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## New storage phosphor for X-ray microscopy for biological application

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### ABSTRACT

The new silverless radiophotoluminescence substances - a single alkaly- halide crystals (non-doped and doped by  $\text{In}^{+2}$ ) - are proposed and tested as X-ray image detectors. Their spatial resolution is not worse than  $2 \mu\text{m}$  and they allow detection of pictures in X-rays with a dynamic range of about 10 000. A stored X-ray image can be read out many times by photoluminescence measurements under UV excitation. The image can be erased completely by heating of phosphor at a temperature of  $400^\circ\text{C}$  during a half an hour. Unique combination of the resolution and the dynamic range of luminosity offers sample scope for x-ray microscopy under biology.

Keywords: X-ray microscopy, radiophotoluminescence, alkaly- halide crystals

### 1. INTRODUCTION

The phenomenon called a radio-photo-luminescence (RPL). is used usually for X-ray dose measuring. A silver-phosphate glasses<sup>1</sup> or  $\text{LiF}$ <sup>2</sup> are used as the registrating media. In the first case centers of luminescence are not destroyed under signal reading out and, in the second case,  $\text{F}_2$  defects in  $\text{LiF}$  are destroyed with reading out process. Action of Imaging Plates<sup>3</sup> widely used at present time, based on this effect too.

High detection efficiency (more than 80% for 8-20 keV photons), a wide dynamic range (about  $10^5$ ), a high count rate capability and other excellent parameters of the Imaging Plates are very useful for registration of X-ray pictures in X-ray diffraction studies, small angle scattering and so on. Unfortunately, the spatial resolution of the Imaging Plates, which is about  $150 \mu\text{m}$  now, and, in principle, may be improved up to several tens of microns, confines their use for the purposes of X-ray microscopy and holography.

During the past few years the alkali halide crystals doped with ions of heavy metals have been extensively studied as a material for image storing. These crystals have attracted the attention to the optical information recording<sup>4</sup>. A single memory cell in these crystals is the color or luminescence center, which has a very small size and allows to record the information with a high spatial resolution.

In this work we report about the media with the stable centers, which arise in alkaly- halide single crystals doped by the ions of  $\text{In}^{+2}$  when they are exposed by X-ray<sup>5</sup>. These centers can be used as single memory cells.

## 2. EXPERIMENT

The authors have proposed and examined the silverless RPL substances of modified alkali-halide crystals <sup>6</sup> as an X-ray area detector. A stored X-ray image is read out by measuring the intensity of photoluminescence with a wavelength of ~ 530-560 nm under the excitation by ultraviolet light with a wavelength of about 365 nm. In opposite to the Imaging Plates, the stored image is not erased upon reading, and therefore the reading can be repeated many times. For a complete erasure of the image the substances must be heated for 0.5 hour at a 400 °C temperature.

In recent years we worked on more wide usage of RPL phenomenon for the X-ray image registration. Single crystals of alkali-halide crystals were used in our studies. Effect of F<sub>2</sub> center creation in pure LiF is used, and effect of stable centers creation in single crystals doped by In<sup>+2</sup> is used too. In the first case the latent image is formed, which can be irradiation by light of 440 nm wavelength. Luminescence of 560 nm wavelength appears and latent image erased, which is like to the imaging plate case. In the second case the 520-560 nm wavelength luminescence (for different type of crystals) is observed under excitation by ultraviolet of 360- 380 nm wavelength. An X-ray image can be read out many times, because at the image is not erased under the reading process. To erase an X-ray image a single crystal must be heated up to a temperature of 400 °C during a half-hour period. For examination of these area detectors, synchrotron radiation (SR) from the VEPP-3 storage ring of the Budker Institute of Nuclear Physics was used. For soft (1-5 keV) X-ray, the investigations were carried out at the X-ray lithography station <sup>7</sup>. The X-ray beam from the VEPP-3 storage ring was fell on the crystal through filters and X-ray mask that contains the pattern of a 0.9- $\mu$ m-thick gold absorber on a 2- $\mu$ m-thick silicon membrane. The radiophotoluminescence substance is 10 mm distant from the X-ray mask. Exposures of crystals were done at 1.2 GeV energy of electrons in the storage ring. Synchrotron radiation from the bending magnet was used with a critical wavelength of 1.26 nm. The processes of recording the information are based on the creation the luminescence centers in crystal lattice by ionizing radiation with the subsequent destruction of F-centers.

Preliminary results indicate that the spatial resolution of the substances is not worse than 2 microns. Examples of luminescent pictures produced by the registered X-ray images in radiophotoluminescence substance of modified LiF are presented in the Fig.1. The width of the narrow black lines on the pictures corresponds to 2  $\mu$ m of the X-ray mask pattern.

The dynamic range of these area detectors is illustrated by the Fig.2, where the luminescent intensity versus the absorbed dose of X-rays is presented. The dependence of the luminescence intensity for after-exposure measurements on non-doped single crystals of LiF is plotted by "x", the dependence for the same samples measured after a half-year delay is plotted by "+", the dependence for luminescence band caused by trace impurities under UV excitation of 365 nm wavelength is plotted by  $\square$ . As it is shown in the Fig.2, the dynamic range is not less than four orders of magnitude. The similar study of these substances with 6-30 keV photons at the X-ray microscopy station of the VEPP-3 <sup>8</sup> perform also that the dynamic range is about 5 000.

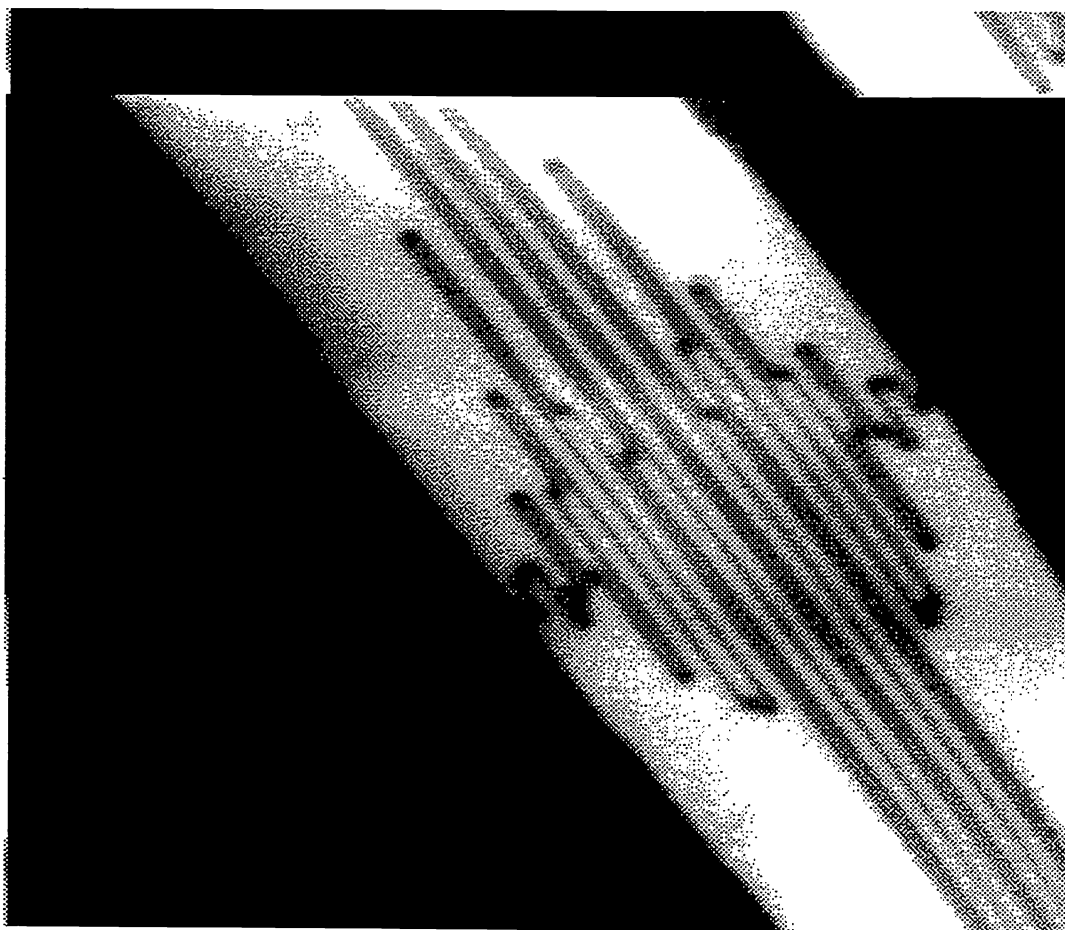


Fig.1. Example of patterns of X-ray images that was read out by the luminescence registration from radiophotoluminescence substances of modified LiF (television system provided X-ray image obtained with the use of an X-ray mask)

### 3. EXAMPLES OF X-RAY IMAGES

The results on the investigation of these image plates (micron spatial resolution, wide dynamic range of registration) stimulated experiments on studying the internal structure of low-contrast biological samples, which did not provide images of enough quality with the use of conventional X-ray films.

To obtain X-ray images a shadow printing was used. Spatial blur in the image of the object structure due to wave nature of X-rays is:

$$\delta \approx \sqrt{\lambda \cdot L}$$

where  $\lambda$  is a wavelength and  $L$  is a distance between image detector and studied structure. Thus, if it is needed to obtain X-ray image with the spatial resolution as high as possible ( $\sim 1 \mu\text{m}$ ), one should use objects with a thickness of about 3 mm with soft X-rays ( $\lambda \sim 0.3 - 1 \text{ nm}$  wavelength).

For more thick objects the exposures were done at X-ray topography station <sup>8</sup> with X-rays of 6 - 30 keV range.

The object being studied were placed directly on the surface of the LiF crystal. Exposure time  $T_{exp}$  was varied from 10 seconds to 10 minutes. So for a mosquito  $T_{exp}$  is about 50 s, for buds  $T_{exp}$  is about 10 min (for the 1.2 GeV energy of electrons in the storage ring), and for frogs  $T_{exp}$  is about 10 s (for the 2.0 GeV energy of electrons in the storage ring).

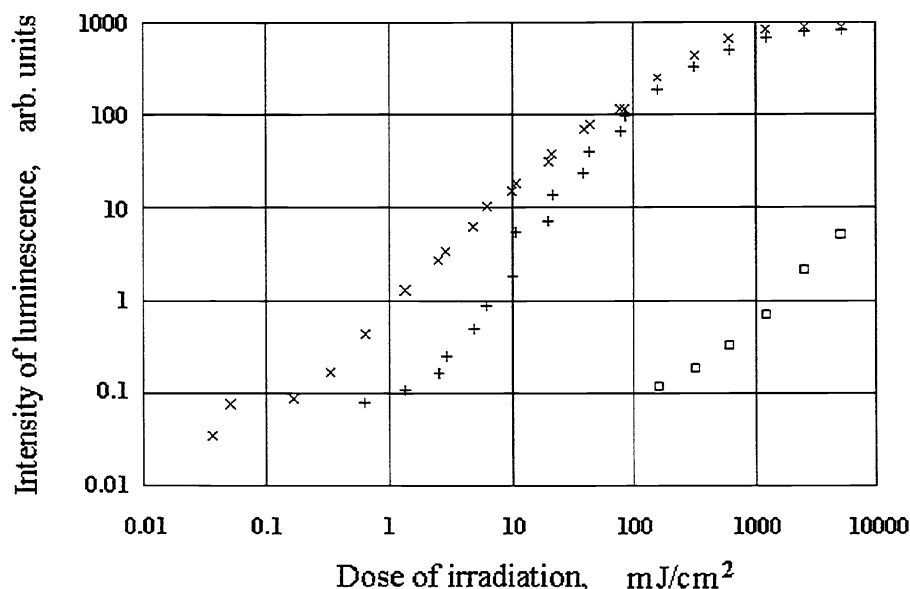


Fig.2. The dependence of the luminescence intensity versus the absorbed dose of X-ray irradiation; × - after exposure measurements for non-doped single crystals of LiF, + - measurements for the same samples after a half-year delay, □ - measurements for luminescence band caused by trace impurities under UV excitation of 365 nm wavelength.

The X-ray images were registered by three ways: the first - by transfer of the magnified image onto photo film, the second - by reading out by a photomultiplier in a count mode with scanning, the third - by reading out by television system. The first way allows to obtain information about object in a relatively short time, but a narrow dynamic range of photofilm registration (less than  $10^2$ ) leads to partial loss of information. This essentially limits abilities of this method. The second way due to high sensitivity and wide dynamic range ( $\sim 10^5$ ) of registration by a photomultiplier allows to read out an X-ray image without information losses, but it have some disadvantages - it takes more long reading time due to scanning, the additional (reference) registration channel for monitoring of excitation light intensity, and a computer with a sufficient memory resources and a data acquisition system are needed. A television system with registration of image in computer memory is most suitable for this purpose. The system must have the ability to register of image

with dynamic range at least  $10^4$ . Our devices have not these parameters and most of the information were obtained by visual study of the images.

#### 4. CONCLUSION

The obtained results show that alkali-halide single crystals (doped by  $\text{In}^{+2}$  and also non doped ones) may be promising medium for the registration of X-ray pictures in different experiments<sup>9</sup>. They are tested as X-ray image detectors. Their spatial resolution is not worse than  $2\ \mu\text{m}$  and they allow to register the X-ray pictures with a dynamic range of about 10 000. The minimum value of the sensitivity is  $20\ \mu\text{J}/\text{cm}^2$  that is in the several orders higher than the sensitivity of negative X-ray resists but considerably lower ( $10^3$  times) than the sensitivity of X-ray films. It seems, the most interesting is the use of LiF single crystal, doped by In, because of the ionizing radiation creates centers of luminescence, which are not destroyed upon reading out process. Therefore the reading can be repeated many times for low light intensities detecting. No degradation of the image luminescence intensity is observed after one-year keeping of these samples. These detectors can be used as the image detectors many times because the stored images can be erased completely by heating the substances up to a temperature of  $400\ ^\circ\text{C}$  during a half-hour time.

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