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Abstract: LiF thermoluminescent material doped with Mg, Cu and Si recently developed by the Korea Atomic Energy Research Institute (KAERI) has shown very good dosimetric properties. Since the thermoluminescence in LiF was found to be dependent on the proper combination of dopants, the investigation of the concentration and type of dopants is very important in developing and characterisation of new TL materials. The aim of this work was to determine the influence of type and concentration of activators on the glow curve structure, sensitivity, reproducibility and on the photon energy response of LiF:Mg,Cu,Si detectors. The energy response was studied in air and on the ISO water phantom in the range of mean photon energies between 33 keV and 164 keV. The morphology and local chemical composition of LiF:Mg,Cu,Si detectors were examined using high resolution scanning electron microscopy (FE-SEM). The results show that type and concentration of activators influence the glow curve and sensitivity. Different dopant concentrations did not show influence on the photon energy response. The sensitivity of LiF:Mg,Cu,Si detector with dopant concentration of Mg=0.35 mol%, Cu=0.025 mol% and Si=0.9 mol% was very high (up to 65 times higher than that of TLD-100). The photon energy response of LiF:Mg,Cu,Si detectors containing all three dopants in various concentrations is in accordance with the IAEA recommendations for individual monitoring.

Cover Letter

Dear Editor-in-Chief

I submit manuscript under the title:

Influence of dopants on the glow curve structure and energy dependence of LiF:Mg,Cu,Si detectors

Ž. Knežević[a], M. Ranogajec-Komor[a], S. Miljanić[a], J.I. Lee[b], J.L. Kim[b] and S. Musić[a]

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Influence of dopants on the glow curve structure and energy dependence of LiF:Mg,Cu,Si detectors

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Abstract

LiF thermoluminescent material doped with Mg, Cu and Si recently developed by the Korea Atomic Energy Research Institute (KAERI) has shown very good dosimetric properties. Since the thermoluminescence in LiF was found to be dependent on the proper combination of dopants, the investigation of the concentration and type of dopants is very important in developing and characterisation of new TL materials. The aim of this work was to determine the influence of type and concentration of activators on the glow curve structure, sensitivity, reproducibility and on the photon energy response of LiF:Mg,Cu,Si detectors. The energy response was studied in air and on the ISO water phantom in the range of mean photon energies between 33 keV and 164 keV. The morphology and local chemical composition of LiF:Mg,Cu,Si detectors were examined using high resolution scanning electron microscopy (FE-SEM). The results show that type and concentration of activators influence the glow curve and sensitivity. Different dopant concentrations did not show influence on the photon energy response. The sensitivity of LiF:Mg,Cu,Si detector with dopant concentration of Mg=0.35 mol%, Cu=0.025 mol% and Si=0.9 mol% was very high (up to 65 times higher than that of TLD-100). The photon energy response of LiF:Mg,Cu,Si detectors containing all three dopants in various concentrations is in accordance with the IAEA recommendations for individual monitoring.

1. Introduction

LiF based materials doped with Mg, Cu and Si were developed long time ago, but they were unnoticed because of relatively low sensitivity compared to TLD-100 and their instability to thermal treatments. (Nakajama et al., 1978). The improved and optimised thermoluminescent material of LiF doped with Mg, Cu and Si (LiF:Mg,Cu,Si) was recently developed at the Korea Atomic Energy Research Institute (KAERI) (Lee et. al., 2007). The

new material showed high sensitivity, low residual signal and good stability to thermal treatments.

For use of a TL material in radiation dosimetry it is very important that the material has a suitable glow curve structure, high sensitivity, tissue equivalency, flat energy response, stability to thermal treatments, good reproducibility etc. Most of these properties are dependent on impurities doped in the host material. Therefore the investigation of the concentration and type of dopants is very important study in the developing and characterisation of new TL materials.

The aim of this work was to determine the influence of type and concentration of dopants in LiF:Mg,Cu,Si on the glow curves, sensitivity, reproducibility and photon energy response in air and on water phantom. Also the morphology and local chemical composition of LiF:Mg,Cu,Si detectors were examined using high resolution scanning electron microscopy (FE-SEM).

2. Materials and methods

2.1. TL detectors and instrumentation

Experiments were carried out using sintered pellets made of LiF:Mg,Cu,Si with different dopant concentrations prepared at KAERI. The concentration of various dopants varied over the following ranges: Mg (0-0.50 mol%), Cu (0-0.03 mol%) and Si (0-1.2 mol%). The optimum concentrations of dopants according to KAERI are as follows: Mg 0.45 mol%, Cu 0.025 mol% and Si 0.9 mol% (Lee et al., 2006). For comparison commercially available standard LiF:Mg,Ti (TLD-100) detectors made by Thermo Fisher Scientific (earlier Harshaw) were used.

The readout was carried out using modified TOLEDO 654 (Vinten) reader (Knežević et al., 2005). The reader connected with a PC contained software which enables detailed analysis and integration of the glow curves with variable integration limits. Before reading the dosimeters were externally annealed at 100 °C for 20 min. In the reader LiF:Mg,Cu,Si detectors were preheated at the temperature of 100 °C for 6 s and then heated with a constant heating rate of 10 °C per second to the temperature T_{max} (280 °C); after that the dosimeter was kept at T_{max} during the time left from the readout cycle (35s). The applied annealing conditions were 10 min at 260 °C in the oven, with rapid cooling on an Al plate.

2.2. *Irradiations*

For calibration, irradiations with ^{137}Cs gamma ray source at the Secondary Standard Dosimetry Laboratory (SSDL) (Vekić et al., 2006) in the Ruđer Bošković Institute were performed (dose was specified as kerma in air). The dose rate was about 57.5 mGy/h at a distance of 1 m. The group sensitivity of each dopant concentration was determined by irradiations with the same ^{137}Cs gamma ray source.

The energy dependence was determined by irradiations with narrow spectra X-ray beams generated by an ISOVOLT 420 X-Ray Unit (40-300 kV, 1-20 mA) at the SSDL. The specified mean energies were obtained by varying the operating potential and added filtration. The following mean energies were used: 33, 48, 65, 83, 118 and 164 keV. The irradiations were performed at a distance of 1 m from the tube. In one series the holders were placed free in air, in the second on the ISO water phantom (ISO 4037-3).

For irradiation the detectors were packed in polymethyl methacrylate (PMMA) holders containing recesses for detectors. The holders were packed in a dark polyethylene foil. The holders with 3 mm and 1.5 mm wall thickness were used for ^{137}Cs and X-ray irradiations, respectively. There were two irradiations with doses of 2 and 5 mGy for every mean energy.

2.3. *Morphology investigations*

The morphology of LiF:Mg,Cu,Si detectors was examined with field emission scanning electron microscopy (FE-SEM Jeol 7000F). The Energy Dispersive Spectrometry (EDS) analysis was performed in order to compare the chemical composition on the surface of detectors with the chemical concentrations specified by KAERI. The following samples with different dopant concentrations were examined: a) TL-1 (Mg= 0.10 mol%, Cu=0.025 mol%, Si=0.9 mol%), b) TL-2 (Mg=0.45 mol%, Cu=0.025 mol%, Si=0.9 mol%) and c) TL-3 (Mg= 0.25 mol%, Cu=0.025 mol%, Si=0.3 mol%).

3. Results and discussion

3.1. *The influence of various dopants concentrations on the glow curves and TL sensitivity*

In Figure 1 dependence of the *glow curves* on different Mg concentrations are presented. The Mg concentrations were 0.0, 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45, and 0.50 mol% while the concentrations of other two dopants were fixed (0.025 mol% Cu and 0.9 mol% Si). *TL sensitivity* expressed as the TL response per unit dose for different Mg concentrations increases with the concentration of Mg. The main dosimetry peak intensity

exhibits a sharp increase at 0.15 mol% Mg, and then rises slightly with increasing Mg concentration. The maximum of TL intensity is at 0.35 mol%. Relative TL sensitivity of this formulation (0.35 mol%. Mg, 0.025 mol% Cu and 0.9 mol% Si) is 65 times higher compared to TLD-100. The sensitivities of LiF:Mg,Cu,Si detectors with different concentrations of various dopants compared to TLD-100 are shown in Table 1.

Figure 2. shows the glow curves and variations of the main peak for different Cu concentrations with fixed Mg (0.45 mol%) and Si (0.9 mol%) concentrations. The Cu concentrations were 0.0, 0.01, 0.02, 0.025 and 0.03 mol%. The results show that Cu concentration did not influence on the main peak in the investigated range of concentrations but the presence of Cu is important for the high sensitivity of LiF:Mg,Cu,Si detectors in that range of concentrations (Table 1). Figure 3 shows the glow curves and variations of the main peak intensity for different Si concentrations with fixed Mg (0.45 mol%) and Cu (0.025 mol%) concentrations. The Si concentrations were 0.0, 0.1, 0.2, 0.3, 0.6, 0.9 and 1.2 mol%. In the investigated range of Si concentrations it is observed that from 0 mol% up to 0.3 mol% there was no essential rise of the main peak TL intensity. The concentration of 0.3 mol% Si is the threshold value because at that concentration relative sensitivity is very high (55 times higher than that of TLD-100) and the small change in concentrations from 0.2 mol% to 0.3 mol% leads to large variation in sensitivity (Table 1). Relative sensitivity of LiF:Mg,Cu,Si detectors with dosimetry system used in this work (reader characteristics, heating treatments etc.) is very high compared to TLD-100. For the optimum concentrations of dopants it is 62 which is even higher than previously published values (Kim et al., 2008 Lee at al., 2006) (Table 1.).

The *residual signal* defined as the percentage ratio of the second readout to the first readout with exactly the same reading programme for the optimum concentrations of dopants was estimated to be satisfactory (0.04 %). The main cause of residual signal in TL materials is the high temperature peak which usually appears after the main dosimetric peak. In this work the high temperature peak in the investigated range of dopant concentrations at the used maximum reading temperature of 280°C and heating rate of 10°C/s was not observed.

Reproducibility of all investigated detectors with various dopant concentrations (except zero concentrations) (expressed as ± 1 standard deviation of the mean values in %) through all measurements was in the range 0.54-5.34% (in one case was higher: 7 %). These values represent good reusability of the detectors. The reproducibility for the zero concentrations of any dopant was not acceptable (16-38%). The sensitivity was decreasing with the repeating measurements (Table 1). Because of their poor reusability –up to 70%

reduction of the readout values after reuse of 5 times they were not included in the energy dependence measurements.

3.2. The morphology of the detectors

The investigation of morphology of LiF:Mg,Cu,Si detectors is shown in Figure 4. Figure 4. shows the surfaces of samples TL-1, TL-2 and TL-3, examined with (FE-SEM). The surface of sample TL-1 (Fig. 4a) consists of big compact grains of about 50 to 100 μm in size with clearly visible boundaries between grains. There are no lot of empty spaces on the surface of this detector, which indicates dense structure characteristic for good ceramic material. Small particles of about 1 to 3 μm in size adhered on the surface of big grains are also visible. Sample TL-2 (Fig. 4b) consists of less compact grains with a lot of empty spaces and rather inhomogeneous surface. Also, a rough surface of grains implies the existence of porous structure in this sample. Sample TL-3 (Fig. 4c) consists of quite heterogeneous, porous surface with poorly visible grain's boundaries.

The Energy Dispersive Spectrometry (EDS) analysis was performed in order to compare chemical composition on the surface of detectors with chemical concentrations specified by KAERI. The results of analysis showed a satisfactory agreement in the case of LiF and Mg, but the proportion of dopants Cu and Si considerably differs from the specified values. The variations in local concentrations of the dopants are in agreement with the results of Lee et al. who found that the proportion of the dopants intended and finally present is different (Lee at al., 2008). The inhomogeneous surface and the surface segregation of dopants may influence the TL properties of detectors.

3.3 Influence of dopants on energy dependence in air and on the phantom

Measured energy responses of detectors with different dopant concentrations irradiated free in air are presented as a function of the mean photon energy in Figure 5. The energy dependence of detectors with zero concentrations of one dopant was not investigated because of their poor reproducibility. Measured values relative to air normalized to 662 keV photons (^{137}Cs) were compared with calculated values of mass-energy absorption coefficients for pure LiF and for LiF with different dopant concentrations and air (Figure 5). The values for LiF:Mg,Cu,Si have been calculated according to the mass energy-absorption coefficients taken from National Institute of Standards and Technology (NIST, 2008). The calculated values of the coefficients were the same for different types and dopant concentrations. Therefore in Figure 5. only the calculated values for the optimum dopant concentrations are

shown. The differences between measured values for different dopant concentrations at the same energy were 1.4-2.8 % which is within experimental errors. The measurement results show that concentrations of dopants have no influence on energy absorption characteristics of LiF:Mg,Cu,Si detectors. Theoretical curves according to the calculated values of mass-energy absorption coefficients showed that addition of dopants increases the mass-energy absorption coefficients of LiF:Mg,Cu,Si compared to pure LiF in the energy range up to 80 keV. Measured values for all dopants concentrations are 11-22 % and 16-26 % lower than the calculated values for pure LiF and for LiF with optimum dopants respectively (Knežević, 2007).

For irradiations on phantom the results are shown in Figure 6. Results for different type and concentrations of dopants are expressed as the mean values of dose measured on phantom relative to delivered doses specified as air kerma free in air. The measured values are compared to personal dose equivalent $H_p(10)/K_a$ values. The personal dose equivalent is the dose equivalent in soft tissue at depth d below a specified point on the body. For the calibration purposes the values are defined in the calibration slab phantom. The results show that the differences between measured values of investigated dopant concentrations at the same energy were 1.4-4.4 %. It is within the experimental error, and the concentration of dopants for the measurements on phantom as well did not influence the energy dependence. The measured values showed 8-55% lower energy responses in the investigated energy ranges compared to theoretical values of $H_p(10)/K_a$, except at the lowest energy (33 keV), where the maximum of absorbed dose is about 18 % higher then the theoretical value. It can be explained by the fact that the dosimeter was placed on the surface of the phantom while $H_p(10)$ is defined and calculated for the depth of 10 mm, and for these two cases the spectra of low energy photons are different (Miljanić et al., 2003).

Lower energy responses as compared to theoretical values of $H_p(10)$ have been explained as an ionisation density effect and is not according to effective atomic number. (Olko et al., 1993). However the results of energy dependence in terms of $H_p(10)$ for the investigated LiF:Mg,Cu,Si detectors are in agreement with the IAEA recommendations (IAEA 1999) for personal dosimetry which indicates that doses of the dosemeters worn on the surface of the body should not differ by more than -33 % or +50 % (at the 95% confidence level) from the dose equivalents that would be indicated by an ideal dosemeter worn at the same point at the same time (IAEA 1999).

4. Conclusions

The influence of type and concentration of dopants on the glow curve structure, sensitivity, reproducibility and photon energy response of LiF:Mg,Cu,Si detectors irradiated in air and on phantom was studied. The main peak intensity depends on the Mg concentration and exhibits a sharp increase at 0.15 mol%. The relative sensitivity for the optimum concentrations of dopants (0.45 mol%Mg, 0.025 mol%Cu and 0.9 mol%Si) as compared to TLD-100 is 62. The absence of even one of dopants caused very low sensitivity and poor reproducibility. Different dopant concentrations did not show any influence on the photon energy response in air and on the phantom. The energy response values in air for all investigated dopant concentrations are lower than the calculated values for pure LiF and for LiF with different dopants. The values measured on phantom compared to theoretical values of $H_p(10)/K_a$ are also lower in the investigated energy range except at the lowest energy (33 keV) where the maximum of absorbed dose is higher. The photon energy response of LiF:Mg,Cu,Si detector for all investigated dopant concentrations satisfies the IAEA recommendations for individual monitoring.

Acknowledgement

The author's wish to express their appreciation to Renata Ban and Branko Vekić for their help with the irradiations

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Figure 1

Figure 1

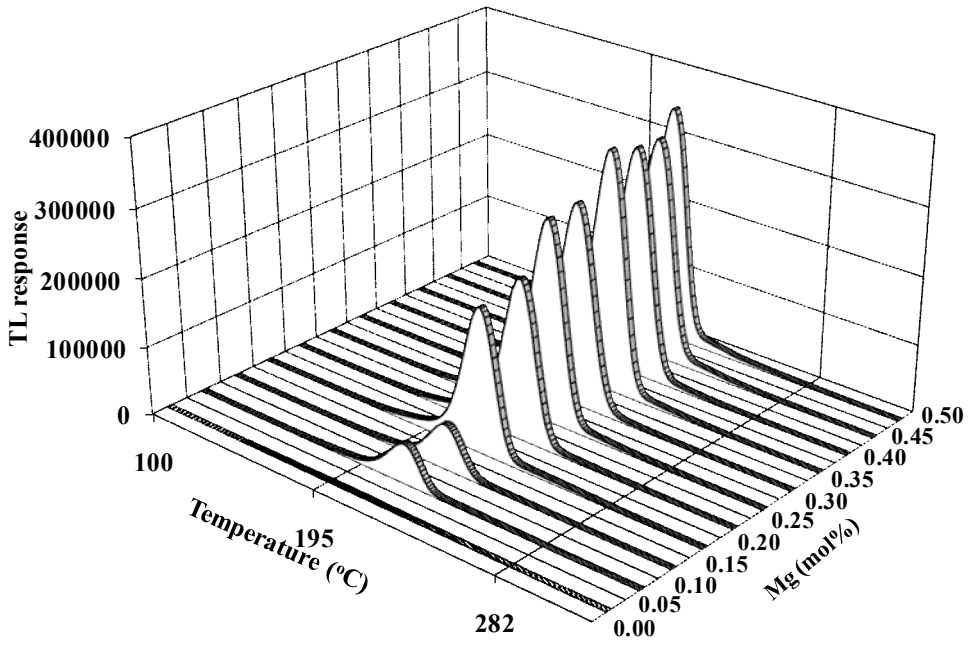


Figure 2

Figure 2

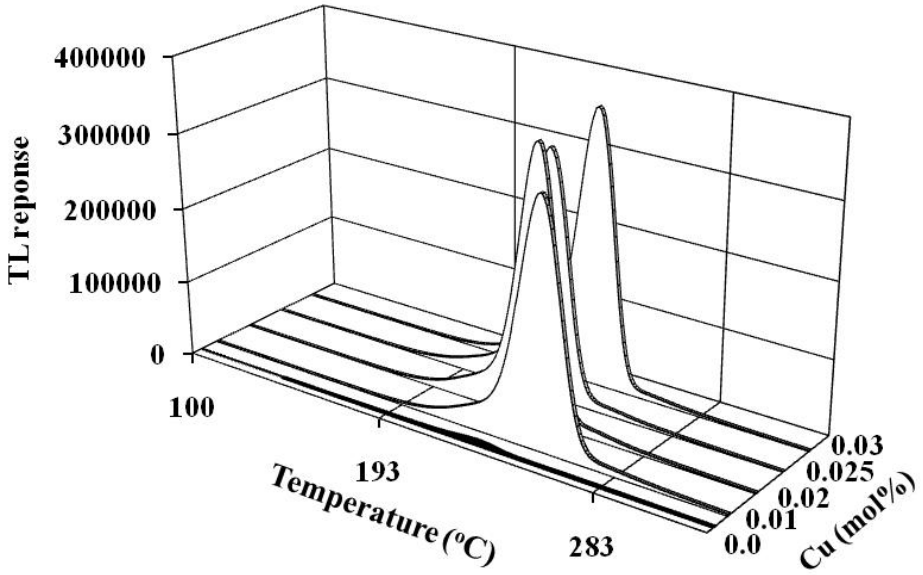


Figure 3 revised

Figure 3

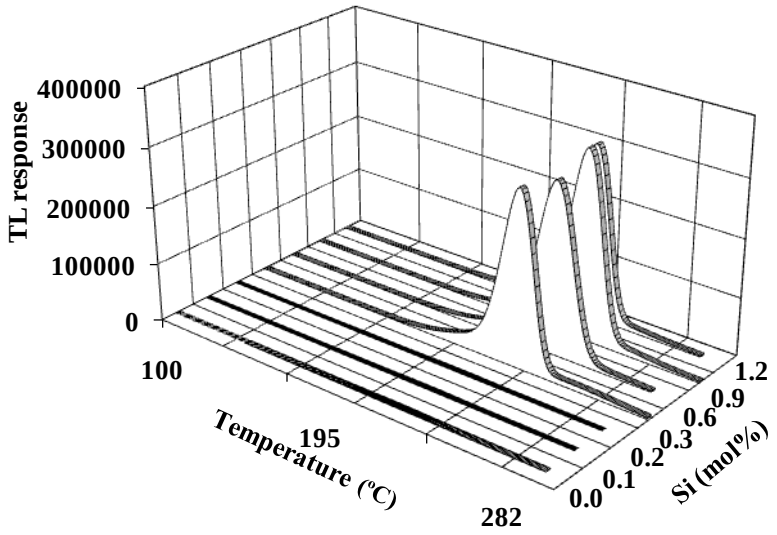
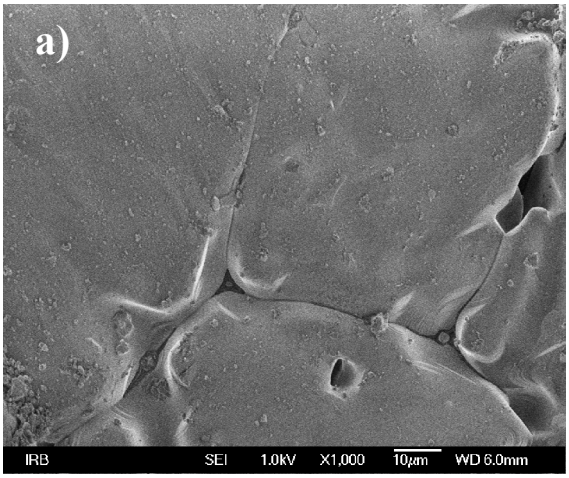
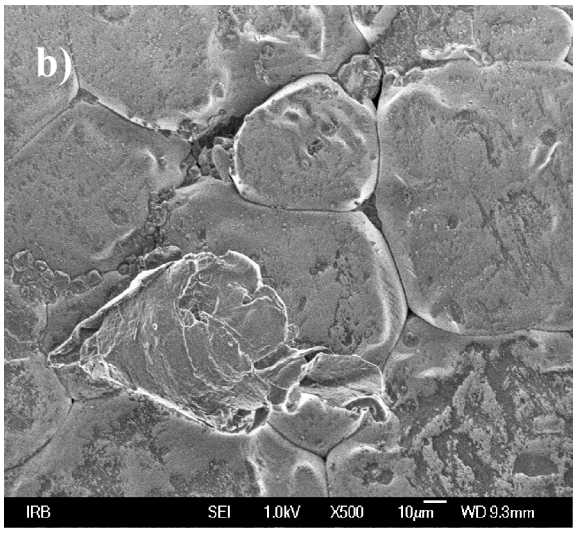


Figure 4

Figure 4





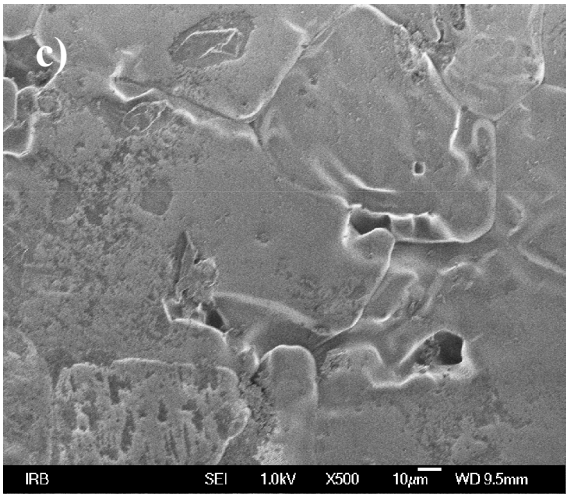


Figure 5

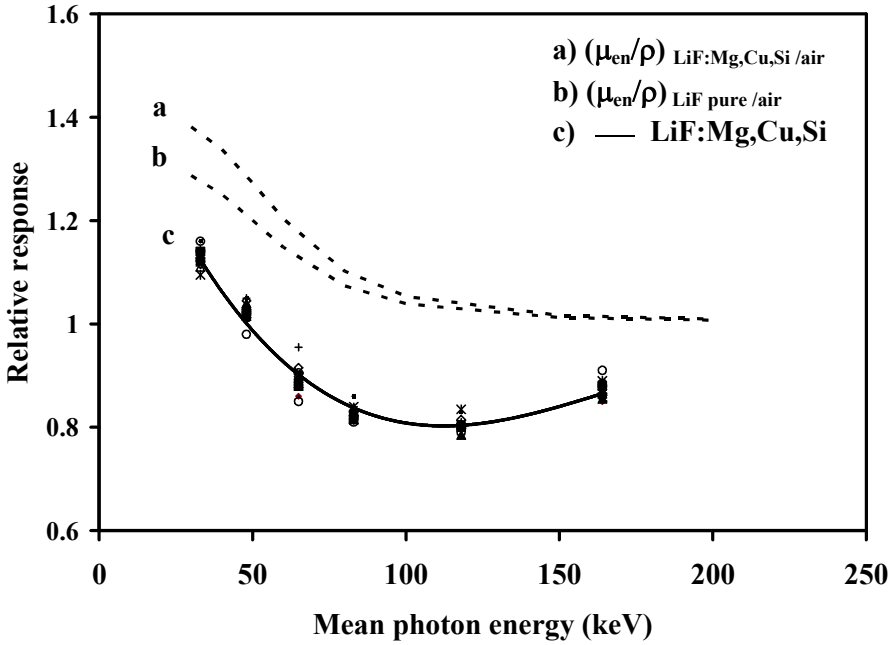

1.6
1.4
1.2
1
0.8
0.6
Relative response
0
50
100
150
200
250
Mean photon energy (keV)
a
b
c
a) (μen/ρ) LiF:Mg,Cu,Si /air
b) (μen/ρ) LiF pure /air
c) —— LiF:Mg,Cu,Si

Figure 6

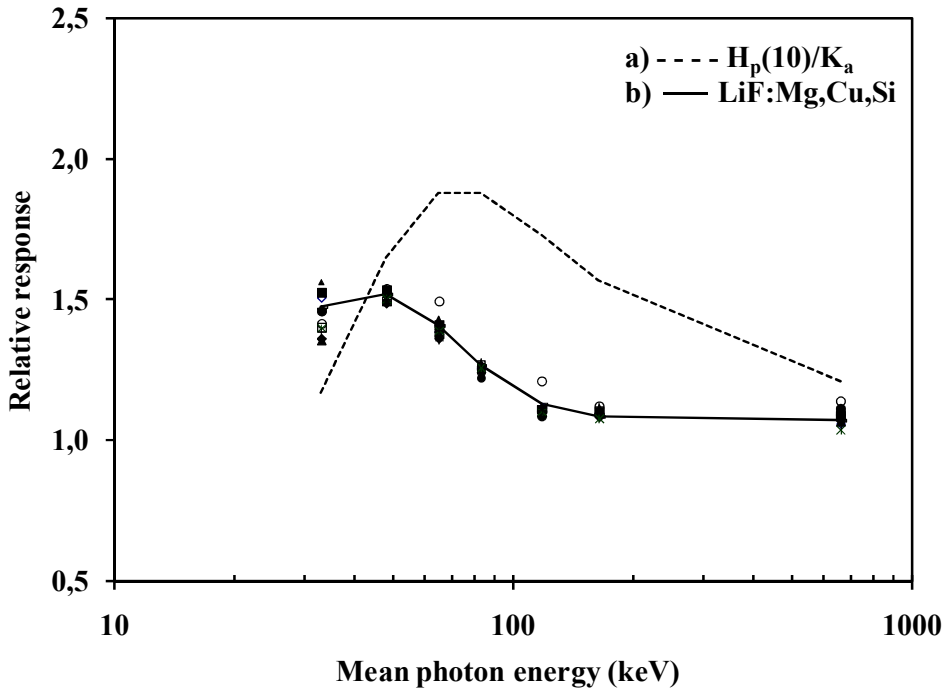

2,5
2,0
1,5
1,0
0,5
Relative response
a) - - - - $H_p(10)/K_a$
b) —— LiF:Mg,Cu,Si
10
100
1000
Mean photon energy (keV)

Table 1

Dopant[a] Mg (mol%)	TL sensitivity relative to TLD-100	Dopant[b] Cu (mol%)	TL sensitivity relative to TLD-100	Dopant[c] Si (mol%)	TL sensitivity relative to TLD-100
0.0	0.3-0.9*	0.0	1.8-2.6*	0.0	0.2-1.3*
0.05	10	0.01	60	0.1	0.2
0.10	12	0.02	64	0.2	0.6
0.15	38	0.025	62	0.3	55
0.20	44	0.03	63	0.6	56
0.25	51			0.9	62
0.30	58			1.2	51
0.35	65				
0.40	63				
0.45	62				
0.50	62				

[a] Cu=0.025 mol%, Si=0.9 mol%

[b] Mg=0.45 mol%, Si=0.9 mol%

[c] Mg=0.45 mol%, Cu=0.025 mol%

* The sensitivity changed in repeated measurements cycles

List of figures:

List of tables

Dear Sir,

As you recommended I revised my manuscript number RADMEAS-D-09-00056

Manuscript title: Influence of dopants on the glow curve structure and energy dependence of LiF:Mg,Cu,Si detectors

Reviewers' comments:

This is an important article that documents the specific dependence of of a relatively new tissue equivalent TLD material on the dopants concentration. It is shown (Table 1) that the relative sensitivity "jumps" to very high values when the dopants concentration deviates slightly from 0%.

What is missing here is some data to show how the sensitivity changes in this critical region close to 0%. For example for Si, what happens between 0.0 and 0.3%? This is critical information that is missing and should be included before this manuscript can be published.

In addition I have a few specific comments:

1. In the abstract, specify what are the "certain activators" that influence the energy dependence the most.

2. Also in the abstract, specify the dopant concentration that gives a sensitivity that is 65 times higher than TLD-100.

3. What specific IAEA recommendation for individual monitoring specifies energy dependence for TLDs?

4. Replace "Harshaw" with "Thermo Fisher Scientific"

5. Explain what is "number of impulses"?

6. Overall the paper is well written but there are still several English errors. These errors should be corrected before publication.

Response to reviewer's comments:

The authors thanks for reviewer's comments. First accept our apologies for slow processing the reviewers comment, but this is because we had to make some additional experiments and also prepare some additional concentrations of Si dopant in cooperation with our co-authors from Korea J.I. Lee and J.L. Kim.

As you recommended we prepared and measured the concentrations of Si dopant in region between 0.0% and 0.3% and included the new data in Table 1 and in the Figure 3 and also in the text about sensitivity changes

Concerning additional comments:

We accept all reviewer comments and made the following changes:

1. In the abstract, specify what are the "certain activators" that influence the energy dependence the most.

   The reviewr has right, the statement is not clear. Therefore the effect of the concentration of the activators on the energy response is preformulated in the abstract.

2. Also in the abstract, specify the dopant concentration that gives a sensitivity that is 65 times higher than TLD-100.

   We specifed in the paper dopant concentration that gives sensitivity that is 65 times higher than TLD-100.

3. What specific IAEA recommendation for individual monitoring specifies energy dependence for TLDs?.

   IAEA Safety Standards Series. 1999. Assessment of occupational exposure due to external sources of radiation Safety Guide No. RS-G.1.3. specifies energy dependence of TLDs used for individual monitoring.

   Doses of the order of the annual dose limits measured by a mumber of dosemeters worn on the surface of the body should not differ by more than -33 % or +50 % (at the 95% confidence level) from the dose equivalents that would be indicated by an ideal dosemeter worn at the same point at the same time (IAEA 1999).

4. Replace "Harshaw" with "Thermo Fisher Scientific"

   We replaced Harshaw with Thermo Fisher Scientific (eralier Harshaw).

5. Explain what is "number of impulses"?

   The phrase „number of impulses“ was replaced by „TL response“ in the text.

I hope that you will accept all the corrections and proceed with publishing the manuscript

Yours sincerely,
Zeljka Knežević