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Inquiry of detector components for electron microscopy

Cathodoluminescent experimental methods for the measurement of efficiencies, emission spectra and decay times of scintillators and imaging screens are presented. Pointed out are light-guiding Monte Carlo simulation methods for efficiency optimization of scintillation detection systems. Finally, sharp edge projection and Monte Carlo methods for the measurement and for the simulation of the spatial resolution of imaging screens, respectively, are described.

1. INTRODUCTION

Scintillators, light guides, photomultiplier tubes (PMT) and imaging screens are opto-electrical components used for detection systems in electron microscopy (EM).

Scintillators, light guides and PMT are used for signal processing utilizing scintillation detection systems in both scanning electron microscopes (SEM) [1] and scanning transmission electron microscopes (STEM) [2]. The scintillator has to be very fast, possess a high efficiency of electron-photon conversion, and it has to emit the light in a spectral region of a high PMT sensitivity. The light guide has to transport the signal from the scintillator to the PMT photocathode with minimum losses, even if it is complexly shaped in order to fit the space available in the microscope chamber. And finally, the PMT has to collect, convert and multiply the signal to be processed by image amplifiers. Strictly speaking, the whole detection system must have high detection quantum efficiency (DQE).

Imaging screens are used for image formation in transmission electron microscopes TEM having a miniature and/or digital imaging system [3] [4]. Imaging screens have to possess a high spatial resolution, high efficiency of electron-photon conversion, but they need not be very fast as is the case with scintillators.

Many methods for the study of detector components properties were developed in our laboratory. Some of them are presented in this paper.

2. CATHODOLUMINESCENT METHODS

To ensure maximum performance of scintillators and imaging screens utilized in EM one must study their cathodoluminescent (CL) properties. These include: (1) CL efficiency (more precisely: energy conversion efficiency), (2) time characteristics (decay time), and (3) spectral emission characteristics. All mentioned CL properties can be measured by using the same equipment. Such an equipment was built in our laboratory and its block scheme is shown in Fig. 1 [5]. The excitation unit is formed by an adapted electron microscope with an electrostatic deflection system and a blanking diaphragm placed above the Faraday cage. In the pulse mode, the excitation electron beam can be deflected outside the blanking diaphragm, so that for 10 keV electrons, the rise and decay times of the excitation pulse are the same, approximately 5 ns. The pulse mode was intended for the determination of kinetic properties, but it can also be used with advantage for the measurement of emission spectra. The CL efficiency is measured in the continuous mode.

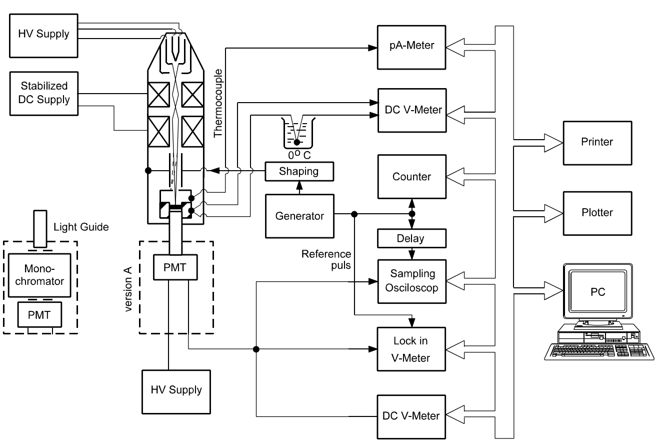

Fig. 1. Equipment used for measuring CL properties.

The investigated single crystal specimen is positioned at the face of the light guide (inside the Faraday cage), and the signal is guided directly toward the entrance window of the PMT, when spectrally non-decomposed CL properties (integral efficiency and decay characteristics) are measured. When spectrally decomposed CL properties (spectral characteristics) are measured, the signal is guided toward the entrance slit of the mirror monochromator. During the measurement of efficiency and decay characteristics, the output of this PMT is connected to the microvoltmeter and the sampling oscilloscope, respectively. For the CL spectra measurement, the PMT is positioned at the output slit of the mirror monochromator, and the signal is processed using a lock-in nanovoltmeter.

The individual instruments are connected to the general purpose interface bus (GPIB, IEEE-488), and the measuring apparatus is controlled by a personal computer which also processes the obtained data. The data measuring and processing software (which contains correction algorithms) was written in Turbo Pascal and Basic. Some tens of different single crystal CL materials were measured at our laboratory [5]. Of these, single crystals of cerium activated yttrium aluminum garnet (YAG:Ce - $Y_3Al_5O_{12}:Ce^{3+}$), cerium activated yttrium aluminum perovskite (YAP:Ce - $YAlO_3:Ce^{3+}$), cerium activated yttrium silicate ($Y_2SiO_5:Ce^{3+}$, which chemically corresponds to the powder phosphor P47), and europium activated calcium fluoride ($CaF_2:Eu^{2+}$) were chosen as the most interesting ones for EM applications.

All detection systems of a SEM should operate at TV frequency. Therefore, the most limiting parameter of CL materials (used as scintillators for SEM) is the CL decay time. It has to be less than 100 ns. So the measurement of decay characteristics is the most important, and, because of short time range, the most exacting measurement. Some typical CL decay characteristics of single crystals for SEM, measured in our laboratory, are shown in Fig. 2. Unfortunately, all single crystals having a short decay time contain oxygen and just belong to those with lower efficiency. The best solution is P47 and YAP:Ce single crystals (having quite satisfactory CL efficiency), whose decay time is 34 ns and 38 ns, respectively. However, the letter single crystal has a multi-exponential decay characteristic, so that it shows the afterglow (1 % at 5 :s after the end of excitation).

3. LIGHT-GUIDING SIMULATION METHODS

Some image modes of SEM or STEM require that the electron detection system should fit into a very small space, sometimes even symmetrically around the primary electron beam. Therefore, scintillation detectors in non-classical arrangements are commonly

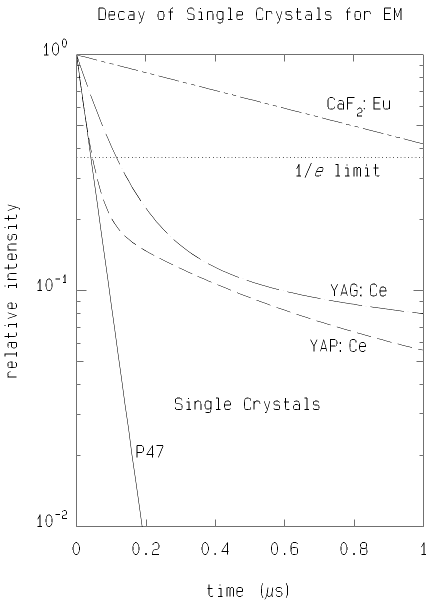

Fig. 2. Decay characteristics of single crystal scintillators for SEM. Excitation pulse duration 10 :s.

applied. The efficiency of these so called edge guided signal (EGS) scintillation detectors is very hard to estimate. For this reason, the Monte Carlo (MC) simulation method has been developed [6]. The method makes use of random generation of photon emission from a luminescent centre and describes the trajectory of photons and the efficiency of their transport toward the photocathode of the photomultiplier tube. The model includes photon generation in a point source, mirror reflection by a metal coated surface, Fresnel reflection by a metal uncoated surface, Fresnel passage through the boundary of different materials, diffusion reflection and passage through a matted surface and optical absorption in material.

Several programs utilizing the described MC model have been debugged at our laboratory. The source code of the programs has been written in Fortran 77 and can be, therefore, run on computers of different platforms. If running the program SCIUNI, version 3.0, the detector system may include all surfaces (or their parts) which satisfy the following demands: (1) Surfaces are given by a rotationally symmetric body or by a plane, (2) the axis of a body of each non-plane surface must be parallel with any axis of the coordinate system and (3) the normal of each plane must be parallel with any plane of the coordinate system. This means that the program enables the calculation of the efficiency of light transport for nearly any configuration of the scintillation detector.

Examples of the MC simulation results, i.e. the results of modelling very simple scintillation detectors are shown in Tab. I. The YAG:Ce single crystal and PMMA were the materials used for scintillators and light guides, respectively. Scintillators with Al deposited electron impact surfaces were connected to the light guide by using optical cement. The circular and the square profiles of the scintillators were 20 mm in diameter and side length, respectively. All light guides were 60 mm long. For comparison, efficiencies of light transport through classical base guided signal (BGS) rotationally symmetric detectors with a disc, conical and hemispherical scintillator, respectively, are also shown in Tab. I.

At our laboratory, the MC light-guiding simulation method is the basic method for the computer optimized designing (COD) of new BSE scintillation detectors [7]. The optimized design for the S 4000 Hitachi SEM, resulting from the application of this method, is shown in Fig. 3. The initial (non-optimized) detector design was given by the size and by the shape of pole pieces and of the specimen holder (Hitachi S 4000). It has been found during optimization, that a great improvement (256 %) has been achieved

description	matted scintill. output surface	hole	EFFICIENCY OF LIGHT TRANSPORT		
			mean	min.	max.
Circular plate scintil-	yes	no	0.0052	1.2e-04	0.0316
lator with strip light	yes	yes	0.0065	8.3e-04	0.0318
guide	no	no	0.0046	2.0e-04	0.0341
	no	yes	0.0066	5.7e-04	0.0341
Circular plate scintil-	yes	no	0.0521	0.0204	0.1440
lator with light guide	yes	yes	0.0526	0.0144	0.1606
widening to circular	no	no	0.0688	0.0100	0.1740
profile	no	yes	0.0685	0.0102	0.1745
Square plate scintil-	yes	no	0.0561	0.0119	0.1674
lator with light guide	yes	yes	0.0562	0.0101	0.1702
widening to square	no	no	0.0649	0.0145	0.1796
profile	no	yes	0.0657	0.0084	0.1850
Disc scintil. with	yes **	no	0.186	0.174	0.196
cylindr. light guide *	yes	no	0.025	0.011	0.035
Conical scintil. with	yes	no	0.138	0.091	0.155
cylindr. light guide *	no	no	0.179	0.126	0.352
Hemisph. scintil.	yes	no	0.0507	0.0406	0.0838
cylindr. light guide *	no	no	0.0680	0.0082	0.1305

* BGS rotationally symmetric system

** no optical cement was used

Tab. I. Efficiency of light transport through EGS and BGS scintillation detectors.

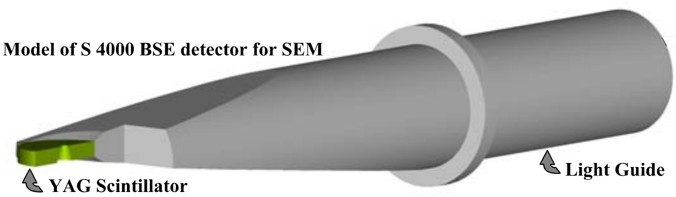

Fig. 3. Computer optimized design of the BSE detector for S 4000 Hitachi SEM. This geometry possesses efficiency of about 400 % compared with the non-optimized one.

after shifting the widening planes as close to the scintillator disc as possible, and after decreasing the angles of these planes. The final refinement (Fig. 3) has been accomplished by integrating a conical light-guiding ring (close to the scintillator) into the widening planes of the light-guide. The resulting efficiency is about 400 % compared with the initial one.

4. METHODS FOR SCREEN RESOLUTION DETERMINATION

YAG:Ce single crystal screens can be used as very small elements for forming a small image intended for further processing. In addition to the CL efficiency, spatial resolution of CL screens is the most important characteristic in such an application. Both theoretical and experimental methods can be used for the examination of spatial resolution.

Calculations can be accomplished by the Monte Carlo (MC) method and can be corrected for electron diffusion. The MC model used for simulation at our laboratory was based on the single scattering utilizing the screened Rutherford cross-section and Bethe slowing down approximation [8]. The MC model simulated

3-dimensional trajectories of primary electrons in the bulk of the investigated solid. Only primary processes were included in the model. Attention was concentrated on the perpendicular impact of primary electrons but it is no problem to simulate an inclined impact. Besides trajectories, the MC program was creating both longitudinal (in the direction of the primary electron beam) and transversal (projected into the surface plane) distribution of the absorbed energy. The MC program was written for and executed on an IBM compatible personal computer.

The outputs of the deposited energy distributions, projected into the surface plane of the YAG:Ce screen, are the basic data taken from the MC simulation. To reduce the statistical errors for these outputs, the total number of primary electrons simulated should be 10^3 at least. Furthermore, to determine the spatial resolution, it is necessary to correct the transversal distribution of energy for the diffusion of electrons by using empirical relations. As a result of such a computation, the distributions of energy deposited by diffused electrons are shown in Fig. 3. With regard to only primary processes involved in the MC model, the results of simulation should be understood as a rough estimate, and the resulting resolutions can be considered as maximum.

In our laboratory, measurement of the spatial resolution was made in the Philips CM 12 TEM using the sharp edge projection on to the examined screen. As a projection object (placed in the specimen chamber), a silicon single crystal plate with an orientation-etched hole was used. As the screen (placed near the column bottom), the YAG:Ce single crystal plate with both sides polished was used. The edge image from the screen was recorded by the optical equipment constructed in our laboratory. The recording optics consisting of an eyepiece-objective system with two prisms enabled us to take a photograph of the screen image with the magnification 40x. The measuring system was calibrated by using the Agar 300 grid as a projection object. Experimental data from photographs of the edge images were converted to intensities of arbitrary units. After the correction for the film emulsion response, the magnitude of the intensity along the direction perpendicular to the edge (edge spread function) was obtained for each energy of the primary electron beam. By differentiation of edge spread functions, the line spread functions of the measured edge responses in the YAG:Ce screen (shown in Fig. 5) were obtained.

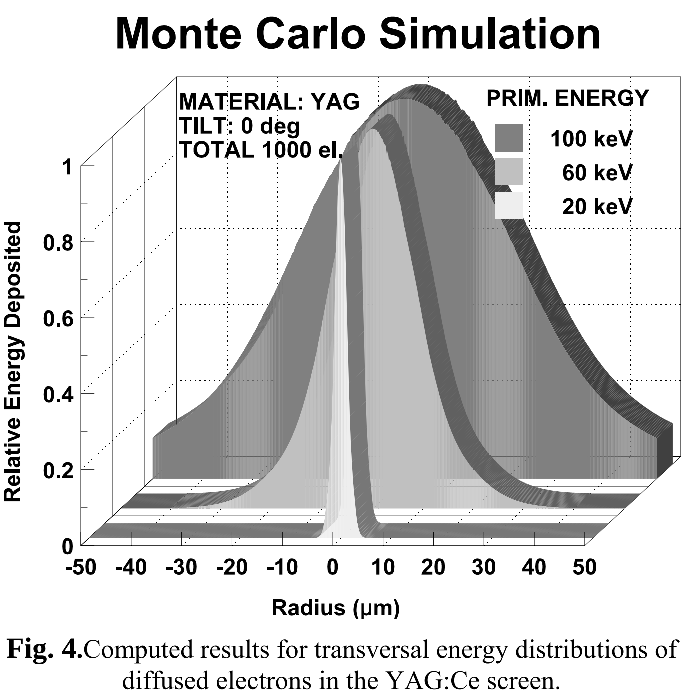

Fig. 4. Computed results for transversal energy distributions of diffused electrons in the YAG:Ce screen.

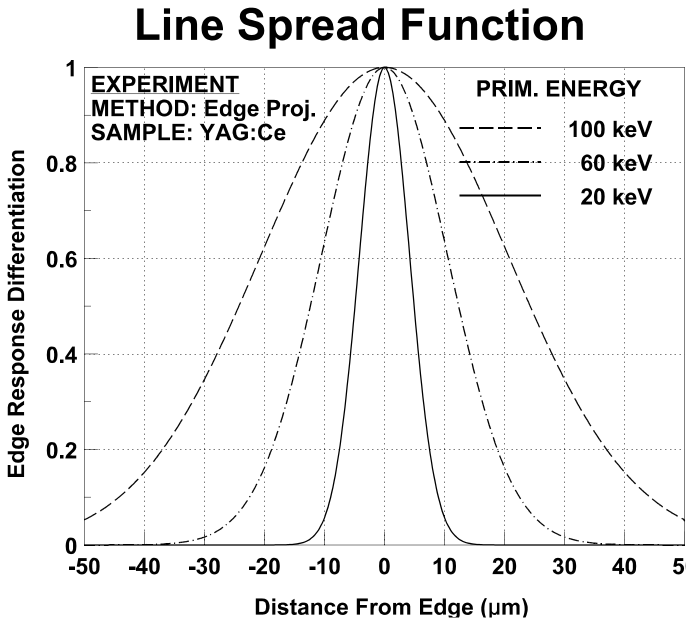

Fig. 5. Experimental results for line spread functions of the edge projections on to the YAG:Ce screen.

5. CONCLUSION

Cathodoluminescent experimental methods, light-guiding simulation methods, and methods for the determination of the screen resolution are the basic tools for the investigation of scintillators, light guides and imaging screens for electron microscopy. The methods mentioned provide results for an optimized design of fast and efficient scintillation detection systems for SEM, and for high resolution imaging systems for TEM.

Acknowledgements

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