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Characterization and imaging performance of nanoscintillator screen for high resolution X-ray imaging detectors

Bo Kyung Cha [a], Seung Jun Lee [b], P. Muralidharan [b], Jon Yul Kim [a], Do Kyung Kim [b], Gyuseong Cho [a,*]

[a] Department of Nuclear and Quantum Engineering, Korea Advanced Institute of Science and Technology, Daejoen, Repulic of Korea
[b] Department of Materials Science and Engineering, Korea Advanced Institute of Science and Technology, Daejoen, Repulic of Korea

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ABSTRACT

We have synthesized europium-doped gadolinium oxide (Gd_2O_3:Eu) scintillators by a precipitation method with advantages of simple process and low-temperature calcinations for high resolution X-ray imaging detectors. The powders with about 15–30 nm particle sizes were obtained by subsequent calcinations of the precursor at a different temperature of 600–800 °C for 10 h. The wavelength of the main emission peak was about 610 nm and as temperature increased, light intensity of the scintillator increased in the X-ray luminescence case. Imaging performance such as X-ray linearity and spatial resolution of both commercial bulk and nanocrystalline-Gd_2O_3:Eu scintillator screens was measured and compared with the X-ray imaging system after coupling these to a CCD image sensor.

1. Introduction

In digital radiography system, an indirect detection method is usually used with a scintillator (or X-ray converter), which converts X-ray to visible light, and an imaging device, including CMOS, CCD and amorphous Si flat panel detector. Scintillators with high light output and spatial resolution are still needed for more detailed inspections such as in mammography, dental and micro-CT applications. Uptil now, both Gd_2O_2S:Tb(Gadox) and CsI:Tl scintillation materials are widely used in indirect digital X-ray imaging detectors [1]. However, because the Gadox scintillator screens of granular type with average 5–10 μm size, the light photons generated by incident X-ray flux are scattered and spatial resolution deteriorates in the final X-ray images. It is reported that high spatial resolution can be achieved by a scintillator with nanocrystalline particles without the scattering of light photons [2,3]. In this work, commercial bulk Gd_2O_3:Eu and our fabricated screens with nanocrystalline particles were used with an image sensor for X-ray imaging performance measurements such as those of light intensity, linearity of X-ray to light and spatial resolution.

2. Experimental procedure

A simple precipitation process was carried out using diethanolamine (DEA) as a precipitant to prepare a 5 mol% Eu-doped Gd_2O_3 powder with nanoparticles. In this procedure, $Eu(NO_3)_3 6H_2O$ and $Gd(NO_3)_3 6H_2O$ were dissolved in absolute ethanol with continuous stirring to form a clear homogeneous solution, 0.25 mol DEA was added to the above solution with continuous stirring and the solution was precipitated at room temperature (RT). A small amount of DI water was added to the precipitate and was allowed to stand for a few hours to ensure complete precipitation and then washed systematically with DI water and ethanol by centrifugation. The obtained white powder was dried at 60 °C for 12 h. A subsequent heat treatment was implemented from 600 to 800 °C for 10 h in an electric furnace [4]. Gd_2O_3:Eu scintillator screens on a silicon substrate were fabricated using the screen printing method by means of mixing and thermal hardening of both commercial bulk (UK63/S-R1, Phosphor Technology, UK) and our synthesized powder, binders such as organic solutions and dispersion agent [5]. Microstructures of as-synthesized and calcinated powders were observed by FE-SEM and high resolution TEM. The crystal structures of our samples were investigated by high resolution X-ray diffraction(RIGAKU Ultima IV) with an analysis 2θ range 20–90°. X-ray imaging performances such as light response of X-ray to light, light intensity and spatial resolution were measured using an optically coupled CCD camera with 43 μm effective pixel size and X-ray source with 50 kV_p peak acceleration voltage and different beam current (LISTEM, BRS-2).

3. Results and discussion

XRD results of nanocrystalline Gd_2O_3:Eu powder synthesized through a precipitation process at different calcination temperatures for 10 h are presented in Fig. 1. The X-ray peaks at (2 1 1), (2 2 2),

* Corresponding author. Tel.: +82 42 350 3821; fax: +82 42 350 5861.
E-mail address: gscho@kaist.ac.kr (G. Cho).

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(4 0 0), (4 4 0) and (6 2 2) are in good agreement with cubic crystal structures of Gd_2O_3 (JCPDF cards 00-012-1797). As the calcination temperature increased, the diffraction width was decreased and showed sharper pattern peaks. However, the X-ray diffraction pattern of commercial Gd_2O_3:Eu powders with microcrystalline particles showed monoclinic phase (JCPDF cards 00-043-1015). The average particle size of our synthesized samples was measured to be 15–30 nm using the diffraction peak full-width at half-maximum (FWHM) of the Scherrer equation. Particle size and luminescent intensity of the fabricated powders were largely affected by calcination temperature. The highest luminescent intensity was observed for the Gd_2O_3:Eu powder with average 30 nm particle size, which was heat-treated at 800 °C temperature for 10 h.

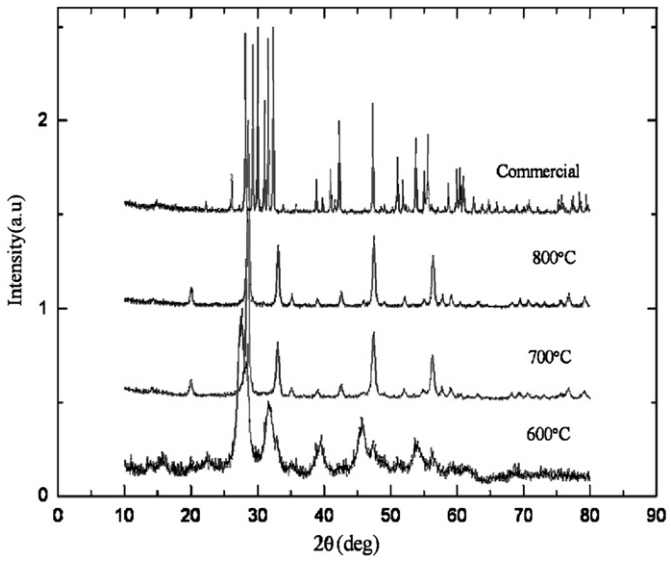

Fig. 1. XRD patterns of Gd_2O_3:Eu nanocrystals at different calcination temperatures.

The morphology and particle size of the commercial bulk and nanocrystalline-Gd_2O_3:Eu powders calcinated at 800 °C temperature are showed in Fig. 2 by SEM and TEM images. The nanocrystalline-Gd_2O_3:Eu powder of spherical shape with average 30 nm size is agglomerated and the commercial bulk powder with average 5 μm particle size is observed in Fig. 2. The above mentioned Gd_2O_3:Eu scintillator powders with bulk and nanoparticle sizes were fabricated as a screen type on a silicon substrate through the screen printing method.

Gd_2O_3:Eu scintillator screens of size $3 \times 3\ cm^2$ and 145 μm thickness were prepared and optically coupled with a CCD image sensor for X-ray imaging performance measurement. Light intensity and X-ray response were acquired by measuring averaged pixel value over the region of interest (ROI) in X-ray image areas as a function of X-ray exposure doses. Light intensity of the Gd_2O_3:Eu scintillator screen with 30 nm particle size was lower than with commercial bulk size. And the light intensity of

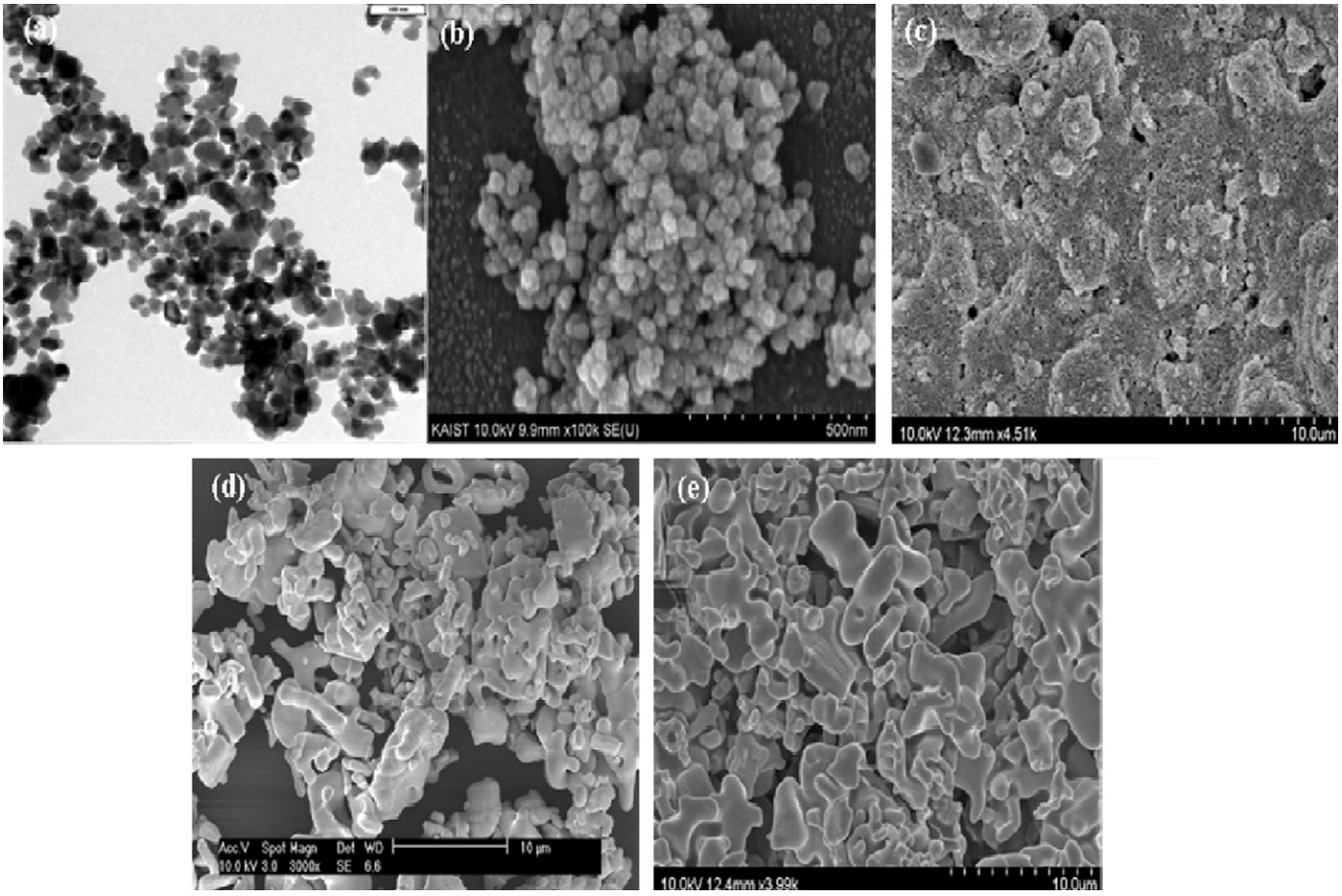

Fig. 2. (a) TEM and (b) SEM images of nanocrystalline-Gd_2O_3:Eu powder, (c) SEM image of nanocrystalline-Gd_2O_3:Eu screen type, SEM images of commercial bulk powder (d) and screen type (e).

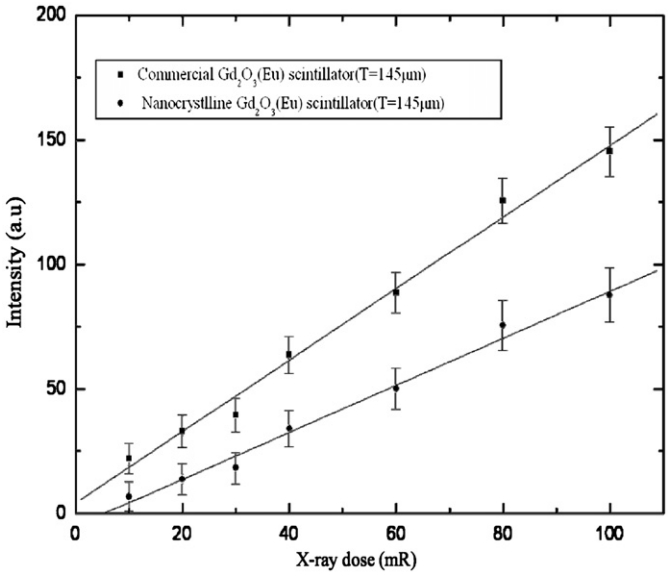

Fig. 3. X-ray linearity of the fabricated Gd_2O_3:Eu scintillator screens.

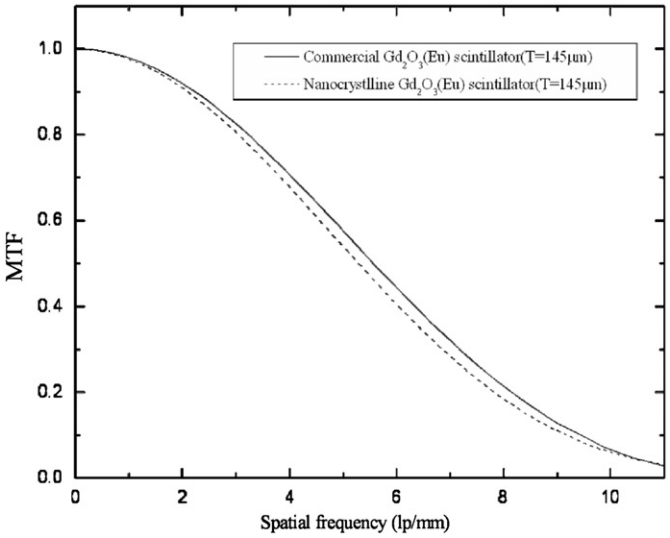

Fig. 4. MTF curves of the fabricated Gd_2O_3:Eu scintillator screens.

both samples was linearly increased as the X-ray exposure dose increased as shown in Fig. 3. The edge method with a tungsten edge phantom (IEC6220-1 standard) was used to measure the modulation transfer function (MTF) for spatial resolution evaluation of the prepared Gd_2O_3:Eu scintillator screens [6]. Spatial resolution of the Gd_2O_3:Eu screen with nanocrystalline particles was lower than that with micron-sized particles in Fig. 4 against expectation. high spatial resolution was expected to achieve because of negligible light scattering by particles of smaller size than the emission wavelength. However, the spatial resolution of the Gd_2O_3:Eu scintillator with nanocrystalline particles due to still existing light spreading could not be improved. However, more research on the resolution of nano scintillators will be undertaken.

4. Conclusion

In this work, a Eu-doped Gd_2O_3 scintillator with nanocrystalline particles was successfully synthesized through precipitation and subsequent calcination process for X-ray imaging detector application. The Gd_2O_3:Eu scintillator calcinated at 800°C temperature for 10 h showed average 30 nm particle size and cubic phase from SEM and XRD results. Its X-ray imaging performance was measured and compared by coupling the prepared Gd_2O_3:Eu scintillator screen with a CCD image sensor. The light intensity and spatial resolution of our nanocrytalline-Gd_2O_3:Eu scintillator were lower than those with commercial micron-size particles. However, we will continue study a proper experimental condition with different calcination temperatures and times through the precipitation method in order to achieve nanocrytalline-Gd_2O_3:Eu scintillators with high light output and spatial resolution for indirect X-ray imaging detector.

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