Gamma irradiation response of photonic crystal and standard optical fibre Bragg grating sensors for radiation dosimetry

Desmond Baccini

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Gamma irradiation response of photonic crystal and standard optical fibre Bragg grating sensors for radiation dosimetry

Desmond Baccini

This thesis is presented in fulfilment of the requirements for the degree of

Doctor of Philosophy

School of Science
Edith Cowan University

2019
USE OF THESIS

The Use of Thesis statement is not included in this version of the thesis.
ABSTRACT

This study investigates the use of Optical Fibre Bragg Grating Sensors (FBGs) for Gamma Radiation Dosimetry. A comparative analysis of responses to gamma irradiation between standard commercial FBGs and new generation FBGs written in Photonic Crystal fibre (PCF) were examined under similar regimes and conditions. Current research suggests that the FBGs performance, when exposed to Cobalt-60 gamma irradiation, can suffer cross sensitivity problems resulting from different external effects such as temperature. However, FBGs written in PCFs may be able to overcome these problems due to their design, flexibility of the shape, and size of the micro-holes in a PCF. Previous research has indicated the Bragg wavelength shift (BWS) of standard FBGs increases with accumulated Gamma dose. This shift appears to be permanent, indicating that gamma irradiation permanently affects the Bragg wavelength of the FBG.

To better understand these effects, and determine the suitability of particular FBGs for use in radiation dosimetry, measurements in relation to the effects of pre-irradiation, dose rates and accumulated dose, and relaxation effects were performed on both sets of FBGs. To simulate real time conditions of a radiation dosimeter, the FBGs are examined through three consecutive radiation stages followed by limited recovery times. There is a lack of research in the areas of small recovery times and multiple periods of irradiation. The experimental regime and setup consisting of the three stages with very limited recovery comparing PCF-FBGs and standard FBGs (STD-FBGs) is unique. The experiments and gamma irradiation were conducted at ANSTO (Australian Nuclear Science and Technology Organisation) using the GATRI (Gamma Technology Research Irradiator) irradiation facility. The responses after exposure to gamma irradiation, including relaxation periods between commercially manufactured FBGs written in Germanium (Ge) doped optical fibres, with and without hydrogen loading, along with the standard SMF-28 fibre with Hydrogen are shown. The FBG inscription in PCF was completed at Interdisciplinary Photonics Laboratories (iPL), University of Sydney. The FBGs in each fibre are written by Ultraviolet (UV) low energy irradiation. In nuclear environments, when FBGs have been exposed to gamma irradiation, changes in the Bragg wavelength occur, although the exact cause or trigger is still unclear. The main outcome of this research has indicated that PCF-FBGs, compared to standard FBGs, are a strong candidate for use in the field of radiation dosimetry. This is due to their very consistent
behaviour and recovery aspects after irradiation exposure. This work will compliment established research and help in the absolute quantitative comparison between the individual standard FBGs and PCF-FBGs. It will help in establishing FBGs as a possible replacement to present physical and chemical sensors currently being used as radiation dosimeters.
DECLARATION

I certify that this thesis does not, to the best of my knowledge and belief:

(i) incorporate without acknowledgment any material previously submitted for a degree or diploma in any institution of higher education.

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CHAPTER 1
INTRODUCTION

This chapter begins with the relevant background and overall scientific objectives of the project in relation to standard STD-FBGs and new generation PCF-optical fibre Bragg sensors. The significance and motivations of the project are also discussed, and the chapter concludes with the research questions.

1.1 Research background

The main focus and scientific objective of this study is to investigate the behaviour of, and help elucidate, the permanent damage mechanism caused by gamma irradiation between current standard commercial SMF-FBGs and PCF-FBGs. For continuity, similar gamma irradiation accumulated dosages, inclusive of short similar relaxation periods is used on standard optical fibre Bragg gratings (STD-FBGs) and PCF-FBGs. This will allow for a direct comparison and help to determine the suitability of which FBG is better suited to act as a dosimeter, both in high dose radiation nuclear areas and low dose environments such as space. There is a gap in the present understanding of the specific causes of gamma irradiation exposure effects to FBGs particularly PCF-FBGs. Results obtained to date indicate that gamma irradiation causes attenuation degradation in various optical fibre types through the generation of defects such as colour centres, causing refractive index changes in the fibre [1]. It is also known that specific types of fibre (e.g. those made with different dopant types such as Germanium (Ge) and Phosphorous (P)) have different responses to ionizing radiation [2]. In this study, Germanium is used, as it is by far the most common dopant. Therefore, a more conclusive comparison will be made when comparing behaviours between the STD-FBGs and PCF-FBGs. What is lacking, is the understanding of the explicit mechanism responsible for the Bragg wavelength shift (BWS) whilst under gamma irradiation. If FBGs are to be applied for the function of measuring radiation levels (dosimetry), it will be necessary to help elucidate the explicit mechanism of the gamma (and other ionizing) radiation damage/effects within optical fibres and FBGs. Of particular interest are the effects of pre-irradiation, temperature and time during the relaxation (no irradiation) or recovery
period, on STD-FBGs and PCF-FBGs. Direct research is lacking with regards to the effects of gamma irradiation on this PCF-FBG fibre technology.

1.2 Overview

Recently, the use of ionising radiation for various applications has increased. Radiation dosimetry is fundamentally important to these processes and is the focus of much research [1]. A dosimeter can generally be defined as any device that is capable of providing an output value that is a measure of the average absorbed dose deposited in its sensitive volume by ionizing radiation [3]. Radiation dosimetry deals with methods for a quantitative determination of the energy deposited in a given medium by direct or indirect ionizing radiation [3]. What is required is a system that is easy to use, and install, to monitor radiation remotely in real time, long lasting and cost effective [1]. In the case of optical fibre sensing in radiation dosimetry, the possible applications and environments in which they could be used are varied [4]. Some of the applications include:

(1) Radiation protection and monitoring of nuclear installations [5]

(2) Space dosimetry [6]

(3) Measurement of the absorbed dose in radiotherapy [7]

(4) Neutron or mixed gamma –ray neutron dosimetry [8]

(5) Evaluation of beam losses in particle accelerators [9]

The basic optical sensing technology includes extrinsic optical sensors. The extrinsic sensors only use the fibre to transmit light to and from a sensing element, whilst intrinsic sensors use the fibre itself as the sensing element. The optical fibres which can be affected directly by radiation are called intrinsic sensor [1]. Primarily radiation dosimeters can be characterised according to the type of interaction between the incident radiation and the sensor. This interaction varies between the different types of radiation dosimeters [1]. There are many factors/parameters to be considered when designing the optimum radiation dosimeter. The main factor which is relevant to this study involves material sensitivity to gamma radiation. The ability for radiation to interact with the material is paramount. The materials utilised in gamma dosimetry must have a high sensitivity to gamma radiation within the dose ranges required for the specific application [4].
Applications may include in-vivo medical measurements, in which the sensor has to be sensitive to small dose rates/total doses, and nuclear installation measurements which require dosimeters capable of measuring at very high doses [1,4]. One relatively new optical fibre sensor in gamma dosimetry involves the use of poly methylmethacrylate (PMMA). It is based on the widely used gamma dosimetry technique which used dyed PMMA slides, which change colour on exposure to gamma irradiation [1]. The resultant radiation-induced attenuation (RIA) change is monitored using a spectrophotometer post-irradiation. The main problem with this system is the lack of real-time information as the PMMA slides had to be removed from the area to be tested in the laboratory [1]. When combining the principle of RIA used by PMMA slides, with PMMA optical fibres, the problem of real time monitoring is overcome.

Standard commercial PMMA plastic fibres have shown significant attenuation following gamma irradiation of doses up to 50 kGy. Above 50 kGy PMMA plastic fibre saturation becomes evident, making them non ideal for high dose areas such as in nuclear facilities [1]. One of the oldest types of dosimeter used in detecting ionising radiation is by scintillation light produced in certain materials. Scintillation dosimeters are based on the phenomenon that the material used is capable of converting ionising radiation into detectable light [1]. A photodiode or photomultiplier tube subsequently converts it into an electrical signal [1]. Optical fibres have now been used in numerous scintillation detection systems to transmit the scintillating emissions to a remote photomultiplier tube [1]. Whilst this system has exhibited the usual dosimetric qualities such as small size, robustness, and good linearity, significant loss in the transmission of light due to the light coupling from the scintillator to the optical has been reported [1]. Again as with the PMMA system, scintillation optical fibres are only capable of measuring at low dose rates.

The third type of dosimeter is known as a thermoluminescence dosimeter. Thermoluminescence (TL) is the emission of light from a solid due to heating after being exposed by radiation [1]. TL optical fibre dosimeters have been shown to accurately measure low doses of gamma radiation. Most TL dosimeters demonstrate a linear response to doses up to a few Gray [1]. After reaching a few hundred gray saturation occurs. The linear region at low dose makes them ideal for medical and environmental applications [1]. Similar to the TL dosimeter there is a dosimeter based on optically
stimulated luminescence [1]. Again this type of dosimeter has the ability to measure very small doses and ideal for use in personal and medical applications. In summary, the literature indicates that optical fibre sensors are becoming important in the field of radiation dosimetry. Most of the optical fibre dosimeters are extrinsic sensors i.e. the fibres alone are used to transmit information. The techniques used are based on luminescence and exhibit a high sensitivity at low accumulated doses. This makes them ideal for use in the medical field. Also some dosimeters rely on the changes in the optical fibre and work by monitoring the change in RIA using spectrophotometry. There is however a problem within this group of optical fibre dosimeters, they are not capable of sensing at high dose rate or accumulated dose facilities.

FBGs manufactured in silica optical fibres however, are now showing the most promise as radiation detectors, especially in the area of high dose rates and accumulated dose [10]. In radiation environments, however, research is still being performed to attempt to confirm the accuracy and reliability of optical sensors when they are used as a form of dosimeter. Results to date indicate that gamma irradiation causes attenuation degradation in various types of optical fibre. The FBG however, seems to be less susceptible to attenuation, making it a strong candidate for a new form of radiation dosimeter. Studies have shown that when exposed to gamma irradiation, a Bragg wavelength shift (BWS) mainly towards the red occurs, by varying amounts depending on the fibre type, dopants used, and inscription methods and temperature. But still there is no definitive answer on the exact cause or trigger for this [11, 175].

The advantages of optical FBG sensors have made them attractive for various industrial sensing applications. Typically, their main attributes compared to electronic, chemical, mechanical and electrical sensors are that they are immune to electro-magnetic interference, are light weight and small, resistant to harsh environments have greater sensitivity, are mass producible and cost effective, and are able to measure, remotely in real time [12]. In the field of radiation dosimetry, FBG sensors have the distinct advantage because of their immunity to electro-magnetic interference as radiation levels by FBGs are relayed using optical signals rather than electrical [1]. The most common radiation dosimeters in use today throughout many industries include film/badge personal dosimeters, thermoluminescent dosimeters (TLDs), electronic and electrical dosimeters and quartz fibre electroscope (QFE) dosimeters. Their disadvantages include, a short shelf
life, high cost, fading due to temperature and light effects, and sensitivity to rough handling [1]. When electrical dosimeters for example are exposed to radiation over long periods, it is found that degradation occurs to the insulation which affects the performance and stability. For industrial applications these problems can primarily increase operating costs. A lot of work has been done investigating and developing radiation resistant FBGs for use in strain and temperature applications [1,13]. They have been considered for various sensing tasks in the nuclear industry such as, structural health monitoring of containment buildings, reactor core temperature, and mechanical stress measurements and long term monitoring of underground waste storage facilities [14]. If FBGs are to be used as dosimeters in environments where there is the presence of high energetic radiation such as the nuclear industry and space, it is essential to evaluate the response of FBGs [14].

1.3 Motivations

Currently there are many published papers on research in the area of radiation dosimetry using FBGs. The continuation of new research in this field could lead to a greater understanding of the mechanisms involved, for the Bragg wavelength shift whilst exposed to gamma irradiation. The results indicate that radiation dosimetry falls into two categories, either low or high dose detection. The primary motivation is to help achieve a circumscribed volume of results that will assist in the eventual use of FBGs in the field of radiation dosimetry. The research and experimental data will be concentrating on gamma radiation in high dose areas such as in nuclear reactors and facilities like the GATRI facility at ANSTO. In order to understand the effects of gamma irradiation, and determine the suitability of FBG's for use as dosimeters, it will be necessary to perform measurements with regards to irradiation effects, dose rates, temperature effects and pre-irradiation effects and recovery aspects. The major motivation is the lack of published research on the irradiation effects in new generation optical fibre Bragg grating sensors such as PCF-FBGs, compared to standard FBGs, and determining the suitability or non-suitability of both. In order to accurately measure the entire range of dose that may be encountered in radiation dosimetry e.g. \( \sim 10^{-4} \text{ Gy} \) for personal use and up to \( 10^7 \) for reactor dosimetry [15], it may be necessary to use two types of FBGs specifically suited to maintain detection depending on the environment. However, for commercial reasons, it would be advantageous to limit detection to one FBG which is possibly able to operate
in both low dose and high dose situations. It is intended that this research will help to clarify this through a strict experimental regime and setup, and determine if the PCF-FBG and/or standard FBG is suitable to act as a radiation dosimeter.

1.4 Objectives

1.4.1 Primary objectives

- To quantify the effect of gamma irradiation and response on new generation optical fibre Bragg grating sensors, in particular PCF-FBGs for gamma dosimetry.

- To examine the effects of pre-irradiation and temperature on the gamma irradiation performance of both Standard optical fibre-based Bragg grating sensors and PCF-FBGs for radiation dosimetry.

- To examine the effects of similar accumulated gamma dose and dose rates on the performance of the FBG’s and to determine the suitability of these sensors for radiation dosimetry.

- To examine the amount of possible strain being produced after full accumulated irradiation, inclusive of recovery.

- To compare the recovery aspects post irradiation between PCF and STD FBGs.

- To determine if any permanent degradation can be identified due to the generation of defects and/or structural damage using SAXS and XRD analysis.

1.4.2 Secondary objectives

- To help elucidate and circumscribe the permanent mechanism that causes the Bragg wavelength shift.

- To determine if and which type of FBGs are compatible and suitable to be used as radiation dosimeters in a nuclear environment.
1.5 Research Questions

1. What are the effects of relaxation in relation to time on FBGs, after irradiation?

2. Does pre-irradiation affect the SMF-FBGs and PCF-FBGs performance?

3. Do the new generation optical fibres (PCF) improve and overcome cross-sensitivity problems?

4. Which FBGs are suitable to act as a dosimeter for high dose radiation?

5. Do the results show that the radiation sensitivity of the FBGs is dependent on the chemical composition (doping) of the optical fibre that they are written in?

6. Which FBGs produce the strongest recovery after irradiation?

1.6 Thesis Outline

The thesis contains eight chapters. Chapters 1, 2, and 3 include the introduction, relevant theory and literature review. It must be noted that throughout the first three chapters, the content relates to the objectives and aim already outlined. Chapter 4 highlights the research methodology and setup used throughout the three experimental stages. Chapter 5 then reports on the irradiation results throughout each stage, in particular, the BWS associated with gamma dose level, accumulated dose, and recovery aspects. A summary of findings is provided at the conclusion of each stage. Chapter 6 looks at the spectroscopic characterization analysis and reports on any structural damage in connection with irradiation and the BWS. Chapter 7 highlights the overall conclusion. Finally, Chapter 8 describes any relevant future work.
CHAPTER 2
THEORY

This chapter outlines the main principles of FBGs, and the various manufacturing techniques used in writing or inscribing the Bragg gratings in various optical fibre, relevant to this research. The effects of radiation in optical fibre and the formation of defect colour centres is also highlighted. It is intended that the information provides a basic platform such that the experimental results and data can be understood.

2.1 Light guidance in Single mode optical fibers

The principle behind the wave guidance property of optical fibre relies on the idea of reflection. If a light ray passes between media of different refractive indexes e.g. a medium with a refractive index of \( n_1 \) to another \( n_2 \) and \( n_2 \) is greater than \( n_1 \) e.g. air to glass, the refractive wave in the second medium will bend towards the normal [49]. Conversely if \( n_2 \) is less than \( n_1 \) the wave will be bent away from the normal. When \( n_2 \) is less than \( n_1 \) there is a point where the incident ray will be totally internally reflected. To achieve light propagation inside SMF, the core refractive index \( n_1 \) has to be higher than the cladding \( n_2 \). In SMF both the cladding and core are made of silica; however, refractive index of cladding is less than that of core. This fulfils the condition for total internal reflection, as shown in Figure 2.1.

![Figure 2.1: Light propagation in optical fibre; showing high and low refractive index [40].](image-url)
The angle where the refracted wave is placed along the surface of the boundary between the two mediums and does not enter the second material is known as the critical angle, $\theta_c$. Light will be reflected at the boundary for all angles of incidence greater than the critical angle. When the incident angle is increased, the refractive wave is turned back into the first medium and total internal reflection occurs. Putting $\theta_2 = 90$ deg in Snell’s law of refraction we can find the critical angle $\theta_c$ seen in equation (2.1) [49].

$$\theta_c = \sin^{-1} \frac{n_2}{n_1}$$  \hspace{1cm} (2.1)

Optical fibre has the ability to collect light. The light gathering ability is known as numerical aperture (NA). The larger the signal of the NA equates to signal loss and decrease in bandwidth. NA can be expressed by the following equations (2.2) and (2.3), where $n_1$ is the index of the core of the fibre and $n_2$ is the index of the cladding. $\theta$ is the half angle of the acceptance cone of the fibre [49].

$$NA = \sqrt{n_1^2 - n_2^2}$$  \hspace{1cm} (2.2)

or

$$NA = \sin \theta$$  \hspace{1cm} (2.3)

An important parameter to consider in relation to radiation effects in optical fibre is that of transmission or power loss. When signals travel through the fibre attenuation can occur. Some of the factors that influence signal loss include impurities in the fibre, colour defect centres or absorption bands that cause variations in the uniformity of the fibre resulting in scattering [38]. The transmitted power through the fibre is given by equation (2.4). $P_0$ is the power into the fibre, $L$ is the length of the fibre and $\alpha$ is the attenuation constant (fibre loss) [49].

$$P_L = P_0 e^{-\alpha L}$$  \hspace{1cm} (2.4)

Typically the fibre loss is measured in units of decibels per kilometre (dB/km), using the relation:
\[ \alpha_{db} = \frac{10}{L} \log \frac{P_f}{P_0} \]  

(2.5)

where \( \alpha_{db} \) equals the loss in decibels.

The fibre loss is a function of frequency which means that fibres will have a greater loss at some frequencies than others. The losses are specified at certain wavelengths rather than at certain frequencies [49].

### 2.2 Light guidance in Photonic Crystal fibre (PCF)

With conventional single mode fibres (SMFs), the geometry inevitably involves a core that is doped primarily with germanium, which is surrounded by a pure silica cladding. This ensures that the core refractive index is higher than the cladding. However, within the geometry of Photonic crystal fibre, which is made typically of a single silica material, the core can be either solid or hollow, surrounded by a microstructured air hole cladding running along their entire length [50,51], as seen in Figure 2.2.

The two types of core seen in PCFs are: (1) Solid core PCFs, which comprise of a solid core surrounded by a periodic array of microscopic air holes and; (2) hollow core PCFs which have an air hole which is surrounded by microscopic air holes [50]. During the manufacturing process and modelling of SMF there is only one parameter to take into account, that is the diameter of the core \( \rho \) seen in Figure 2.3 (a). For PCFs there are three parameters: the core diameter \( \rho \), the diameter of the air holes of the cladding \( d \), the pitch \( \Lambda \) (which is distance between two consecutive air holes) [50], as shown also in Figure 2.3 (b)and (c). PCFs also fall into broad categories of guidance mechanisms: the most common are index guiding PCFs (IG-PCFs) and the photonic bandgap fibres. Each are defined by differing geometry and varied material to cater for these guidance mechanisms. Interestingly, the index guiding PCFs are similar to standard SMF fibres in that they are based on the principle of total internal reflection (TIR) [52].
Solid core PCFs (used in this study) rely on the TIF being modified by the effective cladding refractive index being lowered compared to the core’s refractive index. A typical cross section of a hexagonal structure IG-PCF, and respective refractive profile are shown in Figure 2.4. The light guiding properties in Solid Core PCFs therefore are not from varying the glass composition, as in standard SMF, but from the tiny closely spaced air holes. IG-PCFs structure is defined by two parameters $\Lambda$ and $d / \Lambda$. 
2.3 Fibre Bragg Grating Sensing

A fibre Bragg grating (FBG) is a periodic perturbation of the refractive index along the fibre length which is formed by being exposed to an intense optical interference pattern[16]. The formation of permanent gratings in optical fiber was first demonstrated by Kenneth Hill at the Canadian Communications Research Centre (CRC), Ottawa, Ontario, Canada in 1978 [16]. They achieved this by exposing Germania-doped fibre to intense Argon-ion laser radiation and observed after several minutes an increase in the reflected light intensity occurred until almost all the light was reflected from the fibre. After spectral measurements had been done indirectly by strain and temperature they confirmed that a narrow Bragg filter had been formed over the entire 1m length of fibre [16].

The principle behind the FBG sensor is that when there is a change in temperature, or strain due to stress or pressure, the centre wavelength reflected from the FBG will alter. It is in a sense a type of Bragg reflector constructed or written into the optical fibre. The fibre reflects certain wavelengths of light and transmits all others [17]. The Bragg wavelength is related to the refractive index \( n \) of the material and the grating period \( d \).
A). The grating sensors are therefore based on the reflection and interference of light travelling through the fibre.

When a section of fibre is exposed to axial strain, temperature or pressure changes from an external source eg. during gamma irradiation it will change either or both the refractive index and grating period of the FBG [18]. These affect the Bragg wavelength. This enables changes occurring from pressure or expansion to be detected with the FBG from the shift in the Bragg wavelength [19]. It can be measured by recording the actual change in the reflection coefficient, as shown in the working principle schematic in Figure 2.5.

![Figure 2.5: Basic working principle schematic of FBG Sensor [20].](image)

The Bragg wavelength ($\lambda_B$) is given by,

$$\lambda_B = 2n_{\text{eff}} \Lambda$$

(2.6)

where ($n_{\text{eff}}$) is the effective refractive index of the grating and ($\Lambda$) is the grating period. Changes in the grating caused by strain associated with pressure or expansion results in a wavelength shift [21]. The change in wavelength caused by strain and temperature is given by,
\[ \Delta \lambda = \lambda_B \left[ \varepsilon \left(1 - \frac{n^2}{2} \left[ p_{12} - \nu(p_{12} + p_{11}) \right] \right) + \Delta T \left( \alpha + \frac{1}{n} \frac{dn}{dT} \right) \right] \] 

(2.7)

where \( \nu \) is Poisons’ ratio, \( p_{12} \) and \( p_{11} \) are the strain optic coefficients, \( \varepsilon \) is the applied strain, \( \alpha \) is the thermo-expansion coefficient, and \( \Delta T \) is the change in temperature in equation (2.7), which enables a shift in the wavelength to occur. Equation 2.7 is simplified so that the relative change in Bragg wavelength is,

\[ \frac{\Delta \lambda_B}{\lambda_B} = (1 - p_e) \varepsilon + (\alpha + \eta) \Delta T, \] 

(2.8)

where \( p_e \) is the photo-elastic coefficient and \( \eta \) is the thermo-optic coefficient. Typical values for silica with a core doped with germanium are: \( p_e = 0.22, \alpha = 0.55 \times 10^{-6}/^\circ C \), and \( \eta = 8.6 \times 10^{-6}/^\circ C \) [22]. Therefore, the sensitivity of a FBG with a Bragg wavelength of 1550nm to strain and temperature are [22,23];

\[ \frac{\Delta \lambda_B}{\Delta \varepsilon} = 14.18 \text{ pm } / ^\circ C \] 

(2.9)

\[ \frac{\Delta \lambda_B}{\Delta T} = 1.2 \text{ pm } / \mu^\circ \text{e} \] 

(2.10)

This will be the new FBG wavelength that will be recorded although the theoretical values for strain and temperature above are not absolute, as each FBG from the same fabrication parameters will present slightly differing sensitivity [22]. Much work has been performed investigating the effects of radiation on FBGs. The main area covered for research, is developing radiation resistant FBGs for use in nuclear environments in the areas of temperature and strain measurement applications [1]. For this study the recent research that suggests FBG’s are a candidate as possible high dose radiation sensors is of relevance [10].

The actual sensing ability of the FBG stems from the sensitivity of the refractive index and the grating period within the fibre when being exposed to external forces. These forces affect the response of the FBG directly through expansion and compression changes caused either by strain or pressure [12]. The strain optic effect or the strain
induced change in the glass refractive index, also effects the FBG. An example schematic of the grating period and resultant shift in reflected and transmitted Bragg wavelength before and after external forces (either temperature and or strain) are applied is shown in Figure 2.6, noting the grating period increase.

Figure 2.6: (a) FBG with no external forces, (b) FBG with forces applied noting the grating period increase and resultant wavelength shift.
FBGs can have varying resonant wavelengths depending on the way they are written. This allows them to be multiplexed into a sensor system where varying stresses and temperatures can be measured along the fibre at different intervals [12]. This makes FBGs very attractive as a possible replacement for conventional radiation dosimeters. It is important to note that when FBGs are subjected to both strain and temperature simultaneously, it is necessary to isolate and discriminate them in order to obtain each parameter. The grating period (\(\Delta \Lambda\)) changes through expansion or contraction.

When FBGs are subjected to irradiation, a peak shift can also occur due to the refractive index change (\(\Delta n\)) and to the grating period change (\(\Delta \Lambda\)). A Bragg wavelength shift normally shifts towards longer wavelengths during gamma irradiation. The research indicates that the BWS also saturates at different values at different accumulated doses and dose rates. Gamma irradiation can induce changes in the refractive index (\(\Delta n_{\text{eff}}\)) and the grating period (\(\Delta \Lambda\)). Based on equation (2.6) we can measure the radiation-induced BWS:

\[
\frac{\Delta \lambda_{\text{B}}}{\lambda_{\text{B}}} = \frac{\Delta n_{\text{rad}}}{n_{\text{eff}}} + \frac{\Delta \Lambda}{\Lambda}
\]

where \(\Delta \Lambda\) and \(\Delta n_{\text{rad}}\) are the grating period and radiation-induced effective refractive index changes. For a FBG with a \(\lambda_{\text{B}} \approx 1550\) nm, a refractive index change of \(10^{-4}\) corresponds to a Bragg peak shift of \(\approx 100\) pm [24].

### 2.4. FBG Fabrication

The method known as writing or inscribing is used in the fabrication of FBGs. The fibres refraction index is dependent on the density of the dopant it contains. Germanium (Ge) doped silica fibre is the usual form of dopant used. It is used because it is photosensitive, and when it is exposed to intense ultraviolet light, periodic changes to the refractive index of the fibre core occur [26]. This is achieved by a technique known as laser writing, which has the effect of creating areas of either less or more dense amounts of dopant in the fibre. To write an index grating directly on to the doped fibre, two UV
beams are placed at an angle to produce an interference pattern. One of the first methods used in the fabrication of Bragg gratings was the transverse holographic technique [27]. The holographic technique was achieved by exposing the fibre core without removing the glass cladding to two overlapping ultraviolet light beams [16]. The interfering beams produced a periodic interference pattern, consisting of dark and light bands, causes the change in the refraction index by the movement of the dopants in the fibre [28] as seen in Figure 2.7. The resultant Bragg grating that is photo-imprinted is dependent on the angle between the two interfering UV beams. The holographic technique is successful because the fibre cladding is transparent to UV light compared to the fibre core, being absorbant to UV light [16].

One other method for writing or inscribing an index grating is known as the phase masking technique. This is the technique used to fabricate the gratings used in this study. A phase mask is made from a flat substrate of silica glass which is transparent to UV light [16]. On one side of the surface a one dimensional periodic relief structure is etched using photolithographic techniques such as electron beam lithography [16,22]. The phase mask formed is positioned between a UV light source and photosensitive fibre as shown in Figure 2.8. The phase mask pre-determines the wavelength of the reflection grating by the varying light intensity illuminating the fibre. The shape of the pattern approximates a square wave in profile [16]. The phase mask period (\( \Lambda_{\text{mask}} \)) determines the period of the imprinted grating (\( \Lambda_{\text{mask}} /2 \)). When the fibre is placed close to the corrugations of the phase mask, UV light which is incident normal to the mask passes through and is
The beam is now divided into several diffraction orders. The light in the zero order is suppressed to less than 5%, whilst the +1 and -1 orders prevail with the remaining power, approximately 40% which is divided equally [16]. An actual photograph of a phase mask is seen in Figure 2.9 [30].

The UV method of writing FBG’s is relevant to this study. However for future experiments FBGs manufactured using high energy femtosecond (fs) lasers may be used for comparative purposes. With the normal fabrication method of using UV light, the photons energy of 5eV corresponds to the absorption band of defects in the fibre (usually germano-silicate glass). This fibre is photosensitive due to a high Ge concentration or enhanced by Hydrogen loading [31]. With FBG inscribing, commercial femtosecond lasers are used to produce (fs) UV pulses. The pulses are directed by a phase mask onto the fibre by varying the displacement of the lens in relation to the fibre, as seen in Figure 2.8.
2.10. This allows the variation in UV irradiation intensity. This exposure variation results in the formation of strong FBGs [31].

![Figure 2.10: Femtosecond laser set up with phase mask [30].](image)

2.5 FBG Inscription in Photonic Crystal Fibre

FBGs have proven themselves to be versatile sensing devices capable of measuring a plethora of physical parameters such as temperature, strain, pressure, acoustic noise and much more [20]. As mentioned, FBGs offer a range of attractive attributes such as immunity to electromagnetic interference, high sensitivity, wide dynamic range and their ability to be easily multiplexed. Multiplexing also has been achieved within a single MOF using conventional FBG inscription setups achieving with repeatability and quality that match standard fiber sensing requirements [96]. It demonstrates that the optical properties of the MOF are not compromised in order to produce an array of multiplexed FBGs [96].

This is important as the third stage of experimentation for this study uses a multiplexed PCF. To date, almost all FBG-related research has focused on conventional all-solid, step-index fibres with germanium doped cores. However, there exists another breed of optical fibres that only stands to benefit from the added functionality brought by FBGs: the air-structured fiber family comprises of many members such as Fresnel fibers [97], photonic crystal fibres (PCFs) [98], photonic bandgap fibres [99] and air-clad fibres [100], all of which offer special qualities that set them apart from their step-index counterparts. PCFs for example, offer highly customizable chromatic dispersion [101], endlessly single mode...
operation [102], high optical nonlinearities [103], and the ability to insert liquid or gases into the holes for spectroscopic analysis [104].

The first demonstration of FBG fabrication in an air-structured fibre involved a germanium doped core and pre-pressurization with deuterium to increase photosensitivity [105,106]. FBG inscription in a pure silica PCF was first demonstrated using 193 nm radiation from an ArF laser to directly excite 2-photon absorption at the band edge of silica [107]. Femtosecond lasers operating at 800 nm have also been shown to inscribe FBGs in pure silica PCF [108]. Subsequent work used 193 nm radiation to inscribe type 1n (IIa) gratings in germanium doped PCF and showed they were capable of withstanding temperatures of up to 700 °C [109]. In every case, scattering of the incident inscription light by the fibre cladding air holes is the most limiting factor, as it reduces the efficiency of light reaching the core.

This study has used FBGs fabricated in endlessly single mode (ESM) PCFs by the direct-write method using a 193 nm ArF laser; and combined with hydrogen loading to generate an index change within a stress free PCF. The primary application of these sensors is in gamma dosimetry. FBGs written in pure silica with hydrogen is not new. Albert et al. [110] have reported FBG inscription in hydrogenated (P ~100 atm, t = 18 days, T = 20 °C) all-silica core fibers using a 193 nm ArF laser (tpulse = 10 ns, Eav ~200-400 mJ/cm2). These had a fluorinated cladding with depressed index such that interfacial stress plays an important part in the mechanism. They obtained R = 25 dB and FWHM ~250 pm for a grating of unstated length. Fu et al. [108] have also reported FBG inscription in hydrogenated (180 atm for 7 days at room temperature) all-silica PCF using 267 nm laser (tpulse = 120 fs), for accumulated f ~ 70 kJ/cm2 over 60 min. R = 10 dB depth and FWHM ~300 pm for a grating of unstated length were obtained. Pissadakis et al. [111] have reported the inscription of FBGs into hydrogenated (P = 130 atm, t = 15 days, T = 25°C) ESM PCFs using 248 nm laser (tpulse = 5 ps, Eav ~ 100 mJ/cm2). They report R = 5 dB and FWHM ~ 100 pm for a grating L = 1 cm. The coupling strength, defined here as reflection per unit length, is 5 dB/cm.

For this thesis, we have used 193 nm with silica PCF unconstrained by a cladding producing R = 6 dB, FWHM ~ 80 pm for a grating L = 1 cm, producing a coupling strength of 6 dB/cm, leaving room for further optimization [112]. The response to gamma irradiation compared to STD-FBGs is reported in Chapter 6.

Recently, there has been increased research in microfibers/nanofibres. They have attracted great attention among researchers in the field of optical fibre sensing because of
their attributes which include, flexibility, high sensitivity, low loss, low dimension, high spatial resolution and fast response [113]. The difference between common single mode fibre and microfiber is that essentially the microfiber consists mainly of the fibre core surrounded by air [114]. When light travels along the fibre, it is tightly confined to the fibre core due to contrast between the core and air seen in Figure 2.11. The locations and shapes of the first few rings of air holes are visible with the light strongly confined to the core. The large refractive index allows a large fraction of the guided light to propagate outside the fibre as a evanescent wave, which in turn makes it highly sensitive to the ambient medium [114].

![Figure 2.11: Light guiding confinement properties of PCF[167].](image)

Further techniques have been documented in the literature for the fabrication of FBGs on microfiber. One method is with the use of a femtosecond laser. The successful FBG fabrication using femtosecond pulse irradiation has been achieved in microfibers with diameters ranging from 2 to 10 μm [114]. The microscope image of the FBG in the microfiber with a 10 μm diameter is shown in Figure 2.12. This technology would be of particular interest in future research with regards to radiation dosimetry.
Figure 2.12: Microscope image of the microfiber/nanofiber with diameter of 10 μm[114].

2.6 Types of Gratings

There are several varied types of gratings available in today’s market. They fall into different categories by the format and process from which they are generated. The main or most common gratings are; Type I, Type II, Type IHp (formerly Type IA), and Type In (formerly Type IIA). Fundamentally, there are two types Type I and Type II: those associated with index change below the damage threshold of glass (type I) and those associated with change above the damage threshold (type II) [32]. As mentioned previously, FBGs can be inscribed with either a UV laser (pulsed or continuous wave) or a femtosecond laser (ultrashort pulsed). For this reason, a description of UV-FBGs will be discussed as they were used throughout the experimental stages.

**Type I FBGS**: These are often known as the most common standard gratings, as they are formed in germanosilicate fibre or Ge-doped silica [12]. Type I index changes are associated around the centres excited by UV light or a multiple photon process accessing the same energy levels [32]. In standard Ge-doped fibre, small index changes coincide with oxygen deficiency centre (ODC) absorption bands around 244 nm and 320 nm [32]. These are easily accessible by common light sources such as ArF (pulsed 193 nm) and KrF (pulsed 248 nm) lasers. The formation of FBGs is caused by the continuous or pulsed UV radiation exposure of relatively low intensities below 1-10 MW/cm² in various types of fibres either photosensitized or intrinsically photo-sensitive [24]. To summarize the main characteristics, the formation of Type I gratings is fairly simple especially in
photosensitive doped fibres (mainly germanosilicate) and even doped photonic crystal fibre. Type I gratings are also annealed at temperatures above designed operating temperature to gain stability in their reflectivity [12]. The thermal resistance is stable to ~320 °C. With regards to environments with temperatures above 450 °C, Type I gratings are unsuitable as most of the refractive index change is annealed out at these temperatures [2]. Type I gratings were used throughout the experimental stages in this study.

**Type II FBGS**: Also called damage gratings, are obtained by increasing the energy above the damage threshold. To reach the required intensity levels exposure is performed by high peak power pulses from UV laser sources such as a 248nm KrF excimer laser with a 20 ns pulse duration which corresponds with >50 MW/cm² [24]. The gratings produced with this intensity exceed the damage threshold of the glass leading to fracturing and/or void formation. These gratings show high temperature stability and are stable at temperatures above 1,000 °C making them ideal for ultra-high temperature sensing.

**Type IHp FBGS (formerly Type IA)**: Also known as regenerated gratings, and are usually formed in Ge-doped or B/Ge co-doped fibre [24]. Type IHp also uses Hydrogen loading for the formation of gratings. During the extensive continued UV exposure, bulk refractive index keeps rising although index modulation is small overall. The grating formation is generally quite fast in this process. There is also a large red shift in the central Bragg wavelength (>10nm), and > 10⁻² index change is possible [32]. They have very similar properties to Type IIA gratings which suggests there is anisotropic stress equilibration through anisotropic OH formation [32].

**Type In FBGS (formerly Type IIA)**: Also known as regenerated gratings these are usually formed within high germanium and/or boron co-doped fibers. At the beginning of exposure, a Type I grating is written but with the addition of further prolonged UV exposure this grating is erased and a new one is inscribed [24]. The new secondary grating exhibits a steady blue shift in spectral response; hence there is a negative refractive index change [12]. With optimized fabrication conditions, this type of grating can be produced, making them stable up to 700 °C. Main applications are in the area of high temperature sensing.
2.7 Gamma Radiation

Gamma rays are a form of electromagnetic radiation (EMR). They are the same as X-rays but are distinguished only by the fact they come from the nucleus. EMR can be described in terms of a stream of photons, which are massless particles each travelling in a wave-like pattern and moving at the speed of light [162]. Each photon contains a certain amount of energy, and all EMR consists of these photons. They originate from the nucleus of a radionuclide following radioactive decay whereas X-rays are produced when electrons are rearranged within an atom [162]. The gamma ray is essentially electromagnetic energy or photons which have the highest energy and shortest wavelength in the electromagnetic radiation spectrum [11,162]. Gamma radiation consists of a photon energy being emitted from an unstable nucleus as shown in Figure 2.13.

![Gamma Radiation](image)

Figure 2.13: Gamma radiation: The emission of an high-energy wave from the nucleus of an atom [25]

With having no mass or charge, gamma radiation travels though air much further than alpha or beta, losing on average half its energy every 500 feet [25]. The high energy of gamma rays allow them to pass through most materials as shown in Figure 2.14. Lead and depleted uranium are some of the main materials used to slow or stop the gamma photons. Gamma emitting radionuclides are the most common and widely used radiation sources. The penetrating power of gamma photons has many applications. Whilst gamma rays penetrate many types of materials, they do not make them radioactive. The relatively low radiation emission levels makes it safer for workers in the industrial environment. The most useful radionuclides are cobalt-60 (used in this study), cesium-137, technetium-99m and americium. Cobalt-60 is used e.g. to pasteurise certain foods, sterilise medical
equipment in hospitals, gauge the thickness of metal, and medical radiotherapy. Other applications include the modification of polymers to improve their thermal and mechanical characteristics. Cobalt-60 is convenient and cost effective for industrial use and plays a vital role in the scientific community.

![Figure 2.14: The penetrating capacity of different types of radiation inclusive of Gamma](image)

### 2.8 Silica Optical Fibre Defects

The material used in the fabrication of optical fibre in use today, consists mainly of silicon dioxide (SiO₂) known as pure glass [1] or doped amorphous silica dioxide (a-SiO₂). Plastic fibres and fluoride based glass are also in use, but have limitations. Plastic optical fibre in particular has high attenuation compared to glass fibres, making them less suitable for optical sensing in radiation environments [1]. The amorphous state incorporates the central silicon (Si) atom bonded to four oxygen atoms which occupy the corners of the tetrahedron as shown in Figure 2.15 (a) [33]. The perfect silica structure can be viewed as a continuous random network joined at the corners however the amorphous state shows a lack of periodicity with a degree of randomness also shown in Figure 2.15 (a) [33]. For this study the FBGs were produced in amorphous silica dioxide (a-SiO₂) meaning that during irradiation, defect centres can form in the silica material of optical fibres. These defects are associated with absorption bands within the silica bandgap which decreases the glass or fibre transparency.
Defect structures are related to under-or over coordinated atoms, or substitutional impurities such as Cl or H as seen in Figure 2.15 (b) [33]. Defect centres or colour centres are the main factors in the cause of attenuation loss [34]. The radiation response of silica-based glass has been studied for many years and it is well known that radiation can degrade glasses through varying mechanisms depending on the nature, dose (fluence) and dose rate (flux) of radiations [35]. When exposed to radiation, changes in the optical fibre refractive index occur, due to the damage of the fibre matrix structure, increasing the fibre absorption loss [36]. The defects induce new energy levels inside the band gap, with the result being an increase of absorption of the transmitted signal. The increase of absorption is known as Radiation Induced Attenuation (RIA) [37].

With the addition of dopants, such as Germanium, Fluorine, Boron, Erbium or Phosphorus to the fibre core, it was found that optimization of propagation occurs, along with a reduction of attenuation [38]. The most commonly used core material in optical fibre used today is Germanium(Ge) doped silica or germano-silicate glass [36]. Both germano-silicate and silicate glass show attenuation rates as low as 0.20 dB/km at a wavelength of 1550 nm [39]. Germanium is a dopant used to increase the index of refraction between the core and cladding, providing enhanced light guiding properties.
Germanium doped silica fibre is also highly photosensitive which makes FBG inscription easier.

With the addition of hydrogen loading and boron co-doping, the photosensitivity of the fibre can be enhanced even further [40]. Germanium doping and hydrogen loading in combination is by far the most widely used method to increase radiation sensitivity of commercial fibre. For this reason it is used during the experimental stages.

2.9 Germanium Doped Silica and Associated Defects

The fabrication of glass fibre with core dopants such as germanium, previously mentioned, can lead to a variety of defects. During the manufacturing process, the defects occur because it is not possible for 100% perfection, therefore the deposited chemicals with the core dopant germanium, form suboxides such as GEOx (x=1 to 2) [30]. The best known of the defects are paramagnetic Ge($n$) defects, where $n$ is the amount of neighbour Ge/Si atoms surrounding a Ge ion with an unpaired single electron. In the first structural models proposed the irradiation induced Ge related defects have been named Ge($n$), with $n$=0, 1, 2, 3 [41]. When exposed to UV irradiation, the bond is broken creating what is known as a GeE' centre, as shown in Figure 2.16. A free electron is now free to move within the glass matrix through tunneling or by two photon excitation into the conduction band [40]. The removal or movement of the electron causes a change in the shape of the molecule which may in turn change the density of the material. This causes the formation of colour centres (GeE') or absorption bands, resulting in the change of the refractive index [42]. The relation between these phenomena and the Ge related defects was widely studied as well as the structures and the physical properties of the defects [43,44]. Several experimental studies pointed out that the exposition of the Ge doped silica to ionizing radiation (UV, X, γ or β ray) induces the generation of optical absorption (OA) bands and of different EPR signals [44]. The EPR signals were attributed to three Ge related defects named Ge(1), Ge(2) and E'Ge [44]. The absorption bands are responsible for transmission losses.
2.10 Radiation Effects on Optical Fibres

Research indicates that optical fibre properties change, causing a deterioration in transmission after exposure to irradiation. Radiation exposure leads to the generation of point defects inside the silica glass matrix resulting in three macroscopic changes to the optical properties [45]. The changes observed in silica based glass under irradiation include: 1) Radiation –Induced Attenuation (RIA), 2) Radiation -Induced Emission (RIE), and 3) Compaction [33]. RIA decreases the fiber transmission efficiency through an increase of the linear absorption due to the absorption bands of the radiation induced point defects [33]. RIE corresponds to the light emission within samples when exposed to irradiation. The luminescence can be from pre-existing or point defects caused by the excitation from incoming particles or Cerenkov emission and has the effect of decreasing the signal-to-noise ratio [33,45]. The final change related to irradiation is compaction, which is when the refractive index changes are related to silica density variations ($\rho$). Therefore, after exposure to gamma irradiation in nuclear environments a change in the refractive index (RI) can occur from density changes or from RIA. According to the Kramers-Kronig dispersion relations, an increase of attenuation is accompanied by a change of the RI of the glass ($n$). Overall, the RI change is due to the combination of density change (via the Lorentz-Lorenz formula) and induced absorption effect (via Kramers-Kronig relations) [33]. Therefore, a change in absorption ($\Delta\alpha(\lambda)$) affecting the refractive index ($n(\lambda)$) is explained by the Kramers- Kronig relation [24,164]:

![Figure 2.16: GeE’ centre. Germania defects in Germania doped silica. An electron is released on breaking of the bond [40]](image)
\[ \Delta n(\lambda) = \frac{1}{2\pi^2} \int_{0}^{\infty} \frac{\Delta \alpha(\lambda')}{1 - (\lambda' / \lambda)^2} d\lambda' \]  

(2.12)

The Kramers-Kronig relation only accounts for the changes to the refractive index via the absorption; i.e. a decrease predicted by the colour centre model results in an increase in the refractive index. There can also be structural changes occurring in the matrix, changes in strain after exposure and density changes. The discrepancy attributed to the density \( \rho \) modification effect can be derived from the Lorentz-Lorenz equation for the molar refractivity \( R \) [24]:

\[ \Delta n = \frac{(n^2 + 2)(n^2 - 1)}{6n} \left( \frac{\Delta \rho}{\rho} + \frac{\Delta R}{R} \right) \]  

(2.13)

As far as the density changes related to the FBG inscription are spatially non-uniform they result in stresses, and the corresponding photo-elastic contribution to the refractive index modification must also be considered [24, 46, 47]. The Kramers-Kronig relation in equation (2.12) is precise, and explains the refractive index modifications, provided the absorption changes are known for the whole frequency range from zero to infinity [24]. The density modifications correspond to a shift in the forbidden band edge in the deep UV range. The quantification of this effect is difficult, and equation (2.13) complemented with the corresponding photo-elastic contribution can be considered as a way of performing integration of equation (2.12) for the band gap edge UV range.

Overall the radiation response is dependent on several parameters which include the fibre composition, including the chemical composition and photosensitization technique used [11]. RIA is mainly caused by the excitation of the Ge atoms in the core of the optical fibre when exposed to gamma radiation. Optical fibres guide light through the core due to the total internal reflection which comes from the difference between the index of refraction between the core and cladding of the fibre [26]. Germanium is a dopant used to increase the index of refraction between the core and cladding, providing enhanced light guiding properties [26]. With attenuation, the optical power traveling through the optical fibre decreases with distance as a result of absorption and scattering [48]. It is
defined in units of decibels per kilometer (dB/km). FBG’s however, seem to avoid the broadband radiation induced optical power loss because of the narrow wavelength encoding or narrow spectral range of \(< 5 \text{ nm}\) [11]. When Ge-doped fibres are exposed to gamma irradiation, a change in the effective refractive index occurs, which results in a radiation induced Bragg wavelength shift, and also a lowering of the baseline due to RIA, as shown in Figure 2.17. The radiation-induced loss of gamma radiation on optical fibres is dependent on the wavelength. It is especially pronounced in the 700-1100 nm range and at 1390 nm. At 1500 nm it is lowest [36] as shown in Figure 2.18. This result is relevant to this experiment, as we have used a light source with a centre wavelength of approximately 1550 nm.

![Figure 2.17: Radiation induced Bragg peak shift, with baseline lowered due to RIA [11].](image)
Figure 2.18: Optical spectrum loss curve comparison before and after gamma exposure [36].

2.11 Conclusion

This chapter emphasized enough theoretical information in relation to light propagation, and the formation of FBGs in optical fibre (both standard and photonic), such that the following chapters can be clearly understood. The radiation effects on optical fibres is discussed extensively, outlining the generation of point defects inside the silica glass matrix which result in macroscopic changes to the optical properties.
CHAPTER 3
LITERATURE REVIEW
FBGs in radiation environments.

This literature review incorporates the areas that are relevant to the topic. It includes research that has looked at FBGs being used as possible high dose gamma radiation sensors and examination of radiation damage in FBGs. The effects of pre-irradiation, response during recovery periods (no irradiation), and dose rates are highlighted. The research on the use of different fibres, their behaviour and sensitivity seen by a Bragg wavelength shift to gamma radiation is also covered. This chapter also provides a fundamental hypothesis on the selection of Photonic Crystal fibre-FBGs, to be used as a direct comparison between commercial off the shelf standard SMF-FBGs when both are exposed to gamma irradiation. To date there has been very limited research, if any, involved with the direct comparison of behaviour whilst exposed to gamma irradiation. An area of conjecture is the effect of the amount of time needed during the recovery phase of FBGs after irradiation, and whether full recovery possible after exposure. Comparative research between Standard SMF and PCF–FBGs is regarding recovery phases and responses is minimal at best.

3.1 Significant Journals

A number of Journals can be considered directly related to the topic. These journals are available in full text, and will be used as primary reading sources throughout the candidature. The primary journals include:

- IEEE Transactions on Nuclear Science.
- Journal of Optics, by Institute of Physics.
- Journal of Lightwave Technology, by IEEE.
- Measurement Science and Technology (IOP)
3.2 Databases

There are several major databases that have been used in the sourcing of relevant literature. By doing this it has led to the discovery of other journals where similar subject related material is likely to be found. The most pronounced and significant databases include:

- The International Society for Optical Engineering (SPIE) digital library
- The Institute of Electrical and Electronic Engineers (IEEE) Xplore Digital Library
- The Institute of Physics (IOP) Electronic Journals
- The Optical Society of America (OSA) Digital Library

Other databases such as Science Direct were also used in assisting the search for relevant material.

3.3 Authors and Research Groups

A number of important authors and research groups have been identified. In the field of developing fibre Bragg gratings for radiation environments Henning Henschel, Stefan k. Hoeffgen, A. I . Gusarov, A.Fernandez Fernandez, Udo Weinand, and Katerina Krebber, Sylvain Girard, Adriana Morana, Emmanuel Marin, and their colleagues, are amongst the most prominent. Some of the high profile research groups include:

- Communications Research Centre, Ottawa, Canada.
- Federal Institute for Materials Research and Testing, Berlin, Germany.
- Fraunhofer-INT, Euskirchen, Germany
- Optoelectronics Research Centre, University of Southampton,
- SCK-CEN Belgian Nuclear Research Centre, Boeretang, Belgium
- Hubert Curien Laboratory, Saint-Etienne, France
3.4 FBGs in Nuclear Environments

When optical fibres are exposed to radiation (gamma, neutron or alpha particle radiation), the three main/primary effects that occur to optical fibre are [36]:

(1) there is an increase in optical fibre absorption loss,
(2) changes occur to the refractive index of the fibre, and
(3) optical fibre luminescence occurs.

A study was conducted noting the radiation effects by both gamma and neutron radiation fields in commercial off-the-shelf single mode optical fibres [53]. This study looked at the comparison between gamma and neutron exposure on Ge-doped and pure silica core fibre and tried to establish loss levels, to ascertain if the damage mechanism was consistent. Seven fibres from different manufactures were used. Six were germanium doped and one contained a pure silica core (non Ge doped). The gamma irradiation dose rate is 720 Gy/hr up to a total dose of 100 kGy. The loss levels of all fibres were similar ranging between 0.04 dB/m and 0.06 dB/m at 100 kGy. For doses up to 20kGy, the Sumitomo pure silica core fibre showed a lower loss, although when 100 kGy accumulated dose was reached all fibres were within 0.04-0.06 dB/m [53] as shown in Figure 3.1.

![Figure 3.1: Gamma induced loss at 720 gy/hr up to 100kGy. Fibre b is the Sumitomo pure silica fibre. Fibres a1, a2, c, d, e are germanium doped [53].](image-url)

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The induced loss due to neutron radiation effects indicated that the loss of all fibres is below 0.05 dB/m. By comparing loss versus time for gamma irradiations at different dose rates with results of the neutron irradiation, the mechanism of the loss is similar, as shown in Figure 3.2.

![Figure 3.2: Comparison of loss due to varying dose rates of Gamma and Neutron [53].](image)

Figure 3.2: Comparison of loss due to varying dose rates of Gamma and Neutron [53].

The consistency of the overall shape of the graph indicates the damage mechanism is the same. Whether under exposure by gamma or neutron radiation, the induced losses are consistent with one another, i.e. at 1300 nm the induced losses were below 0.1dB/m [53].

The radiation damage to optical fibre is dependent on the material and type of radiation. When optical fibres are irradiated by gamma radiation, the Compton scattering effect will usually be the most dominant effect [36]. Different photon energies cause a variation of effects in different cross sections in optical fibre, as shown in Figure 3.3. The penetration power of gamma radiation at varying levels of photon energy is also evident in Figure 3.4. The interaction between a high energy photon and an individual electron results in high energy electrons causing radiation damage, due to the gamma ray absorption in the fibre [31]. The high energy electrons increases the concentration of colour centres which results in the additional absorption of light [36].
An important factor when considering the use of FBGs in the field of radiation dosimetry is the effects of long term exposure. A long term response over a period of 8 years to radiation on fibre Bragg gratings in a nuclear reactor has been examined [54]. The FBGs used were fabricated in photosensitive and standard off-the-shelf fibres. They remained in the reactor, during which time it was operational for a total of 4690h. The results indicated that after eight years of exposure, the shape of the gratings spectra and amplitude remained unchanged for fibres without hydrogen loading. The fibres with hydrogen loading showed only slight changes to the grating spectra and amplitude [54]. The study is important as it shows that Bragg gratings can withstand long term exposure.
without significant degradation of reflectivity and with only a slight shift of the Bragg peak [54]. As most research previously conducted have evaluated FBGs in harsh conditions over relatively short time scales of days and weeks, the long term study responses are ideal for comparative purposes. A similar 50 month study showed long term effects on fibre Bragg grating temperature sensors in a low flux nuclear reactor [55]. Results showed that the sensors still functioned and reinforced the idea that FBGs can withstand long-term exposure to at least moderate nuclear radiation. The temperature sensitivity was unaffected by the long term irradiation which resulted in a measurement change with an accuracy better than 3°C, which is acceptable for measurement applications in nuclear installations [55]. The two studies show the viability of FBGs being used as radiation dosimeters long term.

3.5 UV Excitation for FBG Inscription

As mentioned previously the various types of Fibre Bragg gratings can be written by either UV low intensity light (I < 10 MW cm⁻²) or by high intensity UV femtosecond laser. The most common used UV lasers for FBG fabrication are KrF (248 nm) and ArF (193 nm) excimer lasers [56,159]. For standard excimer lasers at 248 nm, the inscription of FBGs is used in conjunction with a continuous UV source at 240 nm. The refractive index produced at these wavelengths is linked with the absorption band of defects found in the germanosilicate glass. The other way to reach a high value of excitation energy is to use the high intensity 264 nm UV laser irradiation. This results in an increase in the photosensitivity because of the additional excitation from the two-photon excitation process, as seen in Figure 3.5 [56].

![Figure 3.5: Photoexcitation and energy levels in Germanosilicate glass [56].](image)

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This sensitivity was highlighted in a study by Gusraov et al.[31]. They studied the cobalt-60 response of FBGs fabricated by high intensity femtosecond UV laser light. The results showed that although the gratings were inscribed in differing fibres, the resultant BWS were very similar [31]. These results are in contrast to gratings written with low intensity UV lasers and also with IR femtosecond laser. This means that the UV femtosecond laser fabrication procedure significantly influences the radiation sensitivity and may offer new possibilities in its tailoring [31]. The most common form for FBG inscription however is by using low intensity UV light with the photon energy of 5eV.

The most common UV sources used to fabricate FBGs are KrF excimer lasers with a phase mask. As UV laser sources have low spatial and temporal coherence, the fibre is placed as near as possible to the phase mask in order to induce the maximum modulation in the refractive index [57]. The greater the distance the fibre is away from the mask, the lower the induced index modulation, which results in lower reflectivity FBGs [57]. Therefore, when actually writing with UV sources and a phase mask, the distance between the lens and the mask should be at a distance where no damage is caused. To produce a clean uniform fringe pattern the fibre, should be placed a specific distance from the mask. The significance of spatial coherence, in the fabrication of FBGs is shown in Figure 3.6. It shows the fibre core is at a distance \( h \) from the phase mask, with the transmitted plus and minus first orders interfering to form the fringe patterns from different parts of the mask referred to as distance \( d \) [57].

As the distance of the fibre from the mask is the same for the two interfering beams, the requirement for temporal coherence is therefore not important for the formation of high contrast fringe patterns [57]. However, as the distance \( h \) increases, the separation \( d \) between the two interfering beams increases as well. Spatial coherence in this instance is critical in the formation of high contrast fringe patterns. If the distance \( h \) extends beyond the spatial coherence, the interference fringe contrast deteriorates, eventually resulting in no interference at all [57].
Figure 3.6: Schematic of Phase mask geometry for FBG inscription. Plus and minus first-order diffracted beams interfere at the fibre core, placed a distance $h$ from the mask [57].

With the advances in high power fs lasers over recent years, the impact on the technology of writing gratings will be enhanced by reducing factors such as, operating costs and improving the flexibility of systems. An advantage fs infrared lasers have is that they are able to produce FBGs in non-photosensitive fibre, as well as through the protective coating, but are very reliant and dependent on the focusing conditions [58]. Presently however, the cost factors compared to using UV sources are high. UV sources currently are easily accessible and the technique is highly controllable [30]. Femtosecond IR lasers also have been shown to be capable of FBG inscription in non-silica and silica based fibres [12]. Writing of FBGs in pure silica fibres is possible but difficult. Therefore fs (IR) lasers have been examined for use, when writing FBGs in radiation hardened fibres with a pure or F(fluorine)-doped silica core without hydrogen loading [59]. The advantage of this is that a F-doped silica core produces fibre that has a low radiation induced attenuation (RIA) which therefore allows reliable strain and temperature measurements.

The overall results, however, when compared to gratings written by UV laser, show that the radiation induced wavelength in comparison with the IR laser were virtually the
The aim was to find the method which produces FBGs that are radiation insensitive and radiation sensitive. For the radiation insensitive FBGs, the radiation hardened fibres showed a BWS between 3 to 7 pm after a dose of 100kGy. The FBGs constructed with radiation sensitive fibres did not show a higher BWS. The main observation is that the same result was obtained by FBGs written in fibre, hydrogen loaded by a UV laser. Therefore, UV lasers and fs-IR lasers produce FBGs capable of measuring high radiation dose values above 100 Gy, but not sensitive ones for measuring low dose up to 100 Gy [59]. In summary, at this point in time, the UV high intensity laser is the most cost effective method although restrictive, in that the fibre must be enhanced by hydrogen to achieve results.

3.6 Radiation effects on Type I UV- FBGs (same manufacturer)

Type I UV-FBGS are the most common FBG used today. The sensitivity and resultant BWS, however, seems to vary greatly depending on the manufacturing and irradiation protocol. The manufacturing parameters can include the composition of the optical fibre, coating used, and whether hydrogen (H₂) loading was used or not. The irradiation parameters can also vary through the dose rates used and accumulated dose. As Type I FBGS are used throughout the experimental stages it is important to highlight the different responses to ionizing radiation, compared to Type II FBGs. Also, a comparison between FBGs written with low intensity UV-lasers and IR femtosecond lasers is included. In most instances, irradiation causes the BWS to shift towards the red (longer wavelengths) and, depending on the accumulated dose and dose rate, at some point there is a saturation tendency to occur. Figure 3.7 highlights the comparative BWS of 12 different fibres that were irradiated up to an accumulated dose of 100 kGy [60,24]. All the Type I gratings used in the reported study were made by one manufacturer (AOS GmbH, Dresden, Germany) under identical conditions, and irradiations which originated from the same source with the same measuring equipment. The fibres were doped with varying elements such as Boron, Phosphorous, Germanium, Cerium and Nitrogen. As this research included fibres that were Germanium doped and made from the same manufacturer, this study is ideal to be used for comparative purposes. In one of the final conclusions of the study by Henschel et al. [60], it was noted that the highest BWS (160 pm) occurred in fibers 5 & 8 doped with medium or high Ge–content (approx. 10 and 21 mol%) and were hydrogen loaded before FBG inscription. Additionally to the previous fibres particular
interest also surrounds fibre no. 2. This is a standard SMF28 with a low to medium GE – content of approximately 4.5 mol%. This fibre produced a BWS of 115 pm, which is significantly lower than that of medium to high Germanium doped fibres. However when compared with fibre 10 (which has no Ge) and the lowest BWS shift of 50pm, the effects of low to medium to high Germanium doping become evident in relation to being suitable for radiation dosimetry.

Figure 3.7: BWS of Type 1 UV-FBGs made in 12 different optical fibres, under identical manufacturing and irradiation conditions. [24]

As UV-Type I FBGs have been used throughout the irradiation stages and results documented in upcoming chapters, it is relevant to include the contrast between UV-Type II gratings. Figure 3.8 compares the BWS highlighting a significantly higher sensitivity in the Type I grating. Type II gratings produce a BWS of ~ 30 pm and saturated between 15-20 kGy making them less suitable for radiation dosimetry, especially in high dose areas.

Figure 3.8: Comparison of the BWS in Type I and Type II gratings [60]
Figure 3.9 highlights the differing responses between Type I UV-FBGs and Type I-IR and Type II –IR gratings with and without hydrogen loading. The significance is that the Type I -UV FBGS, with hydrogen loading  show the most sensitivity to ionizing radiation with a larger induced red shift [61].

Overall, the study highlighted that when gratings are made under identical conditions by the same manufacturer with fibre of differing composition, the radiation induced BWS varied by a factor of three [60]. However, when varying the fibre fabrication parameters, the BWS changed by a factor of approximately ten [60]. The conclusion is that when reporting FBG responses to irradiation, all details about fibre composition and grating fabrication must be included and considered for proper analysis.

3.7 Radiation sensitivity Ge Doped and Hydrogen Loaded/unloaded Type I UV and IR – FBGs (various manufacturers).

One of the main questions with regards to the FBGs response to irradiation is: does the continuity of manufacturing FBGs have a bearing on the sensitivity and how do the results differ if they are produced by varied manufacturers? A study by, Hoeffgen et. al [62] compared the radiation sensitivity of Type I FBGs made by four different manufacturers. The FBG parameters highlighting the different manufacturers, the laser type used, and whether hydrogen loading was used or not, are listed in Table 3.1. The FBGs were written
in Corning SMF-28 and FiberLogix HNA-01 fibres. The Corning SMF-28 is by far the most common and widely used fibre and is cheaply available in large quantities with very good quality and reproducibility [62]. The Corning SMF-28 is about 5mol% germanium doped, while the Fiberlogix has about 9 mol% germanium. The manufacturers AOS, Business Unitec and University of Mons used UV lasers whilst CRC used a fs-IR laser. This data is relevant to this thesis as SMF28H, Ge-doped optical fibres with and without hydrogen loading are used.

Table 3.1: Example of FBG parameters [62]

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Fiber Type</th>
<th>H₂-Loading</th>
<th>Laser Type</th>
<th>Stabilization</th>
</tr>
</thead>
<tbody>
<tr>
<td>AOS, Germany</td>
<td>Corning SMF-28</td>
<td>yes</td>
<td>UV</td>
<td>3 d at 100 °C</td>
</tr>
<tr>
<td></td>
<td>FiberLogix</td>
<td>no</td>
<td>UV</td>
<td>3 d at 100 °C</td>
</tr>
<tr>
<td></td>
<td>HNA-01</td>
<td>yes</td>
<td>UV</td>
<td>3 d at 100 °C</td>
</tr>
<tr>
<td>Business Unitec,</td>
<td>Corning SMF-28</td>
<td>no</td>
<td>UV</td>
<td>none</td>
</tr>
<tr>
<td>Russia</td>
<td>FiberLogix</td>
<td>no</td>
<td>UV</td>
<td>none</td>
</tr>
<tr>
<td></td>
<td>HNA-01</td>
<td>yes</td>
<td>fs IR</td>
<td>4 d at 100 °C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>no</td>
<td>fs IR</td>
<td>4 d at 100 °C</td>
</tr>
<tr>
<td>CRC, Canada</td>
<td>Corning SMF-28</td>
<td>yes</td>
<td>fs IR</td>
<td>4 d at 100 °C</td>
</tr>
<tr>
<td></td>
<td>FiberLogix</td>
<td>yes</td>
<td>fs IR</td>
<td>4 d at 100 °C</td>
</tr>
<tr>
<td></td>
<td>HNA-01</td>
<td>no</td>
<td>fs IR</td>
<td>4 d at 100 °C</td>
</tr>
<tr>
<td>University Mons,</td>
<td>Corning SMF-28</td>
<td>yes</td>
<td>UV</td>
<td>45 h at 100 °C</td>
</tr>
<tr>
<td>Belgium</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The radiation sensitivity of FBGs made in Corning fibre by three different manufacturers, with Ge-doping with and without hydrogen loading, are shown in Figure 3.10. FBGs with hydrogen loading resulted in an increased sensitivity compared to fibres without hydrogen loading. Interestingly, the FBGs written by AOS and Mons with UV laser show virtually the same BWS. All gratings written by CRC with the fs-IR laser produced a BWS 10-20% lower than the UV laser gratings [63]. The similarity in the radiation sensitivity reinforces the assumption that the two grating types result from similar colour centre formation [62,63].

When fibre is hydrogen loaded, the reaction with the Ge ion forms GeH, which changes the band structure in the UV region. The resultant changes after hydrogen loading cause the refractive index profile to change by increasing the core refractive index [64]. When hydrogen loaded standard single mode fibre is exposed to UV, the refractive index changes and increases in excess of 0.011 [65], resulting in a greater sensitivity to gamma irradiation, as seen in Figure 3.11 [30,66].
Figure 3.10: Comparison of Corning SMF-28 BWS with Hydrogen and without Hydrogen. Different manufacturers [62].

Figure 3.11: Change of refractive index of hydrogen loaded fibre before and after UV exposure with pulsed radiation at 248nm [66].

Hydrogen loading pressure variations on the radiation sensitivity of FBGs in relation to the BWS has also been investigated [67]. Again FBGs, (Type I) were inscribed in standard Corning SMF-28 fibre. Before the actual inscription, the fibres were loaded with H₂ for about one week at 50 °C with pressure at 100bar, 200bar and 300bar [67]. The
Bragg wavelength shift as a function of dose rate was measured and compared. In the usual pressure range of 100 bar and 200 bar, the loading pressure shows no distinct influence or difference on the FBGs radiation sensitivity [67]. As seen in Figure 3.12, FBGs with 100 or 200 bar have about the same BWS within the limits of uncertainty. However at 300 bar there is a very slight increase to the radiation induced (RI) BWS [67]. The results again reveal the range of manufacturing parameters that influence the FBGs sensitivity. For this thesis, all standard fibres used were loaded at 100 bar.

![Figure 3.12: Radiation induced BWS of Corning SMF-28 fibres after hydrogen loading at different pressures [67]](image)

3.8 Temperature effects on RI-BWS

A factor to be considered when quantifying FBGs to be used as gamma radiation dosimeters in nuclear facilities is the effect of temperature. A recent study investigated the influence of FBG temperature during irradiation [67]. FBGs were irradiated at room temperature +25 °C, -50°C and +80 °C for a comparison. It showed that at temperatures at -50 °C or below, the radiation induced BWS is twice than at room temperature. When heated to +78 °C or higher, the BWS is approximately 30% lower than that at room temperature, as shown in Figure 3.13 [67]. Temperature effects can also influence the annealing factor after the end of radiation. At low temperatures there is almost no change in relative annealing whilst at elevated temperatures faster annealing occurs [67]. The variation of annealing in relation to varied temperature in FBGs made by Corning SMF-28, are demonstrated in Figure 3.14 [67]. An interesting study (one of the earliest) was performed on Ge-lightly doped without hydrogen FBGs simulating low earth orbit radiation doses [68]. The results indicated that although there was a change in ambient temperature during the experiment of -1.9 °C, the shifts observed induced by gamma
irradiation increase towards longer wavelengths up to a dose of 30 kGy after reaching a BWS of 64 pm, after which saturation occurred. The study indicated that lightly doped Ge-FBGs will perform well in the low dose and dose rate conditions experienced in space environments, providing adequate temperature control is instituted [68].

In summary, temperature plays an important role when qualifying FBGs for measurements in radiation environments. All of the experiments conducted for this thesis were at ~ 23 °C close to room temperature for continuity.

![Figure 3.13: Radiation induced BWS at room temperature, \(-51°C\) and \(+78°C\) of FBGs made of Corning SMF-28 [67]](image1)

![Figure 3.14: Annealing after irradiation; FBG made of Corning SMF-28 [67]](image2)

3.9 Dose Rate Variation: Effect on RI-BWS

A few studies have demonstrated the effect of varying dose rates of gamma irradiation on the BWS [11,69]. The dose rate is important as it reflects the nuclear environment the
dosimeter may be associated with. For the space environment low dose rates and medium dose levels are to be considered, whilst for nuclear facilities high dose rates and high dose levels are relevant. The study showed that the BWS has a clear dose rate dependence, however the Bragg shift saturation behaviour remains even though the dose rate varies from 1 to 25 kGy/h [11]. The FWHM was not affected, which suggests stability under pure gamma irradiation [11]. The FBGs were written in photosensitive optical fibre Spectran SMT-A 1310H, without any pre- or post-writing treatment. The seven FBGs used all showed the BWS shifted towards the longer wavelengths after the 23 hours of irradiation, whilst the dose rates fluctuated as shown in Figures 3.15 and 3.16. Figure 3.15 highlights the BWS of all seven FBGs whilst dose rates are varied and increase in total dose, whilst Figure 3.16 shows the stable FWHM. Also noted is the recovery aspects, which lasts for 12 hours. As can be seen in Figures 3.15 and 3.16 recovery is limited.

![Figure 3.15: Fibre Bragg grating under pure gamma irradiation at several dose rates highlighting BWS. Dose rate increases from FBG1 to FBG7[11].](image1)

![Figure 3.16: Fibre Bragg grating under pure gamma irradiation at several dose rates highlighting stable FWHM. Dose rate increases from FBG1 to FBG7[11].](image2)
Recently, a research group investigated the RI-BWS dependence on the dose rate for a Type I UV-FBG written in a H₂-loaded photosensitive fibre. The FBGs were written using a phase mask and CW Argon ion laser emitting at 244nm [69]. The fibres are Ge-doped at 5 wt% and >15 wt%. The irradiation tests were performed at room temperature with dose rates of 5, 10, and 50 Gy/s up to an accumulated dose of 30 kGy [69]. As shown in Figure 3.17, the higher the dose rate, the larger the RI-BWS. The study also shows that H₂-loaded photosensitive fibre produces a larger BWS.

Along with dose rate, the accumulated dose is important also. The saturation behaviour and point can vary in relation to the final BWS. With regards to saturation behaviour in harsh environments, a study by Morana et.al [70] included three different types of single mode fibres: 1) with a Ge-doped core; 2) with F-doping in core and the cladding, and 3) with a pure silica core and F-doped cladding. The accumulated dose reached was 1 MGy and emphasizes the FBGs BWS responses written by UV and fs. The responses obtained by the FBG written by UV light in Ge-doped fiber is consistent with the reported literature [70] i.e. there is an initial fast increase BWS towards the red followed by a slower tendency to saturate. The fs gratings in the pure silica core are showing less radiation
sensitivity and in this study have resulted in an initial red shift followed by an eventual blue shift as can be seen in Figure 3.18

![Figure 3.18: Radiation-induced BWS as a function of the dose, dose-rate being 50 Gy/s, noting varying response between UV and fs standard Ge and Pure silica core FBGs at high accumulated dose.][70]

3.10 Pre-Irradiation Effects on FBG

Pre-irradiation is an additional parameter that is important in relation to the RI- BWS. When combined with hydrogen loading and Ge-dopants, there is a significant change to the radiation sensitivity of FBGs [71]. A term known as ‘radiation hardness’ is often used in relation to the effects of pre-irradiation. Radiation hardness of the Bragg gratings, resulting from pre-irradiation, can lead to a decrease in sensitivity, and thus a reduction in RI- BWS [71]. Current research also indicates that the reduction in sensitivity via pre-irradiation produces a more stable FBG [72]. One study showed that pre-irradiation treatment can reduce the variation of radiation induced BWS by 8% to 27% at a dose of 50 kGy, as shown in Table 3.2 [72].

<table>
<thead>
<tr>
<th>FBG</th>
<th>Bragg wavelength shift during pre-irradiation/µm</th>
<th>Bragg wavelength shift during second irradiation/µm</th>
<th>Variation in Bragg wavelength shift</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.046</td>
<td>0.035</td>
<td>-27.08%</td>
</tr>
<tr>
<td>2</td>
<td>0.018</td>
<td>0.015</td>
<td>-16.67%</td>
</tr>
<tr>
<td>3</td>
<td>0.025</td>
<td>0.023</td>
<td>-8%</td>
</tr>
<tr>
<td>4</td>
<td>0.030</td>
<td>0.033</td>
<td>-23.33%</td>
</tr>
<tr>
<td>5</td>
<td>0.025</td>
<td>0.020</td>
<td>-90%</td>
</tr>
</tbody>
</table>

Table 3.2: Variation in BWS, dose rate: 0.1Gy/s, total dose: 50 kGy [72]
All fibres were Ge doped and Hydrogen loaded. The only exception is fibre 2, which is without hydrogen. Fibres 2 and 3 have identical Ge concentration and are both the same fibre type (PSF-GeB-125). Figure 3.19 shows that the BWS is higher in FBG3 than FBG2 [72]. This indicates that hydrogen loading, even after pre-irradiation, can increase the radiation sensitivity of FBGs [60].

![Figure 3.19: BWS of FBG2 (a) and FBG3 (b) during the experiment [72].](image)

A further study [71] found the BWS after pre-irradiation decreases by about 7~9 pm for a dose of ~50 kGy. Figure 3.20 shows the RI-BWS difference between pre-irradiated FBGs (FBGs 2-3,1-2,2-1,2-2) and non pre-irradiated (FBGs 3-6,4-5,3-5,3-4). The FBGs with pre-irradiation have a lower BWS up to a total dose of 50 kGy. To again highlight the effect of hydrogen loading, a comparison between the RI-BWS with a pre-irradiated FBG in SMF-28 with H2 loading and GeO2 doping, and a non pre-irradiated FBG without H2 loading and GeO2 doping is shown in Figure 3.21. FBG1-1, although pre-irradiated, still shows a significantly increased sensitivity compared to FBG4-4, which is not pre-irradiated.

![Figure 3.20: Influence of pre-irradiation on BWS [71].](image)
A comparison with the results of a recent study [73] show similarities. The overall average radiation induced BWS was shown experimentally to be 151.6 pm for the first irradiation stage. When the samples were irradiated for a second time, the effects of pre-irradiation were evident. There was a marked reduction in the overall average BWS, dropping to 88.3pm, with the lowest recording of 60 pm occurring in the Ge-doped fibre. Therefore, we can conclude that FBGs exposed to pre-irradiation again result in a lower BWS compared to no pre-irradiation. Pre-irradiation is a possibility for producing radiation hard FBG sensors. Radiation hard sensors due to pre-irradiation seem to be less sensitive, but are however more stable during irradiation. A sample of the results from the two irradiation stages [73] are shown in Table 3.3 and Table 3.4. It shows the BWS in relation to the accumulated dose during the first irradiation and second irradiation stages. The reduction in the second stage can definitely be seen. The large reduction due to radiation hardening from pre-irradiation compared to the previous studies mentioned are more likely due to the larger accumulated dose.

<table>
<thead>
<tr>
<th>Fibre Type</th>
<th>Base Wavelength(nm)</th>
<th>Final Wavelength(nm)</th>
<th>Bragg Peak Shift (pm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+H</td>
<td>1555.060</td>
<td>1555.255</td>
<td>195±12</td>
</tr>
<tr>
<td>Ge + H</td>
<td>1544.950</td>
<td>1545.115</td>
<td>165±12</td>
</tr>
<tr>
<td>Ge</td>
<td>1544.755</td>
<td>1544.920</td>
<td>165±12</td>
</tr>
</tbody>
</table>
Table 3.4: Bragg wavelength shift, for an additional dose of 196.4 kGy (total 403.2 kGy) [73]

<table>
<thead>
<tr>
<th>Fibre Type</th>
<th>Initial Wavelength (nm)</th>
<th>Final Wavelength (nm)</th>
<th>Bragg Peak Shift (pm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+H</td>
<td>1555.210</td>
<td>1555.345</td>
<td>135±11</td>
</tr>
<tr>
<td>Ge + H</td>
<td>1545.070</td>
<td>1545.175</td>
<td>105±11</td>
</tr>
<tr>
<td>Ge</td>
<td>1544.890</td>
<td>1544.980</td>
<td>90±11</td>
</tr>
</tbody>
</table>

The results discussed are a good representation of pre-radiation effects in conjunction with hydrogen loading on FBGs and is relevant to the experimental regime that is used and discussed in Chapter 4.

3.11 Radiation Induced (RI) BWS and Recovery during Irradiation

Significant research has been performed investigating the effects of radiation on standard SMF-FBGs. The main focus has been the development of radiation resistant FBGs for use in nuclear environments for temperature and strain measurement applications [1]. Recently however, FBGs have been investigated as possible high dose and low dose radiation sensors [10].

It has been demonstrated that high dose gamma irradiation causes a shift in the Bragg wavelength. As the BWS increases with radiation dose, we can make use of the radiation sensitivity of FBGs in the area of dosimetry [74]. The current subjects of conjecture are: what mechanism causes this shift, and does the radiation permanently damage the grating? Most studies have observed a cumulative radiation induced (RI) BWS towards the red, after varied irradiation periods, dose rates and accumulated dose. Also reported is the BWS whilst in recovery mode i.e. the removal of an FBG from a radiation field [75] and whether or not the shift recovers to the original base wavelength. What is not reported however, are the effects on the recovery BWS over very small time periods after three consecutive irradiation periods. The premise of this research is to report the effects on the BWS during three irradiation periods followed by reduced recovery times. This is important as for realtime dosimetry under actual working environments there are no actual time restraints (as far as recovery is concerned) when current dosimeters have been used.

Various studies with relaxation periods after irradiation ranging from e.g. 12 hrs [60], 80 days [71], 80 days[72], 24 hrs [76], 100 hrs [31], 12 hrs [11], 60 hrs [77], 200 hrs [78],
60 hrs [79], 44 days [50], 30 hrs [123] have been published. As most studies vary according to the type of gratings, dopant used, and fibre coating, it is hard to make a direct comparison. For this review, the most applicable studies which are relevant to this thesis are included, focusing on irradiation times, recovery times and dosage only. This will give an understanding and lay the foundations in relation to the data/results presented for comparative purposes in the upcoming chapters. As discussed earlier, the most common grating used is a Type I-FBG generated by a spatially modulated UV intensity.

Irradiation studies on FBGs written in various types of fibre material have shown that the BWS at the beginning of irradiation generally results in a small but rapid increase of 20 to 100 pm, depending on the fibre and photosensitization, with saturation occurring after a dose of 80 kGy [80]. Figure 3.22 shows data demonstrating the BWS shift of a Type I SMF-28 hydrogenated germanosilicate FBG. The FBGs were manufactured using a phase mask and 244nm UV illumination. The gamma dose rate was 1.63 kGy/hr with an accumulated dose of 540 kGy reached after 13.8 days. The RI-BWS was monitored during the irradiation stage and through two recovery phases, the initial of 4 days post recovery and then 40 days recovery[80]. The results of this study concluded that although there was a rapid increase in the Bragg wavelength up to 26 pm for day one, the gratings showed insignificant recovery through the initial 4 day period, and no further recovery was noted even after 40 days.

![Figure 3.22: Bragg wavelength shift of type 1 FBGs in H2 loaded germanosilicate fibre before, during and after irradiation [80].](image-url)
A study that is closely linked to the accumulated dosage used in this research was performed by Gusarov et al.[76] looking at the stabilization effects of FBGs against gamma irradiation. A total dose of 50 kGy was achieved over 120 hours at a dose rate of 400 Gy/h, followed by 24 hours of recovery. Type I gratings in standard SMF-28 with hydrogen loading were used. It is known that the RI-BWS saturates at varying levels, and post recovery has been observed but this recovery is incomplete. To address this, a pre-gamma irradiation regime was used of an accumulated dose of 145 kGy at a dose rate of 1.80 Gy/s to potentially radiation harden the FBGs [76]. As seen in Figure 3.23 a BWS is observed in all FBGS, again there is a rapid increase during irradiation up until 140 hours. At this point saturation is beginning. After this a 24 hr recovery period begins and, as can be seen, there again is only a partial recovery.

![Figure 3.23: Change of the Bragg wavelength under radiation for Type I gratings written in the hydrogen-loaded SMF28 fibre [76]](image)

When comparing the RI-BWS, hydrogen loaded fibre consistently show large red shifts when compared with FBGs written in untreated photosensitive Ge–fibre [81]. An example of the comparative RI-BWS and also saturation behaviour between Hydrogen loaded and Ge-doped fibre after an accumulated gamma dose of 1500 kGy reached, is shown in Figure 3.24. It can be seen clearly that the magnitude of the shift is dependent on the chemical composition of the FBG. Also it shows that saturation occurs no matter the fabrication method.
3.12 Coating and Recooating influence on FBGs

Most often when inscribing FBGs, especially with UV lasers, the coating (usually acrylate) of the fibre is removed and then re-applied after the fabrication [82,83]. This is known as being inscribed in bare fibre. This also mainly applies to draw tower gratings written before applying the coating. A few studies have shown that the coating type must be taken into account when correctly interpreting irradiation effects in FBGs [83]. The effect on the RI-BWS and the response of Type I gratings during gamma irradiation is reported by Gusarov et al. [82] and Blanchet et al.[83], and also by Curras et al.(during proton irradiations) [84]. Overall the studies show that the acrylate coated FBGs produce a larger RI-BWS than the bare gratings. The recoating has been shown to produce a slightly higher RI-BWS of approximately 5 pm than bare gratings after 40 kGy of gamma irradiation [82].

The Gusarov et al. [82] study used draw tower gratings before applying 3 differing types of coatings: polymide, acrylate, and ormoecer. A fourth mechanically stripped (removed) ormoecer coated FBG was used for a comparison. The FBGs were subjected to gamma irradiation to an accumulated dose of 40 kGy at a dose rate of 400 Gy/h. The gratings written in the stripped fibre showed the lowest sensitivity whilst the ormoecer coated fibre showed the highest BWS under irradiation as shown below in Figure 3.25.
The various RI-BWS between the FBGs highlight that the coating must be taken into account to allow the correct interpretation of the radiation sensitivity.

Figure 3.25: Change of the BWS after 40 kGY. The coating type: 1 – acrylate, 2 – polyimide, 3 – ormocer, 4 -removed ormocer coating[82].

The study by Curras et al. [84] also showed a clear dependence of the radiation sensitivity on the coating. Again Type I gratings were used that were recoated with acrylate and comparisons were made with polyimide and ormocer coated draw type gratings after proton irradiation. With regards to RIA they reported that the Type I FBG sensor with the acrylate re-coating after irradiation produced a slightly attenuated peak compared to ormocer coated Type I FBGs, as shown in Figures 3.26 and 3.27 [84]. The behaviour exhibited by the acrylate coated FBGs shows that although they are only slightly less radiation sensitive, they are by far the best behaved when attenuation is considered.

Figure 3.26: Bragg peak from the acrylate coated Type I FBG sensor 111 before and after the irradiation (slightly attenuated peak) [84].
Further to the studies previously mentioned, the effects of resolving the cross sensitivity of FBGs with different polymeric coatings was examined in relation to axial strain [86]. The study by Ping Lu et.al. [85] used STD-SMF FBGs which were recoated with acrylate. They found compared with other forms of coating such as polymide, the acrylate coating possesses a larger thermal coefficient which induces a larger change of Bragg wavelength shift under the same axial strain.

3.13 Radiation Effects in Photonic Crystal Fibre

New technology has emerged in the form of Photonic Crystal fibre (PCF) also known as micro-structured fibre (MSF), micro-structured optical fibre (MOF) or Holey fibre. Currently there are two different types of commercialized PCFs in use today; solid core PCF and hollow air core PCF. Normal hollow core PCF has shown a lower attenuation increase than that of conventional SMF during gamma exposure, up to at least 30 times lower after a gamma dose of 10 kGy [86], as shown in Figure 3.28. However, overall application of hollow air core PCFs, is restricted due to limitations with regards to splicing with other conventional fibres and high costs involved [87]. Some advantages of solid core PCFs over the hollow core variety include being easy to handle, and also exhibiting lower attenuation (after splicing), making them an ideal candidate for this study.
Figure 3.28: Attenuation increase comparison during gamma irradiation of Hollow core PCF and standard Ge-doped SMF [86].

Most solid core PCFs are made with pure silica, which is known to be radiation hardened when exposed to gamma irradiation. Therefore, the pure silica PCF-FBGs make them a possible candidate for sensors in radiation dosimetry. The wave guiding properties of this form of optical fibre is obtained, not from varying the glass composition through doping, but from the arrangement of closely spaced tiny holes surrounding a hollow or solid core, which run through the length of the fibre, as shown in Figure 3.29 and Figure 3.30.

Figure 3.29: (a) Example of hollow core PCF (b) Detail of core region [88].

Figure 3.30: PCF with solid core (all silica) surrounded by air channels [88].
As previously mentioned, in standard fibre, light is contained in the core by internal reflection, and to obtain a higher refractive index, doping of the fibre core is needed. The problem with doping is that it increases attenuation. However, in PCF with a solid or hollow core, results indicate that attenuation loss is lower than conventional fibre [88]. Temperature sensitivity is also a governing factor in standard optical fibre in relation to optical fibre sensors. PCFs are made of glass with a uniform composition in the entire cross section. Therefore, there is no thermal stress in PCFs induced by the difference in thermal expansion coefficients between the core and the cladding [90]. This eliminates the consideration of discriminating the temperature sensitivity from other sensitivities, when using PCFs as sensors, compared to standard fibres [90]. The studies and effects of radiation on PCF-FBGs is limited, which is why they have been incorporated in this study.

One study reported the behaviour of a new type of MSF, known as a random hole optical fibre (RHOF), shown in Figure 3.31, under gamma irradiation and their potential use in radiation sensing [91]. In this fibre, thousands of holes that surround the pure silica core are both random in size and location compared to PCF, which have ordered holes occurring in a uniform or regular pattern [91].

The study compared the RHOF with standard SMF (single mode), MMF (multi mode), and pure silica fibre (PSC). Measurements were taken of the radiation induced absorption (RIA) centred at 1550 nm, under a high intensity gamma ray field (4x10^4 rad/hr). The RIA of the RHOF was found to be much lower than standard fibres. RIA of the fibres for the total duration of the experiment inclusive of recovery, is shown in Figure 3.32. The
results indicate that the RHOF have a superior recovery time compared to the other fibres tested [91]. The mechanism for this is not fully understood. The negative RIA represents an improvement in the transmission of the optical signal [91].

One of the first studies on the sensitivity of PCF under gamma irradiation was reported by S. Girard et al. [92]. In particular, they focused on the RIA levels of a PCF at 1.55 \( \mu \text{m} \) with a pure silica core and a cladding made of air holes embedded within a silica background [92]. Optical fibres with pure silica core and fluorine doped cladding are known to be the most radiation hardening optical fibres under gamma irradiation [94]. This is achieved by the absence of core dopants. The tested PCF were irradiated at a dose rate (\( \sim 0.1 \text{ Gy/s} \)) and accumulated dose of (\( \sim 150 \text{ Gy} \)). At the end of the irradiation period, the PCF shows an induced loss of 86 dB/km, as shown in Figure 3.33. This value is high when compared to a 1 dB/km attenuation with fibre made with a pure silica core and fluorine doped cladding. A common germanium doped core fibre without phosphorus in the cladding reaches 4 dB/km. After the recovery phase is complete, shown in Figure 3.33, it can be seen that the losses after irradiation are similar in the pure silica core and in the PCF, and occurs faster than the germanium doped fibres [92]. In summary the gamma induced attenuation in PCF showed high loss at the end of irradiation but recovers more rapidly than germanium doped core fibres.

![Figure 3.32: RIA of sample fibres for total duration showing behaviour under and post irradiation recovery time [91].](image-url)
A very recent study on gamma irradiation effects in pure-silica core photonic crystal fibre by Wei Cai et al [87], has shown more encouraging results with regards to RIA. With the improvement in fabrication methods, the RIA diminished from 27.7 dB/km to 3.0 dB/km. A total dose of 500 Gy and dose rate of 2.38 Gy/min was achieved. It must be emphasized that there is no literature available reporting on the BWS of PCF-FBGs during gamma irradiation. The most similar comparative research however has been reported on the BWS between standard Ge-doped FBGs and pure silica core FBGs by Y.Xu et.al. [94]. During one irradiation stage, a maximum of 180 kGy was achieved at a high dose rate of 10.15 kGy/hour. The results showed that the pure silica core FBGs BWS saturated at 15 kGy after reaching approximately 40pm, with the Ge-doped fibre BWS continuing to increase, as shown in Figure 3.34. The pure silica core FBGs in this instance exhibit strong radiation resistance compared to standard Ge-doped FBGs. This is in keeping with reported research that agree that high purity silica core fibers are less radiation sensitive than Ge-doped FBGs [95]. The studies mentioned are relevant to the experimental results achieved and reported in Chapter 5 as the PCF-FBGs used throughout contain a pure silica core.
3.14 Colour Centre Defects (E’ Centres)

A study [115] has found that, the presence of defect centres can be reduced by pre-irradiation and thermal annealing. In Table 3.5 there are three groups, initial (not irradiated or annealed), irradiated at 20kGy, and irradiated at 40kGy at room temperature. The irradiated samples (by gamma rays) were annealed at different temperatures; 300°C, 500°C, 700°C, 900°C, 1100°C, 1300°C for one hour. Table 3.5 shows that when the dose increases from 20 to 40 kGy, the E’ centre concentrations increase [115].

Table 3.5 E’ Concentration Increased With Dose Increase [115].

<table>
<thead>
<tr>
<th>samples</th>
<th>initial</th>
<th>irradiated (20kGy)</th>
<th>irradiated (40kGy)</th>
</tr>
</thead>
<tbody>
<tr>
<td>E’ concentrations</td>
<td>1.84×10^14 cm(^{-3})</td>
<td>1.55×10^16 cm(^{-3})</td>
<td>3.55×10^16 cm(^{-3})</td>
</tr>
</tbody>
</table>

A comparative study was completed using silica optical fibre showing similar results concerning E’ centre concentration and thermal annealing [34]. It shows that the E’ concentration of optical fibre increases with an increased dose rate, shown in Figure 3.35. Samples were irradiated by gamma rays to a dose of 50 kGy. The increase shows a near
linear trend [34]. The effects of thermal annealing involved samples being irradiated by gamma rays to a dose of 20 kGy, then annealed at different temperatures for 10 minutes respectively. When the samples cool to room temperature, the $E'$ concentration was measured by ESR (electron spin resonance and spectrophotometer). The $E'$ concentration at 25 °C was $1.55 \times 10^{16}$ cm$^{-3}$ and decreased to $3.45 \times 10^{14}$ cm$^{-3}$ at 300°C [34]. There are varying methods to examine the damage in optical fibres after being exposed to gamma irradiation. The most common methods are electron spin resonance (ESR), scanning electron microscopy, and optical time domain reflectometry (ODTR). For this study however, SAXS and XRD analysis is used to determine any identifiable structural changes pre and post irradiation in relation to particle concentration, peak intensity, peak broadening, and scattering patterns. This combination of analysis will help to circumscribe and isolate the primary cause of a BWS. The best known defect to date is the $E'$ centre which are found in silica optical fibre. After being exposed to gamma irradiation, studies have shown through ESR analysis there that is a marked increase in the formation of these defect centres [34].

![Figure 3.35: Increase of $E'$ centre with increase of accumulated dose [34].](image)

An additional study by L.Wenyun et al.[42] measured spectra of two different batches of silica optical fibre pre and post irradiation. Groups 1 and Group 2 were subject to 50 kGy of gamma irradiation. The before and after ESR spectra of the two groups are shown in Figure 3.38. The initial sample of group 1 in Figure 3.36 (a) indicates a weak $E'$ centre
before irradiation (red line) whilst Group 2 does not have any ESR signals. This indicates group 2 is more stable than group 1 [42]. The ESR spectra in both groups has the same g value as the $E'$ centre characterized by $g = 2.0006$. The observable ESR signal is therefore attributed to $E'$ colour centres [42].

![Figure 3.36: ESR spectra of $E'$ centres irradiated with 50 kGy (black line) and the initial (non-irradiated red line). (a) Group 1, (b) Group 2[42]](image)

3.15 Bragg wavelength shift towards shorter wavelengths

Finally, an interesting phenomena which must be mentioned in regard to the radiation induced Bragg wavelength is what is known as a ‘blue’ shift during irradiation. A radiation induced shift to shorter wavelengths was observed in type I gratings by Gusarov et al.[78]. The gratings were written in photosensitive fibre doped with ~ 8 mol% of GeO$_2$. The usual tendency observed in FBGs is that after the end of radiation exposure the long wavelength shifts start to decrease[78]. What was observed however, was a short wavelength shift during the irradiation stage after 120 kGy (~1.0 kGy/hr over 120hrs). They proposed two types of defects may be responsible for the short wavelength shift. One type is responsible for the refractive index decrease during irradiation, while the creation of defects of another type results in the index decrease during annealing.
A recent study by Faustov et al. also reported a blue shift of ~20pm at a total dose of ~100 kGy as shown in Figure 3.37 [116]. This was achieved by gratings written into pure silica core fibres. Such behaviour is characteristic of gratings inscribed in pure silica fibres, where the ionising radiation induced formation of dopant related colour centres is unlikely [116]. The observed blue shift was most likely due to structural changes in the silica glass: a decrease in its density, which leads to a decrease in its refractive index [116]. The absence of a backward shift after irradiation also indicates that the glass matrix undergoes persistent changes [116]. The concurrence of the two types of defects may be a way to obtain high stability of the gratings under irradiation [79].

![Graph](image)

Figure 3.37: Gamma radiation–induced blue shift of resonance peaks; (b) Transmission and (c) reflection spectra of the gratings before (black lines) and after (grey lines) irradiation.[116]

### 3.16 Negative compaction in optical fibre Bragg gratings

As stated previously, during the irradiation phase of optical FBGs, there are three resultant macroscopic phenomena which occur: RIA [117], RIE, and RIC (radiation induced compaction) of the silica matrix [118,119]. The RIA and RIC induce changes in the effective refractive index through the Lorentz-Lorenz and Kramers - Kroning equations. Gamma radiation importantly can also change the FBG period (ΔΛ) [83] and will often lead to compaction (a fractional increase in the density of silica ρ) which alters
both mechanical and optical properties [81]. The dependence of compaction with radiation dose \( D \) in vitreous silica has been found[81,170], and follows a power law:

\[
\frac{\Delta \rho}{\rho} = AD^c
\]  

(3.1)

Where \( A \) is a dose constant and \( c = 2/3 \) for gamma radiation, and \( c = 1 \) for neutron radiation. A negative compaction (dilation) was first observed by Primak et al.[118] in vitreous silica, who found that the \( c = 2/3 \) power law for gamma induced effects depend on the intrinsic glass strain [81]. The study found that compaction or swelling leading to density changes in the glass is dependent on the silica type either amorphous or crystalline [163]. Under fast neutron-gamma irradiation both silica amorphous and crystal also transform towards the “metamict phase” with about a 3% density increase compared to the initial state.

3.17 Concluding Remarks

The literature review reported is closely linked to the objectives of this study. Reported is the manufacturing process, the similar FBGs used, and studies using similar accumulated dose and dose rates. There is however a major gap in relation to PCF-FBGs under similar regimes. The pre-irradiation effects and response to gamma exposure by standard FBGs have also been examined.
CHAPTER 4
RESEARCH METHODOLOGY

This chapter explains the experimental setup, method, and materials used. It starts with the Gamma Irradiation facility, description of FBG samples used during each irradiation phase, and then continues with the actual Bragg wavelength monitoring set up. Also covered, is the method throughout each experiment and spectroscopic techniques used to identify any permanent consistent structural anomalies that affect the structural integrity of the fibre. Overall, there are three irradiation experiments (sessions) with similar regimes. They were conducted over three different time periods due to the availability of the Gamma irradiation facility and the success of obtaining research grants. The regime is designed with continuity in mind, to highlight the effects of pre-irradiation, and recovery aspects between the sample sets; commercial standard FBGS and new generation PCF-FBGs. All experiments were performed as a result of successful AINSE Research Awards: ALNGRA13532 and ALNGRA15540 (2015-R2). Results will follow in Chapter 5.

4.1 Gamma Irradiation Facility

Gamma irradiation was conducted at (ANSTO), the Australian Nuclear Science and Technology Organisation using the Gamma Technology Research Irradiator (GATRI). The same Cobalt-60 radiation source, as shown in Figure 4.1, was used for each irradiation period throughout the three experiments. The cobalt-60 radioactive source is raised out of the water storage pool normally used to shield gamma radiation and items then irradiated either over several minutes or hours depending on the specific accumulated dose required. ANSTO was able to accurately control and monitor the radiation dose throughout. Dose mapping was achieved by placing Ceric Cerous type dosimeters in cylindrical polyethylene holders in an empty polystyrene box. This established the average dose rate which determined the dose absorbed by the FBGs throughout the exposure stages. The precise irradiation services provided by GATRI are unique in Australia. The overall uncertainty associated with an individual dosimeter reading includes both the uncertainty of calibration of the batch of dosimeters and the uncertainty due to variation within the batch and was calculated to be 3%. This expanded uncertainty is based on the standard uncertainty multiplied by a coverage factor of two,
providing a level of confidence of approximately 95%. The uncertainty evaluation has been carried out in accordance with the ‘ISO Guide to the Expression of Uncertainty Measurement’ [120].

Figure 4.1: GATRI’s cobalt-60 radioactive source [121]

4.2 Tested Samples

The purpose of this Thesis is to compare the radiation response in FBGs written in standard off the shelf commercial single mode fibre and new generation PCF-FBGs.

Standard Fibre with FBGs (purchased from Alxenses)

- Std-SMF28H, standard SMF-28 optical fibre with Hydrogen loading.
- Std-Ge, standard Germanium doped fibre without Hydrogen loading.
- Std-GeH, standard Germanium doped fibre with Hydrogen loading

New Generation Fibre Photonic crystal fibre (manufactured by BlazePhotonic (NKT) purchased from Thorlabs)

- ESM 12-PCF, endlessly single mode pure silica photonic crystal fibre (ESM12-01)

The FBG inscription and hydrogen loading in the purchased endlessly single mode (ESM) pure silica PCF was performed through a collaborative arrangement with the University of Sydney, interdisciplinary Photonics Laboratories (iPL), using their FBG writing facility. The results obtained earned a conference presentation and publication.
4.3 FBG Sample Specifications

*Standard Fibre with FBGs:* Each of the FBGs were produced with centre wavelengths of 1539.76 nm (SMF28H), 1544.88 nm (Ge), 1549.98 nm (Ge+H) and 1555.02 nm (SMF28H) for the purpose of spectral separation. Each FBG had a reflectivity of > 90%. The length of each FBG was 10 mm with a bandwidth @-3dB: < 0.3 nm. The hydrogen loading for each fiber was completed at 100bar for seven days. Post-fab annealing to remove the unstable index and also to stimulate out-diffusion of remaining hydrogen assumed completed by manufacturer [76]. The Ge content, as stated by the manufacturer, was approximately in the range of 9-15 mol.%. The centre wavelength tolerances for each FBG were given as ±0.5 nm. The total fibre length for each sample was 30 cm, with the FBG centrally located. All FBGs were written by low-energy UV irradiation. The fibre core diameter is 9 μm whilst the cladding is 125 μm [see Appendix C]. All four optical fibres were coated in Acrylate whilst the FBGs were recoated with Acrylate. After purchasing the FBGs the coating was not removed due to fragility, time constraints, and the scheduling of ANSTO. Could not risk any damage to occur to the FBGs.

*New Generation Fibre (ESM12-01 PCF) and FBG inscription:* Each of the FBGs were produced with centre wavelengths of 1532.860 nm, 1540.806 nm, 1541.020 nm. The endlessly single mode photonic crystal fibre (ESM12-01) as shown in Figure 4.2, has a hexagonal distribution of 54 holes within a ϕ = 125 μm diameter silica fiber, inside a holey region diameter of ϕ = 57.4 μm as shown in Figure 4.2. Additional characteristics include a pitch between holes of Λ = 8.0 μm; a core diameter of ϕ = 12.0 μm, hole diameter relative to pitch of Λ/ϕ = 0.46, and a coating diameter of ϕ = 220 μm (single layer acrylate).

![Figure 4.2: Cross section of Endlessly single mode photonic crystal fiber (ESM12-01) from BlazePhotonics (NKT) highlighting hexagonal distribution[160].](image-url)
The FBGs were inscribed into the PCF using 193 nm pulsed UV radiation of an ArF laser [112]. The inscription parameters were: fluence per pulse, $f_{\text{pulse}} = 248 \text{ mJ/cm}^2$; cumulative fluence, $f_{\text{cum}} = 8.9 \text{ kJ/cm}^2$; repetition rate, $RR = 30 \text{ Hz}$; pulse duration, $\tau_w = 15 \text{ ns}$. Prior to FBG inscription: short ($L = 10-15 \text{ cm}$) sections of PCF were pigtailed at each end with standard SMF-28 fibre using a tailored fusion splicing technique employing a Fittel s175 model Arc fusion splicer shown in Figure 4.3. The pig-tailing of PCF samples served two purposes: it allowed for the PCF to be easily connected to a light source and spectrometer (for FBG interrogation) using standard FC/APC connectors and, more critically, it prevented the rapid out-diffusion of hydrogen after hydrogen-loading.

![Figure 4.3: Actual Fittel s175 model Arc fusion splicer used.](image)

The proximity of the large number of air holes to the PCF core poses a real problem as hydrogen can rapidly out-diffuse through these holes in a matter of minutes [122]; sealing each facet through the splicing of solid fibre therefore circumvents this problem. Using the arc fusion splicer, the fibers are joined together and fused by applying a weak arc power (arc power = 1; arc duration = 250 ms; push distance = 13 $\mu$m). Next the splice strength was intensified by increasing the arc power (arc power =91; arc duration = 250ms; push distance = 0 $\mu$m). Due to mode mismatch between fibers, splice losses were approximately $\alpha = (0.7–0.8) \text{ dB}$. After splicing, hydrogen ($H_2$) loading commenced at pressure $P = 180 \text{ atm}$ and temperature $T = 80 \text{ °C}$ for a time $t = 7 \text{ days}$. The FBG inscriptions commenced immediately after the unloading of fibre samples from the hydrogen vessel. Time taken for each inscription amounted to $\sim 30\text{mins}$. Post thermal treatment was not completed due to the fragility of splice points and time constraints.
However, due to the time interval between the FBG inscription and the first irradiation of a couple of months, hydrogen out-diffusion would have occurred, simulating a form of post-thermal treatment [174]. In this work, we have used 193 nm with silica PCF unconstrained by a cladding producing $R = 6$ dB, FWHM $\sim 80$ pm for a grating $L = 1$ cm, producing a coupling strength of 6 dB/cm. The resultant real time (OSA image) of the reflected spectrum at $\sim 10$ min and final $\sim 30$ min during the inscription process, and graphical characterised reflected spectrum is shown in Figure 4.4.

![Reflection spectra OSA image after 10 mins (top left image) and on completion after $\sim 30$ mins (top right image). Graphical characterized PCF-FBG reflection spectrum (bottom image)](image)

**Figure 4.4:** Reflection spectra OSA image after 10 mins (top left image) and on completion after $\sim 30$ mins (top right image). Graphical characterized PCF-FBG reflection spectrum (bottom image)

### 4.4 Optical Measurement Setup

When determining the Bragg wavelength shift as a function of accumulated dose/time, and as a function of post irradiation relaxation time, the reflected spectra was
recorded using a Agilent86142A Optical Spectrum Analyzer (OSA). The OSA resolution is accurate to within ±0.005 nm (5pm) for wavelength recording. A single line containing up to 4 FBGs in series was connected using FC/APC connectors attached to a 10m patch cable, which was threaded through an access port in the irradiation facility shield. The light emitted from a superluminescent diode (SLD) (Dense Light DL-BZ1-SC5403A) light source with a centre wavelength of 1550 nm, bandwidth of 100 nm, and 25 mW optical power was launched into the fibre. The FBGs will each reflect a specific Bragg wavelength. Reflected spectra as a function of gamma irradiation was then measured via a 3-port circulator using the OSA connected by a GPIB/USB Agilent interface to a PC. A schematic of the experimental setup is shown in Figure 4.5. The only variation of this setup is the number of FBGs in the GATRI chamber. A Matlab data acquisition program (see Appendix A and B) was written to control the OSA and measurement process, and record the results automatically.

![Figure 4.5: Experimental set-up for measuring reflective spectra](image)

To secure the FBGs firmly in position, a polystyrene container was used as shown in Figure 4.6. Before being placed in the chamber, the FBGs were placed between two 5mm thick polyethylene (HDPE) sheets, as seen in Figure 4.7, to improve electron equilibrium through the fibres and to potentially maximise the penetration of the dose and reduce any
fluctuations during irradiation. An additional strip of polyethylene was added covering the FBG–SMFH (base wavelength 1555.02 nm) for comparative purposes.

![Figure 4.6: The polystyrene box with four FBGs set up in series.](image1)

![Figure 4.7: The polystyrene box with FBGs held in place with polyethylene sheets.](image2)

To determine the effect of temperature on the FBGs whilst under irradiation and during the relaxation period (non–irradiation/ recovery period), a temperature sensor (thermocouple) was placed in the side of the sealed polystyrene container as shown in Figure 4.8. This allowed the monitoring of the explicit temperature within close proximity to the FBGs during irradiation and relaxation, compared to the irradiation chamber temperature. The type-T thermocouple with digital display is regularly calibrated by
ANSTO using test equipment MPE156 and was current at the time of experiment. The container was placed on the rig within close proximity to the gamma source in the irradiation cell for maximum exposure.

![Image](image.png)

**Figure 4.8:** The polystyrene box with the thermocouple placed on the bottom right red arrow, and placed inside the GATRI chamber.

### 4.5 Experimental Method

As mentioned previously there were three irradiation sessions. Each session had identical setups as outlined in the Optical Measurement setup section. The three sessions comprised of 3 irradiation periods and 3 relaxation/recovery stages. Prior to the commencement of irradiation characterisation measurements were performed on site at ANSTO using the exact setup as shown in Figure 4.5. Baseline wavelength and reflected spectra were recorded after they had been placed and secured in the irradiation cell via the OSA. Irradiation commencement time was noted, along with the starting wavelength acquired via the OSA. The regimes/method for each session are as follows:
Irradiation Session 1: STD-FBGs; *SMF28H*(x2), *GeH, Ge*, (base wavelengths, 1539.76, 1544.88, 1549.98, 1555.02nm)

Each irradiation period reached an exact accumulated dose of 66.5 kGy over a time period of 21.3 hrs, followed by a recovery periods, two of which were 3.5hrs duration and one 2.0 hrs relaxation (no irradiation) as shown in Table 4.1. The small relaxation periods is intentional to achieve as close as possible, real time conditions of a radiation dosimeter.

Most presented research incorporate large recovery times ranging from 12hrs up to 80days [11,31,50,60,71,76,77,78,79,123]. To the best of my knowledge it is also the first time that FBGs have been examined through three consecutive irradiation periods with the exact same dose rate, accumulated dose and very limited recovery with FBGs produced by the same manufacturer.

### Table 4.1: Session 1, experimental regime

<table>
<thead>
<tr>
<th>Radiation 1 21.3 hrs</th>
<th>Recovery 1 3.5 hrs</th>
<th>Radiation 2 21.3 hrs</th>
<th>Recovery 2 3.5 hrs</th>
<th>Radiation 3 21.3 hrs</th>
<th>Recovery 3 2.0 hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>66.5 kGy</td>
<td>3.5 hrs</td>
<td>66.5 kGy</td>
<td>3.5hrs</td>
<td>66.5 kGy</td>
<td>2.0 hrs</td>
</tr>
</tbody>
</table>

During the irradiation process measurements were recorded every 30 minutes. The relaxation recordings were recorded every 10minutes. A total accumulated dose of 199.5 kGy over 63.9hrs of exposure was achieved with a dose rate of 3.12 kGy/ hr. The different responses to exposure from the gamma irradiation between the Germanium (Ge) doped optical fibres and standard SMF-28 fibre with hydrogen loading are discussed in the results section. At the end of irradiation the temperature was noted and then throughout the relaxation period the temperature was periodically recorded via the digital display every 5 mins. The average irradiation temperature inside the chamber throughout irradiation session one was 22.8 °C.

Irradiation session 2: PCF-FBG; *ESM 12-PCF* (base wavelength, 1540.806nm)

Each irradiation period reached an exact accumulated dose of 50.6 kGy over a time period of 21.0 hrs. followed by 3.0 hrs of relaxation (no irradiation) as shown in Table 4.2. The dose rate equated to 2.41 kGy/ hr. During the irradiation process and relaxation period, measurements were taken and recorded every 30 minutes. A total accumulated
dose of 151.8 kGy over 63 hrs of exposure was achieved. The responses to exposure from the gamma irradiation are discussed in the results section. The irradiation temperature range inside the chamber ranged between 21.5 °C to 23.7 °C.

Table 4.2: Session 2, experimental regime

<table>
<thead>
<tr>
<th>Radiation 1 21.0 hrs</th>
<th>Recovery 1 3.0 hrs</th>
<th>Radiation 2 21.0 hrs</th>
<th>Recovery 2 3.0 hrs</th>
<th>Radiation 3 21.0 hrs</th>
<th>Recovery 3 3.0 hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>50.6 kGy</td>
<td>3.0 hrs</td>
<td>50.6 kGy</td>
<td>3.0 hrs</td>
<td>50.6 kGy</td>
<td>3.0 hrs</td>
</tr>
</tbody>
</table>

Irradiation session 3: PCF-FBGs; *ESM 12-PCF* (base wavelengths 1532.860 nm, 1541.020 nm)

Each period reached an exact accumulated dose of 49.35 kGy over a time period of 21.0 hrs. followed by 3.0 hrs of relaxation (no irradiation) as shown in Table 4.3. The dose rate equated to 2.35 kGy/hr. During the irradiation process and relaxation period measurements were taken and recorded every 30 minutes. A total accumulated dose of 148.05 kGy over 63 hrs of exposure was achieved. The irradiation temperature range inside the chamber for session three ranged between 20.6 and 21.6 °C. The responses to exposure from the gamma irradiation are discussed in the results section.

Table 4.3: Session 3, experimental regime

<table>
<thead>
<tr>
<th>Radiation 1 21.0 hrs</th>
<th>Recovery 1 3.0 hrs</th>
<th>Radiation 2 21.0 hrs</th>
<th>Recovery 2 3.0 hrs</th>
<th>Radiation 3 21.0 hrs</th>
<th>Recovery 3 3.0 hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>49.35 kGy</td>
<td>3.0 hrs</td>
<td>49.35 kGy</td>
<td>3.0 hrs</td>
<td>49.35 kGy</td>
<td>3.0 hrs</td>
</tr>
</tbody>
</table>

4.6 Pre and Post-irradiation analysis spectroscopic techniques

Included are three techniques to help quantify and elucidate the permanent damage mechanism caused by gamma irradiation on FBGs. Physical examination of the fibres was conducted using, SAXS (small angle X-ray scattering), XRD (X-ray diffraction) and electron spin resonance (ESR) spectroscopy. The comparison of structural defects in the silica optical fibre both pre and post irradiation are compared, using three reliable and non-destructive techniques. I believe this is the first report using SAXS and XRD analysis on FBGs, particularly PCF-FBGs pre and post irradiation. Small and wide-angle X-ray scattering are well established standard tools in materials research and recently are gaining popularity due to the complimentary information obtained [124,125].

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4.6.1 XRD: X-ray Diffraction

X-ray Diffraction is a crystallographic technique in which the resultant pattern produced by diffraction of X-rays through the closely spaced lattice of atoms in a crystal is recorded and analysed to reveal the nature of that lattice [126]. It generally leads to an understanding of the material and molecular structure of the substance. The spacing in the crystal lattice can be determined using Bragg’s law [126]. The electrons that surround the atoms, rather than the atomic nuclei themselves, are the entities which physically interact with the incoming X-ray photons [126]. Interpretation of XRD using Bragg’s law also determines the scattering angles at which peaks of strong scattered intensity occur. Normally an ideal crystalline Bragg diffraction peak is a line without width but in reality diffraction from crystal sample produces a peak with a certain width as shown in Figure 4.9. No material has a perfect crystal structure due to their finite size, causing a deviation from perfect crystallinity, which leads to broadening of the X-ray diffraction peaks [127].

An advantage of using XRD in this study is that it provides a way of estimating the crystallite size from the broadening of the XRD reflections [128]. For example if the crystallites in the sample are sufficiently small, the maxima of the diffraction pattern are broadened by an amount that is inversely proportional to the crystallite size [129].

![Figure 4.9: A standard XRD reference pattern peak lines with width and information content of an idealized diffraction pattern [172].](image)
Broadening of x-ray diffraction peaks is apparent in patterns obtained with a diffractometer, and this information can be directly quantified. However, it is important to realize that broadening of diffraction peaks arises mainly due to three factors: instrumental effects, crystallite size and lattice strain. It is well accepted that an increase in the peak intensity of diffraction peaks is related to an increase in crystallinity and the overall broadening of the peak is linked to a decrease in the average crystallite size [130]. Also, if there is a narrowing of a peak, it can imply the sample has a high degree of crystallinity [131]. The broadening is evaluated by measuring the angular width “B”, in radians, at intensity equal to half the maximum intensity (FWHM). Subtracting the instrumental effect from the obtained peak broadening, two main properties: crystallite size and lattice strain, are extracted from peak width analysis.

Crystallite size and lattice strain affect the Bragg peak by increasing the peak width and intensity, and shift the 2θ peak position accordingly [127]. The crystallite size varies as 1/cosθ and strain varies as tanθ from the peak width. The size and strain effects on peak broadening are known from the above difference of 2θ [127]. The crystallite size and lattice strain are the two main properties which can be extracted from peak width analysis. It must be emphasized that crystallite size of the particle is not the same as the particle size. XRD is primarily used to interpret average crystallite size whereas SAXS will identify the average particle size as shown in Figure 4.10.

![Figure 4.10: Crystallite size from XRD and Particle size from SAXS](image)

Size-induced and strain-induced broadenings are known by considering the peak width as a function of 2θ [127]. The crystallite size and lattice strain are estimated by using the Debye Scherrer method. The Scherrer equation [132] is usually the most effective for
crystallite sizes less than 200 nm. Constructive interference results in output x-ray signals at particular angular relationships between the incident beam, the crystal structure, and the specimen orientation. For the calculation of the inter-planar spacing between planes, Bragg’s law is used to define the value between these relationships:

\[ n\lambda = 2d \sin \theta \]  

or

\[ d = \frac{\lambda}{2\sin \theta n(n = 1)} \]  

where \( n \) is an integer representing the order or the diffraction peak, \( \lambda \) is the wavelength of the x-rays, \( d \) is the interplanar separation (d-spacing) for a particular set of crystallographic planes, and \( \theta \) is the angle between the incident x-rays and the crystallographic planes causing the diffraction and not the angle at the actual surface crystal. This is shown schematically below in Figure 4.11.

![Figure 4.11: Schematic for diffraction of Bragg's law showing relationship between wavelength of an incoming ray and the d-spacing of a diffracting crystal [126].](image)

In this study, the crystallite size and lattice strain are estimated by using the Debye Scherrer method using Jade 9 XRD analysis software. The Scherrer equation (Eqn.4.3) is usually the most effective for crystallite sizes less than 200 nm. The average crystallite size and lattice strain are the two main properties also extracted from the peak width
(FWHM) analysis. The average crystallite size $D_p$ is calculated from the XRD line broadening, where $k$ is the Scherrer shape constant (usually taken as unity), $\beta$ is the FWHM of the diffraction peak, $\theta$ is the XRD peak position;

$$D_p = \frac{k\lambda}{\beta \cos \theta} \quad (4.3)$$

When there is a shift in $\theta$, due to changes in inter-planar distance $d$, it is an indication that the lattice parameter is modifying. Along with a change in $\beta$, stresses occur within the structure leading to a change in micro-strain $\varepsilon$ [130], which is given by:

$$\varepsilon = \frac{\beta \cos \theta}{4} \quad (4.4)$$

The FBGs pre and post irradiation are characterized using a Siemens D5000 x-ray diffractometer using copper K-alpha radiation as shown in Figure 4.12. This was to determine if there was any variation in the peak position, shape, FWHM, and estimate the lattice strain and average crystallite size pre- and post- irradiation. Results are discussed in Chapter 6.

![Figure 4.12: Siemens D5000 x-ray diffractometer](image)
4.6.2 SAXS : Small angle x-ray Scattering

SAXS is an analytical non-destructive technique to determine the micro- or nano-scale structure of particle systems in terms of parameters such as averaged particle sizes, shapes, distribution, and surface-to-volume ratio [133]. The materials can be solid or liquid and they can contain solid, liquid or gaseous domains (so-called particles) of the same or another material in any combination [133]. The x-ray source can be a laboratory source or synchrotron light which provides a higher X-ray flux. The basic difference between SAXS and WAXS (wide angle X-ray scattering) is the length scale they correspond to: WAXS detects ordering of individual atoms and SAXS probes larger structures based on electron density differences (e.g. proteins in solution or pores in a solid matrix). Compared to WAXS also known as X-ray diffraction, SAXS measures in smaller angles typically between 0.1° to 10° [133].

The basis of SAXS is when x-rays irradiate a sample, the atoms within the sample scatter the incident radiation in all directions producing a background radiation which is nearly constant at small angles. Clusters of atoms inside the sample also produce additional scattering (known as excess scattering) due to the particles being made of a different material or density (allowing contrast) and are in the size range of the X-ray wavelength. The scattered x-rays are collected by an x-ray detector. The small angle scattering angle is usually defined by the scattering vector $q$ [134] as:

$$ q = \frac{4\pi}{\lambda} \sin \theta $$ (4.5)

Where $\theta$ is half of the scattering angle (in accordance with diffraction, the full scattering angle is defined as $2\theta$), and $\lambda$ is the wavelength of radiation used. The units of the scattering vector $q$ are then in reciprocal length usually in Angstroms [134]. The scattered intensity $I(q)$, is measured in terms of the scattering vector $q$ as seen in Figure 4.13 and can be written as [169]:

$$ I(q) = \frac{N}{V} \Delta \rho^2 \cdot V_{\rho}^2 \cdot P(q) \cdot S(q) $$ (4.6)
In this equation $N/V$ is the number of dispersed particles per unit volume in the sample. $\Delta \rho$ is the excess electron density which is defined as the difference between the electron density of the particles and that of the surrounding medium. $Vp$ is the volume of the particle. $P(q)$ is the form factor which characterizes the single particle scattering and $S(q)$ is the structure factor [169]. An additional factor that affects the scattered intensity $I(q)$, is the actual size of the particle i.e. larger the particle the more intensity will be detected from them. The sample volume also affects intensity linearly. Twice the illuminated sample volume will result in twice the intensity.

It must be noted that it is difficult to find any studies that have used SAXS for analysis of FBGs after gamma exposure. A study reported by Hindle et al.[135] however, concluded through the use of SAXS that femtosecond inscription of standard telecommunications glass (both silica and germanosilicate fibre preforms) is shown to induce significant mesoscopic structure. The structures produced by femtosecond radiation induced features of very small size and at two different scales [135, 136]. The smaller scale had a radius of 20 Å whilst the larger 200 Å [135]. The results are of relevance especially when comparing the SAXS analysis of this study discussed in Chapter 6. It shows that this analytical tool analysis is promising for further future studies in the area of gamma irradiation effects of FBGs.

![Figure 4.13: A typical SAXS pattern [171].](image-url)
The small angle scattering instrument at Curtin University was used in this study is a Bruker NanoStar with a high intensity gallium MetalJet X-ray source as shown in Figure 4.14. The main approach is to measure the scattering intensity patterns for pre and post irradiation and compare the results between PCF-FBGs and STD-FBGs. Results are in Chapter 6.

![Bruker Nanostar SAXS with a high intensity gallium x-ray source.](image)

**Figure 4.14: Bruker Nanostar SAXS with a high intensity gallium x-ray source.**

### 4.6.3 Electron Spin Resonance (ESR)

As mentioned previously the most common and widely used spectroscopic technique in the study of radiation induced defects in SiO$_2$ is Electron Spin Resonance (ESR) spectroscopy [137]. This non-destructive technique is used in the interpretation and measurement of microwave radiation and the energy difference between atomic and/or molecular states. This is achieved by detecting the presence of unpaired electrons from energy splitting when they are placed in a magnetic field. The spin of an electron and its corresponding magnetic moment is the foundation of ESR spectroscopy. When placed in a magnetic field, the electron has two possible spin states (spin down or spin up) resulting in two varying energies. The energy separation between the two spin states is $g\beta B_0$ where $g$ is a proportionality factor. When a microwave frequency is applied the $B_0$ magnetic field is swept with spin-flip transitions occurring when the energy separation between the two electron spin states matches the constant microwave energy [138]. When the separation in energy between the two spin states match the applied microwave radiation, a derivative shaped signal occurs seen in Figure 4.15. The $g$ value (a dimensionless quantity, called the *Lande factor*) governs the magnetic field where the signal appears.
Figure 4.15: EPR transitions occur when the energy contained in the microwave photons matches the splitting between two electron spin states. In the simplest system, this splitting as a function of the magnetic field is $g_e B_0$.

A free electron has a $g$ value ($g_e$) of 2.00231930436153. If $g$ does not equal $g_e$ the conclusion that can be drawn is that the ratio of the unpaired electron’s spin magnetic moment to its angular momentum differs from the free electron value. The area of the ESR peak is directly proportional to the number of unpaired electrons in the sample investigated, and therefore to the concentration of a sample. To calculate a change in the $g$ factor we have to determine the value of $\Delta E$, the energy between the two spin levels. When a paramagnetic sample is placed in a uniform magnetic field $B_0$, the field splits the energy of the ground state by an amount $\Delta E$, where:

$$\Delta E = g \beta B_0 = h \nu$$  \hspace{1cm} (4.7)

The value of $g$ can then be calculated from $\nu$ (in GHz) and $B_0$ (in Gauss) where $h = 6.626 \times 10^{-34}$ J·s; and $\beta = 9.274 \times 10^{-28}$ J·G$^{-1}$ using,

$$g = \frac{h \nu}{\beta B_0}$$  \hspace{1cm} (4.8)

For this thesis ESR measurements in relation to the value of $g$ are recorded by a Bruker ESP 300E Spectrometer operating at 9.45 GHz, 3G modulation and $5 \times 10^{-2}$ mW wave power as shown in Figure 4.16. Results are of a supplementary nature, to highlight pre
and post irradiation values and provide a more thorough understanding of irradiation effects. The results are shown in Appendix D.

4.7 Simulation using OptiGrating

OptiGrating 4.2.3 (released Dec 2014) is software by Optiwave [139] used for modelling integrated and fibre optical devices, and offers different options for analysing and designing typical grating assisted fibres and waveguides. It can also perform layer peeling i.e. deconvolution of the grating structure from the reflection coefficient. For this study, it is used to determine the grating behaviour/properties in relation to temperature and strain. The software allows adjustments to the fundamental properties (shape, length, apodization, index modulation) of a Bragg grating to be modified for analysis [139]. To simulate e.g. strain being produced given original wavelength (before irradiation) and new wavelength (after irradiation), specifications and parameters are entered in optigrating, including known cladding diameters (125μm:STD &PCF) and core diameters (9 μm:STD,& 12 μm:PCF) and core material properties. The refractive index of the core used is related to the base central wavelengths. As previously mentioned, the refractive index is changed in optical fibre by adding dopants such as germanium to the core. This software also allows us to define the material dispersion of the fibre core and cladding. The wavelength dependence of refractive index \((n)\) can be expressed in the Sellmeier equation parameters. The Sellmeier coefficients are stored in the Optigrating library, and when used gives a more accurate
replication of the overall profile. It uses the Sellmeier Equation as shown in equation (4.9) where the coefficients are known for material of just one doping concentration or if the concentration is unknown[139].

\[
n^2 - 1 = \frac{A_1 \times \lambda^2}{\lambda^2 - \lambda_1^2} + \frac{A_2 \times \lambda^2}{\lambda^2 - \lambda_2^2} + \frac{A_3 \times \lambda^2}{\lambda^2 - \lambda_3^2}
\] (4.9)

Where \( A_1, A_2, A_3 \) are the amplitude Sellmeier coefficients, \( \lambda \) is the wavelength, whilst \( \lambda_1, \lambda_2, \lambda_3 \) are the wavelength Sellmeier coefficients used for the simulation. The Sellmeier parameters obtained highlighting the pure silica and Ge-doped coefficients used for the reconstruction of the grating are shown in Table 4.4.

<table>
<thead>
<tr>
<th>Table 4.4 Sellmeier Parameters used for Simulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Silica</td>
</tr>
<tr>
<td>-------------------</td>
</tr>
<tr>
<td>( A_1 )</td>
</tr>
<tr>
<td>( A_2 )</td>
</tr>
<tr>
<td>( A_3 )</td>
</tr>
<tr>
<td>( \lambda_1 (\mu m) )</td>
</tr>
<tr>
<td>( \lambda_2 (\mu m) )</td>
</tr>
<tr>
<td>( \lambda_3 (\mu m) )</td>
</tr>
</tbody>
</table>

To determine the grating properties before and after irradiation, the initial grating produced had zero strain applied. This reflection spectrum profile is now the base profile. The thermo-optics parameter function is switched on to activate the reference temperature. This was kept constant at 23°C (similar to actual chamber temperatures) to overcome cross sensitivity and to isolate the effects of strain. By varying the values of micro-strain \( \mu \varepsilon \) applied only, using the uniform micro-strain option as shown in Figure 4.17 to the original wavelength grating specs in the combined reflection profile (apodization, shape, length, period), a combined profile equal to the wavelength after irradiation resulted.
To correlate the shift in Bragg wavelength with the changes to the grating period \( \Lambda \) only, the base parameters and core material properties and effective refractive index \( n_{\text{eff}} \) were kept constant as per the Optigrating library. Examples of parameters used in Optigrating are shown in Table 4.5, highlighting the base wavelength (before irradiation) and final wavelength (after irradiation) of one FBG used in the actual experiment.

### Table 4.5 FBG parameters in Optigrating

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Base ( \lambda ) (1539.76 nm)</th>
<th>Final ( \lambda ) (1539.94 nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Core Width(( \mu \text{m} ))</td>
<td>4.5</td>
<td>4.5</td>
</tr>
<tr>
<td>Core Refractive index</td>
<td>1.44921</td>
<td>1.44921</td>
</tr>
<tr>
<td>Cladding Width (( \mu \text{m} ))</td>
<td>62.5</td>
<td>62.5</td>
</tr>
<tr>
<td>Cladding refractive index</td>
<td>1.44403</td>
<td>1.44403</td>
</tr>
</tbody>
</table>

Therefore, to correlate the shift in Bragg wavelength \( \lambda \) with changes in \( n_{\text{eff}} \) and the change in grating period \( \Delta \Lambda \) we obtain:

\[
\frac{\Delta \lambda_{B}}{\lambda_{B}} = \frac{\Delta n_{\text{eff}}}{n_{\text{eff}}} + \frac{\Delta \Lambda}{\Lambda}
\]  

(4.10)

If \( n_{\text{eff}} \) is constant and therefore neglected we are left:

\[
\frac{\Delta \lambda_{B}}{\lambda_{B}} = \frac{\Delta \Lambda}{\Lambda}
\]  

(4.11)

As radiation can change the grating period as mentioned previously in Eq. 2.6, a resultant red shift of the Bragg wavelength coincides with an increase in grating period which is shown, and can be confirmed by using Optigrating, after the micro-strain added. Therefore an increase due to an increase of micro-strain that has been produced after irradiation can be obtained:

\[
\frac{\Delta \Lambda_{B}}{\Lambda} \sim \frac{\Delta \lambda_{B}}{\lambda_{B}}
\]  

(4.12)
Using the base wavelength and final wavelength from Table 4.2 we are left with:

$$\frac{\Delta \lambda_B}{\lambda} - \frac{\Delta \lambda_B}{\lambda_B} = \frac{.18 \text{nm}}{1539.76 \text{nm}} = 1.17 \times 10^{-4} = 117 \mu \varepsilon$$ \hspace{1cm} (4.13)

Therefore \(\sim 117 \mu \varepsilon\) was needed to produce the Bragg wavelength shift and combined reflection profile using the same material parameters. Results of all Optigrating profiles are in Chapter 5.

![Figure 4.17: Optigrating Fiber Bragg Grating Sensor dialog box with 117.5 micro strain (red arrow) added.](image-url)
CHAPTER 5
RESULTS OF IRRADIATION STUDIES

This chapter highlights the results obtained from three separate irradiation sessions performed at ANSTO. The data demonstrates the effects of the accumulated dose, dose rate, and temperature during both the irradiation and relaxation periods. Also included are the results of the simulation using OptiGrating software as discussed in Chapter 4. At the completion of each experiment, a summary is provided of the outcomes achieved. The final section presents a direct comparison between results of the standard set and PCF-FBGs. For easier scrutiny, the results for each session are presented in the same sequence of sub headings. Spectroscopic analysis using XRD, SAXS both pre-and post-irradiation will follow in Chapter 6.

5.1 Irradiation Session One

The results from irradiation session one are produced by the standard set of FBGs purchased from Alxenses:

- Std-SMF28H(x2), standard SMF-28 optical fibre with hydrogen loading.
- Std-Ge, standard Germanium doped fibre without hydrogen loading.
- Std-GeH, standard Germanium doped fibre with hydrogen loading

The experiment was conducted at ANSTO, using the gamma irradiation facility under the AINSE research award/grant ALNGRA13532. All results are from an uninterrupted regime of irradiation and relaxation. As mentioned in Chapter 4, each session comprises of three irradiations followed by three relaxation periods. For session one, each irradiation period/stage reached a gamma dose of 66.5 kGy over 21.3 hours, which equated to a dose rate of 3.12 kGy/hr. After three irradiation stages, each of 21.3 hours duration, a relaxation stage/period of 3.5 hours followed. The only variation occurred in the third relaxation stage, which achieved 2.0 hours of relaxation. This was intentional for comparative purposes and will be discussed in section 5.1.2. The actual response and overall cumulative effect during irradiation session one, inclusive of the three irradiation and relaxation periods is shown in Figure 5.1, emphasizing the Ge-FBG (base wavelength, 1544.88 nm). It tracks the response during the complete session, noting the end of each irradiation and relaxation period. The BWS is seen to restart after each relaxation period indicating the effects are cumulative. It must be noted that no error bars are included on graphs throughout the thesis. This is to assist in the visual presentation...
and easier interpretation of FBG responses. Included however, are some examples of logarithmic trends from the various FBGs during irradiation. The overall uncertainty associated with the recorded wavelengths and resultant BWS by the OSA, pre-and-post irradiation is ±0.005 nm (±5 pm) as mentioned in Chapter 4.

**Figure 5.1:** Graph showing FBG (Ge,1544.88 nm) cumulative effect of three irradiation stage inclusive of three relaxation stages. Arrows indicate where the irradiation and relaxation ceased. Total BWS 140pm. Saturation occurred at ~170 kGy (~54.6 hrs of irradiation).

5.1.1 Bragg Wavelength Shift during irradiation

This section reports on the BWS and response of each STD-FBG during the total cumulative session and then the response during the irradiation periods (without relaxation). The cumulative (inclusive of relaxation) total BWS ±5 pm of each FBG during session one is shown in Table 5.1. The starting base wavelength (before irradiation) and final wavelength (after the last relaxation) of each FBG is noted. A breakdown of results highlighting the BWS not inclusive of relaxation for the three irradiation periods only are shown in Table 5.2.
Table 5.1: Cumulative BWS (±0.005 nm or ± 5 pm) for three 66.5 kGy irradiation periods. Total dose 199.5 kGy, inclusive of three relaxation shifts.

<table>
<thead>
<tr>
<th>FBG</th>
<th>START WAVELENGTH</th>
<th>FINAL WAVELENGTH</th>
<th>TOTAL BWS</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+H</td>
<td>1539.76nm</td>
<td>1539.94nm</td>
<td>180pm</td>
</tr>
<tr>
<td>Ge</td>
<td>1544.88nm</td>
<td>1545.02nm</td>
<td>140pm</td>
</tr>
<tr>
<td>Ge+H</td>
<td>1549.98nm</td>
<td>1550.14nm</td>
<td>160pm</td>
</tr>
<tr>
<td>SMF28+H</td>
<td>1555.02nm</td>
<td>1555.14nm</td>
<td>120pm</td>
</tr>
</tbody>
</table>

Table 5.2: BWS (±5 pm) without relaxation for three 66.5 kGy irradiation periods, each of 21.3 hrs duration.

<table>
<thead>
<tr>
<th>FBG</th>
<th>BASE WAVELENGTH</th>
<th>BWS STAGE 1</th>
<th>BWS STAGE 2</th>
<th>BWS STAGE 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+H</td>
<td>1539.76nm</td>
<td>140pm</td>
<td>100pm</td>
<td>60pm</td>
</tr>
<tr>
<td>Ge</td>
<td>1544.88nm</td>
<td>120pm</td>
<td>80pm</td>
<td>80pm</td>
</tr>
<tr>
<td>Ge+H</td>
<td>1549.98nm</td>
<td>100pm</td>
<td>80pm</td>
<td>100pm</td>
</tr>
<tr>
<td>SMF28+H</td>
<td>1555.02nm</td>
<td>120pm</td>
<td>40pm</td>
<td>80pm</td>
</tr>
</tbody>
</table>

Results of the BWS in Table 5.2, indicate that FBG (SMF28H, 1539.76 nm) is less effective under this regime. It shows the reduction of the positive shift (140 pm, 100 pm, 60 pm) during each phase, whereas with the FBGs: Ge (120 pm, 80 pm, 80 pm) & Ge+H (100 pm, 80 pm, 100 pm) the reduction is less. The Ge+H and Ge- FBGs produced a more consistent response and nearly identical BWS. This is again highlighted in Table 5.1, when incorporating the relaxation shifts. The two Ge FBGs produced an almost identical total BWS after 199.5 kGy, of 140 pm and 160 pm. This may be due to the known stabilizing effect of germanium doping in optical fibres [76]. An additional factor to be considered, is the effect of the dose rate. As mentioned previously in Chapter 3, the effect of varied dose rates can affect the response e.g. the greater the dose rate the greater the BWS, or similar dose rate the similar BWS [11]. As shown in Table 5.1, the constant rate of 3.12 kGy/hr during this experiment produced a similar cumulative BWS.

To isolate the BWS of each individual FBG during each irradiation without relaxation, four graphs incorporating each period as a function of accumulated dose have been produced. A graphical representation showing the response of each individual FBG over
the three irradiation periods are shown in Figure 5.2, Figure 5.3, and Figure 5.4, highlighting the BWS without relaxation, and the effects of pre-irradiation leading to eventual onset of saturation. Figure 5.2 shows the BWS of FBG-SMF28+H (1539.76 nm) which produces a near logarithmic trend during the first two periods. The trend continues in the third period, however at the 12.5 kGy (or 145.5 kGy total) dose point definitive saturation begins.

Figure 5.2: BWS for three irradiation periods of FBG (SMF28+H) with 1539.76 nm base. The first irradiation stage (red line, no.1) reaches 140 pm before first relaxation period; BWS second stage (blue line, no.2) shows a slight decrease down to (100 pm); BWS third period (green line, no.3) shows a significant drop to 60 pm shift and eventual saturation.

Saturation occurred at the same point in the FBGs written in (Ge) & (Ge+H). Both saturated during the third irradiation stage at the 37 kGy (or 170 kGy) dose point, as shown in Figure 5.3. The final FBG (SMF28+H, 1555.02 nm) shown in Figure 5.4 produced quite a mixed behaviour. Stage 1 showed there was a logarithmic trend up to the 34 kGy at which point saturation seemed to take place, maintaining a 120 pm shift for the remaining 32.5 kGy. However, the BWS appeared to reduce greatly in period 2 and 3. During period two, a blue shift occurred after the 21.8 kGy point and again during period three at the 12.4 kGy point.
Figure 5.3: BWS for three irradiation periods of FBG (Ge) with 1544.88 nm base shown in (1) and FBG (Ge+H) with 1549.98 nm base shown in (2). Red lines are the first irradiation, Blue second and Green third. Note saturation occurring at a similar dose point in third irradiation at ~37 kGy (or 170 kGy accumulated dose).
Figure 5.4: BWS for three irradiation periods of FBG (SMF+H) with 1555.02 nm base. The first irradiation period (red line) reaches 120 pm before first relaxation period; BWS second period (blue line) shows a decrease to (40 pm); BWS third period (green line) shows a decrease to 80 pm shift.

Radiation induced blue shifts, although rare, have been reported by Maier et al.[80], Gusarov et.al [78], Butov et.al [140] and Niay et.al [141]. Usually during irradiation, the BWS shifts towards the longer (red) wavelengths followed by reduction (blue shift) during the relaxation phase. In this instance, during the second and third irradiation stages, there is a blue shift of ~60 pm as shown in Figure 5.5. Interestingly overall, this FBG displayed differing results from the three also covered by polyethylene sheets. This could be due to the extra strip of polyethylene which maximized the penetration of the gamma dose, as mentioned in Section 4.4. The result may be of significance and can be compared with the Gusarov et.al.[78] conclusion, that radiation sensitivity of FBGs relies to a significant extent on the fibre properties.

Although there was a reduction of the BWS in periods 2 and 3, the BWS during each irradiation resulted in a positive progressive trend in the first hour up to the 3.12 kGy stage. The shift in the first period at the 3.12 kGy stage was 40 pm; the second was 80 pm; at the third 120 pm; resulting in an increase of 40 pm during each period, as shown in Figure 5.6. Pre-irradiation in this instance seemed to have little or no effect especially in the first hour. It seemed to maintain sensitivity, which again may be due to the extra
polyethylene even after the relaxation periods. However at the points previously mentioned the FBG becomes less responsive. The positive progressive trend, albeit at a dose rate of 3.12 kGy, shows that this setup may hold well for low dose dosimetry.

Figure 5.5: BWS for second and third irradiation periods of FBG (SMF+H) with 1555.02 nm base. Blue BWS second period (blue line) begins ~ 21.8 kGy; Blue BWS third period (green line) begins ~ 12.4 kGy. An overall blue shift of ~60 pm.

Figure 5.6: BWS for three irradiation periods of FBG (SMF+H) 1555.02 nm after one hour or 3.12 kGy. Noting the progression, 40 pm, 80 pm, 120 pm.
The data indicates the cumulative effect of the gamma irradiation in three FBGs (1539.76 nm, 1544.88 nm, 1549.98 nm) result in logarithmic trends. An example of the trend is seen in Figure 5.7, which show the fits of the data points of SMF+H FBG (1539.76 nm) for the second irradiation dose; Ge FBG (1544.88 nm) and Ge+H FBG (1549.98 nm) for the third irradiation dose of 66.5 kGy. A logarithmic trend indicates the resultant data rises quickly and then starts to level off. Both Ge based FBGs plots rise to the ~37.5 kGy dose, and then begin to level off which is indicative of saturation. The SMF-FBG plot rises to ~47 kGy, at which point saturation commences. The data has a good fit and correlation with R² values slightly less than 0.90. An overall perspective highlighting the reflective spectra of the BWS after the second irradiation period for the four FBGs is shown in Figure 5.8. An expanded isolated reflective spectra showing the shift for the same second irradiation period from a single FBG is also shown in Figure 5.9.

![Figure 5.7: Graphs showing logarithmic trends: red plot = FBG (SMF, 1539.76 nm), blue plot = FBG (Ge+H 1549.98 nm), green plot = FBG (Ge, 1544.88 nm).](image-url)
To improve the radiation tolerance, and stability of FBGs whilst under gamma irradiation, the area that has shown promise is pre-irradiation. Current research suggests
that pre-irradiation, can reduce the sensitivity of FBGs, leading to a reduction in the BWS but producing a more stable FBG [72]. If FBGs are to be used in radiation environments successfully, they should remain stable. A study by Jing et al. [72] showed that pre-irradiation treatment can reduce the variation of radiation-induced BWS by 8% to 27.08% at a dose of 50 kGy @ 360 Gy/hr). All fibres were Ge doped and hydrogen loaded. The only exception was FBG 2, which was without hydrogen. Fibers 2 and 3 had identical Ge concentrations and were both the same fibre type (PSF-Ge-B-125). The results showed the BWS is higher in FBG3 than FBG2 [72]. This further indicates that hydrogen loading can increase the radiation sensitivity of FBGs [143].

This study is compatible with the results presented by Jing et al., notwithstanding that in this study, an increase in dose (66.5 kGy per period) and dose rate (3.12 kGy/hr) were used. The SMF28H (1539.76 nm) FBG induced BWS reduced by 28.54% in period 2, and the Ge + H (1549.98 nm) and Ge (1544.88 nm) FBGs BWS reduced by 20.0% and 33.3%, respectively in period 2. This study has included three irradiation periods, which effectively amounts to pre-irradiation treatment. The results indicate that the pre-irradiation treatment reduces the radiation sensitivity with FBGs (1544.88 nm, 1549.98 nm; Ge and Ge+H) resulting in increased stability whilst still maintaining a consistent BWS.

5.1.2 BWS during Relaxation versus Time

At the completion of each irradiation period, a stage of recovery/relaxation time followed. The BWS as a function of time compared to the original base wavelength was recorded. The relaxation BWS for the three periods are shown in Table 5.3. The variation in relaxation time periods in Stage 3, shown in Table 5.3 were conducted for comparative purposes. There a two 3.5 hour periods followed by Stage 3 which achieved 2.0 hours relaxation. During each period there was a shift back towards the original wavelength. The results were quite consistent except during period three. Although there was a 1.5 hour reduction in relaxation time, FBG (SMF28, 1539.76 nm) resulted in no shift at all. This may be due to lower photosensitivity and radiation hardening. Comparing with SMF28 (1555.02 nm) between the polyethylene sheets there was a marked difference. This FBG had the highest shift of -60 pm during this period. The remaining two FBGs (Ge and Ge+H) had a BWS’s of -20 pm and -40 pm, respectively. The larger continual
shift that occurred in SMF28 (1555.02 nm) during irradiation Stage 2 (-100 pm) and Stage 3 (-60 pm) may be linked with the ‘blue’ shift mentioned previously in section 5.1.1. As there was a shift towards the shorter wavelengths already occurring during the irradiation stage, it is feasible to suggest that after the cobalt-60 source was removed, the shift was enhanced further, producing larger shifts.

The results also indicate that there is a difference in the relaxation BWS between the SMF28H-FBG and Ge, Ge+H-FBGs. During the first relaxation stage, there are two distinct values/groupings. The two SMF28H FBGs produced a -80 pm shift whilst the Ge FBGs had a -60 pm shift. This is in keeping with established data, which states that, gratings written in Ge-doped fibre are more stable [76], thus producing similar results. Again during the second relaxation period, the SMF28H FBGs resulted in a higher BWS of -80 pm, and -100 pm, compared to the Ge-FBGs BWS of -60 pm, and -80 pm. The stability factor of the Ge-FBGs BWS -20 pm, and -40 pm continues in the third reduced relaxation phase, especially when compared with the SMF28H-FBGs 0 pm, and -60 pm.

A comparison between the two SMF28H-FBGs BWS as a function of time during the three relaxation stages is shown in Figure 5.10. The behaviour during the first relaxation (red plots) and second relaxation (blue plots) seem to be closely correlated and highlighted by the linear regression lines and correlation coefficients ($R^2$) values of 0.8679 and 0.8661 for SMF28H (1539.76 nm). During the third relaxation (green plots) the difference can be seen. Although there was a reduction in the relaxation time, no shift has occurred. It shows that a saturation type behaviour has occurred immediately after the third irradiation stage. Whereas in stage one, saturation began at the 2.5 hour mark and stage two after 2 hours. On average there is a 24.3 pm/hr shift for the first relaxation, and 25.7 pm/hr for the second relaxation periods in the SMF FBGs. For the third stage there is no reduction, which may be due to the reduced relaxation time of 2hrs.

### Table 5.3: BWS(±5 pm/0.005 nm) during relaxation for three stages; Stage 1&2 =3.5 hrs duration whilst Stage 3 = 2.0 hrs duration

<table>
<thead>
<tr>
<th>FBG</th>
<th>Base Wavelength</th>
<th>BWS Stage 1</th>
<th>BWS Stage 2</th>
<th>BWS Stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+H</td>
<td>1539.76nm</td>
<td>-80pm</td>
<td>-80pm</td>
<td>0pm</td>
</tr>
<tr>
<td>Ge</td>
<td>1544.88nm</td>
<td>-60pm</td>
<td>-60pm</td>
<td>-20pm</td>
</tr>
<tr>
<td>Ge+H</td>
<td>1549.98nm</td>
<td>-60pm</td>
<td>-80pm</td>
<td>-40pm</td>
</tr>
<tr>
<td>SMF28+H</td>
<td>1555.02nm</td>
<td>-80pm</td>
<td>-100pm</td>
<td>-60pm</td>
</tr>
</tbody>
</table>
Figure 5.10: BWS during the 3 relaxation stages as a function of time: first stage (red plot, 3.5 hrs), second stage (blue plot, 3.5 hrs), third stage (green plot, 2.0 hrs). (a) BWS of SMF28H (1539.76 nm) during three stages. (b) BWS of SMF28H (1555.02 nm) during three stages.

The relaxation behaviour of SMF28H (1555.02 nm) 25.7 pm/hr, is consistent with SMF28H(1539.76 nm) during the first relaxation stage. During the second stage there is a reduction to 20.5 pm/hr and a poor linear regression trend of $R^2$ value of 0.5831. The third stage is again different, resulting in a further reduction of 24.0 pm/hr ($R^2$ value of...
0.5) compared to no shift in SMF28H (1539.76 nm). The continual reduction may be linked to the blue shifts reported in section 5.1.1. This is reaffirmed in Figure 5.10 (b) when comparing the plots in stage two and three between the 0.5 hour to 2.0 hour mark. The identical response coincides with the blue shifts of stage two and three during the irradiation phase as shown previous in Figure 5.5. The overall average pm/hr for the three stages is 16.67 pm/hr for SMF28H-FBG (1539.76 nm) and 23.40 pm/hr for SMF28H-FBG (1555.02 nm).

A comparison between the three relaxation stages of the Ge and Ge+H based FBGs can be seen in Figure 5.11. The profiles highlighted are very similar with a good linear relationship. The correlation coefficients are very similar. During the stage one relaxation stages both have an identical response of 18.6 pm/hr. After the second relaxation stage the response is slightly different. The Ge+H resulted in 22.9 pm/hr compared to 16.7 pm/hr in the Ge- FBG. This trend continues through to the third stage, with the Ge +H response of 20.0 pm/hr compared to 12.0 pm/hr of the Ge - FBG. The overall average relaxation rate over the three stages is 15.77 pm/hr for the Ge-FBG and 20.5 pm/hr for the Ge+H Fbg. The Ge+H is exhibiting an increased in sensitivity and is more responsive during the relaxation phases even after the pre-irradiation effects of the first irradiation stage compared to the Ge-FBG. The increased photosensitivity due to hydrogen loading is more likely the cause of the stronger response.
5.1.3 Temperature Effects during Relaxation and Irradiation

As the average ambient temperature throughout the irradiation phase for irradiation session one was 22.8 °C inside the chamber, the effects of temperature playing a major role in the BWS during irradiation can more than likely be discounted. With the normal temperature sensitivity of ~ 10 pm to 15 pm/ °C [22,23,123], one could expect only a small red shift. The temperature during the three irradiation stages increased by 1.4, 1.6 and 0.8 °C respectively. Throughout each stage the temperature increased rapidly during the first 30 minutes (to within ~ 90% of max temperature change), and reached the maximum temperature after approximately one hour, at which point temperature seemed to stabilize. The fictive BWS corresponding to the monitored increase during irradiation would range from 8 pm to 24 pm if temperature was the only cause. However with the results previously mentioned of shifts between 120 pm and 180 pm, temperature effects during irradiation can be discounted.

Of interest however, is the possible effects of temperature during the relaxation phase. The temperature was monitored inside the polystyrene container after the gamma source was lowered. The relaxation temperature effects after irradiation session one can be seen in Table 5.4 for the first relaxation period of 3.5 hrs. The overall start temperature inside
the polystyrene container before the first irradiation commenced was 21.9°C. After 21.3 hrs of irradiation the temperature surrounding the FBGs increased to 23.3°C (+1.4°C). When the gamma source was lowered and relaxation commenced the temperature dropped to 20.1 °C (-3.2°C ) after 2 hours inside the chamber. The temperature was recorded every 10mins. After the 2.0hour period, the polystyrene box was removed from the chamber with the sensor and FBGs still in place. This was done to help elucidate the effects of temperature. Outside the chamber the recording continued every 10 minutes for a further period of 1.5 hours. The temperature dropped by 1.6 °C during this period, giving a total reduction of -4.8 °C.

Table 5.4 : Temperature change and BWS ±5 pm during the first 3.5 hours of relaxation after irradiation session one.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Temp. Start / Final</th>
<th>Temp. Drop</th>
<th>BWS</th>
<th>BWS pm/C</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+H</td>
<td>23.3/18.5 °C</td>
<td>-4.8 °C</td>
<td>-80pm</td>
<td>16.7pm/°C</td>
</tr>
<tr>
<td>Ge</td>
<td>23.3/18.5 °C</td>
<td>-4.8 °C</td>
<td>-60pm</td>
<td>12.5pm/°C</td>
</tr>
<tr>
<td>Ge+H</td>
<td>23.3/18.5 °C</td>
<td>-4.8 °C</td>
<td>-60pm</td>
<td>12.5pm/°C</td>
</tr>
<tr>
<td>SMF28+H</td>
<td>23.3/18.5 °C</td>
<td>-4.8°C</td>
<td>-80pm</td>
<td>16.7pm/°C</td>
</tr>
</tbody>
</table>

To further quantify the results, Table 5.5 highlights the temperature change and relaxation BWS versus time after the session one irradiation. It shows the temperature change and BWS over three 60 minute periods followed by one 30 minute period for a total of 210 minutes or 3.5 hours. The 60 minute and 120 minute readings are from inside the GATRI chamber, whilst the 180minute and 210min readings are outside the chamber. For the first 2 hours (-3.2 °C), the BWS of FBG SMF28 (1539.90) reduced by 60pm, which equates to a temperature sensitivity of 18.75 pm/ °C. For the next 1.5 hours (-1.6 °C) the BWS reduced by a further 20 pm to a wavelength of 1539.82 nm, which equates to 12.5 pm/ °C.

Table 5.5 : Temperature change and BWS(±5pm) breakdown through 3.5 time periods during first relaxation after irradiation session one.

<table>
<thead>
<tr>
<th>Time First Relaxation</th>
<th>Temperature Change</th>
<th>BWS SMF+H 1539.76nm</th>
<th>BWS SMF+H 1555.02nm</th>
<th>BWS Ge 1544.88nm</th>
<th>BWS Ge +H 1549.98nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>60min</td>
<td>-1.4°C</td>
<td>-20pm</td>
<td>-20pm</td>
<td>-20pm</td>
<td>0pm</td>
</tr>
<tr>
<td>120 min</td>
<td>-1.8°C</td>
<td>-40pm</td>
<td>0pm</td>
<td>-40pm</td>
<td>-40pm</td>
</tr>
<tr>
<td>180 min</td>
<td>-1.6°C</td>
<td>-20pm</td>
<td>-60pm</td>
<td>0pm</td>
<td>0pm</td>
</tr>
<tr>
<td>210min</td>
<td>0</td>
<td>0pm</td>
<td>0pm</td>
<td>0pm</td>
<td>-20pm</td>
</tr>
<tr>
<td>Total</td>
<td>-4.8°C</td>
<td>-80pm</td>
<td>-80pm</td>
<td>-60pm</td>
<td>-60pm</td>
</tr>
</tbody>
</table>
Overall, there was an 80 pm shift back towards the base original wavelength which can be also seen in the shift of the reflective spectra shown in Figure 5.12. The total temperature drop of 4.8 °C is equivalent to 16.7 pm/°C. The result is consistent with the second SMF-FBG(1555.02 nm) which produced an identical final result of -80 pm throughout the -4.8 °C change. There is a clear distinction between the two Ge FBGs and SMF FBGs. The Ge FBGs produced identical final shifts of -60 pm slightly lower than the SMF-FBGs. The increased responsiveness of the SMF-FBGs may be due to being more sensitive though photosensitization by hydrogen[143]. The normal temperature sensitivity of FBGs is approximately 10 pm/°C at 1550 nm for Ge Doped fibre [123,144]. It is known however, that FBGs with acrylate coating can register temperature sensitivities as high as 14 to 15 pm/°C [142]. The results obtained in the final pm/C column in Tables 5.4 show the SMF-FBGs during relaxation after the stage one irradiation, produced a reduction of Bragg wavelength of 16.7 pm/°C, whilst the Ge-FBGs 12.5 pm/°C. The results on first glance seem to be consistent with a possible thermal dependency in relation to the BWS during relaxation. However, the results in Table 5.5 show otherwise, with inconsistencies (the major ones shown in red) during the hourly periods.

Figure 5.12: Reflection Spectra of SMF28+H; BWS during first relaxation period after 3.5 hrs (4.8°C); Red peak is start wavelength of 1539.90 nm and Blue peak is final wavelength 1539.82 nm which equals 80 pm Blue shift.
After the 180 minutes no temperature drop was recorded. However, the wavelength of the Ge+H FBG continued to reduce by -20 pm. Conversely, between 120 and 180 minutes a temperature change of -1.6 °C occurred, yet no reduction of wavelength was evident in both Ge-FBGs. The same phenomena is apparent for the SMF-FBG (1555.02nm) between the 60 to 120 minute period. Although a temperature change of -1.8°C occurred, no wavelength reduction is evident. Again, during the first 60 minute period a temperature drop of 1.4 °C is shown, however the Ge+H FBG remained stable with no wavelength shift observed. The lack of continuity mentioned indicates that temperature is most likely not playing a major role during the recovery/relaxation phase.

The relaxation temperature effects after irradiation session two can be seen in Table 5.6. After a further 21.3 hours of irradiation the temperature increased from 21.4 to 23.0 °C (+1.6°C). After 2 hours of relaxation the temperature dropped to 20.3 °C (-2.7 °C) in the chamber. The container with the FBGs was again removed to outside the chamber for a further 1.5 hrs whilst recording every 5 mins. A further drop in temperature was recorded to 18.0 °C (-2.3 °C) giving a total of 5.0 °C reduction.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Temp. Start / Final</th>
<th>Temp. Drop</th>
<th>BWS</th>
<th>BWS pm/°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+/H</td>
<td>23.0/18.0 °C</td>
<td>-5.0 °C</td>
<td>-80 pm</td>
<td>16.0 pm/°C</td>
</tr>
<tr>
<td>Ge</td>
<td>23.0/18.0 °C</td>
<td>-5.0 °C</td>
<td>-60 pm</td>
<td>12.0 pm/°C</td>
</tr>
<tr>
<td>Ge+H</td>
<td>23.0/18.0 °C</td>
<td>-5.0 °C</td>
<td>-80 pm</td>
<td>16.0 pm/°C</td>
</tr>
<tr>
<td>SMF28+/H</td>
<td>23.0/18.0 °C</td>
<td>-5.0 °C</td>
<td>-100 pm</td>
<td>20.0 pm/°C</td>
</tr>
</tbody>
</table>

Table 5.7 highlights the temperature change and relaxation BWS versus time after the session two irradiation. It shows the temperature change and BWS over three 60 minute periods followed by one 30 minute period for a total of 210 minutes or 3.5 hrs. The 60 minute and 120 minute readings are from inside the GATRI chamber whilst the 180 minute and 210 min readings are outside the chamber. For the first 2.0 hours (-2.7°C), the BWS of the Ge-FBG (1544.88 nm) reduced by 40 pm, which equates to 14.81 pm/°C. For the second 1.5 hrs (-2.3°C), the BWS reduced by a further 20 pm, which equates to 8.70 pm/°C. Overall there was a 60 pm shift back towards the base through a temperature drop of 5.0 °C which equates to 12.0 pm/°C. The overall result is consistent with the Ge+H FBG which produced a -80 pm shift. The slight increase of reduction is
more likely due to the hydrogen loading and therefore response and sensitivity. There is a distinction between the two Ge FBGs and SMF FBGs. The Ge FBGs produced slightly lower final shifts compared to the SMF-FBGs.

There seems to be a consistency in the results between the two sets of FBGs for relaxation versus temperature in both Stages 1 and 2. The only variation, is by FBG-SMF28H (1555.02 nm) in the second stage, which recorded an overall equivalent wavelength reduction rate of 20 pm/°C, which seems slightly high. Again this may be due to the extra polyethylene sheets and the blue shift previously reported in section 5.1.1. The inconsistencies are again evident in Table 5.7 highlighted in red. During the first 60 minutes of relaxation a 1.1 °C temperature drop occurred, with three FBGs producing a -20 pm wavelength reduction. However, FBG-SMFH(1555.02 nm) produced a -80 pm wavelength shift which is equivalent to ~72.72 pm/°C. A further temperature drop occurred of -1.6 °C during the second 60 minutes with FBG-SMFH (1555.02 nm) recording no shift and FBG-SMFH (1539.76 nm) recording a -60 pm (~37.50 pm/ °C ) reduction. During the third 60 minute period a temperature drop of -2.3 °C resulted, however, two FBGs recorded no shifts. For the final 30 minute period, there was no temperature change, however FBG-GeH (1549.98 nm) produced a -20 pm wavelength shift. In summary, the second relaxation phase has again pinpointed a lack of continuity in relation to thermal dependency.

Table 5.7: Temperature change and BWS ±5 pm breakdown through 3.5 time periods during the second relaxation after irradiation session two.

<table>
<thead>
<tr>
<th>Time Second Relaxation</th>
<th>Temperature Change</th>
<th>BWS SMF+H 1539.76nm</th>
<th>BWS SMF+H 1555.02nm</th>
<th>BWS Ge 1544.88nm</th>
<th>BWS Ge +H 1549.98nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>60min</td>
<td>-1.1°C</td>
<td>-20pm</td>
<td>-80pm</td>
<td>-20pm</td>
<td>-20pm</td>
</tr>
<tr>
<td>120 min</td>
<td>-1.6°C</td>
<td>-60pm</td>
<td>0pm</td>
<td>-20pm</td>
<td>-40pm</td>
</tr>
<tr>
<td>180 min</td>
<td>-2.3°C</td>
<td>0pm</td>
<td>-20pm</td>
<td>-20pm</td>
<td>0pm</td>
</tr>
<tr>
<td>210min</td>
<td>0</td>
<td>0pm</td>
<td>0pm</td>
<td>0pm</td>
<td>-20pm</td>
</tr>
<tr>
<td>Total</td>
<td>-5.0°C</td>
<td>-80pm</td>
<td>-100pm</td>
<td>-60pm</td>
<td>-80pm</td>
</tr>
</tbody>
</table>

The relaxation temperature effects after irradiation session three can be seen in Table 5.8. After a further 21.3 hours of irradiation the temperature increased from 21.9 to 22.7°C (+0.8°C). After 2 hours of relaxation the temperature dropped to 21.8°C (-0.9°C) in the
chamber. The container with the FBGs remained inside the chamber for the reduced time period of 2 hours. With the reduction of temperature being close to 1°C, it is easy to disseminate and compare the results in relation to the accepted sensitivity rates of between 10pm to 14pm/°C [132,144]. Interestingly SMF-FBG (1539.76nm) produced no reduction after 2 hours whilst the three remaining FBGs resulted in high reduction sensitivities between ~ -22 pm to 66 pm/°C. With the BWS breakdown shown in Table 5.9 through two 60 minute periods, the inconsistency is again evident. During the second hour there is consistency i.e. no reduction or recovery recorded. This could be due to increased permanent structural damage caused by the gamma exposure.

Table 5.8: Temperature change and BWS during the third relaxation of 2.0 hours after irradiation session three.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Temp. Start / Final</th>
<th>Temp. Drop</th>
<th>BWS</th>
<th>BWS pm/°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+H</td>
<td>22.7/21.8°C</td>
<td>-0.9°C</td>
<td>0pm</td>
<td>0pm/°C</td>
</tr>
<tr>
<td>Ge</td>
<td>22.7/21.8°C</td>
<td>-0.9°C</td>
<td>-20pm</td>
<td>22.22pm/°C</td>
</tr>
<tr>
<td>Ge+H</td>
<td>22.7/21.8°C</td>
<td>-0.9°C</td>
<td>-40pm</td>
<td>44.44pm/°C</td>
</tr>
<tr>
<td>SMF28+H</td>
<td>22.7/21.8°C</td>
<td>-0.9°C</td>
<td>-60pm</td>
<td>66.66pm/°C</td>
</tr>
</tbody>
</table>

Table 5.9: Temperature change and BWS ±5 pm breakdown through two time periods during the third relaxation after irradiation session three.

<table>
<thead>
<tr>
<th>Time Second Relaxation</th>
<th>Temperature Change</th>
<th>BWS SMF+H 1539.76nm</th>
<th>BWS SMF+H 1555.02nm</th>
<th>BWS Ge 1544.88nm</th>
<th>BWS Ge +H 1549.98nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>60min</td>
<td>-0.9C</td>
<td>0pm</td>
<td>-60pm</td>
<td>-20pm</td>
<td>-40pm</td>
</tr>
<tr>
<td>120 min</td>
<td>0</td>
<td>0pm</td>
<td>0pm</td>
<td>0pm</td>
<td>0pm</td>
</tr>
<tr>
<td>Total</td>
<td>-0.9C</td>
<td>0pm</td>
<td>-60pm</td>
<td>-20pm</td>
<td>-40pm</td>
</tr>
</tbody>
</table>

A comparison of BWS v temperature drop was completed at ECU (Edith Cowan University), with a replication of the conditions and setup inside the GATRI chamber at ANSTO. The fibres are the exact originals that were used at ANSTO. The experimental setup is similar to that described in Section 4.4. The only variation in equipment used was the OSA. At ECU we used a Thorlabs OSA202 Optical Spectrum Analyser. The FBGs were placed in a polystyrene container and the air temperature was heated to 24.7 °C (for run 1) and 25.7 °C (for run 2), by pumping warm air (using a warm air dryer). The temperature inside the container was monitored using a Pasco temperature sensor/probe which was positioned as close as possible (within~ 3 to 4 cm) to the FBGs.
The container was then sealed. The room air temperature was thermostatically controlled at 20.5°C. The Bragg wavelength as a function of temperature was recorded during the cooling/relaxation period every 2 seconds using a PC with a Pasco interface box and Data studio digital temperature setup.

After 30 minutes during each run, the temperature had stabilized to 21.2 and 22.3°C respectively and at this point the BWS had reached saturation. Overall temperature drop was -3.5 and -3.4 °C. A comparison of the saturated BWS and pm/°C between the results on site immediately after Gamma exposure and 10mths after are shown in Table 5.10. The reduction is noticeable and consistent throughout the ECU results. On average, there is a 8.2 pm/°C shift at ECU compared to an average of 14.8 pm/°C immediately after Gamma exposure at ANSTO. This equates to a 44.6% temperature sensitivity reduction. The reduction of wavelength per temperature change could be due to radiation hardening and hydrogen outgassing, reducing the temperature sensitivity of the FBGs [71].

<table>
<thead>
<tr>
<th>FBG</th>
<th>ECU Lab Run1</th>
<th>ANSTO Stage 1</th>
<th>ECU Lab Run 2</th>
<th>ANSTO Stage 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28+H</td>
<td>7.7pm/°C</td>
<td>16.7pm/°C</td>
<td>8.2pm/°C</td>
<td>16.0pm/°C</td>
</tr>
<tr>
<td>Ge</td>
<td>8.4pm/°C</td>
<td>12.5pm/°C</td>
<td>8.5pm/°C</td>
<td>12.0pm/°C</td>
</tr>
<tr>
<td>Ge+H</td>
<td>8.0pm/°C</td>
<td>12.5pm/°C</td>
<td>8.5pm/°C</td>
<td>16.0pm/°C</td>
</tr>
<tr>
<td>SMF28+H</td>
<td>8.0pm/°C</td>
<td>16.7pm/°C</td>
<td>8.5pm/°C</td>
<td>20.0pm/°C</td>
</tr>
</tbody>
</table>

**5.1.4 Amplitude variation**

The amplitude variations of the FBGs after the complete gamma exposure of 199.5 kGy are listed in Table 5.11. The maximum reduction of amplitude is 1.8521 dBm for the Ge + H FBG, whilst the minimum value is 0.9884 dBm for the SMF-H FBG. The overall average amplitude change/reduction is 1.3426 dBm. The average change for the SMF-H FBGs is 1.1781dB and for the Ge FBGs is 1.5071 dB. It can be seen that the amplitude change is very small over the 63.9 hours of irradiation. The results are compatible with previous research [11,72,71,76] showing that the variations are small and that FBGs are able to avoid the broadband radiation induced optical power loss (radiation induced
attenuation) because of the narrow wavelength encoding or narrow spectral range of < 5nm [11].

Table 5.11: Total Amplitude shift (cumulative) after 199.5 kGy of irradiation inclusive of final relaxation period

<table>
<thead>
<tr>
<th>FBG</th>
<th>Reflective power after 199.5 kGy</th>
<th>Reflective power change / dBm</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28H (1539.76)</td>
<td>-24.7785</td>
<td>-0.9884</td>
</tr>
<tr>
<td>Ge(1544.88)</td>
<td>-27.0414</td>
<td>-1.1620</td>
</tr>
<tr>
<td>GeH(1549.98)</td>
<td>-28.1262</td>
<td>-1.8521</td>
</tr>
<tr>
<td>SMF28H(1555.02)</td>
<td>-28.5641</td>
<td>-1.3679</td>
</tr>
</tbody>
</table>

5.1.5 Optiwave reconstruction of FBG properties

To simulate strain being produced given original wavelength (before irradiation) and new final wavelength (after irradiation inclusive of all recovery periods), specifications and parameters were entered in Optigrating software by Optiwave version 4.2.3 (released Dec 2014) [139]. This software can reconstruct an unknown grating from the reflection coefficient using a layer peeling algorithm. It also allows adjustments to be made to the properties of a Bragg grating and to be modified for comparisons and analysis. By varying the values of applied strain from e.g. 0 με (simulated pre-irradiation) to 117.25 με (simulated post irradiation) to the original wavelength grating specifications in the combined reflection profile, a new combined profile equal to the wavelength after simulated irradiation was produced. This gives an indication of strain that has been produced during irradiation to cause the BWS, and also underscores the permanent change in grating period after relaxation.

The profiles including the reconstructed are shown in Figure 5.13, demonstrate a grating period change of (a) 0.532307 μm at 1539.76 nm wavelength to (b) 0.532369 μm at 1539.94 nm. Figure 5.13 (c) shows the matching profile after 117.5 micro-strain added to the original 1539.76 nm grating to achieve 0.532369 μm. The apodization appears to be a similar shape as the original grating as seen in Figure 5.13(a), (b), (c). The change in
the grating period equates to an increase of 62 pm (0.062 nm). This FBG during irradiation produced a final accumulated shift inclusive of relaxation of 180 pm. As can be seen by the optigrating simulation, 117.25 applied micro strain was needed to achieve the matching profile. The established gage factor in standard 125 micron silica fibre with a Bragg wavelength at 1550 nm is known to be ~1.2 pm per microstrain applied to the fibre [23]. Through simulation, our results show that there has been an increase to 1.5352 pm/micro-strain. This is an increase of 28.1% and is a strong indication that during the irradiation period and after relaxation there may be a permanent change to the grating period within the FBG.

For comparison, a simulation was conducted with the parameters of the Ge+H with 1549.98 nm initial wavelength and 1550.14 nm final wavelength. The grating period changed/increased from 0.535848 μm to 0.535904 μm after 103.5 micro-strain was applied. All the apodization profiles appear similar. The change in grating period equated to 56 pm (0.056 nm). The final BWS of this FBG inclusive of relaxation is 160 pm. Through the simulation, the results indicate an increase to 1.545 pm/micro-strain (28.75%). The change to the grating period and increases are shown in Table 5.12. The comparison between the optigrating simulation of the SMF28+H and Ge+H FBGs, emphasize there is continuity in the results, adding credence that there is a permanent change to the grating period, pre and post irradiation.

Table 5.12: Simulated Grating period change and Total change

<table>
<thead>
<tr>
<th>Sample</th>
<th>Final Wavelength</th>
<th>Grating Period Start (μm)</th>
<th>Grating Period Final (μm)</th>
<th>Total Change (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28H</td>
<td>1539.94nm</td>
<td>0.532307</td>
<td>0.532369</td>
<td>+0.062nm</td>
</tr>
<tr>
<td>Ge</td>
<td>1545.02nm</td>
<td>0.534081</td>
<td>0.534130</td>
<td>+0.049nm</td>
</tr>
<tr>
<td>Ge+H</td>
<td>1550.14nm</td>
<td>0.535848</td>
<td>0.535904</td>
<td>+0.056nm</td>
</tr>
<tr>
<td>SMF28H</td>
<td>1555.14nm</td>
<td>0.537595</td>
<td>0.537636</td>
<td>+0.041nm</td>
</tr>
</tbody>
</table>
To simulate and isolate the effects of temperature during relaxation using the Optigrating software, the final reflection profile in Figure 5.12 (c) with the base wavelength of 1539.94 nm was chosen after the second irradiation stage (133 kGy cumulative). The final temperature of 23 °C, as per the actual experiment, was entered in the fibre Bragg grating dialogue box constant temperature box. The strain was isolated with zero being entered in the constant section as shown in Figure 5.14.

Figure 5.14: Fiber Bragg Grating Sensor dialog box with 23°C added. (red arrow).
As mentioned, there was a 5 °C drop recorded within close proximity of the FBGs during the second relaxation period over a 3.5 hour time period. To compare the grating period change in relation to temperature only, a comparison was made with the reflection profiles of the starting wavelength of 1539.94 nm (23 °C) and final profile with 18 °C. The grating period recorded was 0.532369 μm at 23 °C and 1539.940 nm, whilst at 18 °C the grating period reduced to 0.532367 μm and 1539.934 nm resulting in a -0.000002 μm (-0.002 nm) grating period change. To produce the same equivalent profile with strain, a total of -4.2 με (~1.43 pm/με) had to be included. If the temperature was the only factor during the relaxation of wavelength, then the shift would equate to a reduction of only ~6 pm. However, the experimental results show an overall reduction of 80 pm to 1539.86 nm during the same period resulting in -55 με and -0.000029 μm (-0.029 nm) change to the grating period. The experimental results for the relaxation phase produced a figure of ~1.45 pm/με sensitivity @ ~23 °C whilst during simulation ~1.43 pm/με. With the similar strain sensitivity, we can assume that with a reduction of temperature (-5°C) during the relaxation phase, the change in grating period due to temperature is 0.002 nm (2pm), and for strain 0.027 nm (27pm) during the relaxation phase. This implies that during the relaxation period, the reduction of the grating period, and therefore expansion related to ‘negative’ compaction, is most likely due to a combination of strain (93.1%) and temperature (6.9%).

5.1.6 Summary irradiation session 1

In conclusion, a Bragg wavelength shift is observed in all three irradiation stages of the same accumulated dose and dose rate. After each irradiation stage, the BWS reduced in all FBGs. This could be due to pre-irradiation effects and radiation hardening. With very limited recovery times between irradiation, the BWS is compatible with previous research that have used greater recovery times. For the first and second relaxation stages there is a reduction of wavelength back towards the original. The two Ge-FBGs recovery rates combined average reduction equates to, -18.6 pm/hr (first recovery stage); -19.8 pm/hr (second recovery stage); and -16 pm/hr (third recovery stage). The two SMF-FBGs corresponding combined rate average reduction is, -25.0 pm/hr shift (first stage), -23.1 pm/hr (second stage) and -12.0 pm/hr (third stage).
The simulated results indicate there is an increase of strain causing the grating period to increase which in turn produces on average a 28.21% increase of the pm/micro-strain to ~1.5385pm/micro-strain compared to the known standard rate ~1.20pm/micro-strain. This is a strong indication that during the irradiation period and after relaxation there may be a permanent change to the grating period within the FBG. With the effective index kept constant (therefore neglected) the increase of micro strain and resulting BWS may be due to compaction, as it directly impacts the grating period [119]. During irradiation the Bragg wavelengths can show increased red shifts under radiation induced compaction [81].

Particular interest is focused on the relationship between relaxation effects and the temperature effects during the non-irradiation time period. The temperature results during this period show there is a 4.8 to 5.0°C drop in the first two corresponding relaxation periods. When comparing the BWS with temperature drop, the range was 12.0pm/C to 20.0 pm/°C for all FBGs in the corresponding periods. The third relaxation period of 2 hours produced a temperature drop of 0.9C and wavelength range of 0 to 66.67 pm/°C. Normal temperature sensitivity of FBGs is approximately 10 pm/°C at 1550 nm [153]. It is known however that FBGs with acrylate coating however, can register higher temperature sensitivities as high as 14 to 15 pm/°C. The fibres used are coated with acrylate, therefore the inconsistent results reported indicate that the temperature is more than likely not playing a significant role during the relaxation/recovery periods. This is in keeping with several works showing that the temperature sensitivity is not influenced by irradiation [64, 123, 144, 145, 146]. The reduction of BWS (7.7-8.5 pm/°C) during relaxation at ECU can be put down to the increase of relaxation and post recovery times, method and equipment used, in addition to the effects of radiation hardening. To further quantify our results, analysis by SAXS, XRD pre and post irradiation are shown in Chapter 6.

5.2 Irradiation Session Two

The results from irradiation session two are produced by PCF-FBG (base wavelength 1540.806 nm). The FBG was inscribed in endlessly single mode pure silica photonic crystal fibre (ESM 12-01) manufactured by BlazePhotonic (NKT) purchased from Thorlabs). As mentioned the PCF-FBG inscription was performed through a collaborative arrangement with iPL laboratories at the University of Sydney. The experiment was
conducted again at ANSTO, using the same gamma irradiation facility under the AINSE research award/ grant ALNGRA15540. All data is obtained again from an uninterrupted regime of irradiation and relaxation. For session two, each irradiation stage reached 50.6 kGy over 21 hrs, which equated to a dose rate of 2.41 kGy/hr. After each irradiation (x3) of 21 hrs. duration, a relaxation stage of 3 hrs. followed. The actual response and overall cumulative effect during irradiation session two highlighting the BWS, inclusive of the three irradiation and relaxation stages are shown in Figure 5.15. It can be seen clearly that the BWS restarts after each relaxation stage as in the STD-FBGs, however, the major difference being that during the final relaxation almost full recovery is achieved.

![Figure 5.15: Graph showing PCF-FBG (1540.806 nm) cumulative effect over three irradiation periods inclusive of three relaxation periods. Arrows indicate where the irradiation and relaxation periods ceased. Total BWS 12pm. Almost full recovery.](image)

5.2.1 Bragg Wavelength Shift during irradiation

The cumulative (inclusive of relaxation) total BWS of PCF-FBG (1540.806 nm) during session two is shown in Table 5.13. The initial wavelength was 1540.806 nm and the final 1540.818 nm, culminating in a total BWS inclusive of relaxation of just 12pm. The BWS not inclusive of the relaxation for the three irradiation stages for the PCF-FBG in session two are shown in Table 5.14. Overall the results were very consistent except during each irradiation stage. It shows there is only a slight reduction of the positive shift (56 pm, 42 pm, 32 pm) during each irradiation phase.
Table 5.13: BWS (±0.005nm / ± 5pm) cumulative for three irradiation 50.6 kGy stages (151.8 kGy), inclusive of relaxation shifts.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Base Wavelength</th>
<th>Final Wavelength</th>
<th>Total BWS</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF-FBG</td>
<td>1540.806nm</td>
<td>1540.818nm</td>
<td>12pm</td>
</tr>
</tbody>
</table>

Table 5.14: BWS (±0.005nm / ± 5pm) without relaxation for three 50.6 kGy irradiation stages, each of 21 hrs duration.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Base Wavelength</th>
<th>BWS Stage 1</th>
<th>BWS Stage 2</th>
<th>BWS Stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF-FBG</td>
<td>1540.806nm</td>
<td>56pm</td>
<td>42pm</td>
<td>32pm</td>
</tr>
</tbody>
</table>

A graphical representation over the three irradiation stages for session two are shown in Figure 5.16, highlighting the shift without relaxation. It can be seen there is a near logarithmic trend during the three irradiation stages. During the third stage the BWS towards the red is at its lowest. Still however, this result of a consistent BWS (56, 42, 32pm) is compatible with the standard SMF28+H FBG results with a similar accumulated dose of 50 kGy in the study by Jing et.al.[72] which produced a 46 pm shift in their first stage and 35pm shift in their second stage. Saturation occurred fairly consistently at approximately the 40 kGy point during each irradiation.

Figure 5.16: BWS for three irradiation stages for PCF-FBG with 1540.806nm base. (1) Shift equals 56pm before first relaxation period; (2) BWS second stage shows a slight decrease down to (42pm); (3) and third stage a further slight drop to 32pm.
5.2.2 BWS during Relaxation versus Time

At the completion of each irradiation stage a period of recovery/relaxation time followed. The BWS as a function of time compared to the original base wavelength was recorded. The relaxation BWS of PCF-FBG for the three periods are shown in Table 5.15. All relaxation periods for were conducted over a 3.0 hr. time period. During each relaxation period there was a shift back towards the original wavelength to almost full recovery. Total recovery was nearly achieved after 151.8 kGy of gamma irradiation (inclusive of relaxation). The initial wavelength was 1540.806 nm and final 1540.818 nm, culminating in a total BWS inclusive of relaxation of just 12 pm as shown in Table 5.11.

Table 5.15: BWS (±0.005nm or ± 5pm) during relaxation for three stages, each of 3 hrs duration.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Base Wavelength</th>
<th>BWS Stage 1</th>
<th>BWS Stage 2</th>
<th>BWS Stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF-FBG</td>
<td>1540.806nm</td>
<td>-40pm</td>
<td>-36pm</td>
<td>-42pm</td>
</tr>
</tbody>
</table>

A comparison between the BWS as a function of time during the three relaxation stages of the PCF-FBG is shown in Figure 5.17. The behaviour during the first relaxation (red plots), second relaxation (blue) and third relaxation (green) are closely correlated and show a very strong linear relationship shown by the correlation coefficients ($R^2$)>0.9. The first relaxation stage resulted in a rate of 17.0 pm/hr, 13.0 pm/hr for the second relaxation and 11.1 pm/hr for the final relaxation stage. During the relaxation stage the PCF-FBG consistently showed significant recovery to almost the original base wavelength. This is larger than that of conventional fibers under similar regimes and is in keeping with research that pure silica PCF-FBGs has a superior recovery time[92] principally because there is no dopants such as Ge involved. The results show that there is virtually no change in the relaxation BWS shift between each period. The reductions range from -40 pm in the first period, -36 pm in the second period, and -42pm on the third. An overall average equates to a -39.33 pm shift during each irradiation period. On average there is a rate of 13.7 pm/hr during the relaxation stages. The consistency and strong stability factors of the PCF-FBG are evident quantitatively.
Figure 5.17: PCF-FBG (1540.806nm) BWS during the 3 relaxation stages as a function of
time: first stage (red plot, 3hrs), second stage (blue plot, 3hrs), third stage (green plot,
3hrs).

5.2.3. Temperature effects during relaxation and irradiation.

The temperature range in the GATRI chamber recorded during the irradiation periods
ranged from 21.5 to 23.7 °C for this stage. The temperature during the three irradiation
stages increased by 1.2, 1.2, and 1.1°C respectively. As previously mentioned regarding
STD-FBGs, throughout each stage the temperature again increased rapidly during the first
30 minutes (to within ~ 90% of max temperature change), and reached the maximum
temperature after approximately one hour, at which point the temperature seemed to
stabilize. When factoring the PCF-FBGs known temperature sensitivity of ~10.5 pm/°C
[173], the fictive BWS corresponding to the monitored increase during irradiation would
range from ~11 pm to 12.6 pm if temperature was the major factor. However with the
results previously mentioned of shifts between 32 pm and 56 pm, temperature effects
during irradiation can be discounted.

Due to the setup and fragility near the splice points of the PCF-FBGs, monitoring and
characterization was performed at Edith Cowan University, both pre and post irradiation
in relation to BWS v temperature drop during the relaxation period. The fibers were the
exact originals that were used at ANSTO and the experimental setup at ECU was identical
to that described in section 4.4. The only variation was at ECU, with a Thorlabs OSA202 Optical Spectrum Analyser being used to monitor the reflected spectra. The FBGs were placed in a polystyrene container and heated to 30.0°C over three runs. Wavelength as a function of temperature was then recorded during the relaxation period every 2 seconds using a Pasco interface and Data studio digital temperature setup. After 30 mins. during each run, the temperature had stabilized to 23.0°C, and reached saturation. Overall temp drop was -7.0°C. A comparison of the saturated BWS and pm/°C between the results both pre irradiation and post irradiation are shown in Table 5.16. On average there is a 16.3 pm/°C shift (pre irradiated) and 16.5 pm/°C (post irradiation) at ECU. This indicates after gamma exposure the temperature sensitivity remains constant in the PCF-FBGs. For a comparison the relaxation results from onsite have been included. The temperature start and finish for the relaxation phases were recorded whilst inside the container, and inside the chamber only, due to the fragility of the samples. On average, for the PCF-FBGs during the relaxation stages, a -1.5°C temperature drop and a -39.3 pm BWS occurs. This results in an increased sensitivity of 26.2 pm/°C. The sensitivity increase implies, during relaxation, factors other than temperature may be evident.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Temp. Start / Final</th>
<th>Temp. Drop</th>
<th>BWS</th>
<th>BWS pm/°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF(ECU) Not irradiated</td>
<td>30.0/23.0 °C</td>
<td>-7.0 °C</td>
<td>-113.9 pm</td>
<td>16.3pm/°C</td>
</tr>
<tr>
<td>PCF(ECU) Irradiated</td>
<td>30.0/23.0 °C</td>
<td>-7.0 °C</td>
<td>-115.3 pm</td>
<td>16.5pm/°C</td>
</tr>
<tr>
<td>PCF(ANSTO) relaxation stage1</td>
<td>23.7/21.9 °C</td>
<td>-1.8 °C</td>
<td>-40 pm</td>
<td>22.2pm/°C</td>
</tr>
<tr>
<td>Relaxation stage 2</td>
<td>23.1/21.7 °C</td>
<td>-1.4 °C</td>
<td>-36 pm</td>
<td>25.7pm/°C</td>
</tr>
<tr>
<td>Relaxation Stage 3</td>
<td>22.8/21.5 °C</td>
<td>-1.3°C</td>
<td>-42 pm</td>
<td>32.3pm/°C</td>
</tr>
</tbody>
</table>

5.2.4 Amplitude variation

The amplitude variations of the FBG after the complete gamma exposure of 151.8 kGy are listed in Table 5.17. The overall amplitude change/reduction is 0.0011 dBm. It can be seen that the amplitude change is very small over the 63 hours of irradiation. As Silica-
based Photonic Crystal Fibres (PCFs) potentially show lower attenuation loss for conventional fibre such as single mode fibre (SMF) [88], the small variation pre and post irradiation result seems consistent.

**Table 5.17 Total Amplitude shift (cumulative) after 151.8 kGy of irradiation inclusive of final relaxation period**

<table>
<thead>
<tr>
<th>FBG</th>
<th>Reflective power No irradiation dBm</th>
<th>Reflective power after 151.8 kGy dBm</th>
<th>Reflective power Change / dBm</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF-FBG</td>
<td>-59.0736</td>
<td>-59.0747</td>
<td>-0.0011</td>
</tr>
</tbody>
</table>

**5.2.5 Optiwave reconstruction of FBG properties**

By varying the values of applied strain from e.g. 0 με (simulated pre-irradiation) to 8.0 με (simulated post irradiation) to the original wavelength grating specs in the combined reflection profile, the combined profile equal to the wavelength after simulated irradiation was produced. This gives an indication of strain that has been produced during irradiation to cause the BWS, and also accentuates the permanent change in grating period after relaxation. The profiles including the reconstructed are shown in Figure 5.18 (a) (b), underline a grating period change of (a) 0.53266 μm at 1540.806 nm wavelength to (b) 0.532270 μm at 1540.818 nm. Figure 5.18 (c) shows the matching profile after 8.0 micro strain added to the original 1540.806nm grating to achieve 0.532270μm. The apodization appears to be a similar shape as the original grating as seen in Figure 18(a), (b), (c). The change to the grating period equates to an increase of 4pm (0.004nm) as shown in Table 5.18. This FBG during irradiation produced a final accumulated shift inclusive of relaxation of 12pm.

As can be seen by the optigrating simulation, 8.0 applied micro-strain was needed to achieve the matching profile. The established gage factor in standard 125 micron silica fiber with a Bragg wavelength at 1550nm is known to be ~1.2pm per micro-strain applied to the fiber [39]. Through simulation our results reveal that there has been an increase to 1.5 pm/ micro-strain. This is an increase of 25.0% and is a strong indication that during the irradiation period and after relaxation, there may be a permanent change to the grating period within the PCF-FBG.

**Table 5.18: Simulated Grating period change and Total change**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Final Wavelength</th>
<th>Grating Period Start (μm)</th>
<th>Grating Period Final (μm)</th>
<th>Total Change (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF</td>
<td>1540.818nm</td>
<td>0.532266</td>
<td>0.532270</td>
<td>+0.004 nm (4pm)</td>
</tr>
</tbody>
</table>
Figure 5.18: (a) PCF-FBG 1540.806 nm reflection profile (apodization, shape, length, period) before irradiation simulation. (b) profile of 1540.818nm simulating after irradiation, both with no strain. (c) reconstructed profile after 8.0 micro strain entered to original 1540.806 nm profile. Apodization is identical in all three.

5.2.6 Summary Irradiation Session Two

For session two, the BWS is observed in all three irradiation stages of the same accumulated dose and dose rate. After the first irradiation stage, the BWS shift to the red reduced in the PCF-FBG by 25%. After the second the reduction was by only 24% after completing an accumulated dose of 151.8 kGy. We have shown also that, with very limited recovery times between irradiation, the BWS in the PCF-FBG is reduced, but very congruous through each stage. This indicates an overall consistent behavior throughout the process. For the three relaxation stages there is a reduction of wavelength back towards the original, culminating in near recovery, falling short by 12 pm. The BWS as a function of small recovery times during the three relaxation stages on average equates to a -39.33 pm shift during each 3 hr period producing on average a rate of 13.70 pm/hr.

The simulated results indicate that there is an increase of strain causing the grating period to increase, which in turn produces a 25.0% increase in the pm/micro strain to 1.5 pm/microstrain, compared to the known standard rate (1.20 pm/micro-strain). This is a strong indication, that during the irradiation period, and after relaxation, there may be a permanent change to the grating period within the FBG. Compared to the results achieved
in stage one with STD FBGs, the PCF-FBG is showing stronger resistance to the gamma exposure and appears more robust, even with limited recovery times. This resistance and robustness to gamma exposure is also highlighted in the temperature sensitivity consistency both pre, post irradiation. When comparing the pm/ °C results from ECU (16.3pm/°C, 16.5pm/°C), there is little temperature sensitivity difference.

When comparing the BWS with temperature drops on site, the three results over the three relaxation periods are 22.2, 25.7, and 32.3 pm/°C (average 26.7pm/°C). Normal temperature sensitivity of PCF-FBGs is approximately ~10.5pm/°C [173] at 1550nm. The increases in sensitivity and therefore recovery aspects also suggests the PCF-FBGs are strong candidates for sensors in the field radiation dosimetry compared to STD-FBGs. The results of spectroscopic analysis and further verification using SAXS and XRD, are highlighted in Chapter 6.

5.3 Irradiation Session Three

The results from irradiation session three are produced by 2 multiplexed PCF-FBGs (base wavelengths 1532.860 nm, and 1541.02 nm). The endlessly single mode pure silica photonic crystal fibre (ESM 12-01) was purchased from Thorlabs and manufactured by BlazePhotonic (NKT). The FBG inscription was again performed at the iPL laboratories at the University of Sydney. The experiment was conducted at ANSTO using the same irradiation facility under the AINSE research award/grant ALNGRA15540 R-2. No interruptions occurred at any stage during session three where each irradiation stage reached 49.35 kGy over 21hrs, equating to 2.35 kGy/hr. After each irradiation (x3) of 21 hours duration, a relaxation stage of 3 hours followed. The actual response and overall cumulative effect during irradiation session three inclusive of the three irradiation and relaxation stages is shown in Figure 5.19, highlighting the PCF-FBG (base wavelength, 1532.860 nm). It tracks the response during the complete session noting the end of each irradiation and relaxation stage. Total recovery was nearly achieved after 148.05 kGy of gamma irradiation and three stages of relaxation.
Figure 5.19: Graph showing PCF-FBG (1532.860 nm) cumulative effect of three irradiation periods inclusive of three relaxation periods. Arrows indicate where the irradiation and relaxation periods ceased. Total BWS 20 pm. Almost full recovery again.

5.3.1 Bragg Wavelength Shift during irradiation

The cumulative total BWS (inclusive of relaxation) of the two PCF-FBGs during session three is shown in Table 5.19. The initial base wavelength (before irradiation) and final wavelength (after the last relaxation stage) of each FBG is highlighted. A breakdown of the results highlighting the BWS not inclusive of the relaxation stages for the three irradiation stages only are shown in Table 5.20.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Base Wavelength</th>
<th>Final Wavelength</th>
<th>Total BWS</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF-FBG</td>
<td>1532.860nm</td>
<td>1532.880nm</td>
<td>20pm</td>
</tr>
<tr>
<td>PCF-FBG</td>
<td>1541.020nm</td>
<td>1541.040nm</td>
<td>20pm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>FBG</th>
<th>Base Wavelength</th>
<th>BWS Stage 1</th>
<th>BWS Stage 2</th>
<th>BWS Stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF-FBG</td>
<td>1532.860nm</td>
<td>50pm</td>
<td>30pm</td>
<td>30pm</td>
</tr>
<tr>
<td>PCF-FBG</td>
<td>1541.020nm</td>
<td>50pm</td>
<td>30pm</td>
<td>30pm</td>
</tr>
</tbody>
</table>
The BWS results were very consistent except during each irradiation dose. It shows there is a slight reduction after the first dose, however there is still a positive shift towards the longer (red) wavelengths (50 pm, 30 pm, 30 pm) during each irradiation phase. Interestingly both multiplexed PCF-FBGs during each irradiation produced identical shifts.

A graphical representation over the three irradiation stages for session three are shown in Figure 5.20, highlighting the shift without relaxation. It can be seen after the initial irradiation shift, the second and third irradiation periods are identical. This result again underscores a solid BWS (50, 30, 30 pm) which is still compatible with standard SMF28+H FBG results obtained by Jing et.al.[72], having a similar accumulated dose of 50 kGy, which produced a 46 pm shift in their first stage and 35 pm shift in their second.

![Figure 5.20: BWS for three irradiation stages for PCF-FBG with 1532.860 nm base. The first irradiation stage (red line) reaches 50 pm before first relaxation stage; BWS second stage (blue line) shows a slight decrease down to 30pm; BWS third stage (green line) also shows a 30 pm shift.]

\[
y = 9.6282\ln(x) + 12.386 \\
R^2 = 0.8948
\]

\[
y = 5.4462\ln(x) + 7.6214 \\
R^2 = 0.8335
\]
The actual FBG reflection peaks showing the initial shift and recovery shift for PCF-FBG (1532.860 nm) are shown in Figure 5.21 for the first irradiation cycle (49.35 kGy). Both FBGs fell short by 20pm from the initial base wavelength. The initial wavelengths and final wavelength culminating in a total BWS inclusive of relaxation of just 20 pm are shown in Table 5.16.

![Wavelength shift (nm)](image)

**Figure 5.21:** Individual FBG reflection peaks for PCF-FBG 1532.860 nm, (a) before irradiation (blue peak), (b) at the end of the first irradiation period (after 49.35 kGy of gamma irradiation) showing a BWS of $\Delta \lambda = 50$ pm (red peak), and (c) at the end of the first 3 hour relaxation period, showing a BWS relaxation of $\Delta \lambda = 30$ pm (green peak).

### 5.3.2 BWS during Relaxation versus Time

All relaxation periods for session three were again conducted over a 3.0 hr. period for continuity. During each relaxation period there was a shift back towards the original wavelength, again to almost full recovery. The relaxation BWS for the three stages are shown in Table 5.21. During the three relaxation stages, the multiplexed PCF-FBGs consistently showed a strong significant recovery, culminating after the second and third irradiation stages in a full recovery. This is definitely larger than that of conventional fibers under similar regimes, and again is in accord with research, that pure silica PCF-FBGs has a superior recovery time [91]. After the first irradiation stage, the FBGs showed identical results in terms of BWS versus gamma dose, and recovery during relaxation.

<table>
<thead>
<tr>
<th>FBG</th>
<th>Base Wavelength</th>
<th>BWS Stage 1</th>
<th>BWS Stage 2</th>
<th>BWS Stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF-FBG</td>
<td>1532.860nm</td>
<td>-30pm</td>
<td>-30pm</td>
<td>-30pm</td>
</tr>
<tr>
<td>PCF-FBG</td>
<td>1541.020nm</td>
<td>-30pm</td>
<td>-30pm</td>
<td>-30pm</td>
</tr>
</tbody>
</table>

Table 5.21: BWS ($\pm$ 5pm) during relaxation for three stages, each of 3 hrs duration
A comparison between the BWS as a function of time during the three relaxation stages of the PCF-FBG (1532.86 nm) is shown in Figure 5.22. The behaviour during the first relaxation (red plots), second (blue plots) and third (green plots) are closely correlated and exhibit a very strong linear relationship shown by the correlation coefficients ($R^2$) $> 0.90$. During the first relaxation stage a rate of 11.4 pm/hr shift was achieved, followed by 10.7 pm/hr for the second, and 10.0 pm/hr for the final relaxation. The results show that there is very little change in the relaxation BWS between each stage. On average there a 10.7 pm/hr rate during the relaxation stages, underlining the consistent behaviour of the PCF-FBGs. During the relaxation periods, the PCF-FBGs repeatedly produced significant recovery to almost the original base wavelength, falling short by 20 pm. The BWS reductions range from -30 pm in the first stage, -30 pm in the second, and -30 pm during the final stage. An overall average equates to a -30 pm shift during each 3 hr period. The consistency and strong stability factors of the PCF-FBGs have been highlighted again.

![Figure 5.22: PCF-FBG (1532.860 nm) BWS during 3 recovery stages as a function of time: first stage (red plot, 3 hrs), second stage (blue plot, 3 hrs), third stage (green plot, 3 hrs)](image)

5.3.3 Temperature effects during relaxation and irradiation

The temperature range in the GATRI chamber and near the PCF-FBGs during the irradiation periods ranged from 20.6 °C to 21.6 °C. Similar to PCF-FBG irradiation session two, the temperature increased rapidly during the first 30 minutes (to within ~
90% of max temperature change), reaching the maximum temperature after approximately one hour, at which point the temperature seemed to stabilize. When factoring the PCF-FBGs known temperature sensitivity of ~10.5 pm/ °C [173], the fictive resultant BWS corresponding to the monitored increase during irradiation would be ~10.5 pm (+1°C ), if temperature was the main cause. However, with the results previously mentioned of shifts between 30 pm and 50 pm, increases of 2.8 °C and 4.7 °C ring irradiation could be expected. With only a 1°C variation throughout irradiation session three, temperature can be discounted as major factor both during the relaxation and irradiation stages. It must be noted due to the setup and fragility near the splice points of the PCF-FBGs, only session one and two was monitored pre-and-post irradiation in relation to BWS v temperature drop during the relaxation periods.

5.3.4 Amplitude variation

The amplitude variations of the PCF-FBGs after the complete gamma exposure of 148.05 kGy are listed in Table 5.22. The overall average amplitude change/ reduction is 2.615 dB. When comparing the average change with the SMF-H FBGs of 1.178 dB and the Ge FBGs of 1.507 dB, it can be seen that the amplitude change is compatible, although slightly higher. The result is significantly larger than the PCF-FBG change of 0.0011 dB during session two.

Table 5.22: Total Amplitude shift (cumulative) after 148.05 kGy of irradiation inclusive of final relaxation period

<table>
<thead>
<tr>
<th>FBG</th>
<th>Reflective power after 148.05 kGy</th>
<th>Reflective power No irradiation</th>
<th>Reflective power Change / dBm</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF-FBG (1532,860nm)</td>
<td>-57.8280</td>
<td>-59.1279</td>
<td>-2.5848</td>
</tr>
<tr>
<td>PCF-FBG (1541.020nm)</td>
<td>-60.4128</td>
<td>-61.7730</td>
<td>-2.6451</td>
</tr>
</tbody>
</table>

5.3.5 Optiwave reconstruction of FBG properties

By varying the values of applied strain from e.g. 0 με (simulated pre-irradiation) to 13.0 με (simulated post irradiation) in Figure 5.23, to the original wavelength grating specs in
the combined reflection profile, the combined profile equal to the wavelength after simulated irradiation was produced. This gives an indication of strain that has been produced during irradiation to cause the BWS, and also highlights the permanent change in grating period after relaxation. The profiles including the reconstructed, are shown in Figure 5.23 (a) (b), reveal a grating period change of (a) 0.529516 μm at 1532.860 nm wavelength to (b) 0.529523 μm at 1532.880 nm. Figure 5.23 (c) shows the matching profile after 13.0 micro strain is added to the original wavelength 1532.860 nm grating to achieve 0.529523 μm. Also shown in Figure 5.24 (a) (b) is the grating period change from (a) 0.532340 μm at 1541.020 nm wavelength to (b) 0.532347μm at 1541.040 nm. Figure 5.24 (c) shows the matching profile after 13.0 micro strain is added to the original wavelength 1541.020nm grating to achieve 0.0532347 μm. The apodization appears to be a similar shape as the original gratings as seen in Figure 5.23 (a), (b), (c) and Figure 5.24 (a) (b) (c). The change to both grating periods equate to an increase of 7pm (0.007 nm) as shown in Table 5.23. Both PCF-FBGs during irradiation produced a final accumulated shift inclusive of relaxation of 20 pm. As can be seen by the optigrating simulation, 13.0 applied micro strain was needed in each to achieve the matching profile.

The established gage factor in standard 125 micron silica fiber with a Bragg wavelength at 1550 nm is known to be 1.2 pm per microstrain applied to the fiber [20]. Through simulation our results show that there has been an increase to 1.538 pm/ micro strain in both FBGs. This is an increase of 28.16% and is a strong indication that during the irradiation period and after relaxation there may be a permanent change to the grating period within the PCF-FBGs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Final Wavelength</th>
<th>Grating Period Start (μm)</th>
<th>Grating Period Final (μm)</th>
<th>Total Change (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF</td>
<td>1532.880nm</td>
<td>0.529516</td>
<td>0.529523</td>
<td>+0.007 nm</td>
</tr>
<tr>
<td>PCF</td>
<td>1541.040nm</td>
<td>0.532340</td>
<td>0.532347</td>
<td>+0.007 nm</td>
</tr>
</tbody>
</table>
Figure 5.23: (a) PCF-FBG 1532.860 nm reflection profile (apodization, shape, length, period) before irradiation simulation. (b) profile of 1532.880 nm simulating after irradiation, both with no strain. (c) reconstructed profile after 13.0 micro strain entered to original 1532.860 nm profile. Apodization is identical in all three.
Figure 5.24: (a) PCF-FBG 1541.020 nm reflection profile (apodization, shape, length, period) before irradiation simulation. (b) profile of 1541.040 nm simulating after irradiation, both with no strain. (c) reconstructed profile after 13.0 micro strain entered to original 1541.020 nm profile. Apodization is identical in all three.
5.3.6 Summary Session 3

In conclusion a Bragg wavelength shift (BWS, $\Delta\lambda_B$) is observed in all three irradiation periods of the same accumulated dose and dose rate. During the first irradiation period, the Bragg wavelength shift (BWS) of both PCF-FBG(1532.860) and PCF-FBG(1541.020nm) was $\Delta\lambda_B = 50$ pm, followed by a relaxation in the BWS of $\Delta\lambda_B = 30$ pm. During the second irradiation period, the BWS was $\Delta\lambda_B = 30$ pm, and the relaxation shift was $\Delta\lambda_B = 30$ pm. The response of the FBG reached the previous total BWS of the first irradiation stage. A similar result was obtained during the third irradiation period, with a BWS shift of $\Delta\lambda_B = 30$ pm and an identical relaxation of $\Delta\lambda_B = 30$ pm. Hence, it appears that there is an initial radiation hardening period for the pre-irradiated FBG that corresponds to a BWS of $\Delta\lambda_B = 20$ pm. After this pre-irradiation hardening, the FBGs both behaved identically, both in comparison to each other, and for repeated identical irradiations. It can be seen after the initial irradiation shift, the second and third irradiation periods are identical. This result again reinforces a solid BWS ($\Delta\lambda_B = 50$, 30, and 30 pm) which is still compatible with standard SMF28+H results with a similar accumulated dose of 50 kGy which produced a 46pm shift in their first stage and 35pm shift in their second stage [72]. With very limited recovery times between irradiation, the BWS in the PCF-FBGs is slightly reduced, but very consistent through each stage. This indicates an overall stable behaviour throughout the process. For the three relaxation periods there is a reduction of wavelength back towards the original culminating in a complete near recovery, falling short by 20 pm. In relaxation periods 2 & 3 there is complete recovery i.e. 30 pm gain and 30 pm reduction in both periods. The BWS as a function of small recovery times during the three relaxation stages, on average equates to a -30 pm shift during each 3 hr period, producing rates of -10.7 pm/hr, -11.4 pm/hr and -10.0 pm/hr.

The simulated results indicate there is an increase of strain causing the grating period to increase. This produces a 28.16% increase of micro strain to $\sim 1.538$ pm/microstrain compared to the known standard rate of $\sim 1.20$ pm/micro-strain. This is strong indication, during irradiation and after relaxation there is a permanent change to the grating period within the FBG. Compared to the results achieved in irradiation session one with STD FBGs, the PCF-FBGs show they are more resistant to the gamma exposure and appear more robust even with limited recovery. The sensitivity achieved however by the pure silica core PCF-FBGs during irradiation is higher than that reported by Y.Xu et al.[94]
especially when comparing to the accumulated dose of 50 kGy. Although the study by Y.Xu et al. used standard pure silica core FBGs, a fairly good comparison can be made especially due to the lack of results re: gamma effects on pure silica solid core PCF-FBGs. We have reported close to 50pm of BWS with a dose rate of ~2.4 kGy/hr compared to 40pm (after 50 kGy) and which saturated after 15 kGy. Although they had a high dose rate of ~10 kGy/hr, which may have contributed to the faster onset of saturation, the distinguishing factor is that the PCF-FBGs with hydrogen loading are maintaining sensitivity, and producing a logarithmic trend even after three irradiation stages.

5.4 Direct Comparison of Results: ESM 12-PCF and Std-SMF28H, GeH, Ge.

A summary of results achieved and previously discussed are provided in Tables 5.24 to Table 5.30 enabling a direct comparison between the STD-FBGs and PCF-FBGs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Base Wavelength</th>
<th>BWS Stage 1</th>
<th>BWS Stage 2</th>
<th>BWS Stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28H</td>
<td>1539.760nm</td>
<td>+140pm</td>
<td>+100pm</td>
<td>+60pm</td>
</tr>
<tr>
<td>GE</td>
<td>1544.880nm</td>
<td>+120pm</td>
<td>+80pm</td>
<td>+80pm</td>
</tr>
<tr>
<td>GE+H</td>
<td>1549.980nm</td>
<td>+100pm</td>
<td>+80pm</td>
<td>+100pm</td>
</tr>
<tr>
<td>SMF28H</td>
<td>1555.020nm</td>
<td>+120pm</td>
<td>+40pm</td>
<td>+80pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1540.806nm</td>
<td>+56pm</td>
<td>+42pm</td>
<td>+32pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1532.860nm</td>
<td>+50pm</td>
<td>+30pm</td>
<td>+30pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1541.020nm</td>
<td>+50pm</td>
<td>+30pm</td>
<td>+30pm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample</th>
<th>Base Wavelength</th>
<th>BWS Stage 1</th>
<th>BWS Stage 2</th>
<th>BWS Stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28H</td>
<td>1539.760nm</td>
<td>-80pm</td>
<td>-80pm</td>
<td>0 pm</td>
</tr>
<tr>
<td>GE</td>
<td>1544.880nm</td>
<td>-60pm</td>
<td>-60pm</td>
<td>-20pm</td>
</tr>
<tr>
<td>GE+H</td>
<td>1549.980nm</td>
<td>-60pm</td>
<td>-80pm</td>
<td>-40pm</td>
</tr>
<tr>
<td>SMF28H</td>
<td>1555.020nm</td>
<td>-80pm</td>
<td>-100pm</td>
<td>-60pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1540.806nm</td>
<td>-40pm</td>
<td>-36pm</td>
<td>-42pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1532.860nm</td>
<td>-30pm</td>
<td>-30pm</td>
<td>-30pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1541.020nm</td>
<td>-30pm</td>
<td>-30pm</td>
<td>-30pm</td>
</tr>
</tbody>
</table>
Table 5.26: Final wavelength after three irradiation stages, inclusive of three relaxation shifts. Total shift = final (pm) ± 5 pm total from base wavelength highlighting recovery aspects

<table>
<thead>
<tr>
<th>Sample</th>
<th>Base Wavelength</th>
<th>Final Wavelength</th>
<th>Total SHIFT</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28H</td>
<td>1539.760nm</td>
<td>1539.940nm</td>
<td>+180pm</td>
</tr>
<tr>
<td>GE</td>
<td>1544.880nm</td>
<td>1545.020nm</td>
<td>+140pm</td>
</tr>
<tr>
<td>GE+H</td>
<td>1549.980nm</td>
<td>1550.140nm</td>
<td>+160pm</td>
</tr>
<tr>
<td>SMF28H</td>
<td>1555.020nm</td>
<td>1555.140nm</td>
<td>+120pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1540.806nm</td>
<td>1540.818nm</td>
<td>+12pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1532.860nm</td>
<td>1532.880nm</td>
<td>+20pm</td>
</tr>
<tr>
<td>PCF</td>
<td>1541.020nm</td>
<td>1541.040nm</td>
<td>+20pm</td>
</tr>
</tbody>
</table>

Table 5.27: BWS v Temp. average change comparison during relaxation both pre- and post irradiation at ECU and onsite ANSTO

<table>
<thead>
<tr>
<th>SAMPLE (LOCATION)</th>
<th>Average pm /°C for Relaxation period</th>
</tr>
</thead>
<tbody>
<tr>
<td>STD-FBGS (ECU) Irradiated</td>
<td>8.3pm/°C</td>
</tr>
<tr>
<td>STD-FBGS (ANSTO) Irradiated</td>
<td>14.8pm/°C</td>
</tr>
<tr>
<td>PCF (ECU) Not Irradiated</td>
<td>16.3pm/°C</td>
</tr>
<tr>
<td>PCF (ECU) Irradiated post ANSTO</td>
<td>16.5pm/°C</td>
</tr>
<tr>
<td>PCF (ANSTO) Irradiated</td>
<td>26.7pm/°C</td>
</tr>
</tbody>
</table>

Table 5.28: Grating period change and Micro-strain to obtain the combined profile equal to the wavelength after simulated irradiation.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Final Wavelength</th>
<th>Grating Period Change (nm)</th>
<th>Micro Strain (με)</th>
<th>pm/με (normal 1.2pm/με)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28H</td>
<td>1539.940nm</td>
<td>+0.062nm</td>
<td>+117.5</td>
<td>1.5352 (+27.9%)</td>
</tr>
<tr>
<td>GE</td>
<td>1545.020nm</td>
<td>+0.049nm</td>
<td>+90.7</td>
<td>1.5440 (+28.6%)</td>
</tr>
<tr>
<td>GE+H</td>
<td>1550.140nm</td>
<td>+0.056nm</td>
<td>+103.5</td>
<td>1.5459 (+28.8%)</td>
</tr>
<tr>
<td>SMF28H</td>
<td>1555.140nm</td>
<td>+0.041nm</td>
<td>+77.2</td>
<td>1.5540 (+29.6%)</td>
</tr>
<tr>
<td>PCF</td>
<td>1540.818nm</td>
<td>+0.004nm</td>
<td>+8.0</td>
<td>1.5000 (+25.0%)</td>
</tr>
<tr>
<td>PCF</td>
<td>1532.880nm</td>
<td>+0.007nm</td>
<td>+13.0</td>
<td>1.5385 (+28.2%)</td>
</tr>
<tr>
<td>PCF</td>
<td>1541.040nm</td>
<td>+0.007nm</td>
<td>+13.0</td>
<td>1.5385 (+28.2%)</td>
</tr>
</tbody>
</table>
Table 5.29: Final Amplitude shift (cumulative) inclusive of relaxation over three irradiation periods: STD-FBGS=199.5 kGy; PCF (1)= 151.8 kGy; PCF (2)(3)=148.05 kGy

<table>
<thead>
<tr>
<th>FBG</th>
<th>Reflective power No irradiation dBm</th>
<th>Reflective power after irradiation With total relaxation</th>
<th>Reflective power Change / dBm</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>SMF28H (1539.76)</em></td>
<td>-23.7901</td>
<td>-24.7785</td>
<td>-0.9884</td>
</tr>
<tr>
<td><em>Ge (1544.88)</em></td>
<td>-25.8794</td>
<td>-27.0414</td>
<td>-1.1620</td>
</tr>
<tr>
<td><em>GeH (1549.98)</em></td>
<td>-26.2741</td>
<td>-28.1262</td>
<td>-1.8521</td>
</tr>
<tr>
<td><em>SMF28H (1555.02)</em></td>
<td>-27.1962</td>
<td>-28.5641</td>
<td>-1.3679</td>
</tr>
<tr>
<td><em>PCF (1540.806)</em></td>
<td>-59.0736</td>
<td>-59.0747</td>
<td>-0.0011</td>
</tr>
<tr>
<td><em>PCF (1532.860)</em></td>
<td>-59.1279</td>
<td>-61.7730</td>
<td>-2.6451</td>
</tr>
<tr>
<td><em>PCF (1541.020)</em></td>
<td>-57.8280</td>
<td>-60.4128</td>
<td>-2.5848</td>
</tr>
</tbody>
</table>

Table 5.30: Average recovery rate (pm/hr.) per stage.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Base Wavelength</th>
<th>Overall average pm /hr. for relaxation stages</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>SMF28H</em></td>
<td>1539.760nm</td>
<td>-16.67 pm/hr.</td>
</tr>
<tr>
<td><em>Ge</em></td>
<td>1544.880nm</td>
<td>-15.77 pm/hr.</td>
</tr>
<tr>
<td><em>GeH</em></td>
<td>1549.980nm</td>
<td>-20.50 pm/hr.</td>
</tr>
<tr>
<td><em>SMF28H</em></td>
<td>1555.020nm</td>
<td>-23.40pm/hr.</td>
</tr>
<tr>
<td><em>PCF</em></td>
<td>1540.806nm</td>
<td>-13.70 pm/hr.</td>
</tr>
<tr>
<td><em>PCF</em></td>
<td>1532.860nm</td>
<td>-10.70 pm/hr.</td>
</tr>
<tr>
<td><em>PCF</em></td>
<td>1541.020nm</td>
<td>-10.70 pm/hr.</td>
</tr>
</tbody>
</table>
CHAPTER 6
Spectroscopic Characterisation (XRD, SAXS)

This chapter describes a direct comparison between results of the standard set and PCF-FBGs using spectroscopic analysis techniques; XRD, SAXS both pre and post irradiation. The effects of gamma irradiation on the properties of PCF-FBGs and STD-FBGs has been demonstrated.

6.1 X-ray Diffraction (XRD):

The structural studies were carried out using a Siemens D5000 x-ray diffractometer using copper K-alpha radiation (wavelength $\lambda=0.154$ nm) for a range of Bragg’s angle $2\theta$ ($20<\theta<50$) at a scan rate of 10°/min. To correctly calculate the sample displacement, 8 peaks (averaged) over the 2theta range were analysed. Results of the XRD analysis include the peak width (FWHM), peak position (2theta), ‘d’-spacing (lattice parameters), lattice strain and average crystallite size, both pre and post irradiation. A typical example of the peak profiles from the Jade 9 XRD analysis software is shown in Figure 6.1. The XRD pattern of the control SMF-FBG shows the characteristic peaks of SiO$_2$ with tetrahedral structure, with the strongest peaks observed at 20 values of 28.34° (peak#1), 38.34° (peak#4), 42.12° (peak#5), and 47.42° (peak#8). The lattice planes that correspond to the peak positions are (111), (201), (211), (113) and are in agreement with the Joint Committee on Powder Diffraction Standards (JCPDS) card no. PDF 00-11-0695.

![Figure 6.1: XRD peak profile pattern from Jade 9 software, for the control SMF-FBG. Eight peaks(#1 to #8) are clearly visible ranging between angles : 20 (20< θ <50).](image)
The primary area of analysis is identifying any variations from pre and post irradiation to crystallite size and d-spacing, which in turn produce lattice strain and an overall broadening of diffraction peaks. This would indicate any permanent structural damage at the crystalline particle level, and help elucidate which FBGs are more conducive for radiation dosimetry.

6.2 Session One: XRD Results : FBGs ; Std-SMF28H, Std-GeH, Std-Ge

The following XRD results obtained are from irradiation session one, using standard FBGs for analysis. The control SMF-FBG has not been irradiated and is produced by the same manufacturer (Alxenses).

6.2.1 XRD Peak position 2theta and Peak Intensity:

The cumulative Peak position 2theta (2θ deg.) change inclusive of the relaxation stage after the three irradiation stages are shown in Table 6.1. Each stage reached an exact dose of 66.5 kGy over 21.3 hours, which equated to a dose rate of 3.12 kGy/hr. The control FBG (not irradiated) was produced in SMF28H fibre, with the exact specifications made by the same manufacturer as the irradiated FBGs, with a central wavelength of 1539.90nm. According to the Bragg Equation (Eqn.4.1), the 2theta peak position (diffraction angle) corresponds to the distance of the reflection plane.

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>2-THETA CONTROL 2θ (deg.)</th>
<th>SMF28H 1539.94nm 2θ (deg.)</th>
<th>SMF28H 1555.14nm 2θ (deg.)</th>
<th>GE 1545.02nm 2θ (deg.)</th>
<th>GE+H 1550.14nm 2θ (deg.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>29.861</td>
<td>29.843</td>
<td>29.860</td>
<td>29.805</td>
<td>29.821</td>
</tr>
<tr>
<td>3</td>
<td>34.920</td>
<td>34.938</td>
<td>34.901</td>
<td>34.940</td>
<td>34.921</td>
</tr>
<tr>
<td>4</td>
<td>38.341</td>
<td>38.381</td>
<td>38.341</td>
<td>38.359</td>
<td>38.341</td>
</tr>
<tr>
<td>5</td>
<td>42.119</td>
<td>42.140</td>
<td>42.119</td>
<td>42.100</td>
<td>42.120</td>
</tr>
<tr>
<td>6</td>
<td>46.040</td>
<td>46.125</td>
<td>46.041</td>
<td>46.081</td>
<td>46.064</td>
</tr>
<tr>
<td>7</td>
<td>46.460</td>
<td>46.424</td>
<td>46.460</td>
<td>46.441</td>
<td>46.481</td>
</tr>
<tr>
<td>8</td>
<td>47.423</td>
<td>47.441</td>
<td>47.441</td>
<td>47.460</td>
<td>47.423</td>
</tr>
</tbody>
</table>
The peak shift indicates that the lattice constant $d$ has changed post irradiation. When the crystallite is strained the $d$ - spacing will be changed. In the case of compressive stress, the $d$–spacing would become smaller whereas when tensile stress is evident, the $d$-spacing becomes larger. Compressive stress leads to higher diffraction angles, whilst tensile stress results in lower diffraction angles. Comparing SMF28H and Ge+H FBGs peak position change, the Germanium doped FBGs produced an average shift change of 0.024°, whilst the standard SMF28H average increased to 0.032°. Overall there is a $2\theta$ change of 0.028° after irradiation within the STD SMF and Ge doped FBGs. The variation of peak shift is small and tends to fluctuate left, right or no movement between the original $2\theta$. The peak intensity variations are shown in Figure 6.2 and 6.3.

![Figure 6.2: XRD peak profile patterns (#1 to #8) of SMF28H FBG before irradiation control (blue line); and after 199.5 kGy inclusive of relaxation time periods (red line).](image)

The intensities of the observed XRD peaks in both the STD-SMF FBGs and Ge-FBGs reduce as a result of gamma irradiation when compared to the control FBG. This is confirmed further by comparing the area of the peaks, both pre-and post-irradiation. Figure 6.4 shows the variation in this diffraction peak area, highlighting a reduction post irradiation. The data points/grouping post-irradiation of the SMF28+H, Ge, Ge+H
FBGs are very similar, resulting in a clear distinction between the control non-irradiated FBG.

Figure 6.3: XRD peak profile patterns of Ge +H FBG before irradiation control (blue line); and after 199.5 kGy inclusive of relaxation time periods (red line).

Figure 6.4: Variation of diffraction peaks (#1 to #8) area, pre- and post irradiation. orange line (SMF) no gamma; blue (SMF-H), Red (Ge), green (Ge+h) after gamma irradiation (199.5 kGy).
This finding may be attributed to a reduction of crystallinity and further induced lattice defects/disorders post irradiation [147,148]. The prominent peak of the phase (100% peak) at ~28.3° (peak no.1) is used to determine the change of crystallinity. The spectrum is the sum of crystalline peaks and an amorphous peak. The true area of the crystalline peaks and the amorphous peak are determined from the Jade 9 XRD Analysis Software, performing a mathematical deconvolution of the peaks. The crystalline percentage of the STD-FBGs pre-and post-irradiation crystallinity can be determined from equation (6.1) [149]. A significant reduction of crystallinity after irradiation in the STD set of FBGs is clearly evident as highlighted in Table 6.2.

\[
\text{%Crystallinity} = \frac{\text{Area under crystalline peaks}}{\text{Total Area under all peaks}} \times 100\% \quad (6.1)
\]

<table>
<thead>
<tr>
<th>Peak</th>
<th>% Crystallinity Pre-Irradiation</th>
<th>% Crystallinity Post-Irradiation</th>
<th>Change in Crystallinity</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF-H</td>
<td>47.8</td>
<td>40.2</td>
<td>-7.6%</td>
</tr>
<tr>
<td>Ge</td>
<td>47.8</td>
<td>37.7</td>
<td>-10.4%</td>
</tr>
<tr>
<td>Ge+H</td>
<td>47.8</td>
<td>37.4</td>
<td>-10.1%</td>
</tr>
</tbody>
</table>

6.2.2 XRD Inter-planar d-spacing

The inter-planar spacing d spacing (Eq.4.2), before and after irradiation as shown in Table 6.3, indicates a correlation between the 2θ position. The average d spacing over 8 peaks gives a more accurate displacement and information about the sample pre-and post-irradiation. The original non-irradiated sample produced a \(d_0\) spacing at a particular 2θ position. After irradiation, the new d spacing fluctuates from \(d > d_0\), \(d < d_0\) and \(d = d_0\) throughout as signified by the +, - and = signs next to the post gamma d spacing columns in Table 6.3. On average there is an average overall d spacing change of 0.0022 angstroms in the STD FBG samples. The variation of d throughout is consistent with non-uniform micro-strain. In summary, the inter-planar distances were only marginally changed after irradiation, which coincides with marginal change of the 2θ peak positions [130].
Table 6.3: d-spacing change after cumulative 199.5 kGy irradiation, inclusive of relaxation shifts.

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>d0- Spacing</th>
<th>SMF28H 1539.94nm</th>
<th>SMF28H 1555.14nm</th>
<th>GE 1545.02nm</th>
<th>GE+H 1550.14nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.1467</td>
<td>3.1467 =</td>
<td>3.1468 +</td>
<td>3.1466 -</td>
<td>3.1468 +</td>
</tr>
<tr>
<td>2</td>
<td>2.9897</td>
<td>2.9915 +</td>
<td>2.9898 +</td>
<td>2.9952 +</td>
<td>2.9936 +</td>
</tr>
<tr>
<td>3</td>
<td>2.5673</td>
<td>2.5660 -</td>
<td>2.5686 +</td>
<td>2.5659 -</td>
<td>2.5672 -</td>
</tr>
<tr>
<td>4</td>
<td>2.3457</td>
<td>2.3434 -</td>
<td>2.3458 +</td>
<td>2.3447 -</td>
<td>2.3831 +</td>
</tr>
<tr>
<td>5</td>
<td>2.1437</td>
<td>2.1427 -</td>
<td>2.1437 =</td>
<td>2.1446 +</td>
<td>2.1436 +</td>
</tr>
<tr>
<td>6</td>
<td>1.9698</td>
<td>1.9664 -</td>
<td>1.9698 =</td>
<td>1.9682 -</td>
<td>1.9698 =</td>
</tr>
<tr>
<td>7</td>
<td>1.9530</td>
<td>1.9544 +</td>
<td>1.9530 =</td>
<td>1.9537 +</td>
<td>1.9521 -</td>
</tr>
<tr>
<td>8</td>
<td>1.9155</td>
<td>1.9148 -</td>
<td>1.9149 -</td>
<td>1.9141 -</td>
<td>1.9155 =</td>
</tr>
</tbody>
</table>

6.2.3 XRD FWHM (Full Width Half Maximum)

The FWHM of the diffraction peaks after the cumulative dose, inclusive of relaxation is tabulated in Table 6.4. With the identified non-uniform micro-strain, the peak width should have a broadening effect present. The pre-irradiation average FWHM of the control sample is 0.285°. When analysing the post irradiation peaks of the four samples, there is a slight broadening increased average of 0.286°. Even though this is slight, it is consistent and is in keeping with the non-uniform micro-strain indicated by the varying d spacing throughout. Lattice disorders, reduction of crystallinity and particle size are the main reasons for the broadening of the XRD peaks [150].

In the case of isotropic microstrain, the FWHM remains a monatomic function; i.e. the FWHM reflections increase with the diffraction angle [150]. If however there is a fluctuation of the FWHM with an increase of diffraction angle, anisotropic broadenings are observed [150,151]. Anisotropic stresses may also arise due to formations of regions in a fibre due to doping of silica and FBG inscription [152,153,166]. Results indicate that there is variation of FWHM with the increasing peak angle both pre-and-post irradiation. The anisotropy of the widths are clearly seen in Figure 6.5.
Table 6.4: FWHM change after cumulative 199.5 kGy irradiation, inclusive of relaxation shifts.

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>FWHM CONTROL degrees</th>
<th>SMF28H 1539.94nm</th>
<th>SMF28H 1555.14nm</th>
<th>GE 1545.02nm</th>
<th>GE+H 1550.14nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.214</td>
<td>0.209</td>
<td>0.210</td>
<td>0.204</td>
<td>0.218</td>
</tr>
<tr>
<td>2</td>
<td>0.176</td>
<td>0.167</td>
<td>0.207</td>
<td>0.138</td>
<td>0.245</td>
</tr>
<tr>
<td>3</td>
<td>0.229</td>
<td>0.242</td>
<td>0.236</td>
<td>0.227</td>
<td>0.195</td>
</tr>
<tr>
<td>4</td>
<td>0.322</td>
<td>0.332</td>
<td>0.317</td>
<td>0.384</td>
<td>0.345</td>
</tr>
<tr>
<td>5</td>
<td>0.289</td>
<td>0.232</td>
<td>0.258</td>
<td>0.311</td>
<td>0.271</td>
</tr>
<tr>
<td>6</td>
<td>0.455</td>
<td>0.463</td>
<td>0.353</td>
<td>0.485</td>
<td>0.374</td>
</tr>
<tr>
<td>7</td>
<td>0.325</td>
<td>0.325</td>
<td>0.357</td>
<td>0.356</td>
<td>0.353</td>
</tr>
<tr>
<td>8</td>
<td>0.271</td>
<td>0.253</td>
<td>0.396</td>
<td>0.259</td>
<td>0.259</td>
</tr>
</tbody>
</table>

Figure 6.5: Variation of the FWHM. Orange line (SMF) no gamma; Blue (SMF-H), Red (Ge), Green (Ge+h) after gamma irradiation. The anisotropy of the widths of the peaks is clearly evident.

6.2.4 XRD Crystallite Average Size

Table 6.5 compares the average crystallite size throughout the samples using the Scherrer Equation (Eq.4.3). The pre-irradiation overall average crystallite size in the STD- FBGS is 315.940 Å (31.594 nm). Post gamma there is very little change in size to 319.225 Å (31.922 nm). Normally a decrease in crystallite size is indicative of peak broadening. However, due to the non-uniform strain present, and the comparisons made with the FWHM, the result seems consistent as far as the relationship between the very
slight increase and very slight broadening effect. The increase/change in size on average is 0.328 nm or 1.04%, which is primarily caused by gamma exposure.

Table 6.5: Average Crystallite size after cumulative 199.5 kGy irradiation, inclusive of relaxation shifts.

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>AV Crystallite size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Control</td>
</tr>
<tr>
<td>1</td>
<td>39.10</td>
</tr>
<tr>
<td>2</td>
<td>47.58</td>
</tr>
<tr>
<td>3</td>
<td>36.69</td>
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<tr>
<td>4</td>
<td>26.15</td>
</tr>
<tr>
<td>5</td>
<td>29.22</td>
</tr>
<tr>
<td>6</td>
<td>16.62</td>
</tr>
<tr>
<td>7</td>
<td>26.08</td>
</tr>
<tr>
<td>8</td>
<td>31.31</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>SMF28H 1539.94nm</th>
<th>SMF28H 1555.14nm</th>
<th>GE 1545.02nm</th>
<th>GE+H 1550.14nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMF28H</td>
<td>40.04</td>
<td>39.85</td>
<td>41.02</td>
<td>38.38</td>
</tr>
<tr>
<td>SMF28H</td>
<td>50.15</td>
<td>40.46</td>
<td>60.68</td>
<td>34.18</td>
</tr>
<tr>
<td>SMF28H</td>
<td>34.72</td>
<td>35.60</td>
<td>37.01</td>
<td>43.08</td>
</tr>
<tr>
<td>SMF28H</td>
<td>25.37</td>
<td>26.57</td>
<td>21.93</td>
<td>24.41</td>
</tr>
<tr>
<td>SMF28H</td>
<td>36.41</td>
<td>32.74</td>
<td>27.16</td>
<td>31.17</td>
</tr>
<tr>
<td>SMF28H</td>
<td>18.30</td>
<td>24.01</td>
<td>17.47</td>
<td>22.66</td>
</tr>
<tr>
<td>SMF28H</td>
<td>26.08</td>
<td>23.75</td>
<td>23.81</td>
<td>24.02</td>
</tr>
</tbody>
</table>

6.2.5 XRD Microstrain

Micro strain ($\mu\varepsilon$) can be linked to crystallite size and FWHM. As previously mentioned, there is a slight increase in crystallite size post gamma in STD-FBGs. Table 6.6 highlights the average $\mu\varepsilon$ within the lattice. Pre irradiation results indicate an average of 0.00680 $\mu\varepsilon$, whilst after gamma exposure the average lattice $\mu\varepsilon$ increases to 0.00729. Microstrain increases in this instance slightly by 0.00049 or 7.21%. Normally with an increase in particle size the $\mu\varepsilon$ would decrease. However, as seen in Table 6.5, the crystallite size increase is only marginal at best (0.328 nm). The result shows there is a consistency with the small increase in Crystallite Size and little or no change in lattice strain.

Table 6.6: Resultant microstrain after cumulative 199.5 kGy irradiation, inclusive of relaxation shifts

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>Lattice strain Control $\mu\varepsilon$</th>
<th>SMF28H 1539.94nm</th>
<th>SMF28H 1555.14nm</th>
<th>GE 1545.02nm</th>
<th>GE+H 1550.14nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0075</td>
<td>0.0073</td>
<td>0.0074</td>
<td>0.0072</td>
<td>0.0077</td>
</tr>
<tr>
<td>2</td>
<td>0.0059</td>
<td>0.0056</td>
<td>0.0069</td>
<td>0.0068</td>
<td>0.0082</td>
</tr>
<tr>
<td>3</td>
<td>0.0065</td>
<td>0.0069</td>
<td>0.0067</td>
<td>0.0064</td>
<td>0.0055</td>
</tr>
<tr>
<td>4</td>
<td>0.0083</td>
<td>0.0086</td>
<td>0.0082</td>
<td>0.0099</td>
<td>0.0089</td>
</tr>
<tr>
<td>5</td>
<td>0.0068</td>
<td>0.0054</td>
<td>0.0061</td>
<td>0.0073</td>
<td>0.0064</td>
</tr>
<tr>
<td>6</td>
<td>0.0097</td>
<td>0.0099</td>
<td>0.0076</td>
<td>0.0104</td>
<td>0.0080</td>
</tr>
<tr>
<td>7</td>
<td>0.0069</td>
<td>0.0069</td>
<td>0.0076</td>
<td>0.0076</td>
<td>0.0075</td>
</tr>
<tr>
<td>8</td>
<td>0.0056</td>
<td>0.0053</td>
<td>0.0082</td>
<td>0.0054</td>
<td>0.0054</td>
</tr>
</tbody>
</table>
6.3 Irradiation Session Two & Three: XRD Results: PCF-FBGs; ESM 12-PCF

The following XRD results obtained are from irradiation session two and three, using the PCF-FBGs for analysis. The control PCF-FBG has not been irradiated. A summary and comparison between the XRD results of the STD and PCF-FBGs is given at the end of this chapter.

6.3.1 XRD Peak position 2theta and Peak Intensity:

The cumulative peak position 2theta (2θ deg.) change, inclusive of the relaxation periods, for the three irradiation periods are shown in Table 6.7. For session two, an exact dose of 50.6 kGy over three 21.0 hour periods was reached, which equated to a dose rate of 2.41 kGy/hr. During session three, the dose reached 49.35 kGy also over three 21.0 hr periods. The control PCF-FBG (not irradiated) was made with the exact specifications at iPL, The University of Sydney with a central wavelength of 1541.00nm. According to Bragg Equation (Eqn.4.1) the 2theta peak position (diffraction angle) corresponds to the distance of the reflection plane. The average peak shifts post-irradiation compared to the control non-irradiated PCF-FBG is 0.023 degrees.

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>2-THETA Control 2θ (deg.)</th>
<th>151.8 kGy PCF-FBG 1540.806nm 2θ (deg.)</th>
<th>148.05 kGy PCF-FBG 1532.860nm 2θ (deg.)</th>
<th>148.05 kGy PCF-FBG 1541.020nm 2θ (deg.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>29.913</td>
<td>29.822</td>
<td>29.862</td>
<td>29.825</td>
</tr>
<tr>
<td>3</td>
<td>34.938</td>
<td>34.938</td>
<td>34.937</td>
<td>34.937</td>
</tr>
<tr>
<td>4</td>
<td>38.340</td>
<td>38.378</td>
<td>38.340</td>
<td>38.339</td>
</tr>
<tr>
<td>5</td>
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<td>42.119</td>
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<td>42.082</td>
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<tr>
<td>6</td>
<td>46.061</td>
<td>46.059</td>
<td>46.041</td>
<td>46.119</td>
</tr>
<tr>
<td>7</td>
<td>46.461</td>
<td>46.460</td>
<td>46.441</td>
<td>46.461</td>
</tr>
<tr>
<td>8</td>
<td>47.479</td>
<td>47.441</td>
<td>47.441</td>
<td>47.421</td>
</tr>
</tbody>
</table>

The peak shift indicates again that the lattice constant $d$ has changed post-irradiation. The variation of peak shift is very small and tends to fluctuate left, right or no movement between the original 2θ as in the STD-FBGs. The intensities of the observed XRD peaks in the PCF-FBGs reduce slightly as a result of gamma irradiation when compared to the control FBG, as shown in Figure 6.6. This is confirmed further by comparing the area of the peaks, both pre-and post-irradiation. Figure 6.7 shows the variation in diffraction peak...
area, accentuating a small reduction post irradiation by two PCF-FBGs, with one showing an increase. Interestingly the PCF-FBG from session two has a very similar diffraction peak area post irradiation.

![Figure 6.6: XRD peak profile patterns (#1 to #8) of PCF-FBG before irradiation control (blue line); and after 151.8 kGy inclusive of relaxation time periods (red line).](image)

![Figure 6.7: Variation of diffraction peaks (#1 to #8) area pre and post irradiation. Orange line (PCF) no gamma; Blue (PCF session two), Red (PCF session three), Green (PCF session three) after gamma irradiation.](image)
The smaller reduction of crystallinity, compared to the STD set, signifies there are less induced lattice defects/disorders post-irradiation. The prominent peak of the phase (100% peak) at ~28.3° is used to determine the change of crystallinity. The crystalline percentage of the PCF-FBGs pre-and post-irradiation crystallinity can be determined from equation (6.1). The very small change of crystallinity after irradiation in the PCF-FBGs is tabulated in Table 6.8. Interestingly there is only a slight reduction (and even an increase in the first FBG of session three when compared with the STD–FBGs in Table 6.1.

Table 6.8: Change in crystallinity post irradiation

<table>
<thead>
<tr>
<th>2θ (28.32) Peak</th>
<th>% Crystallinity Pre-Irradiation</th>
<th>% Crystallinity Post-Irradiation</th>
<th>Change in Crystallinity</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCF session two</td>
<td>40.7</td>
<td>38.8</td>
<td>-1.9%</td>
</tr>
<tr>
<td>PCF session three</td>
<td>40.7</td>
<td>48.3</td>
<td>+7.6%</td>
</tr>
<tr>
<td>PCF session three</td>
<td>40.7</td>
<td>37.4</td>
<td>-2.1%</td>
</tr>
</tbody>
</table>

6.3.2 XRD inter-planar d-spacing

The inter-planar spacing $d$ spacing before and after irradiation as shown in Table 6.9, indicates a correlation between the $2\theta$ positions. The average ‘$d$’ spacing over 8 peaks gives a more accurate displacement and information about the sample pre and post irradiation. The original non-irradiated sample produced a $d_0$ spacing at a particular $2\theta$ position as shown in the control column in Table 6.9. After irradiation, the new $d$ spacing fluctuates from $d$ throughout as signified by the +, - and = signs next to the post gamma $d$ spacing columns in Table 6.9. There is an average overall $d$ spacing change of 0.0016 angstroms in the PCF-FBG samples. The variation of $d$ throughout is consistent with non-uniform micro-strain. The inter-planar distances only marginally changed throughout because the $2\theta$ peak position varied slightly.
Table 6.9: XRD d spacing distance after cumulative 151.8 & 148.05 kGy irradiation dose, inclusive of relaxation shifts

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>(d_0) spacing</th>
<th>Control Angstroms</th>
<th>151.8 kGy PCF-FBG 1540.808nm</th>
<th>148.05 kGy PCF-FBG 1532.860nm</th>
<th>148.05 kGy PCF-FBG 1541.020nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.1488</td>
<td>3.1487 -</td>
<td>3.1488 =</td>
<td>3.1467 -</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>2.9846</td>
<td>2.9935 +</td>
<td>2.9896 +</td>
<td>2.9933 +</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>2.5660</td>
<td>2.5660 =</td>
<td>2.5661 +</td>
<td>2.5661 +</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>2.3458</td>
<td>2.3435 -</td>
<td>2.3458 =</td>
<td>2.3458 =</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>2.1446</td>
<td>2.1436 -</td>
<td>2.1445 -</td>
<td>2.1455 +</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>1.9689</td>
<td>1.9690 +</td>
<td>1.9697 +</td>
<td>1.9666 -</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>1.9529</td>
<td>1.9530 -</td>
<td>1.9537 +</td>
<td>1.9529 =</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>1.9134</td>
<td>1.9148 +</td>
<td>1.9148 +</td>
<td>1.9156 +</td>
<td></td>
</tr>
</tbody>
</table>

6.3.3 XRD FWHM (Full Width Half Maximum)

The FWHM after the cumulative dose, inclusive of relaxation, is listed in Table 6.10. With the identified non-uniform micro-strain, the peak width should have a broadening effect present. Comparing the pre-irradiation control sample the overall FWHM is 0.329 degrees. When analysing the post-irradiation peaks there is a reduction or narrowing of FWHM down to 0.291 degrees. The reduction is consistent with the crystallite size in Table 6.11. The FWHM change is due to micro-strain, non-uniform strain and the change to crystallite size. Larger crystals produce narrower peaks, as is present in the PCF results. As stated previously, lattice disorders, reduction of crystallinity and particle size are the main reasons for the broadening of the XRD peaks [150]. There is an anisotropic effect also present in the PCF-FBGs due to the fluctuation widths of the FWHM as seen in Figure 6.8. However, due to larger crystallite size post-irradiation, the broadening effect is nullified, as seen by the very small reduction of crystallinity (seen in Table 6.7), peak intensity (seen in Figure 6.7) and area change (seen in Figure 6.8).

Table 6.10: FWHM change after cumulative 151.8 & 148.05 kGy irradiation dose, inclusive of relaxation shifts

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>FWHM Control Degrees</th>
<th>151.8 kGy PCF-FBG 1540.806nm</th>
<th>148.05 kGy PCF-FBG 1532.860nm</th>
<th>148.05 kGy PCF-FBG 1541.020nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.233</td>
<td>0.211</td>
<td>0.223</td>
<td>0.225</td>
</tr>
<tr>
<td>2</td>
<td>0.407</td>
<td>0.349</td>
<td>0.295</td>
<td>0.265</td>
</tr>
<tr>
<td>3</td>
<td>0.234</td>
<td>0.213</td>
<td>0.234</td>
<td>0.197</td>
</tr>
<tr>
<td>4</td>
<td>0.304</td>
<td>0.353</td>
<td>0.331</td>
<td>0.338</td>
</tr>
<tr>
<td>5</td>
<td>0.284</td>
<td>0.211</td>
<td>0.289</td>
<td>0.253</td>
</tr>
<tr>
<td>6</td>
<td>0.591</td>
<td>0.319</td>
<td>0.565</td>
<td>0.331</td>
</tr>
<tr>
<td>7</td>
<td>0.326</td>
<td>0.307</td>
<td>0.332</td>
<td>0.337</td>
</tr>
<tr>
<td>8</td>
<td>0.258</td>
<td>0.262</td>
<td>0.291</td>
<td>0.274</td>
</tr>
</tbody>
</table>
6.3.4 XRD Crystallite average size

Table 6.11 emphasizes the average crystallite size throughout the samples using the Scherrer Equation (Eq.4.3). The pre irradiation overall average crystallite size in the PCF-FBGs is 278.825 Angstroms (27.882 nm). Post gamma, there is an increase in crystallite size to 304.658 Angstroms (30.465 nm). The increase in size is usually indicative of FWHM and peak narrowing, which is confirmed in section 6.3.3. The increase in size on average is 2.583nm or 9.3%, which is primarily caused by gamma exposure.

Table 6.11: Average Crystallite size after cumulative 151.8 & 148.05 kGy, inclusive of relaxation shifts

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>AV Crystallite size (nm)</th>
<th>151.8 kGy PCF-FBG 1540.806nm</th>
<th>148.05 kGy PCF-FBG 1532.860nm</th>
<th>148.05 kGy PCF-FBG 1541.020nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>35.91</td>
<td>39.66</td>
<td>37.52</td>
<td>37.19</td>
</tr>
<tr>
<td>2</td>
<td>20.58</td>
<td>24.00</td>
<td>28.39</td>
<td>31.60</td>
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<tr>
<td>3</td>
<td>35.90</td>
<td>39.44</td>
<td>35.90</td>
<td>42.65</td>
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<tr>
<td>4</td>
<td>27.70</td>
<td>23.86</td>
<td>25.44</td>
<td>24.92</td>
</tr>
<tr>
<td>5</td>
<td>29.74</td>
<td>40.03</td>
<td>29.22</td>
<td>33.38</td>
</tr>
<tr>
<td>6</td>
<td>14.34</td>
<td>26.57</td>
<td>15.00</td>
<td>25.60</td>
</tr>
<tr>
<td>7</td>
<td>26.00</td>
<td>27.61</td>
<td>25.53</td>
<td>25.16</td>
</tr>
<tr>
<td>8</td>
<td>32.89</td>
<td>32.38</td>
<td>29.16</td>
<td>30.97</td>
</tr>
</tbody>
</table>
6.3.5 XRD Microstrain

Micro strain (µε) can be linked to crystallite size and FWHM. As previously mentioned there is a large increase in crystallite size post gamma in PCF-FBGs. Table 6.12 highlights the average lattice strain. Pre irradiation results indicate an average of 0.00872, whilst after gamma exposure the average lattice strain decreases to 0.00743. Lattice strain decreases with an increase in particle size. The result shows that the PCF increased crystallite size after irradiation results in strain throughout the lattice.

Table 6.12: Resultant lattice strain after cumulative 151.8 & 148.05 kGy irradiation dose, inclusive of relaxation shifts

<table>
<thead>
<tr>
<th>PEAK #</th>
<th>Lattice strain Control</th>
<th>151.8 kGy PCF-FBG 1540.806nm</th>
<th>148.05 kGy PCF-FBG 1532.860nm</th>
<th>148.05 kGy PCF-FBG 1541.020nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0082</td>
<td>0.0074</td>
<td>0.0078</td>
<td>0.0079</td>
</tr>
<tr>
<td>2</td>
<td>0.0135</td>
<td>0.0116</td>
<td>0.0098</td>
<td>0.0088</td>
</tr>
<tr>
<td>3</td>
<td>0.0066</td>
<td>0.0060</td>
<td>0.0066</td>
<td>0.0056</td>
</tr>
<tr>
<td>4</td>
<td>0.0079</td>
<td>0.0091</td>
<td>0.0086</td>
<td>0.0087</td>
</tr>
<tr>
<td>5</td>
<td>0.0067</td>
<td>0.0050</td>
<td>0.0068</td>
<td>0.0059</td>
</tr>
<tr>
<td>6</td>
<td>0.0127</td>
<td>0.0068</td>
<td>0.0121</td>
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</tr>
<tr>
<td>7</td>
<td>0.0069</td>
<td>0.0065</td>
<td>0.0071</td>
<td>0.0070</td>
</tr>
<tr>
<td>8</td>
<td>0.0054</td>
<td>0.0054</td>
<td>0.0060</td>
<td>0.0057</td>
</tr>
</tbody>
</table>


A summary of results achieved and previously discussed are provided in Table 6.13 which provides an instant direct comparison between the STD-FBGs and PCF-FBGs. The results in brackets are pre-irradiation values.

Table 6.13 Direct Comparison of Results between STD-FBGs and PCF-FBGs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Av PEAK shifts 2θ Post Gamma</th>
<th>Av d Spacing Angstroms Post Gamma</th>
<th>Av FWHM Degree Post Gamma</th>
<th>Av Crystallite size angstrom Post Gamma</th>
<th>Lattice Strain Post Gamma</th>
</tr>
</thead>
<tbody>
<tr>
<td>STD – FBGS</td>
<td>0.028</td>
<td>0.0022 Å</td>
<td>0.286 (0.285 Pre)</td>
<td>319.225 Å (315.940 Pre)</td>
<td>0.00729 (0.00680 Pre)</td>
</tr>
<tr>
<td>PCF-FBGs</td>
<td>0.023</td>
<td>0.0016 Å</td>
<td>0.291 (0.329 Pre)</td>
<td>304.658 Å (278.825 Pre)</td>
<td>0.00743 (0.00872 Pre)</td>
</tr>
</tbody>
</table>
6.5 Summary XRD

Table 6.13 shows a direct comparison of results pre-and post-irradiation between the STD-FBGs and PCF-FBGs. A reduction of FWHM is seen in the PCF-FBGs after gamma irradiation. A decreasing FWHM reflects the decreasing lattice imperfections (structural defects), and consequently an increase in the average crystallite size in PCF after exposure (average 27.8825 nm before irradiation and 30.4658 nm after). A reduction of FWHM also indicates a reduction of strain/stress in the PCF, indicating strong recovery and radiation resistance. The STD-FBGs remain fairly constant in the FWHM both pre and post irradiation. This also coincides with consistency of the average crystallite size remaining unaffected.

It indicates the STD-FBGs recovery aspects are reduced compared to the PCF-FBGs, indicating more pronounced permanent damage to the structure. Lattice strain decreases with an increase in particle size. Therefore, slight displacements of atoms relative to their normal lattice positions normally imposed by crystalline defects are either reduced or increased. In the case of the PCF-FBG, the lattice strain reduced from 0.00872 (no gamma) to 0.00743.

This shows that crystalline defects are less prevalent in PCF-FBGs, indicating a stronger resistance to Gamma irradiation and improved structural integrity. This is confirmed also by the change of crystallinity pre- and post-irradiation. The STD-FBGs resulted in a large average decrement of crystallinity of -9.4 % after gamma irradiation, compared with the PCF-FBGs, which showed less decrement on average of -2.0 %. In one of the PCF-FBGs, there was an actual increase in crystallinity of +7.6 %. The intensity of diffraction peaks also confirm the variations of crystallinity. The large decrease in peak intensity of the STD-FBGs suggest an evolution toward a more disordered state post-irradiation.

Results for d spacing and 2-theta positions, when compared to the control FBGs, within the STD-FBGs and PCF-FBGs indicate there is a similar interatomic spacing occurring between planes giving an almost similar 2θ peak shift. Therefore, the FWHM and crystallite size pre-and post-irradiation is the main factor with respect to micro-strain. In conclusion, the XRD spectra show the lattice parameters are modified post-irradiation. The ionising radiation is inducing a significant change in crystallinity, particularly in the
STD set of FBGs. The larger reduction is consistent with a metamict phase after irradiation [161]. Analysis of the crystallite size shows the PCF-FBGs increase is large compared to the STD-FBGs. This results in a more disordered system post-irradiation in the STD set compared to the more ordered state in the PCF-FBGs. Anisotropic behaviour is prevalent in both sets pre- and post-irradiation however, the scatter effect is less pronounced post-irradiation in the PCF-FBGs, again emphasizing more order in the system and less of a metamict state than the STD-FBGs.

6.6 Small Angle X-ray Scattering (SAXS)

The small angle x-ray scattering (SAXS) analysis is carried out using a Bruker NanoStar with a high intensity gallium MetalJet X-ray source. This study investigates the effects of gamma irradiation on the shape, size, volume fraction and distribution of particles in the silica based optical FBGs. To correctly calculate and interpret results, a program from SASfit [154] (February 23, 2017) was used. This program accommodates fitting elementary structural models to small angle scattering data. It can fit size distributions with several form factors, including different structural factors. The scattering intensity is compared to see if there are distinct differences between the PCF-FBG and STD-FBGs.

It is known that larger particles scatter x-rays more strongly than small particles. SAXS can measure shapes, and sizes of nanoparticles and large molecules. Also incorporated are results of any fractal–like material which may indicate permanent radiation damage. Each sample was scanned over a period of 3600 secs. (1hr). The scattering angle range is from $0.1 < 2\theta < 5.0$ degrees. The size and shape of the particles or form factor $P(q)$ is recorded. Functions of $q$ are the length of the scattering vector. The units of the scattering vector $q$ are in reciprocal length-units e.g. if the wavelength of the radiation used is entered in Angstrom, $q$ has the units of Å$^{-1}$ [128]. The following results show the scattering patterns of the STD-FBG and PCF-FBGs both pre and post irradiation.

6.7 SAXS Results : FBGs ; Std-SMF28H, Std-GeH, Std-Ge

The SAXS results exemplify the scattering patterns of the STD-FBGs both pre and post irradiation. An increase of scattering intensity represents an increase in size and/or concentration of particles. Most large dimensions occur at small scattering angles and
smaller dimensions at large scattering angles. The data obtained indicates the shape of the particles in the standard SMF FBGs to be hollow cylindrical. The curves were fitted using SASfit for the size distribution and form factors (long cylinder shell model) configuration as shown in Figure 6.9. On the right half of the panel consisting of a form factor, it is indicating a long cylinder shell [155]. The left column tabulates the concentration distribution parameter ‘N,’ which denotes the number of scattering objects involved in the measurement, and the best fit value for the mean radius of the particle which is highlighted in ‘mu’. The long cylinder shell configuration also indicates the shell thickness DR; and cylinder length L. A schematic of the shape of the long cylinder shell and expected scattering intensity profiles are shown in Figure 6.10.

A major consideration for using SAXS is to attempt to ascertain whether or not there is any post irradiation damage. Particles could have fractal (rough) surfaces, or they could have a mass fractal structure through clustering. When analyzing fractal anomalies, SASfit uses the unified exponential power law according to Beacauge [156,157].
This approach describes scattering from multiple size scale structures over $q$ ranges of many orders of magnitude without introducing new parameters other than those used in local fits [157]. The results indicate a large scale structure and, in relation to Porod’s law, $P$, which is the scaling exponent of the power law assigned to the larger structures. Fractals can be categorised into either mass fractals or surface fractals. Generally for surface fractals $4 > P > 3$, and for mass fractals $P < 3$ [158,165]. At the high $(q)$, domain the Porod’s region gives information about the surfaces. 

Typically, in small angle x-ray scattering, the intensity is plotted versus $q$ in a range from $q_{\text{min}}$ to $q_{\text{max}}$. In the low $q$ domain, where the observation window is very large, the so-called Structure Factor $S(q)$ can be obtained. In the intermediate zone, the form factor $P(q)$ can be measured, which includes the size, shape, and internal structure of one particle. At low values $q$ the scattering plots present a region where the intensity can be described by a simple power-equation[168]:

$$I(q) = A/q^d + \text{background} \quad (6.2)$$

where $d$ is related to the fractal dimension (surface fractals $4 > P > 3$, and for mass fractals $P < 3$).

### 6.7.1 Session one  SAXS Scattering intensity patterns

The experimental scattering patterns and data fitting via SASfit both pre-and post-irradiation are shown in Figures 6.11, 6.12, and 6.13 for the STD FBGs. Figure 6.14
highlights that all STD-FBGs show an increase in scattering intensity patterns compared to the control FBG.

Figure 6.11: (a) Experimental Scattering pattern of Control SMF+H with FBG (1539.90nm) no GAMMA (b) applied SASfit pattern (red) plot versus experimental blue plot; profile extracted from SASfit.
Figure 6.12: (a) Experimental Scattering pattern SMF+H with FBG (1539.94nm) after 199.5 kGy GAMMA (b) applied SASfit pattern (red) plot versus experimental blue plot.
Figure 6.13: (a) Experimental Scattering pattern of Ge with FBG (1545.02nm) 199.5 kGy GAMMA (b) applied SASfit pattern (red) plot versus experimental blue plot.
Figure 6.14: Experimental combined scattering pattern profiles of all STD-FBGs after 199.5 kGy of Gamma irradiation. The blue pattern is the control (no gamma).

6.7.2 Summary STD-FBGs SAXS

As shown in Figure 6.14, a comparison between the scattering intensity of the control FBG (blue profile) and irradiated samples can be identified. The yellow and black profiles are from the Ge+H and Ge FBGs, respectively. The green profile and red profiles are from the SMF+H FBGs. This is after the final recovery period and 199.5 kGy accumulated dose. All irradiated scattering intensities are higher than the control FBG. This is exemplified also by the increasing particle number concentration pre-and post-irradiation, as shown in Table 6.14. There is on average a 42.87 % increase of number concentration after irradiation. Results for the types of fractals present, size, shape, pre- and post-irradiation are also shown in Table 6.14. Figure 6.15 shows the 2D scattering intensity profiles obtained from SASfit, pre and post irradiation. The profile shows that after irradiation, the scattering intensity has changed and the anisotropic effect is quite pronounced. It is known the drawing process in the production of optical fibres induces anisotropic stress[119]. This may lead also to anisotropic behavior when they compact under irradiation as mentioned previously in section 5.1.6 [119]. A comparative analysis between the PCF-FBGs and STD-FBGs is in the final summary section 6.10.
Table 6.14: Resulting parameters obtained after SAXS fitting for STD-FBG pre irradiation and after 199.5 kGy irradiation dose, inclusive of relaxation shifts.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Std-FBG Control No irradiation</th>
<th>SMF –H FBGs after 199.5 kGy</th>
<th>Ge +H FBGs after 199.5 kGy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Radius of Particle</td>
<td>23.5277 Å</td>
<td>23.2772 Å</td>
<td>23.9934 Å</td>
</tr>
<tr>
<td>Particle Concentration /cm</td>
<td>6.29483×10^{31} cm^{-3}</td>
<td>9.31088×10^{31} cm^{-3}</td>
<td>8.6767×10^{31} cm^{-3}</td>
</tr>
<tr>
<td>Length of Cylinder</td>
<td>3000 Å</td>
<td>3000 Å</td>
<td>3000 Å</td>
</tr>
<tr>
<td>P factor</td>
<td>3.27285</td>
<td>3.09698</td>
<td>2.97893</td>
</tr>
<tr>
<td>Fractal Nature</td>
<td>Surface</td>
<td>Surface</td>
<td>Surface</td>
</tr>
</tbody>
</table>

Figure 6.15: Combined 2D scattering intensity profile of STD-FBGs. (top left image) STD SMF-FBG no irradiation; (top right) STD SMF-FBG after 199.5 kGy; and (bottom image) Ge-FBG after 199.5 kGy.
6.8 Session Two and Three SAXS Results: PCF-FBGs; \textit{ESM 12-PCF}

The experimental scattering patterns and data fitting via SASfit, both pre- and post-irradiation, are shown in Figures 6.16, 6.17, and 6.18 for the PCF-FBGs. Figure 6.19 displays all PCF--FBGS showing very similar scattering intensity patterns when compared to the control PCF-FBG.

6.8.1 Scattering Patterns PCF SAXS

![Figure 6.16: (a) Experimental Scattering pattern of PCF-FBG (1541.00 nm) NO GAMMA (b) applied SASfit pattern (red) plot versus experimental blue plot.](image-url)
Figure 6.17: (a) Experimental Scattering pattern of PCF- FBG (1540.818 nm) 151.8 kGy GAMMA (b) applied SASfit pattern (red) plot versus experimental blue plot.
Figure 6.18: (a) Experimental Scattering pattern of PCF- FBG (1532.880 nm) 148.05 kGy GAMMA (b) applied SASfit pattern (red) plot versus experimental blue plot.
Figure 6.19: Experimental combined scattering pattern profile PCF-FBGs, 151.8& 148.05 kGy, GAMMA. The blue scattering pattern is the control PCF.

6.8.2 Summary  PCF SAXS

As shown in Figure 6.19 a comparison between the scattering intensity of the control PCF-FBG (blue profile) and irradiated samples can be identified. The red profile is from session two after 151.8 kGy inclusive of recovery. The brown and black profiles are from session three after 148.05 kGy inclusive of recovery. All irradiated scattering intensities are closely configured and much lower in intensity than the STD group. This is underscored also by the null result in determining the particle number concentration pre and post irradiation as shown in Table 6.15. The reduced scattering intensity indicates there are no cylinders present (or too small to detect) and no radiation tracks present (less radiation damage). Fractal analysis however, indicates the surfaces from which we were scattering have a mass fractal nature. Results for the types of fractals present, size, shape, pre and post irradiation are also shown in Table 6.15. Figure 6.20 shows the 2D scattering intensity profile obtained from SASfit pre-and-post irradiation. The profile shows that after irradiation the scattering intensity has changed minimally and the anisotropic effect is minimal. A comparative analysis between the PCF and STD FBGS is in the final summary section.
Table 6.15: Resulting parameters after SAXS fitting for PCF-FBGs pre irradiation and after irradiation dose, inclusive of relaxation shifts.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>PCF-FBG Control No Irradiation</th>
<th>PCF-FBGs after 151.8 kgy &amp; 148.05 kGy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Radius of Particle</td>
<td>Not Determined</td>
<td>Not Determined</td>
</tr>
<tr>
<td>Particle Concentration</td>
<td>Not Determined</td>
<td>Not Determined</td>
</tr>
<tr>
<td>Length of Cylinder</td>
<td>Not Determined</td>
<td>Not Determined</td>
</tr>
<tr>
<td>$P$ Factor</td>
<td>2.48062</td>
<td>2.4075</td>
</tr>
<tr>
<td>Fractal Nature</td>
<td>Mass</td>
<td>Mass</td>
</tr>
</tbody>
</table>

Figure 6.20: Combined 2D scattering intensity profile PCF-FBGs. (left image) PCF-FBG no irradiation; (right) PCF-FBG after 151.8 kGy.

6.9 Direct Comparison of Results: ESM 12-PCF and Std-SMF28H, Std-GeH, Std-Ge. Included are 2d scattering images from experimental data using sasfit.

A summary of results achieved and previously discussed in sections 6.7.2 and 6.8.2 are provided in Figure 6.21, 6.22, and 6.23. This allows a direct comparison between the STD-FBGs and PCF-FBGs. Figures 6.21 (STD-FBGs), and Figure 6.22 (PCF-FBGs), demonstrate the scattering profiles after complete irradiation inclusive of relaxation.
Figure 6.23 shows a direct comparison of the 2D scattering intensity profiles also after complete irradiation inclusive of relaxation.

**Figure 6.21:** Combined scattering pattern profile STD-FBGs

**Figure 6.22:** Combined scattering pattern profile PCF-FBGs
Figure 6.23: Direct comparison 2D scattering intensity profile after complete irradiation and relaxation. (Top left image) SMF28H-FBG; (Top right) Ge-FBG; (Bottom) PCF-FBG.

6.10 SAXS Summary

The results indicate that there are two types of scattering pattern in relation to intensity, and the nature of the surface fractals involved between the STD-FBGs and PCF-FBGs. The PCF-FBG results indicate no presence of cylinders in the scattering pattern, therefore no radiation tracks, which may be indicative of the recovery aspects of the PCF-FBGs. However, the unified model shows signs that the surfaces from which we were scattering have a mass fractal nature. The control PCF-FBG has a P value of 2.48062 pre-irradiation.
and 2.4075 post-irradiation which is under the scaling component $3 < P$. The overall scattering profile intensities are closely linked in relation to the control PCF. There is little fluctuation away from the control which again may indicate the recovery aspects and robustness of the pure silica PCF in relation to gamma irradiation. Comparatively, the STD-FBG scattering intensities are much larger. When using the hollow cylinder model, unified model and a linear background, the surfaces showed signs of having a surface fractal nature. The long shell hollow cylinders present on inspection are long, $\sim 3000\text{Å}$ with a mean radius of $\sim 23\text{Å}$. Interestingly, the mean radius is fairly constant in the STD-FBGs, ranging from $23.5277\text{Å}$ pre–irradiation to an average of $23.6353\text{Å}$ post-irradiation. What is not consistent, however, is the particle number concentration in the STD-FBGs pre-and post-irradiation. There is a marked difference and increase in the number concentration pre-irradiation $6.29483 \times 10^{31}\text{cm}^{-3}$ to $9.3108 \times 10^{31} \text{cm}^{-3}$ and $8.6767 \times 10^{31} \text{cm}^{-3}$ post irradiation.

This is evident in the comparison between the blue intensity scattering pattern of the control STD-FBG, and increased scattering intensity profiles of the irradiated STD-SMF-FBGs and Ge-FBGs. The increase in intensity coincides with the increase in particle number concentration, and with the predominance of large structures at smaller angles in the scattering profile. There seems to be a strong correlation between gamma exposure and a greater influx of structural rough surface fractals (i.e. $> 800\text{Å}$). The control STD-FBG has a $P$ value of $3.27285$ pre irradiation, whilst $3.09698$(SMF+H) and $2.978932$(Ge+H) post irradiation, which is within the scaling component $4 > P > 3$. Although the GE+H is very slightly below the scaling component $P > 3$, it is indicating more of a surface fractal than mass.

The correlation between the SAXS results shows that the PCF has less radiation damage due to the lack of radiation tracks compared to hollow cylinders produced in the STD-FBGs. The scattering patterns are in two definitive groups: PCF and STD. The recovery aspects of the PCFs after gamma exposure seem to be verified with the XRD and SAXS data also in relation to anisotropy. The STD–FBGs display higher scattering intensities post irradiation in conjunction with more pronounced anisotropy.

Although there is anisotropic behaviour present in the PCF-FBGs, as shown in the FWHM variation of the XRD analysis, the change is less pronounced than that of the STD-FBGs. This is clearly seen when comparing the scattering intensity and 2D intensity scattering profiles where little change is displayed. The change in grating periods, as shown in the simulated data, is also corresponding to the spectral analysis. PCFs, are
exhibiting a more stable response and appear more robust during irradiation. They remain sensitive and almost completely recover after irradiation, which make them ideal candidates for radiation dosimetry. They could also become a predictive model, which is lacking, so far as kGy/pm rates are concerned. The rates for the first 50 kGy using PCF-FBGs equate to ~1 kGy/1 pm compared to ~1 kGy/1.8 pm in STD-FBGs.
CHAPTER 7
CONCLUSION

7.1 Conclusions

The gamma irradiation response regarding the Bragg wavelength shift and recovery aspects of new generation PCF-FBGs for the area of radiation dosimetry has been reported and the objectives have been met. A comparison with STD-FBGs under very similar experimental regimes has helped to quantify the differing behaviours. The results indicate the PCF-FBGs are quite suited for use in the area of high dose irradiation areas and even possibly low dose areas. One major problem that has been overlooked in most current research are the recovery aspects over short time periods. Three short consecutive recovery times presented in the study is a first and helps to expand on previous research. If FBGs are to be used eventually as radiation dosimeters, either in nuclear facilities or space applications they should exhibit strong and consistent recovery attributes.

Simulated results in conjunction with experimental data also indicate that strain, more so than temperature, is likely playing a more dominant role resulting in a more permanent change to the grating period. On average there is a 28.21% increase of the pm/micro strain to ~1.5385 pm/microstrain in STD-FBGs, and a 25.0% increase of the pm/micro strain to 1.5 pm/microstrain in PCF-FBGs from the accepted standard rate (1.20 pm/micro-strain) during irradiation. Shown also, with very limited recovery times between irradiation stages, the BWS has progressively reduced. This has highlighted the effects of pre-irradiation. Although overall, the PCF-FBGs and STD-FBGs sensitivity is reduced, the PCFs exhibit a more consistent and stable response through each stage. This indicates that structurally PCF-FBGs are more sound throughout the process.

Collaborative structural analysis by XRD and SAXS confirms this, along with the strong recovery aspects after exposure to gamma compared to the STD-FBGs. The use of XRD and SAXS for analysis pre and post irradiation on both sets of FBGs to my knowledge is a first. Wide angle x-ray diffraction analysis indicates that PCF-FBGs reduction of the FWHM reflect decreasing lattice imperfections in the grating area resulting in a reduction of strain. This strain actually decreases with an increase in particle size as in the PCF-FBGs, which is an indication of less crystalline defects. Within the STD-FBGs, there is an indication that structural damage of a more pronounced nature is
evident, confirmed by the SAXS analysis. The SMF-FBGs show more of a metamict phase and increased anisotropy. Post irradiation results are in accord with this through the presence of surface fractals (large) and increased particle number concentration. Mass fractals however, are present in the PCF, which represents smaller structures and less structural damage.

The results from SAXS, XRD, experiments and simulation, indicate that PCF-FBGs have less radiation damage and seem more robust under these conditions. This allows for strong recovery while still producing sufficient sensitivity with regard to the BWS during gamma exposure. Throughout the literature there are many varied results relating to the behaviour of certain optical fibre Bragg gratings during exposure to radiation. This is normally due to the very broad range of manufacturing parameters which influence the FBGs response. One more important limitation is the lack of continuity i.e., the availability of the same test facilities, equipment and experimental regimes. This all leads to the lack of good predictive models in the behaviour of FBGs. It must be stressed that this study has outlined three irradiation experimental stages using the identical equipment on each occasion, with similar dose rates and identical time periods, both during exposure and recovery, which to my knowledge is a first. Therefore, the results reported in this study come from a regime that overcomes the limitations. In conclusion, the results from this study indicate that PCF-FBGs strong recovery aspects, stability and sufficient sensitivity, make them an ideal candidate as the basis of a predictive model in the field of radiation dosimetry.
CHAPTER 8
FUTURE WORK

The PCF-FBG has shown it is a strong candidate for use as a possible sensor in the field of radiation dosimetry when compared to STD-FBGs. The studies and effects of radiation on PCFs however are limited, which is why further investigation is warranted. Due to limited availability of a gamma source in Australia, testing under varied regimes was difficult. For this reason more research and further testing should be performed varying dose rates (higher and lower) and accumulated dose. The similar multiplexing results obtained from the PCF-FBGs demonstrate stability, sensitivity, and consistency. Further testing is therefore warranted, increasing the amount of FBGs (e.g. to 4 or 5) using wavelength division multiplexing. With all FBGs irradiated at the same time under identical conditions, the time required will greatly reduce and it will also allow greater precision when comparing results. As the results of this study included only two PCF-FBGs that were multiplexed, it would be of interest to compare the results with this increase. Also of interest for future research is the inclusion of hollow-core PCFs, due their lower RIA benefits.

It is also recommended that work be undertaken to improve and enhance the performance of the PCF-FBG fabrication. Using the pulsed UV radiation of the ArF 193nm laser, a coupling strength of 6dB/cm was achieved, leaving room for further optimization. Improvements in the grating fabrication is an area worthy of future research. Currently with respect to time taken using the ArF laser was approximately 30minutes. Recently, there has been increased research in nano-fibres due to their many favourable attributes which include, flexibility, high sensitivity, low loss, low dimension, high spatial resolution and fast responses. Effects of gamma irradiation in this new fibre would also be of interest for comparative purposes.

Finally, because of the attributes and results alluded to in this study during and after irradiation, further research using PCF-FBGs is warranted. They could become a prime candidate as a replacement for the older type dosimeters. The cost factor has always been a barrier in relation to PCF, however, when this fibre is pigtailed at each end with standard SMF-28, using tailored fusion splicing techniques as in this study, the cost factor is reduced dramatically.
REFERENCES


A. Martinez, M. Dubov, I. Khrushchev, and I. Bennion, “Photoinduced Modifications in Fiber Gratings Inscribed Directly by Infrared Femtosecond


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[121] Photograph of ANSTO facility and GATRI cobalt-60 source, available from the ANSTO website: http://www.ansto.gov.au,

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APPENDIX A

As mentioned, the OSA was interfaced using a GPIB/USB to a laptop. The Matlab program was developed, shown below, to control the OSA and the measurement process, and automatically recorded the results of each set of optical measurements for each absorbed dose as a function of time at 1800sec (30min) intervals.

clear all;close all;clc;

% In this program the requires wavelength to be set manually
% Pl turn on light source before executing this program
% Pl enter the primary address of agilent 86141B spectrum analyzer
% instrument data %Enter primary address of OSA

for count=1:350
    fprintf(g, 'sens:wav:star 1537.5nm');
    fprintf(g, 'sens:wav:stop 1557.5nm');
    fprintf(g, 'disp:trac:y:rlev -25dbm');
    fprintf(g, 'sens:bwid:res 0.1nm');
    fprintf(g, 'nit:imm;*opc?'); % Take a sweep, wait for complete%
    t = fscanf(g, 'trac:data:y? tra');
    s=str2num(t);
    % Data processing
    sizer=size(s);
    w=linspace(1537.5,1557.5,sizer(2));
    % saving data
    d=[w ;s];
    save('AnstoData17', 'd');
    Ran=['A' num2str(3*count)];
    xlswrite('AnstoData17.xls',d,1,Ran);
    %xlswrite('data', data_saved);
    c= num2str((3*count)-1);
    xlswrite('AnstoData17.xls',clock,1,c);
    pause(1800)
end
% save('data','s')
APPENDIX B

The Matlab program shown below, controlled the OSA and the measurement process, and automatically recorded the results of each set of optical measurements for each relaxation period (non-irradiation, 3 to 3.5 hrs) at 600sec (10min) intervals.

```matlab
clear all;close all;clc;

% In this program the requires wavelength to be set manually
% Pl turn on light source before executing this program
% Pl enter the primary address of agilent 86141B spectrum analyzer

% instrument data %Enter primary address of OSA
    g = instrfind('Type', 'gpib', 'BoardIndex', 7,
    'PrimaryAddress', 23, 'Tag', '');
    get(g,{'EOSMode','EOIMode'});
    if isempty(g)
        g = gpib('AGILENT', 7, 23);
    else
        fclose(g);
    end
    set(g, 'InputBufferSize', 20000);
    set(g, 'Timeout', 20.0);
    fopen(g);
    fprintf(g,'*IDN?');
    idn = fscanf(g) ;
    for count=1:350
        fprintf(g, 'sens:wav:star 1537.5nm');
        fprintf(g, 'sens:wav:stop 1557.5nm');
        fprintf(g, 'disp:trac:y:rlev -25dbm');
        fprintf(g, 'sens:bwid:res 0.1nm');
        fprintf(g, 'nit:imm;*opc?'); %Take a sweep, wait for complete%
        fprintf(g, 'trac:data:y? tra');
        t = fscanf(g) ;
        s=str2num(t);
        %Data processing
        sizer=size(s);
        w=linspace(1537.5,1557.5,sizer(2));
        %saving data
        d=[w ;s];
        save('AnstoData17', 'd');
        Ran=['A' num2str(3*count)];
        xlswrite('AnstoData17.xls',d,1,Ran);
        %xlswrite('data', data_saved);
        c= num2str((3*count)-1);
        xlswrite('AnstoData17.xls',clock,1,c);
    pause(600)
end
%save('data','s')
```
APPENDIX C

The FBG specifications for the standard set purchased from Alxesnse are shown below.

<table>
<thead>
<tr>
<th>Acrylate FBG (Single FBG)</th>
<th>P/N: AL-S-A-FBG-1540-FA-SMF100bar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Center wavelength: 1540nm</td>
<td>-</td>
</tr>
<tr>
<td>Center wavelength tolerance: +/- 0.5m</td>
<td>-</td>
</tr>
<tr>
<td>Type of FBG: Apodized</td>
<td>-</td>
</tr>
<tr>
<td>Length of FBG: 10mm</td>
<td>-</td>
</tr>
<tr>
<td>Bandwidth @ -3dB: &lt;0.3nm</td>
<td>-</td>
</tr>
<tr>
<td>Reflectivity: &gt;90%</td>
<td>-</td>
</tr>
<tr>
<td>SLSR (dB): &gt;15dB</td>
<td>-</td>
</tr>
<tr>
<td>Fiber length: 30cm with the FBG centrally located</td>
<td>-</td>
</tr>
<tr>
<td>Acrylate coated fiber and Acrylate recoated FBG</td>
<td>-</td>
</tr>
<tr>
<td>Standard SMF-28 fiber and hydrogen loaded with 100 bar</td>
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</tr>
<tr>
<td>FC/APC on both ends</td>
<td>-</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Acrylate FBG (Single FBG)</th>
<th>P/N: AL-S-A-FBG-1545-FA-GW/O</th>
</tr>
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<tbody>
<tr>
<td>Center wavelength: 1545nm</td>
<td>-</td>
</tr>
<tr>
<td>Center wavelength tolerance: +/- 0.5m</td>
<td>-</td>
</tr>
<tr>
<td>Type of FBG: Apodized</td>
<td>-</td>
</tr>
<tr>
<td>Length of FBG: 10mm</td>
<td>-</td>
</tr>
<tr>
<td>Bandwidth @ -3dB: &lt;0.3nm</td>
<td>-</td>
</tr>
<tr>
<td>Reflectivity: &gt;90%</td>
<td>-</td>
</tr>
<tr>
<td>SLSR (dB): &gt;15dB</td>
<td>-</td>
</tr>
<tr>
<td>Fiber length: 30cm with the FBG centrally located</td>
<td>-</td>
</tr>
<tr>
<td>Acrylate coated fiber and Acrylate recoated FBG</td>
<td>-</td>
</tr>
<tr>
<td>Germanium doped fiber and without hydrogen loading</td>
<td>-</td>
</tr>
<tr>
<td>FC/APC on both ends</td>
<td>-</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Acrylate FBG (Single FBG)</th>
<th>P/N: AL-S-A-FBG-1550-FA-G100bar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Center wavelength: 1550nm</td>
<td>-</td>
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<tr>
<td>Center wavelength tolerance: +/- 0.5m</td>
<td>-</td>
</tr>
<tr>
<td>Type of FBG: Apodized</td>
<td>-</td>
</tr>
<tr>
<td>Length of FBG: 10mm</td>
<td>-</td>
</tr>
<tr>
<td>Bandwidth @ -3dB: &lt;0.3nm</td>
<td>-</td>
</tr>
<tr>
<td>Reflectivity: &gt;90%</td>
<td>-</td>
</tr>
<tr>
<td>SLSR (dB): &gt;15dB</td>
<td>-</td>
</tr>
<tr>
<td>Fiber length: 30cm with the FBG centrally located</td>
<td>-</td>
</tr>
<tr>
<td>Acrylate coated fiber and Acrylate recoated FBG</td>
<td>-</td>
</tr>
<tr>
<td>Germanium doped fiber and hydrogen loaded with 100 bar</td>
<td>-</td>
</tr>
<tr>
<td>FC/APC on both ends</td>
<td>-</td>
</tr>
</tbody>
</table>
APPENDIX D

The ESR signals spectra were measured, both pre and post irradiation at room temperature. The experimental results demonstrate, both in the PCF-FBGs and STD-FBGs pre irradiated samples, that no obvious signals of defect centres were detected. Following the complete irradiation regimes inclusive of relaxation, a clear signal of the ESR spectrum was present, indicating that color centres are generated. In the PCF, SMF28H, Ge H, and Ge samples the spectra produced post irradiation are very similar. The ESR spectrums produced g values of 2.0004 (for PCF-FBGs) to 2.0005, 2.0007, 2.0008 (for Ge+H, Ge, SMF28H FBGs) at room temperature post irradiation. An average value between the samples is g= 2.0006. According to the literature [41] the ESR signals reveal Ge–related paramagnetic centres at g = 2.0006 av. which can be assigned to the defect centres of Ge (1).

PCF –FBG before (left); and after gamma exposure (right)
SMF –FBG before (left); and after gamma exposure (right)

Ge –FBG before (left); and after gamma exposure (right)
Ge + H FBG after gamma exposure