SUITABILITY OF PURE NANO CRYSTALLINE LiF AS A TLD DOSIMETER FOR HIGH DOSE GAMMA RADIATION

by

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LiF is an alkali halide that is commonly used in radiation dosimetry utilizing its well-known thermoluminescence property. Pure LiF has very limited use in radiation dosimetry since the density and types of the internal traps are limited. For that reason, LiF is usually doped with different elements such as Mg and Ti in (TLD-100) to enhance its thermoluminescence properties and to be suitable for dosimetry applications. In this work we used ball milling as an alternative to dopants (impurities) to induce structure defects (*e. g.* dislocation) that will play the major role in thermoluminescence process similar to defects caused by dopants. The dislocation density of 1 h ball milled pristine LiF was evaluated at the MCX beamline of the Italian Synchrotron ELETTRA. A ball milled LiF was then compressed in the form of chips, then annealed for 1 h at 600 °C to get rid of low temperature dislocations. The annealed samples showed linear response in the range 50-300 Gy. Fading investigation showed that the integral thermoluminescence intensity almost stabilizes after 12 days from the first irradiation. Results indicate that ball milling is a new promising technique to produce thermoluminescence dosimeters without using any kind of dopants.

Key words: synchrotron radiation, thermoluminescence, LiF, glow curve, ball milling, dislocation, dose response, fading

INTRODUCTION

In nuclear and radiation facilities high levels of gamma radiation are predicted and sometimes are unavoidable, thus the functionality of electric components exposed to such high levels of radiation could be altered significantly [1, 2] and this in turn may disturb the operation of the facility due to equipment malfunction and could lead to radiological accidents. The problem stands for electronic survey meter too such as fixed area monitors in nuclear and radiation facilities, thus inaccurate dose levels readings are probable too. For that reason a suitable and reproducible high dose gamma radiation dosimeter is mandatory, such that it keeps its functionality at very high levels of gamma radiation without deterioration. The best candidates for high dose measurements are passive dosimeters based on thermoluminescence (TL) phenomenon.

TL is the emission of light from an insulator or semiconductor when it is heated. The crystalline forms of some inorganic phosphors store some of the energy imparted to them from ionizing radiation and release that energy as light when the temperature of the crystal is raised. The TL phenomenon is a complex process and many kinetic models have been proposed in literature discussing various TL mechanisms [3, 4]. A simplified explanation could be introduced on the basis of one trap one recombination center (OTOR) model shown in fig. 1.

In OTOR model, the TL material has two kinds of metastable states in the wide forbidden gap between the valence and the conduction bands. These metastable states are introduced either by adding some chemical impurities or by introducing structural defects in the lattice. One of such states exists near the conduction band and acts as a trap for electrons while the other exists near the valence band and acts as a trap for holes. When the TL material is excited by radiation, pairs of electron / hole are generated and are ultimately captured in their respec-

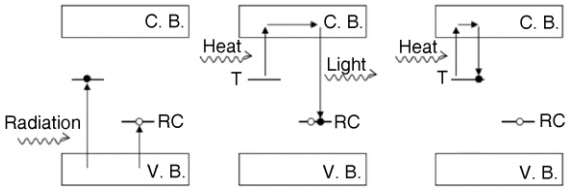

Figure 1. OTOR model (C. B. – conduction band; RC – recombination center; V. B. – valence band)

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tive traps (fig. 1. left).When the TL material is heated up to appropriate temperatures, the electrons are thermally released to the conduction band and such electrons have two possible pathways. The first one is to recombine directly with the trapped holes yielding emitted light (fig. 1. middle); for that reason the metastable state that captures the holes is called recombination center (RC) and the metastable state that captures electrons is called a trap (T). The other pathway for the released electrons is to be re-trapped again by the electron traps (fig. 1. right) before they thermally released again to recombine with respective holes at RC; the process in this case could be simply defined as indirect recombination. The energy required to release the electron from its trap to the conduction band called activation energy. The relationship between the temperature and the intensity of the released light is known as "glow curve" and could be composed of a single or multiple superimposed TL glow peaks and each glow peak corresponds to specific trap *i. e*., specific activation energy. More details will be presented in the next section.

In early 1950s, the TL phosphors used in present day dosimetry were discovered. These included LiF which is the most commonly used TL material [5]. LiF-based TL materials are widely used as personal dosimetric materials because of their low energy dependence, high sensitivity, stability and tissue equivalency. The use of pure LiF is limited in the dosimetry field, so the TL dosimetric (TLD) property is enhanced by adding some impurities to the pure LiF crystal, to be used as a TL dosimeter. For example, LiF doped with Mg, Ti elements (LiF: Mg, Ti) is the most known and efficient TLD materials based on LiF, which is available commercially in the market under brand name TLD-100 [6]. Such material has been developed and studied extensively and it is most widely used in personal dosimetry. Another example of TL phosphor material based on LiF, which uses another kind of dopants, is LiF: Mg, Cu, P. Such material shows very favourable dosimetric characteristics with great potential applications for low level dose measurements in the field of diagnostic radiology. This TL phosphor, in powder form, was first introduced in 1978 by Nakajima *et al*. [7]. Several authors studied and modified its characteristics [8-10]. Heating and cooling rate, readout and annealing temperatures could greatly affect the dosimetric performance of LiF: Mg, Cu, P, residual dose in the phosphor after readout has also been a major problem for dosimetric work. Bacci *et al*. [11] tried to identify the optimal annealing cycle for LiF: Mg, Cu, P, and Pradhan [12] did detailed experiments on the effect of heating rate on the TL response.

The preparation of efficient LiF based TL material using impurities is complicated and the final sensitivities depends significantly on the dopant concentration and on the preparation method, which is not an easy task.

Since the dopants play the main role in TL enhancement by adding defects inside the crystal structure of LiF; thus, the aim of this study is to investigate an easy tool to introduce structural defects inside the LiF crystal without using any kind of dopants and at the same time enhance the TL property of LiF.

There are many methods to create structural defects in materials; however ball milling is a very powerful technique that can introduce structural defects in materials. Plastic deformation *(i. e*., dislocations) is the most common and probable defect type for ball milling as a result of the energetic impacts between powder, vial and balls. In the present work, we prepared LiF using intensive ball-milling method in order to introduce dislocations and investigate its effect on TL properties of LiF material. LiF was selected to perform this analysis because of its well-known TL properties as well as for its quite simple crystal structure that confines the structural defects in only one probable form of defects *i. e*., dislocations.

MATERIALS AND METHODS

Sample preparation

2 g of commercially available LiF (99.99 % from Sigma Aldrich) was ground in a planetary ball milling (Fritsch Pulverisette 6) machine operating at high rotational speed (700 rpm). The pristine (as received) LiF powder was ball milled for one hour in 80 ml agates container. Twenty balls, made of agate, with 1 cm diameter were used. The milled LiF powder was then compressed in the form of discs with diameter of 3 mm and each disk weighted 5 mg approximately. About 500 disks were prepared and distributed on a glass substrate. The disks were subjected to temperature rate of 10 °C min^{-1} and heated up to 600 °C, then annealed at this temperature for 1 hour. The samples were then taken and quenched at the room temperature. About 340 samples were collected in good physical conditions.

Fundamentally, measurement accuracy of any physical quantity depends on the evaluation of measurement uncertainty [13, 14] and its sources should be eliminated or mitigated. In this study, uncertainty of gamma radiation measurement is highly expected from different response of the collected 340 samples due to many factors such as: (a) incomplete sample homogenization during ball milling, (b) different density of defects contained in each of the samples due to ball milling, (c) variation in weights of the collected samples, and finally, (d) different particle sizes contained in each sample. Accordingly, the collected 340 disks should be evaluated and segregated according to their response to the gamma radiation in order to pick up the samples of similar response in terms of integrated TL intensity and hence to reduces the measurement uncertainty.

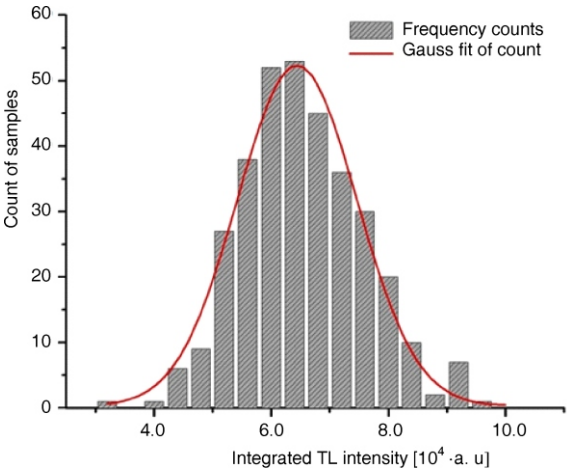

Figure 2. Number of LIF disks as a function of integrated TL intensity

For that purpose, the collected 340 disks were then irradiated with γ-ray dose of 200 Gy using ^{60}Co γ-rays cell model γ-cell-220 at irradiation rate of 2 kGyh^{-1}. The LiF samples were placed inside the γ-cell chamber in such a way that all samples were exposed to the same dose. The TL glow curves of the samples were measured by using a Harshow model 4500 TLD reader, with different linear heating rates, in temperature range from 50 °C to 400 °C. Figure 2 represents the number. of samples as a function of the integrated TL intensity. As shown in the figure, the samples exhibit different response towards the irradiation dose. The overall behavior follows normal Gaussian distribution. Only 150 Samples were taken (3 columns at the center of the curve) for further investigations. The average TL reading of those 150 samples were taken and then a correction coefficient was evaluated for each disk.

SAMPLES CHARACTERIZATION

X-ray powder diffraction (XRPD)

Microstructural characterization of the LiF samples was carried out at the (Microstructural characterization X-ray beamline) MCX beam line [15] of the Italian Synchrotron ELETTRA (Trieste). The measurements were carried out at the photon energy (12 keV) using Debye-Scherrer geometry. This set-up was used to perform fine microstructure line profile analysis at room temperature. A standard sample of NIST SRM 640 a silicon in a borosilicate capillary (0.3 mm in diameter) was used to define the instrumental resolution to be considered in the analysis. Then, each sample was prepared in the borosilicate capillary (0.3 mm in diameter). Diffraction data were collected in the range 15-75° 2Θ in steps of 0.01°. The refined wavelength obtained from the Si standard pattern measured by the MCX diffractometer was 1.03231 Å (1 Å = 10^{-10} m)

The microstructure was analyzed using the whole powder pattern modelling (WPPM) approach [16, 17] through the PM2K program [18]. In the modelling, the instrumental profile parameters were fixed and then the line profiles were modelled by considering the crystal size and the lattice strain in terms of dislocations, where both aforementioned parameters are the main structural sources of broadening. The crystal size was modelled as spherical crystals of lognormal distribution with two refinable parameters, namely mean and variance σ. Equation (1) shows the dislocation average contrast factors A and B for cubic crystals

$$C_{\text{average}} \quad A \quad B\frac{h^2k^2 \quad k^2l^2 \quad h^2l^2}{(h^2 \quad k^2 \quad l^2)^2} \qquad (1)$$

This equation takes into account the anisotropic broadening caused by dislocations as a function of *hkl* planes. The value of the contrast factor A was calculated analytically ($A_{\text{edge}} = 0.195$, $A_{\text{screw}} = 0.162$, $A_{\text{average}} = 0.179$) as proposed by Ungar *et al*. (1999) and then the parameter B, dislocation density ρ and the average cut-off radius R_{eff} (R_{eff} is the effective cut-off radius of the dislocation) were left as free refinable parameters in the strain model. Beside the crystal size and the lattice strain models, a Chebychev polynomial function with refined coefficients was used to fit the background of the diffraction patterns.

XRPD analysis

For pulverized LiF, the one-phase WPPM model was sufficient and stable to fit the collected diffraction patterns, which relates to the samples' homogeneity. Starting from the pristine LiF powder, large crystallite sizes of about 500 nm coupled with an absence of dislocations were observed. After one hour of milling, a strong decrease in the crystal size down to 36 (8) nm was observed, and the dislocation density was found to be about 4.4 (2) $\times 10^{15}$ m^{-2}.

According to WPPM theory [16, 17], the Wilkens factor [19] M [M = Re ($\rho^{1/2}$)] refers to the interactions among dislocations, where smaller M means a larger interaction is likely to take place at the crystal boundaries, and vice versa. The M values after 1h of milling were slightly above 1. Figure 3 shows the WPPM fitting of the annealed LiF sample diffraction pattern as a witness of fitting quality.

RESULTS AND DISCUSSION

Gamma-dose response measurement

The usual method for studying the response of TL as a function of irradiation dose is to irradiate the TL materials at constant dose rate, constant temperature, and then to read out the TL materials at a reason-

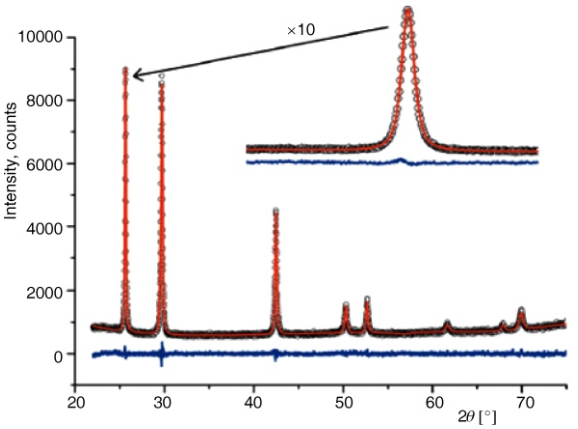

Figure 3. WPPM fitting for the LiF annealed sample; the inset shows the fitting quality of the (111) crystalline plane zoomed in ten times; the open circles refer to the raw data; the continuous line refers to the fitting model and the line below refers to the residual

able heating rate [20]. Figure 4 represents the integrated TL intensity recorded at 5 °C/sec as a function of irradiation dose. As depicted, the prepared TL samples have linear TL response as the irradiation dose increases up to 300 Gy. Figure 5 represents the glow curves over the same dose range. As shown, all the

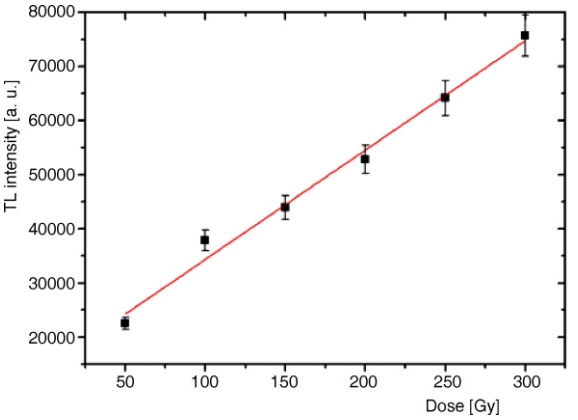

Figure 4. TL intensity as a function of γ-absorbed dose (^{60}Co-source) for LiF, vertical bars represented the standard deviation at each dose

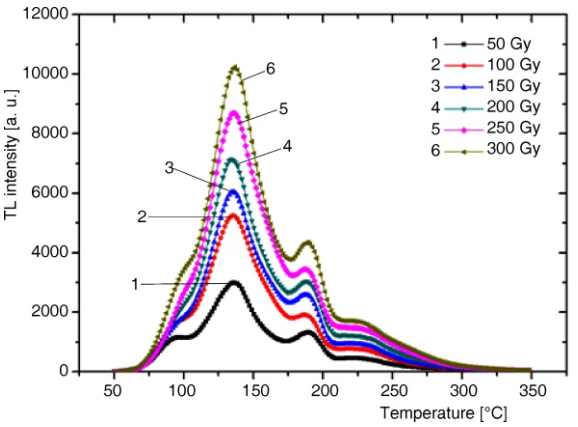

Figure 5. Variations of the glow curves over the dose range from 50 to 300 Gy

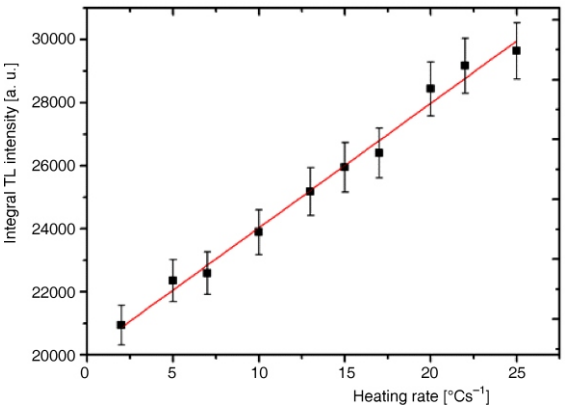

Figure 6. Integral TL intensity as a function of heating rate

glow curves have 4 prominent peaks at around 90 °C, 125 C°, 180 °C, and 225 °C, approximately and their amplitudes are increasing as the dose increases.

Effect of heating rates on TL intensity

Several groups of prepared chips have been measured at different heating rates, namely 2, 5, 7, 10, 13, 15, 17, 20, 22, 25 °Cs^{-1}. About 7-9 chips were measured for each heating rate and then the standard deviations were calculated, as well. The results show that there is an increase in the TL intensity with the increase if heating rate as shown in fig. 6. It has been found that the relation between heating rates and TL response is linear.

Fading during storage in the dark

In most TL materials the TL signal decreases as a function of storage time due to the thermal escape of trapped charge carriers and this characteristic is, therefore, usually referred to as 'thermally induced fading' or simply "fading". In this study, the anomalous-fading was investigated where the prepared LiF chips were irradiated, at a time, with γ-rays from a ^{60}Co source with a dose of 50 Gy. The irradiated chips are then stored in the dark at room temperature. Successive measurements of the TL intensity of the chips were monitored over a period of 2 weeks.

Figure 7 shows the integrated TL intensity recorded at 5 °Cs^{-1} as a function of elapsed time from the beginning of the irradiation. As shown, the integral TL intensity is reduced as the time proceeds and the reduction of the TL intensity as a function of time follows the mathematical formula that could be easily obtained by curve fitting

$$I(t) \quad A_1 e^{\frac{t}{B_1}} \quad A_2 e^{\frac{t}{B_2}} \quad I_{asym} \qquad (2)$$

where A_1, A_2, B_1, and B_2 are fitting parameters, $I(t)$ represents TL intensity at the elapsed time (t), and I_{asym}

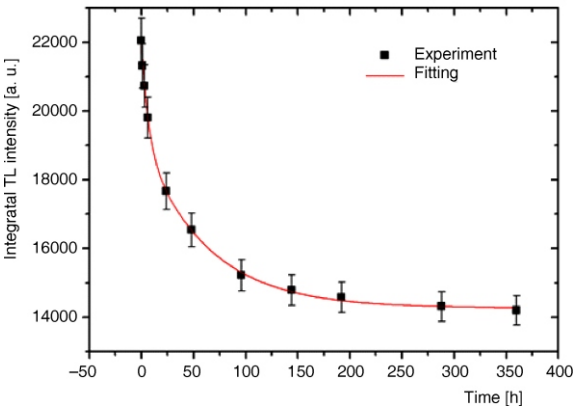

Figure 7. TL integral intensity of 50 Gy irradiated chips as a function of storage time in the dark

Table 1. Fitting parameters of fading curve presented in fig. 7

Fitting parameter	Value	Standard error
I_{asym}	14263.540	72.341
A_1	4913.228	274.741
B_1	61.954	5.867
A_2	2772.391	299.461
B_2	6.320	1.002

represents the asymptotic TL integral intensity after fading process completed approximately after 2 weeks; all the parameters are presented in tab. 1. Figure 8 shows the actual glow curves reordered at heating rate of 5 °Cs^{-1} for irradiated LiF at various selected times from the first irradiation.

From fig. 8 it is very clear that the TL intensity decreases by time and has the main peak keeps the position as the time elapsed. Also it is noticed that the shoulder glow peak at 90 °C disappeared after 6 hour whereas the glow peak at 125 °C decreases with time; the glow peak at 180 °C increases with time and the glow peak at 225 °C remains almost unchanged. This means that, during fading, some of charge carriers released from the low temperature traps are re-trapped again at high temperature traps. This suggests that the TL process during fading is most

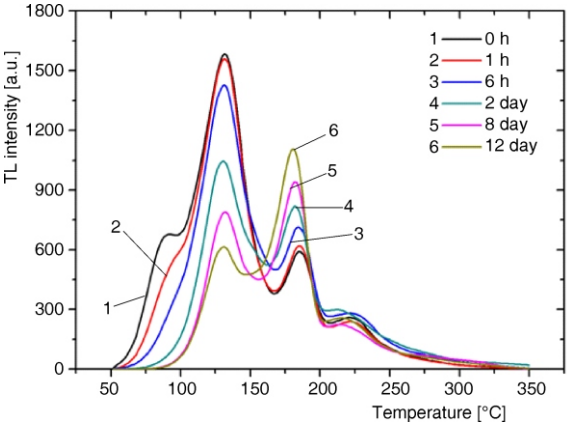

Figure 8. TL intensity of 50 Gy irradiated chips as a function of selected storage time in the dark

probably controlled by 2nd order kinetic based on OTOR model in which the electrons released from the traps are re-trapped in the same, or other kind of traps, before final recombination with the holes. This issue will be further studied in details in near future to determine the kinetic parameters (*e. g.* activation energy and frequency factor) of individual glow peaks

SUMMARY AND CONCLUSION

A 500 nm LiF from Sigma-Aldrich has been ball milled for 1h and the characterized using synchrotron XRD. The diffractogram has been analyzed by using PM2K code and the analysis shows that the crystal size reduced to 37(8) nm with dislocation density about 4.4 (2) × 10^{15} m^{-2}. The TL response of the annealed samples was investigated to study the effect of the dislocation on TL properties. When samples were irradiated at 50 Gy, the observed glow curve has 4 prominent peaks at fixed positions at 90 °C, 125 °C, 180 °C, and 225 °C approximately. Several chips have been measured at different heating rates, namely 2, 5, 7, 10, 13, 15, 17, 20, 22, 25 °Cs^{-1}. It has been found that the relation between heating rates and TL response is linear. At high heating rate, there is a lack between the real temperature of heating element and the temperature of the sample. This in turn leads to appearance of the glow peak at higher temperature than it should be. Worthy to mention is that by using high heating rate the glow peaks are overlapped. For those reasons, the slowest heating rate is utilized for investigating the samples rather than the higher heating rates, although higher heating rates give higher TL intensity. The optimum glow curve intensity is observed at a heating rate of 5 °Cs^{-1}.

The dose-response is found to be linear between 50 Gy and 300 Gy. The amplitudes of the aforementioned 4 prominent peaks increase as the dose increases; this may be explained by the expected change in defect concentration of LiF induced through irradiation.

For the fading studies, one γ-dose of 50 Gy was examined. Successive measurements of the TL intensity from discs were monitored over a period of 12 days, during which the discs were stored in the dark, at room temperature. This study showed that the lower temperature peaks at 90 °C are faded almost completely within 6 hours, the glow peak at 125 °C decreases with time, the glow peak at 180 °C increases with time and the glow peak at 225 °C almost remains constant. This suggests that some of the electrons released during fading from 2nd glow peak are re-trapped by traps responsible for the 3rd peak. The overall integral TL intensity almost stabilizes after 12 days from the first irradiation, showing about 33 % decrease without any further change in glow curve shape.

The results achieved in this study show that the micro structure changes induced by ball milling play important role in enhancing the TL response of LiF. Our results agreed with previous study [21] in which the authors studied the effect of dislocations induced in LiF at different intervals of ball milling and they found that the TL response is significantly enhanced as the ball milling time increased up to certain limit (20 h), then the TL response decreased after this limit. They found that the dislocations play the major rule in affecting the TL response. Such study in parallel to our results, presented in this paper, open up new horizons in producing newly developed TL material without adding any kind of dopants. Although the prepared samples are not optimum from the point of view of TL dosimetry, however, the study confirms the proof of principle of using ball milling as an alternative for dopants to enhance the TL properties, not only in LiF but also in other TL materials. So, efficient and easily prepared free of dopants TL dosimeter could be developed in near future.

AUTHORS' CONTRIBUTIONS

H. A. Amer, M. M. Elashmawy, and H. A. Alazab prepared the samples and carried out TL measurements along with glow curve data analysis, M. M. Elashmawy and H. A. Alazab carried out XRPD measurements at Italian synchrotron ELETTRA. M. M. Elashmawy analyzed the XRPD data using PM2K code. M. R. Ezz El-din supervised the work and wrote the manuscript with M. M. Elashmawy and H. A. Alazab.

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ПОГОДНОСТ ПРИМЕНЕ ЧИСТОГ НАНОКРИСТАЛИНА LiF КАО ТЕРМОЛУМИНИСЦЕНТНОГ ДОЗИМЕТРА ЗА ВИСОКЕ ДОЗЕ ГАМА ЗРАЧЕЊА

LiF је алкал халид који се уобичајено употребљава у дозиметрији зрачења коришћењем његовог добро познатог термолуминесцентног својства. Чист LiF нема велику примену у дозиметрији зрачења јер су густина и врсте јама ограничене. Због овога се у LiF обично уносе различити елементи као што су Mg и Ti (TLD-100) како би му се побољшала термолуминисцентна својства чинећи га погодним за дозиметријске примене. У овом раду користили смо лоптасту дробилицу као замену за нечистоће у циљу индуковања структурних дефеката (дислокација) који ће играти главну улогу у термолуминисцентном процесу, сличну дефектима које производе нечистоће. Густина дислокација после једног сата дробљена чистог LiF оцењена је у MCX снопу италијанског синхротрона ELETTRA. Издробљени LiF је потом компресован у виду жетона и одгреван на 600 °C током једног сата како би се уклониле нискотемпературне дислокације. Одгрејани узорци испољили су линеарни одзив у опсегу 50-300 Gy. Испитивање слабљења показало је да се интегрални термолуминисцентни интензитет скоро стабилише после 12 дана од првог озрачивања. Резултати показују да је лоптасто дробљење нова и обећавајућа техника за производњу термолуминисцентних дозиметара без уноса нечистоћа ма које врсте.

Кључне речи: синхротронско зрачење, термолуминисценција, LiF, крива исијавања, лоптасто дробљење, дислокација, дозни одзив, слабљење