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ULTRAHIGH-TEMPERATURE PHTHALONITRILE NANOCOMPOSITES WITH FUNCTIONALIZED SILICON-BASED NANOPARTICLES: STRUCTURE, DYNAMICS AND PROPERTIES



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series of heterocyclic densely cross-linked hybrid Ananocomposites were synthesized from bisphenol A-based bisphthalonitrile (BPh) with 0.5wt.% aminofunctionalized montmorillonite (MMT) nanolayers, or aminoor epoxy-polyhedral oligomeric silsesquioxane (POSS) nanoparticles. Curing was performed at 260-300°C followed by post-curing at 340-430°C. The reactive nanoparticles were covalently embedded into matrix. FTIR indicated attaining 95% polymerization degree at post-curing and the appearance of phthalocyanine, triazine and isoindoline cycles in the network structure. Nanostructure, dynamics and properties of 0.5 mm thickness films were characterized using scanning transmission electron microscopy (STEM), energy dispersive X-ray spectroscopy (EDXS), FTIR and far-infrared spectroscopy (FIRS), dynamic mechanical analysis (DMA, 0.1-10Hz), differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA). TGA/DMA/ DSC measurements were performed in air or nitrogen mediums. STEM images, EDX spectra and the histograms of Si nanodistribution indicated satisfactory dispersion and uniform distribution of POSS nanoparticles (no nanoclusters) in the matrix. Single MMT nanolayers and 2-3 nanolayers stacks prevailed in the nanocomposites. TGA indicated slight thermal degradation starting from 420-430°C, identically up to 550°C in air and nitrogen mediums. At 550-700°C, TGA curves sharply diverged: thermo-oxidative degradation

in air resulted in dropping mass to char residue of 4-7% whereas 72-74% residual was registered in nitrogen. At low mechanical losses (tan ≤0.03-0.04), four relaxations were registered for post-cured samples: non- or low-cooperative sub-Tg relaxations at 50-100°(I) and 200-250°C(II), and Tg≈460°(III) and 515-540°C(IV); for cured films, Tg=380-390°C. Transition IV was displaced to 560°C in nitrogen. DMA/DSC showed some suppression of matrix dynamics by nanoparticles: Tg increased by 20-60°C. Glass transition manifested highly cooperative ("quasi-phase transition") behavior with effective activation energy Qact>>1000kJ/ mol. Dynamic modulus of the nanocomposites E'≈(2-3)GPa decreased by 20-30% at 500°C. After scanning to 570°C in nitrogen medium, no relaxation spectrum and glass transition were observed, and E'≈3.2GPa was registered at 20-600°C. The studied nanocomposites have a perspective for use in aerospace, microelectronics, etc.

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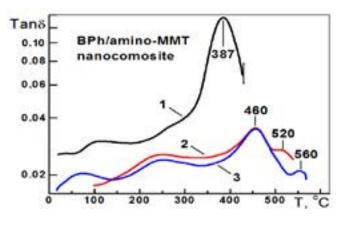


Figure 1: DMA (1 Hz). Relaxation spectra were obtained for the indicated nanocomposite at heating with the rate of 30C/min in air atmosphere up to 430°C for the sample cured up to 300°C (1) and post-cured up to 430°C (2) and for post-cured sample in nitrogen medium (3).

Recent Publications

- 1. Bershtein V et al. (2015) The impact of ultra-low amounts of introduced reactive POSS nanoparticles on structure, dynamics and properties of densely cross-linked cyanate ester resins. Europ. Polym. J. 67:128-142.
- 2. Bershtein V et al. (2015) The impact of ultra-low amounts of amino-modified MMT on dynamics and properties of densely cross-linked cyanate ester resins. Nanoscale Research Lett. 10:165-179.
- 3. Bershtein V et al. (2016) Dynamics and properties of high performance amorphous Cyanate Ester-based subnanocomposites with ultralow silica content and quasi-regular structure. Polymer. 103:36-40.
- 4. Bershtein V et al (2016) Silica subnanometer-sized nodes, nanoclusters and aggregates in cyanate ester based networks: structure and properties of hybrid subnanocomposites. Europ. Polym. J. 85:375-389.
- Bershtein V et al. (2016) Thermostable cyanate ester resins and POSS-containing nanocomposites: influence of matrix chemical structure on their properties. Polym. Adv. Technol. 27(3):339-349.

Biography

Vladimir Bershtein (PhD, 1963, DSc 1980) with his group at loffe Institute of the Russian Academy of Sciences has performed numerous experimental studies during a few decades within such polymer physics problems as the nature of relaxation transitions; physics of strength and plasticity of polymeric materials; dynamics and properties of polymer nanocomposites including thermostable (polyimide and cyanate ester-based) and biocompatible ones, and dynamics of polymer hybrids. He is the co-author of numerous publications, e.g., the first book on "DSC of Polymers: Physics, Chemistry, Analysis, Technology", reviews on Far-IR spectroscopy (Adv Pol Sci 114,1994), on the original method of Laser-interferometric creep rate spectroscopy of polymers (Adv Pol Sci 230, 2010), on polyimides and polycyanurate dynamics, etc.

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