DEVELOPMENTS IN THIN FILM DOSIMETRY

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A thin film thermoluminescent dosimeter (TLD), formed by incorporating the TL phosphor into a plastic matrix of polyethersulphone (PES) (1) has been developed, (2). The advantages of a thin film are reduction of the correction for cavity effects and self-shielding, and reduction in the perturbation of the radiation flux especially at interfaces.

PES is available as pellets or powder, and is resistant to attack by most chemicals. The material dissolves slowly in ketones, esters and some halogenated carbons. In its use as a matrix for the phosphor it is spread into a thin film, incorporating the TL phosphor (usually LiF) at a much higher loading than in PTFE, without any serious loss in sensitivity. A further advantage of these TLDs is that the PES can be dissolved in the solvent using a washing and filtering technique and thus the phosphor may be reclaimed.

The thin film TLD originally developed (the PES/LiF TLD), used the novel method of solvent evaporation to manufacture the film. This has been modified to ensure more control over the product with the aim of achieving the following specifications:-

1. A phosphor grain size of 30μm ± 10μm (LiF loses sensitivity rapidly below 20μm grain size (3).
2. The LiF and PES should be homogeneously mixed.
3. Phosphor loading as high as possible.
4. Uniform film thickness within ± 10%.
5. Physical toughness in order to withstand normal working conditions.

For the work reported in this paper TLD 700 was obtained as a powder which was processed by repeated ball milling and sieving. Grains of phosphor between the 40μm and 20μm mesh sieves were used for dosimeter manufacture and the yield of usable phosphor was approximately 30% of the original weight. A 60% loading of phosphor was achieved with air dried PES dissolved in a constantly stirred 1:1 by volume mixture of Xylene and n-methyl-2-pyrrolidone.

The mixture is spread into a thin film using the Sheen Applicator, a hand tool used in the paint industry for spreading films of paint as test pieces. The thickness of the film is set by the metal shim thickness. All surfaces of the applicator are cleaned with acetone before use. The TLD mixture is deposited on one end of the glass plate and spread in one movement with a straight edged blade. The film is left to dry in air at room temperature for twentyfour hours, after which it is easily peeled off the glass. The dosimeter is cut to the desired size using a metal punch. Each film provides approximately 70 dosimeters of 90μm ± 10% thickness and with an average weight of 6 mg ± 10% at a diameter of 1 cm.

ANNEALING

A common difficulty with thin film dosimeters is the damage which can occur during annealing. The matrix may distort or even melt in the high temperature required for annealing.

The standard anneal cycle for LiF is 1 hour at 400°C followed by 16 to 24 hours at 80°C. Unfortunately the highest temperature that the PES plastic is

claimed to withstand without distortion or discolouration (1) is 200°C. Slightly higher temperatures can be used when the PES is used as a matrix for the dosimeter. Alternative annealing regimes were studied as follows:-

Regime 1 Anneal completed dosimeter for 1 hour at 255°C, the highest temperature at which the TLDs would just survive for 1 hour, followed by 16 to 24 hours at 80°C.
Regime 2 Anneal LiF powder for 1 hour at 400°C followed by 16 to 24 hours at 80°C, before film manufacture. The TLD is used with no further annealing.
Regime 3 Anneal LiF powder for 1 hour at 400°C followed by 16 to 24 hours at 80°C. After film manufacture anneal the TLDs for 1 hour at 200°C.

TLDs from each film and regime were given a calibration irradiation of 410R by a Co^{60} source. During irradiation the TLDs were placed in a perspex TLD holder, 6mm thick, and then covered by a 50μm sheet of black plastic.

The TLDs were read out in a Toledo 654 TLD reader, utilising the standard read cycle of:

16s preheat at 135°C
16s read at 240°C
16s anneal at 300°C

The reproducibility of each regime was compared, for fresh and reclaimed powder, so that a recommended annealing technique for the PES/LiF TLD could be established.

The criteria for the readout of background and irradiated TLDs are as follows:-

1. The standard deviation (σ) of readout of the calibrated TLDs must be less than 10% of the average value of readout.
2. Background readout should be less than or equal to 5% of the average value of readout of the irradiated TLDs.
3. The lower detection limit, equal to 3σ of the background should be less than 1% of the average readout of the irradiated TLDs.

Table 1 gives the background and calibration readout expressed in counts per gramme of TLD.

Regime 3 met these requirements for both fresh and reclaimed powder separately and also as a mixed batch so the recommended method of use for PES/LiF TLDs is:-

1. Anneal LiF in powder form → 2. Film manufacture → 3. Cut out TLDs and anneal (200°C)
↑ ↓
6. Dissolve in solvent and reclaim LiF ← 5. Readout and weigh ← 4. Irradiate

Possible improvements include:-

1. A longer anneal after manufacture (ie 80°C for 16 to 24 hours).
2. Reject TLDs exceeding 100μm in thickness or improve the shim method

of thickness control.

GAMMA MEASUREMENTS IN A REACTOR MOCK-UP

It was necessary to establish the thermal neutron response of the PES/LiF TLD (using TLD 700) before gamma measurements could be made in a reactor environment. The thermal neutron responses were determined in a graphite thermal column (cave D at the NESTOR reactor, AEE Winfrith). The gamma dose was subtracted by using measurements in a lithium carbonate ($^6Li_2CO_3$) pot which was installed in the graphite reflector and contained a graphite shell around the dosimeter. This gave a thermal neutron calibration of 1.3 R Co^{60} per 10^{10}n cm^{-2}. The calibration was repeated at CEN/SCK, Mol in the Cavity Standard Field of the BR1 reactor. In this case a lithium fluoride (^6LiF) pot was used and the calibration yielded 2.9 R Co^{60} per 10^{10} n cm^{-2}. This discrepancy was not important in this application but is being investigated.

The dosimeter was then compared with Beryllium Oxide dosimeters in the NESDIP (Nestor Dosimetry Improvement Programme) facility of the NESTOR reactor where the Pool Critical Assembly - a mock-up of a civil Pressurised Water Reactor shield was installed.

The results showed good agreement between the two dosimeters throughout a series of measurements in the water layer between the thermal shield and pressure vessel and through the steel pressure vessel. The maximum discrepancy between dosimeters was within 2σ in spite of the absence of cavity correction. BeO TLD (4) have been developed for use in graphite, to which they are well matched. In steel and water the mismatch in Z number will invalidate the calibrations of the TLDs which are performed in a Co^{60} field built up in graphite. For this reason the results must be regarded as interim while a fuller energy dependent calibration in different build up materials is being made and understood.

REFERENCES

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TABLE 1 - COMPARISON OF TLDs AGAINST THE MANUFACTURING METHOD
(Exposure 410 roentgens, Co^{60})

Regime	1			2			3		
Source of LiF	Fresh	Reclaimed	Mixture	Fresh	Reclaimed	Mixture	Fresh	Reclaimed	Mixture
Standard Deviation (σ) (% Signal)	5.01	7.66	11.7	8.57	15.82	21.8	4.73	6.15	9.91
Background (% Signal)	2.5	0.93	1.58	0.93	1.5	1.26	0.72	0.41	0.55
Standard Deviation of Background (% Signal)	0.39	0.07	0.07	0.29	1.36	1.36	0.24	0.02	0.02