CATHODOLUMINESCENCE STUDY OF SILICON POLYMERS

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First steps of cathodoluminescence (CL) study of metastable states in poly-(methylphenylsilylene), i.e. PMPhSi are presented in this paper. Besides the explanation of the experimental setup, the attention is paid to the specimen optimization. The degradation of the CL emission as a result of the formation of metastable states is shown.

INTRODUCTION

Excitation of organic molecules, polymers and molecular assemblies by electrons represents interesting phenomena from both the application (detectors on one side and electron resists on the other side) and the basic research point of view. The aim of this paper is to present first steps of our cathodoluminescence (CL) study of the metastable states in poly-(methylphenylsilylene), i.e. PMPhSi.

EXPERIMENTAL SETUP

PMPhSi degradation has been studied using the modular CL equipment [1]. A transparent PMPhSi on a transparent substrate was positioned at the face of the light guide. The CL emission was guided from the substrate side to a photocathode of the photomultiplier (PMT). At the CL spectra measurement, the emitted light was guided to the mirror monochromator, and the PMT was positioned at the output slit of the monochromator. It was necessary to measure in a synchronous mode using the modulated beam and a lock-in nanovoltmeter to eliminate a noise/background. The equipment was controlled by the PC using the GPIB bus (IEEE-488 standard).

SAMPLE PREPARATION

The studied PMPhSi was prepared by the Wurtz coupling polymerization [2]. The low-molecular weight fractions were extracted with boiling diethyl ether. The layers for the CL measurements were prepared from a toluene solution by casting on quartz disk substrates. For electron beams experiments, the PMPhSi must be covered with a conductive film to protect the sample from charging during an electron beam impact. The coating must be thin enough so as not to absorb excitation electrons, and at the same time thick enough to be conductive and to show the high optical reflectivity for the photon collection. The Monte Carlo (MC) simulation was used to estimate the electron energy losses in the Al film. Using the single scattering with the screened Rutherford cross-section and the Bethe energy loss [3] the dependence of thickness of Al film on the electron energy transmitted at 10 keV *(Fig.* 7) was obtained. Even in the 50 nm Al film the losses are acceptable, and in addition, the film has high conductivity. The Fig. 1 also shows the thickness dependence of the matrix method [4] calculations of the internal optical reflection at the PMPhSi-Al boundary. The high optical reflection is significant for low losses at the photon collection. As the best solution, the Al sputtered film of 50 nm, possessing the optical reflection of 89 % and the electron energy yield of 96 % at 10 keV, has been chosen. Before the Al deposition the PMPhSi was purified by precipitation in methanol and toluene solution and centrifuged.

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The PMPhSi is a light material possessing high transmission for fast electrons. The MC simulation of its interaction volume for 10 keV primary electrons is shown in Fig. 2. To estimate the losses for layered specimens at 10 keV, the MC simulated absorbed electron energy versus the depth is plotted in *Fig. 3.* It is seen the thickness of the PMPhSi must be at least 3 μm to have no excitation losses at CL measurements. On the other side, the PMPhSi layer must be thin enough to avoid the losses due to the photon selfabsorption. The degradation of the CL emission of the thickness optimised PMPhSi (2 μm) sample is plotted in *Fig. 4.* It can be found out from this semilogarithmic plot, the material forms metastable states approximately after 7, 27 and 60 minutes of 10 keV electron beam exposure.

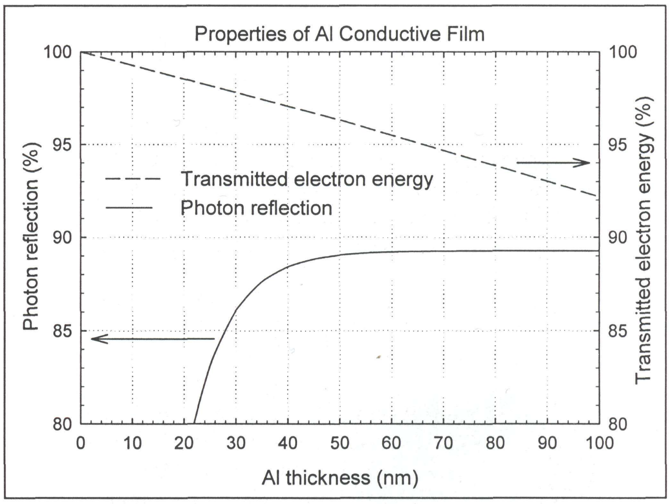

Fig. 1 Simulation of the Al conductive film effect. - - - - MC simulation of the transmitted electron energy gain. —— Calculation of the internal (PMPhSi - Al) photon reflection at the wavelength of 360 nm

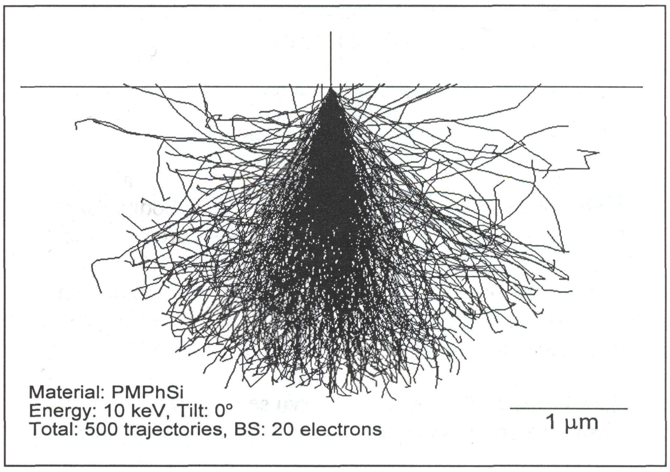

Fig. 2 MC simulation of 2D projection of 3D trajectories in the PMPhSi at primary electron energies of 10 keV

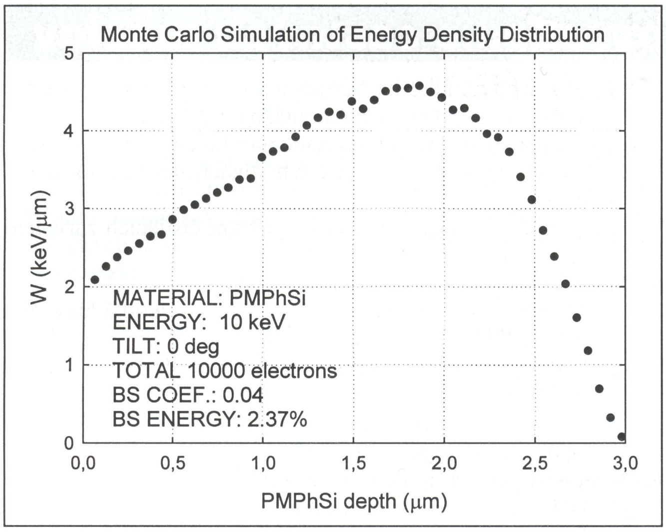

Fig. 3 MC simulation of the depth dependence of the absorbed energy in the PMPhSi. 10 000 trajectories has been evaluated to reduce the statistical errors.

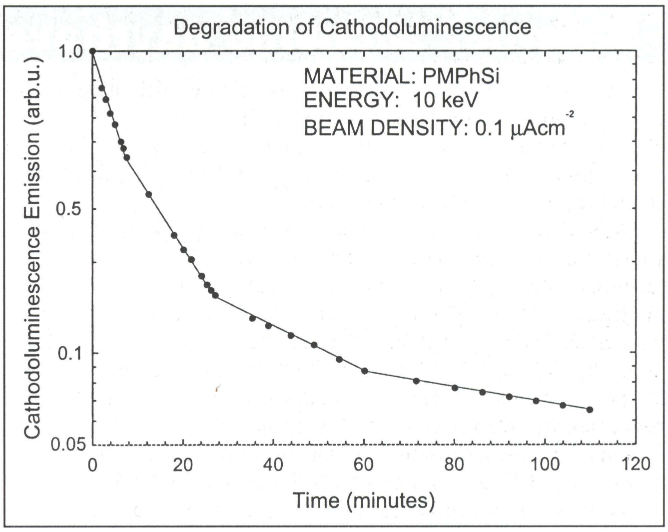

Fig. 4 Experimental results of the time dependence of the CL emission degradation of the PMPhSi (10 keV, 0.1 μAcm^{-2}).

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