LPE grown LSO:Tb scintillator films for high resolution X-ray imaging applications at synchrotron light sources

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Abstract

Within the project Scin^{TAX} of the 6th framework program (FP6) of the European Commission (SCINTAX – STRP 033 427) we have developed a new thin single crystal scintillator for high resolution X-ray imaging. The scintillator is based on a Tb-doped Lu_2SiO_5 (LSO) film epitaxially grown on an adapted substrate. The high density, effective atomic number and light yield of the scintillating LSO significantly improves the efficiency of the X-ray imaging detectors currently used in synchrotron micro-imaging applications. In this work we present the characterization of the scintillating LSO films in terms of their spatial resolution performance and we provide two examples of high spatial and high temporal resolution applications.

PACS. 07.85.Qe; 29.40.Mc ; 87.57.Ce

1. Introduction

Third generation synchrotron light sources offer new possibilities for different X-ray imaging techniques thanks to their high brilliance and the high degree of spatial coherence. These techniques (e.g. microtomography with absorption or phase contrast and holotomography) demand highly efficient X-ray detectors with a spatial resolving power in the micrometer and even submicrometer range [1]. In addition, the increasing number of requests for X-ray imaging experiments at synchrotron light sources world wide demands for shortening the required time for a microtomography sequence to perform a larger number of experiments [2]. A successful approach for achieving X-ray imaging with submicrometer resolution is given by the combination of a transparent luminescent screen (single crystal scintillator) with diffraction limited microscope optics which magnify the luminescence image onto a CCD camera [3, 4]. In this context, both the spatial and the time resolution performances could be improved by increasing the absorption and the luminescence efficiencies of the scintillator material. Within a project of the 6th framework program (FP6) of the European Commission (SCINTAX – STRP 033 427) we have developed a new kind of thin single crystal scintillator for high resolution X-ray imaging. Our research is based on a $Lu_2SiO_5:Tb^{3+}$ (LSO:Tb) layer grown by Liquid Phase Epitaxy (LPE) technique on a suitable substrate [5]. The unique combination of the high density (7.4 g/cm^3) Z_{eff} (65.2) and light yield of this scintillator material significantly improves the efficiency of the X-ray imaging detectors currently used in synchrotron hard X-ray micro imaging applications. As reported in refs. [6, 7, 8]

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the light output of the LSO can be as high as the 130% of the YAG reference sample. This corresponds to an absolute light yield of about 52 ph/keV. The LSO:Tb scintillation spectrum is peaked around 535 nm which is efficiently detected by the front illuminated cameras commonly used in imaging experiments. The used substrate for the LPE growth of the LSO film is free from luminescence contribution, which otherwise may blur the image [9, 10]. Currently, the undesired luminescence by the substrate is the limiting factor for YAG:Ce, LAG:Eu and GGG:Eu scintillating films [11].

In this work we present the characterization of the scintillating LSO films in terms of their spatial resolution performance. In addition we provide two examples of high spatial resolution and high temporal resolution tomography of biological specimens. All the measurements described in this work were performed at the TopoTomo beamline of the ANKA synchrotron source (Karlsruhe, Germany) [12, 13].

2. Experimental set-up

The synchrotron light source ANKA (Angstroemquelle Karlsruhe) is located at the Karlsruhe Institute of Technology (K.I.T.) and is operated with a ring electron energy of 2.5 GeV and beam currents of 180-80 mA. The dipole bending magnets work with 1.5 T magnetic field, the resulting critical energy being E_c=6.2 keV. The ANKA/TopoTomo beamline can work either in white beam or in monocromatic mode. The available white beam energy spectrum ranges between 1.5 and 40 keV (flux at sample position ~10^{16} ph/s, 5 mm x 10 mm beam size at the position of the experiment). The monocromatic energy spectrum is provided by a double-multilayer monochromator (DMM, two coatings available: W/Si, Pd/B_4C) ranging between 6 and 40 keV with an energy resolution $\Delta E/E=10^{-2}$ [13].

The used detector for the spatial resolution measurements is composed of a pco.4000 CCD camera and a so-called *OptiquePeter* monochromatic microscope [14]. The total magnification was set to 25x (10x objective/ NA=0.4 plus a 2.5x eyepiece) with an effective pixel size of 0.36 μm. Several LSO scintillators with thickness between 6 and 74 μm were integrated in the high resolution detector and for each of them we have measured the Edge Spread Function (ESF) by positioning a GaAs edge in direct contact with the scintillator. The edge images were acquired with a photon X-ray energy of 12 keV and the Modulation Transfer Function (MTF) was calculated by using the ImageJ software plugin *"Slanted Edge MTF"* [15].

The exploited detector for the high spatial resolution tomography consists of an *Optique Peter* white beam microscope (10x objective, NA=0.28) which couples the pco.4000 camera to a LSO:Tb scintillating film with 12 μm thickness. The total magnification of the optical system is equal to 22.5x with an effective pixel size of 0.4 μm. The tomography scan was performed by using a filtered white beam (0.8 mm Be filter).

For accomplishing the fast tomography scan we have used a high speed Photron SA-1 CMOS camera, whose highest achievable data rate is equal to 5400 images/s in full frame mode. The camera is coupled to a 74-μm-thick LSO:Tb scintillator through an optical system designed at the Bundesanstalt für Materialforschung und-prüfung (BAM, Berlin, Germany) [13]. It is composed of a Nikon Nikkor 180/2.8 ED eyepiece (f = 180 mm) combined with a Rodenstock TV-Heliflex right-angle objective (f = 50 mm, max. NA = 0.43). The total magnification is equal to 3.6x, which yields an effective pixel size of 5.5 μm. During the tomography scan the sample is mounted on a fast rotary stage capable of creating synchronization pulses, which are used to trigger the image acquisition. The tomography scan was performed with a rotation of 450°/sec at 2000 images/s. Hence, the total acquisition time for a complete tomography scan (800 projections) is equal to 0.4 sec. The tomography scan was performed by using a filtered white beam (0.2 mm Al filter).

3. Results

Figure 1 shows the measured MTF for the a set of scintillating LSO:Tb crystals having thickness between 6 and 74 μm. As it can be seen, the best spatial resolution is achieved when the thinnest

LSO is used with thickness equal to 6 μm. The spatial resolution was measured from the FWHM of the Line Spread Function (LSF) and was determined to be equal to 1 μm.

To validate the performances of the developed LSO:Tb scintillators for high spatial and temporal resolution applications we have recorded the tomographic scans of two biological specimens. Due to their often hard and stiff exoskeleton many insects are difficult to examine by classical morphological methods. Though scanning electron microscopy provides high resolution, internal structures remain hidden. Light- and transmission electron microscopy require slicing the samples, a process drastically altering their condition. Moreover, insect cuticle is particularly difficult to slice and tends to fracture in the process.

As synchrotron-based hard X-ray microtomography allows non-destructive imaging of millimeter-sized objects, it became a powerful tool for entomologists. Virtual slices through the specimen allow taking a look at anatomical details. Additionally, the resulting data can be used to create digital 3D models very useful for morphological and anatomical research.

An example of a reconstructed slice is given in Figure 2a with some morphological details. The achieved spatial resolution was measured by using an X-radia test pattern (type X500-200-30): it was determined to be equal to 1.1 μm (Figure 2b) which is the resolution limit of the optical system.

As far as the fast application of the LSO:Tb scintillator is concerned, Figure 3 reports the tomographic reconstruction of a Peruphasma insect egg. Despite the used high frame rate there is no visible degradation of the image quality. The tomographic reconstruction clearly shows the micropylar plate, a characteristic feature of the eggs of stick insects (order Phasmatodea). This region surrounds the so-called micropyles, two tiny parallel canals traversing the egg shell to allow sperm entry.

4. Summary

In this work we have characterised the newly developed LSO:Tb whose unique combination of high density (7.4 g/cm^3) Zeff (65.2) and light yield (52 ph/keV) improves both the spatial and the time resolution performances of the detectors currently used in synchrotron applications. The value of the LSO:Tb scintillator performances was demonstrated by acquiring the tomographic scans of two biological specimens. The superior performances of the LSO scintillator allowed achieving the required contrast to identify the smallest features in the weak absorbing insects. In addition, it made possible to acquire a complete tomography scan within 0.4 s by using polychromatic synchrotron radiation while retaining at the same time excellent image quality.

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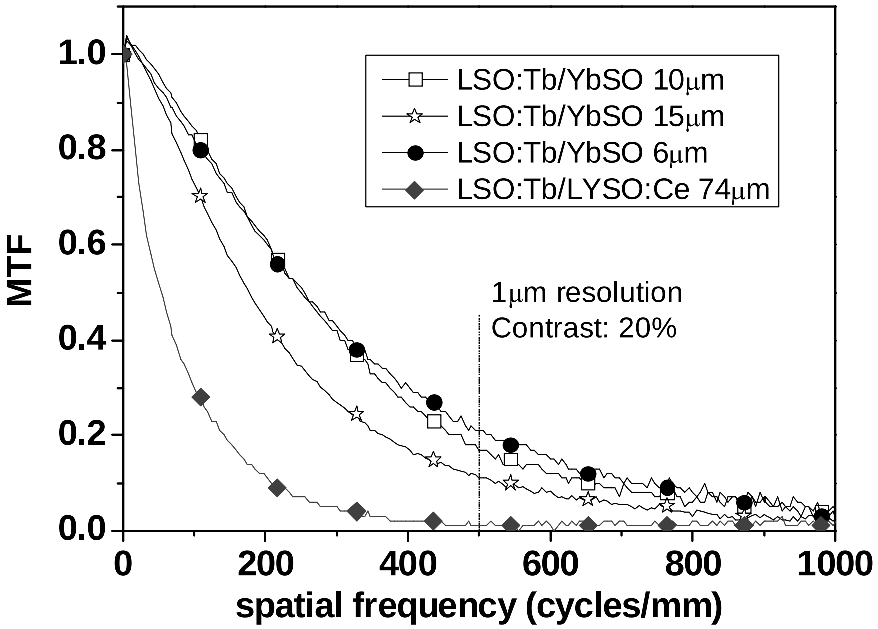

Figure1: MTF comparison for a set of scintillating LSO:Tb with thickness between 6 μm up to 74μm.

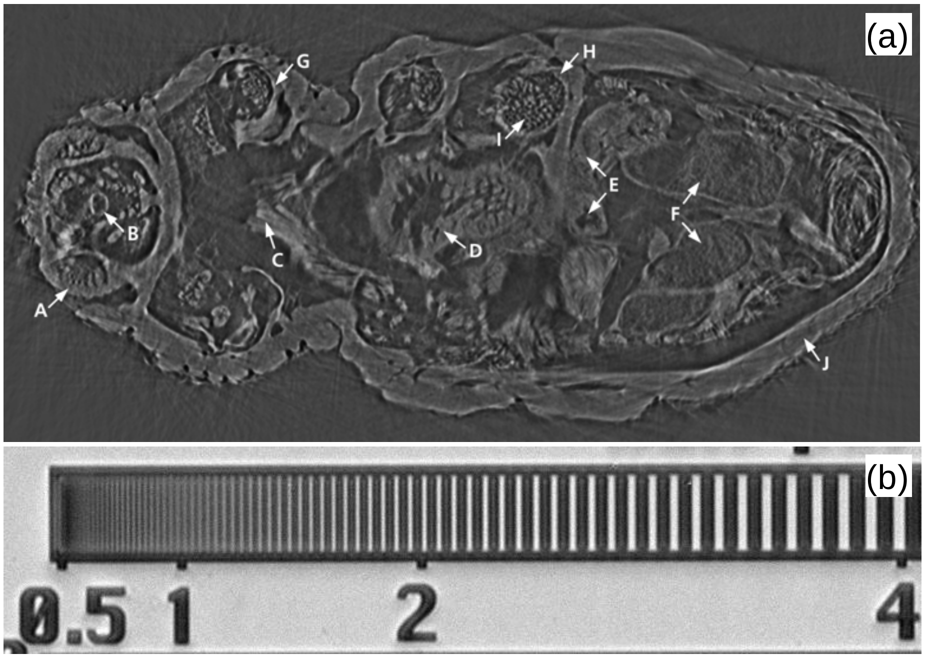

Figure2: (a) Reconstructed slice of a weevil (*Acalles roboris*). A: compound eye; B: oesophagus; C: central nervous system; D: anterior ventriculus (stomach); E: posterior ventriculus; F: developing eggs; G: procoxa (hip of foreleg); H: metacoxa (hip of hindleg); I: metacoxal muscle fibers J: elytron (hardened forewing).

(b) Image of the X500-200-30 resolution test pattern.

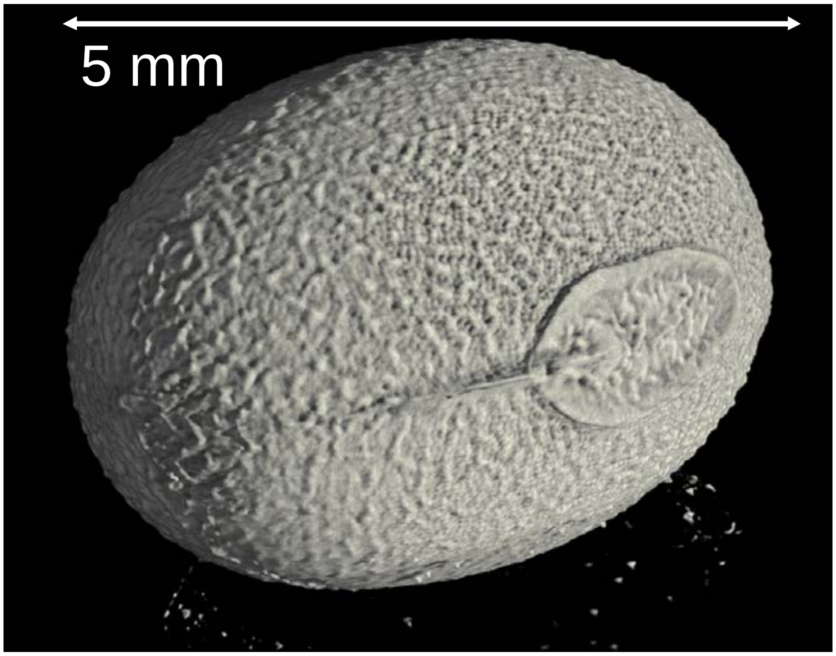

Figure3: Volume rendering of an egg of the stick insect *Peruphasma schultei*