

N 77 - 18102

MATERIALS RESEARCH FOR AIRCRAFT FIRE SAFETY

Demetrius A. Kourtides and John A. Parker
NASA Ames Research Center

Richard W. Bricker
NASA Johnson Space Center

22

SUMMARY

The thermochemical and flammability characteristics of two polymeric composites currently in use and seven others being considered for use as aircraft interior panels are described. The properties studied included: (1) limiting oxygen index of the composite constituents; (2) fire containment capability of the composite; (3) smoke evolution from the composite; (4) thermogravimetric analysis; (5) composition of the volatile products of thermal degradation; and (6) relative toxicity of the volatile products of pyrolysis. The performance of high-temperature laminating resins such as bismaleimides is compared with the performance of phenolics and epoxies. The relationship of increased fire safety with the use of polymers with high anaerobic char yield is shown. Processing parameters of the state-of-the-art and the advanced bismaleimide composites are detailed.

INTRODUCTION

The purpose of this program was to assess the relative flammability and thermochemical properties of some typical state-of-the-art and candidate experimental aircraft interior composite panels, and to develop an understanding of the relationship of flammability and thermochemical properties of these systems. Specifically, aircraft interior composite panels were characterized as to their thermal stability, oxygen index of the composite components, smoke evolution from the panels, fire containment capability or fire endurance, thermal conductivity, identification of the pyrolysis effluents, relative toxicity of the degradation products and mechanical properties such as tensile strength.

As shown in figure 1, composite sandwich panels constitute most of the surface of aircraft interiors as sidewalls, partitions, ceiling panels, and overhead stowage bins. Approximately 1000 m² of the surface area of a typical wide body is made from composite panels weighing approximately 1600 kg. Currently used composite panels meet or exceed regulatory requirements (ref. 1) and offer excellent aesthetic, serviceability, maintenance, and other properties. However, additional improvements are being sought by industry, airframe manufacturers, and government agencies to reduce ignition susceptibility, fuel contribution, smoke and toxic fume emission, and to increase fire containment capability of these panels in selected areas such as lavatories and galleys (refs. 2-8).

Experimental composite panels that could offer improved fire resistance and smoke reduction in aircraft fires are now being developed and tested. In this program, nine different types of experimental composite panels were evaluated in terms of their flammability properties. Two of these were typical state-of-the-art interior panels and seven were experimental.

The composite panels used by most airframe manufacturers as interior paneling are sandwich panels that vary slightly in configuration, component composition, thickness, and density depending on the type of aircraft in which they are used and the specific application. In general, the panel consists of a clear polyvinyl fluoride film which is bonded to a polyvinyl fluoride decorative film bonded to a fiberglass-resin laminate. The complete laminate is bonded to an aromatic polyamide honeycomb core either when the prepreg is uncured or with a suitable adhesive film. The other side of the panel is similar except for the absence of the decorative film. The components of the panels are shown in figure 2.

SYMBOLS

The International System of Units (SI) is used.

D	percent light transmittance
D _s	specific optical density, $D_s = \frac{(131.58) \text{ Log}_{10} 100}{D}$
D _m	specific optical density, maximum
LOI	limiting oxygen index, $O_2/(O_2 + N_2)$
TGA	thermogravimetric analysis
Y _c	char yield, percent weight remaining
T _d	polymer decomposition temperature

DESCRIPTIONS OF COMPOSITE PANELS

Nine types of composite panels were evaluated. Three types of resin systems were used for the fabrication of the laminates used in these composites: epoxy, bismaleimide, and various modifications of phenolic resins. The exact formulation for the phenolic resins was not available from the manufacturers. The epoxy resin used was bisphenol-A-type cured with methylene dianiline. Bismaleimide is an addition-type polyimide based on short, preimidized segments very similar in nature to those of condensation polyimides. The resin is produced by mixing a bismaleimide with a diamine at a specified ratio resulting in a resin with controlled crosslink density. The resin polymerizes thermally without loss of volatiles in contrast to the condensation polymers which cure

with loss of water. The general chemical structure of the laminating resins used is shown in figure 3. The components and composition of the panels are shown in tables I-III.

All composites fabricated were 2.54 cm thick. Composites 1-6 had a decorative surface of polyvinyl fluoride film printed with an acrylic ink and bonded to laminate consisting of various types of fiberglass preimpregnated with various types of phenolic resins. The laminates were adhered to the hexagonal-cell aromatic polyamide honeycomb structure using various types of phenolic resin-fiberglass adhesive ply. Composite 1 was considered a typical state-of-the-art phenolic resin panel.

Resins used in the preparation of the laminates for composites 1-6 were obtained commercially and are designated as phenolic types A through G. The decorative laminates of composites 1-6 were press-bonded to the honeycomb using an adhesive bond ply at 160° C for 12 min at 689.6 kN/M² pressure. The sandwich panel was then cured at 123° C for 1 hr with 50 mm Hg minimum vacuum bag pressure.

Composite 7 was composed of a laminate of bismaleimide-fiberglass adhered to the aromatic polyamide honeycomb which was filled with a polyquinoxaline foam made from quinone dioxime. Processing of this panel is as follows: The aromatic polyamide honeycomb is coated with a pasty mixture of quinone dioxime-phosphoric acid. The amount (dry weight) of coating used is approximately 0.20-0.23 g/cm² (honeycomb face area) for a honeycomb structure with a 0.312-cm cell size. After application, the paste is air dried. The coated honeycomb is heated at a temperature of 150° C for 3 hr to form the carbonaceous char of polyquinoxaline, and any excess amount is removed from the faces of the honeycomb. Prepregs are prepared using the bismaleimide resin and one piece of 181 style E-glass cloth to form flat laminates. The impregnated glass cloth is dried 15 min at 80° C and then 30 min at 93° C. The prepreg is cured in a press at 180° C for 1 hr and subsequently post-cured in an oven at 250° C for 8 hr. The laminates are adhered to the filled honeycomb structure using a polyimide adhesive film using contact pressure in a heated press at 180° C for 1 hr. Processing of this type of panel has been described previously in detail (refs. 9-10). Composite 8 is a typical state-of-the-art composite panel. In general, panel consists of a decorative surface bonded to a laminate and a honeycomb core. The process for producing the decorative surface consists of silkscreening the required decor on a 0.005-cm polyvinyl fluoride film by a continuous web process. After drying, a 0.0025-cm transparent polyvinyl fluoride film, coated on one side with polymethyl methacrylate, is bonded to the decorative film to provide protection for the printed surface. This laminate is then bonded to one ply of epoxy-preimpregnated 181 E glass cloth which may have a canvas or other texture applied during this bonding operation. Time, temperature, and pressure vary depending on the texture applied. The current core material for sandwich paneling is a polyamide, hexagonal-cell honeycomb structure. The cell size varies 0.312 cm, 0.625 cm, or 0.937 cm depending upon strength and application requirements. The current method of bonding the skins to the core consists of using an epoxy resin-preimpregnated bond ply over which is applied the 181 E glass cloth/polyvinyl fluoride decorative laminate. The resin in the bond ply provides the adhesive to bond the skin to

the honeycomb and the decorative laminate to the bond ply. Curing is accomplished at 100° C with 50 cm Hg minimum vacuum bag pressure. For panels requiring decorative laminates on one side only, the bond ply provides the backside skin. Edge close-outs consist of either polyurethane foam or a phenolic microballoon-filled epoxy potting compound. Processing of this type of panel has been described previously in detail (refs. 9, 11, and 12).

The weight distribution of the panel components and some of the thermochemical properties of these components were determined. These included anaerobic char yield, polymer decomposition temperature, and limiting oxygen index. It can be seen in table IV that with the exception of the honeycomb and glass, the other components have a fairly low char yield and a corresponding low oxygen index.

Composite 9 was similar to composite 7 except that the bismaleimide-fiberglass honeycomb used is partially filled with a syntactic foam consisting of a mixture of carbon microballoons and bismaleimide resin. The prepregs for the facesheets are prepared in a manner similar to that described previously for composite 7. The core consists of a bismaleimide-fiberglass honeycomb filled with carbon microballoons bound with bismaleimide resin. The carbon microballoons are prepared by pyrolyzing phenolic microballoons in a nitrogen atmosphere. A stainless steel container is filled with phenolic microballoons and enclosed in a larger stainless steel container with a nitrogen inlet to provide an oxygen-free atmosphere. The assembly is placed in a larger furnace. The pyrolysis cycle is as follows: room temperature to 816° C in 4 hr, hold at 816° C for 4 hr, and cool to room temperature in 2 days. Pyrolyzed carbon microballoons must be cooled to 38° C before removal of the nitrogen blanket to prevent spontaneous ignition of the carbon microballoons. After pyrolysis, the carbon microballoons are no longer free-flowing and are agglomerated as large cakes. To break them into smaller agglomerates, the cake microballoons are placed in a container with isopropanol (ratio of 1 kg balloons/7 liters solvent) and mixed in a paint shaker for 15 min. The slurry is then screened through a 20-mesh screen to remove the larger non-separated agglomerates. The screened isopropanol/carbon microballoon slurry is now ready for core impregnation. The processing cycle for this composite is shown in figure 4.

The equipment shown in figure 5 is used to fill the cores of the fiberglass-bismaleimide honeycomb with the prepared carbon microballoons. A high-density 0.3-cm cell aluminum honeycomb is fitted and restrained on the bottom inside of the vacuum filling box. A nylon screen (120 mesh) is placed between the aluminum support honeycomb and the fiberglass-reinforced polyimide honeycomb to retain the microballoons. High vacuum is not required to effectively impregnate the honeycomb, but a high volume of air displacement is required. A vacuum reservoir chamber is pumped to a vacuum of approximately 10 mm Hg. The filled honeycomb cores, sandwiched between two nylon, fine-mesh screens and between two aluminum support honeycombs, are dried for 16 hr in an air-circulating oven at 93° C. After drying, the microballoon fill is saturated with a solution of bismaleimide resin in N-methyl-2-pyrrolidone solvent. The foamed honeycomb is heated for 2 hr at 93° C and for 1 hr at 204° C to completely cure the bismaleimide binder. For the microballoon resin combination, the resin by weight is approximately 4-10 percent.

The assembly of the sandwich panel consists of bonding the face sheets to the microballoon-filled fiberglass bismaleimide honeycomb panel with a polyimide film adhesive. The assembly is then placed in a platen press at 204° C and cured for 2 hr at 700 kN/m². Afterward, the panel is cured for 24 hr at 254° C to remove volatile materials and to achieve reduced smoke characteristics.

TEST RESULTS AND ANALYSIS

Thermochemical Characterization of Composites

Samples of the nine types of composites were cut to a size of 2.5 cm x 2.5 cm x 2.5 cm and were ground uniformly to approximately 250 mesh. The samples were subjected to the following thermochemical studies in order to (1) determine the relative thermal stability of the samples under anaerobic and oxidative conditions, (2) determine the major volatile products produced from the pyrolysis of the samples in vacuum, and (3) determine the relative toxicity of the pyrolysis effluents by exposing animals to them.

Thermogravimetric Analyses

Thermal analyses of the composites were conducted on a DuPont 950 thermogravimetric analyzer (TGA) using both nitrogen and air atmospheres with a sample size of 10 mg. The thermogravimetric analyses data of 10° C/min heating rate in nitrogen is shown in figure 6.

The pyrolysis of the samples in air and nitrogen atmospheres was conducted to obtain a relative understanding of the pyrolysis of the samples in the furnace used to pyrolyze samples for assessing their relative toxicity as described later in the text. Pyrolysis in an air atmosphere is intended to approximate the environment in the pyrolysis tube at the start of the toxicity test, and pyrolysis in a nitrogen atmosphere is intended to approximate the environment in the pyrolysis tube during the test after the original air has been essentially displaced by pyrolysis effluent. The degradation products are continuously removed from the sample during thermogravimetric analysis, and in the relative toxicity test apparatus described later, they are conveyed only by normal thermal flow. The TGA data in the nitrogen atmosphere are considered more relevant because in the toxicity apparatus, the pyrolysis effluents that evolved at lower temperature have essentially displaced the original air by the time the temperature has reached 700° C.

Composite 9 is the most stable composite and gives the highest char yield in nitrogen. The thermogravimetric analyses data in air are shown in figure 7. All the composites except composite 7 were oxidized completely in air above 600° C and gave constant weight residues.

Analysis of Volatile Products

Samples of the composites were pyrolyzed to determine the degradation products. The apparatus for the pyrolysis is shown in figure 8. The samples were placed in quartz tubes that were 2.5 mm in diameter. Each sample tube was attached to a manifold and evacuated to 10^{-4} torr. A stopcock was inserted between the manifold and the sample tube so that the sample tube could be isolated while gas samples were being collected. An infrared cell was attached to the manifold via a stopcock; a mercury manometer and a trap were also attached to the manifold. At the beginning of a pyrolysis run, the stopcock to the vacuum pump was closed, and a furnace at 700° C was placed around the sample tube. At this point, a timer was started. The pressure of the gases evolved during the pyrolysis was monitored with the pressure gauge. After 5 min the furnace was removed, the stopcock to the sample tube was closed, and the stopcock leading to the infrared cell was opened allowing the pyrolysis gases to enter the infrared cell. After a pressure reading was taken, the stopcock leading from the infrared cell to the gas manifold was closed. Dry air was admitted to the infrared cell so that the total pressure was equal to atmospheric pressure. This was done so that the pyrolysis gases were always measured at the same total pressure, the main portion of which was dry air, thus eliminating the effects of pressure broadening. Infrared spectra were taken using a Perkin Elmer Model 180 infrared spectrometer. Finally, the sample tube was removed from the manifold, broken open, and the residual char was weighed.

Part of the material that was volatile at 700° C condensed on the sample tube as it was removed from the furnace. The analysis of this material is not included in the data presented.

Table V shows the results of the analysis of the volatile species in terms of milliequivalents. These results were obtained from samples that were pyrolyzed in a vacuum. A considerably different distribution of products might have been obtained had the samples been pyrolyzed in air, in which case the products would be a function of the partial pressure of oxygen at the sample, the temperature of pyrolysis, and time that it took the sample to reach the pyrolysis temperature. It can be seen that the maximum amount of volatiles analyzed accounted for only 18 percent, and additional compounds may be present either in the solid particulates or in the condensates. The same volatile products are shown in table VI in terms of milligrams of volatile compound per gram of initial sample.

Thermal Efficiency

The NASA Ames T-3 thermal test (ref. 13) was used to determine the fire endurance or fire containment capability of the composite panels. The apparatus is shown in figure 9. In this test, specimens measuring 25 cm x 25 cm x 2.54 cm thick are mounted in the chamber and thermocoupled on the backface of the specimen. The flames from an oil burner, supplied with approximately 5 liter/hr of JP-4 jet aviation fuel, provide heat flux to the front face of the sample in the range of 10.4 - 11.9 W/cm². Thermocouples are placed on the back of the

composite panel to determine the temperature rise as a function of time. The heat flux produced in this burn is approximately five to seven times as high as that which would normally be encountered in a compartment fire. The test was primarily designed for exterior fuel-fed fires, but it is very useful in the comparative assessment of the fire containment capabilities of aircraft interior composite panels.

The fire endurance capability of the composite panels is compared in figures 10, 11, and 12. The backface temperature rise of the panel is plotted as a function of the time in minutes when the sample is subjected to this type of fire. The dotted line is the furnace temperature in the front surface of the panel. It can be seen in figure 12 that the backface temperature of the conventional composite 8 reached 200° C in 2.5 min, whereas, it took as long as 8 min for the bismaleimide composites 7 and 9 to reach a comparable backface temperature.

Oxygen Index

The oxygen index of the components comprising the composites was determined per American Society of Testing and Materials, Test Method D-2863. The values indicated in table VII are for the laminated or composite components as they are used in the sandwich composite and not for the individual polymers. It can be seen that the laminated facesheets consisting of the bismaleimide resin offer the highest oxygen index as compared with the phenolic and epoxy facesheets. In addition, the filler foams utilized in the honeycomb structure have a very high oxygen index. Among the phenolics, composite 6 exhibited the highest oxygen index.

Smoke Evolution

Smoke evolution from the composites was determined using NBS-Aminco smoke density chambers at two laboratories: laboratory A and laboratory B. The procedure and test method used were essentially those described by NFPA 258-T (ref. 14). A detailed description of the NBS smoke chamber can be found in reference 15.

The test results obtained with the NBS smoke chamber, modified by the incorporation of an animal module accessory (ref. 16), are presented in table VIII. Values of specific optical density (Ds) at 1.5 min, 4.0 min, and specific optical density maximum (Dm) are presented; standard deviations are also given.

Composites 1 and 8 represented the state-of-the-art baseline materials. All the other composites exhibited significantly lower smoke density values, indicating that the phenolic and bismaleimide offer the advantage of smoke reduction.

A comparison of the Ds values obtained by the two laboratories is presented in table IX. In addition to possible differences in apparatus at the two

laboratories, the calculation procedures were slightly different. In laboratory A, the D_s values were obtained from individual test data and then averaged. In laboratory B, an average curve was generated by computer from the data of the individual tests, and the D_s values were obtained from the computer-averaged curve. The smoke density of composite panels similar to composite 8 has also been evaluated by Sarkos (ref. 17). Composition of the panel was essentially the same as composite 8 except the panel was 0.70 cm thick. The maximum smoke level, D_m (corrected) was 54 whereas the average in the present studies was 58.7.

Relative Toxicity

Efforts to obtain relative toxicity information by using the NBS smoke chamber with the animal module accessory were unsuccessful. The mice and rats exposed during the standard smoke tests showed no evidence of death or even incapacitation (ref. 16). The heat flux of 2.5 W/cm^2 used in the standard test procedure appears to be incapable of producing sufficient effluents from these high-performance materials.

Tests were conducted utilizing the NASA animal exposure chamber shown in figure 13 in order to determine the relative toxicity of the composites. The chamber is constructed from polymethylmethacrylate and has a total free volume of 4.2 liters; 2.8 liters are available for animal occupancy. The chamber is fitted with probes for pyrolysis gas sampling and for an oxygen analyzer. In addition, the temperature in the chamber is monitored utilizing the thermometer indicated.

The upper dome section is removable and is connected to the base section by means of a conventional toggle snap ring; the joint is sealed by an O-ring. The upper end of the dome section is provided with an aperture so that test gas can flow completely through the chamber if desired, using the gas inlet passage in the base as the other aperture. In these experiments, the gas outlet was connected to a bubbler to permit venting of pressure exceeding 2.54 cm of water, and to prevent entry of fresh air.

The sample material is pyrolyzed in a quartz tube, closed at one end with a cap and connected at the other end to the animal exposure chamber. A horizontal tube furnace is used for pyrolysis, and the pyrolysis effluents are conveyed to the animal exposure chamber by normal thermal flow. A perforated plate or barrier of polymethylmethacrylate prevents movement of mice into the pyrolysis or connecting tube. The chamber design and the activity of the freely moving mice promote distribution of the gases within the chamber.

A connecting tube between the furnace and the chamber is utilized which reduces the possibility of a significant temperature increase in the animal exposure chamber and reduces conduction of heat to the chamber itself, but it also represents dead space and additional travel distance and provides opportunity for condensation and absorption on the inner surface of the tube and absorption in any condensate present. The procedure for the assessment of relative toxicity has been described previously in detail (refs. 18 and 19).

To provide an indication of relative toxicity, 1.0 g of each of the powdered specimens of the composites was pyrolyzed at a heating rate of 40° C/min in a quartz tube to an upper temperature limit of 700° C, and the effluents conveyed by natural thermal flow into the chamber containing four Swiss albino male mice. The test was continued for 30 min, unless terminated earlier upon the death of all four animals. The highest chamber temperature recorded was 29.5° C, indicating that the pyrolysis gases were adequately cooled before entering the animal exposure chamber. Some condensation of higher boiling vapors in the connecting tube was observed, and some of the effluent gases entered the animal exposure chamber as visible heavy vapors, indicating that some higher boiling compounds did reach the animals and were not lost entirely by cooling. The lowest oxygen concentration recorded was 12 percent, indicating that hypoxia was not a significant factor in animal response. The relative toxicity to mice of the degradation products from the powdered composites when heated in this manner is shown in table X in terms of time to incapacitation and time to death.

During the 30-min exposure period, composite 9 caused no deaths in the test animals. The other composites, that is, 1-8, caused death to all of the animals in times ranging from 19.65 min to 28.31 min.

The test time-to-death was judged as the time elapsed at cessation of movement and respiration of the first test animal as judged by the observer. Time to incapacitation was judged as the time to the first observation of loss of equilibrium, collapse, or convulsions in any one of the animals, whichever came first. As a comparison, 1.0 g of wool fabric causes death to four mice in approximately 9.5 min when tested in a similar manner.

Correlation of Oxygen Index and Smoke Evolution to Char Yield

Parker et al. (ref. 20) have shown a correlation between the flammability properties of polymers and their char yield. A decrease in ease of ignition and smoke evolution was observed with high char yield polymers. The same relationship seems to exist with composites consisting of polymers and inorganic reinforcements.

The smoke density and relative anaerobic char yield of these composites was compared. It can be seen in figure 14 that in general, composites with high char yield had fairly low smoke evolution.

The limiting oxygen index of these composites was compared with their relative anaerobic char yield. It can be seen in figure 15 that in general, composites with very high char yield exhibited a high limiting oxygen index.

Thermophysical Properties

The thermophysical properties of the state-of-the-art and one advanced composite are summarized in table XI. The thermal conductivity of composite 8 was significantly higher than that of composite 9, probably due to the

absence of any insulative material in the honeycomb. The flatwise tensile strength was slightly lower.

CONCLUDING REMARKS

Composite 9, consisting of bismaleimide-fiberglass/bismaleimide honeycomb with carbon microballoons, exhibited the highest fire containment capability.

Advanced composite panels consisting of PVF/phenolic-fiberglass/aromatic polyamide honeycomb/phenolic-fiberglass (composites 2-6) and composites 7 and 9 exhibited lower smoke evolution than the state-of-the-art composite 8.

The results from the toxicity experiments indicated that the relative toxicity of the pyrolysis products of composite 9 was the lowest of all the composites tested. It should be realized, however, that these toxicity measurements are only relative, and no definite conclusions may be drawn from these studies. The methodology developed for assessing the relative toxicity is primarily designed for pure polymers and not for composite systems consisting of various polymers and fibers. Additional studies are being initiated to expose these composite constructions intact to a radiative panel heat source and thus evaluate the relative toxicity of the composite degradation products.

No definite correlation was found between the concentration of the toxic pyrolysis products of the composites and their relative toxicity to animals, indicating possibly that additional toxic species may be present both in the volatile gases, which accounted for only 18 percent of the degradation products, and in the solid particulates. Additional studies will be conducted using both gas chromatography and mass spectrometry to identify these compounds and their relative concentrations.

REFERENCES

1. **Airworthiness Standards: Transport Category Airplanes, Federal Aviation Regulations. Vol. III, Part 25, Transmittal 10. Department of Transportation, FAA, August 1972.**
2. **Aircraft, Engine, and Propeller, Airworthiness, and Procedural Proposals. Department of Transportation, FAA. Federal Register, vol. 40, June 1975, Notice 75-31, pp. 6300-6305.**
3. **Flight Standard, Services, Transport Category Airplanes, Smoke Emission from Compartment Interior Materials. Department of Transportation, FAA. Federal Register, vol. 40, Feb. 1975, Notice 75-3, pp. 6501-6505.**
4. **Flight Standards Service, Compartment Interior Materials, Toxic Gas Emission. Department of Transportation, FAA, Federal Register, vol. 39, Dec. 1974, Notice 74-38, pp. 4540-4544.**
5. **Flaming and Self Extinguishing Characteristics of Aircraft Cabin Interior Materials. Final Rep. NA-68-30 (DS-68013), FAA, July 1968.**
6. **Gross, D.; Loftus, J. J.; Lee, T. G.; and Gray, V. E.: Smoke and Gases Produced by Burning Aircraft Interior Materials. Rep. NA-68-36 (DS-68-16), FAA, June 1968.**
7. **Mott, D. R.; Spruance, W. W.; Danaher, D.; McGuire, R.; Kourtides, D. A.; Bigelow, A.; and Ault, R.: Cabin Interiors - Smoke and Fire. Proceedings of the 12th Annual Conference and Trade Exhibit of the Survival and Flight Equipment Association, Sept. 1974, pp. 114-120.**
8. **Gross, D.: Smoke and Gases Produced by Burning Interior Materials. NBS Building Science Series 18, Feb. 1968.**
9. **Kourtides, D. A.; Parker, J. A.; and Gilwee, W. J.: Thermochemical Characterization of Aircraft Interior Panel Materials. J. Fire and Flammability, vol. 6, July 1975, pp. 373-391.**
10. **Gilwee, W. J.; Parker, J. A.; Kourtides, D. A.; and Hilado, C. J.: Fire Resistant Low Density Composites. Proceedings of the National Technical Conference (NATEC) Meeting, Society of Plastics Engineers, Cleveland, Ohio, Oct. 4-7, 1976.**
11. **Kourtides, D. A.; Parker, J. A.; Hilado, C. J.; Anderson, R. A.; Tustin, B.; Arnold, D. B.; Gaume, J. G.; Binding, A. J.; and Mikeska, J. L.: Fire and Flammability, vol. 7, Jan. 1976, pp. 125-159.**

12. Kourtides, D. A.; Parker, J. A.; Leon, H. A.; Williamson, R. B.; Hasegawa, H.; Fisher, F.; Draemel, R.; Marcussen, W. H.; and Hilado, C. J.: Fire Containment Tests of Aircraft Interior Panels. J. Fire and Flammability, vol. 7, Apr. 1976, pp. 257-258.
13. Riccitiello, S. R.; Fish, R. H.; Parker, J. A.; and Gustafson, E. J.: Development and Evaluation of Modified Polyisocyanurate Foams for Low-Heating-Rate Thermal Protection. J. of Cell. Plastics, vol. 7, no. 2, Mar./Apr. 1971, pp. 91-96.
14. Smoke Density Measurements, NFPA 258-T 1974, National Fire Protection Association.
15. Lee, T. G.: Interlaboratory Evaluation of Smoke Density Chamber. Tech. Note 708, National Bureau of Standards, Dec. 1971.
16. Hilado, C. J.; and LaBossiere, L. A.: Evaluation of Smoke Density Chamber as an Apparatus for Fire Toxicity Screening Tests. J. of Combustion Toxicol., vol. 3, no. 2, May 1976, pp. 356-362.
17. Sarkos, E. P.: Measurement of Toxic Gases and Smoke from Aircraft Cabin Interior Materials Using the NBS Smoke Chamber and Colorimetric Tubes. Department of Transportation, Rep. FAA-RD-76-7, FAA, Mar. 1976, pp. 15-19.
18. Hilado, C. J.: Evaluation of the NASA Animal Exposure Chamber as a Potential Chamber for Fire Toxicity Screening Tests. J. Combustion Toxicology, vol. 2, Nov. 1975, pp. 298-314.
19. Hilado, C. J.: Relative Toxicity of Pyrolysis Products of Some Foams and Fabrics. J. of Combustion Toxicology, vol. 3, no. 1, Feb. 1976, pp. 32-60.
20. Parker, J. A.; Kourtides, D. A.; Fish, R. H.; and Gilwee, W. J.: Fire Dynamics of Modern Aircraft from a Materials Point of View. J. Fire and Flammability, vol. 6, Oct. 1975, pp. 534-553.

TABLE I.- COMPOSITION OF COMPOSITES 1-3

Component	Composite		
	1	2	3
A. Decorative surface, cm thick, percent weight	0.002 PVF clear acrylic ink, 0.005 PVF	Same as 1-A	Same as 1-A
B. Face sheet, resin/fabric, percent weight	Phenolic type A/7581 glass	Phenolic type C/7581 glass	Same as 2-B
C. Bond sheet, resin/fabric, percent weight	Phenolic type B/120 glass	Phenolic type D/120 glass	Phenolic type C/120 glass
D. Core type; thickness, cm; cell size, cm; density, kg/m ³	Aromatic polyamide-paper honeycomb; 2.413; 0.31; 48.06	Same as 1-D	Same as 1-D
E. Core filler; density, kg/m ³	None	None	None
F. Same as C	Same as 1-C	Same as 2-C	Same as 3-C
G. Same as B	Same as 1-C	Same as 2-C	Same as 3-C
H. Same as A	None	None	None
Composite density, kg/m ³	72.410	79.138	70.488

TABLE II.- COMPOSITION OF COMPOSITES 4-6

Component	Composite		
	4	5	6
A. Decorative surface, cm thick, percent weight	0.002 PVF clear acrylic ink, 0.005 PVF	Same as 4-A	Same as 4-A
B. Face sheet, resin/fabric, percent weight	Phenolic type E/7581 glass	Phenolic type F/7581 glass	Phenolic type G/7581 glass
C. Bond sheet, resin/fabric, percent weight	Same as 4-B	Phenolic type F/120 glass	Phenolic type G/120 glass
D. Core type; thickness, cm; cell size, cm; density, kg/m ³	Aromatic polyamide-paper honeycomb; 2.413; 0.31; 48.06	Same as 4-D	Same as 4-D
E. Core filler; density, kg/m ³	None	None	None
F. Same as C	Phenolic type E	Same as 5-C	Same as 6-C
G. Same as B	Same as 4-F	Same as 5-C	Same as 6-C
H. Same as A	None	None	None
Composite density kg/m ³	76.575	76.095	70.968

TABLE III.- COMPOSITION OF COMPOSITES 7-9

Component	Composite		
	7	8	9
A. Decorative surface cm thick, percent weight	None	0.002 PVF acrylic ink, 0.005 PVF	None
B. Face sheet, resin/ fabric, percent weight	Bismaleimide/120 glass	Epoxy type H/181 E glass B + C = 35.9 percent	Bismaleimide/ 181 E glass B + G = 14.1 percent
C. Bond sheet, resin/ fabric, percent weight	Polyimide adhesive	Epoxy type H/120 glass	Same as 7-C C + F = 5.1 percent
D. Core type; thickness, cm; cell size, cm; density, kg/m ³ , percent weight	Aromatic polyamide- paper honeycomb; 2.413; 0.31; 48.06	Same as 7-D 2.413, 0.31; 48.06; 20.5 percent	Bismaleimide- glass honeycomb 2.413; 0.47; 80.1; 30.3 percent
E. Core filler; density, kg/cm ³ , percent weight	Quinone dioxime foam	None	Carbon micro balloons with 5 percent bismaleimide; 112; 50.5 percent
F. Same as C	Same as 7-C	Same as 7-C F + G = 35.9 percent	Same as 7-C
G. Same as B	Same as 7-B	Same as 7-B	Same as 9-B
H. Same as A	None	Same as 7-A A + H = 7.7 percent	None
Composite density, kg/m ³	110	95	130

TABLE IV.- TYPICAL PROPERTIES OF COMPONENT MATERIALS
IN STATE-OF-THE-ART PANELS

Material	Weight, percent	Char yield N ₂ , 700°C, percent	Polymer decomposition temperature, °C N ₂	Limiting oxygen index, $\frac{O_2}{N_2 + O_2}$
Polyvinyl fluoride	7.6	10	400	20
Glass: SiO ₂	41.9	100	-	>70
Epoxy; 4,4'-isopro- pylidenediphenol (bisphenol A)	30.0	23	180	21
Polyamide: poly (N-phenylene isophthalamide)	20.5	48	420	48

TABLE V. - MAJOR PRODUCTS AT 23° C FROM THE PYROLYSIS OF COMPOSITES IN
VACUUM AT 700° C FOR 5 MIN

Composite	Weight loss, percent (a)	Volatile weight loss, percent (b)	Volatiles, meq/g								Total meq/g (d)
			CO ₂	CO	CH ₄	HCO _x	C ₂ H ₆	PH ₃	HC	EC	
1	48.8 ± 2.1	13.8 ± 0.1	2.02 ± 0.53	0.36 ± 0.08	0.74 ± 0.04	0.24 ± 0.03	5.05 ± 0.01	-	1.63 ± 0.1	5.03 ± 0.18	
2	48.8 ± 2.1	12.0 ± .8	1.54 ± .07	.54 ± .09	1.26 ± .20	.32 ± .03	.06	-	1.76 ± .32	5.64 ± .08	
3	39.9 ± 2.2	12.9 ± ?	1.93 ± .14	.50 ± .03	.92 ± .24	.28 ± .02	.04 ± .01	-	2.16 ± .54	5.80 ± .40	
4	41.9 ± 2.8	14.4 ± .6	2.22 ± .17	.51	1.06 ± .02	.26 ± .01	.06 ± .01	-	1.60 ± .22	5.68 ± .05	
5	42.0 ± 1.3	12.6 ± .1	1.89 ± .14	.52 ± .05	.90 ± .30	.26 ± .01	.042 ± .001	-	1.94 ± .46	5.51 ± .51	
6	42.6 ± 1.1	12.4 ± 1.3	1.92 ± .20	.42 ± .09	.80 ± .03	.22 ± .01	.062 ± .005	-	1.72 ± .23	5.02 ± .50	
7	35.8 ± 7.0	10.8 ± 1.8	1.81 ± .34	.32 ± .09	.26 ± .02	.18 ± .03	.04 ± .03	0.28 ± 0.28	.16 ± .02	3.45 ± .31	
8	43.6	11.6	1.95	.22	.54	.12	.53	-	.62	3.56	
9	26.4 ± .3	18.0 ± .1	3.53 ± .10	.51 ± .06	.08 ± .04	.22 ± .02	-	-	.21 ± .12	4.22 ± .01	

$$^a \text{Weight loss, percent} = \left[\frac{(\text{initial wt. sample}) - (\text{final wt. sample})}{(\text{initial wt. sample})} \right] \cdot 100$$

$$^b \text{Volatile weight loss, percent} = \left[\frac{\sum (\text{milliequivalents})_i (\text{molecular wt.}_i)}{(\text{initial wt. sample}) (1000)} \right]$$

^cH₂ determined by difference

$$^d \text{Total volatile, determined from: } \text{meq/g} = \frac{((P)/(R)) (V)}{(P)/(T)} \left(\frac{1}{(\text{initial wt. sample})} \right)$$

TABLE VI.- MAJOR VOLATILE PRODUCTS AT 23° C FROM THE PYROLYSIS
OF COMPOSITES IN VACUUM AT 700° C FOR 5 MIN

Composite	Quantity ^a						
	CO ₂	CO	CH ₄	HCN	C ₆ H ₆	NH ₃	H ₂
1	88.9	10.1	11.8	6.5	3.9		3.3
2	67.8	15.1	20.2	8.6	4.7		3.5
3	84.9	14.0	14.7	7.6	3.1		4.3
4	97.7	14.3	17.0	7.0	4.7		3.2
5	83.2	14.6	14.4	7.0	3.3		3.9
6	84.5	11.8	12.8	5.9	4.8		3.4
7	79.6	9.0	4.2	4.9	3.1	4.8	0.3
8	85.5	6.2	9.4	3.2	4.1		1.2
9	155.3	14.3	1.3	5.9		3.6	

^a Milligrams of volatile compound at 23° C per gram of initial sample.

TABLE VII.- LIMITING OXYGEN INDEX FOR COMPOSITE COMPONENTS

Composite	Composite component	LOI @ 23° C O ₂ /(N ₂ + O ₂)
1	PVF, phenolic A/7581 glass, phenolic B/120 glass	27
	aromatic polyamide-paper	32
	phenolic A/7581 glass, phenolic B/120 glass	26
	average	28.3
2	PVF, phenolic C/7581 glass, phenolic D/120 glass	45
	aromatic polyamide-paper	32
	phenolic C/7581 glass, phenolic D/120 glass	32
	average	36.3
3	PVF, phenolic C/7581 glass, phenolic C/120 glass	38
	aromatic polyamide-paper	32
	phenolic E/120 glass (2 plies)	33
	average	35.3
4	PVF, phenolic E/7581 glass, phenolic F/120 glass	47
	aromatic polyamide-paper	32
	phenolic E/120 glass (2 plies)	30
	average	35.3
5	PVF, phenolic F/7581 glass, phenolic F/120 glass	44
	aromatic polyamide-paper	32
	phenolic F/120 glass (2 plies)	32
	average	36
6	PVF, phenolic G/7581 glass, phenolic G/120 glass	74
	aromatic polyamide-paper	32
	phenolic G/120 glass (2 plies)	36
	average	47.3
7	Bismaleimide/120 glass/polyimide	99
	aromatic polyamide-paper	32
	quinone dioxime foam	100
	bismaleimide/120 glass/polyimide	99
	average	82.5
8	PVF, epoxy H/181E glass, epoxy H/120 glass	29
	aromatic polyamide-paper	32
	epoxy H/181 glass, epoxy H/120 glass	28
	average	29.6
9	Bismaleimide/181E glass/polyimide	62
	bismaleimide/glass	58
	carbon microballoons/bismaleimide	85
	bismaleimide/181 glass/polyimide	62
	average	66.7

TABLE VIII.- SMOKE EVOLUTION FROM COMPOSITE PANELS^a

Composite	Specific optical density			No. of tests
	Ds, 1.5 min	Ds, 4.0 min	Dm	
	Flaming condition			
1	44.7 ± 13.3	51.7 ± 6.9	53.3 ± 8.9	3
2	3.9 ± 3.9	5.7 ± 4.1	8.6 ± 3.0	3
3	11.6 ± .9	13.6 ± 3.2	17.1 ± 6.4	3
4	7.1 ± 5.5	7.2 ± 5.3	8.4 ± 5.8	3
5	9.2 ± 8.2	12.2 ± 8.5	15.3 ± 8.5	3
6	8.8 ± 8.2	10.3 ± 7.3	16.0 ± 6.7	3
7	1.3 ± 1.0	4.5 ± 3.5	20.4 ± 4.7	2
8	48.3 ± 7.4	58.7 ± 6.2	59.1 ± 6.4	3
9	1.0 ± .7	4.9 ± 4.0	18.2 ± 7.9	4
	Nonflaming condition			
1	11.7 ± 4.5	19.2 ± 3.1	20.2 ± 3.3	3
2	.4 ± .2	.7 ± .2	1.6 ± .2	3
3	1.0 ± .7	1.2 ± .7	2.0 ± 1.3	3
4	.7 ± .1	0.7 ± .2	1.5 ± .2	3
5	1.2 ± .2	2.6 ± .3	5.2 ± .7	3
6	2.1 ± .8	2.2 ± .6	2.7 ± .7	3
8	2.6 ± .3	11.2 ± 1.1	19.0 ± 1.9	3

^aNBS smoke chamber, 2.5 W/cm²

TABLE IX.- SMOKE EVOLUTION FROM COMPOSITE PANELS FROM TWO CHAMBERS^a

Composite	Specific optical density											
	Ds, 1.5 min			Ds, 4.0 min			Dm					
	Chamber A	Chamber B	Average	Chamber A	Chamber B	Average	Chamber A	Chamber B	Average			
1	44.7	--	44.7	51.7	--	51.7	53.3	--	53.3			
2	3.9	10.5	7.2	5.7	15.5	10.6	8.6	19.4	14.0			
3	11.6	12.7	12.2	13.6	15.9	14.8	17.9	17.4	17.6			
4	7.1	11.7	9.4	7.2	13.9	10.6	8.4	16.1	12.2			
5	9.2	16.7	13.0	12.2	20.7	16.4	15.3	22.8	19.0			
6	8.8	13.3	11.0	10.3	16.6	13.4	16.0	7.3	16.6			
7	1.3	--	1.3	4.5	--	4.5	20.4	--	20.4			
8	48.3	57.6	53.0	58.7	57.5	58.1	59.1	58.3	58.7			
9	1.0	--	1.0	4.9	--	4.9	18.2	--	18.2			

^aNBS smoke chamber, 2.5 N/cm², flaming

TABLE X.- RELATIVE TOXICITY OF PYROLYSIS PRODUCTS
FROM COMPOSITE PANELS^a

Panel	Test	Time to incapacitation, min	Time to death, min
1	1	18.1	28.31 ± 1.67
	2	21.9	25.21 ± 3.51
	3	16.3	25.83 ± 1.02
	4	18.9	22.90 ± 1.42
	Mean	18.8	25.56 ± 2.76
2	1	20.9	26.74 ± .89
	2	21.0	24.90 ± .11
	Mean	21.0	25.82 ± 1.13
3	1	19.0	24.52 ± .69
	2	22.1	25.35 ± .97
	Mean	20.6	24.94 ± .90
4	1	20.5	24.17 ± 3.01
	2	19.3	23.48 ± .31
	Mean	19.9	23.82 ± 2.01
5	1	20.3	26.18 ± 1.83
	2	19.7	22.48 ± .52
	Mean	20.0	24.33 ± 1.17
6	1	17.1	19.65 ± .31
	2	20.9	22.90 ± .96
	Mean	19.0	21.28 ± .63
7	1	22.8	27.40 ± 1.46
	2	24.8	28.28 ± .70
	Mean	23.8	27.84 ± 1.16
8	1	18.5	27.50 ± 1.86
9	1	8.7	N.D. ^b
	2	N.I. ^c	N.D.

^aFour swiss albino mice in 4.2 liter exposure chamber, 30 min exposure; 1.0 g powdered specimens pyrolyzed at 40 C/min to 700° C.

^bN.D. - No deaths.

^cN.I. - No incapacitation observed.

TABLE XI.- PROPERTIES OF COMPOSITES 8 AND 9

Property	Composite 8	Composite 9
Bulk density range, kg/m ³	90-96	109-113
Flatwise tensile strength, at 23° C, kN/m ²	690	500-610
Vertical burn test	Passes	Passes
Thermal conductivity at 23° C, W-cm/cm ² -°C	1.296 - 1.44 × 10 ⁻³	4.932 × 10 ⁻⁴
Smoke density (specific optical density, flaming condition, 2.5 W/cm ²)	Ds 1.5 min:53.0 Ds 4.0 min:58.1 Dm:58.7	1.0 4.9 18.2
Limiting oxygen index, O ₂ /(N ₂ + O ₂)	Epoxy-fiberglass:29 Aromatic polyamide:32 Composite: 29.6	Bismaleimide-fiberglass/ Polyimide:62 Carbon microballoons, Bismaleimide:85 Bismaleimide-fiberglass:58 Composite:66.7

TABLE XI.- Concluded

Property	Composite 8	Composite 9
Relative toxicity of pyrolysis products (1.0 g, 40° C/min to 700° C, 4 mice in 4.2 liter exposure chamber, 30 min exposure), time to incapacitation; time to death (min)	18.5; 27.5 ± 1.86	>30; >30
Fire endurance, NASA Ames T-3 test, time to reach backface temp. of 204° C at front face heat flux of 10.4-11.9 W/cm ²	2 min 20 sec	7 min 45 sec
Volatiles at 23° C from pyrolysis in vacuum at 700° C for 5 min Mg of volatile per gram of initial sample	CO ₂ : 85.5 CO: 6.2 CH ₄ : 9.4 HCN: 3.2 C ₆ H ₆ : 4.1 NH ₃ : -- H ₂ : 1.2	155.3 14.3 1.3 5.9 -- 3.6 --
TGA, percent weight remaining at 700° C (N ₂ , 10° C/min)	51.0	73.5

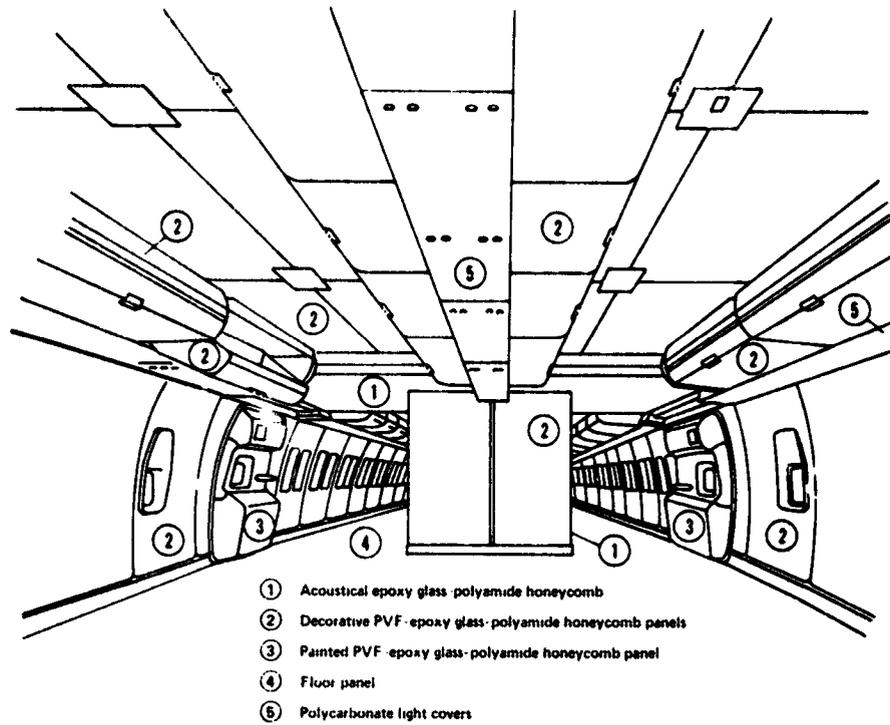


Figure 1.- Typical wide body interior materials.

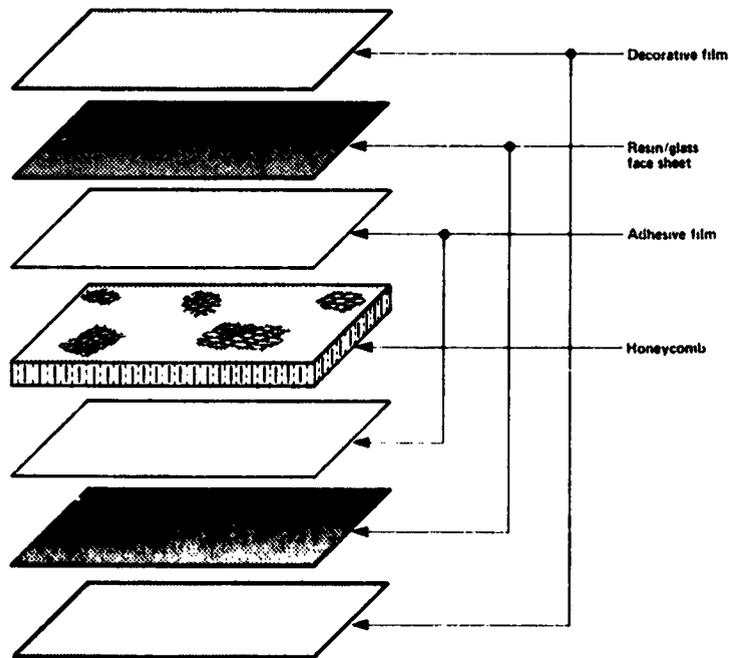
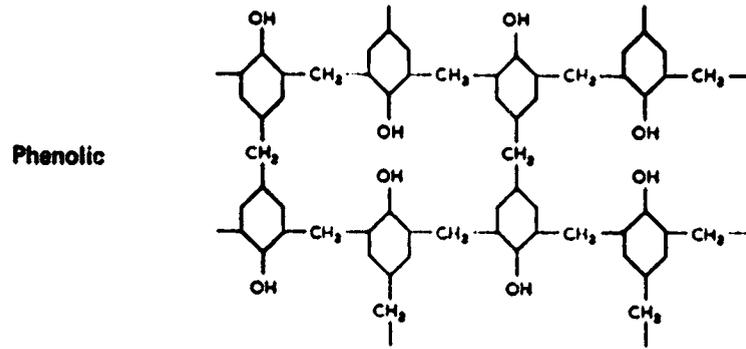


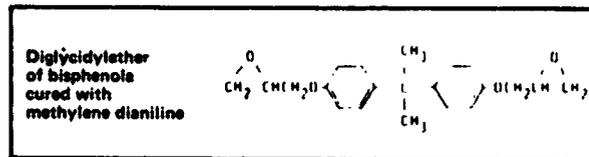
Figure 2.- Typical composite configuration of aircraft interior panels.

ORIGINAL PAGE IS
OF POOR QUALITY

Composites 1-6



Composite 8



Composites 7 and 9

Bismaleimide

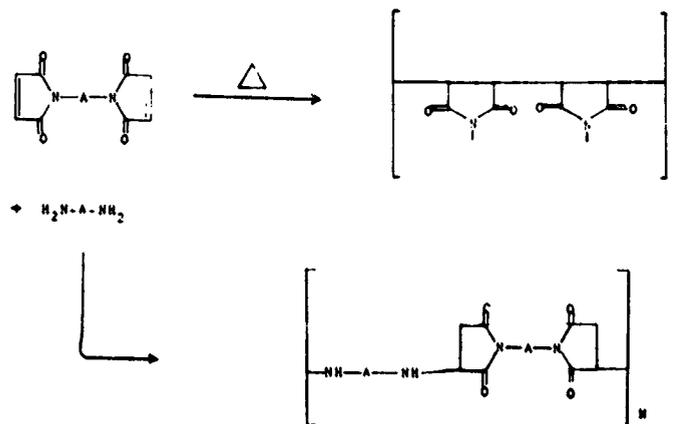


Figure 3.- Chemical structure of laminating resins.

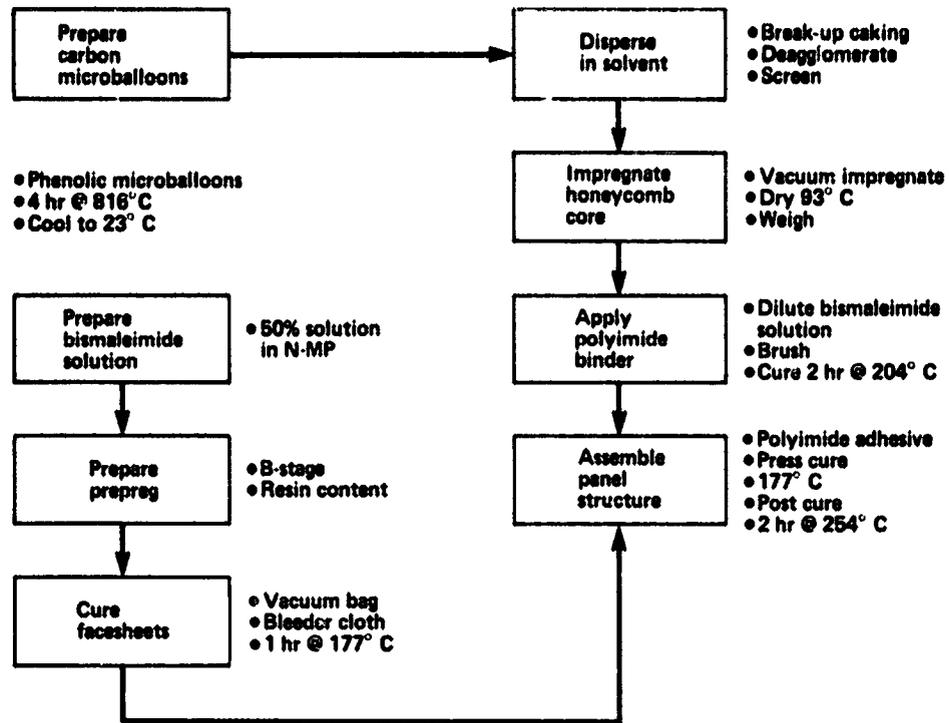


Figure 4.- Fabrication process for composite 9.

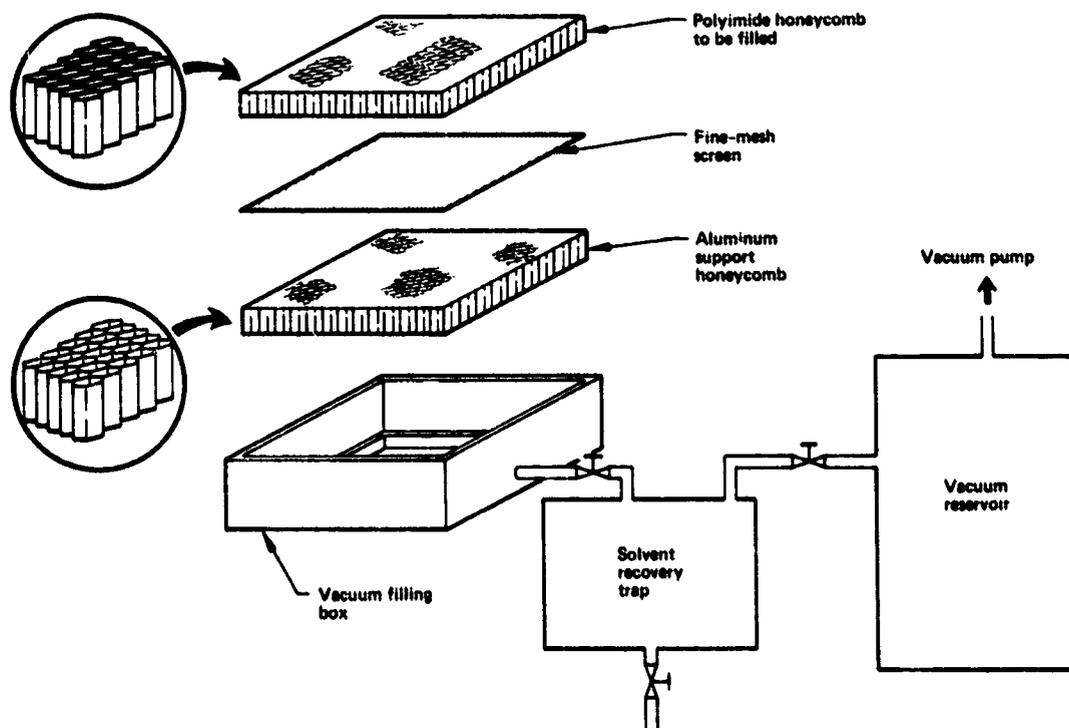


Figure 5.- Honeycomb core impregnation equipment.

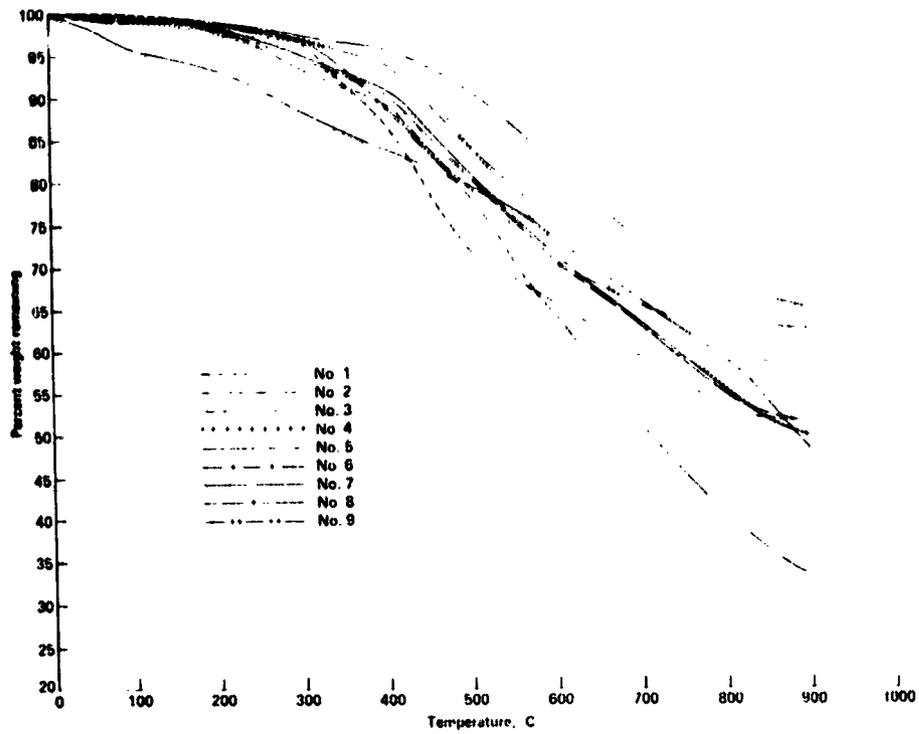


Figure 6.- Dynamic thermograph of composites 1-9 (heating rate 10⁰ C/min, N₂).

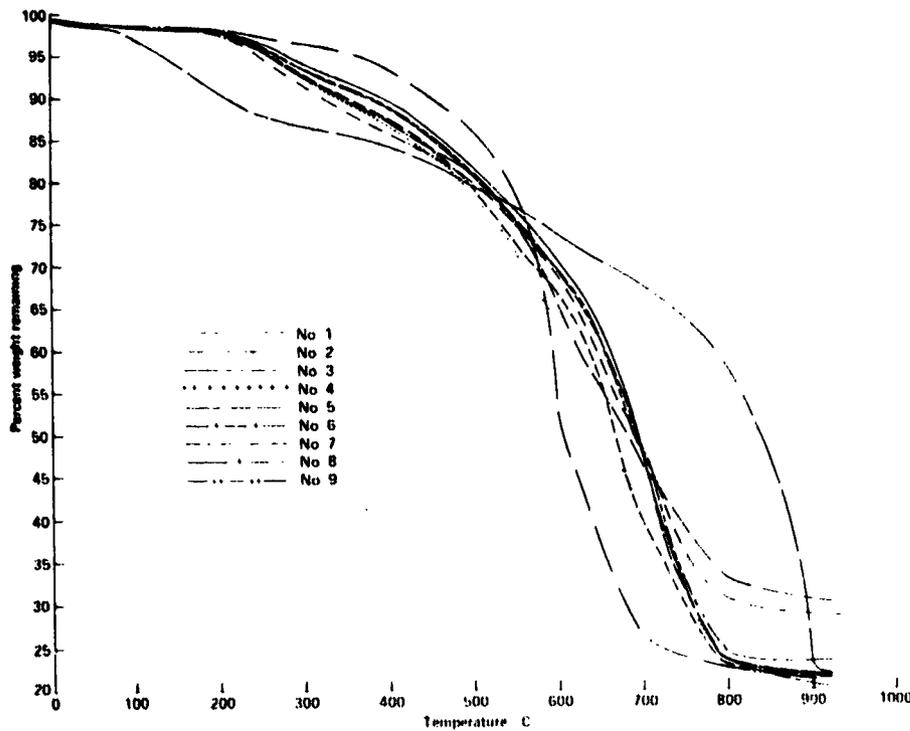


Figure 7.- Dynamic thermograph of composites 1-9 (heating rate 10⁰ C/min, air).

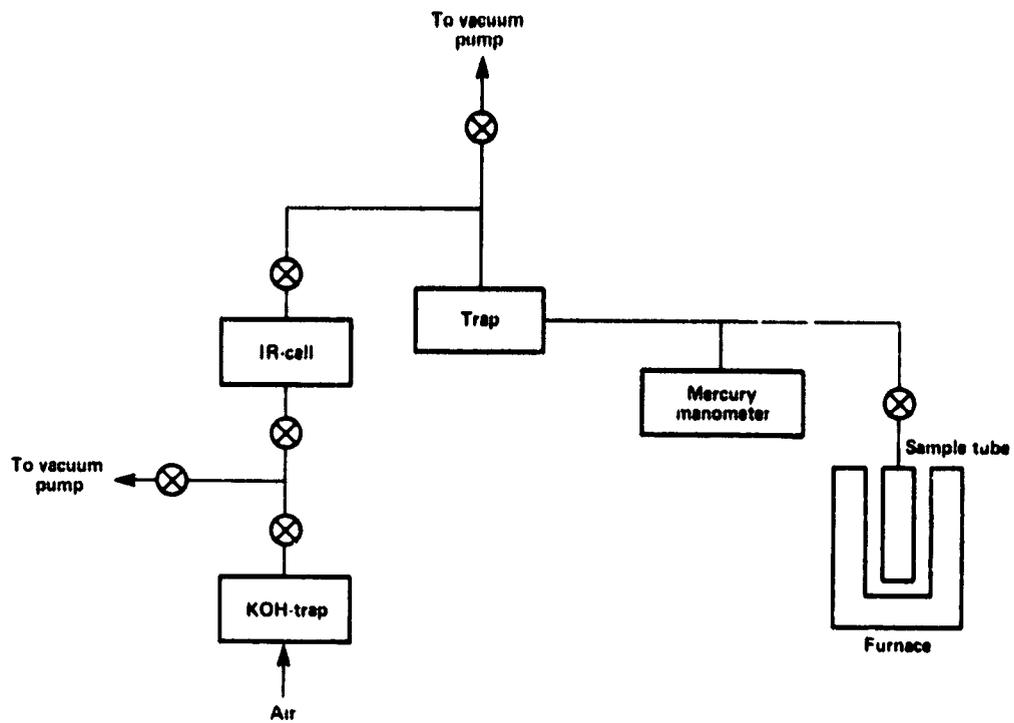


Figure 8.- Apparatus for pyrolysis of materials.

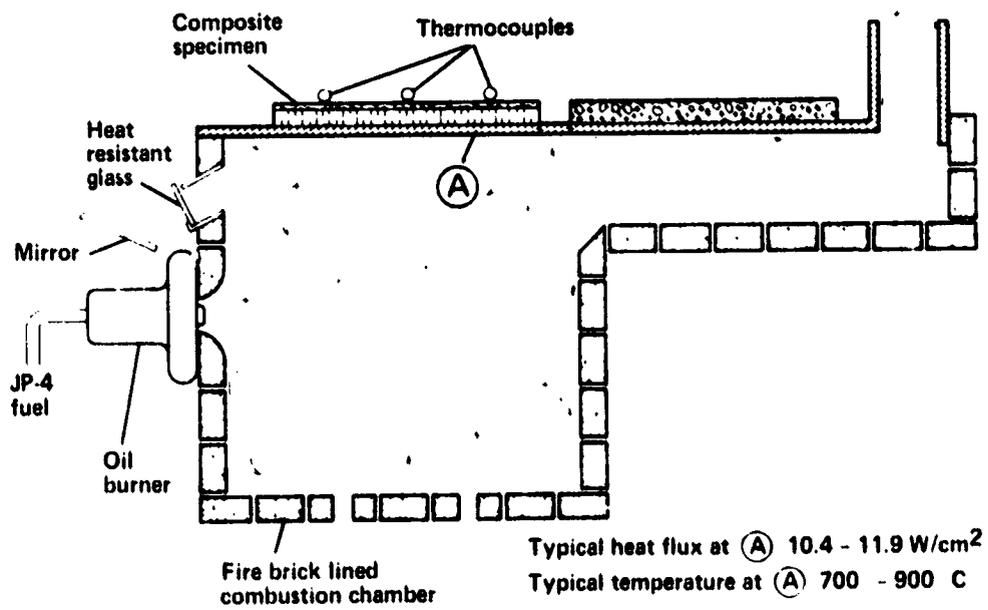


Figure 9.- NASA Ames T-3 thermal efficiency apparatus.

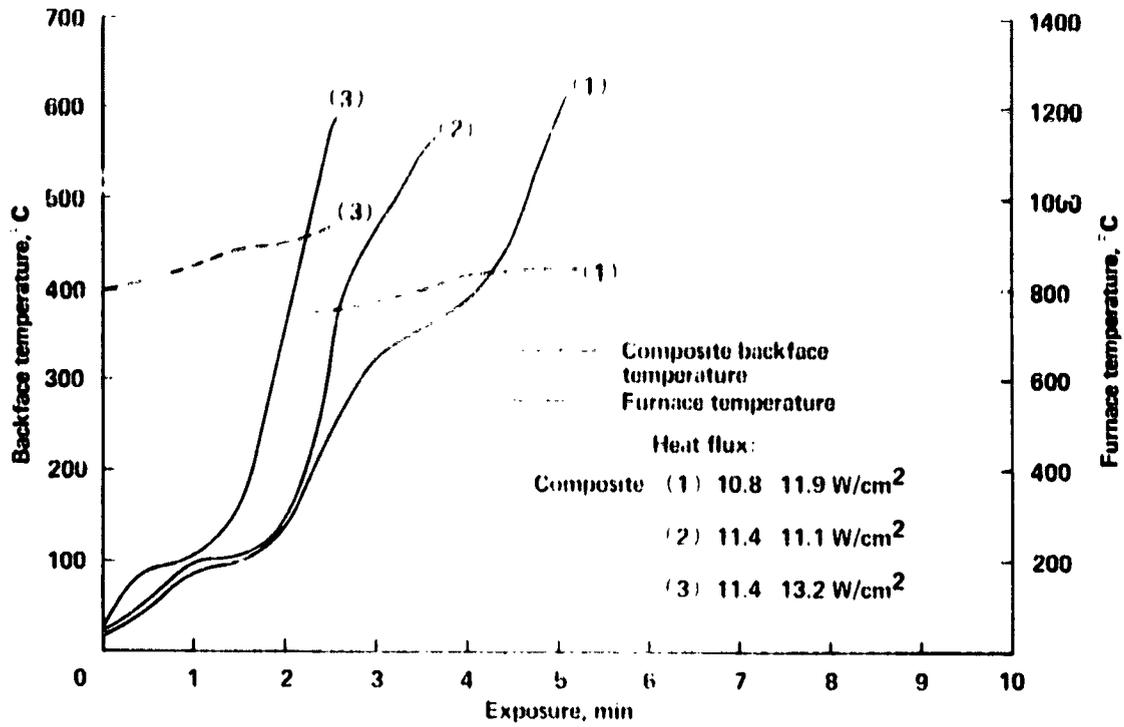


Figure 10.- Thermal efficiency of composites 1-3.

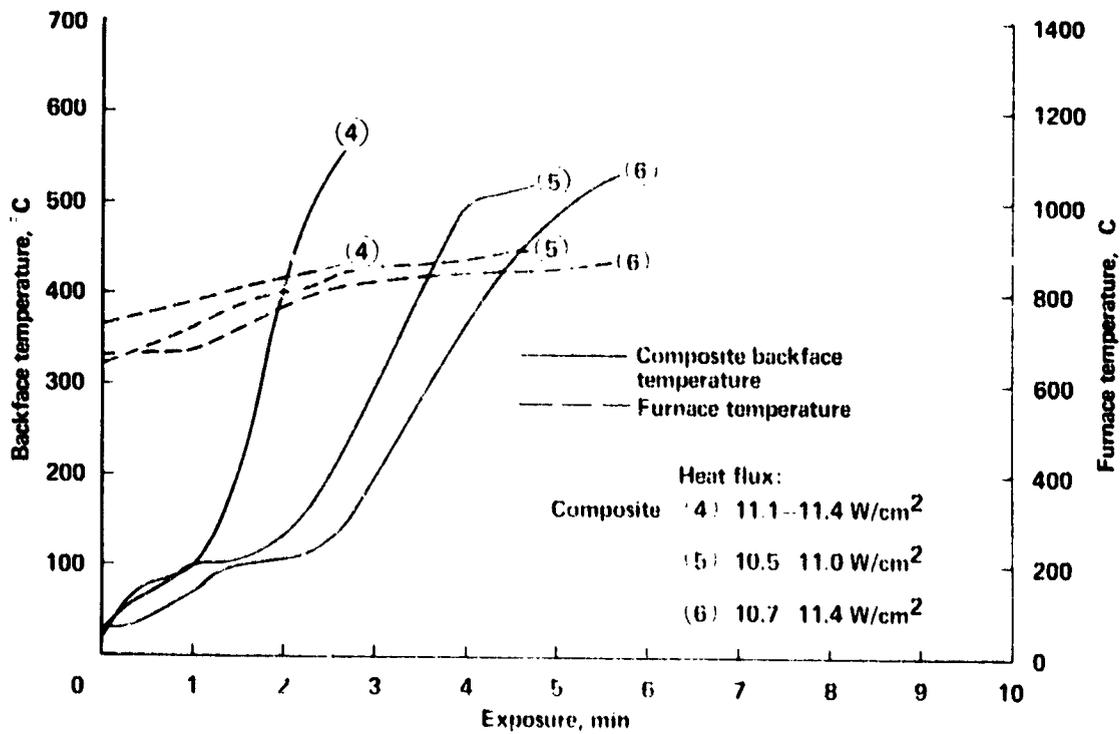


Figure 11.- Thermal efficiency of composites 4-6.

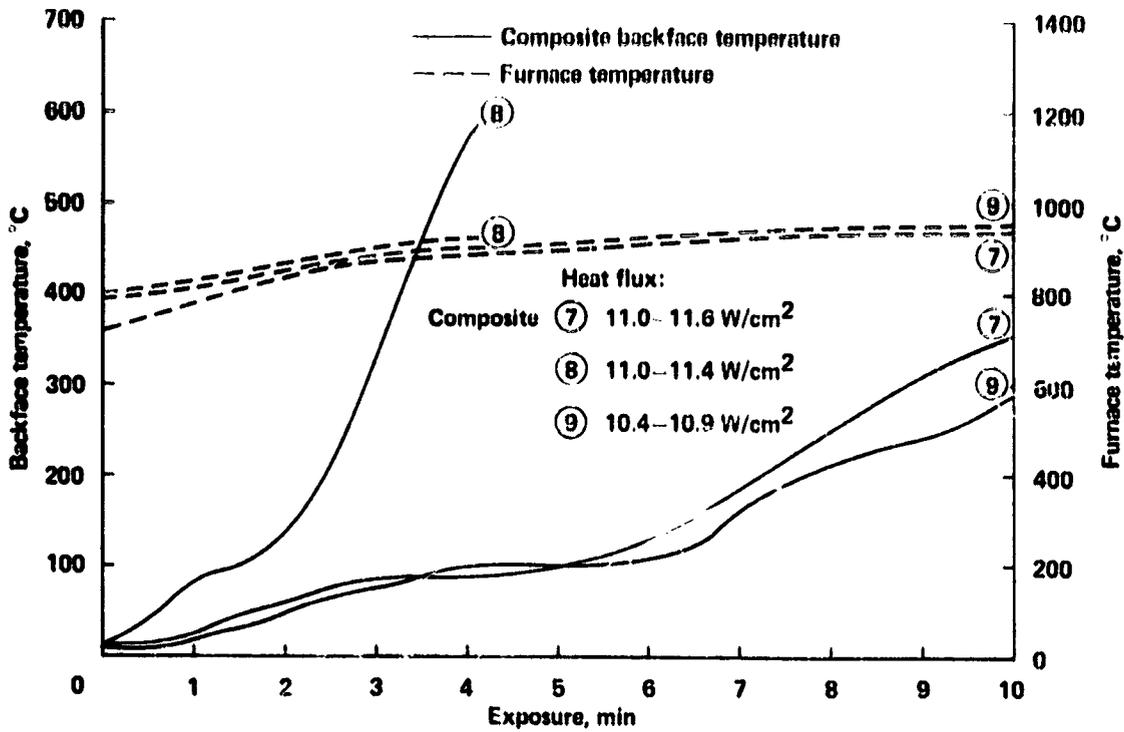


Figure 12.- Thermal efficiency of composites 7-9.

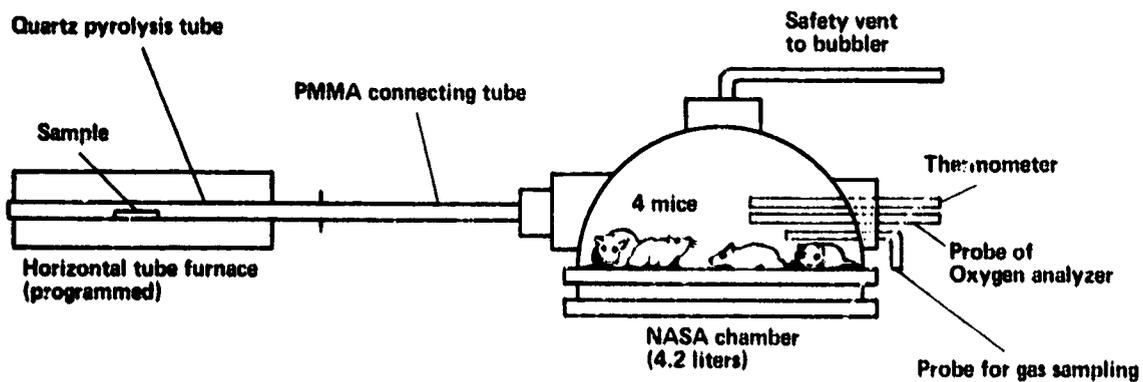


Figure 13.- Pyrolysis toxicity apparatus.

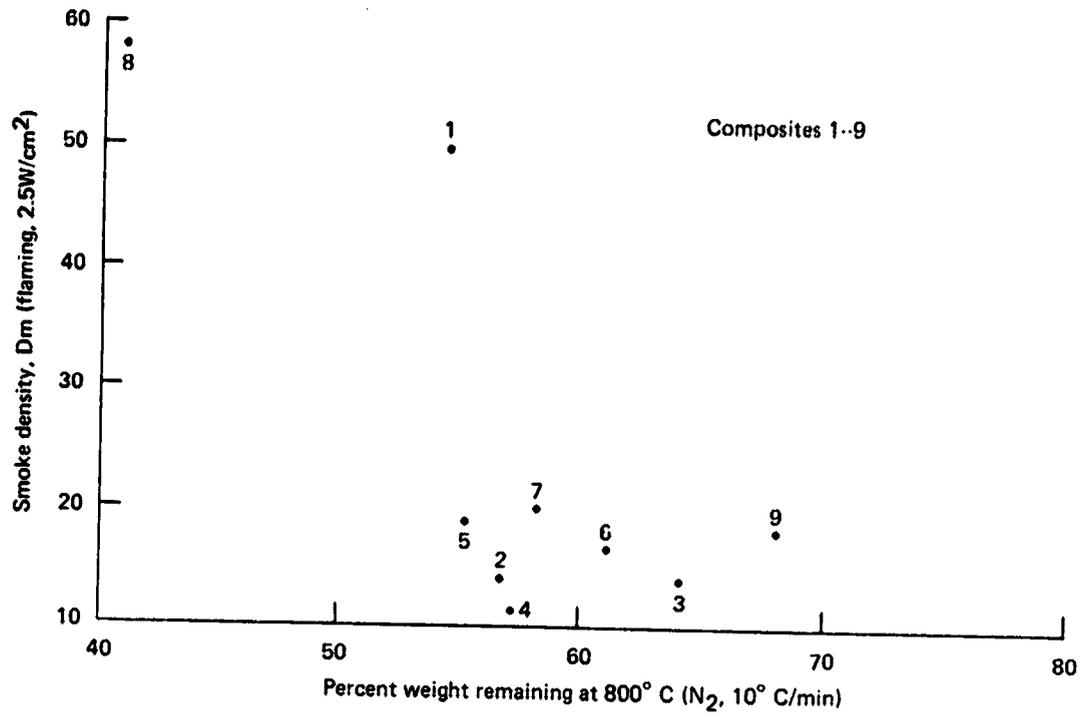


Figure 14.- Effect of char yield of composites on smoke evolution.

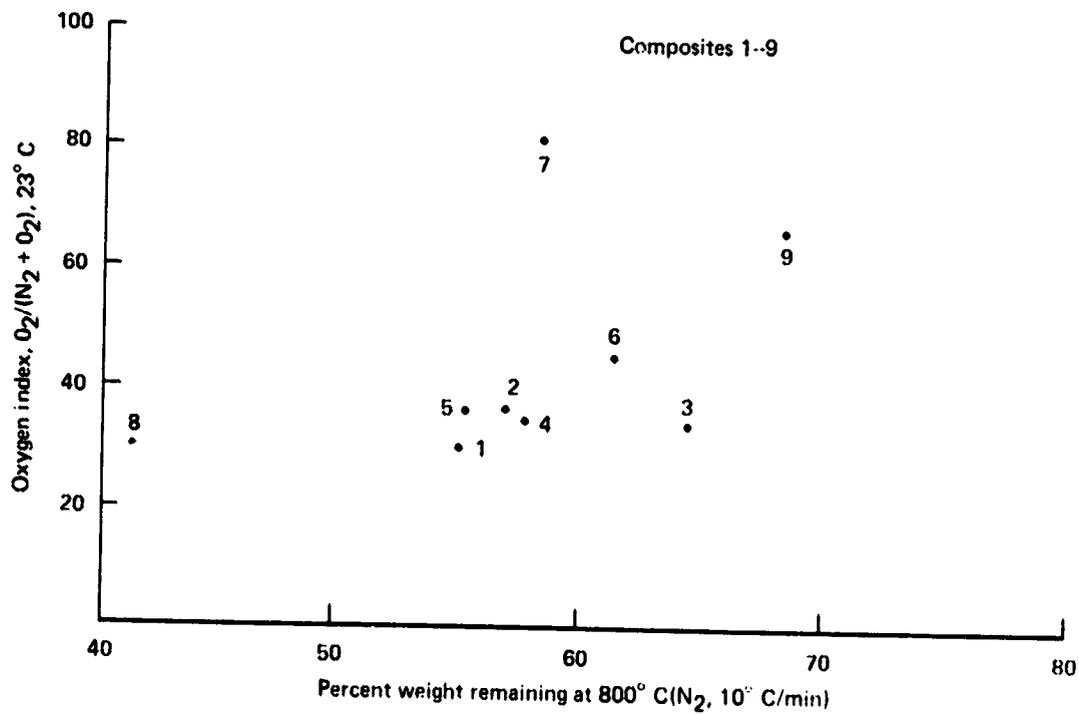


Figure 15.- Effect of char yield of composites on limiting oxygen index.