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AGENZIA NAZIONALE PER LE NUOVE TECNOLOGIE, L'ENERGIA E LO SVILUPPO ECONOMICO SOSTENIBILE

# *Rivelatori a lettura di fotoluminescenza in fluoruro di litio per la caratterizzazione di ottiche policapillari in un sistema table-top di imaging a raggi X*

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http://www.frascati.enea.it/UTAPRAD/lss_imaging.htm
http://www.frascati.enea.it/UTAPRAD/statosolido_film.htm

# Outline

**LiF-based radiation detectors**

Principle of operation – optical reading

Peculiarities and applications

**LiF detectors for X-ray imaging - X-ray tubes combined with polycapillary optics and LiF detectors as a compact X-ray imaging facility**

X-ray polycapillary optics characterization by using LiF detectors

Confocal fluorescence microscopy for advanced optical reading of LiF detectors and 3D optical imaging

Contact X-ray micro-radiography on LiF detectors by X-ray tubes combined with X-ray polycapillary optics (test and geological samples)

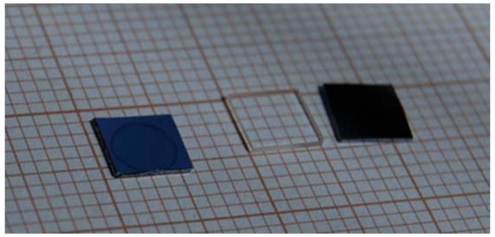
**Conclusions**

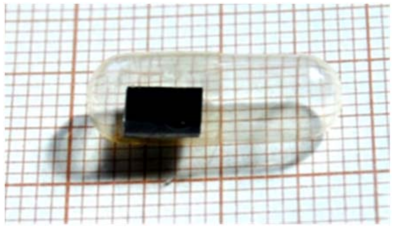
# LiF imaging detectors peculiarities and applications

**Novel thin-film radiation imaging detectors** based on **photoluminescence (PL)** of radiation-induced **ligth-emitting color centers** in **lithium fluoride (LiF) thin layers and crystals**

**PECULIARITIES**

- **Multi-purpose: X-ray (20 eV-30 keV), thermal neutrons, low-energy electrons,...**
- **high spatial resolution (intrinsic < 2 nm, standard < 250 nm )**
- **large field of view ( > 1 $cm^2$ )**
- **wide dynamic range ( > $10^3$)**
- **efficient optical readout process (optical fluorescence microscopy)**
- **easy handling:** no development needs and no sensitivity to visible light
- **versatility:** film geometry compatible with protective layers and different substrates





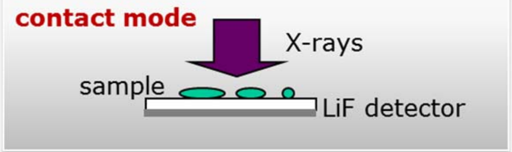
| | LiF-based imaging detectors | Photographic films | photoresist | Phosphor-based imaging plates |
|---|---|---|---|---|
| Soft X-rays | + | + | + | - |
| Hard X-rays | + | + | - | + + (linear response) |
| Reading technique | Optical (PL) | Optical (OD) | AFM | Optical (OSL) |
| No development | + | - | - | + |
| No light sensitivity | + | - | + | - |
| Image stability | + | + | + | - (fading) |
| Spatial resolution | + + | + | ++ | + |
| Dynamic range | + | + | - | + |
| Wide field | + | + | - | + |

**APPLICATIONS**

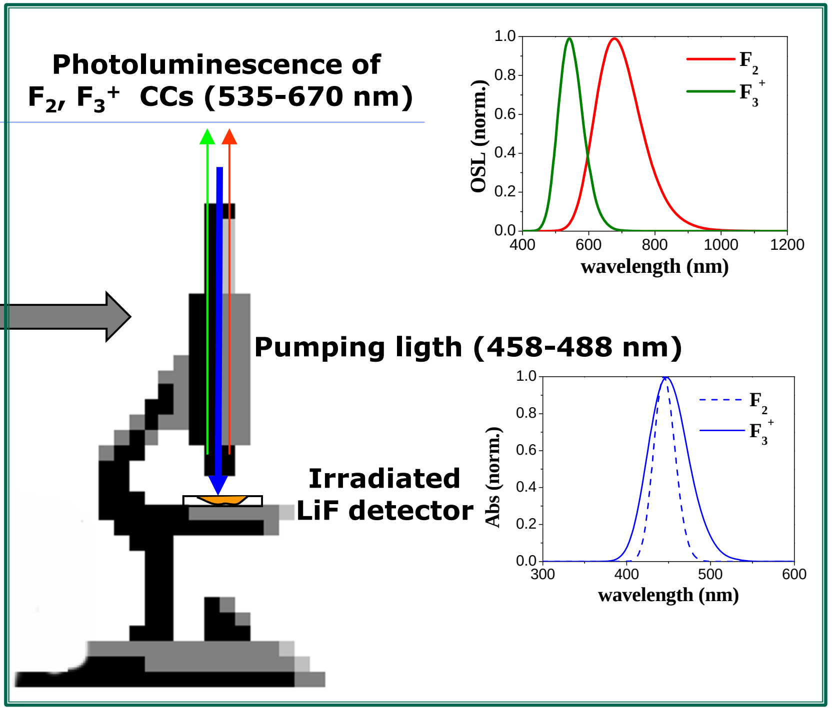
**- photonics and nanotechnologies**
**- materials science & micro-devices characterization**
**- processes diagnostics & characterization of radiation sources for energy, medicine, space...**
**- life sciences: X-ray micro-radiography for bio-medical imaging, even for *in vivo* samples.**

# Reading process of LiF detectors based on fluorescence detection

## Reading process
optical reading of CCs photoluminescence signal

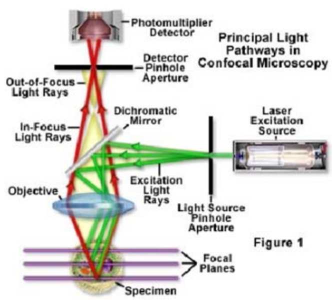



X-ray microradiography on LiF

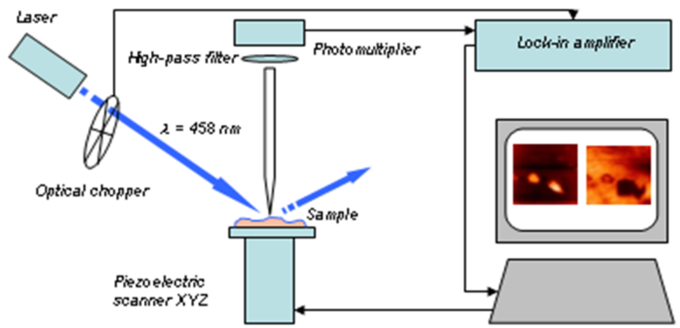



## Advanced Fluorescence microscopy

**Confocal Laser Scanning Microscope – SSL Lab ENEA**



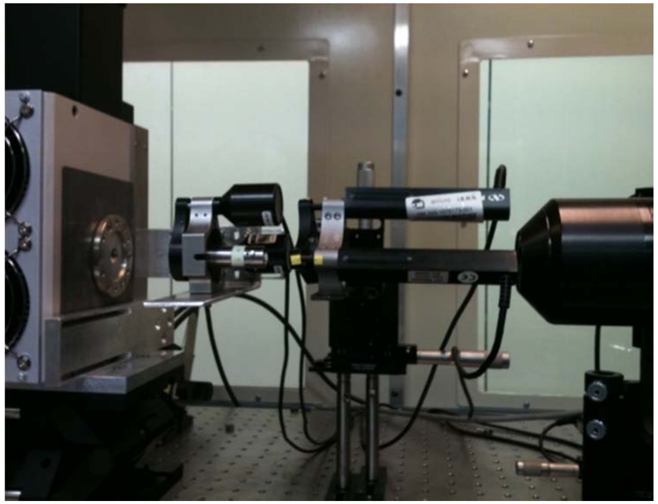

**SNOM collection mode - (CNR-ISM)**



# X-ray tubes combined with X-ray polycapillary optics and LiF detectors as a compact X-ray imaging facility

## X-ray table-top source (XLab – INFN-LNF)

The experimental set-up is based on a conventional X-ray tube (Oxford Apogee 5000, Cu target, 50 Watt) **combined with a polycapillary semi-lens.** With the polycapillary semi-lens it is possible to obtain a quasi-parallel beam from a divergence source.



### X-ray polycapillary lens and half-lens

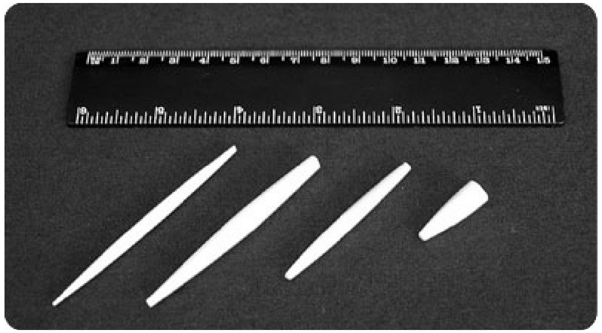


Photo of polycapillary lens and half-lens (http://unisantis.com/kumakhov-optics.html)

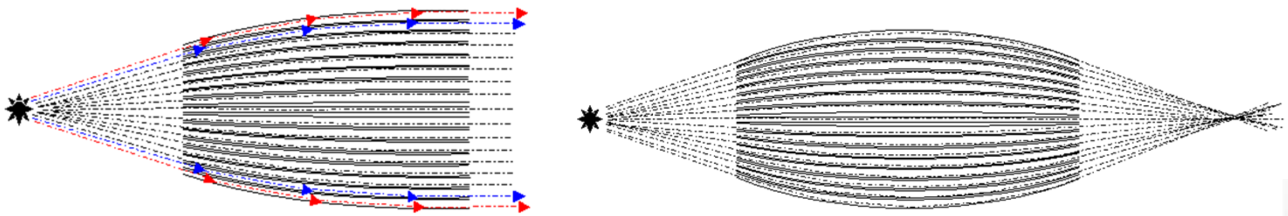
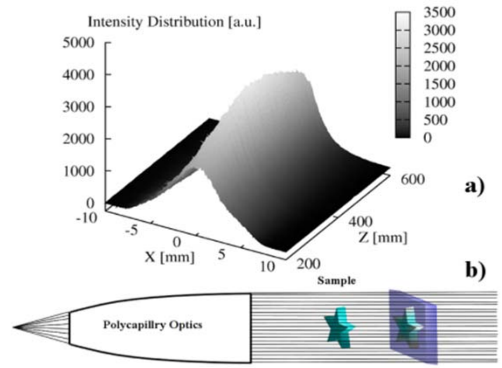


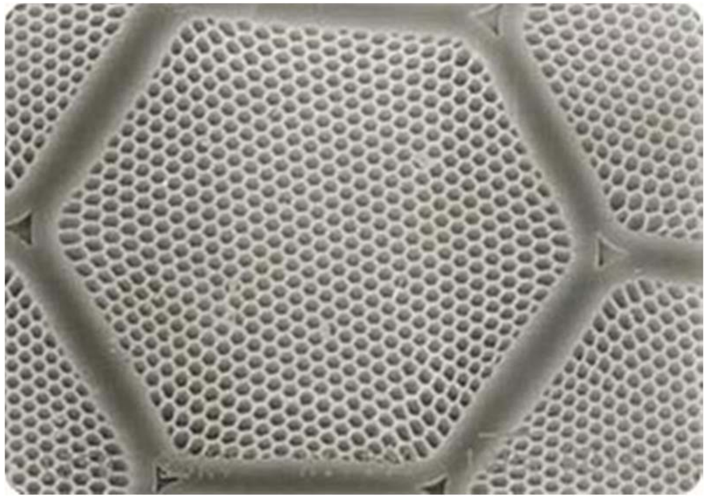
*Fig. 4 Transformation of a divergent beam from X-Ray source into a parallel beam*

Transformation of a divergent beam from X-ray source into a parallel or a focused beam by a polycapillary lens

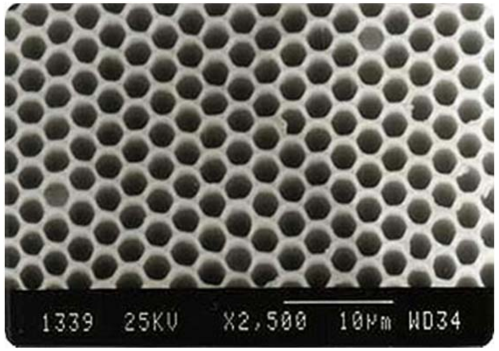


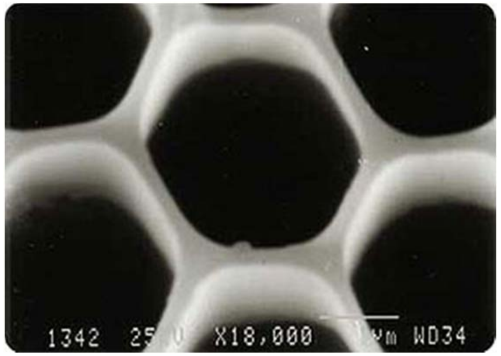

a) Measured X-ray intensity distribution contour behind polycapillary half lens
b) Scheme of X-ray imaging with parallel beam produced by a polycapillary optics

# Characterization of X-ray polycapillary optics with LiF detectors



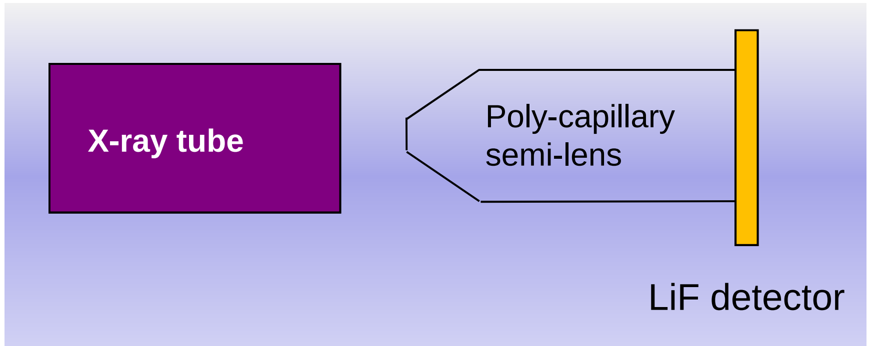
**Electron microscope image of polycapillary lens transversal section (http://unisantis.com/kumakhov-optics.html)**

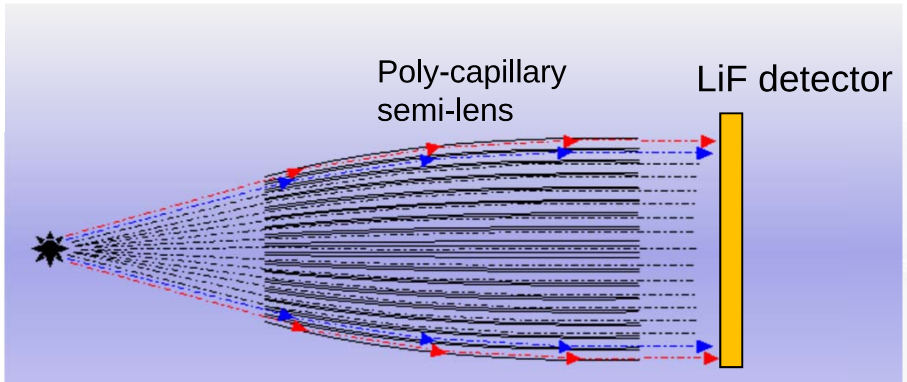



The polycapillary optics is composed by many bundles containing thousands of channels with a length of about 60 mm; each single channel is characterized by an average diameter of about 4 µm.

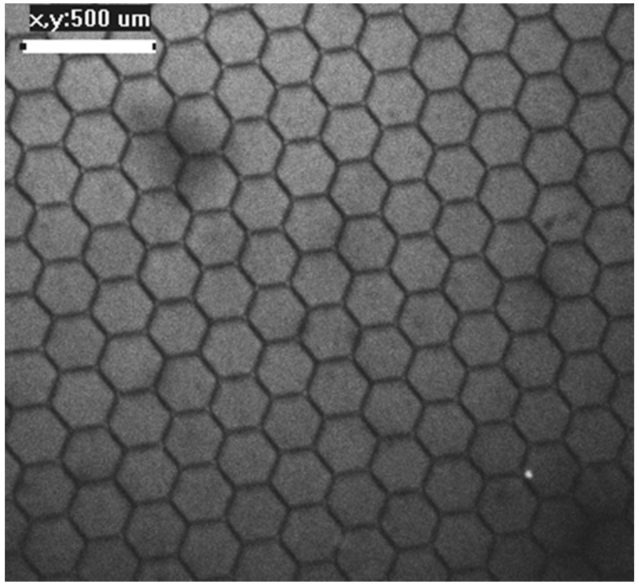
LiF crystal placed at the semi-lens exit



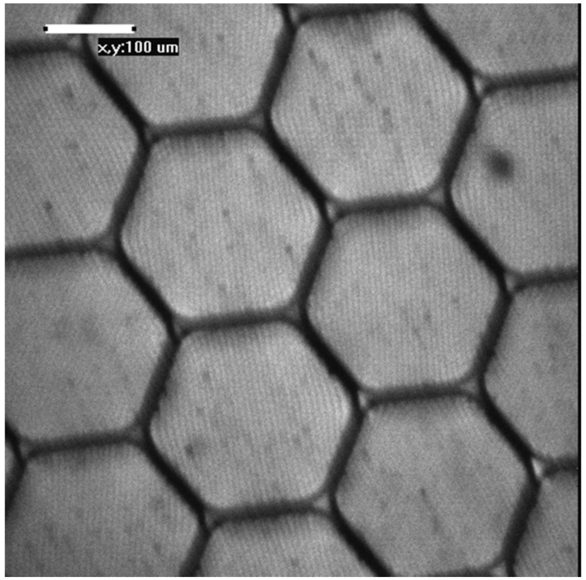

Transformation of a divergent beam from X-ray source into a parallel beam by a polycapillary lens

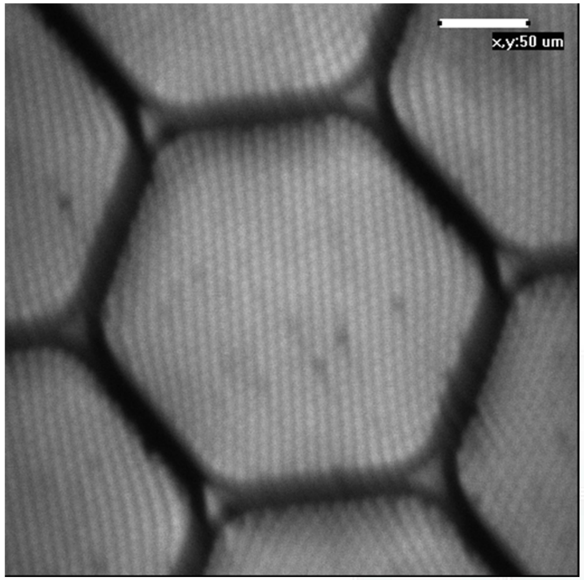
# Detection by LiF detectors of the transmitted X-rays by a policapillary semi-lens




## Fluorescence image stored in a LiF detector of the transmitted X-ray by a polycapillary semi-lens

The fluorescence image is obtained by an optical reading of LiF detector by a confocal microscope in fluorescence mode at different magnifications



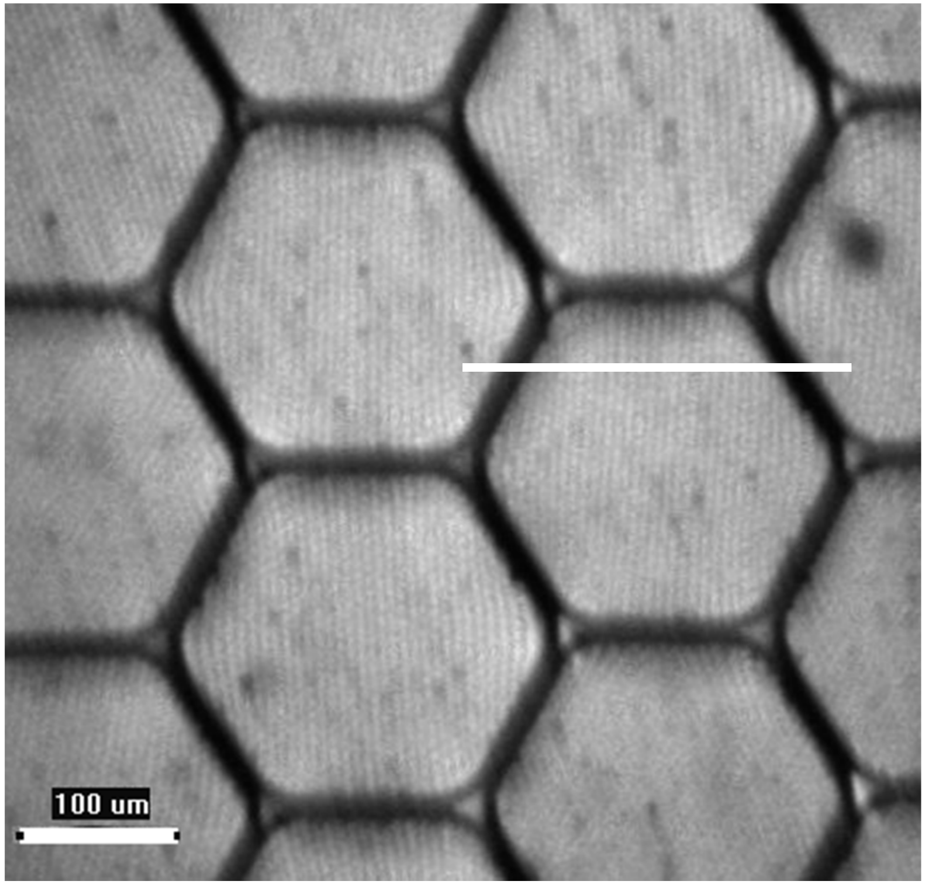



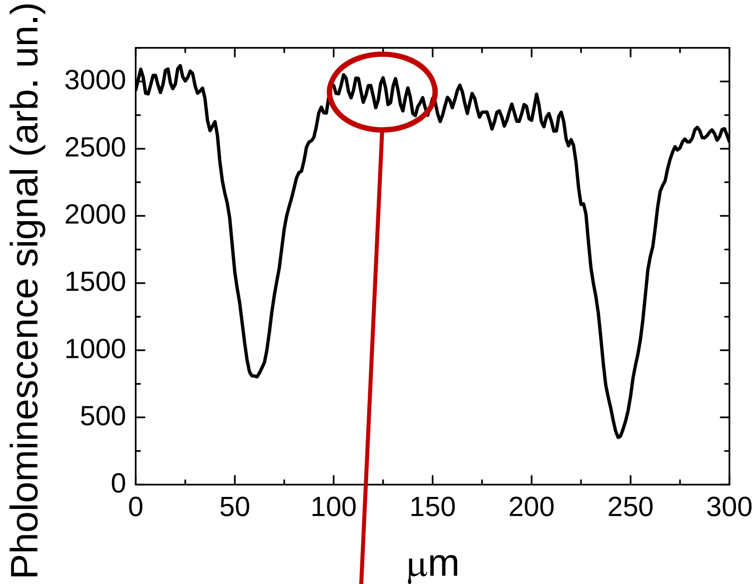

Each glass channel, even if it is stretched due to the melting during the lens drawing process transports effectively X-radiation resulting in a quite uniform X-ray distribution behind the optics.
Additionally, it confirms that the channels remain mainly opened along the entire optics length.
The possibility of LiF detectors to store images with high spatial resolution on a wide field of view simultaneously, in this case allowed to obtain images of the transmitted X-rays through the entire lens and through the single channels.

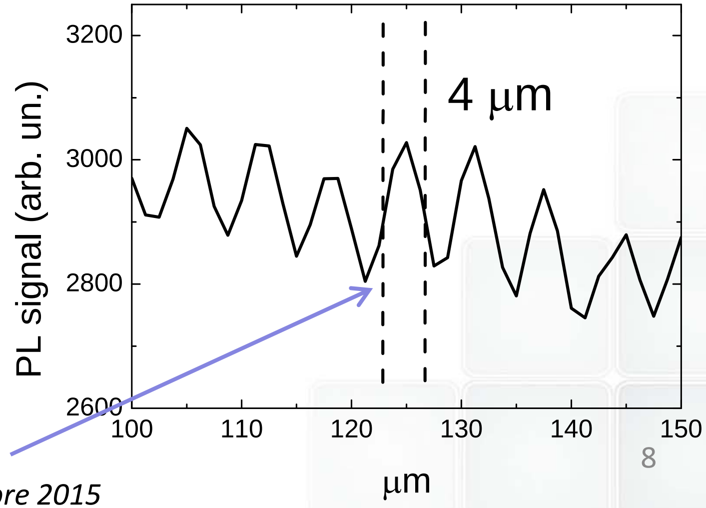
D. Hampai, F. Bonfigli, S.B. Dabagov, R.M. Montereali, G. Della Ventura, F. Bellatreccia and M. Magi, et al. *LiF Detectors-Polycapillary Lens for Advanced X-ray Imaging*, Nuclear Instruments & Methods In Physics Research A, Vol. 720, p. 113-115, (2013).

# Single channel X-ray transmission of the semi-lens stored by LiF detector

## X-ray image on a optical reading LiF detector



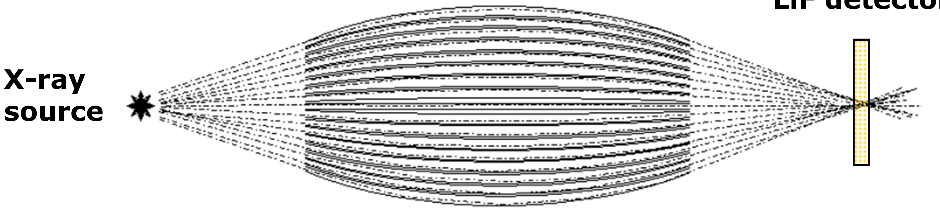




CCs PL signal produced by transmitted X-rays from a single channel of the optics

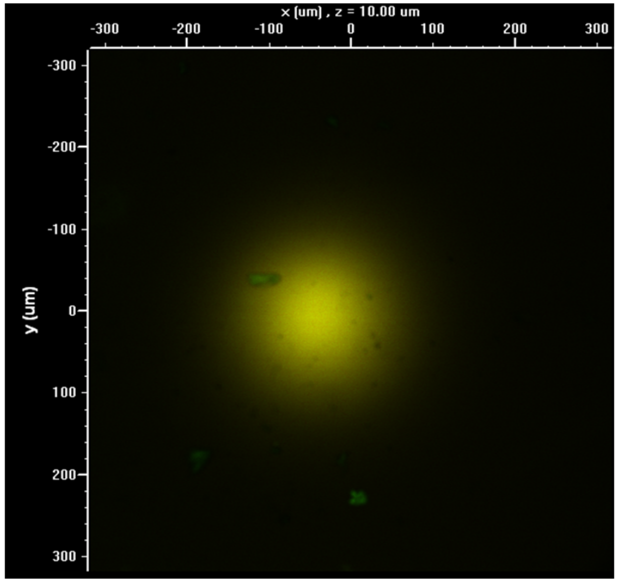
# Investigation by 3D confocal fluorescence microscopy of coloured LiF crystals by a focused X-ray beam

LiF crystal (5x5x0.5)mm$^3$ was irradiated with the Cu X-ray tube ("Oxford Apogee 5000") at XLab (INFN-LNF) (exposure time of 30 min, parameters of X-ray tube: 50 kV, 1 mA, 50 W).

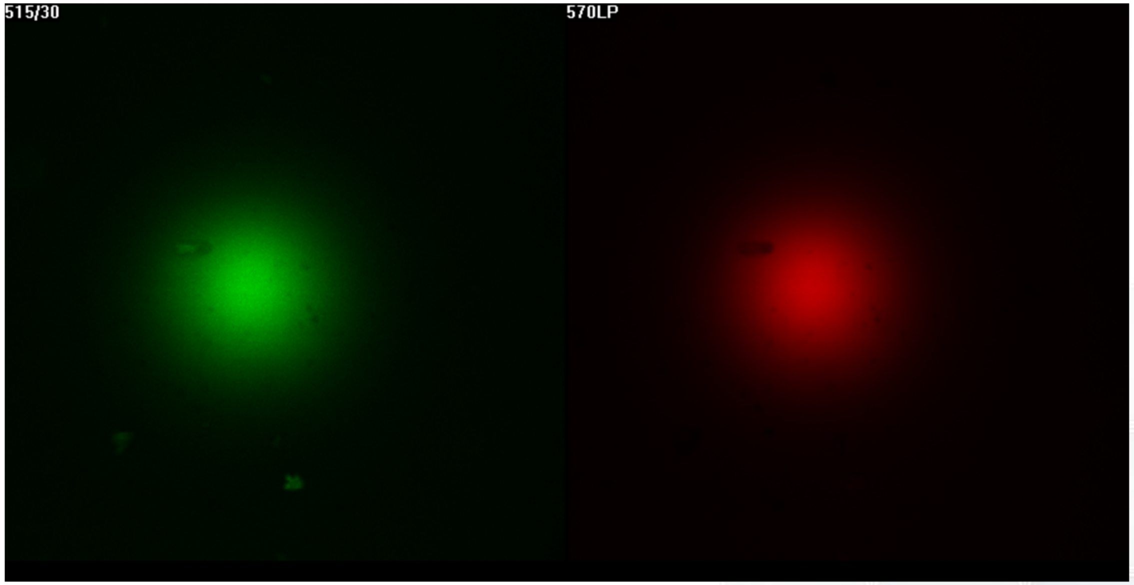



X-ray beam diamenter on lens focus (theoretical) ~ 100 μm

## 2D confocal fluorescence image of CCs produced on the LiF crystal surface by the focused X-ray beam




PL signal of $F_2$ and $F_3^+$ CCs

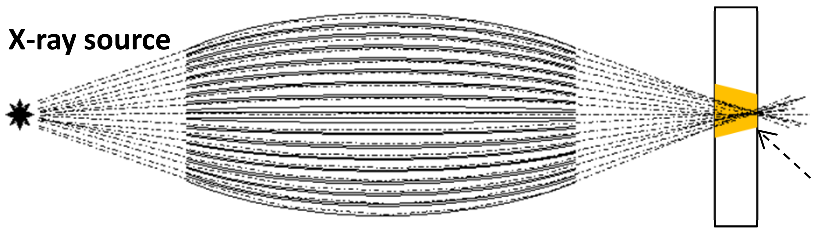



PL signal of $F_3^+$ CCs

PL signal of $F_2$ CCs

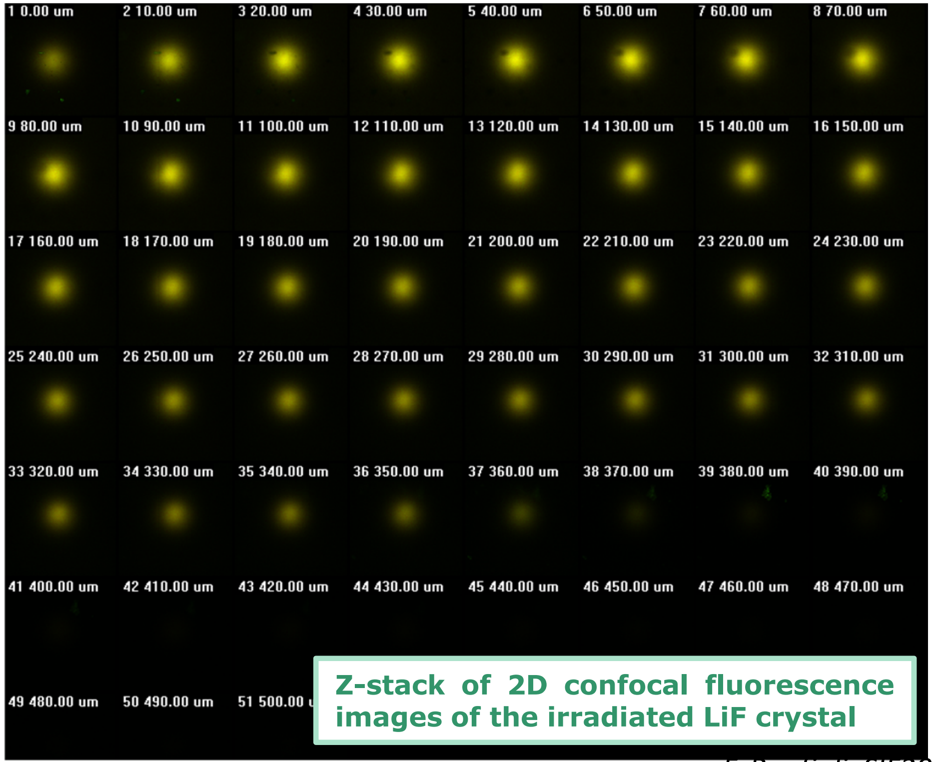
# Investigation by 3D confocal fluorescence microscopy of coloured LiF crystals by a focused X-ray beam

## 3D confocal fluorescence image of CCs produced in LiF a crystal by the focused X-ray beam

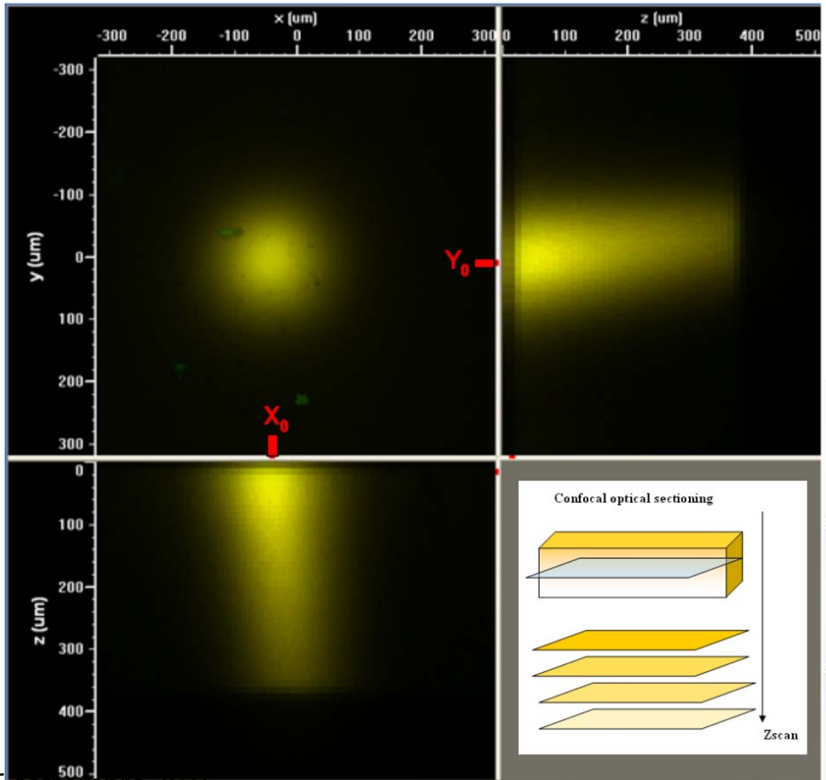



The optical sectioning was performed by an objective 20x (N.A. 0.5).
The samples were analysed over a Z interval of 500 µm acquiring 50 2D-images with a step size of 10 µm.

The green and the red channels were detected simultaneously measuring both $F_3^+$ and $F_2$ PL signals.

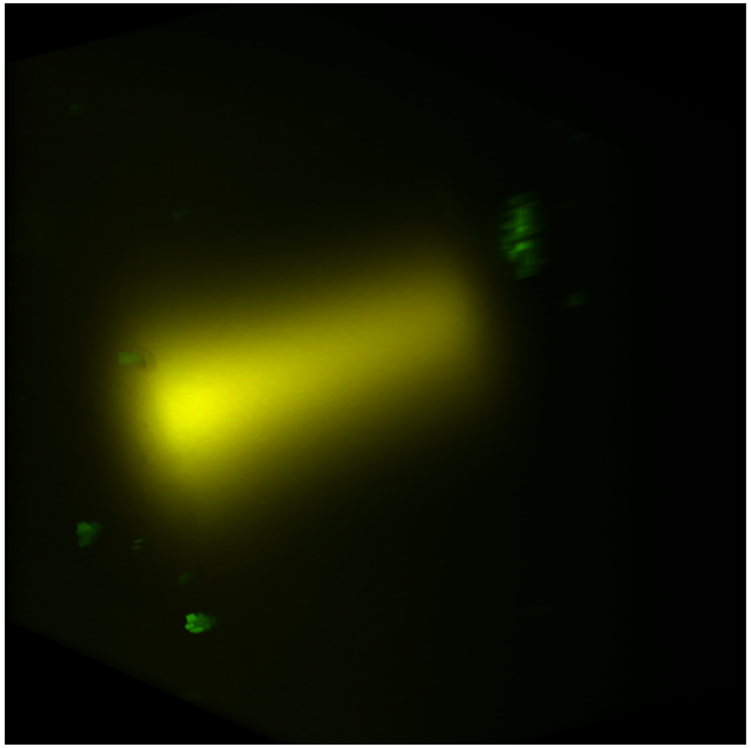


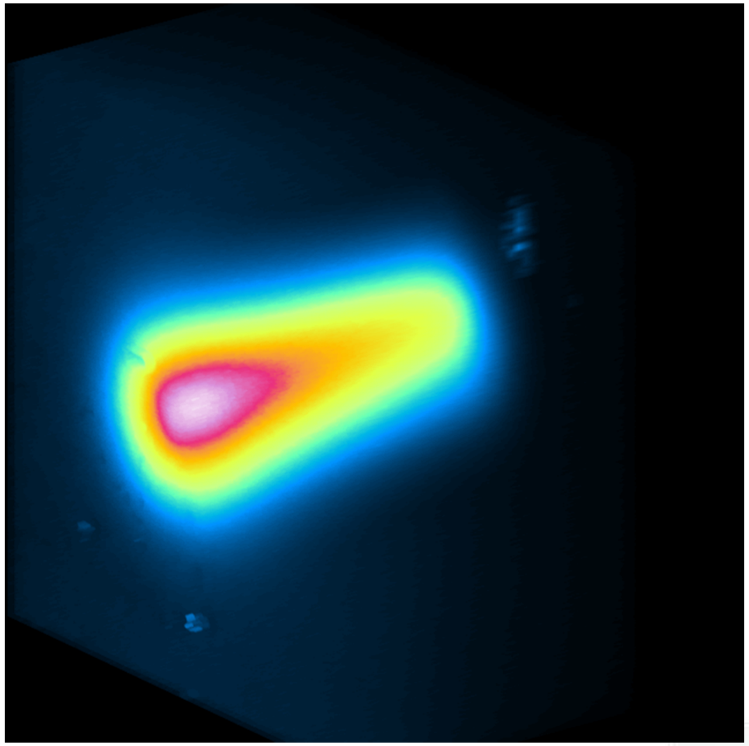
Z-stack of 2D confocal fluorescence images of the irradiated LiF crystal



# Investigation by 3D confocal fluorescence microscopy of coloured LiF crystals by a focused X-ray beam

**3D confocal fluorescence image of CCs produced in LiF a crystal by the focused X-ray beam**





# Investigation by 3D confocal fluorescence microscopy of coloured LiF crystals by a focused X-ray beam

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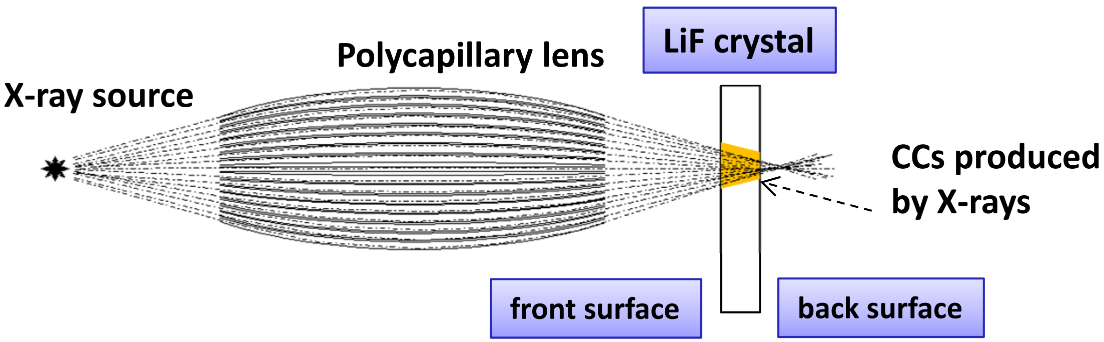
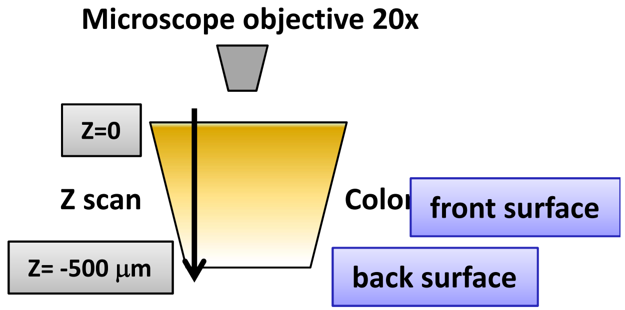
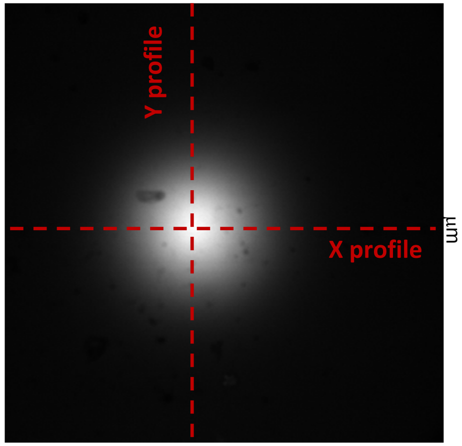
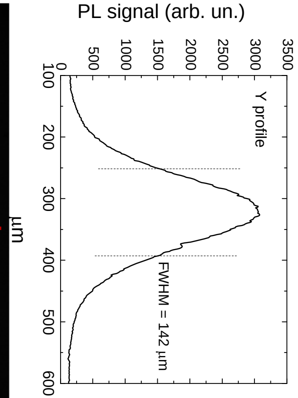


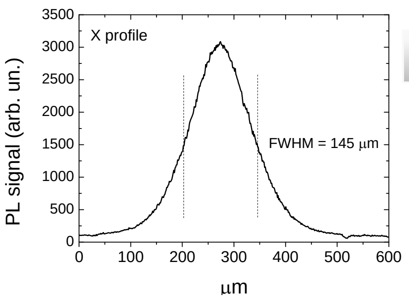




**Z=0** front surface



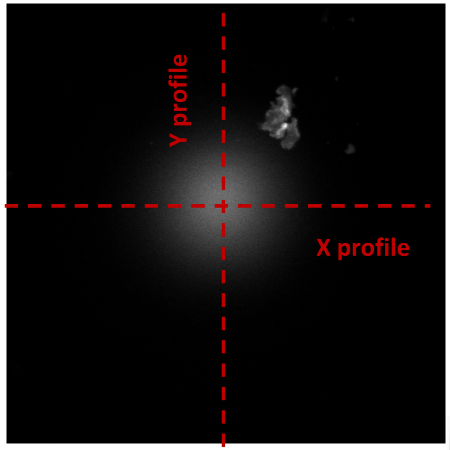



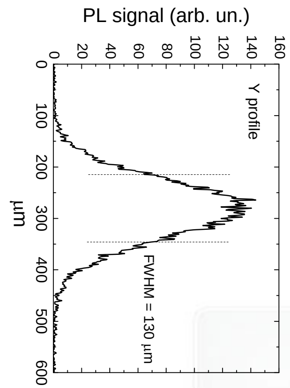

**Y profile – FWHM = 142 µm**



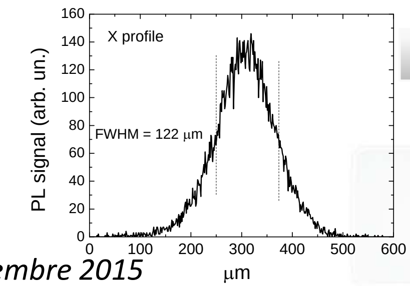

**X profile – FWHM = 145 µm**

**Z= -500 µm** back surface



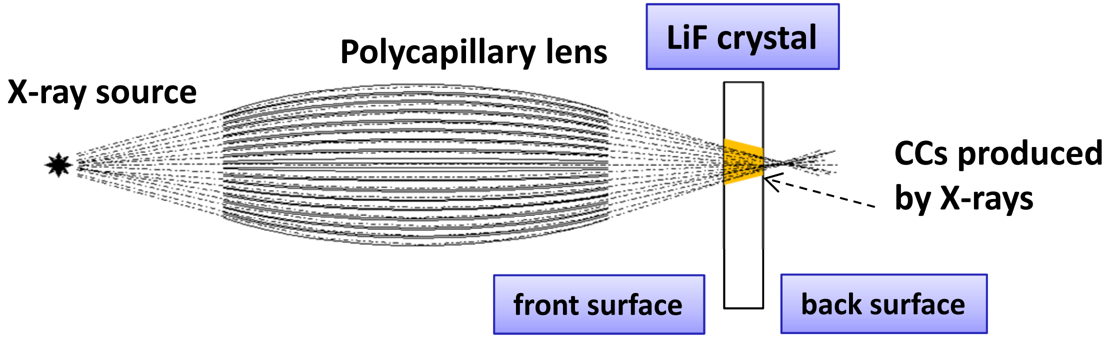

**Y profile – FWHM = 130 µm**

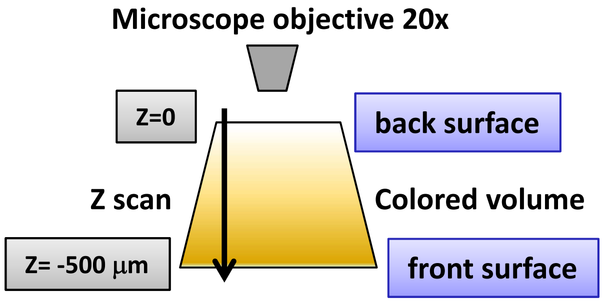



**X profile – FWHM = 122 µm**

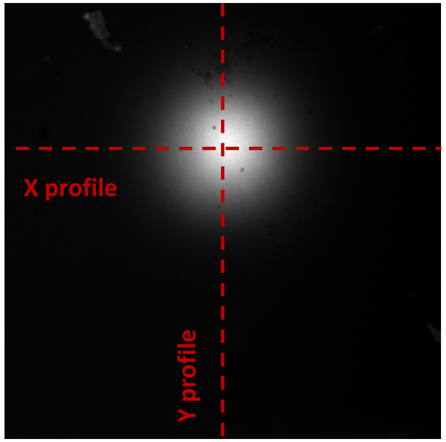
Focusing angle (measured) ~ 30 mrad

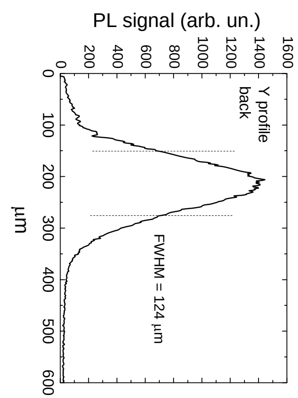
# Investigation by 3D confocal fluorescence microscopy of coloured LiF crystals by a focused X-ray beam



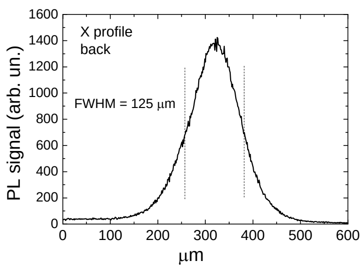




**Z=0** **back surface**



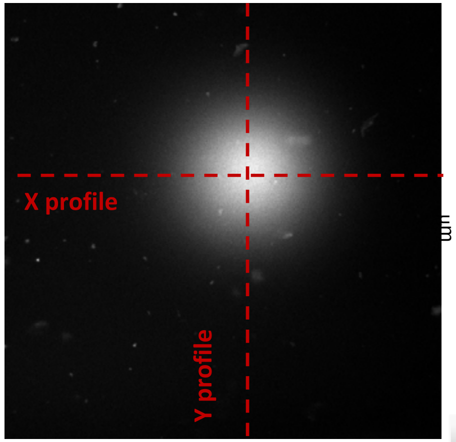



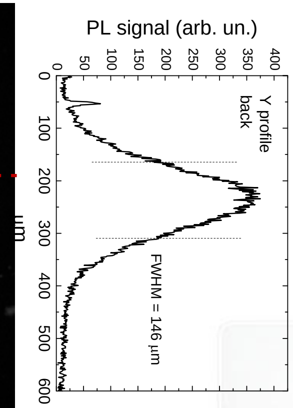

**Y profile – FWHM = 124 μm**



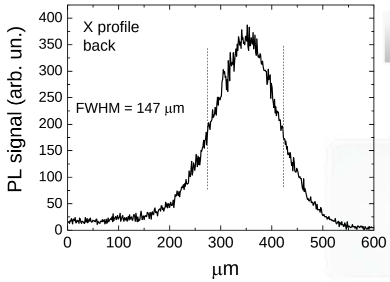

**X profile – FWHM = 125 μm**

**Z= -500 μm** **front surface**



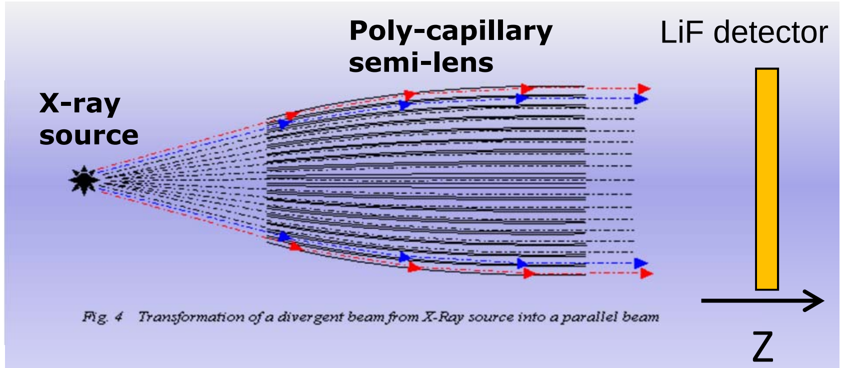



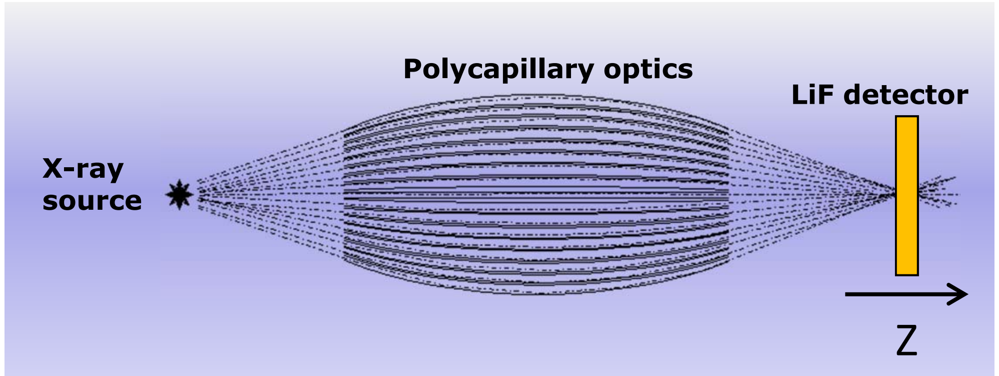

**Y profile – FWHM = 146 μm**

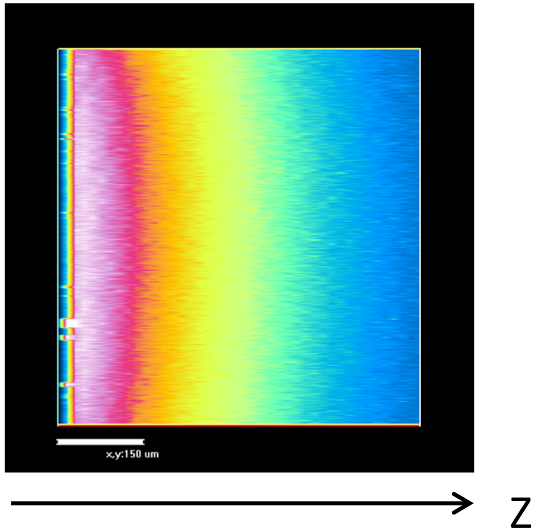



**X profile – FWHM = 147 μm**

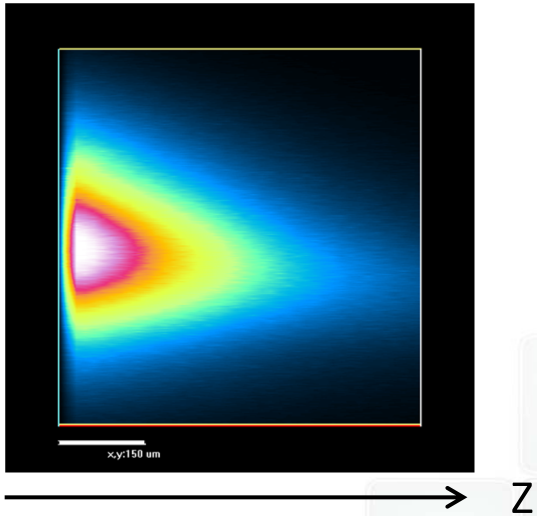
# CLSM Z distributions of CCs in a LiF crystal depth coloured by a X-ray beam



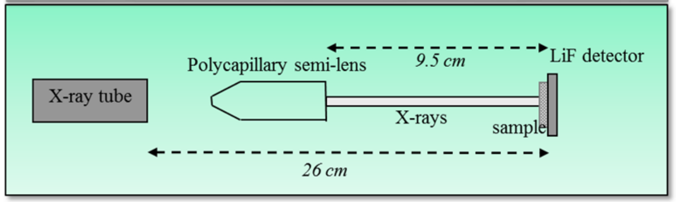

CLSM Z distributions of CCs in a LiF crystal depth of a irradiated LiF crystal by a parallel X-ray beam.

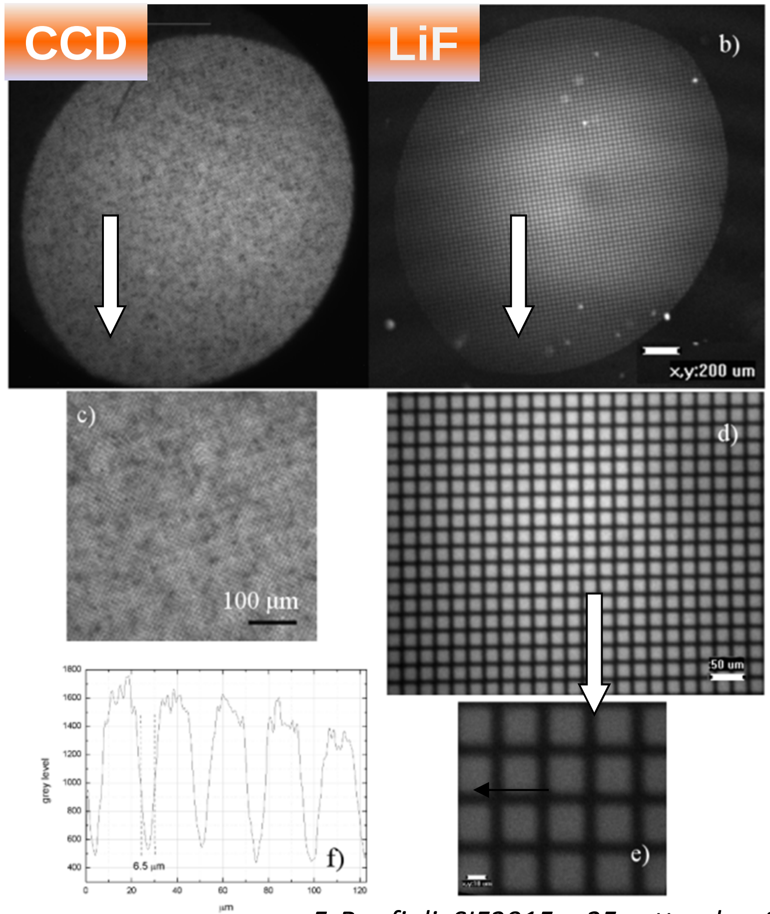



CLSM Z distributions of CCs in a LiF crystal depth of a irradiated LiF crystal by a focused X-ray beam.

# LiF detectors and X-ray polycapillary optics as a new approach for advanced X-Ray imaging

Experimental configuration for contact X-ray micro-radiographies on LiF detectors




**X-ray image of Au mesh 1000 lines/inch (hole width: 19 µm - wire width: 6 µm) acquired by a CCD camera and by a LiF crystal read by a CLSM system.**

CCD: Photonic Science - FDI 1.61:1, sensitive area of (4×3)mm² and (3.5×3.5) µm² pixel size, 12-bit

LiF: crystal (5x5x0.5) mm³

a, b) X-ray image of Au mesh acquired by a CCD camera and by a LiF crystal, respectively. The fluorescence image acquired by LiF detector, b), was read by the CLSM system.
c), d) Magnified images of CCD and LiF X micro-radiographies, respectively.
f) Magnified image of LiF X micro-radiograph and e) intensity profile along an horizontal line of the luminescent patterned image in LiF of f).

**The X-ray micro-radiography stored in the LiF crystal has been read by a CLSM in fluorescence mode**

The better quality of X-ray radiography on LiF detector with respect to the CCD one, in terms of resolution and contrast, is evident, although the signal digitalization (12 bits) is the same.

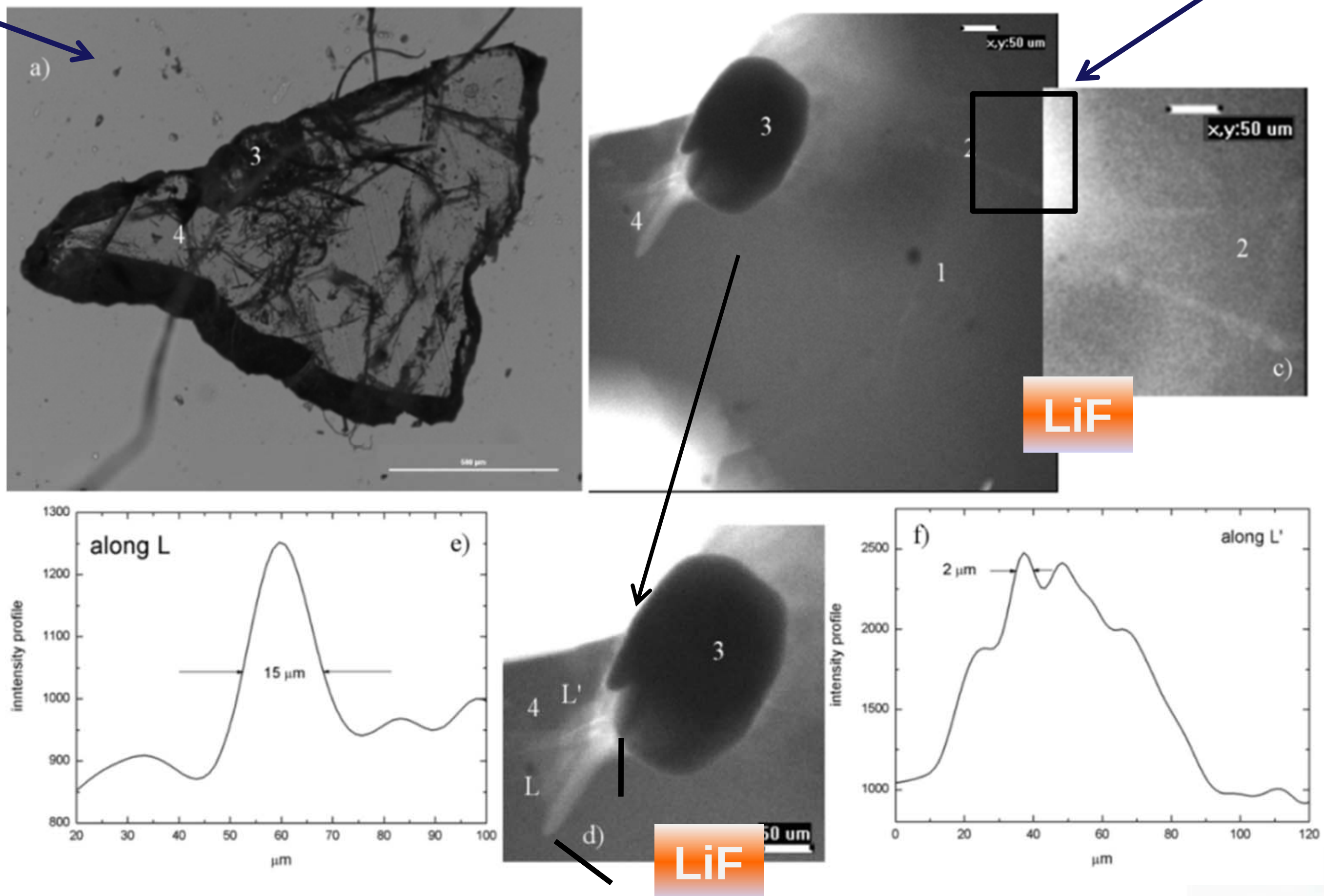

D. Hampai, S.B. Dabagov, G. Della Ventura, F. Bellatreccia, M. Magi, F. Bonfigli and R. M. Montereali, *High Resolution X-ray Imaging by Polycapillary Optics and Lithium Fluoride Detectors Combination*, Europhysics Letters 96 (2011) 60010p1-p4.

# Contact X-ray micro-radiographies of geological samples on LiF detectors

Sample: Cordierite section (140 mm) – Silicate with ideal composition $(Mg, Fe)_2 Al_4Si_5 O_{18x} (H_2O, CO_2)$.

**Optical image under white light illumination in transmission mode**

**X-ray micro-radiography stored in a LiF crystal and read by a CLSM in fluorescence mode**




D. Hampai, S.B. Dabagov, G. Della Ventura, F. Bellatreccia, M. Magi, F. Bonfigli and R. M. Montereali, *High Resolution X-ray Imaging by Polycapillary Optics and Lithium Fluoride Detectors Combination*, Europhysics Letters 96 (2011) 60010p1-p4.

## Conclusions

We presented **high performance X-ray imaging detectors** based on photoluminescence of CCs in LiF for X-ray imaging.

**High-resolution fluorescent images** stored in LiF crystal detectors of the **transmitted X-ray beam by a poly-capillary semi-lens** were obtained to characterize both propagation and transmission of X-rays through these optics. The peculiarity of LiF combined with the performances of the optical reading system enabled us obtaining the high spatial resolution images on a wide field of view, that is fundamental for the full optics characterization.

Confocal fluorescence microscopy was used as powerful tool for advanced optical reading of LiF detectors and 3D imaging reconstruction of X-ray image stored in LiF detectors.

High-resolution solid-state imaging detectors based on photoluminescent colour centres in lithium fluoride have been successfully used for **X-ray imaging performed by conventional X-ray tube combined with poly-capillary optics.**

**Contact X-ray micro-radiographies** of geological samples and test objects by means of a low divergent X-ray beam from a poly-capillary semi-lens with LiF crystals detectors have been successfully performed.

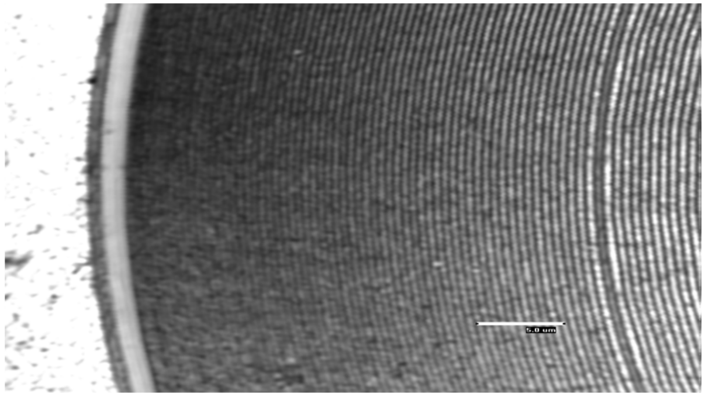
The peculiarities of LiF detectors, like **high spatial resolution over a large field of view, wide dynamic range, simplicity of use, and efficiency of the reading technique**, can be exploited for contact X-ray microscopy (soft and hard), compact X-ray imaging facility, X-ray optics characterization, X-ray source characterization, etc.

**Thank you for your attention**

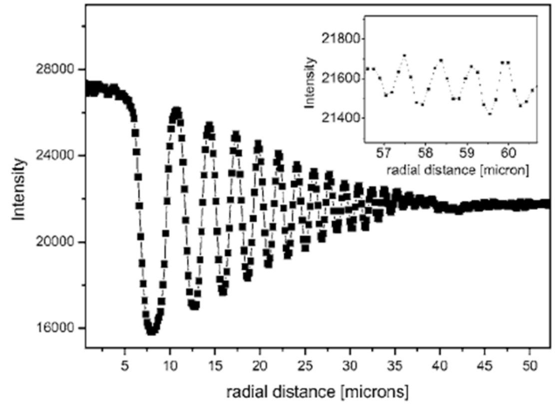
# Spatial resolution of contact X-ray micro-radiographies on LiF by advanced optical reading

## Optical reading by Confocal Laser Scanning Microscope

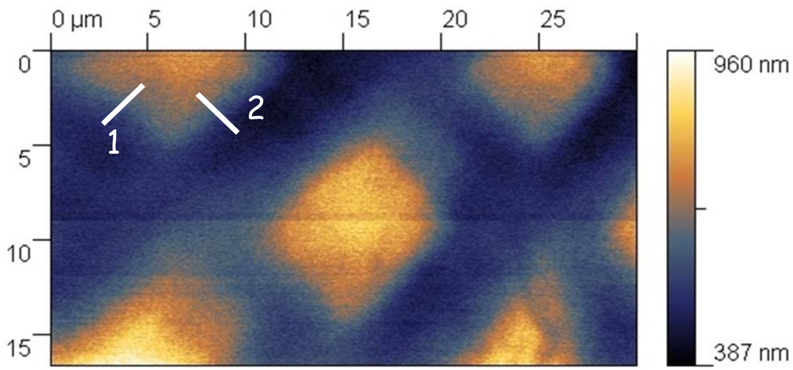
**~ 250 nm**
**Spatial resolution**



Confocal image of a contact X-ray micro-radiography of a Zone Plate stored in LiF film

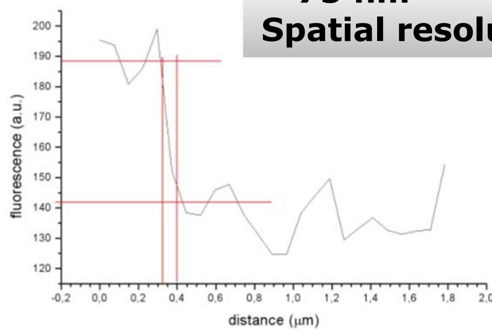



## Optical reading by Scanning Near Field Microscope




SNOM image of contact X-ray micro-radiography of a mask stored in a LiF film

**~ 75 nm**
**Spatial resolution**



# X-ray microradiographies on LiF detectors

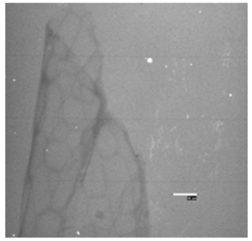
**X-ray sources -** EUV, soft and hard X-rays (20 eV – 30 keV)

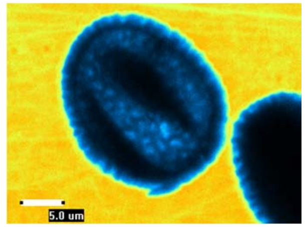
synchrotrons

X-ray lasers

laser plasma

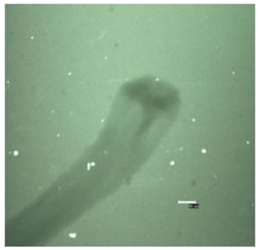
X-ray tubes

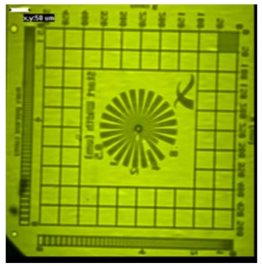


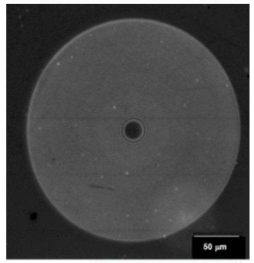



### Investigated Samples

- **biological samples** (*in vivo* cells, mosquito wings, pollens, leafs, human hairs, onion cataphils, etc.)
- **geological samples**
- **micro-nanostructures** (masks, Phase Fresnel Zone Plate PFZP, X-ray polycapillary optics. etc.)







**Irradiation technique:** lensless imaging, contact mode in absorption contrast, projection in phase contrast