

In-situ study of network creation in polymers during polymerization initiated by light

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The *o*-Ps probe is a very sensitive tool for monitoring changes in the microstructure of polymers. For slow processes over a period of hours and more, Positron Annihilation Lifetime Spectroscopy (PALS) detects different effects over time without major problems of sufficient integral counts of annihilation events for appropriate evaluation of data. In the case of fast processes (on a typical scale of minutes or shorter times), PALS can characterize the state of the microstructure only at the beginning (before starting the monitored process, for example the reaction) and at the end of the process investigated. In certain cases, if the monitored process (reaction) can be interrupted, PALS can successfully monitor changes in the microstructure over time and characterize the individual stages of the polymer network formed. In this way, it is possible to study the course of crosslinking in the case of conventional light-initiated polymerization, in which the reaction runs only during the illumination of the reaction volume. An example is intermittent photopolymerization, where after a short light pulse, which supplies energy to generate radicals and triggers a crosslinking reaction, the stage of crosslinking is measured in the dark using an *o*-Ps probe. From the lifetimes and their distribution, it is possible to determine the free-volume characteristics of different stages of crosslinking of the examined sample as well as the reaction kinetics. An example is the crosslinking of a commercial mixture of three types of acrylates in SPOT LV [1]. By setting a low-light intensity, it is possible to slow down the reaction so that intermittent illumination lasting a few seconds achieves multiple and repeated gradual crosslinking of the polymer until the stage of complete curing. The individual degrees of crosslinking achieved were characterized by *o*-Ps lifetime spectra in "dark" intervals. The both evolutions of microstructural shrinkage (Fig. 1) and free volume distribution for different stages of the investigated system were found: from liquid monomers, through the gel stage to the cured polymer.

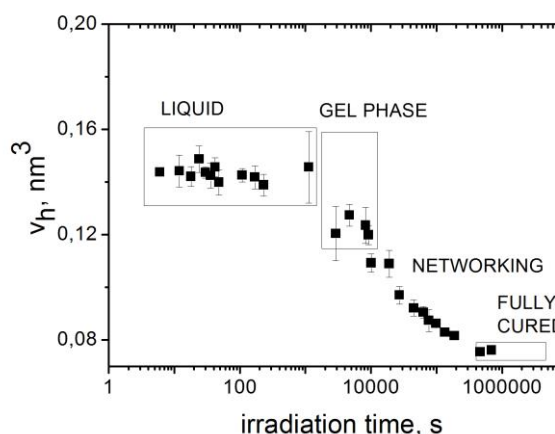


Fig.1. Microstructural shrinkage versus irradiation time during photopolymerization of SPOT LV.

[1] H. Švajdlenková, O. Šauša, G. Peer and C. Gorsche, *RSC Adv.* **8**, 37085 (2018)