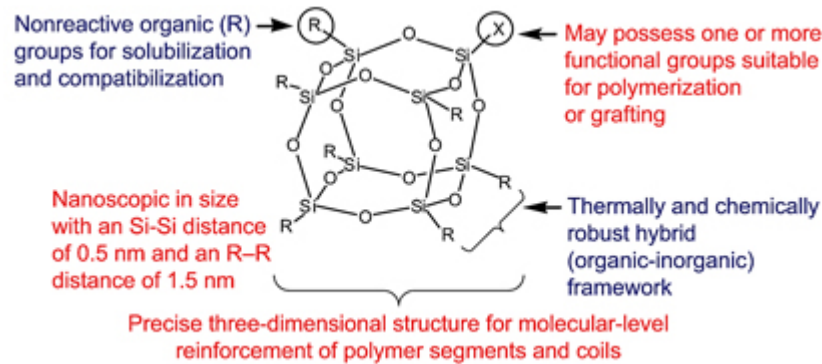


Properties of PMR Polyimides Improved by Preparation of Polyhedral Oligomeric Silsesquioxane (POSS) Nanocomposites

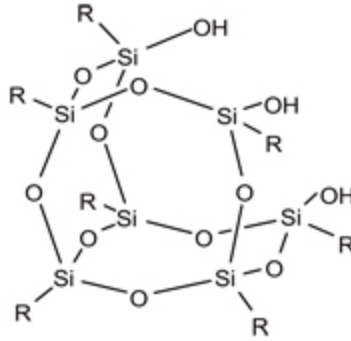


Anatomy of a POSS chemical.

Long description of figure 1. POSS has a cage structure composed of silicon and oxygen atoms. The general formula is $(\text{RSiO}_{1.5})_8$. Diagram shows nonreactive organic (R) groups for solubilization and compatibilization. The structure is nanoscopic in size with a silicon-silicon distance of 0.5 nanometers and an R-R distance of 1.5 millimeters. It may possess one or more functional groups suitable for polymerization or grafting. It has a thermal and chemically robust hybrid (organic-inorganic) framework.

The field of hybrid organic-inorganic materials has grown drastically over the last several years. This interest stems from our ever-increasing ability to custom-build and control molecular structure at several length scales. This ability to control both the composition and structure of hybrid materials is sometimes broadly referred to as nanocomposite systems. One class of hybrid (organic-inorganic) nanostructured material is polyhedral oligomeric silsesquioxane (POSS), shown in the preceding diagram. The hybrid composition gives POSS materials dramatically enhanced properties relative to traditional hydrocarbons and inorganics. An important benefit of this technology is that it makes possible the formulations of nanostructured chemicals with excellent thermal and oxidative stability. This is largely due to the inorganic component.

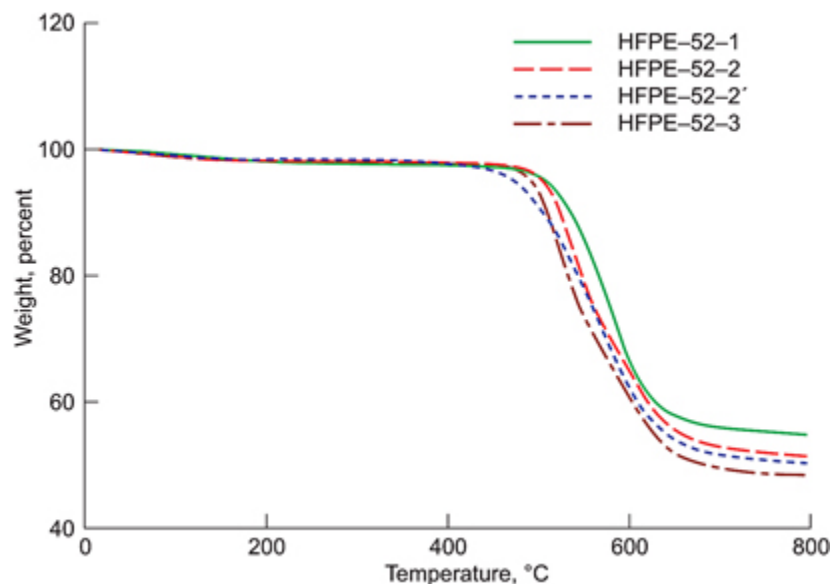
Researchers at the NASA Glenn Research Center investigated adding two different functional POSS triols (R: ethyl and phenyl) to improve the properties of a polymerization of monomeric reactants (PMR) resin (see the following diagram). Both of these POSS-triol monomers are easily dissolved in methanol, which makes them well suited for typical PMR formulation approaches.



Chemical structures of POSS triol.

The PMR polyimide resin (HFPE-II-52, a NASA second-generation polyimide resin) was prepared with different amounts of POSS triol. Both neat resins and carbon-fiber-reinforced polymer matrix composites were processed using the currently optimized HFPE-II-52 processing cycle. Physical and mechanical characterization--differential scanning calorimetry, thermogravimetric analysis (TGA), and dynamical mechanical analysis (DMA)--of resins with POSS incorporation were performed and compared with the control.

Initial experiments were performed to analyze any influence the addition of POSS to HFPE may have on the chemistry of the system. Differential scanning calorimetry results indicated that the addition of POSS did not affect the cross-linking reaction of the endcaps. In addition, Fourier transform infrared microscopy analysis showed no effect on imidization.

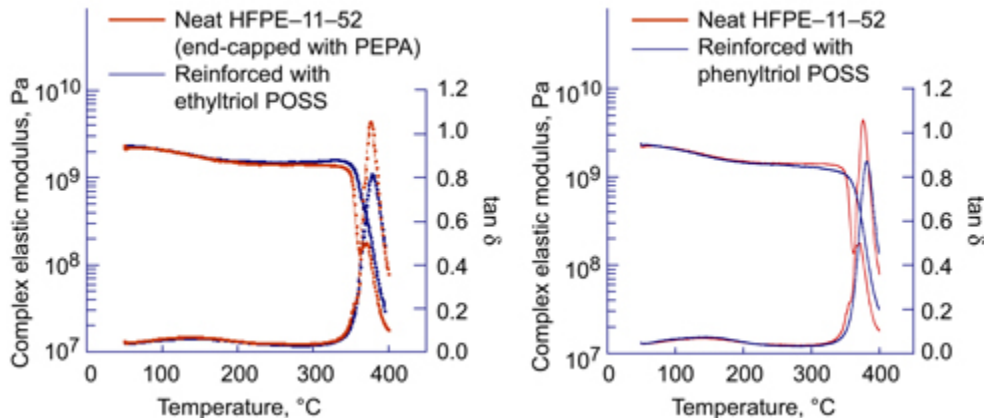


TGA traces of HFPE-II-52 containing with 15 wt% of two moieties of POSS triol.

A critical issue with aerospace resins is the high-temperature stability of the material. The preceding graph shows the TGA traces of HFPE-II-52 with POSS triols added and compared with the control sample.

By TGA, HFPE-II-52 with POSS inclusions showed an enhanced thermal stability in a nitrogen (N₂) environment. These experiments will be repeated in normal air and oxygen (O₂) environments to examine the oxidative stability.

DMA was performed to examine the thermomechanical properties of these hybrid organic-inorganic polyimides. The following graphs show that samples reinforced with POSS triol exhibit an improved high-temperature dimensional stability in comparison to the control HFPE-II-52.



Left: DMA of neat HFPE-II-52 and HFPE-II-52 reinforced with 15 wt% of ethyltriol POSS. Right: DMA of neat HFPE-II-52 and HFPE-II-52 reinforced with 15 wt% of phenyltriol POSS, where $\tan \delta$ is the (loss modulus)/(storage modulus): that is, (energy lost as heat)/(energy stored elastically).

In summary, it has been demonstrated that nanostructured chemicals such as POSS can be incorporated into high-performance resins using the standard PMR approach. The incorporation of these hybrid nanostructured chemicals will improve the thermal and dimensional stability of high-temperature thermoset polyimides.

This work was performed at the NASA Glenn Research Center by Professor Andre Lee from Michigan State University. Professor Lee worked at Glenn through the NASA Faculty Fellowship Program.

Find out more about this research at

<http://www.grc.nasa.gov/WWW/MDWeb/5150/Polymers.html>

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Programs/Projects: VSP, UEET