# SOME METHODS FOR INVESTIGATION OF DETECTOR COMPONENTS FOR ELECTRON MICROSCOPY

P. Schauer, R. Autrata

Institute of Scientific Instruments, Academy of Sciences of the Czech Republic, Královopolská 147, CZ - 612 64  Brno, Czech Republic

Scintillators, light guides, photomultiplier tubes (PMT) and imaging screens are opto-electrical components used for detection systems in electron microscopy (EM). Many methods for the study of their properties were developed in our laboratory. Some of them are presented in this paper.

## 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 (Fig. 1) [1]. 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.

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
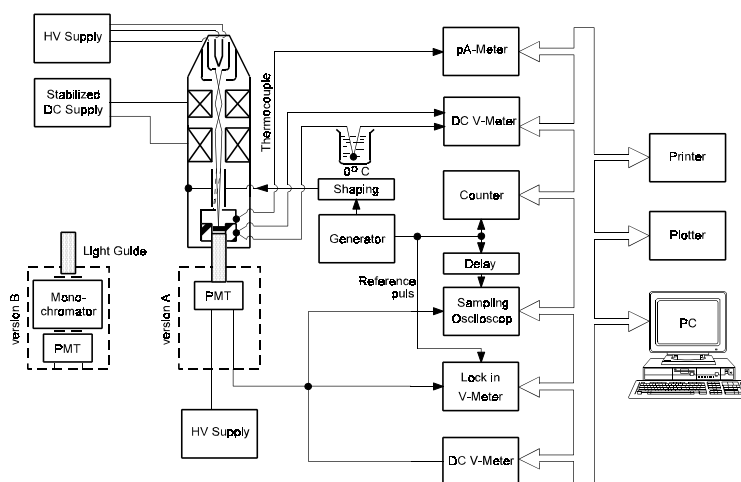


**Figure 1.** Equipment used for measuring CL properties.

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 processed 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 [1]. 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.

**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 a Monte Carlo (MC) method and can be corrected for electron diffusion. The MC model used for simulation in our laboratory was based on the single scattering utilizing the screened Rutherford cross-section and Bethe slowing down approximation [2]. 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 the 486DX2/66 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. 2. 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.
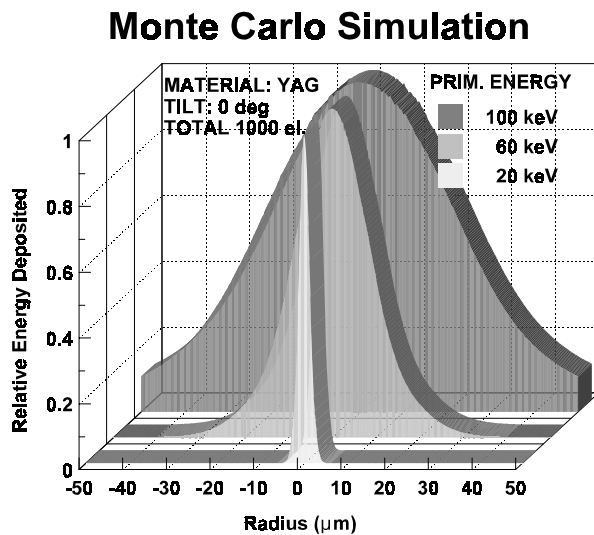
**Figure 2.** Computed results of transversal energy distributions of diffused electrons in the YAG:Ce screen.
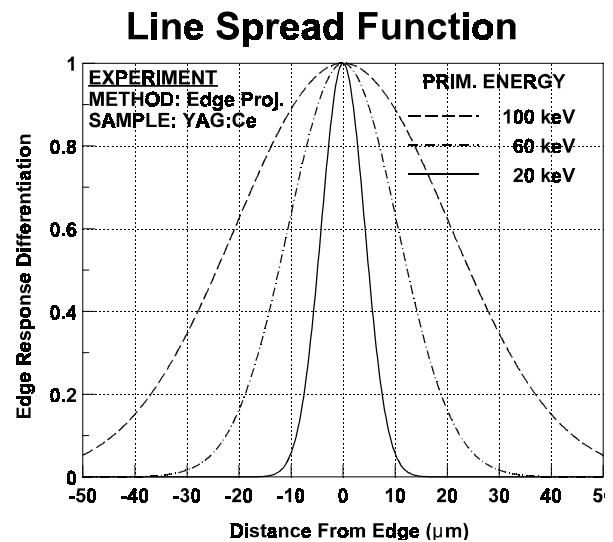
**Figure 3.** Experimental results of line spread functions of the edge projections on to the YAG:Ce screen.

In our laboratory, measurement of the spatial resolution is 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), the 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 to 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. 3) were obtained.

## Light-guiding simulation methods

Some image modes of SEM or STEM require that the electron detection system be fitted into a very small space, sometimes even symmetrically around the primary electron beam. Therefore, scintillation detectors in non-classical arrangements have to be applied. Especially, the efficiency of 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 [3]. 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. In 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. The source code of the program has been written in Fortran 77 and can be, therefore, run on computers of different platforms.

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 BGS rotationally symmetric detectors with a disc, conical and hemispherical scintillator, respectively, are also shown in Tab. I.

| description | matted scintil. output | hole | EFFICIENCY OF LIGHT TRANSPORT | | |
|---|---|---|---|---|---|
| | | | mean | min. | max. |
| Circular plate scintil-lator with strip light guide | yes | no | 0.0052 | 1.2e-04 | 0.0316 |
| | yes | yes | 0.0065 | 8.3e-04 | 0.0318 |
| | no | no | 0.0046 | 2.0e-04 | 0.0341 |
| | no | yes | 0.0066 | 5.7e-04 | 0.0341 |
| Circular plate scintil-lator with light guide widening to circular profile | yes | no | 0.0521 | 0.0204 | 0.1440 |
| | yes | yes | 0.0526 | 0.0144 | 0.1606 |
| | no | no | 0.0688 | 0.0100 | 0.1740 |
| | no | yes | 0.0685 | 0.0102 | 0.1745 |
| Square plate scintil-lator with light guide widening to square profile | yes | no | 0.0561 | 0.0119 | 0.1674 |
| | yes | yes | 0.0562 | 0.0101 | 0.1702 |
| | no | no | 0.0649 | 0.0145 | 0.1796 |
| | no | yes | 0.0657 | 0.0084 | 0.1850 |
| Disc scintil. with cylindr. light guide * | yes ** | no | 0.186 | 0.174 | 0.196 |
| | yes | no | 0.025 | 0.011 | 0.035 |
| Conical scintil. with cylindr. light guide * | yes | no | 0.138 | 0.091 | 0.155 |
| | no | no | 0.179 | 0.126 | 0.352 |
| Hemisph. scintil. cylindr. light guide * | yes | no | 0.0507 | 0.0406 | 0.0838 |
| | no | no | 0.0680 | 0.0082 | 0.13055 |

* BGS rotationally symmetric system
** no optical cement was used

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

**References**

1. Autrata R., Schauer P., *Scanning Microsc. Suppl.* **9** (1996), 1-12.
2. Joy D.C., *Proc. EUREM 88, York, England*, (1988), 23-32.
3. Schauer P., Autrata R., *Scanning* **14** (1992), 325-333.