FAST NEUTRON DOSIMETRY BY ACTIVATION OF TL PHOSPHORS

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Thermoluminescent phosphors are used as beta dosimeters to measure the dose from the decay of radioactive nuclei produced in the phosphor by fast neutrons via threshold reactions. The following reactions have been used: $^{19}\text{F}(\text{n},\text{Zn})^{18}\text{F}$, threshold=12 MeV; $^{24}\text{Mg}(\text{n},\text{p})^{24}\text{Na}$, threshold=7 MeV; $^{64}\text{Zn}(\text{n},\text{p})^{64}\text{Cu}$, threshold=4 MeV; $^{32}\text{S}(\text{n},\text{p})^{32}\text{P}$, threshold=2.5 MeV; together with activation at thermal thresholds of ^{164}Dy , ^{55}Mn , and ^{68}Zn . These nuclei were incorporated in the following phosphors: LiF , $\text{CaF}_2:\text{Dy}$, $\text{CaF}_2:\text{Mn}$, $\text{CaSO}_4:\text{Dy}$, $\text{Mg}_2\text{SiO}_4:\text{Tb}$, and ZnO:Tm . At 14 MeV, neutron fluences as low as 10^7 n/cm^2 can be detected. When the TL is read with photon counting techniques, one or more photons are detected for each beta disintegration so that the fundamental limits are the number of activations and the intensity of background sources, and not the TL sensitivity (LiF is an exception). Practical limitations are discussed.

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FAST NEUTRON DOSIMETRY BY ACTIVATION OF
THERMOLUMINESCENT PHOSPHORS

By: D. W. Pearson and P. R. Moran

INTRODUCTION

A standard method of fast neutron dosimetry is the activation of foils through threshold reactions. Such threshold activation detectors have the advantages of small size, reasonable sensitivity, and complete gamma discrimination. Simultaneous measurements with several detectors of different threshold energies allow one to reconstruct the incident neutron spectrum. However, measurement of the weak, neutron induced radioactivity of the detector foils requires a sensitive counting system. Only one detector can be counted at a time, and extensive measurements such as field scans or depth dose measurements thus would require vast arrays of counters. This disadvantage is most severe for low flux sources when the weak induced activity requires long counting times per detector which precludes serial counting.

Thermoluminescent (TL) phosphors are sensitive radiation detectors which can be used to measure low level radioactivity¹. Mayhugh and co-workers reported using a TL phosphor to measure its own thermal neutron induced radioactivity.² The technique, briefly, is to expose an appropriately chosen TL phosphor to thermal neutrons, inducing radioactivity as with ordinary activation foils. The TL produced directly by the neutrons and any other radiations, that is, the "prompt dose", is thermally removed and the phosphor samples - dosimeters - are left in a cool, dark place while the neutron produced radioactive nuclei decay. The subsequent TL signal is related to the total dose received from the decay of these nuclei. This neatly combines the activation detector and counter functions in one small, solid state device. Since each detector is its own integrating dosimeter, one has the equivalent of a virtually limitless array of counters. Reading each dosimeter

requires a short time which is independent of the signal level. Since the dosimeters can be read after the induced activity has decayed, the exact moment when the readout occurs need not be critical. Thus one can record serially, at ones convenience, data which were acquired in parallel by the individual dosimeters.

This technique is also effective for fast neutron activation via threshold reactions. However, since the fast neutron cross sections are considerably smaller than thermal neutron cross sections, the parent nuclei should be major constituents of the phosphor. We describe the direct use of TL phosphors as fast neutron activation detectors. The phosphors LiF , $\text{CaF}_2:\text{Dy}$, $\text{CaF}_2:\text{Mn}$, $\text{CaSO}_4:\text{Dy}$, $\text{ZnO}:\text{Tm}$, and $\text{Mg}_2\text{SiO}_4:\text{Tb}$ are considered.

PROCEDURE

I. Activation of TL Phosphors

The exposure of TL phosphors to fast neutrons results in the activation of certain nuclei according to the standard differential equation:

$$\frac{dN_A(t)}{dt} = -\lambda_A N_A(t) + N_P \int_0^{E_{\max}} \sigma(E) \phi(E, t) dE \quad 1.$$

$N_A(t)$ is the number of nuclei of isotope A at time t, λ_A is the decay constant, N_P is the number of parent nuclei, assumed essentially constant, $\sigma(E)$ is the differential cross section for the reaction. $\phi(E, t)$ is the differential neutron flux. Two conditions give simple solutions to equation 1. A short exposure time, $t_e \ll \lambda_A^{-1}$, gives the solution:

$$N_A = N_P \int_0^{E_{\max}} \sigma(E) \bar{\phi}(E) dE \quad 2,$$

where $\bar{\phi}(E) = \int_0^{t_e} \phi(E, t) dt$, the differential neutron fluence. If a constant flux rate is maintained, $\phi(E, t) = \phi(E)$, one has the other simple solution:

$$N_A(t_e) = \frac{N_P}{\lambda_A} \int_0^{E_{\max}} \sigma(E) \phi(E) dE (1 - e^{-\lambda_A t_e}) \quad 3.$$

Table I lists some of the common TL phosphors and the fast neutron reactions available in them. Only reactions whose products have half-lives from a few hours to several days are likely to be useful. Shorter lived isotopes require rapid handling and greater precision in timing the exposures and annealings. Long lived isotopes have consequently lower activity which gives a low rate of TL production, increasing the relative importance of background radiation and requiring a long wait for data. The threshold energies given in Table I are the values at which the cross section attains about ten percent of its maximum or its 14 MeV value.

In addition, Table I shows three thermal neutron induced reactions. Although the Dy and Mn isotopes occur only as TL activators, their low relative abundances of a few parts in 10^4 are compensated by their large capture cross sections for low energy neutrons. Hence the CaF_2 phosphors have complex activation functions and detect both fast and slow neutrons. For the $\text{CaSO}_4:\text{Dy}$ and ZnO:Tm phosphors, the difference in half lives between the thermal and fast neutron produced isotopes allows one to measure their activities separately.

Because the microscopic cross sections are all quite small, self-shielding can be neglected. The induced activity is also small so that contamination with exogenous radioactivity must be carefully avoided. Containers such as small gelatin capsules which can be discarded after use are convenient, and free of activating contaminants.

II. Annealing

In addition to the induced radioactivity, the exposure of TL dosimeters to fast neutrons produces a large TL signal from both fast neutron reactions and the interactions of their companion gammas. This prompt dose TL is two to three orders of magnitude greater than the TL from the subsequent decay of the neutron induced activity. This prompt dose TL must be removed completely, preferably in a time short compared to the half-life of the induced activity, and in a manner which preserves the sensitivity of the phosphor. Thermal annealing to 500°C in air serves this function for the phosphors used here.

For ease in handling, the solid dosimeters were placed in holes milled in aluminum blocks and numbered to identify individual dosimeters. Blocks of 16 or 20 dosimeters can thus be annealed simultaneously. The blocks reach 500°C about three minutes after being placed in the furnace. Two minutes later they are removed and cooled on a lead block which takes about two minutes. The powder phosphors which were exposed in gelatine capsules were transferred to aluminum foil packets to be annealed and then re-encapsulated.

The time interval, t_a , between the end of the exposure and the cooling of the dosimeters to ambient reduces the initial activity by a factor of $\exp \lambda t_a$. As a result, the annealing should be as short as possible and careful account must be kept of t_a .

II. Self Dose

After cooling, the annealed dosimeters are placed in lead containers to minimize background radiation while the neutron induced radioactivity decays. The TL signal grows with time according to the equation.

$$T(t_R) = k \left[\sum_A N_A(0) (1 - \exp(-\lambda_A t_R)) D_A f_A + D_B + f_B t_R \right] \quad 4.$$

The time from annealing to readout is t_R . The constant k describes the reader sensitivity. The summation is over the A different unstable isotopes whose initial numbers are $N_A(0)$. D_A is the average dose from one decay of isotope A , and f_A is the efficiency of TL production for this dose. D_B and f_B refer to the background dose rate and TL efficiency.

The average dose per decay can be written as:

$$D_A = \sum_i n_{Ai} \bar{E}_i \epsilon_i \quad 5.$$

The summation is over the i different particles emitted, with n_{Ai} being the relative abundance of the i^{th} particle of mean energy $\bar{E}_i$. ϵ_i is the fraction of this energy actually absorbed by the dosimeter.

Because of the small size of TL dosimeters, only betas contribute appreciably to the self dose. In addition, the ranges of these betas are not small compared to dosimeter dimensions, so that the absorbed fractions, ϵ_i , depend on dosimeter geometry and backscatter from the surroundings. The solid dosimeters have fixed geometry and one need only provide consistent backscatter to obtain reproducibility. This is another reason for the aluminum block holders mentioned in the previous section. For powder phosphors, #5 gelatine

capsules provide reproducibly shaped samples large enough for 5 or 6 TL readings. Lead foil 0.2 mm thick wrapped around the capsules gives reproducible backscatter and excludes light. Most TL phosphors show some light sensitivity and so were handled under red safelight.

Several considerations influence the length of time one should wait for the self dose. First, from equation 4, one sees that after a time t_R equal to six half lives of an isotope, more than 98% of the potential self dose from that isotope has been delivered. Since other uncertainties in the measurements are usually greater than 2%, the maximum signal is attained in about six half lives. Second, if the activity is high, one can wait any convenient time which gives an adequate TL signal, and use the factor $(1 - \exp(-\lambda_A t_R))$ to correct to the total initial activity. Third, for low activities, the only source of error which is a function of t_R is the uncertainty in the dose from the background radiation. This uncertainty grows as $t_R^{1/2}$ but is smaller than instrumental uncertainties for times less than about a day. We commonly use one day for the ^{18}F and ^{165}Dy isotopes, three days for the ^{64}Cu and ^{24}Na isotopes and 14 days for the ^{32}P isotope. These times are sufficiently short that TL fading effects are not significant.

IV. Readout of the TL

Since the self dose is small - a few milli rads for a fast neutron fluence which would deliver rads to tissue - , the TL reader must have high sensitivity and stability. The photon counting technique offers the ultimate in sensitivity, precision and stability.³ The intrinsically digital nature of a small TL light signal is preserved for convenient computation. We use a modified Harshaw mode 2000A reader with an SSR 1120 photon counting amplifier-discriminator. A temperature gated, up/down counter sums the photon counts from the appropriate glow peaks. Glow curves and integration limits are shown in Figs 1 through 6. A second heating cycle subtracts the PM tube current and heater thermal incandescence emission. The heating chamber is flushed with helium gas to improve the thermal contact and suppress spurious TL. Flushing with an inert gas to eliminate oxygen has long been known to suppress spurious TL.⁴ Helium provides increased thermal contact to give more reproducible heating, and cools the heater twice as fast as nitrogen. The reader is described in detail elsewhere⁵.

The solid dosimeters are carefully positioned directly on the platinum heating planchet. Powder phosphors are read as 20 mg to 40 mg samples spread evenly in silver pans (12 mm diam) placed on the planchet and subsequently weighed. The powders should be mixed thoroughly before dispensing since the self dose irradiation is nonuniform. Careful mixing by pouring between two gelatine capsules does not introduce measurable spurious TL for the $\text{CaSO}_4:\text{Dy}$ and ZnO:Tm phosphors.

V. Analysis

Eventually a set of TL measurements must be related to the original neutron fluence. One can rewrite Eq. 4 for the short exposure case of Eq. 2 as :

$$\text{TL}(t_R) = k \sum_A N_p (1 - \exp(-\lambda_A t_R)) D_A f_A \int_0^{E_{max}} \sigma(E) dE + k D_B f_B t_R \quad 6.$$

Defining a sensitivity function

$$S_A(t_R) = k N_p (1 - \exp(-\lambda_A t_R)) D_A f_A \quad 7.$$

and calling the TL from background, B:

$$B = k D_B f_B t_R \quad 8.$$

we get

$$\text{TL}(t_R) - B = \sum_A S_A(t_R) \int_0^{E_{max}} \frac{1}{V_A(E)} \Phi(E) dE \quad 9.$$

Likewise for the constant flux case one has

$$\text{TL}(t_R) - B = \sum_A S_A(t_R) (1 - \exp(-\lambda_A t_R)) \lambda_A^{-1} \int_0^{E_{max}} \frac{1}{V_A(E)} \phi(E) dE \quad 10.$$

The sensitivity, $S_A(t_R)$, can be measured with any calibrated neutron source having adequate flux rate at an energy for which the activation cross sections are well known. Judicious choice of the neutron energy reduces the summation over A to a single isotope.

The net TL signal, $(\text{TL}(t_R) - B)$, is a measure of the activity which decayed between the end of the annealing, t_a , and the time of readout, t_R . To correct this to the activity at the end of the exposure, one multiplies by $\exp \lambda_A t_a$.

For phosphors with two activated isotopes, the summation in equations 9 and 10 can be eliminated in different ways. If the decay rates of the isotopes are sufficiently different, $\lambda_1 \geq 10 \lambda_2$ as in the case of ^{32}P and ^{165}Dy in the

CaSO₄:Dy phosphor, their activities can be measured separately by reading the phosphor at two different times. The first reading at time $t_{R1} \geq 4\lambda_1^{-1}$ measures all the short lived activity plus a fraction $(1 - \exp(-\lambda_2 t_{R1}))$ of the longer lived activity. Subsequent rereading at time t_{R2} determines the long lived activity. One has then

$$(TL(t_{R1}) - B(t_{R1}) - (1 - \exp(-\lambda_2 t_{R1})) S_2(t_{R1})) \exp \lambda_1 t_a = S_1(t_{R1}) \int_0^{E_{max}} \sigma_1(E) \Phi(E) dE \quad 11.$$

and

$$(TL(t_{R2}) - B(t_{R2} - t_{R1})) \exp \lambda_2 t_{R1} = S_2(t_{R2}) \int_0^{E_{max}} \sigma_2(E) \Phi(E) dE \quad 12.$$

If the decay rates are comparable as for the ¹⁸F and ¹⁶⁵Dy isotopes in the CaF₂:Dy phosphor, one can construct an effective cross section:

$$\sigma_{eff}(E) = \frac{S_{Dy}(t_R)}{S_F(t_R)} \sigma_{Dy}(E) + \sigma_F(E) \quad 13.$$

and use $S_F(t_R)$ as the effective sensitivity.

Thus one can always obtain a set of equations of the form:

$$TL / S = \int \sigma \Phi dE \quad 14.$$

Several methods exist to solve this set of equations for the neutron fluence $\Phi(E)$, developed for conventional threshold activation dosimetry which can be used directly.⁶

EXPERIMENTAL

The sensitivities, $S_A(t_R)$, defined in section V can be measured with any calibrated neutron source of sufficient intensity. Using a monoenergetic source simplifies the calculations. The ³H(d,n)⁴He reaction yields essentially monoenergetic 14.7 MeV neutrons. We used a 2 cm diameter beam of 200 kV deuterons on a thick TiT, 1:1, target. A constant flux rate is difficult to maintain with this machine.⁷ Therefore, exposures short enough to approximate the pulsed exposure case of equation 2 while providing large enough fluences for accurate determinations of the sensitivities were indicated. Ten minute exposures, 10 cm from the target in the forward direction, gave fluences of approximately 10^{10} n/cm². At first fluences were measured with conventional activation detectors of ³²S, ⁶³Cu and ¹⁹F, which agreed with one another within 10%. The ¹⁹F(n, 2n)¹⁸F reaction was the most convenient and served subsequently as the standard for inter-exposure normalizations.

The sensitivities were calculated from the TL data with equation 9 using the cross sections in Table I. The sensitivities listed in Table II are average values of $S_A(t_R \approx 4\lambda_A^{-1})$, except for ³²P in the CaSO₄:Dy phosphor where $S_A(t_{1/2})$ is given. The solid dosimeters of LiF, CaF₂:Dy and CaF₂:Mn are of questionable parentage and variations in sensitivity between dosimeters of as much as 25% was found. These variations in sensitivity paralleled variations in their response to ¹³⁷Cs gammas. Typical values of the gamma responses are also shown in Table II for comparison.

The thermal column of the UW nuclear reactor provided the thermal neutrons used to measure the thermal neutron sensitivities given in Table II. Exposures

of two minutes gave fluences of 10^{10} n/cm² as measured by activation of calibrated gold foils. The sensitivities $S_A(t_R \geq 4\lambda_A^{-1})$ were calculated from equation 9 in the same manner as the fast neutron sensitivities.

Repeated exposure of the same dosimeter to 10 mR of ¹³⁷Cs gammas resulted in the standard deviations given in Table III. Standard deviations of the neutron sensitivities for different dosimeters given by their respective gamma responses are shown in Table III. For similar TL light sums, these errors are seen to be comparable.

DISCUSSION

Limitations

There are both experimental and fundamental limits on the accuracy with which the neutron fluence can be determined from TL measurements.

From equation 9 we get

$$(TL - B)/S = \int \sigma(E) \bar{I}(E) dE \quad 15.$$

The quantities σ and S are measured parameters which may contain unknown systematic errors. The values of TL and B exhibit statistical errors as well. Experimentally, the determination of $(TL - B)$ involves four independent measurements. Explicitly

$$(TL - B) = \underset{1}{(TL + D)} - \underset{2}{D} - \underset{3}{((B + D))} - \underset{4}{D} \quad 16.$$

where D is the photomultiplier dark current and the thermal emission of the heater and phosphor during that portion of the heating cycle in which the glow curve is integrated. The statistical error for the TL measurement is given by

$$\sigma_{(TL-B)}^2 = \sigma_{(TL+D)}^2 + \sigma_{(B+D)}^2 + 2\sigma_D^2 \quad 17.$$

There are also more fundamental limitations. There is the well known statistical uncertainty for N decay betas, $\sigma^2 = N$. There is also a more subtle error introduced by the assumption

$$E = \frac{1}{N} \sum_{i=1}^N E_i \quad 18.$$

implicit in equation 5 for the dose. Converting from the measured dose to a number of decay particles involves inverting equation 18 to obtain

$$N = \frac{1}{E} \sum_{i=1}^N E_i \quad 19.$$

For large N equation 19 is acceptable. However for small N, the distribution in decay energy of the betas introduces an additional uncertainty in the value of N determined from equation 19 of the amount

$$\frac{\sigma^2}{N} = \frac{\langle E^2 \rangle}{\langle E \rangle^2} - 1 \quad 20.$$

where $\langle E \rangle$ and $\langle E^2 \rangle$ are the first and second moments of the beta decay energy spectrum. This is basically because the energy deposited over-represents the high energy decays. The expression in parenthesis in equation 20 can be of the order of .4 for an allowed beta decay spectrum. The smallest error possible for determining N particles from a dose measurement is then

$$\frac{\sigma}{N} = \frac{\sqrt{\langle E^2 \rangle}}{\langle E \rangle} \quad 21.$$

It should be noted that equation 20 derives only from using a deposited energy or dose to determine a number of fluence of particles. It is valid for other particles having an energy distribution such as protons from n-p scattering and for other readout techniques such as TSC or TSEE. The N in equation 20 is the number of primary particles interacting with the detector. Increasing the efficiency of conversion of these into measured particles, eg TL photons, conduction electrons or exoelectrons, does not decrease this uncertainty.

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7. The machine is an Activatron 211 M from Accelerators Inc.

TABLE I

PHOSPHOR	REACTION	THRESHOLD	CROSS SECTION	DECAY MODE	HALF-LIFE
LiF	$F^{19}(n,2n)F^{18}$	12 MeV	50mb @ 14 MeV	β^+ , .64 MeV	109 min
$CaF_2:Dy$	$F^{19}(n,2n)F^{18}$	12 MeV	50mb @ 14 MeV	β^+ , .64 MeV	109 min
	$Dy^{164}(n,\gamma)Dy^{165}$	Thermal	2.8 kb	β^- , 1.3 MeV	140 min
$CaF_2:Mn$	$F^{19}(n,2n)F^{18}$	12 MeV	50mb @ 14 MeV	β^+ , .64 MeV	109 min
	$Mn^{55}(n,\gamma)Mn^{56}$	Thermal	13 b	β^- , 2.8 MeV	2.5 hr
$CaSO_4:Dy$	$S^{32}(n,p)P^{32}$	2.5 MeV	250mb @ 14 MeV	β^- , 1.7 MeV	14 day
	$Dy^{164}(n,\gamma)Dy^{165}$	Thermal	2.8 kb	β^- , 1.3 MeV	140 min
$ZnO:Tm$	$Zn^{64}(n,p)Cu^{64}$	4 MeV	200mb @ 14 MeV	EC, β^+ , .6 MeV	12.8 hr
	$Zn^{68}(n,\gamma)Zn^{69}$	Thermal	1 b	β^- , .9 MeV	57 min
$Mg_2SiO_4:Tb$	$Mg^{24}(n,p)Na^{24}$	7 MeV	200mb @ 14 MeV	β^- , 2.8 MeV	15 hr

TABLE II
SENSITIVITIES

PHOSPHOR	MASS mg	S (^{137}Cs) photons/mR	S THERMAL photons/n/atom	S 14 MeV photons/n/atom
LiF	23	360	nil	3.5×10^{19}
$CaF_2:Dy$	27	1.3×10^4	1.5×10^{17}	8×10^{20}
$CaF_2:Mn$	50	5.7×10^3	6×10^{18}	3.5×10^{20}
$CaSO_4:Dy$	40	1.5×10^4	6×10^{16}	2×10^{20}
$ZnO:Tm$	40	2.5×10^4	2×10^{18}	4.5×10^{20}
$Mg_2SiO_4:Tb$	20	1.6×10^4	*	5.5×10^{20}

* It has not been determined which isotope is responsible for the thermal neutron sensitivity of this phosphor.

TABLE III

PHOSPHOR	MEASURED STANDARD DEVIATIONS	
	10 mR ^{137}Cs	10^9 n/cm^2 (14 MeV)
LiF	4 %	6 %
$\text{CaF}_2:\text{Dy}$	1.5 %	3 %
$\text{CaF}_2:\text{Mn}$	2 %	4 %
$\text{CaSO}_4:\text{Dy}$	5.5 %	11 %
$\text{ZnO}:\text{Tm}$	6 %	7.5 %
$\text{Mg}_2\text{SiO}_4:\text{Tb}$	5 %	6 %

FIGURES

Figures 1 through 6 show the glow curves for the phosphors investigated. The curves are on the same scale and the listed exposures give an idea of the relative sensitivities. The arrows show the limits of integration.

Figure 1. Glow curve of LiF.

Figure 2. Glow curve of $\text{CaF}_2:\text{Dy}$.

Figure 3. Glow curve of $\text{CaF}_2:\text{Mn}$.

Figure 4. Glow curve of $\text{Mg}_2\text{SiO}_4:\text{Tb}$.

Figure 5. Glow curve of $\text{ZnO}:\text{Tm}$.

Figure 6. Glow curve of $\text{CaSO}_4:\text{Dy}$.