Investigation of the luminescence, crystallographic and spatial resolution properties of LSO:Tb scintillating layers used for X-ray imaging applications.

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# Abstract

In this work, a group of Lu<sub>2</sub>SiO<sub>5</sub>:Tb (LSO:Tb) scintillating layers with a Tb concentration between 8% and 19% were investigated by means of synchrotron and laboratory techniques. The scintillation efficiency measurements proved that the highest light yield is obtained for a Tb concentration equal to 15%. At higher concentration, quenching processes occur which lower the light emission. The analysis of the reciprocal space maps of the (082) (280) and (040) Bragg reflections showed that LSO:Tb epilayers are well adapted on YbSO substrates for all the investigated concentrations. The spatial resolution tests demonstrated the possibility to achieve a resolution of 1  $\mu$ m with a 6  $\mu$ m thick scintillating layer.

# Introduction

Thanks to their high brilliance and coherence, third generation of synchrotron light sources offer new possibilities for different X-ray imaging techniques. These

techniques, including microtomography with absorption or phase contrast and holotomography [Nugent et al. 1996, Cloetens et al. 1996, Cloetens et al. 1999, Snigirev et al. 1995], demand for X-ray detectors with a spatial resolution in the micrometer and even submicrometer range. In addition, micromotomography calls for fast working detectors due to the necessity to collect quickly and efficiently several hundred images for a single tomogram. X-ray energies are typically between 5-20 keV for absorption radiography and up to 60 keV in phase contrast imaging [Koch et al. 1998, Koch et al. 1999, Martin et al. 2006, Bonse et al. 1996, Stampanoni et al. 2002].

A successful approach to achieve X-ray imaging with submicrometer resolution is based on the use of a scintillator combined with diffraction limited microscope objectives [Koch et al. 1998]. The crucial elements for the spatial resolution of this kind of detector are the resolution power of the optics and the luminescent screen. The resolution power of the visible light coupling limits the spatial resolution to a value of 0.4 µm [Born and Wolf 1999]. Luminescence screens may be either based on a powder phosphor or on optically transparent, i.e. no scattering single crystal scintillators. The spatial resolution achievable with powdered scintillators is limited to  $2-3 \mu m$  due to the scattering of the visible light photons from the scintillator powder grains. On the contrary, it is possible to achieve micrometer and submicrometer resolution by using scintillating single crystals [Martin et al. 2006]. A comparison of the spatial resolution performance achievable with powder and crystal scintillators is shown in Figure 1. A Gadox (Gd<sub>2</sub>O<sub>2</sub>S:Tb) powder and a LSO:Tb scintillators were integrated in an indirect X-ray imaging detector composed of a microscope optics (4x objective/NA=0.16 and 2.5 eyepiece) and a SensicamQE CCD detector. The effective pixel size was equal to 0.64  $\mu$ m. The spatial resolution of the two scintillators was compared by acquiring the X-ray images of an X-radia standard calibration pattern X500-200-30 at an X-ray energy of 12 keV at the beamline ID06 of ESRF (Grenoble, France). The X-ray image recorded with the crystal scintillator is sharper than the image acquired with the phosphor powder, proving the better performance of the scintillating crystal.

Currently, Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>:Ce (YAG) and Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>:Ce/Eu (LuAG) single crystal scintillators are used as radiation detectors in X-ray microradiography with either

synchrotron sources or micro-focus X-ray tubes [Tous et al. 2010]. These scintillators are becoming less and less attractive for several reasons: LuAG scintillators are characterized by a low light yield and YAG crystals have low absorption efficiency due to their low density and atomic number [Crytur, 2013]. To overcome those limitations, new scintillating materials need to be developed with higher light yield and higher absorption efficiency than the scintillators currently used in synchrotron X-ray imaging applications. The Lu<sub>2</sub>SiO<sub>5</sub> oxyorthosilicate (LSO) is a suitable candidate for that because of its high density and atomic number that reverberate in higher absorption efficiency than LuAG and YAG. In Figure 2 the LSO absorption coefficient measurements are compared to the theoretical curves of LSO, YAG and LuAG [Martin et al. 2009]. As it can be seen, the experimental results are closely matched to the theoretical LSO absorption efficiency and LSO absorption efficiency is significantly higher than the ones of the other scintillators. The comparative study of LSO:Tb YAG:Ce and LuAG:Ce afterglow is described in [Douissard et al. 2010]. Results demonstrated that in the case of YAG:Ce and LuAG:Ce, the phosphorescence limits the applicability of the scintillators to a dynamic range to 8 - 10 bit. In contrast to this, LSO:Tb can be used with a 16-bit CCD camera and has an excellent fast decay of the phosphorescence component.

In this work a set of LSO:Tb scintillating layers were considered having a Tb concentration between 8% and  $19\%^1$  and a thickness between 3 µm and 9 µm [Douissard et al. 2010, Cecilia et al. 2011]. The LSO:Tb scintillation and luminescence properties were investigated by means of laboratory techniques for each layer composition. The lattice mismatch between the LSO:Tb and YbSO substrate and the spatial resolution tests were accomplished by X-ray synchrotron radiation techniques.

## Samples and experimental set-up

### a) Samples

The LSO:Tb scintillating crystals studied in this work were deposited by liquid phase epitaxy (LPE) growth on YbSO substrates with [010] oriented surface. The details of

<sup>&</sup>lt;sup>1</sup> The percentage refers to the weight fraction Tb/(Tb+Lu)

the growth procedures can be found in [Douissard et al. 2010]. The thickness and Tb dopant content of all the samples are summarised in Table I.

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Tb concentration	substrate (µm)	Layer (µm)
(%)		
8	500	8.75
12	500	9.13
14	500	2.66
15	500	7.00
19	500	3.41

Table I: LSO/YbSO samples

# *b) Scintillation efficiency*

The scintillation efficiency of the LSO:Tb layers was measured in the laboratory of the detector group at the European light source ESRF (Grenoble, France). The LSO:Tb scintillation light was excited with a steady state X-ray source (operated at 20 kV, 40 mA with a Cu anode). A 25  $\mu$ m Cu X-ray absorption filter was used to select the monochromatic 8 keV emission line of the copper anode. The scintillation light was collected by a PCO SVGA Sensicam CCD camera coupled to a microscope optics (4x objective, 0.16 NA, 2x eyepiece) and for each sample, a CCD image was acquired with an integration time of 45 s.

The intrinsic scintillation efficiency was calculated in two steps:

1) for each sample, the mean value of the image counts was normalised to the layer absorption efficiency at 8 keV;

2) the calculated value was compared to the mean value of the counts detected with a calibrated YAG scintillator having a light yield of 35 ph/keV.

#### c) Photoluminescence

The LSO:Tb photoluminescence measurements were performed in the laboratory of the Institute of Physics, Academy of Science of Prague.

The experimental setup for the emission/excitation measurements is composed of a steady-state deuterium lamp, two monochromators and a Schimadtzu photomultiplier (working range 200-800 nm). The emission spectrum is recorded with the excitation

light impinging the sample at  $45^{\circ}$  with respect to the surface and the emission light detected at  $45^{\circ}$ . During each measurement, the excitation wavelength is kept fixed and the emission wavelength is varied with the monochromator.

The equipment for the decay time measurement includes a microsecond pulsed xenon lamp (Horiba Jobin Yvon) two monochromators and the Schimadtzu photomultiplier mentioned above. The scintillation light excited with the xenon lamp is detected with the photomultiplier. The photomultiplier signal is then sent to a timer followed by a 1024 channels multichannel scaler.

## d) High resolution synchrotron diffraction

The crystallographic adaptation of the LSO:Tb layer on the YbSO substrate was investigated by high resolution synchrotron diffraction. The measurements were carried out at the SCD beamline of the ANKA synchrotron (Karlsruhe Institute of Technology) using a 6-circle diffractometer. The energy was set to 10 keV X-ray. The unit cell axes (a, b, c) and the monoclinic angle ( $\beta$ ) of the YbSO and of the undoped LSO are listed in Table II [Cong et al. 2009]. The lattice mismatch between the LSO layer and YbSO substrate was calculated along the a, b and c crystallographic axes by measuring three coplanar Bragg diffractions (040), (280) and (082).

YbSO	LSO
a= 12.38 Å	a= 12.36 Å
b=6.666 Å	b=6.644 Å
c=10.28 Å	c=10.25 Å
β=102.54°	β=102.4°

Table II: Unit cell settings of the monoclinic LSO and YbSO

## e) Spatial resolution measurements

The spatial resolution measurements were performed at the ANKA/TopoTomo beamline [Rack et al, 2009] by using a high resolution detector composed of a CCD camera pco.4000 and a monochromatic microscope from OptiquePeter [Optique Peter, 2013]. The total magnification was set to 25x (10x objective/ NA=0.4 in combination with a 2.5x eyepiece) resulting in an effective pixel size of 0.36 µm. The investigated LSO:Tb scintillators contain a Tb concentration equal to 15%, that

corresponds to the highest light production, as it will be discussed in the next section. The scintillating layers with thickness between 6  $\mu$ m and 74  $\mu$ m were integrated in the high resolution detector and for each of them the Edge Spread Function (ESF) [Zhu et al. 1995] was measured by positioning a GaAs wafer edge in direct contact with the scintillator. The edge images were acquired at an X-ray energy of 12 keV and the Modulation Transfer Function (MTF) was calculated by using the ImageJ software plugin "*Slanted Edge MTF*" [ImageJ 2013].

#### **Experimental results and discussion**

### *a) Scintillation efficiency*

The scintillation efficiency of the LSO:Tb scintillators is reported in Figure 3 for all the investigated compositions. The light production increases between 10 ph/keV and 45 ph/keV where it is maximum. At higher Tb content, the scintillation efficiency decreases again.

## *b) Photoluminescence*

The photoluminescence spectra of the LSO:Tb scintillators were excited at a wavelength equal to 236 nm within the 4f-5d absorption band of Tb<sup>3+</sup> centre. The spectra of the samples are characterised by four lines peaking at 488 nm, 542 nm, 584 nm and 620 nm due to the radiative transition of Tb<sup>3+</sup> from the <sup>5</sup>D<sub>4</sub> excited state to the ground <sup>7</sup>F<sub>x</sub> multiplet (Figure 4). The LSO scintillation spectrum is centred around 540 nm, matching the quantum efficiency of most of the CCD detectors currently in use in X-ray imaging experiments. The emissions from the higher lying level <sup>5</sup>D<sub>3</sub> (in the spectral region between 370 nm and 480 nm) are quenched due to cross-relaxation processes. In the enlarged view of the <sup>5</sup>D<sub>3</sub>  $\rightarrow$  <sup>7</sup>F<sub>x</sub> luminescence emission (Figure 4, inset), an unknown weak broad band can be observed between 320 nm and 340 nm. A possible explanation for this band could be the presence of lead related centres in the crystal matrix that can be formed due to the use of PbO:B<sub>2</sub>O<sub>3</sub> flux during the LPE growth [Gorbenko et al., 2012].

The decay of the LSO:Tb scintillators was measured at the emission wavelength equal to 542 nm, that corresponds to the most intense peak in the photoluminescence spectrum. The photoluminescence decay turned out to be perfectly single exponential

for all the investigated Tb concentrations and the decay time constant was typically of the order of few ms (Figure 5).

The dependence of the decay time constant on Tb concentration is shown in Figure 6. As it can be seen, it increases slightly from 1.78 ms and 1.88 ms for a Tb concentration between 8% and 14%. At higher Tb dopant content the decay time decreases again.

The observed increase of Tb time decay can be explained by considering a weak radiation trapping effect, which has been extensively studied for rare earth ions whose electronic structure is very similar to Tb ( $Yb^{3+}$ ,  $Er^{3+}$  and  $Ho^{3+}$ ). This process consists in the re-absorption and re-emission of the ion fluorescence, caused by the superposition of the absorption and emission bands [Auzel et al. 2003, Ehrmann et al. 2002, Balaji et al. 2012].

At Tb concentrations higher than 15 % the decay time shortens. Such behaviour typically appoints to a concentration quenching process. When the luminescent ions become closer to each other below a critical distance, the excitation energy starts to migrate among them and becomes progressively lost at unspecified non radiative traps (lattice defects) [Nikl et al. 2003].

### c) High resolution diffraction measurements

The reciprocal space maps (RSM) of the (082) Bragg reflections are shown in Figure 6 for the the LSO:Tb doped with a Tb concentration equal to 8%, 12%, 15% and 19%. The RSM of the (280) and (040) Bragg reflections were also measured and they turned out to be qualitatively very similar to the RSMs of the (082) Bragg reflections shown in Figure 7. For a better visibility of the peaks, the diffraction intensity is displayed using the same logarithmic scale for all the samples. In each map, the YbSO substrate diffraction peak was marked as (1) and the diffraction peak of the LSO:Tb layer was indicated as (2).

As it can be seen, the intensity of the YbSO diffraction peak is 2-3 orders of magnitude lower than the diffraction peak intensity of the LSO:Tb layer due to the attenuation of the X-rays in the LSO layer.

The diffraction intensity distribution of the YbSO peaks indicates the presence of grains in the substrate region under investigation. A similar intensity distribution is

replicated in the LSO:Tb layer peaks, pointing out the propagation of the substrate crystal defects in the layer during the LPE growth.

The comparison of the relative position of the LSO:Tb and YbSO diffraction peaks in Figure 7 demonstrates that the diffraction peak of the LSO:Tb layer approaches the one of YbSO as the concentration of Tb increases. In the sample doped with the highest Tb concentration (19%) the diffraction peaks are no longer distinguishable.

The in-plane lattice mismatch was calculated from the RSM of the (082) (280) and (040) Bragg reflections. In a monoclinic crystal system, the intensity of the reciprocal space vector ( $|Q_{hkl}|$ ) detected at the reciprocal lattice point (hkl) is related to the length of the crystallographic axes a, b and c through the following relation [International Tables for crystallography, 2005]:

$$|Q_{hkl}|^{2} = \frac{h^{2}}{(a \cdot \sin \beta)^{2}} + \frac{k^{2}}{(b)^{2}} + \frac{l^{2}}{(c \cdot \sin \beta)^{2}} + 2\frac{hl}{ac(\sin \beta)^{2}} \cdot \cos(\pi - \beta)$$
(1)

By measuring  $|Q_{280}| |Q_{082}|$  and  $|Q_{280}|$ , and introducing the simplifying hypothesis that  $\beta$  is always constant for the diffraction Bragg reflections of the same sample, equation (1) has been solved for the a, b and c crystallographic axes of the LSO:Tb layer and of the YbSO substrate. From the knowledge of the LSO:Tb and YbSO crystallographic unit cell parameters, the in-plane lattice mismatches ( $\delta L_{//a}$  and  $\delta L_{//c}$ ) were calculated that are defined as the difference in the in-plane cell edges of the layer and the substrate [Baumbach et al., 2004].

The values of  $\delta L_{l/a}$  and  $\delta L_{l/c}$  are reported in Figure 8 and Figure 9 versus the concentration of Tb. Qualitatively, the dependence of  $\delta_{L/a}$  and of  $\delta_{L/c}$  on the Tb doping concentration is very similar. In both cases, the adaptation of the LSO:Tb layer on the YbSO substrate is very good for all the Tb concentrations.

The observed effect is likely due to the partial substitution of Lu ions (ionic radius = 0.085 nm) with the larger Tb ions (ionic radius = 0.0923 nm) in the LSO matrix. The higher ionic radius of Tb could favour the elongation of the LSO in-plane unit cell and thus the lattice matching between the YbSO and LSO:Tb.

The difference in in-plane crystallographic axes between LSO:Tb and YbSO is already compensated in the LSO:Tb sample with the lowest Tb concentration (8%). Consequently, an extension of the LSO:Tb unit cell is expected along the out-plane

direction as the content of Tb is increased between 8% and 19%. The vertical extension of LSO:Tb unit cell was quantified by using the following equation:

$$\delta_{L\perp} = \frac{b_{LSOTD} - b_{LSO}}{b_{LSO}} \tag{2}$$

Where,  $b_{LSO:Tb}$  is the interplanar distance of the scintillating LSO:Tb layer and  $b_{LSO}$  is the interplanar distance of the undoped LSO crystal. As shown in Figure 10, the vertical extension of the LSO:Tb unit cell increases at higher Tb concentration and it is highest (0.3%) in the sample containing the highest Tb concentration (19%).

e) *Spatial resolution measurements*: the MTF measurements are shown in Figure 11 for a group LSO:Tb crystals having thickness between 6  $\mu$ m and 74  $\mu$ m. The thickest LSO:Tb layer was grown up on a LYSO substrate to test the possibility to deposit LSO:Tb layers on commercial substrates. The comparison of the curves demonstrates that the best spatial resolution is achieved when the thinnest 6  $\mu$ m thick LSO:Tb scintillator is used. The spatial resolution was measured from the FWHM of the Line Spread Function (LSF) and was determined to be equal to 1  $\mu$ m. The contrast at 500 cycles/mm is equal to 20%.

The MTF was also measured at 24 keV for LSO:Tb and LuAG:Eu scintillating films both having a thickness of 10  $\mu$ m; the results are reported in Figure 12. The YAG substrate is known to emit intrinsic scintillation light, thus a FGL495 filter (Thorlabs) was used to cut this parasitic emission below 500 nm. Despite the use of the filter, the remaining scintillation from the substrate deteriorates the spatial resolution performance and induces a tail in the LSF (see inset Figure 12).

The benefit of the chosen YbSO substrate is proved by the decrease of the tail in the LSF of the LSO:Tb. The FWHM of the LSF for the LSO:Tb is equal to 3 pixels which corresponds to  $1.1 \mu m$ . The spatial resolution can be further improved by reducing the thickness of the scintillator at expense of efficiency.

### Conclusions

The aim of this work was investigating the scintillation, crystallographic and resolution performances of LSO:Tb scintillators for high spatial resolution X-ray imaging applications. A group of selected LSO:Tb samples with a Tb concentration

between 8% and 19% was studied by means of synchrotron and laboratory techniques.

In the Tb concentration range between 8% and 15%, the scintillation efficiency of LSO:Tb increases from 10 ph/keV to 45 ph/keV, due to the higher density of Tb luminescence centres available in the LSO matrix. The 45 ph/keV conversion efficiency of LSO:Tb is 1.3 and 3.7 times higher than the YAG and LuAG scintillators, respectively. This result is very promising, especially for fast applications. In fact, the absorption efficiency of a 5  $\mu$ m thick LSO crystal is 6 times higher than the efficiency of 5  $\mu$ m YAG:Ce and 1.5 times higher than the one of a 5  $\mu$ m LuAG:Ce crystal at an energy of 30 keV. The combination of the LSO:Tb higher absorption efficiency and higher light yield results in a better conversion efficiency of the detection system and thus allows reducing the measurement time.

At Tb concentration higher than 15%, the LSO:Tb scintillation efficiency decreases. The LSO:Tb photoluminescence spectra turn out to be very similar for all the investigated compositions and they are characterised by four emission lines at around 486 nm, 540 nm, 581 nm and 620 nm, that are typical of the  ${}^{5}D_{4} \rightarrow {}^{7}F_{x}$  Tb transitions. The photoluminescence decay of LSO:Tb was studied at the emission wavelength equal to 540 nm, where most of the energy deposited in the scintillator is reemitted in the form of light. The photoluminescence decay is perfectly exponential for all the investigated compositions. The decay time constant depends on the Tb concentration and it increases from 1.78 ms and 1.88 ms in the Tb concentration range between 8% and 15%, while it shortens to 1.72 ms at higher Tb content.

The observed increase of the time decay constant at Tb cocentration lower than 15% indicates the occurrence of a weak radiation trapping effect, while the shortening of the decay time at concentration higher than 15% proves the occurrence of a concentration quenching process that may also explain the decrease of the scintillation efficiency in the same Tb concentration range.

The synchrotron diffraction measurements show that LSO:Tb layers are well adapted on YbSO substrates for all the considered compositions, likely because of the elastic deformation of the LSO layer accomplished by the substitution of Lu ions (ionic radius equal to 0.085 nm) with the larger radius Tb ions (ionic radius equal to 0.0923 nm). The compensation of the lattice mismatch by means of elastic deformation

prevents the formation of misfit dislocations, that appear when the layer preserves its lattice parameters. The layer and the substrate are quite well adapted already in the sample with the lowest Tb concentration equal to 8%. The further increase of Tb elongates the LSO unit cell along the out-plane direction, leaving unchanged the in-plane crystal properties.

The spatial resolution tests performed on LSO:Tb showed that the highest achievable spatial resolution is equal to 1  $\mu$ m. The comparison of the LSO:Tb with a LuAG:Eu layer proves the superior performance of the LSO:Tb with respect to LuAG:Eu.

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## **Figure legends**

Figure 1: X-ray image of a X-radia test pattern recorded with a Gadox powder screen

with 5 µm thickness and with a LSO:Tb screen having a thickness equal to 48 µm.

Figure 2: Absorption efficiency of a 5  $\mu$ m thick scintillators vs. X-ray energy. The curves are calculated for LSO, LuAG, YAG. For LSO the theoretical values were verified experimentally [14].

Figure 3: Scintillation efficiency of LSO: Tb at different Tb dopant concentration.

Figure 4: Room temperature photoluminescence emission spectra of scintillating LSO, excited at  $\lambda exc = 236$  nm. Inset: enlarged section of  ${}^{5}D_{3} \rightarrow {}^{7}F_{x}$  emissions.

Figure 5: Photoluminescence decay curve of the 542 nm emission line of Tb for the LSO:Tb (8%)

Figure 6: Dependence of LSO: Tb decay time on Tb concentration.

Figure 7: RSM of LSO/YbSO reciprocal space maps (RSM) of the (082) Bragg reflections for Tb concentrations of 8%, 12%, 15%, and 19%. The peak positioned as (1) corresponds to the YbSO substrate and the peak marked as (2) is related to the LSO layer.

Figure 8:  $\delta L/a$  calculated from the RSM the (040), (280) and (082) Bragg reflections.

Figure 9:  $\delta L/c$  calculated calculated from the RSM the (040), (280) and (082) Bragg reflections.

Figure 10: Vertical extension  $\delta L \perp$  vs. Tb doping concentration, calculated from the diffraction of the (040) symmetrical plane.

Figure 11: MTF of LSO:Tb with thicknesses between 6  $\mu$ m and 74  $\mu$ m measured at 12 keV.

Figure 12: MTF measured at 24 keV on LSO:Tb and LuAG:Eu with thicknesses of 10µm each. The inset shows the LSF.