Combination of LiF crystals with optical confocal micro-spectroscopy for advanced 2D-3D X-ray detection

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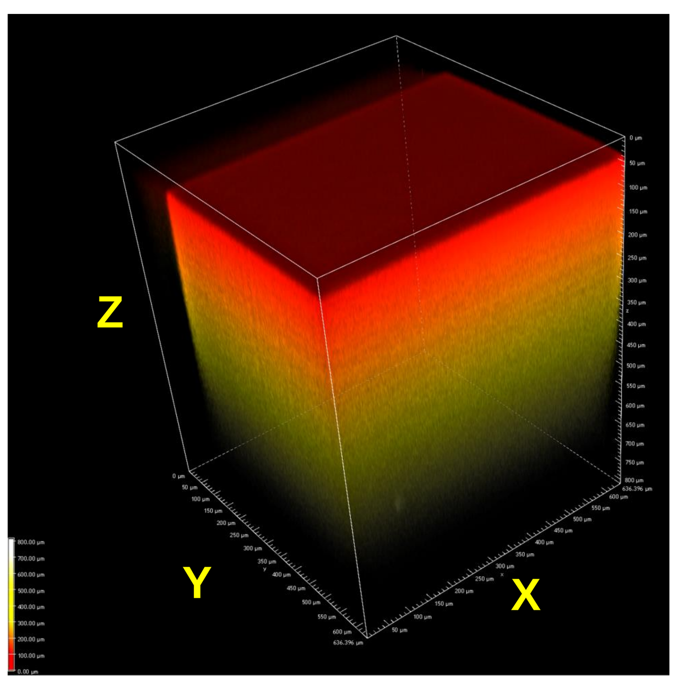
We investigated the capability of lithium fluoride (LiF) crystals to register volumetric X-ray mapping through the local formation of radiation-induced electronic point defects (color centers) and their combination with confocal microscopy-spectroscopy reading techniques based on fluorescence/Raman signals detection. The investigated LiF crystals were irradiated with monochromatic (8 keV) X-ray beam at KIT synchrotron light source (Karlsruhe, Germany) and with the broadband white beam spectrum of the synchrotron bending magnet. In the framework of **TECHEA** (Technologies for Health) project, the optical sectioning operations of confocal microscopy working in fluorescence mode and confocal Raman micro-spectroscopy have allowed to obtain 3D reconstructions of the X-ray colored volumes [1]. Among the peculiarities of these LiF-based detectors, noteworthy one is their very high intrinsic spatial resolution related to the atomic scale of the radiation-induced defects (the lattice constant of LiF is the shortest among all alkali halides, i.e. 0.2013 nm). The presented results provide interesting perspectives for 2D and 3D X-ray imaging by the combination of LiF plates with advanced reading techniques based on optical confocal micro-spectroscopy.

Lithium fluoride (LiF)-based detectors represent a versatile tool for X-ray imaging and for characterization of X-ray beams and optics. These detectors have been used with several X-ray sources, such as compact sources [2,3,4], large-scale facilities [5] and for X-FEL beam monitoring [6,7]. Among the peculiarities of LiF-based detectors, noteworthy ones are their very high intrinsic spatial resolution across a large field of view, wide dynamic range and versatility. The peculiarities of these LiF-based solid-state detectors - high spatial resolution on a large field of view, wide dynamic range, versatility and simplicity of use - combined with opportunities offered by the fast development of soft and hard X-ray sources (laser plasma sources, X-ray lasers, table-top X-ray tubes) as well as large scale facilities (synchrotrons, free electron lasers) make their application very attractive in physics as well as in life sciences. The characteristics of LiF imaging detectors overcome some of the limitations of the standard ones and can be exploited for X-ray microscopy in very simple configurations, such as lensless techniques, even for *in vivo* investigations of biological samples. R&D activities are going on to improve the performances of these promising radiation imaging detectors and to study the best approaches and conditions for imaging experiments, including in contact and phase contrast techniques.

Commercial LiF crystals in the form of squared plates polished on both surfaces were irradiated at the synchrotron light source of KIT. A LiF crystal (10x10 mm^2, 1 mm thickness) was irradiated on a uniform area with the 8 keV monochromatic X-ray beam.

CONFOCAL FLUORESCENCE MICROSCOPY

Nikon Eclipse 80i-C1



3D reconstruction (XY = 636 × 636 μm^2, Z = 1 mm) of the photoluminescent volume acquired by CLSM of the 8 keV X-ray irradiated LiF crystal (10x10x1 mm^3) on a uniform area.

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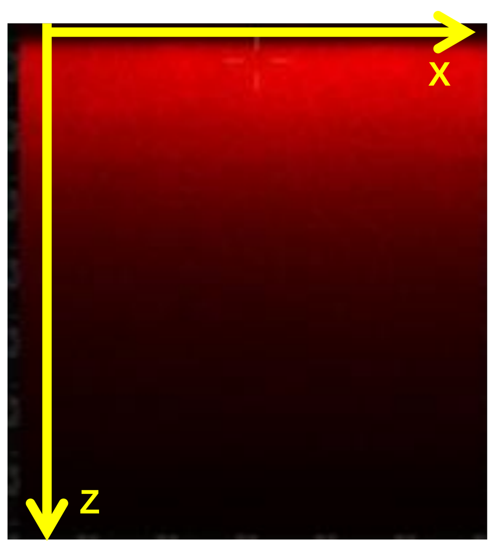
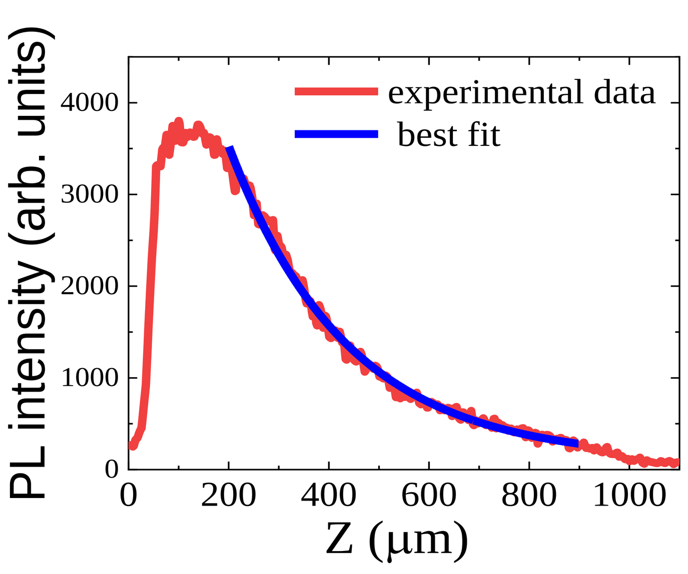


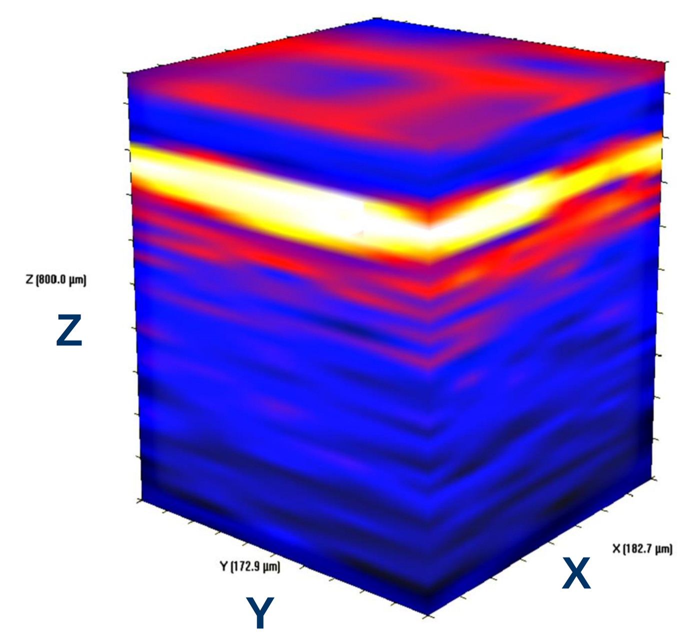


XZ slice of the 3D reconstruction showing the Z distribution of F_2 CCs fluorescence intensity along the thickness of the LiF crystal (1 mm) and the corresponding photoluminescence PL intensity profile along Z scan optical axis with its best fit by a single exponential curve.

According to the X-ray transmission properties in solids [8], the energy deposition of the X-ray beam exponentially decreases along the penetration depth in LiF. Assuming that CC concentrations are proportional to the deposited energy, the confocal fluorescence microscope is an appropriate technique to investigate the X-ray colored profile along the crystal thickness (Z direction). The experimental F_2 PL profile (red curve) is shown in Figure 5c together with its best fit with a single exponential curve (blue curve), a value of (236 ± 3) μm, corresponding to the 1/e of the maximum, being obtained. The theoretical X-ray attenuation length in LiF corresponding to an X-ray energy of ~ 8 keV is 331 μm [8]. Since possible re-absorption phenomena of pumping laser by CCs or reabsorption of the CC PL during CLSM Z-scanning measurements could occur in this 3D characterization, a comparison with the confocal Raman micro-spectrometer was performed.

CONFOCAL RAMAN MICRO-SPECTROSCOPY

Horiba XploRA Plus



3D Raman maps (XY = 182 × 172 μm^2, Z = 0–1 mm) of an uniform colored area of the 1-mm thick LiF crystal irradiated with 8 keV X-rays.

A value of (221 ± 3) μm corresponding to the 1/e of the maximum, was obtained. This value is close to that obtained with red fluorescence signal in the CLSM systems.Further investigations regarding optical sectioning of the confocal systems applied to X-ray colored LiF crystals are in progress.

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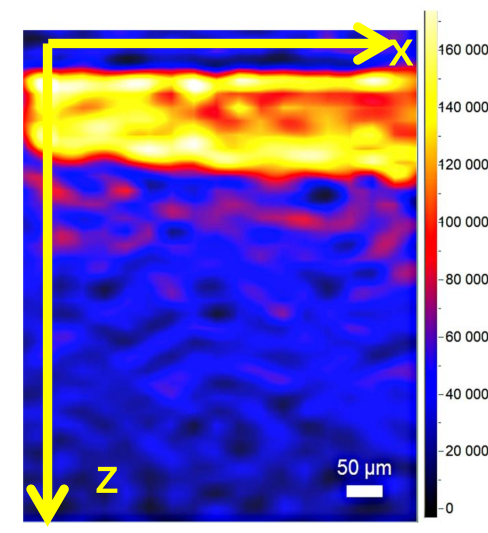
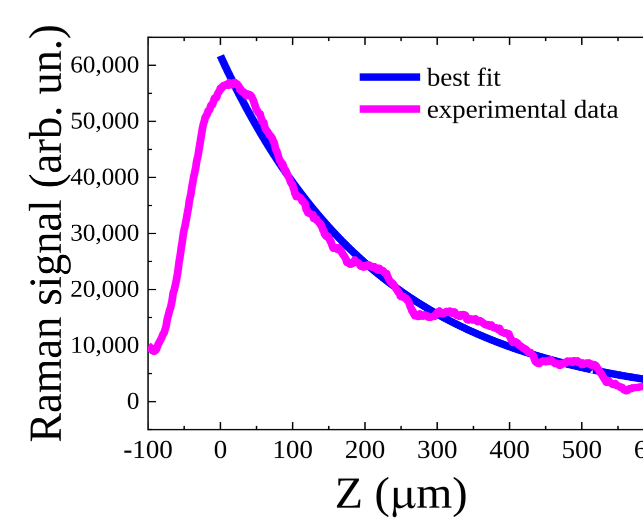
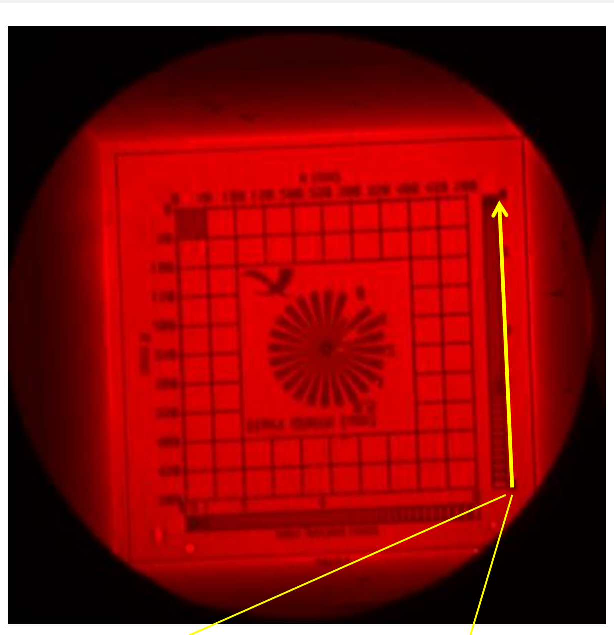


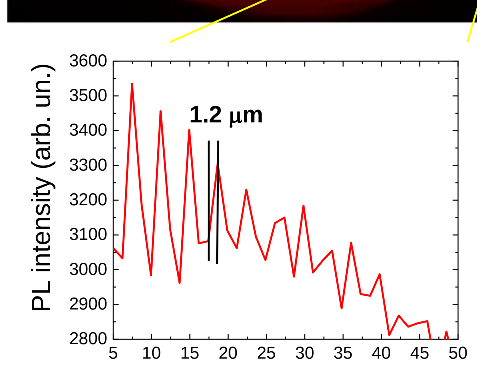


XZ slice of the 3D Raman map and the corresponding Raman intensity profile along Z direction together with its best fit.

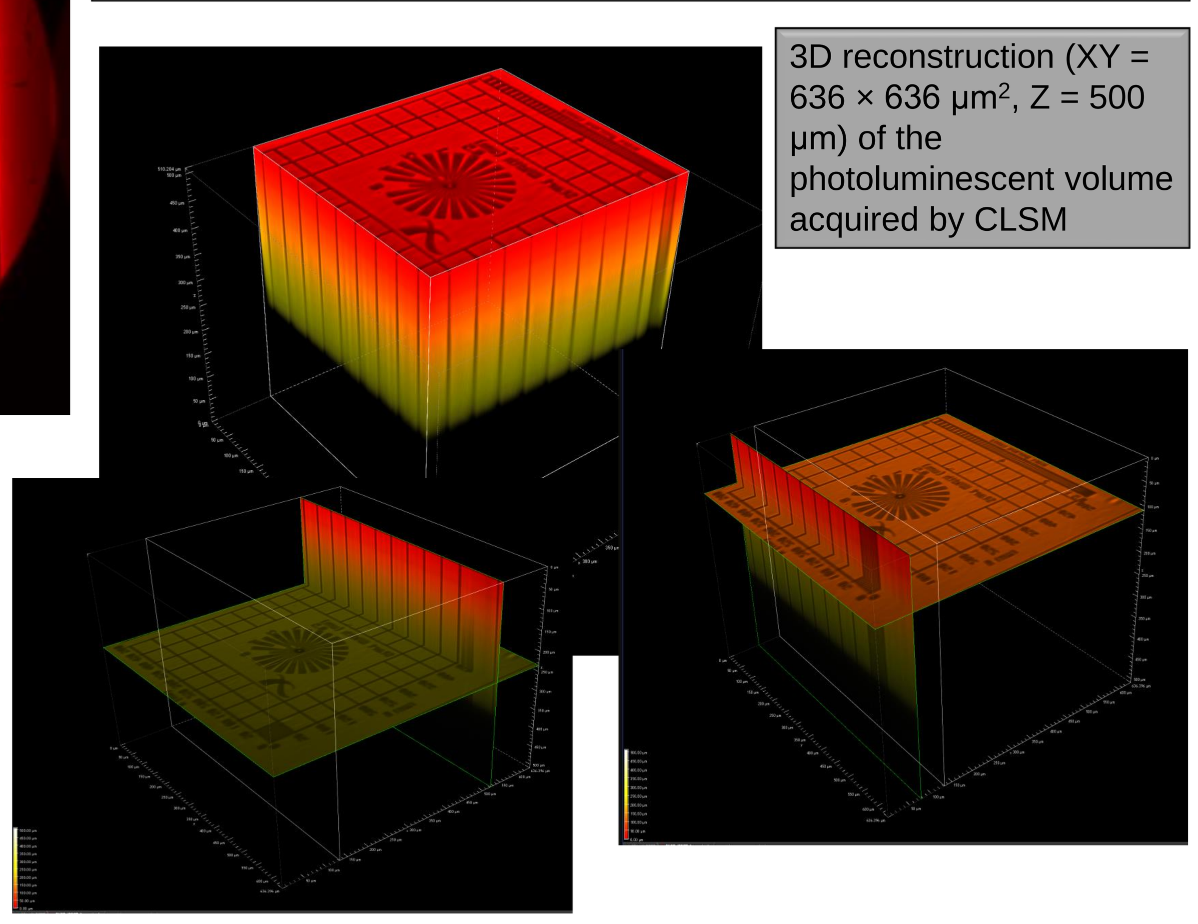
A LiF crystal (5x5 mm^2, 0.5 mm thickness) was irradiated with the broadband white beam spectrum of a synchrotron bending magnet. During the irradiation, a commercial test pattern X500- 200-30 (Xradia, Pleasanton, CA, USA), consisting of 330 nm thick gold mask deposited on a (500 x 500) μm^2 Si_3N_4 window, was positioned at a distance of 26 cm from the exit of the beam (at 29 m from the irradiation source). The propagation distance between the test pattern and LiF sample was set equal to 17.5 cm, thus performing a lens less projection imaging experiment [9]. To impress the X-ray image of the test pattern on the LiFC1 detector, the test pattern was irradiated with several exposure times between 1 s and 60 s. The beam size was set equal to (800 x 800) μm^2.



2D fluorescent image acquired by the CLSM of the irradiated LiF crystal (5x5x0.5 mm^3): image of the radiography of a test pattern stored in the LiF crystal by irradiating with the white broadband X-ray beam for 30 s irradiation time.



PL signal intensity profile along the yellow arrow on a pattern with decreasing spatial period.



3D reconstruction (XY = 636 × 636 μm^2, Z = 500 μm) of the photoluminescent volume acquired by CLSM

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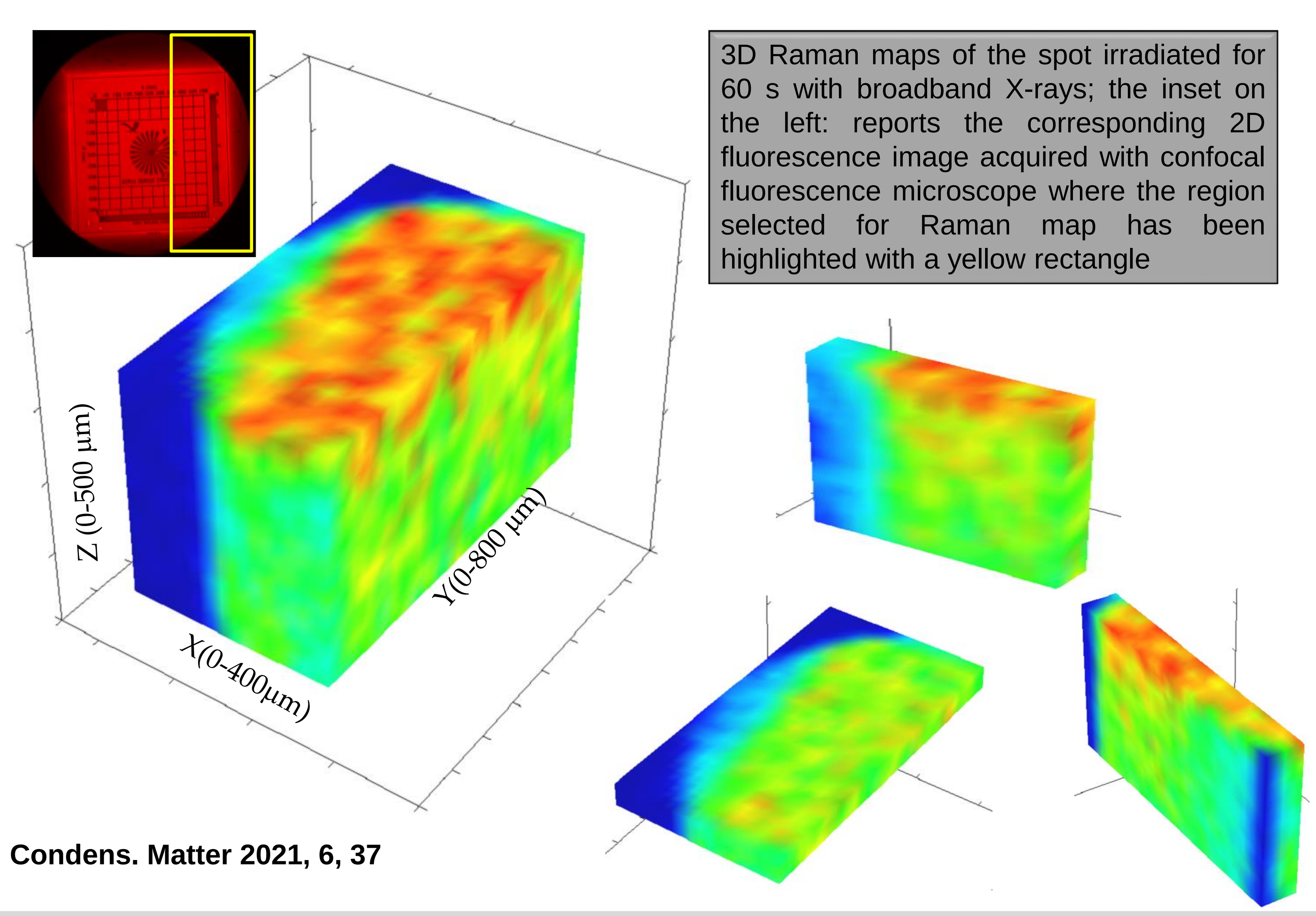
The X-ray image of the pattern is well resolved along the thickness of the irradiated LiF crystal.



3D Raman maps of the spot irradiated for 60 s with broadband X-rays; the inset on the left: reports the corresponding 2D fluorescence image acquired with confocal fluorescence microscope where the region selected for Raman map has been highlighted with a yellow rectangle

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Conclusions

X-ray detectors based on radiation-induced color centers (CCs) locally produced in lithium fluoride (LiF) crystals have been discussed. LiF crystals irradiated with broadband and monochromatic X-rays of synchrotron light source at KIT (Karlsruhe Insitute of Technology, Germany) have been investigated with confocal fluorescence microscopy confocal Raman micro-spectroscopy.

The penetration depths in LiF of the X-rays used for irradiation allowed to produce volumetric distributions of CCs in the crystals. 3D fluorescence and Raman maps of colored LiF crystals have been performed obtaining volumetric reconstructions of the X ray-induced CC distributions. The combination of capability of a LiF crystal to store volumetric information with the optical sectioning operations of optical confocal techniques has allowed performing 3D reconstructions of the X-ray colored volumes and it could provide advanced tools for 3D X-ray detection.

Acknowledgment

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