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Application of High Temperature Silica Yarns as Thermoluminescence Radiation Dosimeters

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Abstract - There is a growing number of applications where ionizing radiation is used and for this reason its detection is of great importance. One way of measuring the absorbed dose is by thermoluminescence dosimetry (TLD). In this, a phosphor is employed to record ionizing radiation. A suitable technique is then used to thermally extract this information to determine the radiation dose the phosphor was exposed to. TLDs are usually inexpensive and physically small which makes them ideal to use in both personal and environmental dosimetry. These devices are well researched but have drawbacks such as a high processing cost and not being able to detect overall dose received by wearer since they only cover small part of person's body. This leads to a lower than actual dose readings when dosimeter is, for instance, shielded by another material during radiation exposure. In this project a silica yarn was explored to determine its suitability in dosimetry use. This material has promising applications in both, personal and environmental dosimetry because of its physical flexibility. This can be utilized for innovative applications such as development of a dosimeter in the form of clothing. The primary goal was characterization of the relationship between irradiation of the material by a known dose and its subsequent reading by the process of thermoluminescence (TL). Correlation factor of 0.9 was found which confirms usability of silica yarn for TL dosimetry. Further work is proposed to develop a custom made reader for fabric type phosphors in order to achieve accurate detection of absorbed dose.

Keywords - High Temperature Silica Yarns; Thermoluminescence; Radiation Dosimeters.

1 Introduction

Ionizing radiation reaches every person in the form of background radiation. Even though a direct link between effects of natural radiation to human health is not clearly proven, dose as low as 1 mGy can produce double strand break in DNA in some cells [1]. This, coupled with advances in technologies involving ionizing radiation, used by modern society, justify a need for accurate determination of dose received. Thermoluminescence dosimetry is one type of widely used forms of radiation detection for environmental and personal monitoring. It can detect levels of μGy in environmental detection as well as several Gy used by radiotherapy treatments [2]. They are useful in variety of applications due to their small size and ability to operate without external power source. Popular materials used in this field are often various compounds, such as lithium fluoride, [3] where doping process is used to enhance its ability to detect ionizing radiation. They mostly take a physical form of a disc, film

or even powder [4]. TL phenomenon requires ability of a material to lock electrons and holes in place, therefore crystals, with impurities acting like traps, are predominately used in TLD. These materials, unlike metals, have almost no free electrons and so any electrons found in conduction band are due to irradiation. Silica yarn explored in this project comes with over 94% SiO2 content and it is therefore predetermined to be a good TLD. Further, it has two intrinsic properties that differentiate it from most materials used in this field; high temperature resistivity of over 1000°C and physical flexibility. The second is the primary reason for experimenting with it. Because it is essentially a piece of string it is proposed to use it in the form of a clothing with ability to record ionizing radiation. There is no commercially available TL reader on the market that would allow for scanning of fabric based materials. Toledo 654 TL reader made by D.L. Pitman Ltd was used in this project because its design allows for research activity of this type. It has a heated planchet on which a sample can be placed. A secondary part of the project was therefore to develop a suitable holder for silica yarn that would serve as an adapter between the sample and the planchet.

2 Theory

2.1 Thermoluminescence effect

Thermoluminescence dosimetry uses phenomenon seen in some materials in which they emit light during heating after previous exposure to ionizing radiation. Theory behind this effect can be conveniently described by energy bands. TLD materials, crystals or insulators, have large band gaps in which ionized electrons can be locked in place and effectively store radiant energy [5]. The process is pictorially described in Figure 1. During the irradiation, ionized electrons are released into the material (a), travel in the conduction band (CB) and get trapped inside the crystal's defects (b). Positively charged holes left after departure of the electrons are known as hole traps [6].

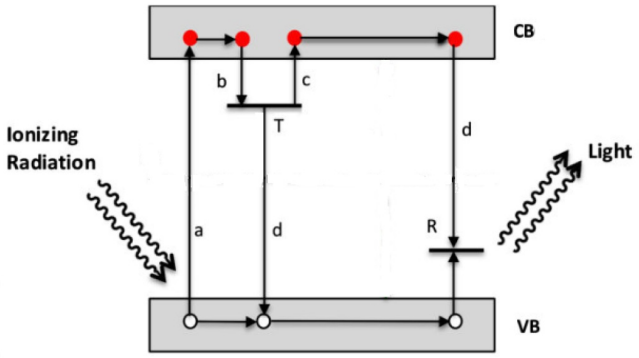

Figure 1: Thermoluminescence process - band model for crystalline structure Source: www.intechopen.com

Trapped electrons will stay in these metastable states if they are located deep enough below the conduction band, otherwise room temperature may cause them to recombine back to CB. Large amount of trapping centers act as a memory because continuous irradiation will lead "to progressive buildup of trapped electrons" [7]. This ability to accumulate absorbed dose is the main reason why it is desirable for any TLD materials to have large amount of recombination centers or large band gaps. Thermoluminescence effect takes place when large enough external thermal energy is introduced to the material for electrons to move from traps to conduction band (c), where they travel until they recombine with a hole in recombination center R and radiate photon (d). Visible light is produced with energy difference of 3-4 eV [7]. There is also a possibility of an electron recombining directly to valence band. The process described here for electrons is similar for holes. Therefore the amount of electron-holes pairs created during irradiation should be proportional to the amount of photons released. This fact can be used to plot a chart representing light output as a function of heating rate of the material. This is called glow curve.

2.2 Materials

Amorphous silica SiO_2 is popular material for manufacturing electronic components such as transistors where it acts as the dielectric [8]. Its non-strict crystalline structure give rise to defects so important to TL effect. Structure defects arise mainly from indefinite distance between Si-O bonds [9]. 2-fold oxygen must always connect to 4-fold silica creating chains and rings (Figure 2).

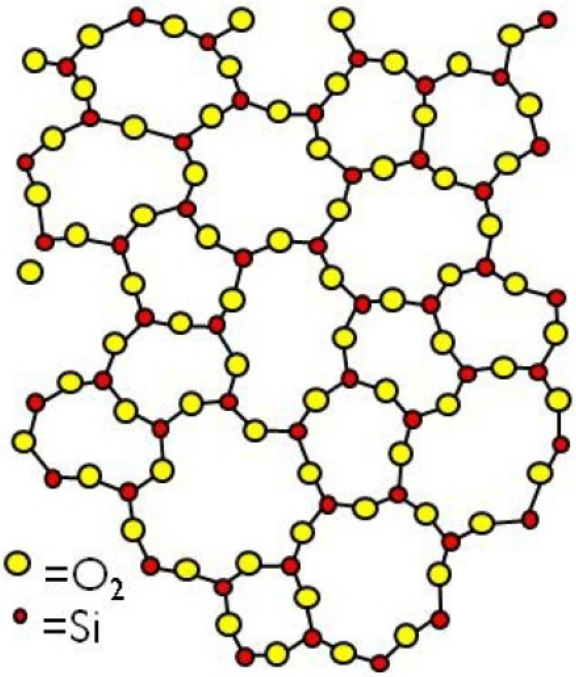

Figure 2: Figure 2: The structure of amorphous SiO_2 Source: nanoHUB.org

With this structure oxygen vacancies are certain. Oxygen related defects associated with Si dangling bond are also frequent due to this structure. Defect in the form of Interstitial oxygen molecules may be formed as a result of irradiation [10]. Further to intrinsic structure defects there are other caused by external effects such as temperature change or irradiation [10]. These can form new defects or alter existing ones. Aforementioned Si dangling bond and non-bridging oxygen are among the most common defects induced by ionizing radiation in this material [11].

2.3 Fading

Stability of recorded dose over time is important factor for any TLD. Loss of this information between irradiation and reading is known as fading. According to [6] loss of signal of 5% per months is deemed acceptable. Temperature of the environment in which TLD is stored is among the most influential in data decay and so is external light source. Any external source of energy can cause shallow traps to empty because energy needed to liberate an electron to the conduction band is small. One of the ways to deal with these signals is to preheat the sample prior to the readout to empty shallows traps.

2.4 Instrumentation

To extract information about absorbed dose from TLD, a suitable TL readers is used. A typical reader consists of heating device and some form of light detection. Reader used in this experiment is Toledo 654 (Figure 3) which has a circular metal planchet on which a samples are placed. After closing the drawer, heating element is moved into position with the planchet.

Figure 3: Toledo 654 TLD reader used in this experiment.

Thermocouple controls the temperature. Nitrogen gas flows into the chamber during heating to minimize a risk of false signal detection due to possible contaminants present on the sample surface i.e. dust. Heating rate is adjustable with temperature setting going up to 400°C. Photomultiplier tube then records the amount of light which is then processed in analogue/digital converted for analysis. The above apparatus belongs to the standard group of readers designed for low light detection since most TLDs exhibit highest light output at around 300°C. Bilski et al. have pointed out [12] some distortion in results during high light output signal - high dose measurements, with such readers. TL readers tested in their experiment had small errors in low level readings but varied considerably with high dose measurements. Worse still, errors varied with different manufacturers.

2.5 Data acquisition

Charges are trapped at different depths within the band gap so different energies are required to release them into conduction band in order for luminescence effect to take place. These energies are provided by heat. It is expected that higher temperatures will result in more light being produced. During the reading the heating process is gradual and fast (in order of seconds). Therefore one can expect a correlation between light intensity and a temperature [13]. If we were to plot the temperature as a function of light intensity we get a line called Glow curve. Many readers are able to compute the area under this curve. This area directly corresponds to the amount of measured photons and it is therefore a good indicator of a number of activated recombination centers. This can be used to estimate a strength of the original radiation that produced these centers. The process of interpreting glow curves is complicated and resembles a statistical process. This is because, during ionizing radiation, once the electron enters a CB it may be re-trapped on crystal's impurity or recombined. Although recombination is more frequent than re-trapping [2], result of

this activity is probabilistic. Especially considering that radiation energy itself can cause loss of traps. Production of recombination centers does not necessarily stop after removing radiation. Traps interact with each other so charges can be captured by other sites. Important parameters affecting this process can be mathematically described. Energy 'E' is the energy required for an electron located below the conduction band to escape to CB. This is known as trap depth. Frequency factor 's' describes how often will the charge try to escape its position. This could also be due to lattice vibrational frequency. We can describe the probability of an electron leaving its site as:

$$p = s \cdot e^{\left(\frac{-E}{kT}\right)} \tag{1}$$

Where k and T are Boltzmann constant and Temperature respectively. It can be seen that the particle liberation probability is higher with increasing T but lower with the depth of the trap. If we consider an amount of electrons 'n' locked inside the band gap, then, provided the temperature of material is the same, this number will decrease with time:

$$\frac{dn}{dt} = -p \cdot n \tag{2}$$

This, of course, assumes that all electrons that escaped from their positions recombined radiatively. Relationship between trap depth and the speed in which traps are emptied is achieved by substituting Eq.1 Into Eq.2, and integrate:

$$n = n_0 \cdot e^{\left(-s \cdot t \cdot e^{\left(\frac{-E}{kT}\right)}\right)} \tag{3}$$

with n_0 amount of electrons at time 0. Behavior of this equation shows that shallow traps will depopulate faster than deeper ones. It is important to realize that in real life situation the amount of trapped electrons in the material is finite. Probability of electrons leaving traps increases with temperature but there will be a point where p = 1, i.e. thermal energy supplied is large enough for all traps to be emptied. At this point dn/dt will begin to decrease and the curve on the temperature/light intensity graph takes shape of a bell curve. There is a number of theories and computer tools to interpret glow curves. Often, theoretically calculated curve is matched to experimental result. Each model comes with some constraints and assumptions while some look at geometrical properties of the curve. Resultant curves in this experiment have large inconsistencies to be considered for those methods although an attempt was made and can be found in the result section.

3 Methodology

3.1 Overview of the experiment

Characterizing a new material for dosimetry requires methods that allow for unexpected findings or complications and ensures reproducible results. Silica yarn was theoretically predicted to be a suitable material by consideration of its crystalline structure. This is further supported by experiments already carried out with silica containing materials such as glass beads [14].

Figure 4: Silica yarn sample

Material used in this experiment was silica yarn, 1.6 mm in diameter, containing 94% silicon oxide, able to tolerate temperatures of up to 1000°C. It is made from 3 separate continuous filaments of amorphous silica fibers twisted together with a thin metal wire entangled lengthwise, for reinforcement. The detailed composition was not provided by manufacturer. Three separate experiments were performed, each with some variations in method, described separately in following sections. Samples were annealed before irradiation process.

3.2 Experiment 1

Six samples of 10 cm long pieces of yarn were placed into aluminum foil of square shaped envelopes to allow for even irradiation and to protect the samples from light exposure. Traceably calibrated linear accelerator radiotherapy treatment unit, Varian Clinac, at a nominal 6MV energy was used for irradiation. Each envelope was placed inside a solid water phantom and irradiated by uniform 10 x 10 cm2 treatment field beam. Each pack received a different dose -18, 12, 6, 3, 1, 0.5 Gy and was labeled. Readout took place 2 weeks later with Toledo 654 TL reader, Figure 3, described previously. The scanner has the flexibility to select desired heating parameters. Temperature was set to 370°C, the highest experimentally chosen value the apparatus could handle without errors. Preheating cycle was deemed unnecessary, at this stage of the experiment, because it could cover up low energy response characteristics of the material and so was not used. Heating rate was 30°C s-1 up to the temperature of 350°C, followed by the rate of 3.4°C s-1 up to the final value. This 2-way rate system could not be altered. Placing samples loosely on the metal tray, as it is common practice with other solid TLDs, was not possible due to a physical nature of the silica yarn. Fabric materials do not conduct heat very well and must be pressed onto the heating planchet for TL effect to take place. This was confirm experimentally. To tackle this challenge, a holder was developed, that would lock the sample tightly in place and ensured good heat transfer. The holder was circular in shape, 8 mm in diameter, to fit well within the planchet in the reader, made from high quality quartz glass as recommended by Oberhofer and Scharmann [4].

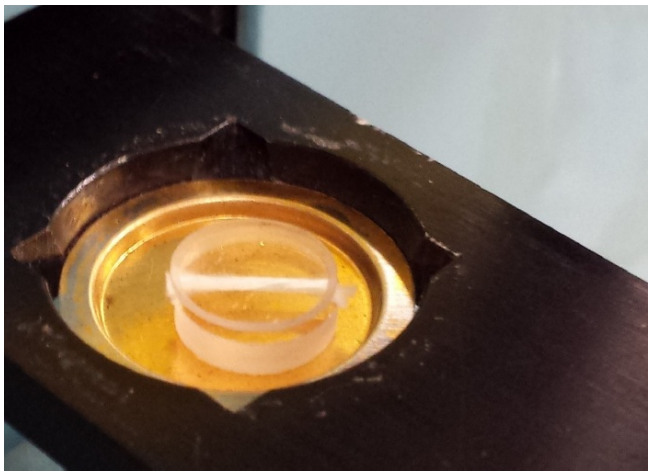

Figure 5: Sample holder - Quartz Glass

This material has good heat resistance, withstands sudden temperature changes and offers excellent optical qualities. This design consisted of two glass discs, the bottom portion is 2 mm thick with a groove for the yarn placement and the top disc, 1 mm thick, loosely placed on top to sandwich the sample inside for improved thermal contact. Samples were handled by plastic tweezers to avoid scratching the material which could introduce localized phosphor excitation and leading to potential errors [2]. Once in the position the sample was cut to size by fingernail clipper. Several readouts were conducted for each dose to evaluate consistency of results.

3.3 Experiment 2

Poor thermal conductivity of quartz glass (1.3 W/(m K) and its thickness was to blame for poor results acquired in the experiment 1. Improvements were made as follows. The base of the holder was substituted by stainless steel material which has better thermal conductivity (16 W/m K). Its thickness was chosen arbitrary to 0.9 mm to improve heat transfer further and for ease of construction. Guiding rails were designed with the aim of keeping the top disc in place (Figure 6) as opposed to laying it loosely on top as in previous experiment.

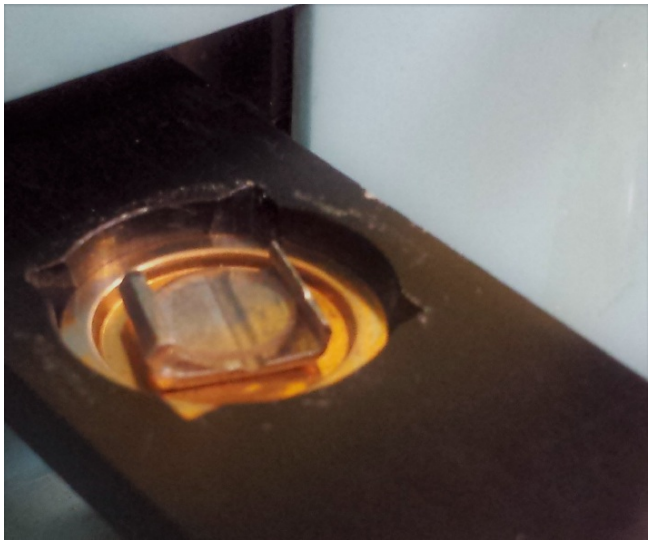

Figure 6: Sample holder - Stainless Steel, version 1

New set of samples was obtained and irradiated by strontium 90 source. Four separate batches of yarn were created and exposed to the source, each for the period of 5,10,15, 20 minutes which produced an estimated dose of 5,10,15,20 Gy respectively. Because the equipment has a narrow sample opening, the 10 cm long yarn pieces were wrapped around a piece of paper to achieve some homogeneity of radiation field sent through it. Option of cutting the string before the irradiation was not possible because silica yarn frays easily. Samples were read in the same manner as described in previous experiment. But this time within 10 minutes of irradiation to eliminate fading. Due to unforeseen circumstances guiding rails have snapped at the end of the experiment day, rendering the holder useless for subsequent use.

3.4 Experiment 3

Following satisfactory results obtained with the previous holder design a new base was fabricated using similar specifications. Heat transfer was enhanced by reducing stainless steel thickness further to 0.5 mm. Guiding rails were bent more for tighter fit around the top disc. Samples were irradiated in the same manner as outlined in experiment 1, this time with a wider range of doses. Arbitrary values were chosen: 40, 20, 10, 5, 3, 2, 1, 0.5, 0.2 Gy.

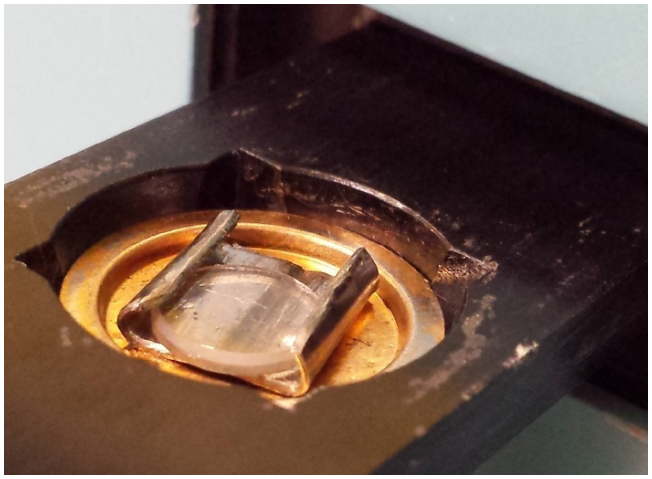

Figure 7: Sample holder - Stainless Steel, version 2

Despite the improvements the top glass failed to lock the yarn in place in the same reproducible manner. This has caused quite a significant errors during readout. Also noted was large inconsistency developed by the TLD reader itself when checking background signal (Table 1). This has only happened during this experiment and the reason of this is unexplained.

3.5 Further remarks

Sample holder forms an integral part of this project. Several ideas for its design were discussed and evaluated theoretically before the experiment begun. The most promising ones were put to the test in the reader itself. Thin end of a disposable soda-lime glass pipette (Figure 8), with the 0.8 mm diameter seemed a good candidate to house silica yarn firmly inside. This attempt failed for two reasons; a round wall of the pipette meant not enough material was in contact with the planchet to transfer heat to the sample. More worryingly, this extremely lightweight solution caused the pipette to be lost inside the reader on many occasions.

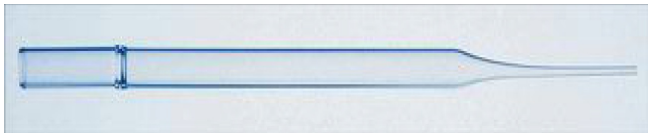

Figure 8: Sample holder - Disposable soda-lime glass pipette Source: Fishersci.com

This is believed to be due reader's automated system of bringing the heater element in contact with the planchet which is accompanied by a knock leading to pipette's ejection.

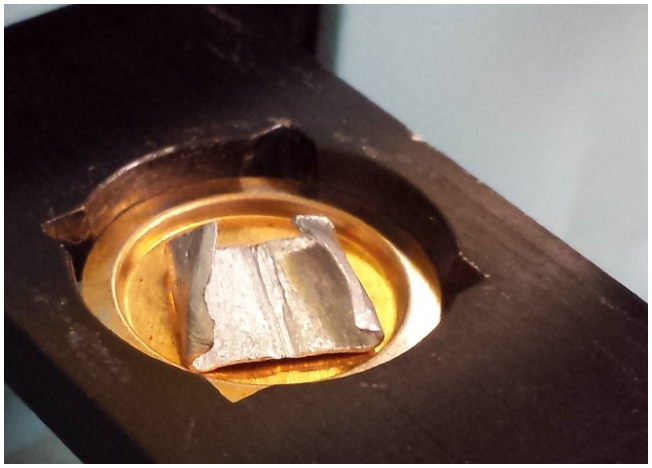

Figure 9: Sample holder - Aluminum, deformation due to heat

Aluminum was suggested as a material for holder base because of it desirable thermal conductivity of 222 W/m K. Trials revealed that coefficient of linear expansion (24 x 10^{-6}/K) is too high for the temperature used by TLD reader. Aluminum base has arched on the planchet, loosing contact with it. Guiding rails shrunk, locking the glass inside.

4 Results and Discussions

Before any reading session background levels were established by taking 4 consecutive measurements of sample holder containing non-irradiated silica yarn. Average background radiation was then calculated. Even though the background level values are not vital for the purpose of this experiment it helped to uncover large irregularities in measurements of the reader. These were noted especially towards the end of the experiment days. Possibly due to reader overheating. Figure 10 shows an example of one such result where flat glow curves were expected indicating no signal and Table 1 shows corresponding area under the curves of these background tests.

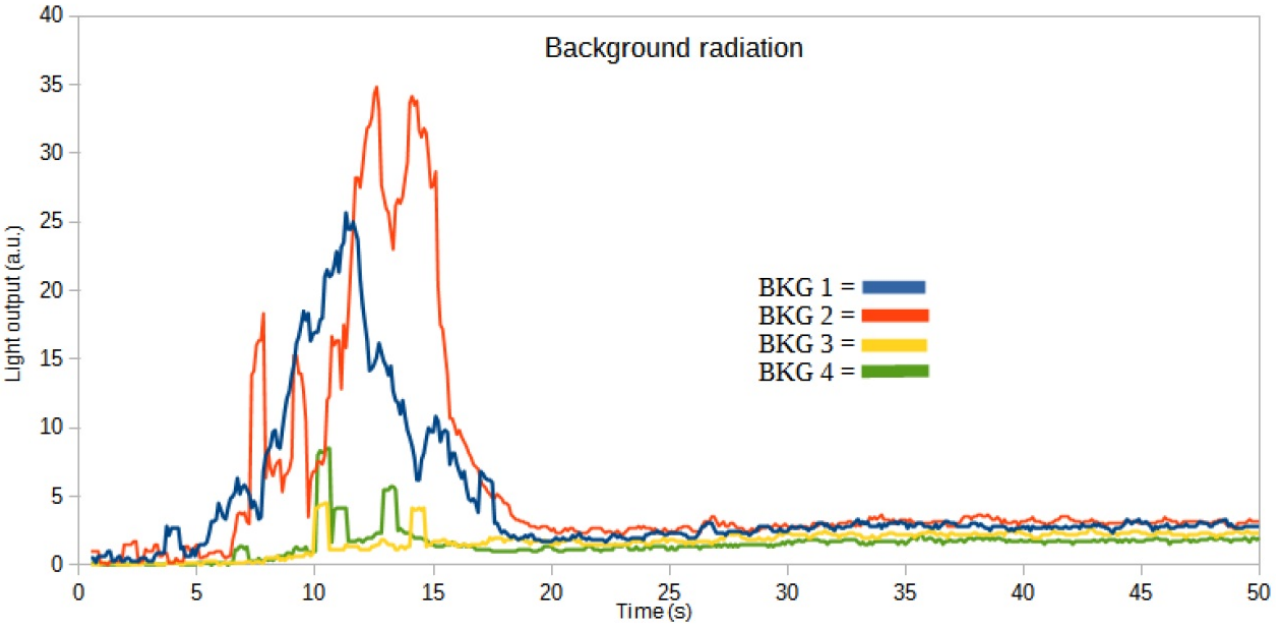

Figure 10: Background radiation glow curves.

Analyzing glow curves plotted by TL reader is uneasy task with no one specific method. Shape of the curve as well as the area under it reveal not only information about the dose received by the sample but also characteristics of the material itself.

Measurement No.	Area under curve
1	2100
2	2757
3	986
4	866
Average ± S_m	1677 ± 525

Table 1: Background radiation measurements.

One of those methods is to compare acquired glow curve to mathematically modeled one. Advantage of curve fitting is that the entire curve is used for analysis which possess many data points for accurate investigation. Chen and McKeever highlight two points [15] that, if not adhered to, negatively affect the result:

- The need for accurate determination of temperature of the sample. This is not possible in our case due to long delays of heat transfer from the planchet to the sample. Further, Toledo reader uses thermocouple to read temperature of the planchet, not the sample.

- Requirement for linear heating temperature gradient. Toledo reader, as described in theory section, does not follow this pattern for high end of the temperature scale.

Above arguments are apparent in experiment 1 as shown on Figure 11. Toledo TL reader reaches 350°C in 12 seconds as measured by the thermocouple on the planchet. Maximum points in all curves come 3 seconds later due to poor thermal conductivity of the glass holder. Further, there is a significant amount of traps being emptied after the maximum temperature attainment. This is in contradiction to equation 2 calling for diminishing number of traps after maximum temperature. This was a clear indicator that called for a different holder design for the next experiment. One compelling evidence came out from this experiment and that is the correlation between the dose recorded by the silica yarn and the area under the corresponding curve which is plotted in Figure 12. Not only the areas increase with dose but height of peaks are also raising. R2 figure of 0.9 shows good responsiveness of silica yarn to different dose levels.

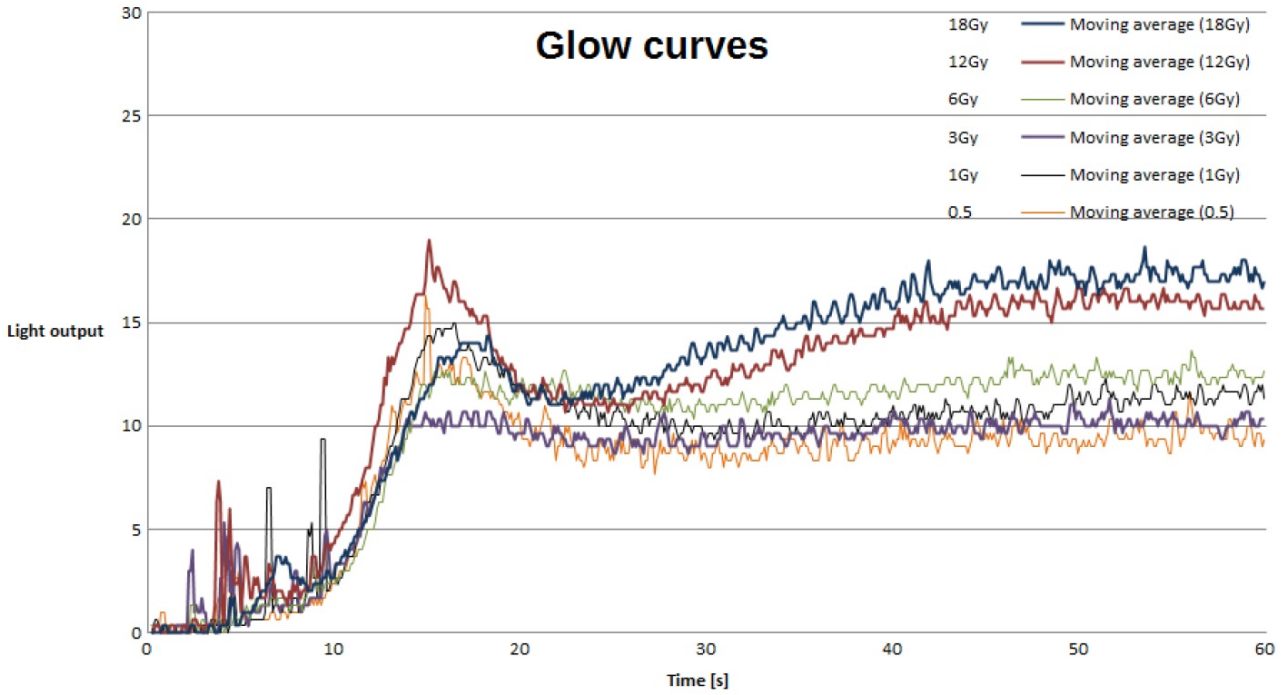

Figure 11: Results from experiment No. 1.

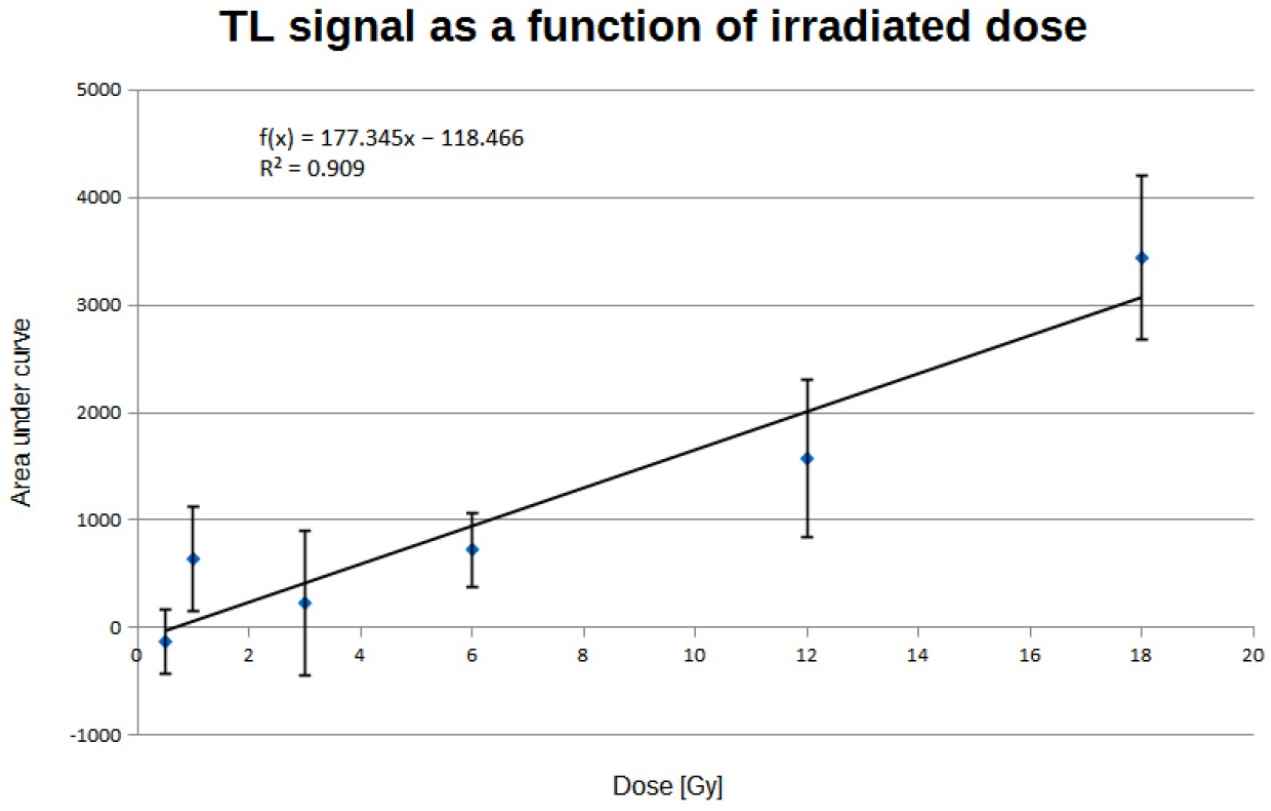

Figure 12: Area under the curve expressed as function of recorded dose, Experiment 1.

Encouraging results from previous experiment led to major changes as outlined in methodology section. Figure 13 shows one example of 20 Gy irradiated sample by Strontium 90 (beta source). A distinct peak is apparent followed by a decay to almost background levels. This is in a stark contrast to the previous results and is a good indication of all traps being emptied. A temperature value peaks close to the highest point of the glow curve further confirming very good heat transfer through the improved holder.

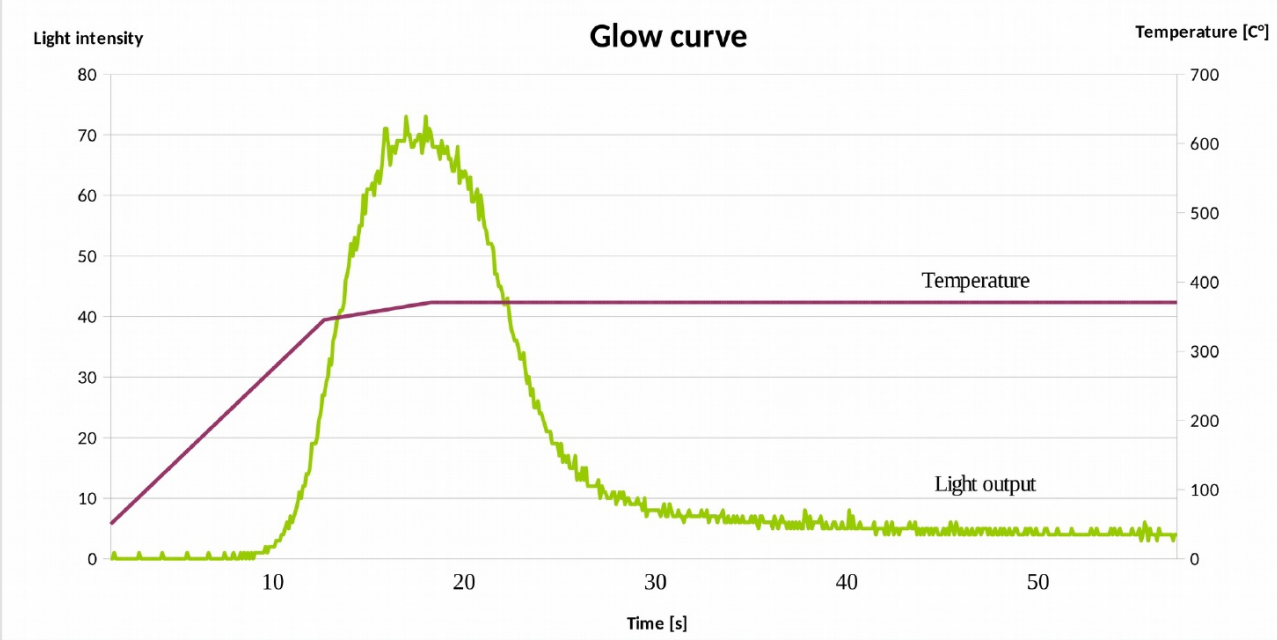

Figure 13: Glow curve with temperature plot - 20 Gy irradiated sample.

Despite the much improved glow curves there were large inconsistencies found in them even within the same dose batch. This is because silica yarn was wrapped around a piece of paper during irradiation which resulted in parts of it being fully exposed to strontium whilst other parts were shielded by the paper. This prevented plotting of TL signal as a function of dose chart for this experiment. However, the presence of well defined peak allows for interpretation of the acquired signal. As stated in theory section, electrons can recombine during heating, causing thermoluminescence effect, or they can be re-trapped [4]. This is probabilistic and dependent on the material itself. Both situations have been modeled and can be used to fit such model to experimental curve to investigate the material and find trapping energy. First order kinetic model is based on assumption that electrons predominately recombine [2]. Also assumed is that all traps are in single depth. With this in mind one can deduce that Light output 'I' must be proportional to de-trapping rate and multiply equations 1 and 3 to get:

$$I(t) = n_0 \cdot s \cdot e^{\left(\frac{-E}{kT}\right)} \cdot e^{\left(-s \cdot t \cdot e^{\left(\frac{-E}{kT}\right)}\right)} \tag{4}$$

With further assumption of constant heating rate $\beta = dT/dt$ and integrating from t_0 to t, Furetta and Pao-Shan [2] obtained:

$$I(T) = n_0 \cdot s \cdot e^{\left(\frac{-E}{kT}\right)} \cdot e^{\left(\frac{-s}{\beta} \cdot \int e^{\left(\frac{-E}{kT}\right)} dT\right)} \tag{5}$$

The result returns a rather narrow bell curve. Kitis et al. [16] have adapted the above equation for use in computer script or spreadsheet which requires parameters of maximum TL intensity and the temperature at that point, both of which are easily obtained from experimental data. Figures 14-16 shows example of silica yarn sample irradiated by 10 Gy and three attempts to fit first order kinetic curve over it. This was done in spreadsheet by adjusting the parameter E. This calculation can be found in appendix.

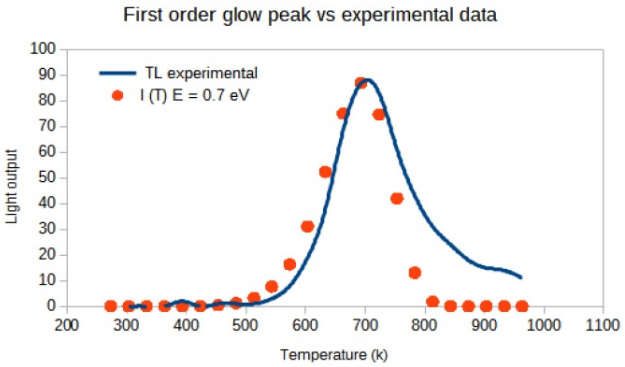

Figure 14: First order kinetic fit, E = 0.7 eV.

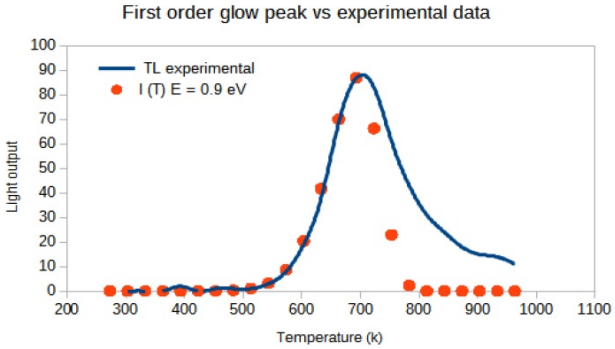

Figure 15: First order kinetic fit, E = 0.9 eV.

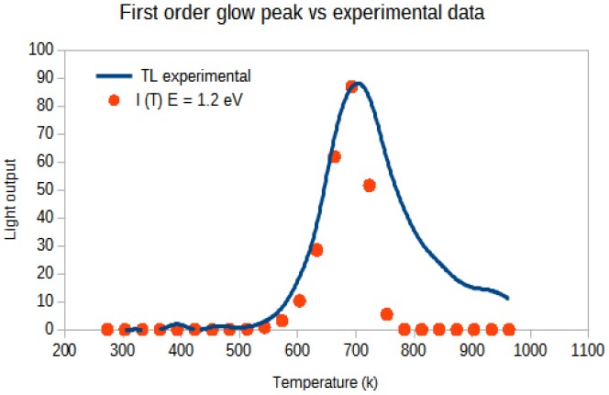

Figure 16: First order kinetic fit, E = 1.2 eV.

It is found that with low values of E (e.g. 0.7) points on leading side of the glow curve are above it. Similarly, higher values (e.g. 1.2) lead to dots lying below the curve. In all cases, however, no fit is achieved because the glow curve is too wide for this model as it is apparent in all cases. Second order kinetics yields better results as the curve is wider (Figures 18-19). This model is based on idea that both, re-trapping and re-combining of electrons take place.

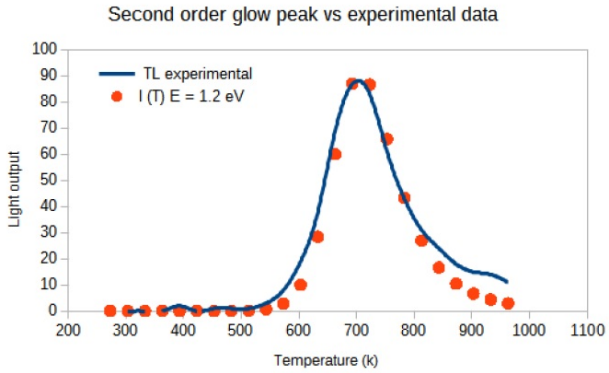

Figure 17: Second order kinetic fit, E = 1.2 eV.

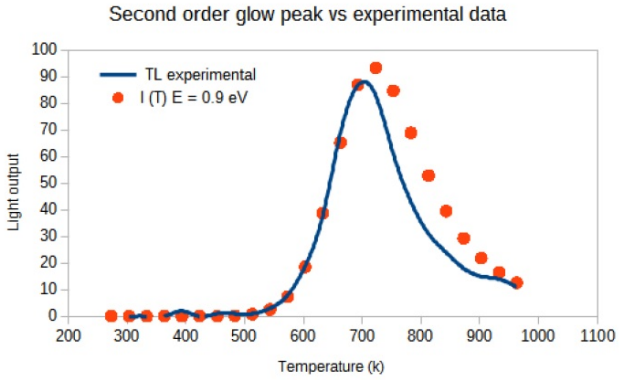

Figure 18: Second order kinetic fit, E = 0.9 eV.

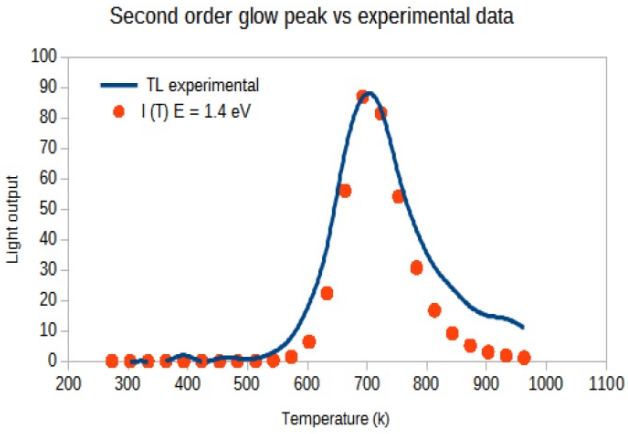

Figure 19: Second order kinetic fit, E = 1.4 eV.

The process of adjusting value E to fit the curve is the same as in previous case. Also in this situation no suitable fit was found in order to obtain trapping energy E. Factors behind the failure of curve fitting in this experiment are non-linear heating rates and large variation in curve shapes. There are many other models available but their utilization in this case was deemed unnecessary due to known experimental errors. Figure 20 shows a selection of best curves obtained in third experiment where 0.5 mm thick holder was used. Despite all efforts to make a precision holder, this version could not hold the glass disc in reproducible manner. Guiding rails were too wide allowing the glass to slide in at a different angle every time. This has caused a varying pressure on silica sample during readings rendering results unusable for analysis. Glass disc has been scratched by guiding rails multiple times in attempt to lock the sample inside. This in itself caused excitation of the material leading to more errors.

Metastatic states in all readings were not emptied and many readings did not resemble glow curve.

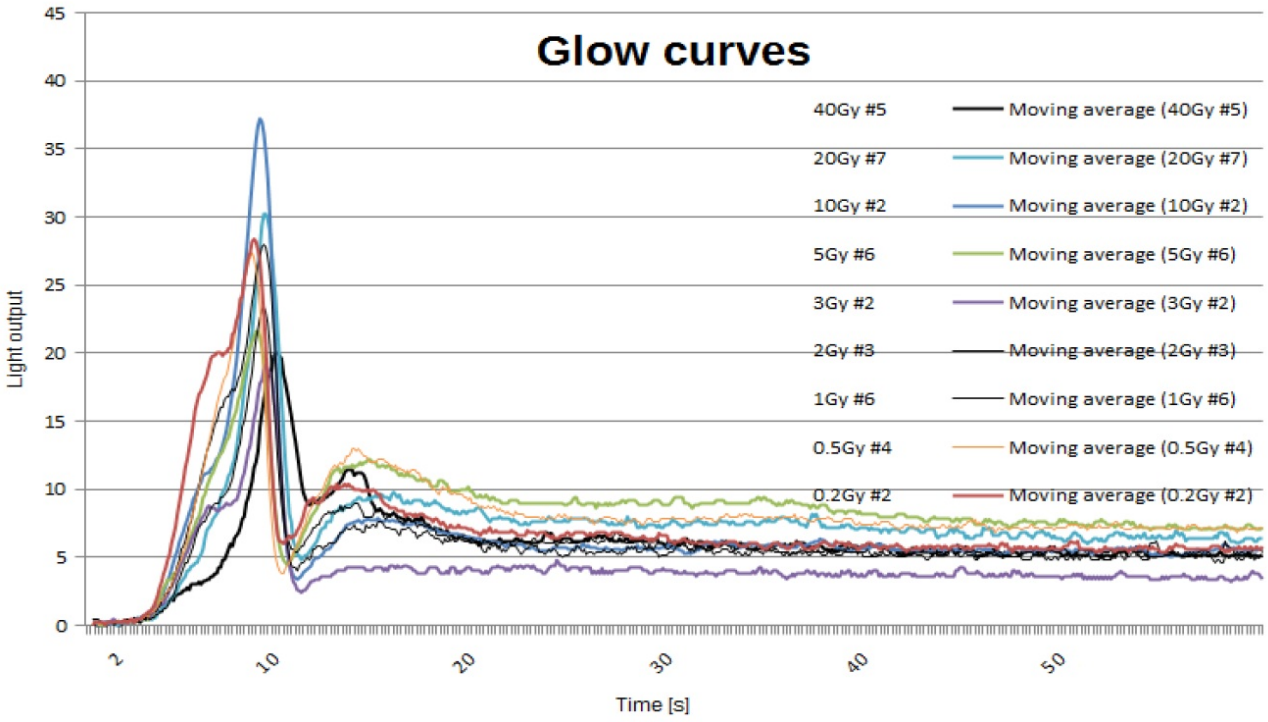

Figure 20: Experiment No. 3, selection of best glow curves.

5 Conclusions

Experimenting with silica yarn has shown its suitability for thermoluminescence dosimetry. The primary concern of this research was investigating silica yarn's response to radiation and subsequent dose extraction from it. Important linear radiation-dose characteristics was confirmed. So was the ability to react to wide dose range. Although the primary goal has been achieved, intricacies when working with any TLDs were felt. Negative results were down to improvisation with equipment inadequate for this purpose. Development of a suitable holder was a key to success in this project and even though all care has been taken to provide it, a high level of precision was not achieved. Development of a particular reader capable of scanning fabric type TLDs is essential in order to characterize silica yarn accurately. This is the future work of this project. Besides many advantages most TLDs feature, such as re-usability, low cost, and small size, silica yarn differentiate itself from most by physical flexibility. Fabric based nature of this material offers unique applications in dosimetry. It can be useful in medical environment during cancer therapy treatment as it can adapt to any shape of a patient' body. Or in personal dosimetry where sewing it into clothes can provide convenient wearable multidirectional detection device.

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