discernible defects is different between the two sources of radiation. The highest defect center amplitude shifts neutron radiation dominant dataset shows a substantial tail past 3.0 eV. Note the partial Gaussian close to 1.6 eV in the γ radiation predominant dataset. This feature is minimal for the same total dose where neutron radiation dominates.

Radiation Dose due to n° (%)	Peak Value (eV)	Width (eV)	Amplitude (eV)
75.29			
Defect 01	1.30	0.16	0.35
Defect 02	1.58	0.13	0.80
Defect 03	2.09	0.35	6.00
Defect 04	2.55	0.33	14.50
Defect 05	2.70	0.20	1.60
18.37			
Defect 01	2.32	0.47	22.84

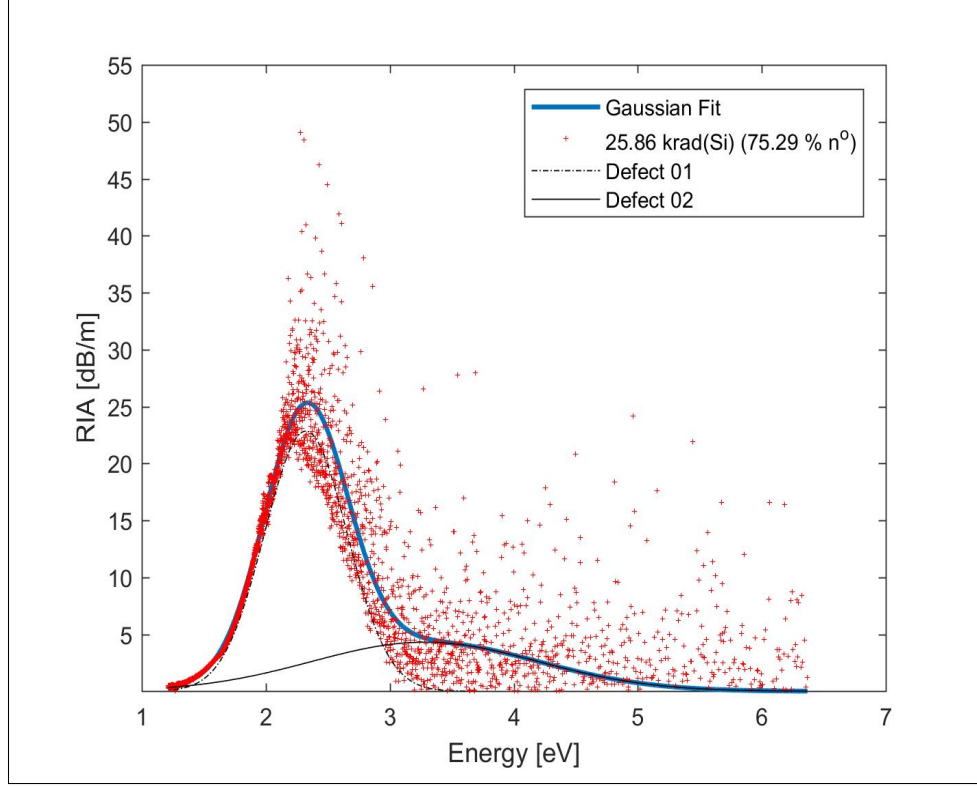


Figure 27. Two partial Gaussians are predicted by the Curve Fitting App in MATLAB, however the program does not account for experimentally observed defect centers. Note the shift in the highest defect peak located between 2-3 eV and the clustered points past 3 eV in this figure in comparison to the almost linear decay of the right tail in Figure 28.

Comparing the total dose instead of the dose rate after a certain time highlights the features that are prevalent for neutron radiation and γ radiation dominant doses. Unlike Figures 26, where the RIA is calculated in both using the initial intensity and the intensity at about 800 seconds, Figures 28 and 27, which have different dose rates but similar total dose, show much smoother Gaussians with definitive peaks that correspond to features in the respective total Gaussian. There is more similarity between these two than when taking a reading at a certain time for different shielding widths. Therefore, we can speak of similar peaks being shifted, with the γ radiation dominant dataset having an absolute peak to the right of the neutron radiation dominant dataset. The scales between these two figures differ because the

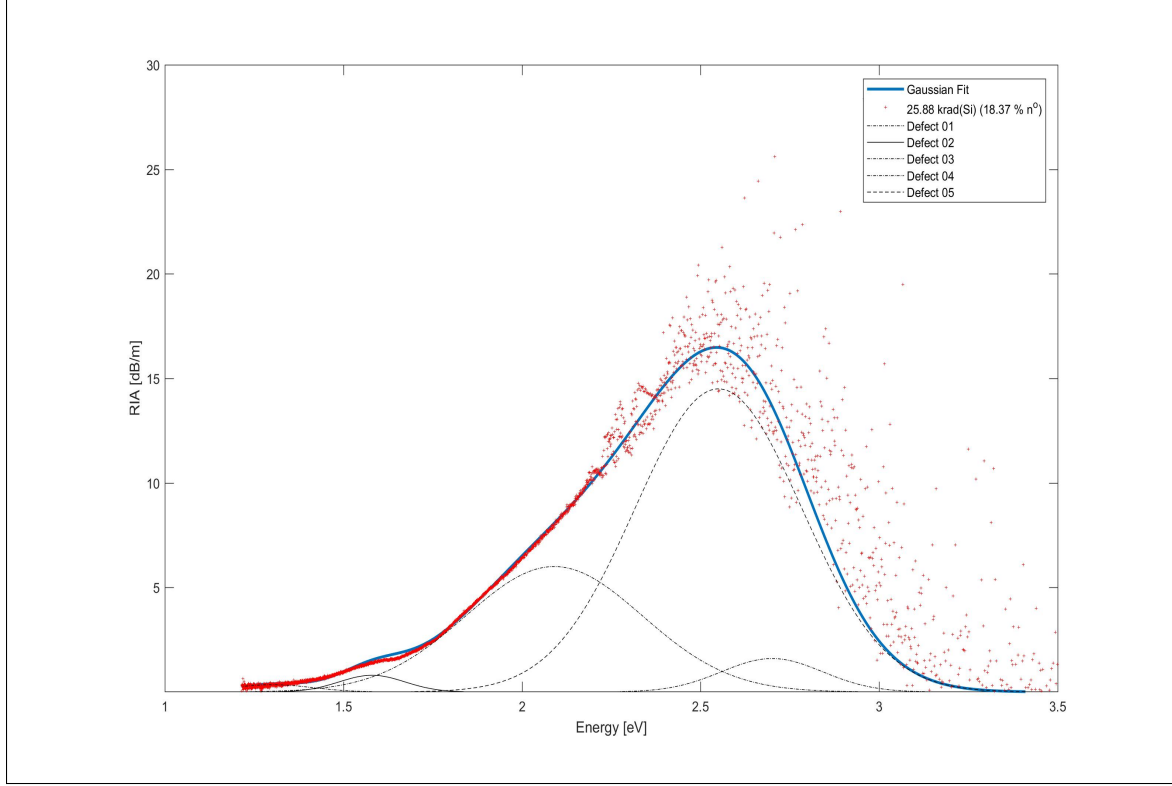


Figure 28. Several partial Gaussians are predicted by Curve Fitting App in MATLAB, however the program does not account for experimentally observed defect centers. Because of this, only the location of the peaks for the two radiation sources are taken into account.

left tail of Figure 27 has small, but significant features.

4.3 20/400 YDF Irradiation

Unlike the YDF 6/125, the YDF 20/400 barely had any demonstrated “RIE”. This was true regardless of the amount of shielding used. Borman did not show any RIE in her specimens, however the irradiation times for this experiment and [6] are different. As to the attenuation of the intensity as time/dose rate increased, with the exception of the 6.35 cm shield run (358.32 krad(Si)/hr), the graphs were as expected, with the heavily shielded fibers experiencing slow attenuation. The 6.35 cm shielded sample had a significantly larger integration time, which may result in this dataset showing more attenuation than a less shielded sample. Because these fibers were

larger, they tended to break upon removal from SPUD-D, and recovery data could not be obtained sequentially. However, just as in the YDF 6/125, an intermittent period of approximately 15 minutes was used to record the recovery of the fibers after a certain amount of radiation (accounting for shielding already), as evident in Figure 29.

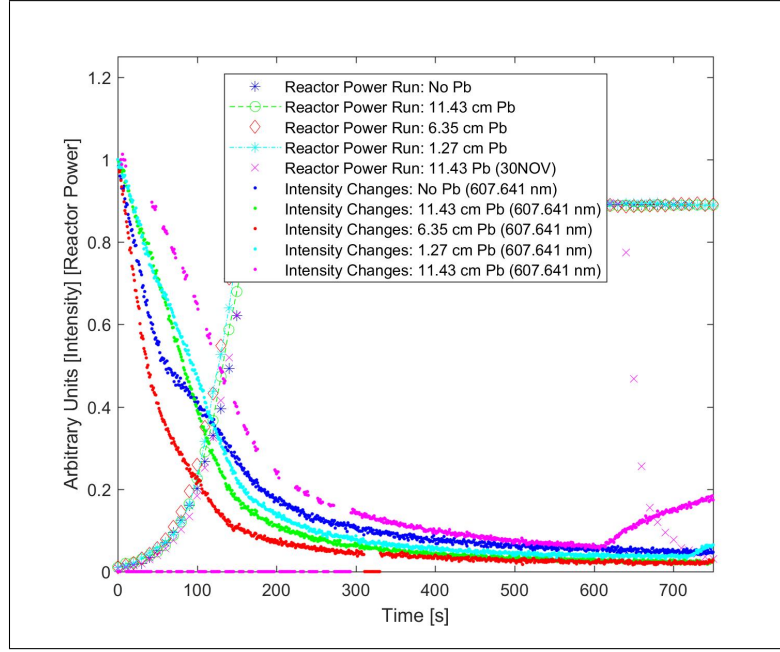


Figure 29. This wavelength was chosen as one of several that represent a defect center peak. Although the data was absolutely characterized and the integration time of each data set was taken into account, it is clear that the photon attenuation does not follow an exponential increase with increased shielding, exactly.

Data used to find RIA resulted from taking the initial intensity reading when the reactor first surpassed 1% power and when the total dose approached approximately 25.8 krad(Si). This was to ensure uniformity in total dose, as using the same time resulted in drastically different total doses as well as dose rates. Figure 29 shows the intensity values normalized as the initial intensity changed as SPUD-D was lowered into the 7-inch dry tube. It is difficult to identify a pattern using intensity readings, as the highest shielded fiber run did not result in the largest intensity readings. One interesting feature results around 90 seconds on the unshielded sample, where the

rate of decrease in intensity slowed, and then resumed the original slope at about 180 seconds.

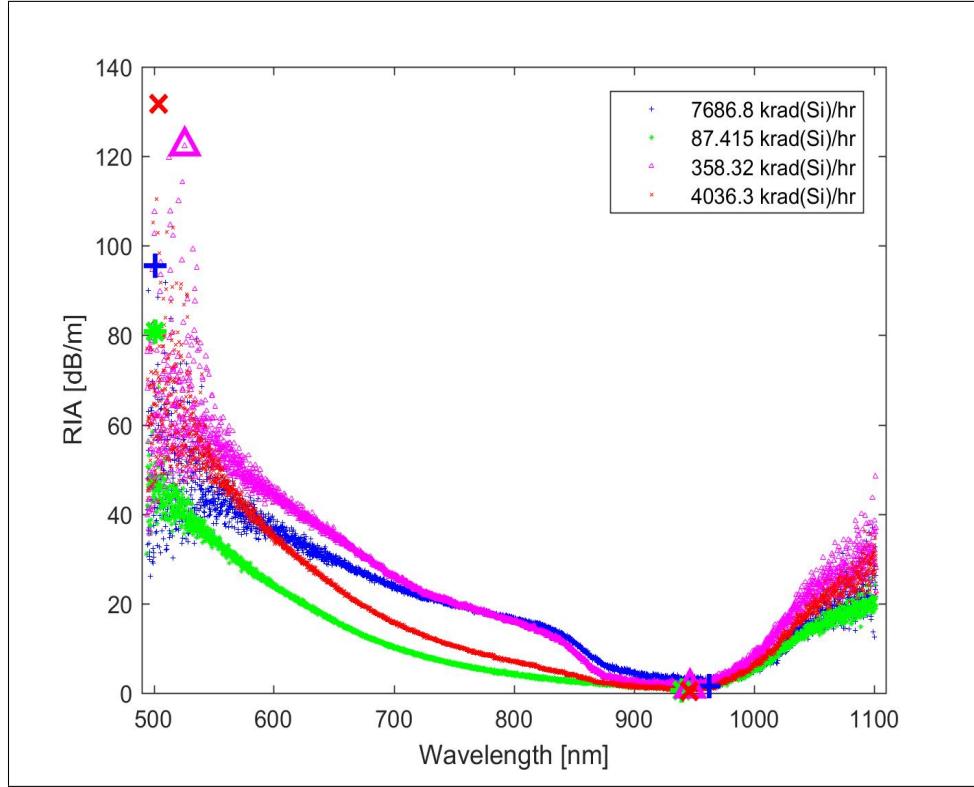


Figure 30. The RIA calculated for the CCS 200 wavelength 900 seconds after the OSURR surpassed 1%. A more defined stable RIA area is seen for the 20/400 YDF fiber. Note the clustering of minimum and maximum RIA centers. In particular, a γ radiation specific defect may be seen from 700-875 nm for doses above 1000 krad(Si)/hr. This corroborates the hypothesis that γ radiation begins to create defects above 600 nm, as stated in [6].

RIA calculations were less affected by large RIA variance (small intensity values) in the YDF 20/400. Defect features, especially those indicating a possible correlation to the initial findings of [6] concerning wavelength ranges for neutron radiation and γ radiation, were present in these fibers. A significant feature, seen in Figure 30 at about 800 nm, shows a marked difference in RIA for the Mrad(Si)/hr doses versus the 100 krad(Si)/hr doses. Additionally, clustering of minima and maxima occurs at smaller ranges of wavelength for this fiber. Unfortunately, defects near 500 nm are again masked by the small intensity values associated with the lower visible (VIS)

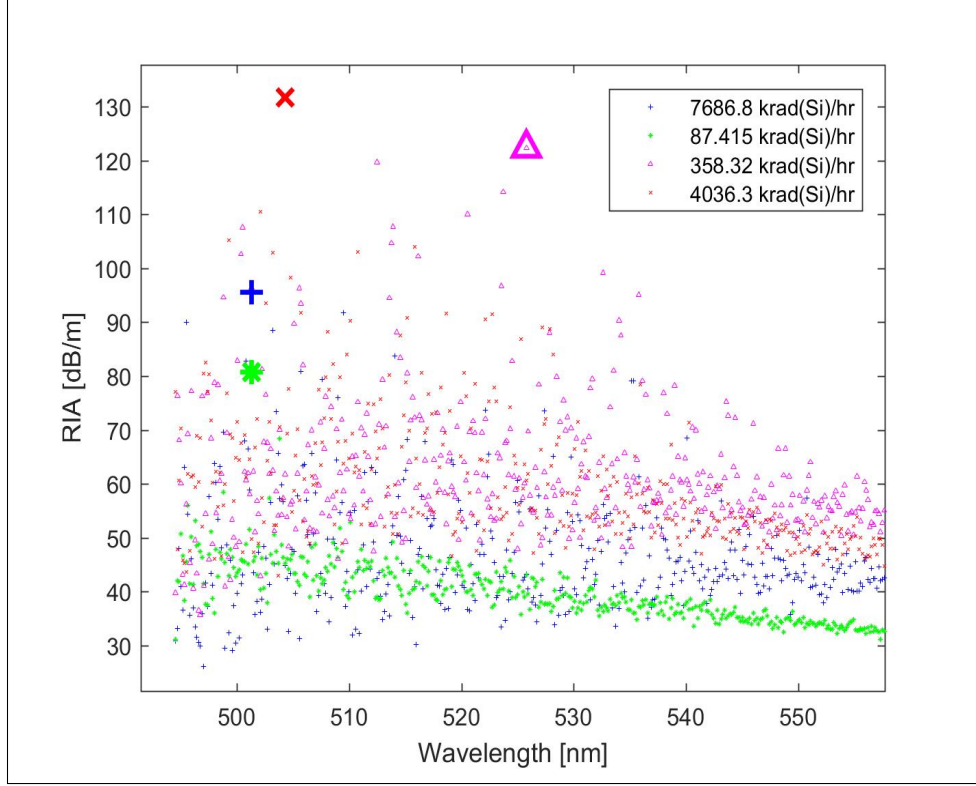


Figure 31. The RIA calculated for the CCS 200 wavelength 900 seconds after the OSURR surpassed 1%. It appears that in this fiber, the clustering of maxima occurs at a slightly higher wavelength bracket as compared to the 6/125 YDF. POHC defect centers are typical from 300-550 nm [18]. However, note that in this fiber, a fully shielded fiber has the lowest RIA.

spectrum.

For the maxima seen in Figure 31, the largest values are still associated with wavelengths near 500 nm, however, unlike the YDF 6/125 fiber, this fiber shows a lower RIA at 11.43 cm, albeit only about 20 dB/m. This does not support the idea that secondary ionizations due to neutrons cause an increase in attenuation. Both of these fibers were irradiated at the same time, in the same container, and almost at the same distance to the centerline of the fuel case of the reactor. Of the four maxima in Figure 25, the three lower ones cluster between 25 and 45 dB/m for the 6/125 YDF. The lowest two values of the maxima plotted in Figure 31 for the 20/400 YDF are at 80 and 100 dB/m, respectively. Both have highest maxima around 130

dB/m.

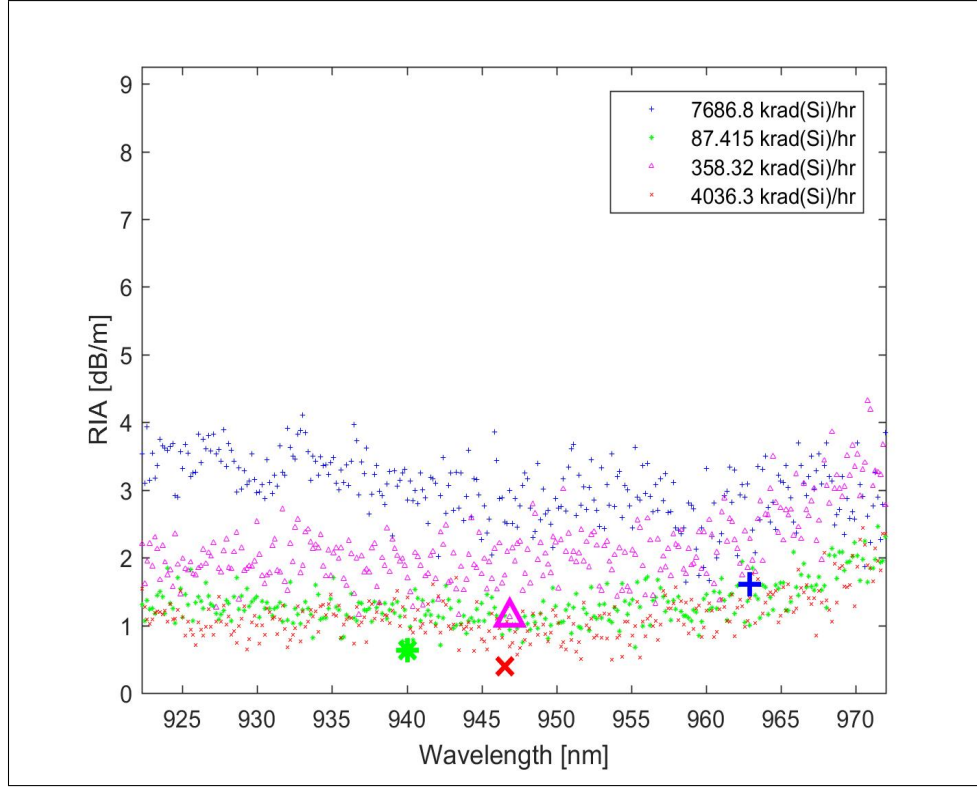


Figure 32. The RIA calculated for the CCS 200 wavelength 900 seconds after the OSURR surpassed 1%. As in the 6/125 YDF, we see a region of low RIA where Yb has absorption/emission features, indicating the use of Yb doped fibers as good pumping REDF.

The minima shown in Figure 32 also cluster at similar wavelengths as the 6/125 YDF, indicating again the Ytterbium is an RE dopant whose sensitivity is attributable to the codopants in the host matrix. The RIA values in the 20/400 YDF are larger than in the 6/125 YDF, which maintains an RIA close to 0 for these wavelengths. The highest RIA in this case was seen in the unshielded fiber.

Gaussian deconvolution in the 20/400 YDF was more successful than in the 6/125 YDF as it results in several Gaussian bands at or near defect centers identified in prevailing literature. However, for both Gaussian deconvolutions, the associated width with each energy peak for the defect centers was typically larger for the calculated Gaussian deconvolution than those values given in [18], for example. This may result

in smaller features being hidden by broad Gaussians. The specific features in Figures 33 and 34 show defects presented in [6] as well as unknowns. Shifts in defect peaks were not calculated for these Gaussians, however it appears that the peak at about 1.5 eV may be exclusively attributed to γ radiation dose.

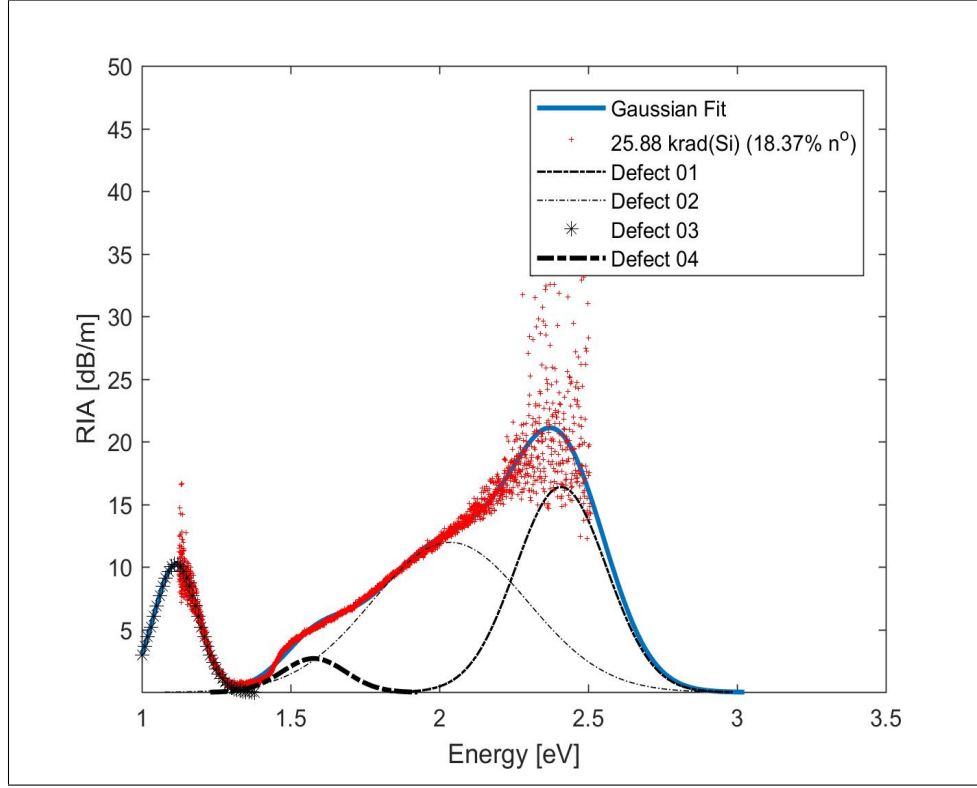


Figure 33. The 20/400 YDF provides a much more detailed deconvolution. Only an unknown peak close to 1.6 eV remains, however we can attribute this defect to γ radiation from the feature we saw around 800 nm in Figure 30. The two relative absolute peaks around 1.1 eV and 2.4 eV may be attributed to POHC and ALOHC, depending on the presence and concentration of these elements within the fiber. This Gaussian has an R^2 equal to 0.99.

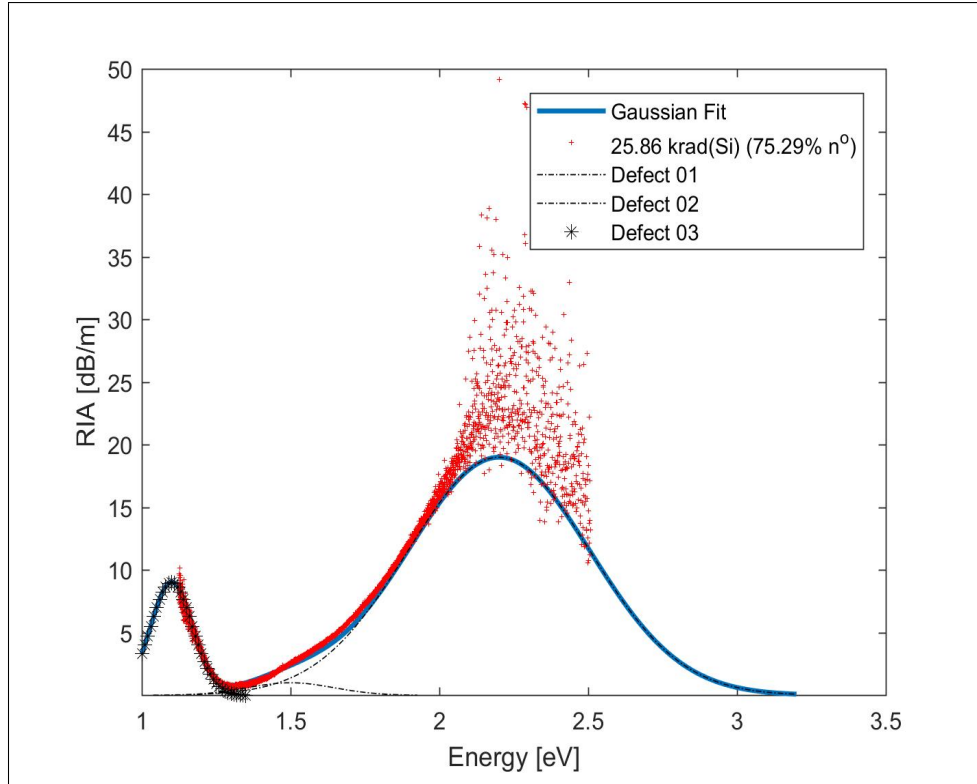


Figure 34. The 1.6 eV is no longer evident in this graph, where the fiber was heavily shielded and the majority of the dose received was from neutron radiation. There is also another peak close to 2.5, within the hypothesized neutron radiation dominant spectrum. The Gaussian curves close to 2.5 eV are also narrower as compared to the curve in Figure 33, a similar feature seen in the 6/125 YDF. This Gaussian has an R^2 equal to 0.99.

V. Conclusions and Recommendations

5.1 Neutron Radiation Effects

A significant amount of oscillation at low intensity readings by the 500 nm point and below, mentioned in [6] resulted in not being able to identify the true RIA at this wavelength. Gaussian analysis supports the hypothesis that neutron radiation acts below 550 nm while γ radiation acts above this point, as specifically demonstrated in Figures 33 and 34. A rate of formation may be interpreted from these two plots using the same attenuation curve for the γ radiation dose rate calculations. It is not unexpected that using high-Z shielding will result in attenuated γ radiation, and thus a higher neutron to γ radiation ratio. The unexpected inability to appropriately characterize the amount of dopant and co-dopant concentration leaves the identification of the type of defect centers observed to future work.

5.2 Photon Radiation Effects

The fact that several wavelengths of light demonstrated supposed “RIE”, the number of which changed as the shielding changed, is not accounted for in the literature for continuous radiation. Whether artifact or due to a fluorescence effect, again, the concentration of Yb in the fiber could enlighten why so many wavelengths, and only in the YDF 6/125 fibers, demonstrated recovery past the initial pre-radiation reading. A particular defect center around 1.5 eV is evident in plots with Mrad(Si)/hr dose rate while absent in dose rates at the order of krad(Si)/hr (i.e. shielded reactor runs).

5.3 Expected Contributions

Specific contributions to the existing body of knowledge in fiber research include:

1. *Develop a technique to maximize the signal-to-noise ratio during radiation experiments.* Leaving independent pieces such as BFT1s in a rig is extremely difficult to control. As mentioned in Chapter 2, there are many ways to reduce the SNR, in addition to the inherent high integration times of some samples. Developing SPUD-D ensured the fibers survived intact, for the most part, but adding spliced ends will optimize fiber research utilizing the OSURR. Additionally, the ensure a deconvolution is accurate, equipment response curves outside those for the fiber in question should be subtracted out. In this case, the grating efficiency produced intensity patterns whose variation from those in the given fiber are unidentified.
2. *Provide an absolute characterization of YDF after radiation defect generation.* The use of patch cable for this experiment should be limited in the future. The amount of loss due to mismatched ends between the CCS, which has a specific NA it was calibrated to, and the fibers was two orders of magnitude. Passive matched fiber cables are cheaper than patch cable and can be jacketed independently. Attaining the spectrum of the light source and removing it from the fiber did show different features in the fiber, however these were then hidden by the patch cable.
3. *Identify neutron radiation specific defects and their rates of formation.* It appears that the significant feature at 1.6 eV mentioned previously shows up as the photon fluence was modified (and the neutron fluence was not). This feature indicates a neutron independent defect center at wavelengths above 600 nm. Minute shifts leftward in the defect peaks may be attributable to neutron radiation dominant dose rates as this tendency is seen in both of the fibers when comparing the two types of radiation. It also appears that the γ radiation dominant source produces more broad defect widths than the neutron radiation

source, with the latter having the narrower dominant peak but also a significant right tail. The rates of formation appear inconclusive at this time as significant differences in RIA for similar times in the fibers were observed. However, we can say that the neutron fluence produced by the neutron generator, 2×10^9 n/s, was insufficient to produce defects in the fiber, making this a lower limit. We also determine that the Yb interaction with both sources of radiation does not significantly increase RIA close to the absorption/emission points for YDF. Slight shifts in the minima and maxima are discernible though. As such, the preponderance of defect creation in the host matrix is due to the addition of codopants.

5.4 Recommendations for Future Work

Prior to beginning research on fiber, it is important to know the concentrations of dopants and the techniques used to insert the dopants into the host glass. SEM-EDX has been cited in recent literature as a way to characterize both the presence of dopants in fiber as well as the concentrations of each. To adequately determine the number of defects in a sample without interpolation using Gaussian deconvolution, a mass must be determined. Fibers of this size are not able to be stripped to their core without a chemical treatment or other, more advanced, form of stripping, therefore instruments such as SEM-EDX or EPS may be used. Perhaps fibers manufactured to certain doping level for the host matrix and the RE dopant may be used to accurately identify the defect centers.

A different technique is needed, perhaps a pulsed source, to determine the reason behind the apparent RIE in the fibers. Since no discernible pattern in regards to affected wavelengths could be determine, a pulsed source and power meter may be used to determine the behavior at specific wavelengths immediately after irradiation.

A more formal analysis of the dose rate due to neutron and γ radiation is necessary to precisely determine defect rates and defect centers. This can be accomplished by completed the NAA flux wire analysis with the OSURR (for neutrons). As for the γ radiation, an MCNP program may be needed to model this reactor to ensure non-forward biased γ radiation is considered.

Bibliography

1. *A Guide to the Use of Lead for Radiation Shielding*. Technical report, 1984.
2. *Hitachi S-4700 SEM Training & Reference Guide*. Technical report, 2007.
3. *nanoDot Dosimeter*. Technical report, 2017.
4. Adelphi. “Adelphi Technology Inc.”, 2003. URL http://www.adelphitech.com/pdfs/DD108-9-generator_Flier_24-Nov-14_A.PDF.
5. Attix, F. H. *Introduction to Radiological Physics and Radiation Dosimetry*. Wiley-VCH, Weinheim, GE, 1986.
6. Borman, O. *Gamma versus neutron radiation effects on Ytterbium-doped optical fibers*. Master’s thesis, AFIT-ENP-MS-16-M-057, Department of Engineering Physics, Air Force Institute of Technology, 2015.
7. Boyd, R. W. *Radiometry and the Detection of Optical Radiation*. John Wiley & Sons, New York, 1983.
8. Bridgeman, C. J. *Introduction to the Physics of Nuclear Weapons Effects*. US DOD, Ft. Belvoir, 2001.
9. Bryant, J. F. and J. A. Wall. “Radiation Effects on Fiber Optics”. (627), 1975.
10. Cheymol, G. et al. “Dimensional measurements under high radiation with optical fibre sensors based on white light interferometry - Report on irradiation tests”. *IEEE Transactions on Nuclear Science*, 61(4):2075–2081, 2014.
11. Considine, G. D. and P. H. Kulik. “Van Nostrand’s Scientific Encyclopedia: Optical Fiber Systems”, 2008.
12. Devine, R. A. B. and J. M. Francou. “Extrinsic- and intrinsic-defect creation in amorphous SiO₂”. *Physical Review B*, 77(2):189–212, 1990.
13. Dignonnet, M. J. F. *Rare-Earth-Doped Fiber Lasers and Amplifiers*. 2001.
14. Edwards, A.H. and W. Beall Fowler. “Theory of the peroxy-radical defect in a-SiO₂”. *Physical Review B*, 26(12):6649–6660, 1982.
15. Fanciulli, M. et al. “Phosphorous oxygen hole centers in phosphosilicate glass films”. (Mi):1–10, 2006.
16. Fox, M. *Optical properties of solids*. Oxford University Press, Oxford, 2010.
17. Friebele, E. J. et al. “Fundamental defect centers in glass: The peroxy radical in irradiated, high-purity, fused silica”. *Physical Review Letters*, 42(20):1346–1349, 1979.

18. Girard, S. et al. "Radiation Effects on Ytterbium- and Ytterbium/Erbium-Doped Double-Clad Optical Fibers". *IEEE Transactions on Nuclear Science*, 56(6):3293–3299, 2009.
19. Girard, S. et al. "Radiation effects on fiber amplifiers: design of radiation tolerant Yb/ Er based devices". *Proceedings of SPIE*, 7914(February 2011):79142P–79142P–6, 2011.
20. Girard, S. et al. "Design of radiation-hardened rare-earth doped amplifiers through a coupled experiment/simulation approach". *Journal of Lightwave Technology*, 31(8):1247–1254, 2013.
21. Girard, S. et al. "Radiation effects on silica-based optical fibers: Recent advances and future challenges". *IEEE Transactions on Nuclear Science*, 60(3):2015–2036, 2013.
22. Glasstone, S. and P.J. Dolan. *The Effects of Nuclear Weapons*. US DOD, D.C., 3rd edition, 1977.
23. Griscom, D. L. "E' center in glassy SiO₂, O-17, H-1, and very weak Si-29 superhyperfine structure". *Phys. Rev. B*, 22(9):4192, 1980.
24. Griscom, D. L. "Nature Of Defects And Defect Generation In Optical Glasses". *Proceedings of SPIE*, (December 1985):38, 1985.
25. Griscom, D. L. "Self-trapped holes in amorphous silicon dioxide". *Physical Review B*, 40(6):4224–4227, 1989.
26. Griscom, D. L. "Growth and decay kinetics of defect centers in high-purity fused silicas irradiated at 77 K with X-Rays or 6.4-eV laser light". *Nuclear Inst. and Methods in Physics Research, B*, 46(1-4):12–17, 1990.
27. Griscom, D. L. "γ-Ray-induced visible/infrared optical absorption bands in pure and F-doped silica-core fibers: Are they due to self-trapped holes?" *Journal of Non-Crystalline Solids*, 349(1-3):139–147, 2004.
28. Griscom, D. L. "Self-trapped holes in pure-silica glass: A history of their discovery and characterization and an example of their critical significance to industry". *Journal of Non-Crystalline Solids*, 352(23-25):2601–2617, 2006.
29. Griscom, D. L. "On the natures of radiation-induced point defects in GeO₂-SiO₂ glasses: reevaluation of a 26-year-old ESR and optical data set". *Optical Materials Express*, 1(3):400, 2011.
30. Griscom, D. L. "Trapped-electron centers in pure and doped glassy silica: A review and synthesis". *Journal of Non-Crystalline Solids*, 357(8-9):1945–1962, 2011.

31. Griscom, D. L. and E. J. Friebele. “Fundamental defect centers in glass: Si29 hyperfine structure of the nonbridging oxygen hole center and the peroxy radical in a-SiO2”. *Physical Review B*, 24(8):4896–4898, 1981.
32. Griscom, D. L. and E. J. Friebele. “Fundamental radiation-induced defect centers in synthetic fused silicas: Atomic chlorine, delocalized e centers, and a triplet state”. *Physical Review B*, 34(11):7524–7533, 1986.
33. Griscom, D. L. and M. Mizuguchi. “Determination of the visible range optical absorption spectrum of peroxy radicals in gamma-irradiated fused silica”. *Journal of Non-Crystalline Solids*, 239(1-3):66–77, 1998.
34. Griscom, D. L. et al. “Radiation-induced defects in glasses: Origin of power-law dependence of concentration on dose”. *Physical Review Letters*, 71(7):1019–1022, 1993.
35. Griscom, D. L. et al. “Evaluation of fast-neutron radiation effects in optical fibers for fusion reactor diagnostics”. *RADECS 93. Second European Conference on Radiation and its Effects on Components and Systems (Cat. No.93TH0616-3)*, 280–286. 1994.
36. Griscom, D. L. et al. “Model for the Dose, Dose-Rate and Temperature Dependence of Radiation-Induced Loss in Optical Fibers”. *IEEE Transactions on Nuclear Science*, 41(3):523–527, 1994.
37. Hecht, J. *Understanding Fiber Optics*. Laser Light Press, Auburndale, 5th edition, 2006.
38. Henschel, H. et al. “Radiation-induced loss of rare earth doped silica fibres”. *RADECS 97. Fourth European Conference on Radiation and its Effects on Components and Systems (Cat. No.97TH8294)*, 45(3):1552–1557, 1997.
39. Henschel, H. et al. “Comparison between fast neutron and gamma irradiation of optical fibres”. *IEEE Transactions on Nuclear Science*, 45(3):430–438, 1998.
40. Hess, W. N. et al. “Cosmic-Ray Neutron Energy Spectrum*”. 116(2), 1959.
41. Hubbell, J. H. “Photon Mass Attenuation and Energy Absorption Coefficients from 1 keV to 20 MeV”. *Int. J. Appl. Radiative Isotopes*, 33(I):1269–1290, 1982.
42. Hubbell, J. H. “Electron-positron pair production by photons: A historical overview”. *Radiation Physics and Chemistry*, 75(6):614–623, 2006.
43. Hubbell, J. H. and S. M. Seltzer. “Cross section data for electron-positron pair production by photons: A status report”. *Nuclear Instruments and Methods in Physics Research, Section B: Beam Interactions with Materials and Atoms*, 213:1–9, 2004.

44. Imai, H. et al. "Generation of E' centers and oxygen hole centers in synthetic silica glasses by γ irradiation". *Physical Review B*, 48(5):3116–3123, 1993.
45. Just, F. et al. "Ytterbium-doping related stresses in preforms for high-power fiber lasers". *Journal of Lightwave Technology*, 27(12):2111–2116, 2009.
46. Kannan, S. et al. "Analysis of recovery in radiation-induced loss in rare-earth-doped fibers through master curve/demarcation energy diagrams". *OFC/IOOC 1999 - Optical Fiber Communication Conference and the International Conference on Integrated Optics and Optical Fiber Communication*, volume 3, 93–95. Lucent Technologies, Somerset, 1999.
47. Khan, F. M. and J. P. Gibbons. *The Physics of Radiation Therapy*. Wolters Kluwer, Philadelphia, 5th edition, 1953.
48. Kirchhof, J. and S. Unger. "Codoping effects in fibers for active applications". *OFC/IOOC 1999 - Optical Fiber Communication Conference and the International Conference on Integrated Optics and Optical Fiber Communication*, 2:196–198, 1999.
49. Koponen, J. J. "Progress in direct nanoparticle deposition for the development of the next generation fiber lasers". *Optical Engineering*, 50(11):111605, 2011.
50. Krane, K. S. *Nuclear physics*. John Wiley & Sons, Hoboken, 1998.
51. Lagomacini, J. C. et al. "Growth kinetics of AlOHC defects in γ -irradiated silica glasses". *Journal of Non-Crystalline Solids*, 403:5–8, 2014.
52. Laperle, P. et al. "Relations between phosphorus/aluminum concentration ratio and photodarkening rate and loss in Yb-doped silica fibers". *Proceedings of SPIE*, 7580(May):75801Y, 2010.
53. Lezius, M. et al. "Radiation Induced Absorption in Rare Earth Doped Optical Fibers". *IEEE Transactions on Nuclear Science*, 59(2):425–433, 2012.
54. Liekki. "Yb1200-6/125 Single Mode Ytterbium Doped Fiber", 2017. URL <http://www.nlight.net/nlight-files/file/DatasheetsV2/OpticalFiber/nLIGHTLIEKKILargeModeAreaPassiveFibersv2.pdf>.
55. Luo, W. et al. "Defect center characteristics of silica optical fiber material by gamma ray radiation". *Proceedings of SPIE*, volume 8307, 83072H. 2011.
56. Mashkov, V. A. et al. "Fundamental Role of Creation and Activation in Radiation-Induced Defect Production in High-Purity Amorphous SiO₂". *Physical Review Letters*, 76(16):2926–2929, 1996.
57. Morana, A. et al. "Influence of neutron and gamma-ray irradiations on rad-hard optical fiber". *Optical Materials Express*, 5(4):898, 2015.

58. Nelson, G. and D. Reilly. "Gamma-ray interactions with matter". *Passive Non-destructive Analysis of Nuclear Materials*, (I):27–42, 1991.
59. Norouzi, M. et al. *A survey on rare earth doped optical fiber amplifiers*. Technical report, 2013.
60. Ollier, N. and V. Pukhkaya. "Point defect stability in gamma irradiated aluminoborosilicate glasses: Influence of Yb³⁺+doping ions". *Nuclear Instruments and Methods in Physics Research, Section B: Beam Interactions with Materials and Atoms*, 277:121–125, 2012.
61. Pacchioni, G. et al. *Defects in SiO₂ and related dielectrics: Science and technology*. Kluwer Academic Publishers, Dordrecht, 2000.
62. Paschotta, R. et al. "Ytterbium-doped fiber amplifiers". *IEEE Journal of Quantum Electronics*, 33(7):1049–1056, 1997.
63. Patel, P. "Lockheed Martin Shows Off High-Power Fiber Laser Weapon". 1, 2014. URL <https://spectrum.ieee.org/tech-talk/aerospace/military/lockheed-martin-shows-off-highpower-fiber-laser-weapon>.
64. Pedrotti, F. *Introduction to Optics*. Pearson Prentice Hall, Upper Saddle River, NJ, 3rd edition, 2007.
65. Reghioua, I. et al. "Cathodoluminescence Characterization of Point Defects in Optical Fibers". *IEEE Transactions on Nuclear Science*, 64(8):2318–2324, 2017.
66. Richardson, D. J. et al. "High power fiber lasers: current status and future perspectives [Invited]". *Journal of the Optical Society of America B*, 27(11):B63, 2010.
67. Schmelzer, J. W. P. *Glass - Selected Properties and Crystallisation*. Rostock, 2014.
68. Sen, R. "Rare Earth Doped Fibers for Fiber Laser: Opportunities and Challenges". *International Conference on Fibre Optics and Photonics*, 978–980. Kolkata-23, 2012.
69. Shu, H. and M. Bass. "Calculation the Guided Modes in Optical Fibers and Waveguides". *Journal of Lightwave Technology*, 25(9):2693–2699, 2007.
70. Siegel, J. A. et al. "Calculating the absorbed dose from radioactive patients: the line-source versus point-source model." *Journal of nuclear medicine: official publication, Society of Nuclear Medicine*, 43(9):1241–1244, 2002.
71. Sigel, G. H. and M. J. Marrone. "Photoluminescence in as-drawn and irradiated silica optical fibers: an assessment of the role of non-bridging oxygen defect centers". *Journal of Non-Crystalline Solids*, 45(2):235–247, 1981.

72. Simmons, K. D. et al. "Correlation of defect centers with a wavelength-dependent photosensitive response in germania-doped silica optical fibers." *Optics letters*, 16(3):141–3, 1991.
73. Singleton, B. J. *Radiation Effects on Ytterbium-Doped Optical Fibers*. Ph.D. thesis, AFIT-ENP-DS-14-J-15, Department of Engineering Physics, Air Force Institute of Technology, 2014.
74. Singleton, J. *Band Theory and Electronic Properties of Solids*. Oxford University Press, New York, 2014.
75. Söderlund, M. J. et al. "Specialty fibers: Direct nanoparticle deposition builds active fibers". *Laser Focus World*, 2008(1):1–10, 2006.
76. Sreckovic, M. et al. "Properties of Fiber Optical Materials". *Microelectronics (Proceedings)*, 1:12–1, 1995.
77. Staff, Ohio State University Research Reactor. "Neutron Spectrum Measurements", 2014.
78. Staff, Ohio State University Research Reactor. "Neutron Fluxes and Neutron and Gamma Dose Rates", 2015.
79. Staff, Ohio State University Research Reactor. "OSURR 7" Dry Tube Vertical Flux Distribution". 2017.
80. Suzuki, S. "Highly ytterbium-doped silica fibers with low photo-darkening." *Optics express*, 17(12):9924–9932, 2009.
81. Tammela, S. et al. "Direct Nanoparticle Deposition process for manufacturing very short high gain Er-doped silica glass fibers". *2002 28TH European Conference on Optical Communication*, (4):1–2, 2002.
82. Tammela, S. et al. "The potential of direct nanoparticle deposition for the next generation of optical fibers". 6116:61160G, 2006.
83. Taylor, N. *Analysis of a High Temperature Fission Chamber Experiment for Next Generation Reactors*. Thesis, Ohio State University, 2017.
84. Thorlabs. "SLS201L(M)/SLS202L(M) Stabilized Tungsten Light Sources User Guide". URL https://www.thorlabs.com/newgrouppage9.cfm?objectgroup_{_}id=7269.
85. Thorlabs. "LDC2xx Series Operation Manual", 2011.
86. Thorlabs. "Spectrometer, CSS Series, Operation Manual", 2014. URL <http://www.thorlabs.de/thorcat/21600/PDB450C-Manual.pdf>.

87. Thorlabs. “Ytterbium Doped Fiber Specification Sheet”, 2018. URL https://www.thorlabs.com/newgrouppage9.cfm?objectgroup_{_}id=336.
88. Titterton, David H. *Military Laser and Technology Systems*. Artech House, Norwood, 2015.
89. TrippLite. “User’s Manual for TrippLite S2200XLa UPS”, 2017. URL www.triplite.com.
90. Trukhin, A. N. et al. “Luminescence of phosphorus doped silica glass”. *Journal of Non-Crystalline Solids*, 462:10–16, 2017.
91. Trump, Donald J. “State of the union address”, 2018. URL <https://www.whitehouse.gov/briefings-statements/president-donald-j-trumps-state-union-address/>.
92. Uchino, T. et al. “Structure and generation mechanism of the peroxy-radical defect in amorphous silica”. *Physical Review Letters*, 86(20):4560–4563, 2001.
93. Unger, L. M. and D. K. Trubey. “Specific Gamma-Ray Dose Constants for Nuclides Important to Dosimetry and Radiological Assessment”. *Defense*, 1982.
94. Vastag, M. R. “Mode Field Diameter and Effective Area”, 2001. URL https://www.corning.com/media/worldwide/global/documents/sfiberWP7071_{_}ModeFieldDiamandEffArea_{_}10-01-3.pdf.
95. Verdeyen, J. T. *Laser electronics*. Prentice Hall, Upper Saddle River, NJ, 3rd edition, 1995.
96. Wang, T. et al. “Influences of Thermal Annealing Temperatures on Irradiation Induced E’ Centers in Silica Glass”. *IEEE Transactions on Nuclear Science*, 55(5):2685–2688, 2008.
97. Zervas, M. N. “High power ytterbium-doped fiber lasers fundamentals and applications”. *International Journal of Modern Physics B*, 28(12):1442009, 2014.

Vita

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14. ABSTRACT Studies of amorphous silica fibers bombarded with neutral particles indicate that both photons and neutrons have the same mechanism for creating defects, however rate of creation and affected wavelengths for each type is unclear [38] [39]. Some studies indicate that photon radiation induced attenuation (RIA) is higher than neutron RIA, while others indicate the reverse due to secondary effects by neutrons. Additionally, it appears that the two neutral particle radiation sources produce defects at significantly different rates despite using the same dose rate [39]. In comparing neutron dominant and γ dominant radiation sources for a similar total dose, the peak between 2-3 eV in the 6/125 YDF shifts approximately 0.4 eV, with a smaller defect peak deconvoluted around 3.2 eV in the neutron dominant radiation source results. The 20/400 YDF deconvolves to more Gaussian peaks, with a 0.1 eV shift for the 1-1.5 eV peak and a 0.15 eV shift for the 2-2.5 eV peak. Noticeably, the γ radiation dominant source data shows a peak around 1.6 eV significantly more pronounced than in the neutron radiation dominant source dataset.						
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