

Microscale Calorimetric Method for Determining Fire Properties of Materials

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Research Objective: To develop an instrument and methodology for evaluating the flammability of research quantities of materials (< 1g) being developed for aircraft through the Fire Research Program. The data will be correlated with bench-scale fire calorimetry data of materials to allow prediction of full-scale fire performance from milligram samples.

Approach: Separately reproducing the solid state (pyrolysis) gas phase (combustion) chemical processes of flaming combustion in a laboratory test (1-3) provides a method for studying the effect of material composition on flammability and allows for a quantitative determination of the heat release as it occurs in fires. A schematic of the microscale calorimeter is shown in figure 1. In order to approximate surface conditions in a real fire samples are thermally decomposed in an inert environment at a constant heating rate of several degrees Kelvin per second. The volatile thermal decomposition products are swept from the pyrolyzer by nitrogen and mixed with excess oxygen prior to entering a high temperature combustion furnace (~ 1000°C) to force complete combustion. The products of combustion (water, carbon dioxide, halogen acids, etc.) are removed from the gas stream and the remaining oxygen/nitrogen mixture is analyzed to measure oxygen depletion. The proportionality between oxygen depletion and heat of combustion allows direct calculation of the rate of heat release and total heat release. A size- and rate-independent material flammability parameter, the heat release capacity (J/g-K), is obtained by dividing the peak heat release rate (W) by the initial sample mass (g) and linear heating rate (K/s). The char yield of the polymer, obtained by weighing the sample before and after the experiment, allows calculation of the heat of combustion per unit mass of evolved gas.

Accomplishment Description: Using this test method, a database on material heat release has been generated, a portion of which is shown in figure 2. Standard materials, including those currently used in aircraft, have been used to benchmark the fire performance of new materials being developed for use in aircraft through the Fire Research Program. Order-of-magnitude improvements in the fire performance have been observed in research materials with potential for use in commercial aircraft.

Significance: This test has greatly accelerated the development of ultra fire resistant materials by providing for the first time a rapid, quantitative, cost effective method for determining fire properties of materials which are available only in research (gram) quantities. The results of this test have guided the materials development activities in the Fire Research Program.

Expected Results: Combustion microcalorimetry testing of commercial, precommercial, and research materials will provide the chemical structure-fire property correlations needed to design next-generation, fire resistant cabin materials. Data from combustion microcalorimetry tests correlates well with data (4) from the FAA bench scale heat release rate test FAR 25.853(a-1) as shown in figure 3. Candidate fireproof materials for next-generation aircraft have been identified and are being scaled up for production.

References:

1. R.N. Walters and R.E. Lyon, *A Microscale Combustion Method for Determining Flammability Parameters of Materials*, Fire Resistant Materials: FAA Progress Report 1998.
2. R.E. Lyon and R.N. Walters, *A Pyrolysis-Combustion Flow Calorimeter Study of Polymer Flammability*, The Ninth Annual BCC Conference on Flame Retardancy 1998.
3. R.E. Lyon and R.N. Walters, *Microscale Heat Release Rate of Polymers*, Fire and Materials '98, 1998.
4. W. Bassett, *Flame Retardant Thermoplastics Materials for Aircraft Interiors*, Fire Retardant Engineering Polymers and Alloys, 1989.

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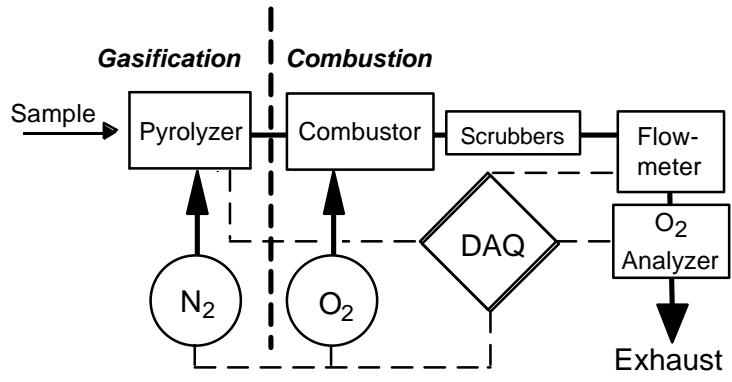


Figure 1: Schematic of Microscale Calorimeter showing gasification and combustion stages.

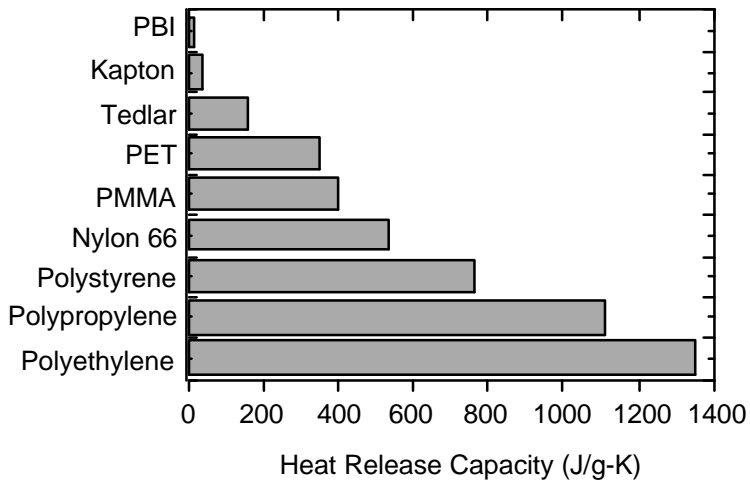


Figure 2: Range of heat release capacities measured in the microscale calorimeter for several polymeric materials.

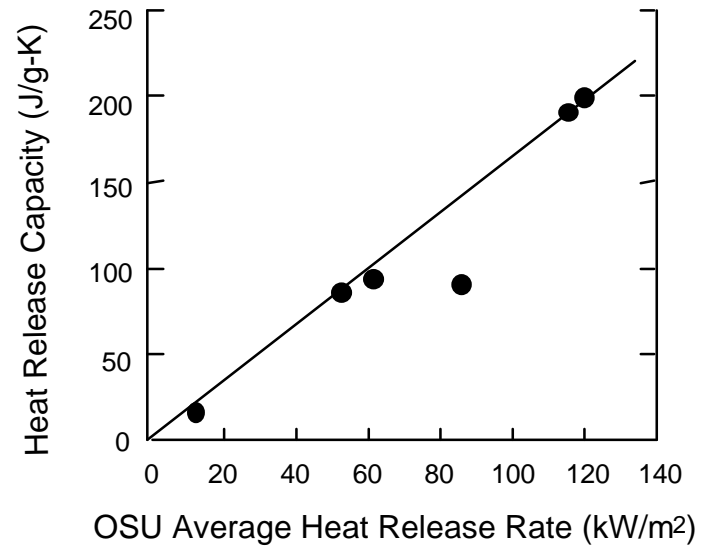


Figure 3: Plot of heat release capacity measured in the microscale calorimeter to the average heat release measured in the OSU calorimeter for the same material.