CHARACTERISTICS OF SOME TLD SOLID SYSTEMS

by

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INTRODUCTION

In the early TL research by Daniels^(1,2) and his coworkers, it was noted that the TL sensitivity of single crystals varied a good deal, even by a factor of two within the same crystal. Professor Daniels overcame this difficulty by grinding the crystals into powder and mixing well in order to average out this variability. Loose powder has been used for a number of years by placing it in plastic tubing, plastic capsules, etc. during irradiation from which it was removed for the reading process. The use of loose powder has an advantage in its flexibility; it can be used in various shapes and forms or even spread over the surface. It has also been uniformly distributed in a gelatin mixture in order to determine the average amount of radiation received in a relatively large volume. On the other hand, it has the disadvantage of being less convenient to handle and has to be measured out accurately for reading. Professor Daniels and his graduate students experimented with compressing the LiF powder into various metal pans approximately 1 cm in diameter by 2 mm thick. These pellets suffered from the disadvantage of having poor reproducibility as well as having a greater and more variable low temperature component in its glow curve⁽³⁾. Schulman and Attix⁽⁴⁾ from the Naval Research Laboratory

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experimented with putting $\text{CaF}_2\text{:Mn}$ and LiF powder into small glass capillaries approximately 1 mm od by 1 cm in length. These have been used by Roswit⁽⁵⁾ to measure radiation in vivo. The cost of these glass capillaries originally was approximately \$5.00 each; but they are now produced commercially by E.G. & G. Company, Goleta, California, at about \$2.00 each. E.G. & G. has also made rather elaborate dosimeters by coating the $\text{CaF}_2\text{:Mn}$ on a heating element inside an evacuated or Argon filled chamber. These units were made especially for personnel dosimetry and have not been widely used up to the present time. In the last year or so five different commercial concerns have developed solid approaches to the packaging problem. This paper is a preliminary report on three of these systems; the other two systems will be tried in the near future.

The first system involves the use of Teflon as a matrix in which about 30% weight of LiF powder is embedded. It is available in various forms, but for our measurements pieces were cut from tape 25 mm wide by 0.4 mm thick. This material has been developed by Controls for Radiation Inc., Cambridge, Massachusetts, and the product will be identified in this paper as LiF-T . A similar system, in which about 30% weight of LiF powder is embedded in a Silicon rubber product, has been developed by Madison Research, Inc., Middleton, Wisconsin, and will be denoted by LiF-S . A third approach is that by the Harshaw Chemical Company, the producers of the TLD-100 dosimetry powder which is most in current use. They have heated the crystals and extruded them through dies a couple of mm in diameter. Pieces of this material are then polished into rectangular parallelepipeds and appear transparent. This material

will be referred to as LiF-E in this report.

The fourth approach, which has not yet been tried, is a new method of packaging in glass by the Radiation Detection Company of Mountain View, California. The fifth approach is the glass needle by E.G. & G.

The first two systems (LiF-T and LiF-S) are embedded in materials which cannot withstand the standard annealing procedure of 400°C for one hour. It is also possible that the embedding procedure has changed some of the physical TL characteristics of the LiF. These two materials do, however, have the advantage of flexibility of use since they can be cut into a variety of shapes. All of the systems appear to be reusable under proper conditions. This report will give results on studies of the reproducibility of LiF-T, LiF-S, and LiF-E and will show changes of glow curves. Of the three types tested, it appears that the LiF-E has the most desirable characteristics as far as reproducibility and glow curve structures are concerned. It should be pointed out that LiF-E is not currently on the market and is undergoing further development. The studies reported here on LiF-E involve only about 6 samples of about 14 mg each. In the errors reported, it should be kept in mind that some of this error may be attributable to measuring apparatus independent of the material being measured; however, the integrated measurement from a light standard built into the reader gave results which were always within one per cent maximum deviation from the mean.

I, Reproducibility of LiF-T

Twenty-five samples of about 5 x 5 mm, weighing about 21 mg, were cut from the 0.4 mm thick tape. Each sample was given an identifying number, and its response was recorded for a series of 100R exposures by ^{137}Cs . They were read within 1 hour on the Clinical Reader described in an earlier publication⁽⁶⁾. The samples were not annealed between reading cycles. It was found that the first readings were lower by about 15% than the subsequent readings. If the virgin samples were given one reading cycle before irradiation, the first readings were the same as subsequent readings. The standard deviation of 5 repeated readings, excluding the initial reading, varied from 1.6% to 7.9% with an average of 4.7%. The standard deviation of the ct/mg/100R of the 25 samples was 4.7%. When the samples were marked to keep the same side up during each reading, the average standard deviation was reduced to 3.2%. When the samples were annealed at 100°C for 5 minutes after irradiation and prior to reading, the average ct/mg/100R was reduced by about 30% indicating a large low temperature component of the glow curve. This study will be repeated using the manufacturers recommended annealing cycle⁽⁷⁾ which reduces the low temperature contribution with a 2 hour annealing at 80°C. (See results of this annealing in Section V). The low temperature peak appears to contribute about 10% after 2 hours at 80°C annealing.

II. Reproducibility of LiF-S

Twenty-five samples of 2 x 4 mm weighing about 8 mg were cut from the 1 mm thick sheet. The samples were given multiple exposures of 100R and read repeatedly without any annealing between

readings. It was found that the first readings were lower by about 30% than the subsequent readings. This increase of sensitivity was not observed when the virgin samples were given one reading cycle before irradiation. The standard deviation of 4 repeated readings on the individual samples varied from 1.7% to 15.1% with an average of 6.7%. The standard deviation of the ct/mg/100R was 5.9%. When the samples were marked to keep the same side up during each reading cycle, the average standard deviation was reduced to 3.8%. Several annealing procedures are being studied in order to improve the reproducibility.

III. Reproducibility of LiF-E

Two samples of about 1 x 1 x 15 mm were received from the Harshaw Chemical Company, and each sample was cut into three pieces of about 5 mm in length. The average weight was 13.7 mg. The virgin samples were read for one cycle and then given a series of 100R exposures without any annealing between reading cycles. The standard deviation of 4 repeated readings varied from 1.2% to 3.2% with an average of 2.2%. The standard deviation of ct/mg/100R was 2.4%. The LiF-E had the best reproducibility among the three systems studied. It should be noted, however, that the number of samples was small and may not be indicative of the results to be obtained when they are produced in larger quantities.

IV. Glow curves of LiF-T, LiF-S, and LiF-E

Figures 1 and 2 show the glow curves of LiF-T and LiF-S, respectively. The LiF-T was a disc 5 mm in diameter and weighing about 14 mg. The LiF-S was a disc 7 mm in diameter and weighing about 37 mg. For the purposes of identifying the glow peaks, the glow

LIF-T

Fig. 1 Glow curves of LiF-T

Fig. 2 Glow curves of LiF-S

LiF TLD-100

— 100R ON VIRGIN
--- 100R REPEATED
W/O ANNEALING
... 100R REPEATED
100 °C FOR 10 MIN.

LIGHT EMITTED (ARB. UNIT)

Fig. 3 Glow curves of LiF(TLD-100) powder

curves of TLD-100 powder (~26 mg) are shown in Fig. 3. The glow curves of virgin samples all show the main peak 5. Repeatedly giving 100R on the same samples without any annealing, all the other peaks appear. Peak 3 and peak 4 are indistinguishable in both LiF-T and LiF-S; they are called peak 3,4 in this report. In LiF-T peak 5 decreased, which can be recovered by annealing at 80°C for a few hours (see Section V). Annealing at 100°C for 10 minutes after irradiation removed peak 1 and peak 2. The glow curves clearly show the increase of sensitivity after the first reading and the large decrease of sensitivity produced by annealing at 100°C after irradiation. Peak 1 decays at room temperature in the order of 10 minutes.

Figure 4 shows the glow curves of LiF-E. Due to the heat treatment during the manufacturing process, all of the five glow peaks appear in the initial glow curve. When one repeatedly gives 100R without any annealing, the low temperature peaks become more prominent and the high temperature peaks decrease slightly. Annealing at 100°C after irradiation removes peak 1, peak 2, and part of peak 3. Annealing 1 hour at 400°C followed by 24 hours at 80°C produced a glow curve similar to the usual shape of virgin LiF crystals such as shown in Fig. 3.

LIF-E

Fig. 4 Glow curves of LiF-E

V. Effect of 80°C Pre-annealing on the Glow Curves of LiF-T and LiF-S

LiF-T discs (about 14 mg and 5 mm in diameter) and LiF-S discs (about 36 mg and 7 mm in diameter), which had been previously irradiated and read, were annealed at 80°C for different lengths of time. The samples were then given 100R and read within 4 minutes after irradiation.

Figure 5 shows the change in LiF-T glow curves as a function of annealing time at 80°C. Peak 3,4 and peak 5 increase, and peak 1 and peak 2 decrease as the annealing time increases. After 3 hours at 80°C, peak 3,4 and peak 5 have been fused to become a single peak. Peak 1 and peak 2 continue to decrease with time. After 14 hours at 80°C, the glow curve has almost been restored to the shape of the virgin sample glow curve in Fig. 1.

Fig. 6 shows the change in LiF-S glow curve as a function of annealing time at 80°C. Peak 5 increases and the other peaks decrease as the annealing time increases. Peak 3,4 is no longer distinguishable after 1 hour at 80°C. After 2 hours at 80°C, the main peak has been essentially restored to its virgin shape. The low temperature peaks continue to decrease with time. After 14 hours the shape of the glow curves does not change with time.

Fig. 5 Effect of pre-irradiation annealing on the glow curve of LiF-T.

Fig. 6 Effect of 80°C pre-irradiation annealing on the glow curve of LiF-S.

VI Thermoluminescence of Silicon Rubber

The Silicon rubber without LiF embedded is slightly thermoluminescent and is sensitive to room light. The Silicon rubber discs and the LiF-S discs, which were the same size, were annealed at 300°C for 5 minutes to remove the TL due to previous exposure to room light or radiation. The samples were exposed to fluorescent room light on the working bench for 3 hours and then read. Figure 7 shows the TL produced by room light compared with that produced by 100R exposure of ^{137}Cs . Note that a scale change of 30 is involved. Since LiF is not light sensitive, it appears that Silicon rubber is. The integrated TL from LiF-S exposed to room light for 3 hours is about equivalent to that given by 0.5R. However, since the TL induced by room light has a low temperature glow peak, it can be almost completely removed by annealing at 100°C for 10 minutes before reading. If LiF-S is used in the low R range, the samples must be annealed at 100°C just before reading. LiF-T showed no light-induced TL

Fig. 7 Radiation and room light-induced TL:

- A. TL from LiF-S exposed to 100R
- B. TL (x30) from Silicon rubber exposed to fluorescent room light for 3 hours
- C. TL (x30) from LiF-S exposed to fluorescent room light for 3 hours

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