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In-depth visualisation of Triglycine-Sulfate's domain structure

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The domain structure is an important characteristic of ferroelectrics. One of the most intensively studied ferroelectrics is triglycine sulfate (TGS). Although many methods have been developed for the observation of the domain structure in TGS, it still cannot be visualized routinely.

We suggest a new method for observation of the domains in TGS, in particular digital in-line holographic microscopy. A big advantage of the new method is its ability to extract information about the change of the domain structure in depth from one digital hologram only.

The domain structure of TGS monocrystals doped with Yb was visualized by digital holographic microscopy for the first time.

Keywords: digital in-line holographic microscopy, DIHM, Triglycine sulfate, TGS, domain structure, Yb

INTRODUCTION

Triglycine sulfate $(NH_2CH_2COOH)H_2SO_4$ (TGS) is one of the most intensively investigated ferroelectrics because of its excellent ferroelectric and pyroelectric properties. Single crystals of TGS are considered to be the most suitable material for developing pyroelectric infrared sensors [1-3]. Hoshino et al. [4] described in detail the crystal structure of pure TGS. An extensive list of related references is given by Nakatani [5].

The basic principle of holography was introduced by Gabor [6], who invented the in-line holography. The new principle was used soon afterwards with visible light [7]. Its power has been revealed after discovery of the laser, which delivered much higher contrast in the holograms, and later with the implementation of the CCD camera as a recording device. This type of holography, which uses CCD (or CMOS) camera to record a hologram, is called digital in-line holography.

Digital in-line holographic microscopy (DIHM) is a relatively new microscopic technique showing many advantages over conventional microscopy. Digital holography is a non – destructive, marker – free imaging method offering a full field of view. Unlike conventional light microscopy, in-line holographic microscopy can give visual information about an object not only in the focal plane. From the captured hologram the real image is reconstructed by means of numerical deconvolution. The method provides virtual focusing throughout the depth of the sample from a single hologram as quantitative information about the intensity and the phase distribution.

The advantages of digital holographic microscopy listed above encouraged the authors to investigate its suitability for observation of ferroelectrics domains in TGS.

The present methods to observe the domain structure of ferroelectrics are not easily applied. As a common feature they need preliminary preparations of the object and the observing system and are generally expensive. We suggest an application of digital holographic microscopy as a quick and inexpensive, but very sensitive method to directly observe the ferroelectrics domains and determine their dimensions.

Digital in-line holographic microscopy (DIHM) was used for characterization of domain structure of TGS doped with Yb.

EXPERIMENTAL

A digital in-line holographic microscope (DIHM) was developed at the Agricultural University of Plovdiv. The light source is a diode laser (Lasiris) with wavelength of 673.2nm and

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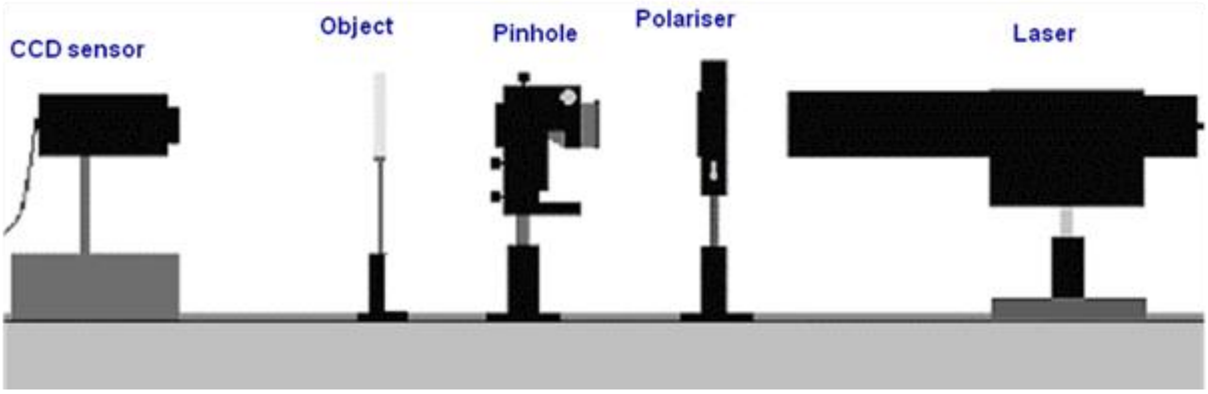

Fig. 1. Optical set-up of the digital in-line holographic microscope

output of 6.98 mW. The intensity of the illumination, focused onto pinhole is controlled via a polarizer (Fig. 1).

The spherical wave emerging from the pinhole illuminates the object. The perturbed by the object and the unperturbed wave interfere and are recorded as a hologram on a CCD sensor and stored in a computer. The intensity and the phase of the object are reconstructed by numerical computer calculations using the back light propagation method of HoloRec3D: A free Matlab toolbox for digital holography [8].

TGS single crystals, doped with Yb, were grown by the dynamical method in the ferroelectric phase [9]. All samples used in these experiments were grown from aqueous solution. The dopant salt was in the form of sulfate. The concentration of $Yb_2(SO_4)_3$ in the solution was 1 % and 4 % for the different monocrystals. The crystals of TGS show a clear cleavage plane perpendicular to its ferroelectric axis.

The samples for the present investigations were plates, cleaved perpendicular to the polar axis [010] from clear regions far from the seeding area. The principal optical axis X coincides with the polar crystal axis. According to the notation of Damen et al. [10] our experiments were performed in the x(yy) geometry.

RESULTS AND DISCUSSIONS

We report four examples of application of the DIHM for TGS's domain structure investigation. The four holograms and their amplitude reconstructions are given in Figs. 2 - 5. Figs. 2 – 3 show results for two different samples of TGS monocrystal, doped with 1% Yb in the solution of growth, while Figs. 4 – 5 show results for two samples of TGS monocrystal, doped with 4% Yb in the solution of growth.

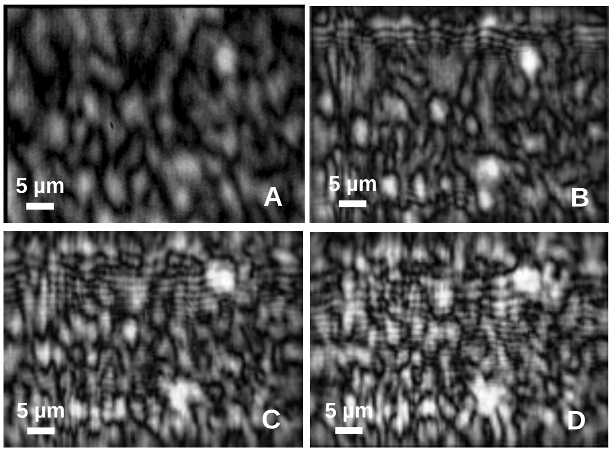

Fig. 2. A – a digital hologram of TGS monocrystal, doped with 1% Yb in the solution of growth; B – reconstruction of the intensity of hologram 2A on the surface of the sample, C - reconstruction of the intensity of the hologram 2A at 1 μm depth inside the TGS sample and D - reconstruction of the intensity of the hologram 2A at 2 μm depth inside the TGS sample.

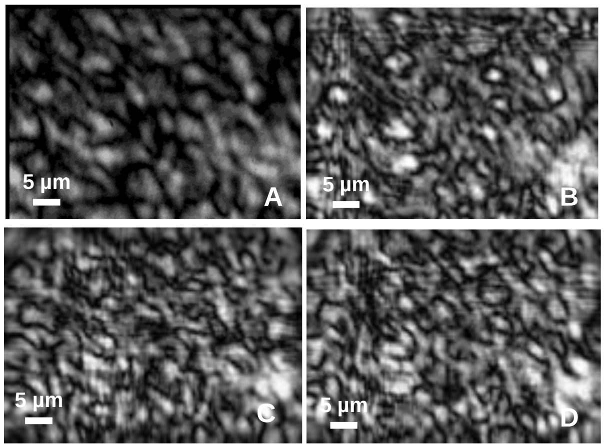

Fig. 3. A – a digital hologram of TGS monocrystal, doped with 1% Yb in the solution of growth; B – reconstruction of the intensity of hologram 3A on the surface of the sample, C - reconstruction of the intensity of the hologram 3A at 1 μm depth inside the TGS sample and D - reconstruction of the intensity of the hologram 3A at 2 μm depth inside the TGS sample.

It is important to note that using samples in the form of plates with thickness of 1 mm, cleaved perpendicular to the polar axis [010] of the monocrystal, we should observe the cross-sections of the domains in the TGS material. It is known that the domains in TGS are parallel to its ferroelectric axis [11].

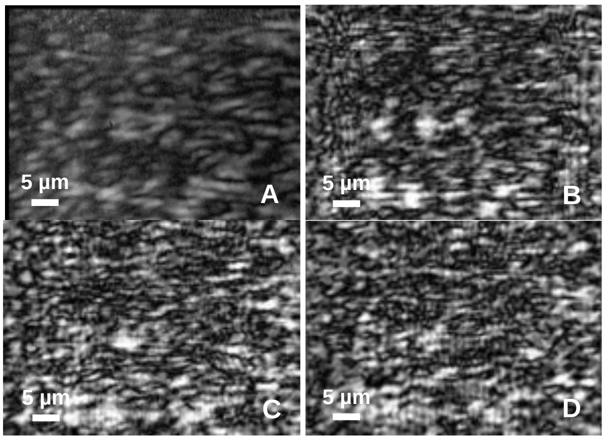

Fig. 4. A – a digital hologram of TGS monocrystal, doped with 4% Yb in the solution of growth; B – reconstruction of the intensity of hologram 4A on the surface of the sample, C - reconstruction of the intensity of the hologram 4A at 1 µm depth inside the TGS sample and D - reconstruction of the intensity of the hologram 4A at 2 µm depth inside the TGS sample.

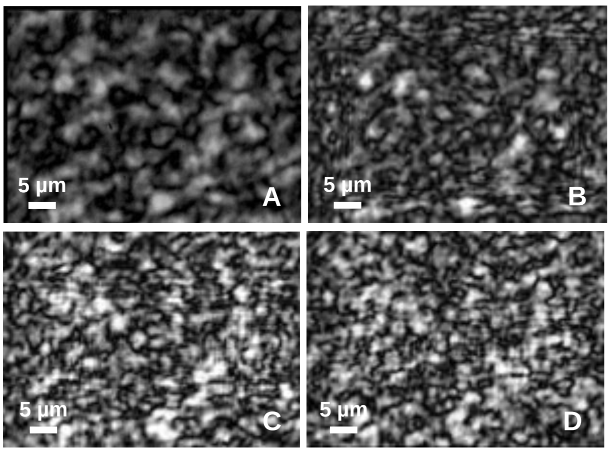

Fig. 5. A – a digital hologram of TGS monocrystal, doped with 4% Yb in the solution of growth; B – reconstruction of the intensity of hologram 5A on the surface of the sample, C - reconstruction of the intensity of the hologram 5A at 1 µm depth inside the TGS sample and D - reconstruction of the intensity of the hologram 5A at 2 µm depth inside the TGS sample.

Figs. 2 – 5 show that the diameters of the single domains (cross sections) in TGS monocrystals doped with Yb vary between 2 µm and 4 µm for the thickness of the cross section and between 3 µm and 7 µm for the length of the cross section. It was established that the domains became thinner and longer with increasing the concentration of the dopant.

CONCLUSIONS

The domain structure of TGS doped with Yb was visualised by digital in-line holographic microscopy for the first time. The DIHM was employed without any preliminary preparation of the triglycine sulfate samples. It was established that the diameters of the single domains (cross sections) in TGS monocrystals doped with Yb vary between 2 µm and 4 µm for the thickness of the cross section and between 3 µm and 7 µm for the length of the cross section. It was established that the size of the domains changes with increasing the concentration of the dopant – they became thinner and longer.

The DIHM was successfully used for observation of the TGS domains in depth. The digital virtual focusing inside the TGS crystals doped with Yb show the same dimensions for the ferroelectric domains as observed on the top surface of the samples.

We conclude that DIHM is a promising novel technique for domain visualisation and can be easily used for structural studies in all transparent ferroelectrics.

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ВИЗУАЛИЗАЦИЯ НА ДОМЕННАТА СТРУКТУРА НА ТРИГЛИЦИНСУЛФАТ В ДЪЛБОЧИНА

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(Резюме)

Доменната структура е важна характеристика на сегнетоелектриците. Един от най-интензивно изучаваните сегнетоелектрици е триглицинсулфата (ТГС). Въпреки, че много методи са разработени за наблюдаване на доменната структура в ТГС, тя все още не може лесно да бъде визуализирана. Ние предлагаме нов метод за наблюдение на доменната структура в ТГС, по-точно цифрова линейна холографска микроскопия. Голямо предимство на новия метод е възможността да се извлича информация за промяната на доменната структура в дълбочина само от една цифрова холограма. За първи път доменната структура на монокристали от ТГС легирани с Yb е визуализирана с цифрова холографска микроскопия.