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FIRE TEST METHOD FOR GRAPHITE
FIBER REINFORCED PLASTICS

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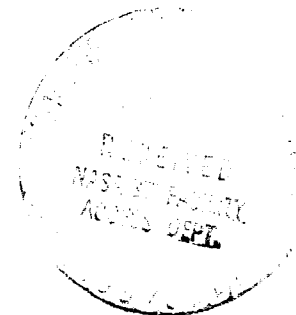
FIRE TEST METHOD FOR GRAPHITE FIBER
REINFORCED PLASTICS

by Kenneth J. Bowles

ABSTRACT

A potential problem in the use of graphite fiber reinforced resin matrix
composites is the dispersal of graphite fibers during accidental fires. Air-
borne, electrically conductive fibers originating from the burning composites
could enter and cause shorting in electrical equipment located in surrounding
areas. A test method for assessing the burning characteristics of graphite
fiber reinforced composites and the effectiveness of the composites in retain-
ing the graphite fibers has been developed. The method utilizes a modified Ohio
State University Rate of Heat Release apparatus. The equipment and the testing
procedure are described. The application of the test method to the assessment
of composite materials is illustrated for two resin matrix/graphite composite
systems.

INTRODUCTION

The relatively recent emergence of advanced technology fibers and resins has
greatly increased the potential for the use of composites in the fabrication
of primary structures in aircraft. Previously, the application of fiber rein-
forced composites has been limited to secondary, low stress structures.
Graphite reinforced composites are now being considered for highly stressed
structural members such as those found in aircraft engine fan frames and
ducts. Graphite fibers, used with the recently developed PMR polyimide
(ref. 1) represent one of the composite materials of primary interest for
these applications. The thermo-oxidative stability of both the fibers and the
resin make it possible to use these materials in those sections of aircraft
engines where temperatures can reach $500^\circ F$ (ref. 2). Their combination of
very high specific strength and stiffness can result in considerable weight
savings in aircraft engines, and thereby lead to significantly decreased
fuel consumption.

The graphite fibers used in composite fabrication possess two unique features.
They are small in diameter and of low density. These two features allow in-
dividual fibers to easily become airborne and to be carried extraordinary
distances by air currents. Graphite fibers possess excellent electrical con-
ductivity. Thus, airborne, electrically conductive graphite fibers can create
a hazardous environment for electrical and electronic circuitry. In order for
such a situation to occur, the fibers must be released from the composite mate-
rial. This can possibly occur during an accidental fire. Potential hazards
from graphite fiber release are described in detail in reference 3, 4, and 5.

The purpose of this paper is to describe a fire test method which can be utilized
to assess fiber release characteristics and fiber containment concepts. Fiber
containment concepts evaluated in the fire tests included resin structure modi-
fication and fiber containment by resin filler materials. The effects of selected

variables such as burning time, char formation and char stability (resistance to oxidation) were evaluated. Burn test requirements for the composites are described as well as test procedures and equipment. Typical results of the burn tests are included. Fiber retention characteristics are assessed primarily by the amount of free graphite fibers visually observed after a sample has been subjected to a standardized burning test.

Burn Test Requirements

The requirements for burn testing the graphite reinforced composites are as follows:

1. A controllable heat source.
2. Reproducibility of burning processes.
3. Graphite fiber dislodgement.
   a. Air stream
   b. Mechanical impact
4. Fiber and fragment collection

A controllable heat source is necessary to attain burning reproducibility. A method of monitoring the burning process during the test is necessary for confirming burning reproducibility and for assessing the fire performance of the composites being tested. The "worst condition" for an aircraft has been envisioned as a crash-fire resulting in a fuel-pool fire (ref. 6). The pool fire would envelop at least part of the aircraft. Explosions could occur subsequent to the onset of burning. A model developed for such a situation predicts maximum flame velocities of about 49 feet per second (ref. 7). In order to simulate this series of events in laboratory tests, a controlled airstream across the sample surface and a mechanical impacting device are required. These two features would tend to disturb any free fibers exposed by the burning of the samples and cause them to become airborne.

Equipment

The Ohio State Rate of Heat Release (OSU-RHR) apparatus was chosen as the testing apparatus for the graphite fiber reinforced composites burning test program at the Lewis Research Center. This equipment is shown in figures 1 and 2. The basic design of the equipment provides easy adaptability to produce the versatile research tool required by this type of materials study.

The OSU-RHR apparatus and its operation is fully described in reference 8. The air flowing through the test chamber is heated by the energy released by the burning sample. The amount of heat released by the burning sample is evidenced by an increase in temperature of the outlet gas. By utilizing a heat and mass balance of the air moving through the test chamber, and including the heat losses from the apparatus walls, the heat released by the burning sample can be measured as a function of time.

Some modifications were made to the apparatus to adapt it to the anticipated requirements of the burn tests. Provisions were made for the use of either air or nitrogen as the testing gas. The solid sample holder positioning rod was replaced by a hollow tube. An impacting rod, actuated by an air cylinder, was positioned inside the tubing. The rod was used to impact the back of the composite sample at any desired time during the test. A coarse metal screen (1/8 inch mesh) was positioned in the air exit duct to collect whole fibers which may become airborne during the burning tests. A fiberglass cloth filter over the entrance of the hood ducting was used to trap those fibers and pieces of fibers which might get through the coarse screen. Both filters could be quickly removed and replaced anytime during the test.

A tube, positioned to direct an auxiliary flow of air across the exposed surface of the sample was also installed inside of the test chamber. This air flow system was separate from the chamber air flow required for the heat release determination. The air and nitrogen flow through the burning chamber were metered through rotameter type flow meters. Temperature differences were measured with a thermopile across the air inlet and air outlet ports and the thermopile output was recorded by recorders with a variable chart speed. The radiant heat flux was measured with a radiometer at the beginning of a test and again after the test was completed.

Materials

Burn tests have been conducted using two resin/fiber composite systems. An epoxy/graphite composite (Hercules 3501-6/Hercules HTS-II) was studied because it is typical of the composite material presently being used in the aircraft industry. A polyimide/graphite composite material (PMR-15/HTS-II) was tested because it is one of the advanced technology composite materials.

At this time, the concept of utilizing composite particulate filler materials to retain graphite fibers during burning has been the only design studied to any great extent. Particulate filler materials having a relatively low melting temperature are dispersed in the resin matrix. Heat generated during burning melts the filler allowing flow and encapsulation of the fibers. This type of action would then be expected to cement the fibers together precluding their release during burning. Also, any impact fragments which may be formed would be in the form of large pieces that would unlikely become airborne. The evaluation of this concept requires visual examination of burned sample surfaces for the presence of free graphite fibers. It also requires sample weight change measurement and the demonstration that fragmentation of the burned sample produces only the type of fragments described above.

Figure 3 shows a plot of the results of thermogravimetric analysis of some carbonaceous materials from reference 9. The thermal resistance of two types of fibers (AS fiber and GY-70 fiber) and an epoxy resin are shown in this figure. The difference between the thermal resistances of the two types of fibers can be explained by evidence that the AS fiber is less graphitic than the GY-70 fiber. This figure indicates that fire testing in air at temperatures greater than 1427°F would result in the rapid oxidation of the fibers

along with the matrix. This situation is not desirable since we are looking for the "worst case" conditions where the resin material would be burned completely but the graphite fibers would remain either as free fibers or trapped in the resin/filler char. It is not to be inferred that the fiber material would not degrade. The rate of degradation would be slow in comparison to the degradation of the resin, however. Based on these considerations, the burn tests were conducted at temperatures below 1427°F. Test conditions which provide the desired temperature level for burn tests of graphite fiber reinforced composites were determined to be a radiative heat flux of 5.3 Btu/ft^2-sec, with an air flow of 21 cu. ft/min. through the test chamber.

Test Procedure

The air flow through the burn chamber was adjusted to a value of 21 cu. ft/min. through the airline flowmeter. This volumetric flow rate corresponds to a linear flow velocity of 4.2 inches per second over the test sample in the chamber. Figure 4, from reference 6 lists six categories of fibers that have been found after burning and impacting tests. Also listed are the settling rates for these six different fiber categories. Based on these calculated numbers, the air flow rate through the chamber would cause only the single fibers to be carried out of the chamber and to the filtering system. All other fibers which would be in the form of bundles or clumps would fall to the bottom of the burn chamber. The 21 cu. ft/min. air (or nitrogen) flow rate was chosen because it was found to have no effect on RHR measurements (ref. 9). However, it is worth noting that the flow rate can be utilized to separate the single fibers from heavier debris when specimen impacting within the chamber is required.

The gas pilot burner was adjusted to burn 2 cu.ft. per hour of natural gas. The gas pilot served to ignite the volatiles from the sample as they were released.

The silicon carbide heater current was then adjusted until a heat flux of 5.3 Btu/ft^2-sec. was attained. Under these conditions, the rate of composite degradation was low enough to allow sufficient time to observe and document the degradation process.

The composite laminates to be burned were cut into 3 in. by 6 in. samples and weighed. The actual resin content of each composite was determined gravimetrically (ref. 10). The thickness of each sample was also measured and recorded.

Each sample was placed into the sample holder of the OSU-RHR apparatus and inserted into the burn chamber. The temperature difference between the chamber inlet air and the outlet air was recorded as the sample burned. All samples remained in the OSU-RHR apparatus for at least five minutes. This was sufficient time to allow sample flaming to cease naturally. The sample was then removed from the apparatus. The filters were removed and visually examined for trapped graphite fibers. The burned composite was weighed, and the exposed surface examined visually for bare graphite fibers. In some instances,

new filters were installed and the sample was reinserted for another period of time. Generally, the sample was reinserted to continue the degradation so as to obtain weight loss data as a function of time. During the second insertion, the auxiliary air flow device or the impacting device could be operated.

Some of the tests were conducted with nitrogen flowing through the test chamber to obtain anaerobic char data.

DISCUSSION

Heat Release

Heat release data and total heat release data provide a history of the burning event to confirm test reproducibility. Also, the data provide a means for comparing the burning processes of different composite materials. Figure 5 shows heat release rate recordings for two types of composite materials included in this study. Figure 5(a) shows the heat release rate curve for a standard bill of material epoxy/graphite composite. Figure 5(b) shows the heat release rate history of graphite/PMR, a polyimide/graphite composite laminate.

Figure 6 shows heat release curves for two panels of graphite/epoxy composite material. Figure 6(a) is that for the standard epoxy/graphite panel and figure 6(b) is the heat release rate curve for similar panel filled with about 10% boron powder. From the appearances of the two curves, there is no significant difference in the two burning processes. These curves were recorded for a burn time of five minutes during exposure to a radiant flux of 5.3 Btu/ft^2-sec. Figure 7 shows the surfaces of the two panels. The surface of the panel without the boron filler consists of a mat of loose graphite fiber. The other panel, with the boron filler, exhibits a smooth, shiny surface with no observable loose fibers. While the boron powder does promote retention of the graphite fibers within the solid combustion products of the resin, it does not appear to affect the heat release characteristics during the initial flaming of the sample.

Resin Weight Loss

All resin weight losses from burning were calculated based on the sample weight before burning, the post-test weight, and the as-fabricated resin content of the sample. At least two samples of each type of composite were tested. One was decomposed anaerobically in nitrogen in the OSU-RHR apparatus. The anaerobic testing was performed to assess the results of composite modification in producing a maximum amount of char. It was reasoned that the anaerobic decomposition tests would produce the maximum amount of char for each resin tested. Figure 8 shows weight loss data for the epoxy and polyimide matrix composites for decomposition and burn times up to 35 minutes. For these tests in air, the actual sample flaming time was completed after five minutes of testing. It can be seen in figure 8 that the amount of char available for containing the graphite fibers in the polyimide composites is significantly greater than the amount of char in the epoxy composites after five minutes of burning in air. However, if burning is allowed to continue, the char residue from both

resins disappears. In figure 8, the polyimide produces significantly more anaerobic char than the epoxy resin. Based on these data, neither short time burn tests in air nor anaerobic decomposition tests can be used by themselves as a simplified method for predicting the fiber retention effectiveness of composite systems in air. Figure 9 shows resin weight loss data for three polyimide/graphite composites. One contains a boron powder filler and was burned in air. The other two contained no boron filler. One of these was burned in air and one was decomposed in nitrogen. From the results shown in figure 9, it appears that the boron powder causes the composite to burn anaerobically, possibly by oxidizing, then melting to form a molten B_2O_3 coating which protects the resin char. This type of residue appears to be very effective in retaining graphite fibers as illustrated in figure 7.

Fiber Release

Burn tests with 21 cu. ft. of air flowing through the burning chamber resulted in the collection of insignificant amounts of graphite fibers in the filtering system, even when the sample surface was covered with a heavy layer of loose fibers. Apparently, the disturbance caused by the air flow was not enough to pull individual fibers from the entangled mass of surface fibers. A number of variables have been found to strongly influence fiber release even when the sample was subjected to mechanical impacting and exposur to varying air flows over the surface of the burned sample. These variables and fiber release mechanisms are described in references 11 and 12.

The criterion established in this study for assessing the fiber retention effectiveness was based on the amount of free fibers exposed on the surface of the specimen at the conclusion of the tests. On this basis, only the boron filled composite was effective in retaining the graphite fibers as illustrated in figure 7.

CONCLUDING REMARKS

The burn testing procedures developed for graphite composites provided a semi-quantitative means for assessing burning characteristics and fiber retention effectiveness. These testing procedures also effectively monitored the burning processes and contributed significantly to the understanding of the methods by which fiber containment could be accomplished. The test data were found to be reproducible.

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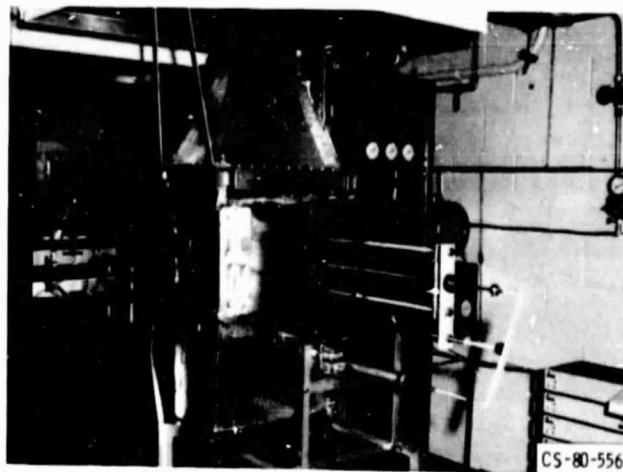   Reinforced Plastics," M.S. Thesis, Ohio State University, 1979.
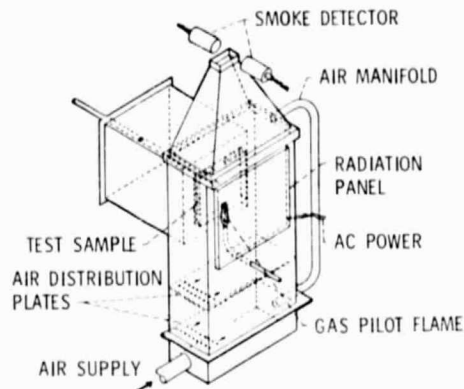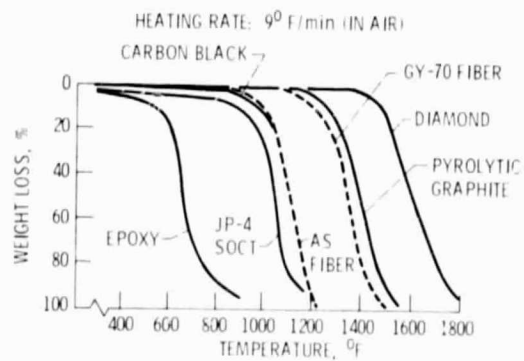
Figure 1. - OSU-RHR apparatus.

Figure 2. - OSU-RHR apparatus.

Figure 3. - Thermogravimetric analysis of carbonaceous materials.