X-ray image detectors with a micron spatial resolution

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Abstract

The new silverless radiophotoluminescence substances - a LiF single crystals non-doped and doped by In $^{+2}$ - are proposed and tested as X-ray image detectors. Their spatial resolution is not worse than 2 μ m and they allow to register the X-ray pictures with a dynamic range of about 10 000. A stored X-ray image can be read out many times by photoluminescence registration under UV excitation. The image can be erased completely by heating substances up to a temperature of 400 0 C during a half-hour time.

X-ray images of some biological objects are demonstrated.

I. INTRODUCTION

X-ray absorption and luminescence in visible region take place simultaneously in the luminescent techniques used usually for X-ray image registration. However, these phenomena can be time-separated and secondary luminescence can be caused by ultraviolet irradiation. This phenomenon is called a radio-photo-luminescence (RPL). Usually RPL is used for dose measuring. A silver-phosphate glasses [1] or LiF [2] are used as registrating media. In the first case centers of luminescence are not destroyed under signal reading out and, in the second case, F₂ defects in LiF are destroyed with reading out process. Action of Imaging Plates [3] 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 10⁵), 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 µm now, and, in principle, may be 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. A single cell of the memory 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. If ionization radiation creates in the crystal lattice a pair of centers - impurity hole center (Me) and F-center, the information can be read out by means of measuring the recombination luminescence when the crystal is exposed to excitation radiation such as the visible light. In the case

of such information recording, the stored data will be lost after the reading out process or when the crystal is kept at room temperature in visible light.

In this work we report about the new stable centers, which arise in LiF single crystals doped by the ions of In +2 when they are exposed by X-ray. These centers can be used as single memory cells.

II. EXPERIMENT

The authors have proposed and examined, as an X-ray area detector, the silverless RPL substances of modified LiF [4]. A stored X-ray image is read out by measuring the intensity of photoluminescence with a wavelength of $\sim 530-560$ 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 reading out can be repeated many times. For a complete erasure of the image the substances must be heated for 0.5 hour at a 400 $^{\circ}$ C temperature.

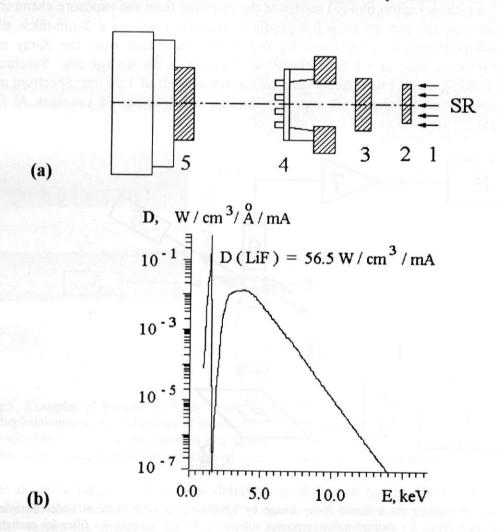


Fig.1. (a) The scheme for testing of X-ray image registration. 1 - X-ray beam from VEPP-3 storage ring, 2 - the 28- μ m-thick beryllium filter, 3 - the 13- μ m-thick kapton film, 4 - the X-ray mask (a 2- μ m-thick silicon membrane with the pattern of a 0.9- μ m-thick gold absorber), 5 - the radiophotoluminescence substance. (b) Spectral distribution of absorbed power in the LiF crystal under SR exposure through an additional 58 μ m-thick Al foil at the VEPP-3 storage ring operated at 1.2 GeV energy.

In recent years we lead works on more wide usage of RPL phenomenon for the X-ray image registration. Single crystals of LiF were used in our studies. Effect of F₂ center creation in pure LiF is used, and effect of stable centers creation in single crystals doped by In ⁺² is used too. In the first case the latent image is formed, which can be light out by irradiation 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 nm wavelength luminescence is observed under excitation by ultraviolet of 380 nm wavelength. An X-ray image can be read out many times, as image is not erased under reading out. To erase an X-ray image a single crystal must be heated up to a temperature of 400 °C during a half-hour time.

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 [5]. The scheme of the test image registration is shown in the Fig.1a. There is the X-ray beam (1) from the VEPP-3 storage ring. A 28-μm-thick beryllium filter (2) separates high vacuum chamber of the storage ring from the SR beamline, and a 13-μm-thick kapton film (3) separates the beamline from the exposure chamber. An X-ray mask (4) contains the pattern of a 0.9-μm-thick gold absorber on a 2-μm-thick silicon membrane. The radiophotoluminescence substance (5) is 10 m 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. Spectrum of SR dose absorbed in the LiF crystal under SR exposure through an additional 58 μm-thick Al foil is shown in the Fig. 1b.

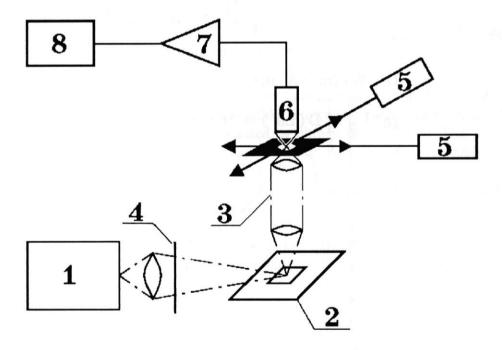


Fig.2. The scheme for reading out a stored X-ray image by luminescence measurements under ultraviolet excitation: 1 - mercury lamp, 2 - radiophotoluminescence substance, 3 - microscope, 4 - filter for excitation light, 5 - scanning system, 6 - photomultiplier, 7 - preamplifier, 8 - acquisition system with a computer.

The processes of recording the information are based on creation the luminescence centers in crystal lattice by ionizing radiation with the subsequent destruction of F-centers. The scheme for reading out a stored X-ray image by the luminescence registration under the ultraviolet excitation is

shown in the Fig.2. A radiophotoluminescence substance (2) is illuminated by a mercury lamp (1) through a filter (4). The luminescent picture is observed by a microscope (3) through a filter which cuts off the excitation light. The picture formed by luminescent light can be registered either by a photofilm or by a scanning system (5) with a photomultiplier (6). In the case of reading out by PMT, a signal comes through preamplifier (7) and acquires by computer (8).

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.3a. The width of the narrow black lines on the pictures corresponds to 2 µm of the X-ray mask pattern. Intensity of a luminescence light along the scan line, crossing a 2 µm pattern in the X-ray image, is shown in Fig.3b. Space resolution of the scanning system corresponds to a 5 steps per micron.

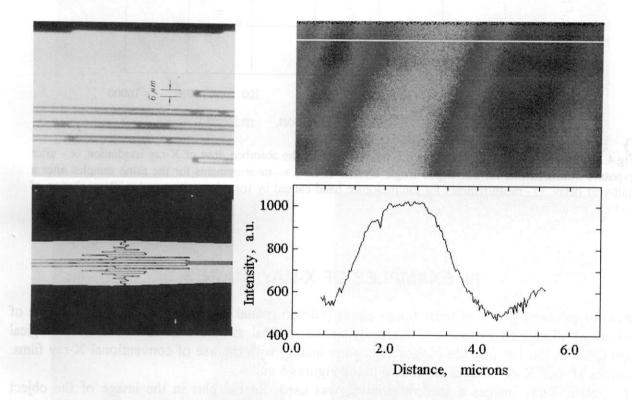


Fig.3. Examples of patterns of X-ray images that were read out by the luminescence registration from radiophotoluminescence substances of modified LiF; (a) - microphotographs of an X-ray image obtained with the use of an X-ray mask, (b) - intensity of a luminescence light along scan line, crossing a 2 μ m pattern in the X-ray image. Space resolution of the scanning system corresponds to a 5 steps per micron.

The dynamic range of these area detectors is illustrated by the Fig.4, where the luminescent intensity versus the absorbed dose of X-rays is presented. The dependence of the luminescence intensity versus the absorbed dose of X-ray irradiation 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 "\(\sigma\)". As it can be seen from the Fig.4, 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 [6] showed also that the dynamic range is about 5 000.

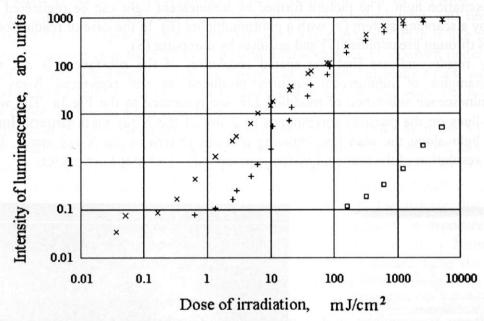


Fig.4. The dependence of the luminescence intensity versus the absorbed dose of X-ray irradiation; × - after exposure measurements for non-doped single crystals of LiF, + - measurements for the same samples after a half-year delay, \Box - measurements for luminescence band caused by trace impurities under UV excitation of 365 nm wavelength.

III. EXAMPLES OF X-RAY IMAGES

Results on 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. Examples of such X-ray images are shown in the Figures 5 and 6.

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 λ 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 (~1 µm), one should use objects with a thickness of about 3 mm with soft X-rays (λ ~ 0.3 - 1 nm wavelength). For more thick objects exposures were done at X-ray topography station [6] with X-rays of 6 - 30 keV range.

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

The X-ray images were registered by two ways: the first - by tranfer of the magnified image onto photo film, the second - by reading out by a photomultiplier in a count mode with scanning. The first way allows to obtain information about object in a relatively short time, but a narrow dynamic range of photofilm registration (less than 10²) leads to partial loss of information. This essentially limits

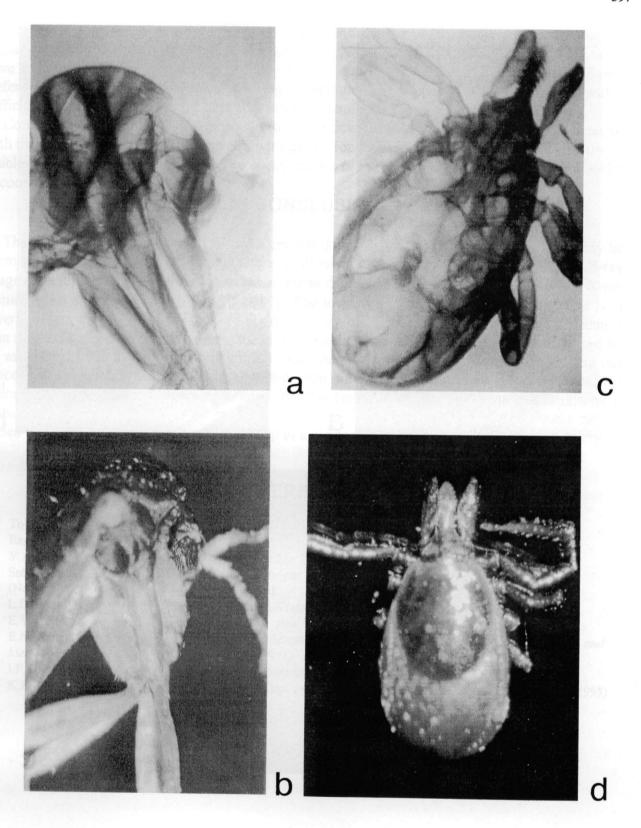


Fig.5. Comparison of the images (a,c), obtained by the X-ray image detector with the use of 0.3 - 1 nm wavelength synchrotron radiation, and microphotographs (b,d), obtained with the use of a visible light; (a,b) - mosquito, (c,d) - tick.

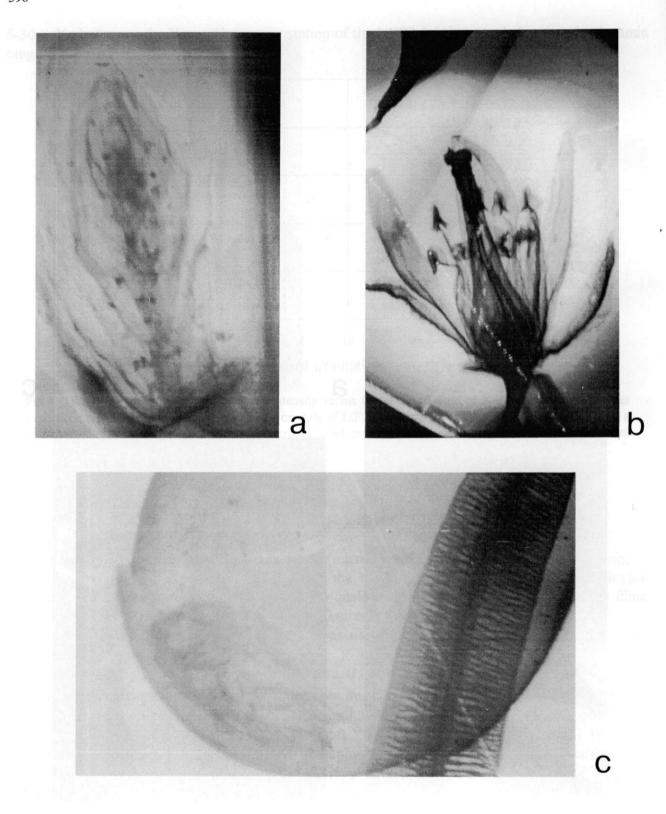


Fig.6. The images, obtained by the X-ray image detector with the use of 0.3 - 1 nm wavelength synchrotron radiation; (a) - the bud, (b) - the flower bud, (c) - the spider cocoon.

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 loss, but it have some disadvantages - it takes more long reading out time due to scanning, it takes additional (reference) registration channel for monitoring of excitation light intensity, it takes computer with a sufficient memory resources and a data acquisition system.

Comparison of the images of a mosquito (a) and a tick (c), obtained by the X-ray image detector with the use of 0.3 - 1 nm wavelength SR, with the microphotographs (b,d), obtained with the use of visible light, is shown in the Fig. 5. The X-ray images of a bud (a), a flower bud (b) and a spider cocoon (c) are shown in the Fig. 6.

IV. CONCLUSION

The obtained results show that LiF single crystals doped by In ⁺² and also non doped ones may be promising for the registration of X-ray pictures in different experiments [7]. They are tested as X-ray image detectors. Their spatial resolution is not worse than 2 µm and they allow to register the X-ray pictures with a dynamic range of about 10 000. The low edge of sensitivity is 20 µJ/cm ² that is in several orders higher than sensitivity of negative X-ray resists but considerably lower (10 ³ times) than sensitivity of X-ray films. It seems, the most interesting is the use of LiF single crystal, doped by In, as ionizing radiation creates centers of luminescence, which are not destroyed upon reading out process. Therefore the reading can be repeated many times for detecting low light intensities. No degradation of the image luminescence intensity after one-year keeping these samples is observed. 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 ^o C during a half-hour time.

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