Fire Resistant Nanocomposites ¹ **Jeffrey W. Gilman** Building and Fire Research Laboratory National Institute of Standards and Technoloby Gaithersbug, MD 20899

Research Objective: To determine the effect of clay nanoparticles on the flammability of thermally-stable polymers with potential for zero heat release rate in flaming combustion, and determine the mechanism of action.

Approach: The solid-state mechanism of combustion modification of clay-polymer nanocomposites will be investigated. Clay-polymer nanocomposites with both delaminated and intercalated clay structure will be prepared using organic-modified montmorillonite clays in combination with melamine. Recent results show excellent promise for this approach. The mechanical properties of clay-polymer nanocomposites will also be characterized. The polymers to be investigated as clay-polymer nanocomposites are cyanate ester thermosetting resins and polyetherimide thermoplastics.¹

Accomplishment Description: We have found that the phenolic-based cyanate ester resins have unique flammability properties. Although, they are a very high char yield system, they are relatively flammable if not flame retarded. The large amount of char (60 % to 70 %) appears to form too slowly to prevent the violent degradation and rapid mass loss characteristic of these materials. During combustion we observed, as others have that the char cracks under the pressure from rapid volatilization. The combustion samples burn in a characteristic and unusual manner. As the resin heats up some char forms, however, it appears to crack and many gas "jets" form on the surface of the sample. These "jets" ignite and burn violently much like small blow-torches. This results in a 30 % mass loss in only 1-2 minutes. However, the flammability behavior of the cyanate ester nanocomposites is different. The char which forms during combustion appears to crack less, and the number and intensity of the gas/flame "jets" is reduced, this gives a 50 % lower heat release rate.

In other nanocomposite systems we have found, using XRD and TEM, that a common carbonaceous-silicate nanocomposite char forms during combustion. The d-spacing of this multi-layer carbonaceous-silicate char is 1.3 nm for chars derived from nylon-6 clay nanocomposite and epoxy clay nanocomposites. We see the same type of char here. For several of the cyanate ester clay nanocomposites the ordered multi-layered carbonaceous-silicate char structure observed by XRD has a d-spacing of 2.1 nm. This

¹ Certain commercial equipment, instruments, materials or companies are identified in this paper in order to adequately specify the experimental procedure. This in no way implies endorsement or recommendation by NIST.

corresponds to a factor of 2 or 3 times as much carbonaceous material sandwiched between the silicate layers as we found in previous nanocomposite chars..

Significance: Because of this FAA funded program polymer-clay nanocomposites are currently under investigation in several industrial groups as a new general approach to reduce the flammability of a wide range of polymers useful for composites, adhesives, fabric, and foam materials used in aircraft interiors. In addition an Industry- Government Consortium has been formed to investigate the flammability properties of these unique materials.

Expected Results: We will be using radiative gasification coupled with TEM and other techniques to further study the mechanism of flammability improvement in polymer clay nanocomposites. We will also be preparing polyetherimide-clay nanocomposites to evaluate the effectiveness of this approach in these very low heat release materials.

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Figure 1. Heat release rate (HRR) data for cyanate ester resin PT–15 and PT–15 with: sodium montmorillonite (Na-MMT), methyl, tallow, bis-2hydroxyethyl, ammonium montmorillonite (OH–MMT), dimethyl, ditallow ammonium montmorillonite (A–MMT). A mass fraction of 10 % treated clay was used in each case. This data shows the importance of using the appropriate organic treatment on the clay. The OH-MMT treatment increases flammability of the resin while the other treatments (A-MMT, and Na-MMT) significantly reduce the heat release rate.