

CATHODOLUMINESCENCE OF POLYSILANES

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Polysilanes - a broad class of organic materials whose basic building block is a chain built up of silicon substituted by alkyl or aryl groups. Properties of these substituents significantly affect properties of polysilanes. This material is very interesting because of its chemical, electrical and optical properties [1]. In spite of great research interest in recent years cathodoluminescent (CL) properties of polysilanes were not yet studied. Poly[methyl(phenyl)silane] (PMPHSi) is a typical representative of polysilanes which was prepared by the Wurtz coupling polymerization. Thin layers of PMPHSi were prepared from toluene solution by a spin coating technique [2]. The material was applied on the quartz glass substrate and covered with the aluminium (Al) film. The Al film protected the specimen from charging and reflected photons emitted under the specimen surface towards the quartz glass substrate.

The method of PMPHSi study was based on the measurement of CL intensity after passing through the specimen (Figure 1). Electron beam, emitted from a wolfram cathode, accelerated and focused in the excitation part, struck the Al deposited specimen. Emitted photons which passed through the specimen and through the substrate were led by a light guide to the detection part.

The excitation part was based on the rebuilt electron microscope TESLA BS 242. Electron beam energy was variable from 1 to 60 keV. The deflecting system allowed modulation of the electron beam and enabled measurement, not only in the continual mode, but also in the pulse mode. This was very important for the measuring in the synchronous mode as well. The detection part was based on a photomultiplier tube [3].

The first PMPHSi specimens of the thickness of 2 μm covered with 50 nm of the Al film were measured using the photomultiplier tube TESLA 65 PK 415. The excitation energy of 10 keV was used for cathodoluminescence intensity measurement in the continual mode (i.e. the deflecting system is switched-off). Current density of the incident electron beam was approximately 5 nA/mm^2 . The CL emission from PMPHSi was smaller by 2 orders of magnitude in comparison with the CL emission from the YAG:Ce single crystal and measured under the same conditions [4]. Strong influence of the background, above all of the cathode light, was proved and also measured by switching off high voltage (only wolfram filament is heated).

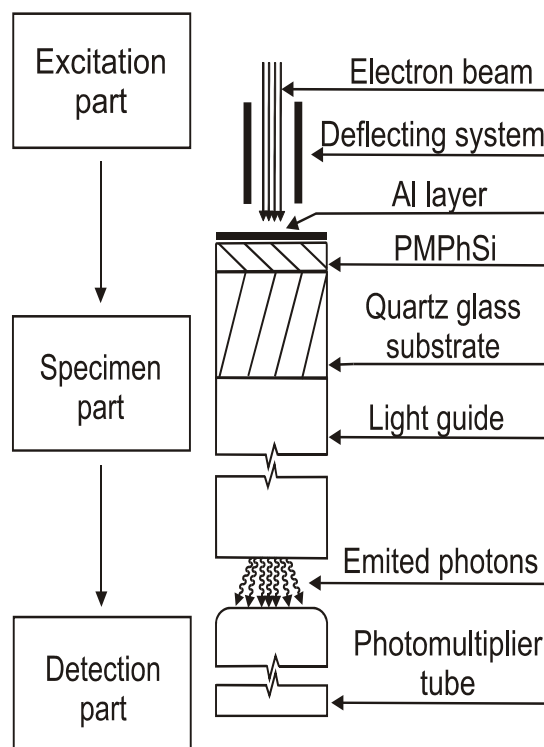


Figure 1 Layout of the experimental arrangement for the study of cathodoluminescent properties of polysilanes including the specimen part details.

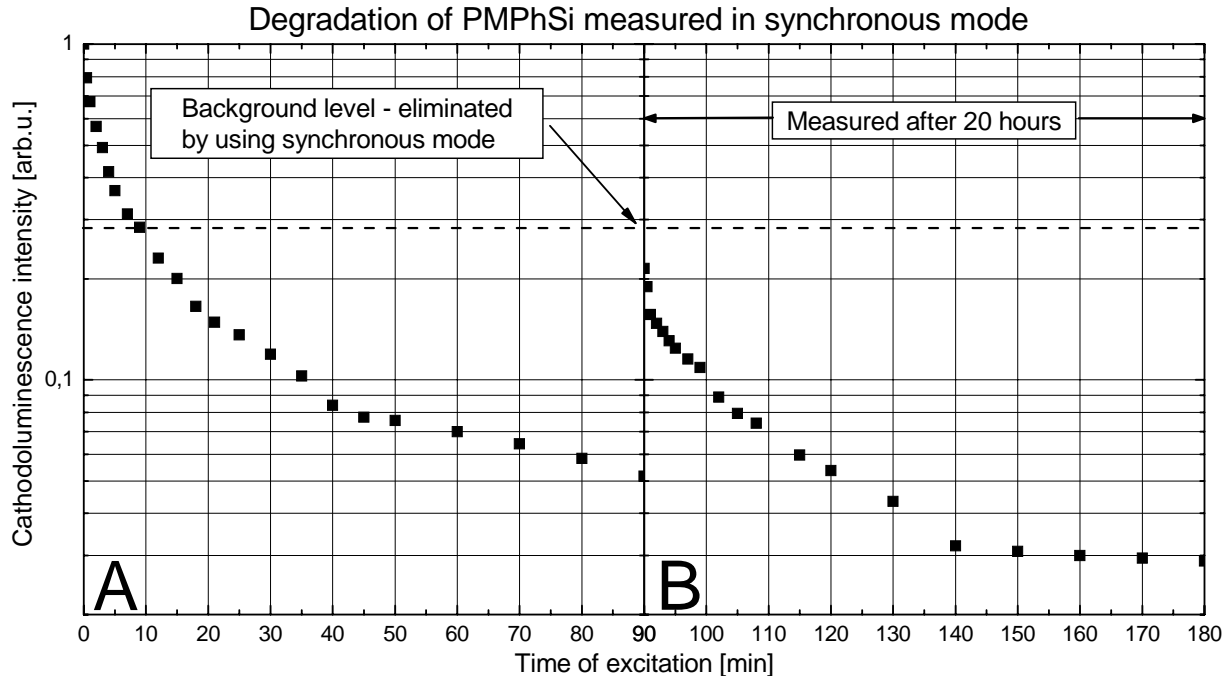


Figure 2 Time dependence of PMPPhSi CL intensity measured for 90 minutes (part A) and subsequently after 20 hours of leaving in a vacuum chamber of microscope at room temperature for next 90 minutes (part B).

Measuring in the synchronous mode was used to eliminate the influence of the background and noise. The electron beam was modulated using the pulse generator TR-0331. The frequency of modulation was 1 kHz. The reference pulse taken from the trigger output of the pulse generator was connected to the reference input of the lock-in nanovoltmeter UNIPAN 232 B. The change of the PMPPhSi CL intensity was measured using this lock-in nanovoltmeter and plotted on semilogarithmic graph (Figure 2). The experimental conditions at this measurement were as written above (10 keV, 5 nA / mm², 1 kHz).

The decrease of CL intensity of PMPPhSi was assigned to a material degradation. The fastest degradation of the new specimen (part A of the graph) was seen in the first 10 minutes (about 70% decrease of the intensity). The degradation of PMPPhSi was still going on, but much more slowly, after 50 minutes of excitation (about 1% in 10 minutes). After 20 hours of no excitation, with the specimen left in the vacuum chamber of the microscope at room temperature, the PMPPhSi was again excited by the electron beam under the same conditions (part B of the graph). Partial recovery of the CL intensity (20%) was observed. The CL intensity value in 120th minute was nearly identical to that in 90th minute of the previous measurement. After 140 minutes of electron beam excitation, the CL intensity was almost invariable, but reached only 1% of the initial intensity value. The changes of the slope of the curve in the 10th and 40th minute (part A) as well as in the 140th minute (part B) may indicate creating metastable states.

References

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