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OSL Characteristics of Cu Activated $NaLi_2PO_4$ Phosphor

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Cu-activated $NaLi_2PO_4$ phosphor material was successfully synthesized through solid state diffusion method. It was mainly doped with Cu_2Cl_2 and $CuCl_2$ salts for impurities in the form of Cu^+ and Cu^{2+} respectively. Powder X-ray Diffraction (PXRD) was carried out for the confirmation of single phase of synthesized phosphor material. Dosimetric properties of the material were investigated using the Optically Stimulated (OSL) techniques. We have found maximum sensitivity of Cu^{2+}: $NaLi_2PO_4$ samples at concentration 1000ppm (0.1 mol%) in OSL with annealing temperatures 1073K. Whereas for Cu^+: $NaLi_2PO_4$ samples, 873K was considered as the optimization annealing temperature with impurity concentrations 0.08mol%. The phosphor material exhibits good dosimetric characteristics. It was found that $NaLi_2PO_4$:Cu^{2+} is little less (3 times) and $NaLi_2PO_4$: Cu^+ is 4.2 times less sensitive than that of STD. Al_2O_3:C (Landauer Inc., USA) respectively. For further OSL studies only $NaLi_2PO_4$:Cu^{2+} used. Low TL fading, for $NaLi_2PO_4$:Cu^{2+}, only 10.74% fading takes place in 10days after that it negligibly fade. Also, wide OSL dose response (10-15kGy), easy optically bleaching for reusability and easy to handle are some of the other significant qualities observed in our samples. Simple method of preparation and tissue equivalence (Zeff~10.8) adds on the advantages of $NaLi_2PO_4$: Cu phosphor. This type of phosphor is also suitable for high energy radiation dosimetry used in OSL techniques.

Keywords: Optically Stimulated Luminescence (OSL), Dosimetry, Phosphor, Solid State Diffusion, $NaLi_2PO_4$: Cu, Sensitivity, Reusability, Optically Bleaching

1 Introduction

Optically stimulated luminescence (OSL) technique is broadly used in high energy radiation dosimetry[1-4]. It depends on the recombination of electrons and holes pairs on stimulation got trapped in defects produced during exposure to ionizing radiation of the host material. Intensity of thus produced luminescence is correlated to the energy deposited in the detector material. OSL, the stimulation was done with the help of optical means. It has also established itself as technique for studying redox reactions, phase transitions, colour centres and other kind of defects in samples. In this technique pre- irradiated (ϒ-source Co^{60} source) phosphor used. In Optically Stimulated Luminescence (OSL) technique, single wavelength light lying in the visible region is stimulated on the already irradiated phosphor sample which emits light. Formerly to read the sample, the light from the stimulated source and later from the emitted light was separated by placing appropriate filters in front of the source and detector. The intensity v/s time is plotted and the graph is known as OSL decay curve. The intensity of the curve is proportional to the dose absorbed by the material. CW-OSL is more advanced technique compared to TL due to its speedy multiple readouts used in dosimetry[5-7].This technique has become more convenient because of numerous benefits over TL dosimetry, such as totally optical nature, efficient power consumption in LEDs (green, Blue) stimulations, possible application in online measurements, advantage of re-evaluation of irradiation in situation of doubts and less chances of structural changes during heating due to which some TLD's lose their reusability[8]. However, the continuous wave (CW; Stimulating light constant) OSL assembly are light sensitive and should be put in a dark paper until the OSL readouts are taken. Difficulty in synthesis of many commercially phosphors, makes them costlier. For examples Al_2O_3:C requires very high temperature furnaces with vacuum facilities as the doping is not easy in air atmosphere for example such as an OSL phosphor BeO is also tissue equivalent but very special measures are required for its preparation and management due to its toxicity to human lungs[5].

The challenges are therefore underway to develop new OSLD phosphors. There are a few such highly sensitive phosphors, e.g., $NaMgF_3$:Eu^{2+} [9],

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$NaLi_2PO_4:Eu$[10], $NaLi_2PO_4:Ce$[11], $SiO_2:Cu$[12], $LiMgPO_4:Tb,B$[13], $Y_3Al_5O_{12}:C$[14], $Na_2SiF_6:Cu,P$[15], $Al_2O_3:B$[16], $LiAlO_2$[17], MgO:Tb[18] etc., developed in different laboratories are reported in the literature but are not yet to be used commercially. The basic conditions for a good OSL phosphors are that the emission should lie within the range 350-425nm (because sensitivity of detectors is mostly high in this range). Also, it should bear defects with more photoionization cross-section in the blue region (450-550) then compared to green region/IR region (from 650 to 800 nm) [1]. The sensitivity of CW-OSL material depends upon various kind of defects, ionizing sources, filters and detectors (usually with PMT wide bandwidth).

In the present paper, we report the Cu doped $NaLi_2PO_4$ as highly sensitive and low-Z material. The phosphor is observed with many promising characteristics such as simple process of synthesis, low-Z, highly sensitive, nontoxic in nature, low fading, wide dose response, excellent reusability. All these properties of $NaLi_2PO_4$:Cu are comparable with that of the commercially available OSLD phosphors which makes it a very useful candidate for high energy radiation monitoring devices.

2 Materials and Methods

2.1. Synthesis

Microcrystalline form of Cu^+ and Cu^{2+} doped $NaLi_2PO_4$ phosphor prepared by solid state method. LiOH and NaH_2PO_4 (99.0% CDH, India) are using as initial materials. The materials were made taking into the consideration of the below chemical reaction by using solid state reaction method.

$$2\mathrm{LiOH} + \mathrm{NaH_2PO_4} + \mathrm{CuCl_2} \longrightarrow \mathrm{NaLi_2PO_4{:}\,Cu^{2+}} + \mathrm{H_2O} + \mathrm{Cl_2}$$

$$2\mathrm{LiOH} + \mathrm{NaH_2PO_4} + Cu_2\mathrm{Cl_2} \longrightarrow \mathrm{NaLi_2PO_4{:}\,Cu^{+}} + \mathrm{H_2O} + \mathrm{Cl_2}$$

Initially, suitable amount of $CuCl_2.H_2O$ and $Cu_2Cl_2.H_2O$ were taken as Cu^{2+} and Cu^+ impurities respectively. Then, LiOH and NaH_2PO_4 taking into molar ratio of 2:1 were mixed well in an agate motor. The mixture in powder forms was initially heated for 12h at 673K and cooled slowly to 300K. For final preparation of the material, it was crushed to fine particles and given heat treatment at 873K and 1073K for the same interval of time and then, slowly cooled down to 300K again. The prepared sample was finally crushed in motor pestle and sieved (by using sieves of different BSS sizes) to obtained different particle size ranging from 45µm-350µm. The best results were obtained for the particle size in the range between 63 µm-150 µm of $NaLi_2PO_4:Cu^{2+}$. All the prepared samples were annealed at different ambient temperatures ranging from 373K to 1273K for 1.0h and quenched to 300K (room temperature) by placing quartz container on a metal block to improve sensitivity. These materials were used for further studies, as well. Effective atomic number ($Z_{eff.}$) of the material was determined by using these formula $\mathrm{Zeff.} = \sqrt[3]{\frac{\sum n_i X_i^4}{\sum n_i X_i}}$ where n_i is the fractional part by weight of whole compound occupied by the element, Xi is the atomic number[19] and it was came out to be =10.8 for $NaLi_2PO_4$.

3 Results and Discussion

3.1. XRD (X-ray diffractometer)

The PXRD of the microcrystalline $NaLi_2PO_4$: Cu phosphor material was matched with that of the reported data in the literature "JCPDS file no.#80-2110" with lattice parameter a=6.884, b= 9.976, c= 4.927 and $\alpha = \beta = \Upsilon = 90°$. This material is having a direct band gap of 5.169eV. It was also confirmed that phosphor has single phase (Nalipoite) having space group Pmnb (62) and orthorhombic structure. The peaks of the synthesis material in XRD pattern were also indexed with the help of miler indices (h,k,l). The intensity (number of counts) of different diffraction peaks were identified and recorded using a point detector (scintillation counter). It could be seen from (Fig. 1) that the experimental data matches exactly same with the standard JCPDS file #80-2110. No additional peak related to impurity is present.

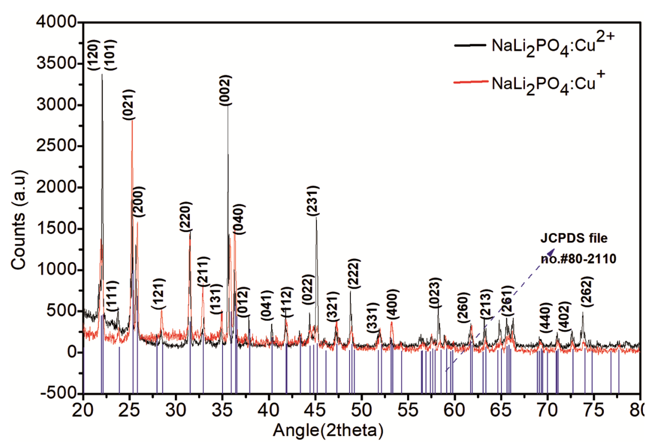

Fig. 1 — XRD form of the $NaLi_2PO_4$ phosphors (our material). The data available in the literature" JCPDS File# 80-2110" is also given for comparison.

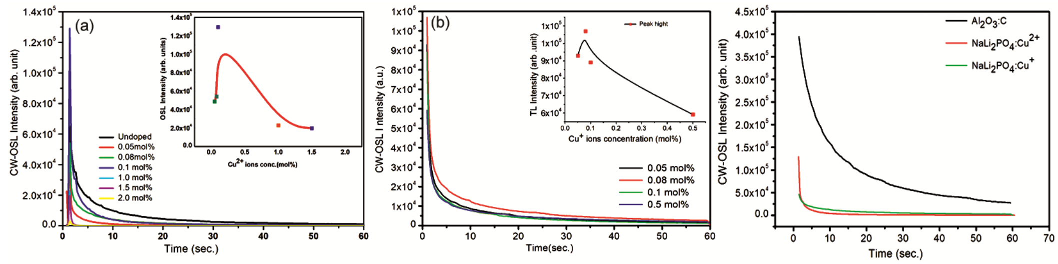

Fig. 2 — (a) Optimization of CW -OSL sensitivity of $NaLi_2PO_4$ with doping concentration (Cu^{2+} ions) ranging from 0.05-2.0 mol% phosphor material annealed for 1.0 hr at 1073K and irradiated with gamma ray Co^{60} source at 10 Gy. (b) Optimization of OSL sensitivity of $NaLi_2PO_4$ with doping concentration (Cu^+ ions) ranging from 0.05-0.5 mol% phosphor material annealed for 1.0hr at 873K and irradiated with gamma ray Co^{60}source at 10 Gy, Combined graphs of Cu doped $NaLi_2PO_4$ Sensitivity comparison with STD Al_2O_3:C ("Luxel TM, Landauer Inc., USA") at 10 Gy gamma ray Co^{60} source.

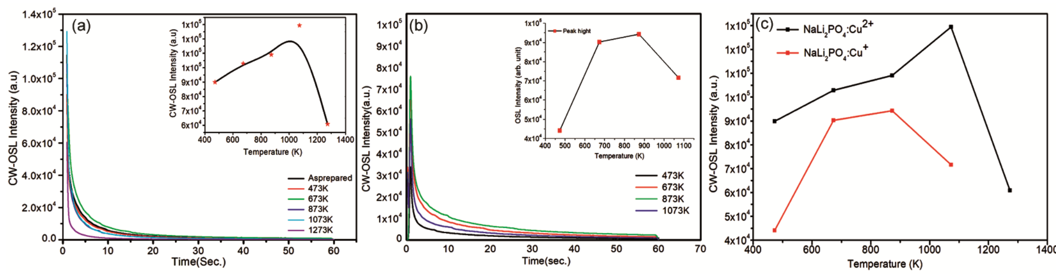

Fig. 3 — (a) Annealing temperatures effect on OSL intensity are shown in decay curves. Variation of different annealing temperatures with OSL intensity is shown in the inset. The sample of 0.1 mol% Cu^{2+} (optimized concentration) used for these studies and were irradiated at 10 gray by gamma ray Co^{60} source, (b) Annealing temperatures effect on CW-OSL sensitivity of Cu^+ doped $NaLi_2PO_4$ are shown in decay curves. The sample of 0.08 mol% Cu^+ concentration (which shows the maximum CW-OSL sensitivity) used for these studies and were irradiated at 10 Gray by gamma ray Co^{60} source, and (c) Combined graphs of Cu doped $NaLi_2PO_4$ with different annealing temperatures.

3.2. Dopant concentration effect on OSL properties of Cu activated $NaLi_2PO_4$

Variation of doping concentration (0.05-2.0 mol%) with OSL intensity of $NaLi_2PO_4$:Cu^{2+} are shown in (Fig. 2(a)). It was observed that if we increase doping concentration, OSL intensity also increases up to 0.1mol% after that it starts decreasing due to concentration quenching effect.

We have found that Cu^{2+}: $NaLi_2PO_4$ sample is little 3.17 times less sensitive than STD. Al_2O_3:C (Landauer Inc., USA) shown in (Fig. 2(a)) But in case of Cu^+ doping the maximum concentration was found at 0.08mol% when annealed at 873K. Its sensitivity is 4.2 times less than that of STD. Al_2O_3: C shown in (Fig. 2(b)). It was observed from combined (Fig. 2(c)) graphs that $NaLi_2PO_4$:Cu^{2+} is more sensitive than $NaLi_2PO_4$:Cu^+ . $NaLi_2PO_4$:Cu^{2+} was used for further studies.

3.3. Effect of annealing temperature on CW-OSL intensity

$NaLi_2PO_4$:Cu^{2+} material (0.1 mol%) was annealed at different ambient temperatures (ranging from 373K-1273K. It is mentioned here that material showed maximum sensitivity at 1073K which is shown in (Fig. 3(a)). Similarly, $NaLi_2PO_4$:Cu^+ material (0.08 mol%) was annealed at different ambient temperatures (ranging from 373K-1073K) It is mention here that when it was studied later as an OSLD phosphor, the same material showed maximum sensitivity at 0.08 mol% with same annealing temperature.

3.4 OSL measurements

Effect on OSL sensitivity of stimulation sources

For optically stimulation we want to use two different type of excitations sources (blue LED cluster 80mWcm^{-2}, green LED cluster 40mWcm^{-2}) to see which is more appropriate. The results can be seen from (Fig. 4). It is observed that optically stimulation by blue LED yielded more (3.3 times) OSL intensity (signal) compared to the green LED for the samples exposure by ϒ-ray Co^{60} source at 10Gy. Since, ionization power of blue cluster LED is just double to that of green cluster LED, It is observed from (Fig. 8) that the stimulation power of Blue LED is more suitable than green LED due to its photo-ionization cross-section (also given in table 1). It is difficult to determine the difference between the blue and green

LEDs accurately since there is a difference between their power intensities due to difference in their wavelengths (energies). However, blue LEDs are used for dosimetric purpose also.

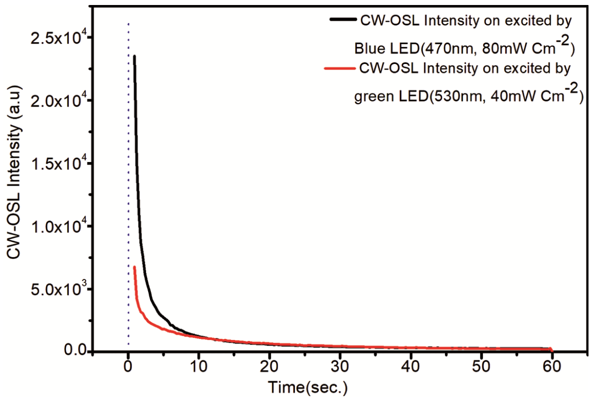

Fig. 4 — Effect of the stimulation source on the CW-OSL decay curves of $NaLi_2PO_4:Cu^{2+}$ stimulation by Blue LED (80mWcm^{-2}) and green LED (80mWcm^{-2}). The material was irradiated at 10Gy of ϒ- ray Co^{60} source.

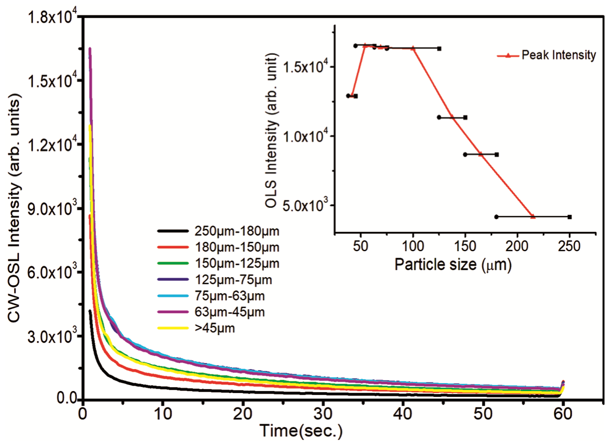

Fig. 5 — Particle size effect on $NaLi_2PO_4:Cu^{2+}$ irradiated at ϒ-ray Co^{60} source.

3.5 Particle size effect

To obtain the best particle size of the phosphor material, was initially Cu^{2+} (0.05-2.0 mol%) doped LiOH and NaH_2PO_4 into 2:1 molar fraction was mixed in ethanol by using agate motor and pestle for better mixing. The temperature programmable controller muffle furnace (West 6400) taking temperature stability ±1K was operated for synthesis of $NaLi_2PO_4$ phosphor. Initially the appropriate amount of mixture was heated at 673K for 12 hours in air atmosphere and cooled to 300K, then crushed again to obtained fine powder. This process was repeated again at 873K and 1073K for same time period. Finally, to obtain best particle size (ranging from 45 μm -250μm), the sample were crushed and sieved. It can be seen from Fig. 5. that the best particle size was found to be within the range of 63 μm -125μm. All the sample were annealed at 1073K for further characterization [20-21].

3.6 Dose response

3.6.1. Optically stimulated emission (OSL) dose response of $NaLi_2PO_4$: Cu^{2+}

$NaLi_2PO_4$: Cu^{2+} samples with 0.1mol% impurity concentration of Cu^{2+} ions annealed at 1073K annealed for 1.0 hr were irradiated with dose ranging from 10-12kGy with gamma ray Co^{60} source and then, CW-OSL decay curves were recorded. The result is shown in (Fig. 6(a)). There is no appreciable change in the shape of the decay except for the intensity increases as dose increases linearly[1]. The response curves are taken for the excitation given by using blue LED (470nm, 80mW cm^{-2}) light source. It can be seen from (Fig. 6(a)) that the response curve is linear up to 12kGy but after that it starts to decrease. This implies that there is no saturation up to high dose

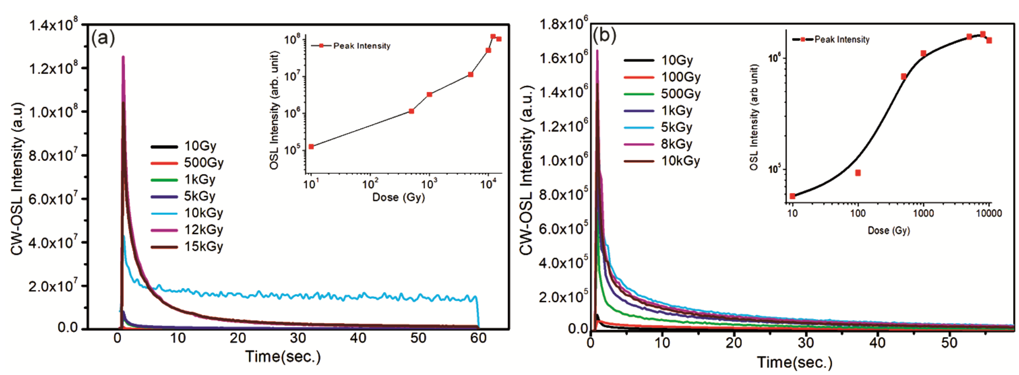

Fig. 6 — (a) OSL Dose response curve (ranging from 10Gy-15kGy) of $NaLi_2PO_4:Cu^{2+}$ stimulated by the blue LED (80mW,470nm) before irradiation the 0.1mol% phosphor material annealed at 1073K for 1.0 h, (b) OSL Dose response curve (ranging from 10Gy-8kGy) of $NaLi_2PO_4$: Cu^{+} stimulated by using blue LED (470nm, 80mW). The phosphor was annealed at 873K for 1h before irradiation with 0.08 mol%.

such as 12kGy and is useful in finding applications in high radiation dose monitoring systems.

3.6.2. Optically stimulated emission (OSL) dose response of $NaLi_2PO_4$: Cu^+

Cu^+ doped $NaLi_2PO_4$ samples with 0.08mol% impurity concentration of Cu^+ ions at 873K annealing temperature for 1 h were irradiated with dose ranging from 10 Gy -15kGy irradiate with gamma ray Co^{60} source and then, CW-OSL decay curves were recorded. The result is shown in (Fig. 6(b)). There is no appreciable change in the shape of the decay except for the intensity increases as dose increases linearly. The response curves are taken for the excitation given by using blue LED (470nm, 80mW cm^{-2}) light source. It can be seen from (Fig. 6(b)) that the response curve is linear up to 8kGy but after that it starts to decrease. This implies that there is no saturation up to high dose such as 8kGy and is useful in finding applications in high radiation dose monitoring systems.

3.7 Theoretical fitting of the CW-OSL decay curve and photoionization cross-section.

The theoretical fitting CW-OSL curve which is stimulated by using blue light LED (470nm, 80mW) is shown in (Fig. 7). The figure of merit of the theoretical fitting decay curve was approximate 0.09%. It can be seen that stimulation by blue light decay curve consists of mainly 3 components -fast, medium and slow component. The sum of all these components was done by theoretically fitting of graphs (decay curve with dotted line in orange colour)[1,22].

The CW-OSL intensity of the decay curve stimulated by blue LED can be denoted by given equation.

$I_{OSL\ (blue)} = B1exp.\ (-x/\tau1) + B2exp.\ (-x/\tau2) + B3exp.\ (-x/\tau3)$ [13]

Where Y0=582.39666, B1=4230284.77603, t1=0.191±0.04425s, B2=65544.21081 t2=1.50389± 0.08466s, B3=14044.93945, t3=6.82362±0.35198s "Photo ionization cross-section (σ)" can be determined by the following formula (σ)=1/ (φ X τ), where τ and φ are decay constant (s) and incident flux (number of photons incident per cm^2 per second) respectively. The resultant values of photo ionization cross-section and decay constant of the sample are mentioned in Table 1. It can be concluded from the table that "photoionization cross- section" depends on stimulated energy as well as on the incident flux[1].

Table 1 — Photoionization cross- section & decay constants data for OSL response stimulated by using Blue LED (470nm, 80mW) are shown in below table.

OSL components	Coefficients	Decay constant(s)	Photoionization cross- section σ(cm^2)
Fast	4230284.77 (B1)	0.1 (τ1)	$0.00737X10^{-14}$
Medium	65544.21 (B2)	1.50 (τ2)	$0.000936X10^{-14}$
Slow	14044.93(B3)	6.82 (τ3)	$0.000206X10^{-14}$

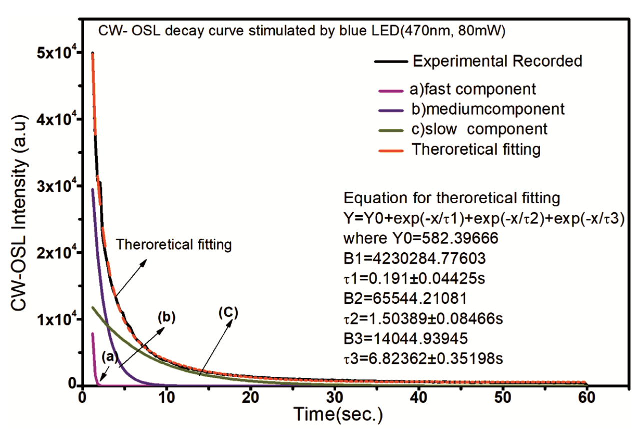

Fig. 7 — Theoretically fitted OSL decay graphs stimulated by using blue LED light (470nm,80mW). The theoretical curve is shown by dotted blue line and experimental curve is shown by solid black line. The component is; (a) 1st order de-convolution curve (red solid line), and (b) 2nd order de-convolution curve (pink solid line).

3.8 Cu^{2+} doped $NaLi_2PO_4$ readouts many times exposure with ϒ-ray Co^{60} source at 10 Gy.

It is clear from (Fig. 8) that the OSL sensitivity reduces drastically after 1st readout itself. However, it can be found from figure that it does not completely diminish even after 5 readouts. Therefore, it is concluded from Fig. 8 that some of its optically

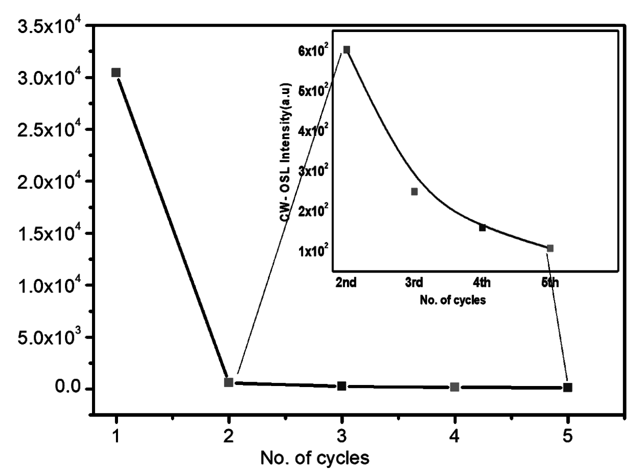

Fig. 8 — Cu^{2+} doped $NaLi_2PO_4$ Readouts many times irradiated with 10 Gy Co^{60} ϒ-source. Shows OSL Intensity v/s no. of cycles. It is seen from Fig. 8 that during first readouts, more than 50% intensity was vanished. An expanded view of the successive readouts is shown in the inset of the figure.

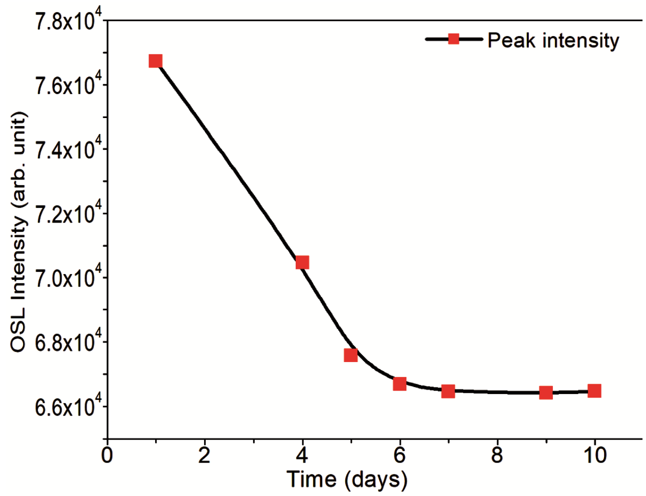

Fig. 9 — Typical CW-OSL decay curves of $NaLi_2PO_4:Cu^{2+}$ taken 1.0hr after irradiation and storing in dark at room temperature (300K). Fading was taken starting from the same day up to 10 days.

sensitivity still retains inside the sample and gets over only after many subsequent readouts.

3.9 Fading

The results showing the OSL fading effect in our samples $NaLi_2PO_4:Cu^{2+}$ are shown in (Fig. 9) Hence, it can be concluded that only 10.74% fading of the CW-OSL signal was recorded in 10 days demonstrating negligible fading even after 7 to 10 days. This proves the efficacy of our sample in the field of medical and personal dosimetry.

3.10 Reusability

Reusability is one of the most important properties to satisfy for a good OSLD phosphor. It is not considered good for a material to be dosimeter, If its sensitivity changes at every time at each no. of cycle of irradiation and readouts. Therefore, the newly developed $NaLi_2PO_4:Cu^{2+}$ OSLD phosphor was supposed to be tested on such grounds. Pallets of the above sample were exposed to 10 Gy of ϒ-rays followed by the quickly cooling it to the room temperature. Same sample was again irradiated second time with the same amount of dose and readouts were done by using same heating procedures. Several such cycles of irradiations and readouts were performed on the same sample by keeping all other parameters constant. No significant changes have been observed in the OSL sensitivity the material. Therefore, a very good reusability is seen in phosphor thus paving its way to radiation dosimetry using optically stimulated luminescence. The material showed excellent reusability ability.

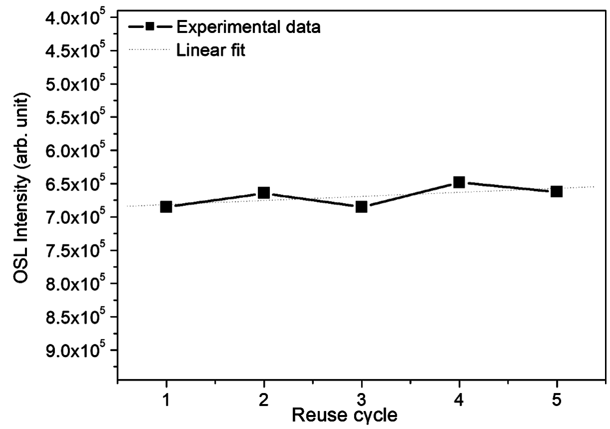

Fig. 10 — OSL intensity graph for repeated reusability was irradiated by 10 Gy Co^{60} source. Above figure shows the normalised graph of CW-OSL signal intensity for repeated reusability of Cu^{2+} doped $NaLi_2PO_4$ phosphor material, irradiated by 10 Gy from Co^{60} source. The sample was irradiated and readouts were conducted 5 times again and again by the same dose to check our material's reusability characteristics. The dotted line represents the linear fitting.

Results proving the same are shown in the (Fig. 10) below.

3.11 Optically bleaching

Bleaching means it is a procedure of removal of internal stress/residual signal with exposure to high intense blue light. Several pellet of the Cu^{2+} doped $NaLi_2PO_4$ OSLD phosphor were prepared for Optically bleaching. The phosphor was irradiated at 10 Gy by using gamma ray Co^{60} source.

The samples were bleached in between every OSL readouts to remove the leftover OSL signal completely. After first readout itself, the remaining signal was found to be only of the order of a few Gy. The overall time sufficient for bleaching was found to be around one and a half hours. Several samples were irradiated for different times at various trial doses, with the same repetitive procedure. The OSL was taken after every bleaching cycle.

4 Conclusion

Cu^{2+} doped $NaLi_2PO_4$ phosphor shows excellent OSL characteristics suitable for the high- energy radiation dosimetry. The highly sensitive material was compared with STD. Al_2O_3:C ("Luxel^{TM}, Landauer, USA"). It is concluded that the $NaL_2PO_4:Cu^{2+}$ phosphor material is little less sensitive (3.0 times less) than STD. Al_2O_3: C. Simple synthesis-process makes it cost effective. But in case of Cu^+ doping the maximum concentration was found to at 0.08mol% when annealed at 873K. Sensitivity is 4.2 little less times than STD. Al_2O3:C but with wide dose response(10-10kGy) makes its useful to high radiation dosimetry. The OSL fading in the material of $NaL_2PO_4:Cu^{2+}$ was found to be around 10.74% in 10 days, Excellent reusability. It is comparable with the commercially available OSLD phosphor and thus it is acceptable for dosimetry. Dose response was observed to be linear up to high dose~10kGy and then after that it starts decreasing. Due to its wide dose response, it can be used for high dose monitoring devices. Cu^{2+} doped $NaLi_2PO_4$ phosphor shows excellent OSL characteristics suitable for the high-energy radiation dosimetry. It is comparable with the commercially available OSLD phosphor and thus it is acceptable for dosimetry. Irradiation generates electron-hole trap centres which act as luminescent centers. Due to its wide dose response, it can be used for high dose monitoring devices.

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