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Study of spatial resolution of YAG:Ce cathodoluminescent imaging screens

Petr Schauer *, Jan Bok

Institute of Scientific Instruments of the Academy of Sciences of the Czech Republic, v.v.i., Department of Electron Microscopy, Kralovopolska 147, CZ-61264 Brno, Czech Republic

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ABSTRACT

The aim of this paper is to find spatial resolution of YAG:Ce single crystal cathodoluminescent imaging screens at primary electron energies in the range from 20 to 100 keV using theoretical simulation as well as the experimental method. Calculations have been based on the MC model for energy distribution of excited electrons. Measurement of the spatial resolution was realized using the sharp edge projection method. As the projection object, the silicon single crystal plate with the hole made by the anisotropic etching was prepared and used. The edge of this object was examined at the magnification of up to 125,000×. For the edge projection method, the experimental system with the screen specimen cartridge and with the light-microscopic module using the magnifying objective and the CCD camera has been constructed and used. The simulated as well as experimental results have been processed and are presented in the form of line spread function (LSF). The resulted image qualities were quantified using modulation transfer function (MTF). Finally, the spatial resolutions of YAG:Ce single crystal imaging screens were determined as the number of lines per mm for the contrast of 50% and primary electron beam energies of 20, 60 and 100 keV.

1. Introduction

Cerium activated single crystals of yttrium aluminum garnet (YAG:Ce) are sought more and more as for materials for cathodoluminescent (CL) imaging screens in TEM [1]. Owing to their well-defined optical properties, including the homogeneity of the CL emission and also due to the possibilities of quite precise and even complex shaping, YAG:Ce single crystal screens can be applied as very small elements, which provide a very small and relatively perfect image intended for further processing [2]. At such use of YAG:Ce, unlike the utilization in scintillation detector systems, there are no high demands for the speed of the imaging element. In addition to the CL efficiency, the spatial resolution of CL screens is the most important characteristic in such an application.

During the last years, only a few papers dealt with the spatial resolution of imaging screens, but none of them studied the resolution of single crystal screens. Cavourase et al. [3] evaluated YAG powder screens excited by X-rays at the energy of 60 keV. The objects of interest were screens employed in medical diagnostic radiology. From earlier articles, the study of Nishi et al. [4] dealing with an experimental determination of the spatial resolution using observation of luminous broadening is notable. Unfortunately, the study is without simulation support, and moreover it is for electron beam excitation energy as high as 0.5–2 MeV. A simulation of elastic and inelastic multiple scattering in a thin YAG single crystal attached on a glass plate has been done by Kotera and Kamiya [5]. However, only basic primary processes were included in their Monte Carlo model. In addition, the simulation had not been done for electron beam energy below 100 keV in their paper. Even more articles related to the screen resolution in connection with CCD imaging have been published, but either monolithic material was not investigated or electron beam excitation energies less than 80 keV were not used in these articles. For example, Fan and Ellishman [6] have presented optimization of thin-foil based P20 powder phosphor screens for CCD imaging in TEM at the electron beam energy of 80–400 keV, and theoretical support is based only on a simple simulation of the electron trajectories.

The aim of this paper is to find the resolution of YAG:Ce single crystal CL screens at the primary electron beam energies (excitation energies) in the range of 20–100 keV using theoretical simulation as well as an experimental method.

2. Simulation of the spatial resolution of imaging screens

It is very difficult to interpret the spatial resolution from experimental results without support by a calculation. And vice versa, it is courageous enough to interpret spatial resolution without experimental support. A good starting step for the calculation of the CL spatial resolution of the YAG:Ce may be the use of the Monte Carlo (MC) model of the electron interaction with the matter.

* Corresponding author. Tel.: +420 541514313; fax: +420 541514404.
E-mail address: Petr@ISIBrno.Cz (P. Schauer).

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The MC model used for simulation of distribution of deposited excitation electrons was based on the single scattering utilizing the screened Rutherford cross-section and Bethe slowing down approximation [7]. Also secondary processes associated with diffusion of excited electrons were included in the model. When applying the MC model, it should be also considered whether it is necessary to include diffusion of the emitted light. Effect of light diffusion essentially depends on the surface finishing and thickness of the screen. If the screen in the shape of a disc with the thickness of about 100 μm is studied (better would be thinner, but it is practically impossible), and if both flat surfaces of the screen are as much polished as possible, light diffusion can be expected in the area around 1 μm. This has been confirmed in the frame of screen optimization mentioned in the Section 3.2. This implies that for the thin and perfectly polished screens the MC model without inclusion of the emitted light diffusion can be used with the acceptable error for electron beams with the energies greater than roughly 15 keV.

The used MC model simulated 3-dimensional processes associated with interactions of primary electrons in the bulk of the investigated YAG single crystal. The Ce activator can be ignored during the simulation because its representation is only 0.3 mol%. Besides trajectories, the MC program also calculates the longitudinal (in the direction of the primary electron beam) and transversal (projected to surface plane) distributions of the deposited excitation energy. Attention was concentrated on the perpendicular impact of primary electrons in this paper, but it is no problem to simulate an inclined impact. The algorithms have been incorporated into the program code and compiled as an application called "SCATTER", which is applicable under OS Windows (32 bit). Calculations can be done for different energies of primary electron beam.

2.1. Simulation of electron trajectory

The MC output of the 2-D projection of the 3-D electron trajectories in the YAG single crystal at the primary electron energies of 20, 60 and 100 keV is shown in Fig. 1. The surface boundary and trajectories of the primary beams are indicated by horizontal and vertical lines, respectively. The shorted outgoing trajectories indicate secondary electrons (SE) as well as backscattering electrons (BSE). Using this view, the SEs and BSEs cannot be distinguished, but the MC model is able to determine both their energies and their directions. The presented simulation of the trajectories makes sense only if the plotted trajectories can be distinguished. Therefore only 100 trajectories were chosen for each beam energy. This figure of trajectories provides good visual information about interaction volumes, but it supplies only poor data for analysis of energy deposition. Nevertheless, it can be concluded that interaction volumes in the screen can be very large, especially in high voltage TEM. Even in low voltage TEM the resolution can be limited by the screen, because in this case a relatively small image on the screen is considerably magnified by light optics [2].

2.2. Simulations of longitudinal and transversal energy distribution

The outputs of the deposited energy distributions in YAG screen, taken from the MC simulation, are the basic data used to determine spatial resolution of screens. To reduce the statistical errors of these outputs, the total number of primary electrons simulated should be at least 10^3. As a result of such MC simulation, two different energy distributions can be obtained: (i) the longitudinal distribution that is deposited in the direction of the primary electron beam, (from the surface to the depth of the screen) and (ii) transversal distribution that is a projection of absorbed and consequently diffused energy on the surface of the screen.

The longitudinal distribution is not of primary importance when assessing CL screen resolutions. However, it could be helpful in choosing the appropriate thickness of the screen. This distribution should also be used if the transport of photons from luminescence centers is incorporated, which was neglected in the model used. Longitudinal distribution of absorbed electron energies using the primary electron beam of 20, 60 and 100 keV is in Fig. 2. It is very difficult to obtain a simulated value of the energy deposited near the surface of the single crystal, since these values are strongly affected by surface effects such as SE and BSE emission. Although the behavior of the SEs and BSEs was included in the MC model, their influence on the longitudinal energy distribution was omitted near the surface. The reason is that it would represent an unnecessary complication and significantly prolong the simulation time, while it would not provide any relevant results for the study of the spatial resolution of the single crystal screens. Therefore, the energies deposited at the surface were dropped from processing.

The transversal distribution of energy deposited by excited electrons in YAG screen for the primary electron beam of 20, 60 and 100 keV is shown in Fig. 3. In these results secondary processes associated with diffusion of excited electrons are also included, which is necessary for the spatial resolution determination. The presented graphs were obtained as outputs from the mentioned extended MC simulation in the form of the 2-D projection of the deposited energy on the surface of the YAG:Ce screen in dependence on the radius from the point of electron beam impact. The transversal distribution shown is of primary importance in

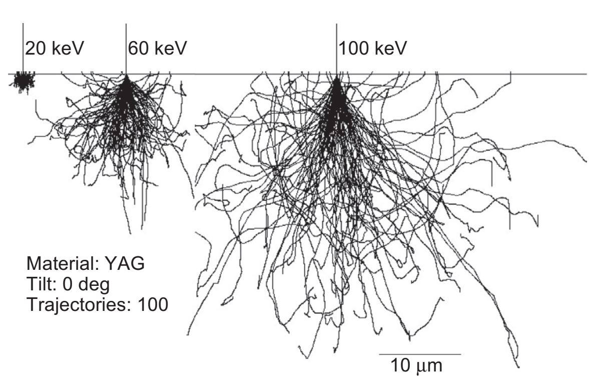

Fig. 1. MC simulation of electron trajectories in the YAG:Ce single crystal.

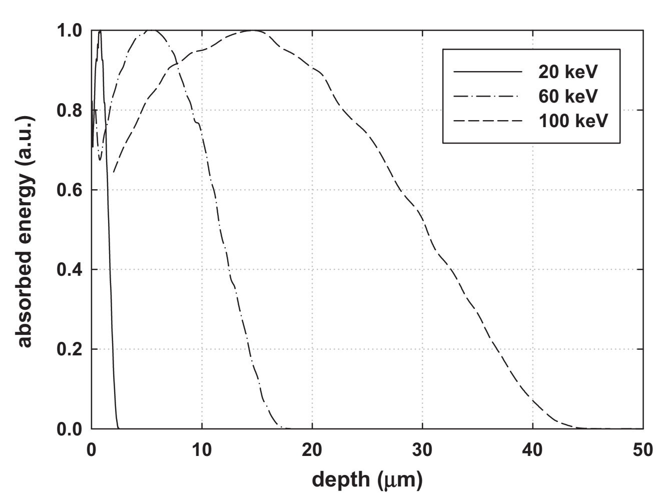

Fig. 2. MC simulation of longitudinal distribution of deposited electron energy in the YAG:Ce single crystal.

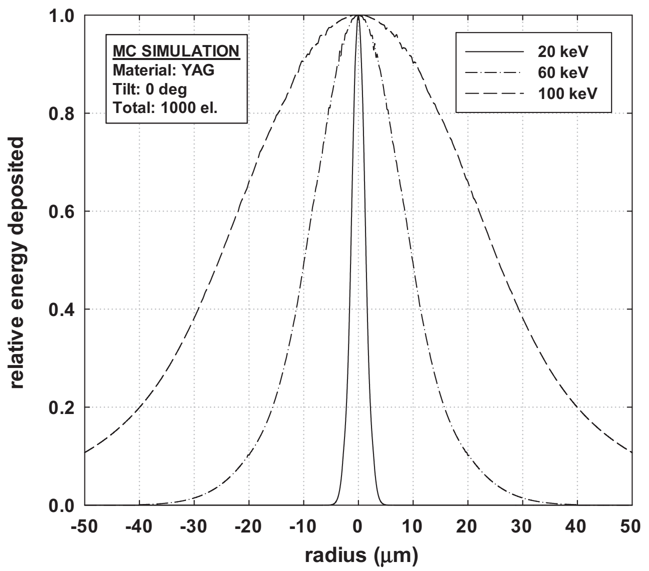

Fig. 3. MC simulation of transversal energy distributions of diffused electrons in the YAG:Ce single crystal.

assessing the resolution of the imaging screen. Of course, if the simulation does not include all processes, these results are only of limited predictive value. In particular, we must be careful in interpreting such phenomena, which may be more or less affected by various subsequent processes. With regard to this, the results of simulation should be understood as a rough estimate, and the true resolutions may be worse.

In the graphical expression in Fig. 3 symmetry from the primary electron beam is respected, as this expression is better suited for further processing and also for comparison with experiments. The presented excited energy distribution actually corresponds to the line spread function (LSF), and, in fact, it expresses the degree of blurring. There is pandering to determine the full width at half maximum (FWHM) of the plotted curves, but to assess the spatial resolution the FWHM is a less informative quantity than the LSF. For quantification, it is necessary to work with a contrast and especially to take into account a spatial frequency. So far, it is preferable to convert the LSF to an optical transfer function used for evaluating the image quality, which will be done later in comparing these results with the experimental ones.

3. Experimental examination of spatial resolution

Experimental examination of the spatial resolution of YAG:Ce can be performed using more different methods. The simplest one is the contact mask method, where a line or grid mask is placed on the surface of the studied screen, and the resolution is determined using a direct capture of mask borders. To get correct results with this method, one must have the mask with a sufficient absorption capability. But especially for high energy electron beams, the spread of electrons in the mask is very large, and it is quite impossible to stop all electrons within the mask [8]. For example, in experiments with the beam energy of 100 keV a mask with a thickness of several tens of microns is needed. Therefore, it is far better to use the edge projection method.

3.1. Edge projection method

At our laboratory, measurement of the spatial resolution was realized using the edge projection method. Unlike the contact mask method, using the edge projection method eliminates all problems associated with electron passage through the mask. The edge projection method utilizes projection (imaging) of a very sharp edge onto the examined YAG:Ce screen using the image mode of the TEM Philips CM 12 with relatively small magnification in the order of some tens or hundreds. In such a way the focused image of the edge situated in the first optically conjugate plane is obtained on the examined YAG:Ce screen situated in the second optically conjugate plane of the TEM used. The scheme of the experimental setup of this method is shown in Fig. 4

To obtain good results the key is a good projection object, which must have the flatness of the edges by a few orders of magnitude better than the resolution of the screens studied. As the projection object (placed in the specimen chamber of the TEM Philips CM 12, situated in the first optically conjugate plane), a silicon single crystal plate with a hole made by the anisotropic etching was used [9]. The prepared silicon plate with a pyramidal hole marked out by a large area of crystallographically flat and accurately defined geometry. The hole mentioned possesses the sharp edge with an inclined wall of 54.7°, which is shown in Fig. 5. The edge of such geometrically perfect hole used was examined by TEM Philips CM 12 and the results are presented in Fig. 6.

It is necessary to record the projection of the edge displayed on the screen with maximum possible magnification. This has to ensure the recording system based on a light-microscopic device, consisting of a magnifying objective and a CCD camera. Consequently, it is necessary to process the obtained record in a computer. Initial information must be processed into line spread function (LSF), and subsequently into the modulation transfer function (MTF) as outlined in Fig. 4 Using the primary beam energy of only a few tens of keV the resolution of several tens of lines per mm can be expected. In this case, it is necessary not only to magnify the image projected onto the screen as much as possible, but also to avoid vibrations which occur in insufficiently rigid and poorly fixed optical systems. In principle, it is possible to use a classic camera, but this is very uncomfortable and creates uncertain results.

For experimental determination of the spatial resolution a special compact system for the edge projection method (Fig. 7) has been built in our laboratory. The system was designed to hold the studied YAG:Ce single crystal screen in the second optically conjugate plane of the TEM Philips CM 12, to create a vacuum flange for TEM Philips CM 12 and to capture the image from the screen. It allows very precise mechanical and optical settings of

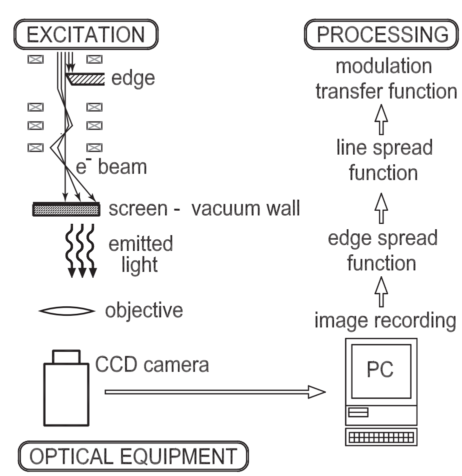

Fig. 4. Arrangement for the study of single crystal screen properties. The silicon single crystal plate with the anisotropically etched hole (situated in the first optically conjugate plane) is used as the projection object, and the YAG:Ce single crystal disk (situated in the second optically conjugate plane) is used as the investigated imaging screen.

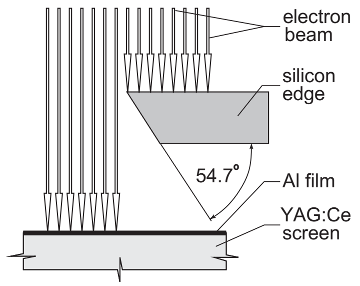

Fig. 5. Outline of the arrangement for unit step image creation using the edge projection method.

the whole experiment as well as on-line processing of the very small image from the screen without the loss of details. The image created on the screen was magnified by the small Olympus S Plan objective, and it was recorded with use of the Sony high resolution 1/2'' B/W CCD (CCIR) camera and the frame grabber card Matrix Vision Pcimage-SC plugged in the PC. All four objectives were situated in the four-position lens turret, and so the magnification was very easily changeable. The objectives and the CCD camera were jointed very rigidly. Therefore the focusing of the recording system could be accomplished readily by the fine adjustment element. The investigated screen was positioned in the bayonet joint cartridge at an otherwise heavily accessible TEM chamber, which makes the quick, simple and safe exchange of an examined screen possible. The cartridge was equipped with a tilt mechanism to ensure perfect perpendicular screen orientation towards the optical axis. The recording system was equipped with an x–y manipulator, which makes selection of the observed edge and/or moving the objective system out for the screen replacement possible. The image was digitized using the frame grabber installed in the PC. The final digital image was saved in an image file format for further processing and interpretation.

3.2. YAG:Ce specimen

Experimental examination of spatial resolution of the YAG:Ce screens has been carried out on the single crystal specimen of cerium-activated yttrium aluminum garnet (YAG:Ce – $Y_3Al_5O_{12}:Ce^{3+}$) [10,11]. The YAG:Ce single crystal was pulled by the Czochralski method using the molybdenum crucible, resistance heating and a 98% Ar + 2% H_2 protective atmosphere in the company Crytur. The initial material contained less than 10^{-4} wt% impurities. The crystal possesses the Ce concentration of 0.32 mol%.

The as-grown single crystal was cut and ground into the form of a very thin disc of $\varnothing 10 \times 0.1$ mm. The thickness of the disc must be as thin as possible to short imaging photon trajectories to the maximum possible extent and thus eliminate their scattering, which would have great influence on the screen resolution. But at the same time the screen must be self-supporting and it must be mechanically resistant, because it forms the vacuum barrier in the system. This puts very high demands on the production and handling of the screen. The entire screen surface was perfectly polished to prevent unwanted image distortions due to photon scattering at the disc boundary. Geometry and surface finishing of the screen have been optimized using the MC simulation of the light transport in the YAG:Ce single crystal disc [12–14]. The polished specimen was cleaned in organic solvents and additionally annealed in reducing atmosphere (H_2 fired) at a temperature of 1500-1700 °C. Although this treatment leads to a slight

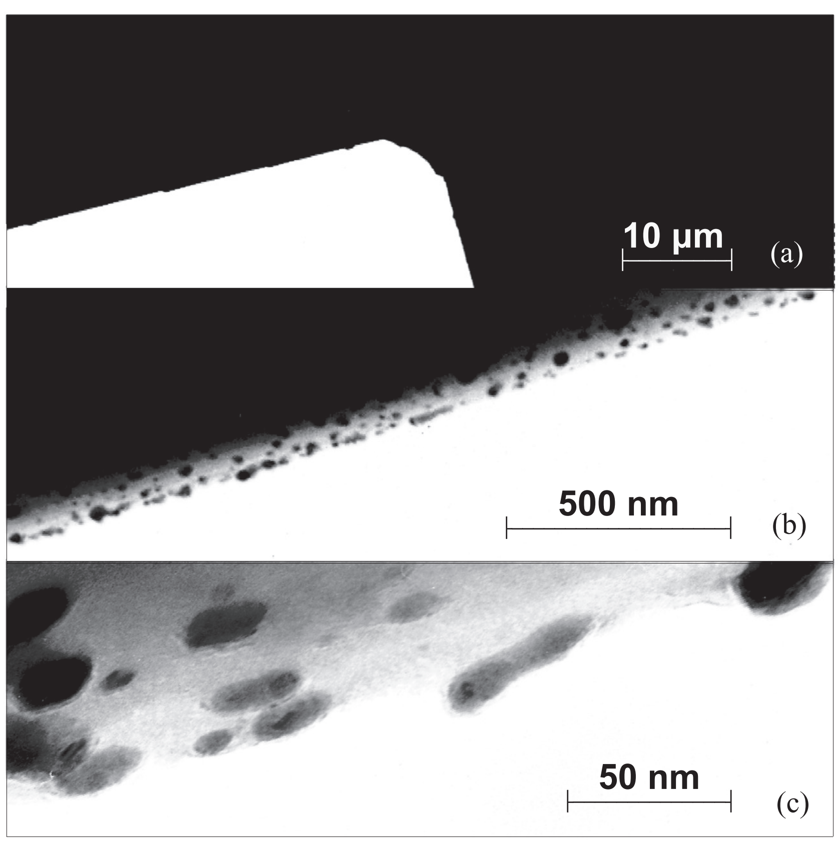

Fig. 6. 120 keV TEM images of the anisotropically etched hole in the single crystal silicon. (a) The corner at the magnification 450×. (b) The edge at the magnification 13000×. (c) Detailed structure of the edge at the magnification 125000×.

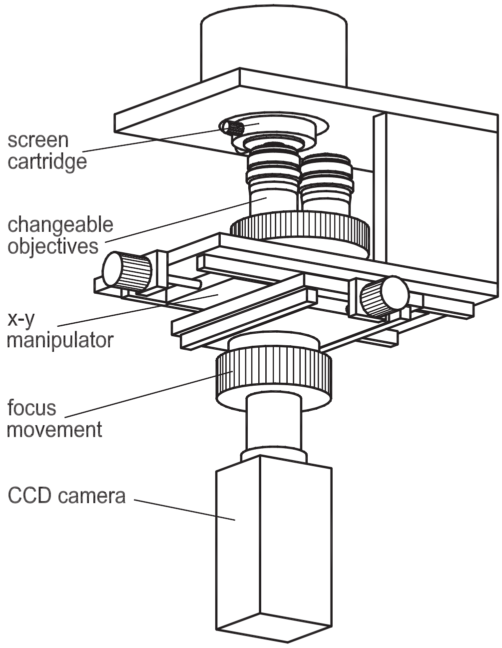

Fig. 7. Recording system for the edge projection method. The system allows handling and positioning the screen specimen using the screen cartridge, setting and focusing the capture module, and taking pictures using changeable objectives and the high resolution CCD camera.

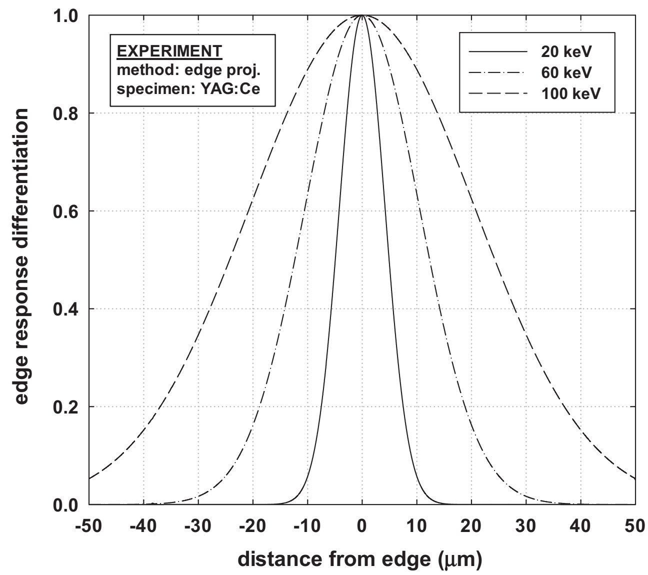

Fig. 8. Line spread function (LSF) of the YAG:Ce single crystal imaging screen measured using the edge projection method.

degradation of material time response (which is irrelevant here), it provides maximum CL efficiency of YAG:Ce single crystals [15]. The side of the YAG:Ce disc intended for the electron impact was coated with a thin aluminium film (50 nm) to prevent the charging of the surface on the one hand, and to increase the light signal collection efficiency [16,17] on the other hand.

3.3. Experimental results

The initial digital picture of the edge projected onto the studied YAG:Ce screen was captured using the experimental system described in the previous chapter. It was of the form of a blurred-edge image. For quantification, the mentioned images of the blurred edges were converted to intensities of arbitrary units using Tescan Atlas image processing software. Thereby, edge spread functions (ESF) were obtained. By differentiation of the edge spread functions (ESF), the line spread functions (LSF) of the measured edge response in the YAG:Ce screen were obtained. The measured LSFs of the YAG imaging screen are plotted for the primary beams of 20, 60 and 100 keV in Fig. 8. Although the LSF is not the best quantitative expression of the spatial resolution, it provides a basis to obtain some transfer function that is ideal for image quality evaluation.

In accordance with the simulated results, the strong dependence of the edge blurring on the electron beam energy is obvious from the LSFs shown in Fig. 8. In the case of the edge projection method used (unlike the method using the mask) the results are much more accurate for the higher beam energy. The reason is that using the projection method the accuracy grows to a certain limit with the size of the blurred edge because the larger studied area is under the smaller influence of limiting resolution of the light-optical recording system. Contrary to this, the accuracy of the mask method is rather given by the ability of the electron beam masking, which decreases with increasing of the electron energy. However, when interpreting blur at the beam energy of 20 keV a great caution must be taken, because the experimental values of blur are in the order of microns. In such a case, the LSF of the whole measurement system must be considered, which may not be negligible in comparison with the LSF of the investigated screen. In other words, the results can be effected by relatively large measurement error using the 20 keV electron beam, especially owing to the limited magnification of the optical objectives Olympus S Plan.

4. Comparison of results using MTF

Comparison of simulation results with those obtained experimentally has a number of drawbacks because, especially at lower primary beam energies, both methods are imperfect. In the case of the simulation, simplification does not include minor processes. And in the case of the experiment, limited optical magnification reduces the resolution of the recording system. Independently of this, it is desirable to transform the LSFs into a more useful form.

Image quality can be best expressed by an optical transfer function (OTF). The OTF characterizes the overall ability of the imaging system without the need to know the influence of individual optical events. The OTF is generally composed of its size (module) and of its phase. In the case of imaging screens the phase of OTF is of little significance. Therefore, to calculate the spatial resolution it is sufficient to use the module of OTF, which is called modulation transfer function (MTF). Using MTF all details of an image transfer can be ideally described, including the ability to change adjacent pixels from black to white (or vice versa) in response to spatial frequency changes. In other words, the MTF gives information about the ability to display fine details with a necessary contrast.

To determine the MTFs of the YAG:Ce imaging screens for the primary beam energies of 20, 60 and 100 keV, Fourier transform of the LSF modules presented in Fig. 3 and in Fig. 8 were used, and the results obtained are shown in Fig. 9. In this graph, the MTFs are plotted in dependence on the spatial frequency. For practical assessment of the results presented here, it is better to interpret the spatial frequency as the number of distinguishable lines (or dots) per given length period (for example per mm). Experimental and theoretical results are in perfect consistency for the primary beam energy of 100 keV and in quite good consistency for the energy of 60 keV. Mismatch in results at the energy of 20 keV may be caused by a number of inaccuracies in both simulations and experiments. For example, scattering of photons from luminescent

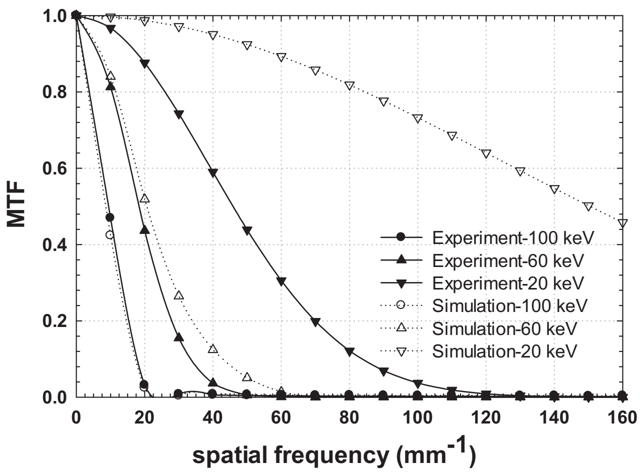

Fig. 9. Modulation transfer function (MTF) of the YAG:Ce single crystal imaging screen for different electron beam energies. Solid lines represent the experimental results and dotted lines represent the simulated results.

centers probably cannot be neglected in simulation volumes of a few microns. As already discussed in the previous chapter, problematic are also the experiments at the energy of 20 keV, where quite high demands are placed on precision magnification of the projected images. In such a case the MTF of the measurement system may affect the MTFs of the imaging screen. It can be deduced from the results of MTFs presented in Fig. 9 that for contrast of 50% the spatial resolution of YAG:Ce CL imaging screens is approximately 8 lines per mm at the primary beam energy of 100 keV and about 18 lines per mm at the energy of 60 keV. Determination of the resolution at the energy of 20 keV is problematic due to inaccuracies in both simulations and experiments, but it can be roughly estimated that the resolution is somewhere around 100 lines per mm.

5. Conclusion

Determination of spatial resolution of the YAG:Ce CL imaging screens is quite a demanding task. It is very difficult to interpret the spatial resolution from experiment without calculation support. It is not only very difficult but also quite reckless to interpret the resolution of the simulation without experimental support.

To determine the spatial resolution of the imaging screens the MC simulation of the primary electron trajectories has relatively little meaning. Somewhat better information on the spatial resolution provides the distribution of the absorbed energy of the primary electrons, but even that does not give useable results. To obtain acceptable results for a quantification of the spatial resolution of the imaging screens at least major diffusion processes affecting the excited electron energy distribution must be included in the MC simulation. The results of such MC simulation that can be easily transformed into line spread function (LSF) give good information about blurring of lines on the screen for different energies of the primary electron beam.

The preferred method for experimental determination of the spatial resolution of the imaging screens is the edge projection method. Compared to the mask method the edge projection method is not degraded by electron scattering in the shielding object during edge demarcation. The drawback of all experimental methods is the necessity of high magnification of the small image of only slightly blurred edge at the low energies of the primary electron beam. For the energies below about 30 keV, it is necessary to magnify substantially the interaction region that is much smaller than 10 μm, but it can run into limits of the magnifying objectives. Nevertheless, the experimental edge projection method provides very useful results that can be easily transformed into blur characterizing line spread function (LSF).

Because the LSF is not a tool which would adequately quantify imaging possibilities of the YAG:Ce screens, it is necessary to transform LSF to modulation transfer function (MTF) that is used to describe all the details of the image transfer, including the spatial resolution. Using the MTFs obtained with both the MC simulation and the experimental edge projection method, the values of spatial resolution of the YAG:Ce CL imaging screens were found as approximately 8 lines per mm at the primary beam energy of 100 keV, approximately 18 lines per mm at the energy of 60 keV, and roughly estimated as about 100 lines per mm at the energy of 20 keV.

Acknowledgments

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