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Structure characterization and luminescence studies of MgO:Li calcinated at different temperatures via solution combustion and sol-gel methods

Veysi GUCKAN[1*], Volkan ALTUNAL[1], Adnan OZDEMIR[1], Zehra YEĞİNGİL[1]

1Department of Physics, Faculty of Art and Sciences, 01330 Balcali Saricam, Adana, Turkey

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Abstract: In the present study, lithium (Li) doped magnesium oxide (MgO) samples were prepared using Solution Combustion Synthesis (SCS) and Sol-Gel (SG) methods. Their photoluminescence (PL) and thermoluminescence (TL) behaviors were determined after the different calcination temperatures. The aim of this study is to investigate the effect of different calcination temperatures on the PL and TL sensitivities of lithium doped MgO (MgO:Li) samples in pellet and powder forms prepared by SCS and SG methods. The structural characterization analysis of MgO:Li powder and pellets were carried out using X-ray diffraction (XRD) and scanning electron microscope (SEM) methods. The results of these structure analysis showed that MgO:Li samples have different crystal properties when changing calcination temperatures were applied during the preparation of the samples. Luminescence properties of the MgO samples which were synthesized at different calcination temperatures were investigated by the Photoluminescence (PL) and TL techniques. The maximum TL intensity of the samples was obtained at a calcination temperature of 800 and 1000 oC for the SCS and SG methods, respectively. Uncontrolled chromium impurities were observed in MgO samples by using PL measurements. On the PL spectrum, peaks indicating chromium (III) (Cr3+) transmission in the red portion of the spectrum at 672, 698 and 721 nm are clearly evident. We investigated high dose sensitivity in these samples. This study presents optimum calcination temperatures in order to obtain maximum PL and TL sensitivity of MgO sample. It is also clear that it will contribute to the literature that it can be studied as a new dosimetric material.

1. Introduction

Magnesium oxide (MgO) is a wide band-gap (E_g=7.8 eV) insulator under ambient pressure. MgO has high chemical and thermal stability, and high surface reactivity. These properties make it a promising material for application in sensors, catalysis, paints, and additives (Shukla et al., 2004). MgO has a high melting point of about 2800 ^oC. Its density is about 3.58 g/cm^3 and the Mg ions occupy the octahedral sites within the anion close-packed structure (Klein & Hurlbut Jr, 1999).

MgO has long been accepted as a luminescence dosimetry material, mainly for use with the thermoluminescence (TL) technique. In order to increase the number of materials suitable for optically stimulated luminescence (OSL) dosimetry, many researchers have investigated the luminescence characteristics of this material (Bos, Prokić, &

*İlgili yazar: veysiguckan@gmail.com ORCID: 0000-0001-7693-7770

Brouwer, 2006; Oliveira, Doull, & Yukihara, 2013; Oliveira, Milliken, & Yukihara, 2013). There are many reports of MgO doped with lanthanides or transitions metal ions to increase the TL or OSL signals and to improve its dosimetric characteristics (Oliveira, Doull, et al., 2013; Oliveira, Yukihara, & Baffa, 2019; Yukihara et al., 2013).

In this study, the effect of calcination conditions on TL and OSL intensities of MgO phosphors produced by the SCS and SG methods were presented. Li-ion was doped into the MgO phosphors during the synthesis process as dopants and different calcination temperatures and times were performed to obtain high sensitivity MgO luminophors that can be for TL and CW-OSL dosimeters. The structure and morphology of the synthesized materials were checked by XRD and SEM methods. The TL and OSL curves of Li doped MgO phosphors calcinated at different temperatures and times were compared with each other.

2. Materials and methods

Material preparation

This study presents the calcination effect on MgO samples in pellet form prepared by SCS and SG methods. All chemicals were supplied from Sigma Aldrich. Li was used as the dopant with 0.1% mol for both methods. The calcination temperatures and times used in both production methods are 800, 900, 1000, 1100 and 1200 ^{o}C for 2, 4, 6, 8, 10 and 24 hours. After the bulk material was produced, the material was divided into 5 equal parts and then calcined at different temperatures, keeping the time constant for 4 h. In this work, MgO samples were studied in pellet form for having more settled PL and TL signals and for easy handling. MgO pellets were performed as a 6 mm diameter and 0.6 mm thickness as a shape of disk form using 25 milligrams calcinated powders. In all TL measurements, readings were taken after the sintering of all types of pellets at 1600 oC for 4h. The calcination temperature giving the best TL signal was selected and the time experiment was performed (It is not given). The same process was repeated for the time experiment.

Characterization

A few characterization analyses of MgO samples obtained by SCS and SG methods were performed. All of the analysis was performed at the room temperature. The phase evaluation and crystal structure characteristics of pellet-shaped MgO samples were studied by means of the XRD technique. The measurements were performed using XRD PANalytical EMPYREAN branded diffractometer equipped a copper and cobalt X-ray tube, and copper Kα radiation wavelength was 0.1541 nm. Diffraction angles were adjusted from 20° to 90° (scan mode, Δ2θ=0.02°). Qualitative phase analyzes were performed by comparison of the experimental diffraction patterns with the standard ones from the International Centre for Diffraction Data (ICDD). The surface morphology of the samples and microstructures of MgO pellets were examined using a FEI branded Quanta 650 model field-emission SEM with 30–100 kV accelerating voltage and 100nA probe current.

Luminescence measurements

PL measurements were performed using a monochromatized xenon lamp as the excitation source. All PL spectra were obtained at room temperature. The PL and PLE spectra were measured using a Horiba/Jobin-Yvon Fluorolog-3 spectrofluorimeter. It has a continuous xenon lamp (450 W) and a photomultiplier tube (Hamamatsu R928P). The measured PLE spectra were corrected by the xenon lamp emission spectrum.

TL measurements were carried out using a Risø TL/OSL reader model DA-20 (Risø National Laboratory, Denmark). Luminescence emission were measured in the VIS region using a Schott BG-39 filter (300–700 nm) and UV region using a bandpass filter (Hoya, U-340, transmittance range from 250 to 390 nm, max 340 nm) in front of the PM tube. In order to the detection of light, a bialkali photomultiplier tube (PMT) was used (model 9235QB, Electron Tubes Ltd., Uxbridge, UK). Samples were irradiated at room temperature using an in situ 90Sr/90Y beta source. This ionizing irradiation source emits beta particles with a maximum energy of 2.27 MeV. All TL measurements were performed at a heating rate of 5 ^{o}C/s.

3. Results

Crystallographic characterization and morphology of the samples were studied by XRD and SEM. The XRD pattern of Li doped MgO pellets are given in Fig. 1. Fig. 1 shows that the pattern matched well with ICDD 98-064-2714 in terms of peak positions indicating the formation of single phase. It has cubic structure with lattice parameters of a =b=c=4.2080 Â. XRD spectra of all MgO:Li pellets having the diffraction peaks (111), (200), (220), (311) and (222) located at 2theta = 36.9°, 42.9°, 62.3°, 74.7°and 78.7°, respectively. It was observed that the different calcination temperatures applied did not cause any difference in the phase in the crystallographic structure.

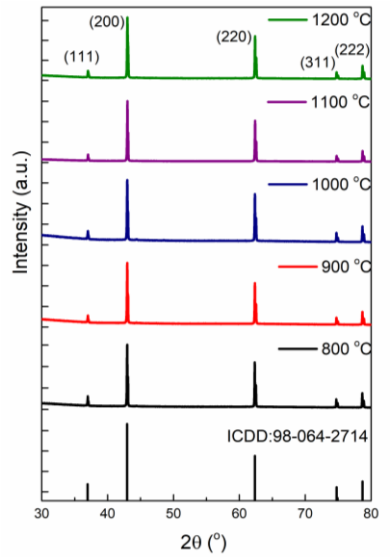

Figure 1. XRD analysis of MgO:Li pellets calcinated at different temperatures.

The SEM images revealed the morphological characteristics of Li doped MgO pellets produced by sol-gel methods. The SEM images of produced samples are shown in Fig. 2. There is homogeneous distribution among particles for all the MgO:Li pellets. These images do not clearly show us how the calcination temperature creates differences in the morphology of the MgO pellets. It needs further studies.

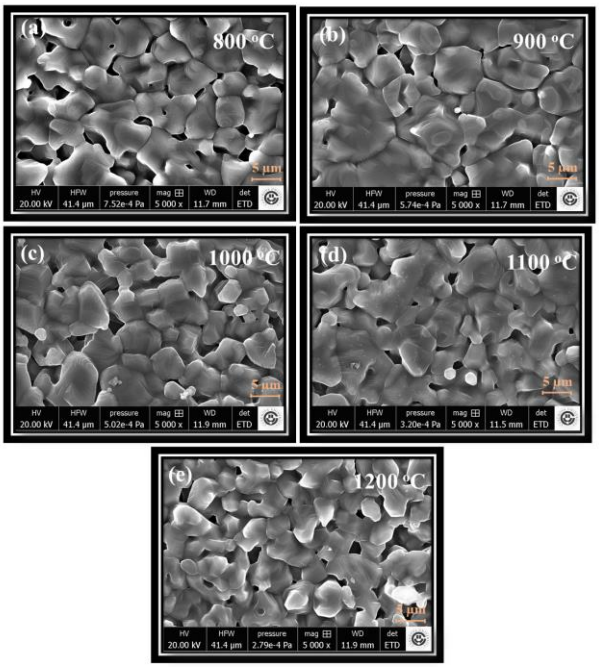

Figure 2. The typical SEM images of MgO:Li pellets calcinated at; (a) 800 °C, (b) 900 °C, (c) 1000 °C, (d)1100 °C and (e) 1200 °C for 4 h

The room temperature photoluminescence emission spectrum of MgO:Li pellets prepared by combustion and sol-gel methods excited with a wavelength of 330 nm were shown in Fig. 3. MgO:Li pellets prepared using the SCS method have a broad emission peak centered at ~660 nm (1.88 eV, orange region). The pellets produced using the sol-gel method have both a relatively narrow peak at 660 nm and a peak at 720 nm in some of the samples. Interestingly, uncontrolled chromium impurities were observed in MgO samples. When the samples excited with 440 nm, peaks indicating chromium (III) (Cr^{3+}) transmission in the red portion of the spectrum at 672, 698 and 721 nm are clearly evident (see Fig. 4) [1, 2].

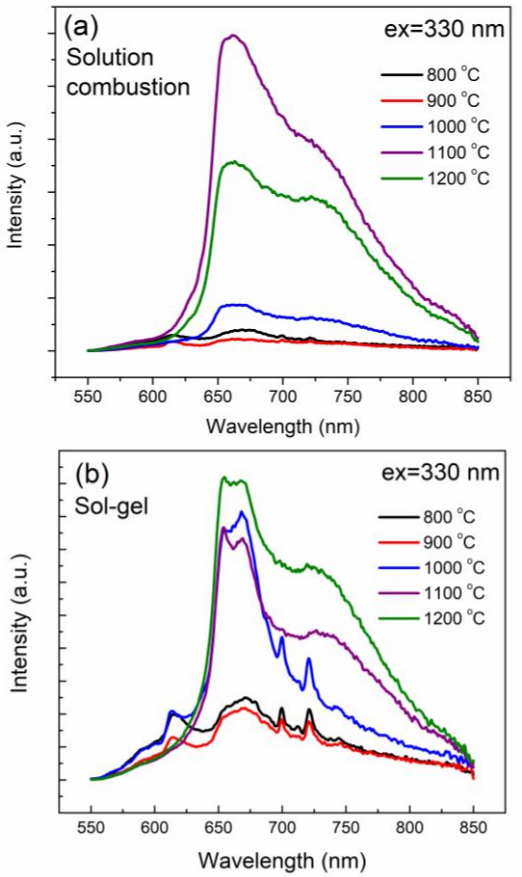

Figure 3. The PL spectra of the MgO:Li pellets

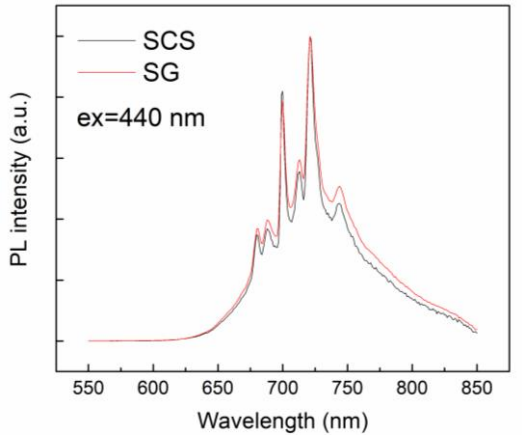

Figure 4. The PL spectra of the MgO:Li pellets under 440 nm excitation.

Fig. 5 shows the TL glow curves from MgO:Li pellets after 2 Gy beta irradiation in comparison with

calcinated samples at different temperatures. TL readouts were performed by heating each sample from room temperature up to 450 °C with a heating rate of 5 °C/s. As is seen in Fig. 4a, maximum trapped charge populations were obtained from MgO:Li pellets prepared using SCS method with calcination temperature at 800 °C for 4 h. Fig. 4b shows that the maximum intensities of MgO:Li pellets were recorded from samples calcinated at 1000 °C for 4 h.

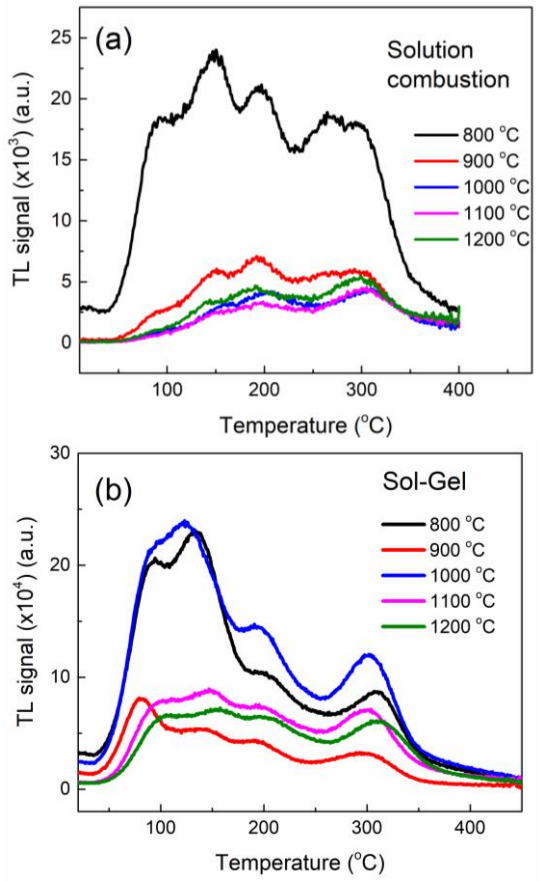

Figure 5. The TL glow curves from MgO:Li pellets prepared using (a) solution combustion method and (b) sol-gel method calcinated at 800 °C, 900 °C, 1000 °C, 1100 °C and 1200 °C for 4 h.

4. Conclusion

Li doped MgO pellets produced using solution combustion synthesis and sol-gel methods were verified by XRD measurements. MgO samples did not contain different phases were highly compatible with the reference number ICDD 98-064-2714 in terms of peak positions. SEM images showed that all samples were homogeneous with no hollow structure between the grains. According to TL measurements, for the Li doped MgO samples prepared using SCS and SG methods, the maximum trapped charge populations were obtained from MgO:Li pellets with calcination conditions of 800 °C. and the maximum intensities of MgO:Li pellets were recorded from samples calcinated at 1000 °C, respectively. However, it is evident that the trap structures formed in the material could not be formed at high energy levels by the dopants. Therefore, TL peaks were obtained at temperatures lower than desired. It was observed that the change in calcination temperature did not affect the TL glow curve. It is clear that the effect of calcination temperature on TL signals needs further study.

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