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UNIVERSITY HONORS PROGRAM

SENIOR PROJECT - APPROVAL

Name: Stephen RussCollege: Engineering Department: MSEFaculty Mentor: Matt FerberPROJECT TITLE: Development of a high
temperature stress rupture testing apparatus
for small diameter ceramic fibers.

I have reviewed this completed senior honors thesis with this student and certify that it is a project commensurate with honors level undergraduate research in this field.

Signed: Matt Ferber, Faculty MentorDate: 5/13/97

Comments (Optional):

Outstanding work!!

Introduction

The efficiency of many thermal processes is directly related to the operating temperatures of the system, which in turn is limited by the materials used in a given design. A great deal of research and development had gone towards creating materials that push the envelope of performance at high temperatures. Of particular concern are mechanical properties at high temperatures over an extended time span. Ceramics have a service temperature limit a full 200-300 °C above the highest performing metallic superalloys, and also have a higher hardness, strength, elastic modulus, and a lower coefficient of thermal expansion. The greatest drawback of ceramic materials is their low fracture toughness or tolerance for crack-like defects. Therefore, a critical need exists to increase the fracture toughness of ceramic materials.

One of the major approaches undertaken by the government, academia, and industry, to increase the fracture toughness of ceramics is via the use of ceramic matrix composites. Composites are by definition materials that have a chemically and/or physically distinct phase distributed within a continuous phase. Ceramic composites consist of a ceramic matrix (the continuous phase) and a reinforcement consisting of particles, whiskers, short fibers, or continuous fibers. One major program undertaken through the United States Department of Energy, has brought together DOE labs, universities, and industry to undertake the development and characterization of viable continuous fiber ceramic composites (the CFCC program). In a CFCC material, cracks that begin to propagate are impeded when they run against a reinforcing fiber. The fracture toughness of the material is subsequently increased as energy is absorbed through a variety of mechanisms including fiber/matrix debonding, crack deflection, and fiber pullout.

These fracture-toughening mechanisms are sensitive to the properties of the fiber reinforcement. If the fiber is not strong, then very little energy is absorbed in one of the above-mentioned mechanisms before the fiber simply fails and the crack continues. The fiber must also be flexible enough to be processed in a weave. Extremely small diameter fibers fit these two needs, as their small cross-section and high aspect ratio allow them to have a higher strength than the bulk material of the same composition as well as a high degree of flexibility.

A common reinforcement used in CFCC materials is a β -SiC fiber manufactured in Japan under the tradename Nicalon. Nicalon has an average diameter between 12-20 μ m, and has a circular cross-section which allows it to be easily mathematically modeled in relation to a irregularly shaped fiber. The major drawback of Nicalon is its probable thermodynamic instability of composition and microstructure above 600 °C. Not much

data exists on the properties of individual Nicalon fibers above room temperature, and that which does has mainly been extrapolated from tests conducted upon large bundles of the fiber. Tests on single fibers that are as small as Nicalon are extremely difficult to conduct due to problems such as gripping the fiber. Due to the popularity of Nicalon, and to better understand its basic properties for modeling purposes, the design of an apparatus to conduct stress rupture tests was undertaken.

Discussion

A variety of requirements were considered to be essential to the testing apparatus. They are summarized as follows:

- Ability to conduct test up to 1000 °C.
- Automated data acquisition of some sort to record the time to failure of each fiber.
- Variable gauge lengths that are subjected to a uniform temperature.
- Load applied in such a manner that the fiber is in 'pure' tension.
- A method of gripping the fiber that does not damage it.
- Capability of applying a wide range of loads to the fiber.
- Handling process accomplished in such a way to minimize fiber damage
- Ability to measure the diameter of each fiber to accurately determine a stress state.
- Capacity to run a large number of tests concurrently, and to load new tests without effecting the other tests running.
- Cost

Previous attempts at single fiber tensile testing had failed due to problems associated with gripping the fiber while not damaging it, and applying a uniform tensile load. After several designs were contemplated and tested, the following method for fiber testing was contrived:

1. A single fiber, roughly 12 inches in length, is removed from a larger bundle by attaching a small piece of transparent tape to one end.
2. The fiber is placed perpendicularly on a 6 inch wide Teflon block placed flush on the end of a table. The tape attaches one end of the fiber to the side of the Teflon away from the edge of the table, and the rest of the fiber is allowed to hang over the edge of the block/table.
3. Another piece of transparent tape is used to attach a small paper clip to the free end of the fiber, essentially placing it in a "pre-loaded" condition.

4. The fiber is now stretched across the Teflon block, and two single small drops of a high temperature quartz adhesive are placed at the required gauge distance apart. Typically the drops of adhesive are on the order of 0.5 cm in diameter.
5. A larger Sigma fibers ($>100\text{ }\mu\text{m}$ in diameter) is then placed into the end of a drop of adhesive, in line with the Nicalon fiber. This is repeated with another Sigma fiber for the other drop of adhesive.
6. The adhesive is allowed to dry at room temperature for 6 to 24 hours, followed by a cure at $120\text{ }^{\circ}\text{C}$ in a furnace for 6-12 hours. The curing process is essential, as otherwise retained water boils off during the test, and under stress loaded condition this cracks the adhesive and the fiber pulls out.
7. The Nicalon fibers protruding from each end of the adhesive, next to the Sigma fibers, are cut using a sharp razor blade. After this step what is left, in linear order, is a Sigma fiber, a small drop of adhesive, the gauge length of Nicalon, another small drop of adhesive, and the other Sigma fiber.
8. One of the Sigma fibers is glued into a $2\frac{1}{4}$ inch long hollow alumina tube, with a thumbtack glued into the opposite end, using the same high temperature quartz adhesive.

This process leaves you with a load train that is schematically illustrated in Figure 1. The furnace was then planned around the use of this loading scheme.

In order to maximize uniformity of heating within the furnace, a circular furnace design was considered. Other considerations included the need to run off of 120 Volts AC instead of 240, and enough inside volume to test a large number of fibers simultaneously, and expense. A furnace from the Watlow Corporation was found to best meet the requirements, with a $2.0\text{W}/\text{cm}^2$ power density supplied through a series of looped, nested coils inside of an alumina matrix. The service temperature extends to $1100\text{ }^{\circ}\text{C}$, which is high enough for any foreseeable testing. A full cylinder two-lead furnace 12" high, 12" OD, with an 8" ID was ordered.

A top and bottom for the furnace were machined from plate stock alumina with a lip to fit into the furnace. A hexagonal array of 42 holes, spaced one inch apart were drilled into the top and bottom to allow specimens to be lowered into the furnace, and to be removed again if necessary. A hexagonal array was used as it created the highest packing density possible with the constraint that the holes must be 1 inch apart. The center hole is for the controller thermocouple, and one of the rim holes for the overtemp controller, leaving forty available test sites. The entire furnace was placed on a steel rim, with legs

12” long to allow the weights to hang free underneath. Placing the furnace high also allowed one end of the Nicalon fiber to fall clear of the furnace with the weight upon failure, meaning that it does not sit inside and continue to oxidize after failure.

To control the furnace, a 965 series Watlow 1/16 DIN microprocessor-based auto-tuning controller and an SCR were purchased. The controller featured auto-tuning for heat outputs, ramp to set point for gradual warm up of the thermal system, and automatic/manual capability with bumpless transfer. All information is stored in a non-volatile memory, and the controller accepts J,K,T,N, or S thermocouples in a single input channel. A Eurotherm Model 92 alarm unit was purchased after some concern about the possibility of overheating due to thermocouple failure, and placed in series as an overtemp controller.

The most common way to apply loads in a stress rupture test is via a hanging “dead” weight. For a material with a marked change in cross-sectional area upon loading, or high degree of deformation, this method may not be appropriate due to the fact that the stress condition is unknown or changing during the test. Due to Nicalon’s low strain to failure (around 1.5%), and the fact that testing temperatures are below $\frac{1}{2} T_m$, so creep is not a major concern, the free weight method is viable. Weights needed to be made from a material readily available that offered a high density due to limited space and a melting point that did not put the weight at risk if it was placed in the vicinity of a 1000 ° environment. Lead offered an exceptionally high density, but was rejected due to its low melting temperature of 327.5 °C and the hassle involved in its use since it is classified as a toxic material. Copper was chosen as suitable alternative with the relatively high density of 8.96 g/cm³, and its 1083.4 °C T_m .

The weights were machined with a conical section of material removed from the top, to guide a Sigma fiber towards a drilled hole (Figure 2). When attaching the weight to the bottom of the load train, the conical area is filled with a 5-minute epoxy and the weight is raised using a lab jack until the Sigma fiber contacts the adhesive. After the epoxy has cured, the load of the weight is transferred smoothly to the fiber by slowly lowering the lab jack. The use of the lab jack is critical after it was found that abrupt application of the load caused the Nicalon fibers to break.

The weights were cut into 9 different lengths to obtain different loads ranging from 5 to 50 grams. The weights were “fine-tuned” to various specific and individually distinct

masses by removing different amounts of material via a small grinder. Each weight was then assigned a number and demarcated with a permanent marker. A load cell was placed underneath the furnace, with an aluminum plate on top of it to catch weights as the fibers failed. A data acquisition system utilizing Labview® software is used to collect load versus time data. These data may then be analyzed to determine which weight fell onto the plate at which time, as the load cell is sensitive enough ($\pm$) to differentiate each individual weight from another. After a fiber has failed, it is taken to a scanning electron microscope to measure its exact diameter, in order to establish the stress it was under at the fracture point. This information, along with the time to failure, is used to generate a stress rupture plot. An overall schematic of the stress rupture testing apparatus may be found in Figure 3.

Further Work

Initial testing has been successful. Surprisingly, all but the tests conducted in the 5-10 gram range fail almost immediately, and only one test has lasted for more than 24 hours. For this reason, more of the lighter weights are being machined. The greatest concern at the moment is to conduct enough tests to create a viable stress rupture plot for 700 °C. Further testing will then be done at other temperatures. Another variable which it may prove interesting to control is the composition of the atmosphere that the fibers are placed in. Designs have been created for a Plexiglas case to surround the furnace, in which an inert gas could be kept at a slightly positive pressure.

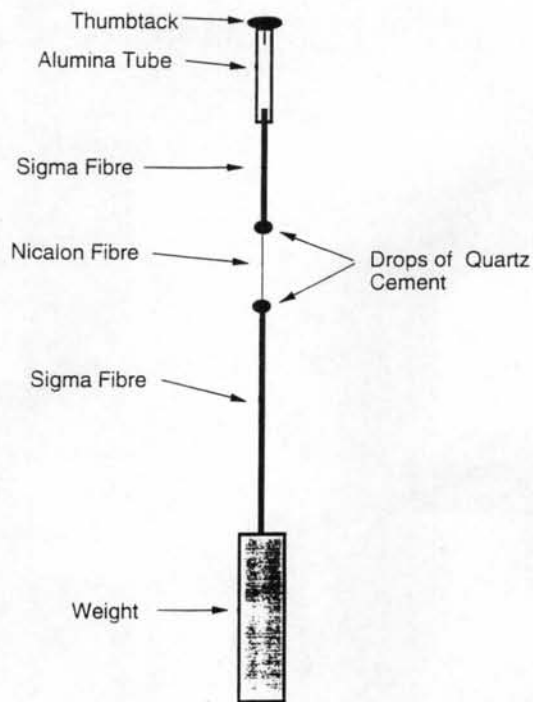
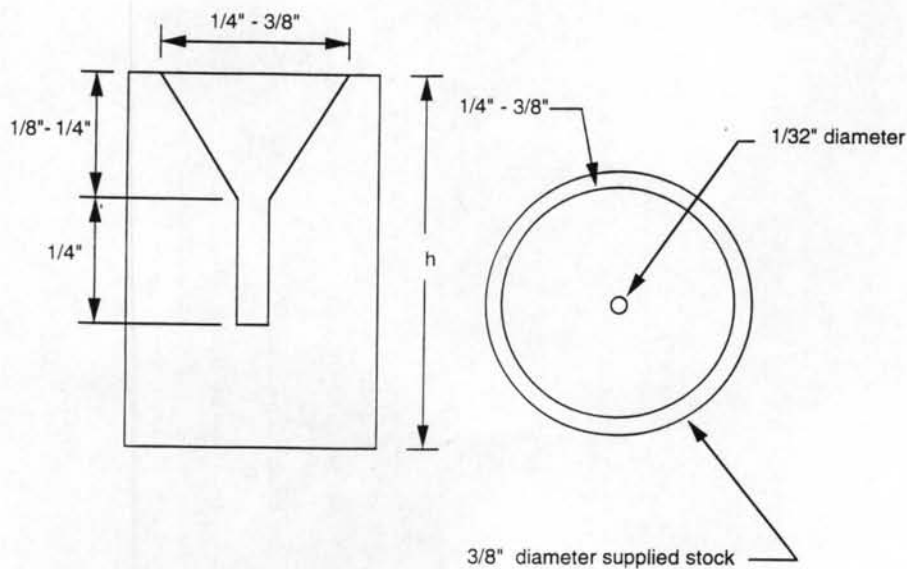


Figure 1. Schematic of stress rupture load train.



h varies

5 Pieces Each:
 $1/2''$, $3/4''$, $1''$, $1\ 1/4''$,
 $1\ 1/2''$, $1\ 3/4''$, $2''$, $2\ 1/4''$,
 $2\ 1/2''$

Figure 2. Diagram of weight showing hole and conical guide for the Sigma fiber.