

FRactal Structure of Epoxy-Silica Nanocomposites Synthesized by the Sol-Gel Method

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Sol-gel synthesized epoxy-silica nanocomposites find applications in many fields due to the possibility to widely range the structure and properties of such systems. The goal of this work was to investigate the morphology of anhydride cured epoxy-silica hybrids with different ways of silica sol formation (in the absence and in the presence of epoxy resin).

The polymeric matrix was formed by combination of EPONEX 1510 diepoxide, curing agent *iso*-methyltetrahydrophthalic anhydride and accelerator of curing reaction 2,4,6-*tris*-*N,N*-dimethylaminomethylphenol. The sol of silica particles was obtained by acid-catalyzed hydrolytic polycondensation of tetraethoxysilane and 3-glycidoxypropyltriethoxysilane in 2:1 molar ratio. The silica particles' content in the composites varied from 0.5 to 6.0 wt. %. The sol was formed in the absence and in the presence of epoxy resin at ambient temperature during 24 h. After that it was evacuated from volatiles (for systems with sol formation in the absence of epoxy oligomer it was added before evacuation, accompanied by thorough homogenization) and cured stepwise from 120 to 180 °C.

The structure of the composites was investigated by scanning electron microscopy (SEM) and small-angle X-ray scattering (SAXS). As it was shown by SEM results in both cases of sol formation the samples with uniform distribution of silica particles were obtained at SiO₂ content up to 1.5 wt.% (Fig.). The higher the nanofiller content the larger particles were formed, especially for the composites with sol formation in the absence of epoxy resin.

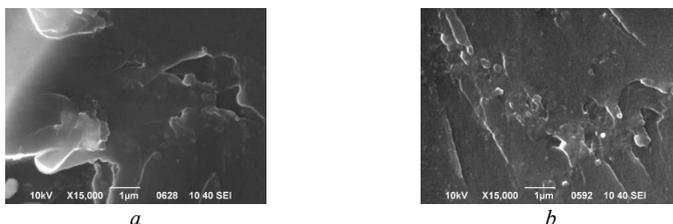


Fig. SEM images of the samples with 1 wt.% of silica with sol formation in the presence (a) and absence (b) of epoxy resin ($\times 15\,000$)

The analysis of SAXS profiles of the composites has shown the formation of the systems with fractal hierarchy. Most of the samples demonstrate one structural level. For the composites with sol formation in the absence of epoxy resin the silica particles were branched mass fractal objects. At the same if the sol of silica particles was formed in the presence of epoxy oligomer, the primary particles in the composites were rough surface fractals. And for the samples with concentrations of SiO₂ 3.0 and 6.0 wt.% the primary particles formed larger aggregates that were mass (3.0 wt.%) and surface fractals (3.0 wt.%).

Thus, it was shown that the procedure of sol formation resulted in the composites with different size of the particles and the fractal structure of the aggregates with one or two levels of structure organization, depending on content of SiO₂.