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PRE — AND POST — ANNEALING PROPERTIES OF LOW TEMPERATURE GLOW PEAKS 2 AND 3 IN LIF:Mg

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PRE- AND POST-ANNEALING PROPERTIES OF LOW TEMPERATURE

GLOW PEAKS 2 AND 3 IN LIF:Mg

Shigueo Watanabe*

ABSTRACT

Properties of the low temperature glow peaks 2 and 3 in LiF:Mg have been investigat ed after removing the main peaks, 4 and 5, by annealing at 400° C for ⁻¹ hour followed by 175°C for 9 to 17 hours. EG & G 'LiF mini-dosimeters were used. A definite supralinearity was observed for both peaks beginning between 500 and 1,000 rad of $^{-37}$ Ce -ray, with peak 3 presenting a slightly more pronounced supralinearity. S/S₀ values, where S is the sensitize ed TL (thermoluminescence) value and S is the unsensitized one for a same exposure, were also measured after post-annealing at 280°C for one-half and one hour, or at 175°C for one hour, in each case after a sensitizing irradiation. S/S₀ and S depend upon both the tempera ture and duration of the post-annealing. Also, the main peak number 5, first removed by preannealing at 175°C, is restored with sensitizing irradiation and 280°C annealing. The height of this restored peak is a function of the initial sensitizing dose.

Thermoluminescence properties of ⁷LiF mini-dosimeters that were annealed repeatedly at 400°C for one hour, 175°C for 10 hours, 280°C for 10 hours, sometimes irradiated to 100 rad and read in between annealings, were also investigated. The shape and the height of glow peaks depend on the last annealing temperature. The dependence of the TL response on the pre annealing temperature for a fixed annealing time of 10 hours was also investigated. Mini-dom meters irradiated to high dose, read, and then annealed at 80°C for 1/2 hr to 3 hr present a large increase in their sensitivity. A similar result was obtained with annealing at 175°C for 1 hour followed by 80°C for 1 hour, instead of 80°C annealing only.

1 - INTRODUCTION

An extensive study has been carried out by Zimmermann, Rhyner and Cameron¹ concerning the pre-irradiation annealing effects on dosimetry LiF, mainly considering peaks 4 and 5 called main dosimetry peaks.

Among several isothermal pre-irradiation annealing temper atures, the following ones present the most interesting properties:

(a) 80°C pre-annealing for times longer than 10 hours

reduces the low temperature peaks 1, 2 and 3, leav-Instituto de Energia Atômica and Instituto de Física da Universidade de Sao Paulo, Sao

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ing the main peak with 95% of its normal height;

- (b) 175^oC pre-annealing for times longer than 3 to 4 hours reduces peaks 4 and 5 to almost zero and enhances peaks 2 and 3 by a factor of about 4;
- (c) 280°C or 400°C pre-annealing for times less than 24 hours has little effect on the shape or size of the glow curve other than a slight increase in the height of peak 2;
 - (d) 400[°]C annealing for 1 hour restores the original shape and size of the glow curve of dosimetry LiF

after any kind of pre-annealing treatment at or below 400° C, except for LiF annealed at or above 400° C for more than 48 hours.

Cameron et al.² reported an enhancement in the sensitivity of LiF by 280° C annealing for 1 hour after irradiation to 3,000 to 5,000 rad.

All these results indicate that thermal annealing has an important role in creating or destroying trapping centers. For example the reduction of the main peaks by $175^{\circ}C$ pre-annealing and the restoration by $400^{\circ}C$ annealing are essentially thermal destruction and creation of trapping sites. On the other hand, the supralinearity appears to depend only on the radiation and not on the heat treatment.

Since little attention was given to low temperature peaks, and since such study may contribute to the elucidation of the the<u>r</u> moluminescence mechanism, such an investigation was carried out using 175°C pre-annealing to eliminate peaks 4 and 5 from the glow curve.

2 - EXPERIMENTAL PROCEDURES

The EG & G* miniature dosimeters TL-23 (⁷LiF dosimeters)

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^{*} Edgerton, Germenhausen & Grier Inc., Santa Barbara Division.

and the EG & G model TL-3B reader were used throughout this work. In every measurement the samples were pre-annealed at 400°C for one hour followed by 175° C for times which varied between 9 and 17 hours. Two 137Cs γ -rays sources, one with dose rate 50.5 rad / min and the other one with 7,600 rad/min were employed.

In Fig. 1, curve <u>a</u> represents the glow curve of mini-dosi meters irradiated to 100 rad, and curve <u>b</u> to 500 rad. It is evident that peaks 4 and 5 are absent. The sharp peaks indicate changes in the scale.



FIGURE 1 - Glow curve of mini-dosimeter annealed at 400°C for 1 hour and then at 175°C for 17 hours, (a) 100 rad, (b) 500 rad. Sharp peaks indicate changes in the scale.

. 3.

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3 - SUPRALINEARI

Fifty-four mini-dosimeters treated as described in the previous Section, were divided into 9 groups. Each one was then irradiated to different absorbed doses varying between 10 rad and 25,000 rad. The upper limit is dictated by the upper limit in the reading scale of the EG & G reader. The dosimeters were read out within 2 hours after irradiation. The TL reading increased slight ly (less than 6%) as the time elapsed since irradiation increased.

Figure 2 shows TL vs. absorbed dose for peaks 2 (curve <u>a</u>) and 3 (curve <u>b</u>) of the mini-dosimeters. Each experimental point is an average of 6 readings. The dashed line represents linear response; hence, we see that supralinearity begins between



FIGURE 2 - TL vs. rad curves of mini-dosimeters, (a) peak 2, (b) peak 3.

500 and 1,000 rad. The height of peak 3 is always lower than that of peak 2; however, peak 3 is slightly more supralinear than peak 2.

4 - MEASUREMENTS OF S/S

Several mini-dosimeters annealed as described in Section 2 were irradiated to different dose levels varying from 10 rad to 400,000 rad. At each level they were divided into 3 groups. One was annealed at 280° C for half an hour, the second group amealed at the same temperature for one hour, and the last one at 175° C for one hour. They were then exposed to a 100 rad test dose and read 30 minutes later. The mini-dosimeters were read after first series of irradiations, but, before 280° C annealing.

Figures 3, 4 and 5 show S/S_c vs. previous dose curves for the above post-annealings (here, "post-annealing" means anneal



FIGURE 3 - S/S vs. previous dose for mini-dosimeters post-annealed at 280°C for 1/2 hr (a) peak 2, (b) peak 3.

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FIGURE 4 - S/S_0 vs. previous dose for mini-dosimeters post-annealed at 280^oC for 1 hour, (a) peak 2, (b) peak 3.



E-GURE 5 - S/S₀ vs. previous dose for mini-dosimeters post-annealed at 175°C for 1 hour, (a) peak 2, (b) peak 3.

ing after the sensitizing dose is given, but, before giving the test dose). Again each experimental point is an average of 5 to 6 readings.

It is interesting to note that with 280°C post-annealing (Figs. 3 and 4) peak 3 is higher than peak 2 at every level while with 175°C post-annealing, above 10,000 rad sensitizing dose, the opposite is true.

Figure 6 presents glow curves of mini-dosimeters annealed at $175^{\circ}C$ after being irradiated to 1,000 rad (curve <u>a</u>) and to 400,000 rad (curve <u>b</u>). Curve <u>b</u> shows a peak beyond the main peaks.



FIGURE 6 - Glow curves of mini-dosimeters post-annealed at 175°C for 1 hour, (a) previous dose - 1,000 rad, (b) previous dose - 400,000 rad.

In Fig. 7 glow curves of mini-dosimeters annealed at 280°C for half an hour after irradiation to 1,000 rad and at 280°C for one hour after 25,000 rad are presented. Both dosimeters received a 100 rad test dose after being read. In each case restoration of main peaks was observed, but we could not infer whether there is any peak beyond the main peaks.



FIGURE 7 - Glow curves of mini-dosimeters post-annealed at 280°C, (a) 1/2 hour postannealing and 1,000 rad previous dose, (b) 1 hour post-annealing and 25,000 rad previous dose.

5 - DEPENDENCE OF S ON POST-ANNEALING TIME OF SENSITIZED LIF MINI

DOSIMETERS

After being irradiated to 100,000 rad, several mini-dosi meters, pre-heated the same way as above, were separated into groups and were annealed at 280° C for 1/2, 1, 2, 5 and 10 hours. They were then irradiated to 100 rad test dose and their TL respon se S was measured. Each group consisted of 5 to 6 samples.

Due to relatively large fluctuation encoutered in the readings for peaks 2 and 3 no definite conclusion can be drawn. If there is a drop in sensitivity with increasing post-annealing time, it must be very small.

Since 175° C pre-annealing eliminates peak 5, S_o for peak 5 is zero. Therefore, we plotted in Fig. 8 and Fig. 9, S vs. pre-vious dose peaks 2, 3 and 5, rather than S/S_o.



FIGURE 8 - S vs. previous dose for mini-dosimeters post-annealed at 280°C for 1/2 hour

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FIGURE 9 - S vs. previous dose for mini-dosimeters post-annealed at 280°C for 1 hour.

In Fig. 10, S vs. post-annealing time is represented. In all three cases, the post-annealing temperature is 280°C. The out standing result is that the traps causing peak 5 are recreated due to the post-annealing treatment; furthermore, peak 5 appears to reach its largest height after about one hour annealing at 280°C. Another striking result is that the restored peak remembers the level of irradiation to which the sample was subjected.

6 - THERMAL TRANSFORMATION OF TL TRAPS

Based on results found in Ref. 1 and the present work, one can assume that thermal annealing causes a change in the nature as well as in the number of traps. Such a change can be interpreted as a creation or destruction of complexes comprising trapping centers. The destruction of a center is not necessarily an actual dissociation of a complex; it may be an association



FIGURE 10 - S vs. post-annealing time at 280°C. Previous dose - 10,000 rad.

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that changes the existing center into another kind of center. Thus, with a pre-annealing at some temperature, all or a part of the traps corresponding to a given peak can be destroyed, and traps corresponding to other peak or peaks can be formed. Those traps destroyed by annealing at one temperature can be restored by annealing at another temperature.

To investigate to what extent TL response is dependent on thermal annealing alone, the series of measurements described below was performed.

Several mini-dosimeters were pre-annealed at 400°C for one hour followed by 175°C for 10 hours. They were then subjected to a succession of annealings, irradiations, or readings according to the scheme presented in Table 1. Every reading took place 30 minutes after the 100 rad test dose.

TABLE 1 - Scheme of annealings, irradiation and read-outs



The capital letters (P), (Q), (R), etc. indicate groups of 5 or 6 mini-dosimeters read-out at the stage shown in Table 1 $175^{\circ}C-10$ means 10 hours annealing at $175^{\circ}C$. Thus the group (Y), for example, was irradiated to 100 rad and read after a succession of $400^{\circ}C$ for one hour, $175^{\circ}C$ for 10 hours, $280^{\circ}C$ for 10 hours,

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 400° C for 1 hour, while the groups (P) and (K) were annealed at 400° C for 1 hour, 175° C for 10 hours, irradiated to 100 rad, and then read-out (group P), subsequently annealed at 280° C for 10 hours, irradiated to 100 rad, and then read-out again (group K).

Figure 11 shows the glow curves corresponding to group (P), (R), (W), (V), and (U), all of them with the last annealing temperature of 175° C for 10 hours. They were, however, treated differently prior to the final 175° C annealing.



TIME

FIGURE 11 - Glow curves of groups (P), (R), (W), (V) and (U) characterizing the last anneal ing temperature of 175°C (for 10 hours).

Figure 12 presents those ending with 400° C for 1 hour groups (Q), (X), (Z), and (Y) and Fig. 13, those of (T), (K),(S), (O), (M), and (N), all ending with 280° C annealing. We see that the shape and the size of glow curves depend on the last preannealing temperature only, if the irradiating dose does not exceed the value which gives rise to supralinearity. Looking at the glow curve one can usually guess what kind of pre-annealing a dosimeter was subjected to.

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FIGURE 12 - Glow curves of groups (Q), (I), (Z), and (Y) characterizing the last annealing temperature of 400°C (for 1 hour). Due to the change in scale the form of the glow curves looks different, although they are not.



FIGURE 13 - Glow curves of groups (T), (K), (S), (O), (M), and (N), characterizing the last temperature of 280°C (for 10 hours).

7 - PRE-ANNEALING TEMPERATURE DEPENDENCE OF TL RESPONSE FOR A

FIXED ANNEALING TIME OF 10 HOURS

It is evident from Ref. 1 that for a fixed pre-annealing time, the TL response of LiF is a function of the pre-anneal ing temperature. In order to find more explicitly this temperature dependence, TL measurements were carried out, using LiF mini dosimeters first annealed at 400°C for one hour. The duration of subsequent annealing was fixed at 10 hours at 60, 80, 100, 105,



FIGURE 14 - TL vs, pre-annealing temperature for 10 hours annealing time.

110, 115, 120, 130, 140, 150, 165, 190, 220, 240, 280, 315, 342, and 360°C.

Figure 14 presents curves showing the way the height of peaks 2, 3 and 5 change when the pre-annealing temperature is var ied. Each experimental point is an average of 6 readings.

In Fig. 15 the glow curves for 100°C to 165°C pre-anneal ing temperatures are shown. A gradual shift of peak 5 toward a high temperature region can be seen, indicating that the peak called number 6 in Ref. 1 may merely be peak 5 shifted from its usual position. In fact, this gradual shift can also be seen in



Fig. 6 of Ref. 1 at 175°C pre-annealing temperature as the anneal ing time is increased. On the other hand, Fig. 11 of Ref. 1 shows that post-annealing at 132°C for 3 hours or longer, resolves peak 5 into peak 5 itself and peak 6. There is the possibility that what is considered to be peak 6 is just peak 5 displaced, while what is thought as being peak 5 is nothing more than peak 4 displaced by post-annealing treatment.

Peak 4 was observed distinctly only at pre-annealing temperatures higher than 280°C. Its measured TL values at 280°C, 315°C, 342°C, and 360°C are ~7 in the units of Fig. 14.

There seems to be a strong correlation between peaks 2 and 3. Figure 14 confirms this statement in that peaks 2 and 3 always come and go together. This may be because they originate from the same complex.



TIME

FIGURE 16 - Glow curves of mini-dosimeters post-annealed at 175°C for 1 hour followed by 80°C for 1 hour, (a) 10,000 rad previous dose, (b) 50,000 rad previous dose (c) and (d) 100,000 rad previous dose. Test dose - 100 rad.

8 - OTHER POST-ANNEALING EFFECTS

Several mini-dosimeters pre-annealed at 400° C for 1 hour followed by 175° C annealing for 10 hours were used for these experiments. In the first series of experiments 15 mini-dosimeters were divided into 3 groups. The first one was irradiated to 10,000 rad, the second one to 50,000 rad, and the last one to 100,000 rad. They were then read and read again to verify that almost zero residual thermoluminescence remained. After this procedure they were post-annealed at 175° C for 1 hour and then at 80° C for one hour. In a second series of measurements, 20 mini-dosimeters were irradiated to 10,000 rad read and checked as above, and post-annealed at 80° C, 5 of them for half an hour, 5 for one hour, 5 for 2 hours, and the last 5 for 3 hours. All these dosimeters were then irradiated to 100 rad test dose and read out.



TIME

FIGURE 17 - Glow curves of mini-dosimeters irradiated to 10,000 rad and post-annealed at 80°C, (a) 1/2 hour, (b) 1 hour, (c) 2 hours, (d) 3 hours. Test dose - 100 rad.

Somewhat unexpected results were obtained in these measurements and they are presented in Figs. 16 and 17 The principal result is that the TL response is much larger than expected.

9 - SUMMARY OF RESULTS

Let us restate the initial treatment which the ⁷Lif mini dosimeters received. They were first annealed at 400° C for 1 hour and then annealed at 175° C for more than 7 hours. With such a heat treatment peaks 4 and 5 are removed.

Let us now present a summary of results.

(a) Peaks 2 and 3 present a supralinearity curve as well as an S/S_o curve very similar to those of peak 5. Peak 3 is slightly more supralinear than peak 2.

(b) For a previous dose of 10^5 rad and post-annealing at 280°C for one hour, Cameron et al.² found a maximum S/S_o value of ~6.5 for the main dosimetry peak. We found the maximum S/S_o = 5.4 for peak 3 and 2.3 for peak 2, with a post-annealing at 280°C for 1/2 hour. They reduce to 3.3 and 1.8, respectively, for one hour of post-annealing. With 1 hour of 175° C post-annealing, however, S/S_o = 6 for peak 2 and 4.5 for peak 3, for a previous dose of 70,000 rad.

(c) The ratio of the height of peak 2 to that of peak 3 is less than 1 for 280°C post-annealing, but, it is larger than 1 for 175°C post-annealing.

(d) For a given sensitizing dose, say 10,000 rad, S varies with both temperature and duration of post-annealing. Large fluctuations of peak 2 in these experiments preclude any definite conclusion as to the dependence of S on time.

(e) The most interesting result is that, with 280°C post annealing peak 5 not only is restored, it remembers whether the crystal was subjected to a previous high dose or not. The maximum height of peak 5 is attained within about one hour of post annealing, then it decreases for longer times. Such a restoration is not observed for 175°C heat treatment.

(f) The shape and the size of glow curves depend on the last pre-annealing temperature. They are independent of whether or not the dosimeters were irradiated prior to the pre-annealing. The irradiating dose should not, however, exceed the value which gives rise to supralinearity.

(g) The dissociation of one kind of trap and the formation of another one under a given pre-annealing treatment is a reversible process, except for some special annealings like the one at 400°C for times longer than 48 hours.

(h) For 10 hours pre-annealing time, the heights of peaks 2 and 3 as a function of pre-annealing temperature, present maxima and minima in the range between 60° C and 400° C. The height of peak 5 decreases rapidly from 60° C to about 160° C, then vanishes to reappear at about 240° C, increasing very rapidly as the temperature increases toward 280° C. From 280° C on it increases slowly (Fig. 14).

(i) Figure 15 shows glow curves for several pre-annealing temperatures between 100° C and 165° C. It shows a gradual shift in the position of peak 5 as the temperature increases, besides the fact that its height decreases as we noted already.

(j) With respect to the experiments described in Section8, we can list following results:

(j₁) In every case a very large reading resulted for peaks 3 and 5; in some cases it is almost 100 times larger than the expected value.

 (j_2) There is quite a change in the glow curve of the sample post-annealed at 175°C, followed by 80°C, compared

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to that of a mini-dosimeter post-annealed at 175°C only. In the former peak 5 (or 4) is restored and peak 3 is removed; furthermore, peaks at higher temperature (at least two of them) are revealed.

 (j_3) With 80^oC post-annealing peak 2 is removed, but peaks 3 and 5 (or 4) are observed,

10 - DISCUSSION

The fact that peaks 2 and 3 behave almost the same way as peak 5, as far as supralinearity is concerned, indicates that the corresponding trapping centers do not compete significantly with each other. The mechanism of electron (or hole) capture by these centers must be equal.

Material pre-annealed at 175°C for more than 7 hours does not present peaks 4 and 5 no matter how intensely it is irradiated, demonstrating that the radiation does not create trapping centers corresponding to peaks 4 and 5. This conclusion applies equally to peaks 2 and 3 after pre-annealing at 80° C for 24 hours. On the other hand, a post annealing at 280°C restore peak 5, which means that this heat treatment recreates the traps responsible for peak 5, which were destroyed by 175°C annealing. This result, summarized in (e), indicates that the radiation causes an internal change of the crystal so that with 280°C post annealing, the restoration of the peak 5 takes place and so that the height of peak 5 depends on the previous dose. Apparently some part of this internal change in the crystal depends on the previous dose. On the other hand, 175°C post-annealing hinders the restoration of peak 5 probably for the same reason that 175°C pre-annealing removes them.

Several people³ have pointed out the role played By Mg, a key impurity in dosimetry LiF. The divalent Mg²⁺ impurity enters the crystal substitutionally, creating cation vacancy to keep charge neutrality. This vacancy behaves as a negative center and forms with the impurity a dipole which can be a trapping center. It is evident that more complex aggregates can be formed, giving rise to other trapping centers. In view of the results found in the present work and in Ref. 1 we can assert that heat treatment can form or dissociate such aggregates, there being an optimum temperature for which the dissociation of a given kind of trapping centers takes place. The diffusibility of defects in the crystal must be related to this process.

The nature of the peak labled number 6 is not yet clear. The result shown in Fig. 11 of Ref. 1 seems to indicate that it is a peak distinct from peak 5. On the other hand according to our Fig. 15 it may simply be peak 5 displaced toward high temperature as the annealing temperature is increased. Recently a model was proposed at this laboratory, which assumes that the TL traps corresponding to a given peak, are distributed continuously in energy around a value E_0 , for example in Gaussian form. Such a model predicts a displacement of a peak, for instance, as a function of the post-annealing time. The application of this model to try to explain the results of Fig. 11, of Ref. 1 is now in progress.

We have not found any reasonable explanation for the results summarized in (j).

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sions concerning the present paper. He is also indebted to Dr. Michael R. Mayhugh for reading the manuscript.

RESUMO

As propriedades dos picos de emissão 2 e 3 de temperatura baixa, no LiF:Mg, foram in vestigadas, depois de remover os picos principais 4 e 5, através de recozimento a 400°C por uma hora seguido de 175°C por 9 a 17 boras. Foram usados os mini-dosímetros da EG & G do ti po 7LiF. Uma nítida supralinearidade foi observada para os dois picos começando em doses ar tre 500 e 1.000 rad de raios gama de Cs-137, com o pico apresentando uma supralinearidade - ligeiramente mais pronunciada. Valôres de S/S₀, onde S é o valor da TL (termoluminescência) sensibilizada e, de S₀, o valor não sensibilizado para uma dada exposição, foram também, me ados, depois de um pos-recozimento a 280°C por meia e uma hora, ou a 175°C por uma hora, - após uma irradiação sensibilizante, em cada caso. S/S₀ e S dependem tanto da temperatura co mo da duração do pos-recozimento. O pico principal número 5 é restaurado com uma irradiação sensibilizante e recozimento de 280°C, quando êle havia sido removido por pré-recozimento a 175°C.

As propriedades de termoluminêscência dos mini-dosímetros de ⁷LiF, que foram recozidos alternadamente a 400°C por uma hora, 175°C por 10 horas, 280°C por 10 horas, às vêzes irradiados a 100 rad e lidos entre aquêles recozimentos, foram, também, investigados. A for ma e a altura dos picos de emissão dependem da temperatura do último recozimento. A dependência da resposta TL com a temperatura para um tempo fixo de recozimento de 10 horas foi também examinada. Mini-dosímetros irradiados a alta dose, lidos e então recozidos a 80°C por meia hora a 3 horas apresentam um aumento grande na sua sensibilidade. Um resultado semelham te foi obtido com pós-recozimento a 175°C por uma hora, seguido de 80°C por uma hora, ao im vés de 80°C somente.

RÉSUMÉ

Les proprietés des pics 2 et 3 d'émission thermoluminescente à basse température de Lif:Mg furent examineés, aprés avoir éliminé les pics principaux, 4 et 5, en utilisant la technique de recuit à 400°C pendant une heure, suivi de 175°C pendant 9 a 17 heures. Les mi ni-dosimètres EG & G de 'LiF furent employés. Une nette supralinéarité fut observeé pour les deux pics, pour les rayonnements gamma de Cs-137 supérieur a 500 rad. On a trouvé une supra linéarité plus prononceé pour le pic 3 que pour le 2. Les valeurs de S/So, où S est le valeur de thermoluminescence (TL) de l'échantillon sensibilisé et S₀, celui de l'échantillon non sensibilisé, furent mesurés uprés un post-recuit à 280°C pendant demi-heure et une heure, ou à 175°C pendant une heure. Ici post-recuit signifie un recuit aprés irradiation de sens<u>i</u> bilisation. S/S₀ et S dépendent de la témperature et de la duréé de post-recuit.Le pic pri<u>n</u> cipal numéro 5, d'abord éliminé par le recuit à 280°C. La hauteur de c. pic récupéré par une irradiation très intense suivie de recuit à 280°C. La hauteur de c. pic récupéré est une fonction de la dose antérieure de sensibilisation.

Nous avons étudié les propriétés thermoluminescentes des mini-dosimètres de ⁷LiF, qui ont subi une succession de recuit à 4000C pendant une heure, 175° C pendant 10 heures, 280°C pendant 10 heures et quelques fois irradiés avec 100 rad et mésurés entre deux recuits con sécutifs. La forme et la hauteur des pics d'émission dont dépendants de la dernière tempéra ture de recuit. Il fut aussi étudié la dépendance de la TL avec la température de recuit – avant l'irradiation pendant un temps fixe de 10 heures. Les mini-dosimètres irradiés avec une dose éléveé, puis recuits à 80°C de une demi-heure a 3 heures, présentent une augmentation considerable de leurs sensibilité. Un pareil résultat fut obtenu quand le recuit est fait à 175°C pendant une heure, suivi de 80°C pendant une heure, au lieu d'un recuit simple à 80°C.

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