

OPTICALLY STIMULATED LUMINESCENCE CHARACTERISTICS OF BeO NANOPARTICLES SYNTHESIZED BY SOL-GEL METHOD ¹

Sol-jel Yöntemi Kullanılarak Sentezlenen BeO Nano Parçacıklarının Optik Uyarmalı Lüminesans Karakteristikleri

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ÖZET

Sol-jel yöntemi kullanılarak sentezlenen BeO nano parçacıklarının Optik Uyarmalı Lüminesans özellikleri araştırıldı. Üretilen malzemelerin kristal fazlarını belirlemek için X-Işını Difraktometresi analizleri gerçekleştirilmiştir. Ayrıca, morfolojik yüzey analizleri Taramalı Elektron Mikroskopu (SEM) kullanılarak sentezlenen malzemelerin morfolojisini ve parçacık boyutlarını incelemek için yapılmıştır. . Diğer yandan, Fourier Dönüşümlü Kızılötesi Spektrofotometre (FT-IR) analizleri her örnek için üretilen fazların fonksiyonel gruplarının titreşimsel modlarını gözlemlemek için gerçekleştirilmiştir. Buna ilave olarak, Diferansiyel Taramalı Kalorimetre (DSC) ve Termogravimetrik (TG) analizleri sıcaklık ölçümlerinde malzemenin içerisinde veya yüzeyinde gerçekleşen fiziksel ve kimyasal değişimleri incelemek için kullanılmıştır.

Bu çalışmada, katkılı ve katkısız BeO peletlerinin OSL ölçümleri "Risø TL/OSL okuyucu (Model TL/OSL-DA-20)" ile yapılmıştır. BeO peletleri okuyucu içerisinde bulunan 90Sr / 90Y beta kaynağı ile ışınlanmıştır. Her malzeme için OSL bozunum eğrileri gösterilmiş ve bu eğrilerin analizleri kullanılarak her dozimetrik malzemenin doz cevap, fading (solma), yeniden kullanılabilirlik ve tekrar tekrar okunabilirlik özellikleri belirlenmiştir. Diğer yandan, OSL sisteminin ölçebileceği minimum doz değerleri (MDD) her malzeme için hesaplanmıştır. OSL ve TL sinyalleri arasındaki ilişkiyi gözlemlemek için, malzemelerin TL sinyalleri ve OSL ölçümünün TL sinyalleri üzerine etkisi araştırılmıştır. Işığa hassas TL piklerinin ve OSL sinyallerinin termal aktivasyon enerjileri hesaplanmış olup, sonuçlar kendi aralarında karşılaştırılmıştır.

Anahtar Kelimeler: Berilyum Oksit (BeO), Sol-Gel Sentez Tekniği, Nanofosfor, Lüminesans Dozimetre, Optik Uyarmalı Lüminesans (OSL).

ABSTRACT

Optically Stimulated Luminescence (OSL) properties of the sol-gel synthesized nanopowders of Beryllium Oxide (BeO) were investigated. Luminescence properties of BeO nanoparticles are dependent on the crystal structure, particle size and morphology and therefore strongly dependent on the way of synthesis, thus the way of preparation was modified to enhance the OSL signal from the material. Structural, thermal and morphological properties of BeO in nanopowder and pellet forms were studied using X-Ray Diffraction (XRD), Fourier Transform Infrared Microscopy (FTIR), Simultaneous Thermal Analysis STA (Thermo Gravimetric Analysis (TGA) / Differential Scanning Calorimetry (DSC)) and Scanning Electronic Microscopy (SEM) techniques. XRD and STA analysis suggested an enhancement in crystallinity and thermal stability of the BeO with increasing sintering temperature.

OSL dosimetric properties of the pellets prepared by pressing the synthesized powders were investigated in detail. Thermal stability of the signal as determined with a pulse heating experiment has shown that the OSL signal was stable up to depleted at around 300 °C,

¹ Aynı başlıklı Yüksek Lisans tezinden üretilmiştir.

suggesting the suitability for dosimetric purposes. Dose response of OSL signal intensity was observed to be linear in the dose range 0.1 to 100 Gy. The minimum detectable dose limit was estimated as about $92 \pm 1 \mu\text{Gy}$. Replicated measurements of the samples irradiated with the same dose indicated a good reproducibility. Light induced and dark fading of the OSL signal intensity was also studied.

Key Words : Beryllia, BeO, Optically Stimulated Luminescence, OSL dosimetry, luminescence, nanoparticles

Introduction

OSL phenomenon is based on the emission of light from a solid, previously exposed to ionizing radiation, upon stimulation with a light of proper energy. Radiation dosimetry using OSL relies on the similar types of processes involved in thermoluminescence (TL) dosimetry (Bøtter-Jensen et al., 2003; Yukihiro and McKeever, 2011). Reusability, multiple readability, ease of readout and possibility of point stimulation can be listed among the advantages of OSL dosimetry (Yukihiro and McKeever, 2011). BeO as a luminescent phosphor with an effective atomic number close to that of tissue (for human tissue $Z_{\text{eff}} = 7.42$), $Z_{\text{eff}} = 7.13$, (Scarpa, 1970; Scarpa et al., 1971; McKeever et al., 1995) has been explored for radiation dosimetry.

Based on the characteristic light-sensitive thermoluminescence of BeO, Albrecht and Mandeville (1956) were the first to report the measurement of optically stimulated luminescence from X-ray irradiated BeO using visible photons for stimulation. Tochilin et al. (1969) and Rhyner and Miller (1970) further investigated the potential of BeO (discs and powder) as a luminescence dosimeter. However, these studies were based on the measurement of the optically stimulated afterglow (or delayed OSL) where the luminescence is measured after the exposure to stimulation light. A systematic prompt OSL study, where the luminescence emission was measured during stimulation, has been carried out some decades later. Bulur and Göksu (1998) investigated the continuous-wave (CW) OSL from BeO ceramics and discussed the OSL signal and its characteristics relevant for radiation dosimetry.

Great majority of the experimental work that can be found in the literature have been performed on some selected types of commercial BeO ceramics which are suitable for radiation dosimetry. The number of studies focused on the OSL characteristics of synthesized BeO (especially nano-powders) is rather limited. In this work, we present the results of a study concerning the synthesis of BeO nano-powders prepared using the sol-gel technique. Structural, morphological and optically stimulated luminescence properties of the BeO nanopowders and the pellets produced using these nano-powders were investigated concerning the requirements for radiation dosimetry.

Material and Method

Material

In nanopowders, since small particles are closer together, there are more contact areas and less distance to travel to fuse together (Schubert and Husing, 2000). This in turn means that the preparation of nanomaterials and investigation of their properties have become the main direction of the development of promising novel materials. In this work, we prepared nanopowders of BeO using the polymeric sol-gel route due to some special characteristics namely nanoparticle size leading to high reactivity and low processing temperatures, high purity and homogeneity.

Sol-gel method applied for preparing BeO nanopowder was accomplished using beryllium sulphate tetra-hydrate ($\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$) (Sigma >99.0 %) and citric acid ($\text{C}_6\text{H}_8\text{O}_7$) salts (Sigma 99.5 %) as reactants and ethylene glycol ($\text{C}_2\text{H}_6\text{O}_2$) (Sigma 99.8 %) as solvent. All the samples employed were prepared from high purity raw materials. Beryllium sulphate

tetra-hydrate ($\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$) was used as the precursor for the inorganic component. Citric acid played the role of complexing agent. It was employed as a chelating agent (Hwang et al., 2001) which provides the mixing of cations at the molecular level in this sol-gel process.

Method

Sol-gel formed BeO materials as a result of the applied sintering temperatures were characterized using X-Ray Diffraction (XRD), Fourier Transform Infrared Microscopy (FTIR), Simultaneous Thermal Analysis STA (Thermo Gravimetric Analysis (TGA) / Differential Scanning Calorimetry (DSC)) and Scanning Electronic Microscopy (SEM) methods. The same structure analysis were repeated for the BeO ceramic pellets.

The crystal phase and cell parameters of prepared samples were determined by X-ray diffraction (XRD) technique using a Rigaku, Smart Lab x-ray Diffractometer (XRD) with Cu-K α ($\lambda=1.5406 \text{ \AA}$). A Fourier Transform Infrared (FTIR) spectrometer (ThermoScientific, NicoletS10, equipped with the Universal ATR sampling accessory) was used to determine the vibrational bonds in the 4000–550 cm^{-1} region and diluted sample powders with potassium bromide.

OSL and TL measurements were performed using an automated TL/OSL reader (Model Riso TL/OSL DA-20, National Laboratory, DTU-Denmark) which is equipped with a bialkali photomultiplier tube (Electron Tubes 9235 QA). Blue LEDs ($\lambda_p \sim 470 \text{ nm}$, FWHM $\sim 30 \text{ nm}$) were used for stimulation. OSL measurements were made using continuous wave stimulation at a power density of $\sim 50 \text{ mWcm}^{-2}$. The intensity of the emitted luminescence was measured by a light detection system using a 7.5 mm thick UV band pass filter (Hoya U-340; THK Photo Products.Inc., Long Beach, CA, USA) placed in front of the PMT. After each measurement, the background was measured at the same conditions and this background was subtracted from the OSL measurements. Thermoluminescence measurements were performed by heating the sample from room temperature to 450 $^{\circ}\text{C}$ at a rate of 3 $^{\circ}\text{C/s}$ in nitrogen atmosphere.

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Research and Discussion

In this work, after synthesizing BeO nanopowder material and producing BeO ceramic pellets, OSL signals of samples were measured to ensure that the signal has a convenient monotobous decay. In Fig.1 a typical OSL decay curve measured from a 0.5 Gy irradiated BeO pellet is shown as an example. As seen from the figure, the OSL signal exhibits a fast decay and the back ground levels are reached in a few seconds (see the inset of Fig. 1). In this paper, much more detailed discussion of the analyses of the OSL decay curves will be given later.

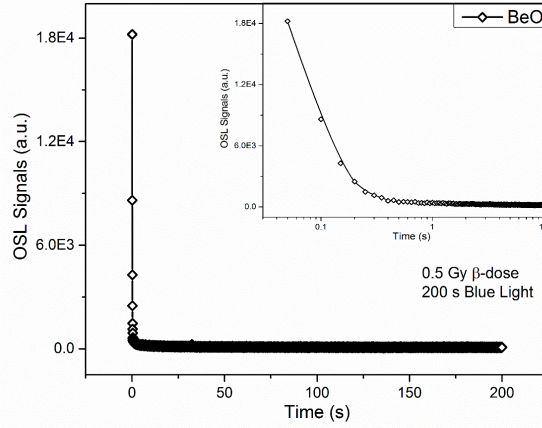


Figure 1. A typical OSL decay curve of BeO ceramic pellets following 0.5 Gy β irradiation. Inset: semilogarithmic plot of the initial part of the OSL decay curve.

In order to decide the optimum sintering temperature, the sensitivity change of the OSL signal from BeO nano-powders were investigated as function of sintering temperature. For this purpose, calcinated BeO nano-powder samples were sintered in air, at four different temperatures (800 oC, 900 oC, 1000 oC and 1100 oC) with the retention time of 4 h. After each sintering step, BeO pellets were irradiated to 1 Gy and OSL signals were recorded by stimulating the sample with blue light and monitoring the luminescence using 290-370 nm band pass filters.

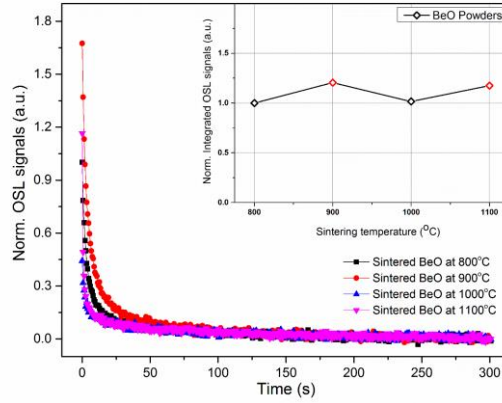


Figure 2. Typical OSL decay curves of BeO nanopowder samples on exposure to β -particles (dose=1 Gy) at different function of sintering temperature 800, 2) 900, 3) 1000, and 4) 1100 oC. A comparison of OSL signals of samples produced using sol-gel method and sintered at different temperatures due to their maximum OSL counts. Stimulation time is 300 s.

The nanopowder samples exhibited similar sensitivities to the beta radiation for all sintering temperatures. Therefore it was not possible to decide the sintering temperature using these data; by considering the XRD patterns (will be mentioned later) a sintering temperature of 1100 oC was decided. Rest of the study was performed using the pellets sintered at 1100 oC for 4h.

In order to investigate thermal behavior of pellet form BeO, the thermogravimetric differential scanning calorimetry and thermogravimetry analysis were carried out in the temperature range of 25 - 900 °C. In Fig. 3, TGA and DSC results are given for BeO pellets. In TGA results, a weight loss was found to be about 2.7 % between the temperatures from 25 to around 73 °C and the percentage of the residual weight loss of BeO pellets after this temperature became 0.2 % at maximum sintering temperature which resulted from the dehydration and decomposition of residues (organics, sulfate, etc.) remained from sol-gel synthesis environment.

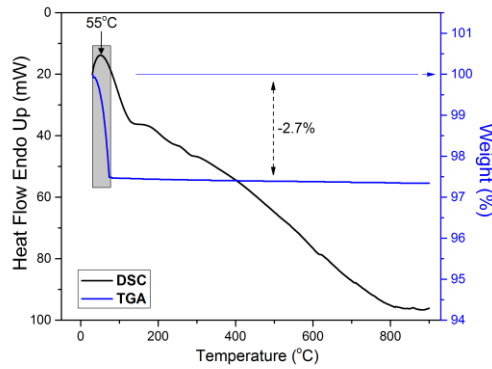


Figure 3. DSC and TG curves of BeO pellet form (10 °C/min in air).

XRD patterns of the synthesized BeO nanoparticle (non-sintered) and sintered BeO nanoparticles are given in Fig. 4. Analysis of the powder XRD patterns indicated that the patterns belonging to all samples matched with the data of BeO reported in ICDD (PDF2.DAT) Card No 01-078-1562 and also little amount of amorphous phase (2 θ : 15 -30 deg) were seen in the main structure. The results are in good agreement with the literature (Norazlina et al., 2013; Zahedifar et al., 2012; Wang et al., 2011). In addition, Fig. 4 shows that amorphous phase decreased with increasing the sintering temperature and completely disappeared in the structure of BeO as pellet form which were synthesized at 1100 °C for 4 h. Our pre-investigation indicated that the highest OSL intensity was obtained from the BeO sintered at 1100 °C, meaning the positive effect of using higher sintering temperature. Therefore, the sintering temperature was chosen as 1100 °C in the present study.

In order to support XRD results, IR transmission spectra of the obtained BeO samples were also investigated. The IR spectra of the BeO nano -powders are presented in Fig. 5. The IR spectra of all obtained materials are in good agreement with each other. It can be seen that the band positions in the IR spectra of all samples were almost constant, indicating a temperature-independent character. The observed peaks became narrower for all sintered BeO nano-powder samples. This case is attributed to difference of the crystal morphology. Besides, the peak at about 600 cm⁻¹ are assigned to the Be-O vibration in sintered BeO pellet.

SEM investigations were conducted to examine the effect of sintering temperature on the particle size and morphology of the synthesized powders, as shown in Fig. 6. Through SEM analysis, regularly shaped particles and narrow size distribution were observed. The grain sizes of non-sintered particles were detected as between 45-85 nm (see Fig. 6a). To investigate the effect of sintering temperature on the sizes of the nano-powder particles, the diameter of the rounded particles in the SEM pictures were measured

and shown in Fig. 6 b,c,d,e and f. It was found that the BeO powders with a particle size of less than 100 nm are uniformly and completely produced with 800 oC sintering temperature (see Fig. 6b). The particle sizes of 81-120 nm (Fig. 6c), 130-245 nm (Fig. 6d) and 245-685 nm (Fig. 6e) were determined for the sintering temperatures of 900, 1000 and 1100 oC, respectively.

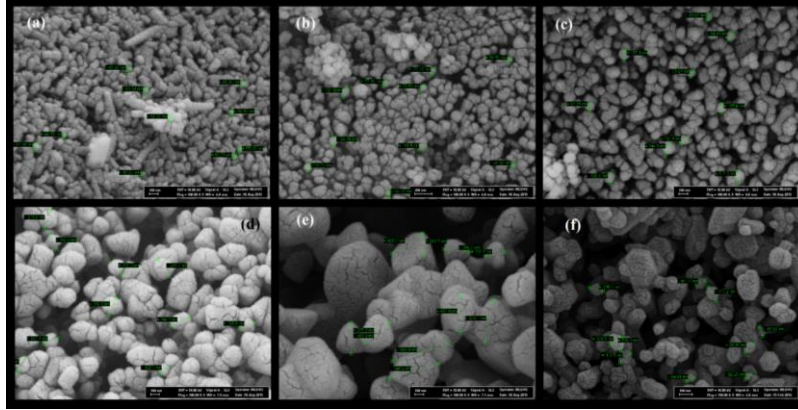


Figure 4. a) SEM images of non-sintered BeO powders. SEM images of BeO powders sintered 4hours at b) 800 oC, c) 900 oC, d) 1000 oC, e) 1100 oC, f) BeO pellets sintered at 1100 oC for 4h.

A monotonous decay of the OSL signal from the synthesized BeO has already been mentioned above. The characterisation of the OSL decay curve is an essential starting point in attempting to understand the luminescence production mechanisms. OSL decay curve of a 0.5 Gy irradiated BeO sample is given in Fig. 7. Fig. 7 also shows the background level (measured using the same experimental parameters) recorded after the OSL measurement.

Exposure of BeO ceramic pellets to high temperatures may induce changes in the sensitivity of the OSL signal concerned. In addition, the total depletion of the traps populated by irradiation (some of which may not be erased by optical exposure) requires a study on the thermal annealing characteristics of the signal considered for OSL dosimetry. The changes induced by high temperature annealing procedures are important since the normal processing of the powder during production requires high temperatures. They indicate (or display) the extent of the modification of the luminescence characteristics when repeated irradiation and annealing steps are involved.

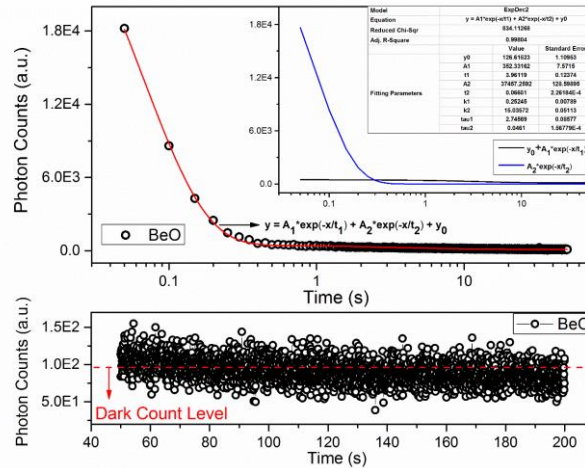


Figure 5. OSL decay curve of a 0.5 Gy irradiated BeO ceramic pellet (preheating at 100 oC for 10 s).

Thermal annealing behaviour of the OSL signal was studied by annealing the irradiated samples at a set temperature and monitoring the OSL at room temperature. The experiments were carried out using 2 Gy irradiated samples annealed at different temperatures ranging from 100 oC to 500 oC with 100 oC steps and for the duration of 10 minutes for each annealing temperature.

One of the very important characteristics of a luminescence signal, considered for radiation dosimetry, is the thermal stability. The luminescence signal should be stable enough to hold the radiation dose information. In addition, possible interference of unstable components of the radiation induced signal should be removed with a proper preheating regime. The thermal stability of the OSL signal measured of the synthesized BeO samples was studied in the temperature range from 50 to 350 oC. For this purpose, samples irradiated with 1 Gy were heated to a preheat temperature and the remaining OSL signal was measured. This procedure was repeated with increasing preheat temperatures for freshly irradiated samples. A plot of OSL signal intensity against the preheating temperature is given in Fig. 9a (upper plot).

In the literature, preheating experiments carried out using commercial BeO ceramic samples (Thermalox 995) have shown that the OSL signal was stable up to a preheat temperature of 250 oC (Bulur and Göksu, 1998; Bulur and Yeltik 2010, Yuki-hara and McKee-ver, 2011; Bulur and Saraç, 2013). Here, in synthesized BeO nano-powders, the system involved in the OSL production comprise at least two different types of trapping states with different thermal stabilities. In a recent study, in BeO ceramics irradiated with high doses Bulur (2014) has observed a similar -two step decay- structure in the preheating profile.

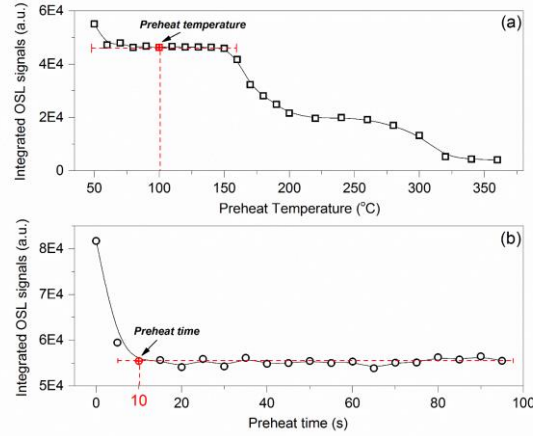


Figure 6. a) Preheating temperature dependence of the OSL signal intensity b) Preheating time dependence of the OSL signal intensity (temp=100 oC).

Radiation dose responses of the BeO samples were determined by monitoring the OSL signals using beta doses of 0.1, 0.2, 0.5, 1, 2, 5, 10, 20, 50 and 100 Gy. As mentioned above, irradiated samples were preheated at 100 oC for 10 s before the OSL measurements. Following the OSL measurements the samples were depleted by measuring the thermoluminescence (450 oC at a rate of 3 oC/s). A plot of integrated OSL signal intensity against the absorbed radiation dose is shown in Fig. 10. As seen from the plot, the dose response of the OSL intensity is nearly linear in the range considered. The slope of logOSL versus logDose curve (which is also defined as the supralinearity index) was found as about 0.96.

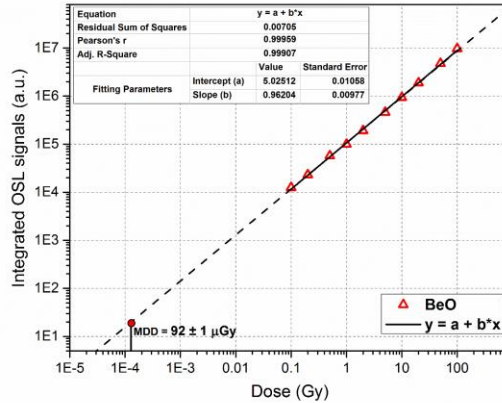


Figure 7. Dose response of BeO pellet dosimeters exposed to ⁹⁰Sr/ ⁹⁰Y radiation.

In dosimetry applications, the OSL dosimeters should be reproducible, i.e., the OSL signal should exhibit the same response when it is re-measured under the same experimental procedures/conditions. In order to investigate the reproducibility, the OSL

signals were measured from five annealed samples of BeO ceramic pellets by a 200 s blue-light stimulation at room temperature after given identical exposures (1 Gy beta dose), with providing 100 oC for 10 s preheating before reading, under carefully controlled laboratory conditions. After each OSL measurement, the samples were depleted by a TL measurement up to 450 oC. The reproducibility of repeated readings of individual samples for an exposure of 1 Gy was tested for ten consecutive measurements.

The storage of dose information over periods of time is an essential property of a passive integrating dosimeter. It was mentioned above that, the luminescence signal is thermally stable up to a temperature of 150 oC (obtained using short preheating times) in the mid or long term storage, the thermal decay of the stored charges has a non-zero probability at room temperatures. Another possibility of losing charges that keep the record of radiation dose information is the anomalous fading which is thought to be due to escape of charges from trapping states via quantum mechanical tunneling.

Here, short term fading characteristics of the OSL signal was tested by storing a group of 1 Gy irradiated pellets in dark for various storage times up to a month.

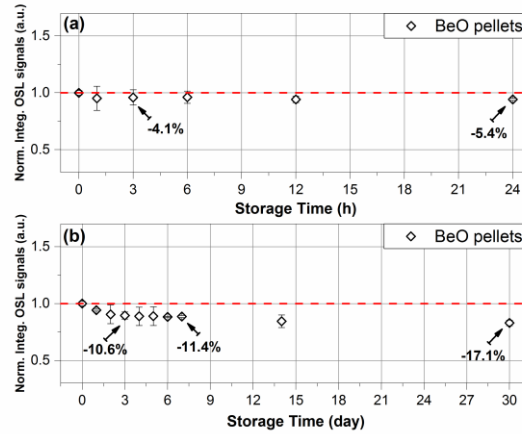


Figure 8. Stability of BeO ceramic pellets over a) 24 hours b) 4 weeks period of storage at room temperature following a 1 Gy beta-ray exposure.

Conclusions

Luminescence properties of BeO ceramics have been investigated for decades both to gain a better fundamental understanding of the mechanisms involved and to develop/use it in a variety of applications. Currently, many groups worldwide are involved in research on BeO due to its superior luminescence properties as compared to those of conventional dosimetric materials. In this study, alternative to the commercially available BeO ceramics, nano-powder BeO has been prepared using sol-gel route and characterized using various techniques including X-ray diffraction, SEM and FT-IR spectroscopy.

OSL characteristics of BeO pellets were investigated in detail to test whether the material is suitable for the purposes of radiation dosimetry or not. The radiation induced OSL signal observed to have a bright, fast decaying component and a slower one with decay constants 0.06 s and 3.96 s using blue LED stimulation at a power density of 50 mW/cm².

Preheating experiments have shown that the OSL signal intensity is stable up to nearly 150 oC exhibiting a plateau region between 50-150 oC and partially decays in the temperature region 150-200 degrees. A second plateau region was observed in the interval 200 to 250 oC and the depletion of the signal was reached at temperatures around 350 oC.

In summary, the OSL signal was observed to be thermally stable and sensitive to radiation dose (with a nearly-linear dose response in a six decade dynamic range) indicating the possibility of usage in the field of radiation dosimetry. However, it is evident that the results presented here are of preliminary in nature. More studies are necessary to develop a better information about the dosimetric properties of the material. These studies may include the determination of the response to radiation energy and type.

In addition, at the moment very little is known about the luminescence mechanism. The nature of the charge storage centers and the luminescence centers is to be studied. Emission and excitation/stimulation spectroscopy studies should be carried out. Furthermore, the luminescence properties can further be enhanced using some dopants to induce more defects in the materials which is an ongoing research in the laboratory.

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