# Light extraction from Scintillating Crystals enhanced by FIB patterned PhC structures.

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# Abstract—

Photonic Crystals have been used in a variety of fields as a structure for improving the light extraction efficiency from materials with high index of refraction. In previous work we already showed the light extraction improvement of several PhC covered LYSO crystals in computer simulations and practical measurements. In this work, new samples are made using different materials and techniques which allows further efficiency improvements. For rapid prototyping of PhC patterns on scintillators we tested a new method using FIB (Focused Ion Beam) patterning. The FIB machine is a device similar to a SEM microscope, but it uses ions (mainly gallium) instead of electrons for the imaging of the samples' surface. The additional feature of FIB devices is the option of surface patterning in nano-scale which was exploited for our samples. Three samples using FIB patterning have been produced. One of them is a direct patterning of the extraction face of a 0.8 x 0.8 x 10 mm<sup>3</sup> LYSO crystal. The second and third were TiO<sub>2</sub> coated LYSO crystal with a 1.4 x 1.4 mm<sup>2</sup> out coupling face partly covered with highquality photonic crystals. TiO<sub>2</sub> has high refractive index (around 2.1 - 2.4) – which shows the best results in our light extraction simulations. In this paper, scanning electron microscopy (SEM) images of the samples will be presented together with measurements of excited crystals which show unrivalled performance for such scintillator samples.

Patterning of all crystals was performed in the laboratory of Nanometrology Group of Faculty of Microsystem Electronics and Photonics, which is part of Wroclaw University of Technology, Poland.

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### I. INTRODUCTION

 $\mathbf{P}^{\text{ET}}$  (Positron emission tomography) and detectors in HEP (High Energy Physics) both use scintillating crystals and

face similar problems when it comes to light extraction. A large fraction of light produced in the crystals is trapped inside the crystal due to total internal reflection caused by their high refraction index [1].

Scintillating crystals and other high refractive index materials (e.g. LED) patterned with PhC (Photonic Crystals) shows better light output [2-5] compared to unpatterned samples. Snell's law states that light can only pass from the medium with higher refractive index to a medium with lower refractive index, when the angle of interaction is smaller than critical angle  $\theta_c$ . This can be partly overcome by nano-structuring of the interface between materials. By using PhC - gratings it is possible to extract part of the light which normally would be internally reflected [4][5], this increase the light output from the crystal which can improve the quality of medical imaging. Future accelerators and particle physics experiments can benefit from this technology, which help them to cope with high luminosity and timing constraints.

We used a FIB microscope for nano-structuration of the crystal surface. The technique is perfectly adequate as a fastprototyping method with a very direct path from computer simulations of the photonic pattern to first working prototypes. Because of those benefits FIB technique has been used for prototyping of both electrical and optical based nanostructure devices [7-10]. The FIB machine is a device similar to a SEM (scanning electron microscope), but it uses ions (mainly gallium) instead of electrons. For imaging purposes, the FIB operates at small currents (pA) [10-12], while for the patterning of surfaces in nano-scale uses high currents (nA) [7-12]. We have produced three prototype samples using FIB patterning. In this work, the production methods and materials are presented as well as the results of first measurements of light output gain.

## II. FOCUSED ION BEAM SAMPLES PRODUCTION

Focused Ion Beam is a microscopy technique which can both image and structure surface of the materials – see figure 1, with the smallest spot size of 2.5 nm it is possible to manufacture very complex nanostructures in almost any inorganic conductive and unconductive materials [7-12][14]. All this makes FIB device an excellent fast prototyping tool in



Fig. 1. Focused beam of gallium ions "bombards" surface of the sample and sputters a small hole in the material. The material leaves the hole in the form of secondary ions (i+ or i-) or neutral atoms

the area of nanopatterning and nanophotonics [7-10] – in our case for production of PhC on the surface of scintillating crystals. A LYSO crystal is an extremely unconductive material, what makes it a difficult substrate to pattern with FIB. To improve the quality of surface imaging and the crystal patterning, the samples were covered with a 10 - 20 nm layer of chromium which was removed afterwards. To figure out all the parameters of the FIB procedure, we had to conduct a series of quality and time optimization experiments. First, the optimisation of the beam power and the focusing time were performed. We used different beam current, with the aim of obtaining holes of the same geometry in the shortest possible time, see results in figure 2. Next conducted trial was a beam



Fig. 2. Beam power and quality test - all the holes in the image were supposed to be the same size, produced with different beam power



Fig. 3. Beam power test – different depth and diameter of the holes. The goal of the test was to obtain the highest quality detail in the shortest time.

power test to achieve the highest quality of patterning in the shortest time – see example in figure 3, where geometries were tested at 0.24 nA current and 30 kV acceleration voltage.

#### **III.** PHOTONIC STRUCTURES DESIGN AND SIMULATIONS

The photonic patterns of our samples were designed in several steps. On each step of the process we were adapting our designs accordingly to the possibilities of the FIB machine to produce fast patterns. All the simulations were performed using CAMFR, a freely available software which is based on the eigenmode expansion principle, which divides the simulated structure in a number of layers where the refractive index does not change in the 'z' direction [13]. Such operation allows treatment of each layer as a sum of the local eigenmodes of the particular layer, which leads to a very compact representation of the layer and therefore smaller need for computation power [13].

# A. Designed patterns

The three different patterns were designed to achieve maximal gain in the light out-coupling performance. The first sample is a directly engraved LYSO scintillating crystal with PhC square matrix of holes - see figure 4. This was the first sample used as the proof of concept of usage of FIB for fast prototyping of photonic structures on scintillating crystals. Second sample is as well patterned with a matrix of holes positioned in a square pattern, but the PhC are engraved in a layer of high refractive index TiO<sub>2</sub> material deposited on top of the crystal, which is beneficial for the light extraction parameters of the structure – see figure 5. The third sample is an optimized version of the  $2^{nd}$  configuration with TiO<sub>2</sub> material and a more efficient hexagonal pattern.

#### B. Simulation results

The simulation results indicate, that there is no extracted photons at angles larger than critical angle  $\theta_c$  for an unpatterned crystal, regardless of air or glue contact – see figure



Fig. 4. Directly engraved matrix of Photonic Crystals holes on the surface of a scintillator



Fig. 5. Photonic crystals holes engraved in layer of  $TiO_2$  deposited on top of a scintillating crystal

6 & 7. For patterned crystals, the simulated gain value is 1.062 for single pass (photons directly leaving the crystal) and 1.285 for multiple incidences (photons collected after several internal reflections) in glue contact – see figure 6. Similarly 1.320 and 1.522 in the air contact case – see figure 7. The gain values were simulated for a simple square pattern of holes where, the hole dimensions are: diameter – 400 nm, height – 300 nm and period of 500 nm. These parameters can be seen



Fig. 6. Photonic crystals holes engraved in the layer of  $TiO_2$  deposited on top of the scintillating crystal – glue contact



Fig. 7. Photonic crystals holes engraved in the layer of  $TiO_2$  deposited on top of the scintillating crystal – air contact in our first test sample – see figure 9.

## IV. PRODUCED SAMPLES

Three successful samples have been produced using FIB patterning; in first example we produced a directly engraved pattern on the surface of the scintillating crystal - see figure 8 & 9. For the two other samples - see figures 10 to 13, a layer of 450 nm of TiO<sub>2</sub> was deposited and patterned. TiO<sub>2</sub> has high refractive index and it is beneficial for light extraction enhancement – increase of gain [1][2].

# A. Directly engraved crystal

The first sample is a 0.8 mm x 0.8 mm x 10 mm LYSO crystal with the PhC structure directly engraved on its surface - see figure 8 & 9. Twenty four photonic crystals patches 100  $\mu$ m x 100  $\mu$ m each, are engraved on the surface of the crystal (see figure 8) and the patch production time was approximately 67 minutes. Each patch contains 40000 holes arranged in a simple



Fig. 8. SEM image 0.8 mm x 0.8 mm out-coupling face of the scintillating crystal with 24 (100  $\mu$ m x 100  $\mu$ m) directly engraved patches of PhC



Fig. 9. SEM image – zoom on the surface of the patterned crystal, hole diameter: ~320 nm, depth: ~300 nm, and period of 500 nm



Fig. 10. SEM image 1.4 mm x 1.4 mm out-coupling face of the scintillating crystal with 178 (50  $\mu$ m x 50  $\mu$ m) patches of PhC



Fig. 11. SEM image – zoom on the surface of the patterned crystal, hole diameter: ~200 nm, depth: ~300 nm, and period of 500 nm



Fig. 12. SEM image 1.4 mm x 1.4 mm out-coupling face of the scintillating crystal with 664 (25  $\mu$ m x 25  $\mu$ m) patches of PhC



Fig. 13. SEM image – zoom on the surface of the patterned crystal, hole diameter: ~600 nm, depth: ~450 nm, and period of 800 nm.

square pattern and their geometry is as follows: hole diameter:  $\sim$ 320 nm, depth:  $\sim$ 300 nm, period between holes – 500 nm - see figure 9. FIB beam parameters used for patterning were I = 0.4 nA and U = 30 kV. Before patterning, the sample was deposited with 20 nm of chromium. It was partly removed during the pattering process, whereas un-patterned areas were cleaned afterwards.

# B. Engraved $TiO_2$ thin layer

After a successful first sample, we produced two more samples with PhC structure engraved on top of 1.4 mm x 1.4 mm x 7.2 mm scintillating crystal with around 450 nm thick layer of TiO<sub>2</sub> to enhance the light extraction. Sample 2 had 178 patterned patches of 50  $\mu$ m on 50  $\mu$ m each – see figure 11. Each patch contains 10000 holes arranged in a simple square pattern, their geometry is as follows: hole diameter: ~200 nm, depth: ~300 nm, period between holes – 500 nm - see figure 12. Last sample has 664 small patches of 25  $\mu$ m on 25  $\mu$ m each with optimized hexagonal pattern – see figure 12. Each patch contains 3515 holes, their geometry is as follows: hole diameter: ~600 nm, depth: ~450 nm, period between holes – 800 nm - see figure 13.

# V. RESULTS

For the measurement of the gain from the PhC we excited crystals with UV light and analysed the difference in the light intensities between zones with and without photonic



Fig. 14. Sample 1 - UV excited out-coupling face of directly engraved crystal. Measured gain:  $\sim$ 30%, crystal size – 0.8 mm x 0.8mm



Fig. 15. Sample 2 - UV excited out-coupling face of crystal with PhC in  $TiO_2$  layer on the surface. Measured gain: ~70%, crystal size 1.4 mm x 1.4 mm



Fig. 16. Sample 3 - UV excited out-coupling face of crystal with PhC in  $TiO_2$  layer on the surface. Measured gain: ~80%, crystal size 1.4 mm x 1.4 mm

patterning – see figures 14 to 16. In our setup, an UV diode was placed on top of the crystal, this is why for the results comparison we choose patterned and unpatterned zones which were in the same distance from the diode. Measured gain in air contact ranges from 35% in the directly engraved scintillating crystal up to 80% in the optimised pattern with  $TiO_2$  layer on top of the crystal – for results and excited crystals pictures see table 1 and figures 14 - 16.

### VI. CONCLUSIONS

Our first samples have proven focused ion beam machining to be an efficient tool for the production of PhC pattern prototypes on top of scintillating crystals. The process of

	TABLE I	
Measured gai	n values in air surrounding for all sam	ple

Sample number	Measured gain	Patterned material
1	30%	Directly engraved LYSO
2	70%	TiO2
3	80%	TiO2

patterning needed several tests and optimization procedures. After adaptation of pattern design and beam parameters, we managed to produce 3 samples on the pixel sized crystals. Our measurement results are in accordance with simulated values. For the first – directly engraved sample, the gain was 30% higher comparing to unpatterned areas of the crystal. For the PhC created in the  $TiO_2$  layer on top of the crystal, the measured gain was as high as 70% for the square pattern sample and 80% for the hexagonal pattern. Measurements were conducted with UV excitation of crystals and in air surrounding.

In our next steps we will produce samples with the whole out-coupling face covered with a photonic structure; this will give us a chance to measure the light gain not only in the UV excitement setup, but additionally on a classical light yield bench with gamma excitation and PMT (Photo Multiplayer Tube) measurement. This will allow us to measure in glue contact which is not possible with the current samples.

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