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### FIBER REINFORCED COMPOSITES FOR INSULATION AND STRUCTURES

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#### **Introduction**

The work involves two areas:

Composites – optimum fiber placement with initial construction of a pressure vessel, and The general subject of insulation, a continual concern in harsh thermal environments.

### **Insulation**

During a first meeting about insulating materials, a question was asked about fibers for a resilient insulating blanket. As we (at Auburn) have more than 10 years experience in the development of high temperature, resilient, insulating batting (Figure 1), I prepared and presented a lecture on fibrous insulation and the technology of its production. I also had some crimped carbonaceous fiber at Auburn University, and provided a quantity of this fiber for use by NASA. That collaboration is continuing.

Since the temperature requirements for materials used by NASA are often substantially more severe than the testing previously done on the carbonaceous fiber batting (Figure 1), a test was performed on this batting in a plasma torch located at MSFC. That test burned through the sample in about 4 seconds (1000 seconds is considered acceptable for the more severe applications). Even a carbon-carbon composite does not survive the plasma torch test unless it is coated with a temperature/oxidation resistant coating.

Figure 1. Demonstration of temperature/flame resistant carbonaceous batting –

Dr. David Hall, Professor Emeritus, Textile Engineering, Auburn University.



A temperature/oxidation resistant material, suitable for use as an adhesive and coating in the carbonaceous batting was sought. The primary material selected was an alumina/silicate based binder/coating (Pyropaint 634 AL-L from Aremco Products Inc). A phenol-formaldehyde resol used in the fabrication of carbon/carbon composites was also obtained for trial as an adhesive/coating,

The alumina paint is a two component mixture – Part A is reported by the manufacturer to be a low viscosity suspension of colloidal silica (sol) in water. Part B is an alumina powder. Mixing instructions suggest three parts powder to one part silica suspension in

water. According to the manufacturer, a small amount of the liquid can be added as a thinner if needed. The major concern with thinning (having too little of the powder) is the tendency of the silica binder to shrink and crack as it dries. When applying to a porous material like a batting, there is also a concern that the liquid will be pulled into the porous batting leaving a pigment rich (alumina) coating on the surface that will flake off easily. The manufacturer has experience with application of their refractory paint to porous ceramics and metals, but does not seem to have experience using the materials as a binder in nonwoven fibrous felts where the paint can penetrate very well and the pigment may be filtered out to various degrees with the penetration.

Some initial trials were made to see how the coating would perform. First a moderate density carbonaceous batting was simply painted with the paint, mixed to the manufacturers specifications. Subsequently attempts were made to saturate the batting with the paint thinned with water. This approach allows sufficient liquid to saturate the batting without loading it with excessive binder or pigment, and also without changing the binder/pigment ratio. As the battings were allowed to dry, some of the binder and pigment migrated under the influence of gravity to the bottom of the samples. This appeared to give a coating more on the individual fibers rather than producing a solid painted surface on the batting. The samples were allowed to dry at room conditions for 25 hours before putting them in an oven at ~300 F for ~ 1 hour. The weight add-on of the samples was measured and is shown in Table 1. Further experimentation will be necessary to select the best process and conditions for optimum performance. Samples of the coated and the saturated felt were tested in the plasma torch to see what if any improvement in performance is observed.

Samples of the carbonaceous felt were saturated with a phenol formaldehyde resol dissolved in methanol. The resol was diluted with methanol to allow an easy distribution of the fluid in the felt. The methanol readily soaks into the felt which essentially behaves as a sponge for the solution, and could be wrung out just like a sponge or wash cloth soaked in liquid. The amount of liquid added was limited therefore and the felt simply dipped in the solution on one side. All the liquid was readily absorbed into the felt. Subsequently the felt was inverted and squeezed to distribute the liquid binder. The solids content of the resol solution was not known and initially was not measured. The weight gain of the sample was measured and results shown in Table 1 – samples 1-5.

Samples 6 - 10 consist of felt treated with an alumina/silicate binder. Samples 2 and 3 (saturated with phenolic resin) were subsequently treated by painting the alumina/silicate on the surface. Samples 2 and 10 were tested in the plasma torch.

Both samples failed at about 100 seconds, more than an order of magnitude better than the untreated sample, but still well short of the desired time to failure. The sample bonded with phenolic and subsequently painted with alumina/silicate on the surface seemed to perform better over the longer time period, indicating the desirability of an impervious high temperature coating over the whole surface. Some additional tests will be run to try to improve the thermal resistance.

Sample number	Resin/Coating	Resin on weight of carbon fiber (%)	~ Density (lb/ft <sup>3</sup> )	Comments
1	Phenolic	21	6.3	
2	Phenolic	35	7.0	Coated*
3	Phenolic	37	7.1	Coated*
4	Phenolic	75	8.6	
5	Phenolic	165	13.8	Squeezed out excess
6	Alumina/silicate	147	12.8	Painted on surface
7	Alumina/silicate	145	12.7	Saturated
8	Alumina/silicate	98	10.3	Saturated
9	Alumina/silicate	218	16.6	Saturated + painted
10	Alumina/silicate	132	19.0	Saturated

 Table 1: Glue/resin added to carbonaceous batting

\* Subsequently coated with alumina-silica composition

#### Fiber Placement and Composite Tank

We continued a project begun by Dr. David Beale, to cover an aluminum tank liner/pressure vessel with braided Kevlar. Dr. Beale had suggested a braid angle approaching 50degrees; however, it became obvious that the braided structure would not lie close to the tank surface in the neck region if that braid angle was maintained.

The initial work undertaken was to learn how to set the machine controls. The two primary controls are the mandrel speed and the rotational speed of the yarn carriers. The results of this effort are shown in Figures 2 and 3.

A simple geometric model of the braided structure was developed assuming a circular yarn cross section and a 65 % packing factor for fibers in the yarn bundles. Using 18 axial yarns of 1000 denier Kevlar and 18 yarns spiraling in each direction, a model was developed for the size of the braid as a function of braid angle (Figure 4). This model combined with the graphs below allowed the selection of the machine settings, and the

settings appeared to be approximately correct – until we began to vary the yarn tensions on the machine. At that point, the assumptions about yarn shape became obviously untrue (Figure 5).

# Figure 2. Machine setting vs rotational speed

Figure 3 Machine setting vs mandrel speed



#### Figure 4: Braid diameter as a function of braid angle – with minimum yarn spacing



Figure 5. Yarn crossing illustrating flattening of the lower tensioned yarn



At this point we discarded the model and adjusted the machine by intuition about the desired result and the adjustments that would achieve it. The most satisfactory result was produced by using 18 axial yarns until the tank neck begins to flare outward, then another 18 axials were added. The axial yarn tensions were significantly higher than the spiral varns. A nonwoven Kevlar felt was placed under the braid and an epoxy wetted carbon fiber layer was placed over the braid (see Figures 6 and 7). The tanks will be tested by overpressure until failure and with a rifle shot while under pressure. Results from these tests are not yet available.

## the braiding machine







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