Properties of Ceramic Filters

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Introduction

The mechanical integrity of ceramic filter elements is a key issue for hot gas cleanup systems. To meet the demands of advanced power systems, the filter components sustain thermal stresses of normal operations (pulse cleaning), of start-up and shut-down, and of process upsets such as excessive ash accumulation without catastrophic failure. They must also survive various mechanical loads associated with handling and assembly, normal operation, and process upsets. For near-term filter systems, the elements must survive operating temperatures of 1650 °F for three years.

Schumacher F40, Refractron 442T, and Coors alumina mullite candle filters tested at American Electric Power's PFBC at the Tidd plant and the Ahlstrom-Pyropower PFBC in Karhula, Finland resulted in failure of some candles. Coors monolithic ceramic filters were susceptible to thermal stresses. Refractron 442T and Schumacher F40 filters showed substantial creep and degradation of the binder material. Test results obtained at SRI showed that microcracking due to thermal stresses generated during pulse cleaning could occur when the temperature drop on the I.D. of the candle is 100 °F to 200 °F. Tensile creep tests indicated that the clay-bonded materials began to creep at ~1400 °F. Degradation of mechanical properties was measured in clay-bonded materials after exposure in Tidd and Karhula.

New Schumacher and Refractron candle filters - Schumacher FT20 and Refractron 326 - have been tested at SRI. These materials have a different binder intended to decrease the creep rate. Axial tensile, hoop tensile, tensile creep, and thermal expansion properties of these materials are presented here. One Refractron 326, one Schumacher FT20, and one Coors alumina mullite candle filter were sent to SRI after ~540 hours in service at Karhula. Hoop tensile strength was measured on several rings taken from various axial locations on each used candle filter. Hoop tensile strengths of the used and as-manufactured candle filters were compared to evaluate strength degradation after 540 hours in service.

Objectives

Objectives of the testing conducted at SRI were as follows:

1. Measure basic physical, mechanical, and thermal properties of candle filter materials and relate these properties to in-service performance.

2. Perform post-exposure testing of candle filter materials after service at Tidd and Karhula and compare post-exposure results to as-manufactured results to evaluate property degradation.

3. Based on measured properties and in-service performance, develop an understanding of material requirements for candle filter materials and help establish property goals.

4. Establish a test protocol for evaluation of candle filter materials.

Approach

Based on the hot gas cleanup conditions and the in-service performance of candle filters to date, several issues have been identified as critical issues for hot gas filter materials. Candle filters must posses sufficient mechanical strength to withstand handling and assembly and to withstand the weight and side loading due to pulse cleaning, start-up, and shut-down. Creep is an issue because ash accumulation has generated side loads leading to excessive creep in Schumacher FT20 and Refractron 326 materials. The materials must operate in the hot gas cleanup environment without excessive property degradation, and the candles must filter effectively. Some of these issues are not applicable to all materials. The materials received and tested to date at SRI and the critical issues for each material are summarized in Table 1.

Southern's approach is to measure basic material properties of candle filter materials and predict in-service performance based on the measured material properties. The properties measured address the critical issues discussed. For example, mechanical strength is addressed by the tensile and compressive strength while thermal stress susceptibility is addressed by measuring tensile stress-strain response and thermal expansion. A summary of critical material issues and Southern's methods for evaluation of each issue is given in Table 2.

Table 1

Summary of Critical Issues for Hot Gas Cleanup Filter Materials

Material	Mechanical Strength	Thermal Stress	Creep	Property Degradation	Filtration/ Pressure Drop
Clay-Bonded (Schumacher, Refractron)	X	X	X	X	X
Monolithic Ceramics (Coors)	X	X	X [1]	X	X
3M Composite	X	X	X		
Dupont/Lanxide PRD-66	X	X		X	X
Dupont/Lanxide Composite	X	X		X	X
Industrial Filter and Pump	X			X	X
Blasch	X	X		X	X

[1]Slow crack growth

Table 2

Critical Material Issues and Methods of Evaluation

Material Issue	Methods for Evaluation
Mechanical Strength, "Toughness"	Tensile Strength Compressive Strength Fracture Toughness
Thermal Stress Susceptibility	Tensile Stress-Strain Curve Thermal Expansion
Creep	Tensile Creep/Heat Deflection
Property Degradation	Tensile Stress-Strain After Exposure Ring Tensile After Exposure Microstructure
Filtration/Pressure Drop	Permeability

Test results presented in this paper are for Schumacher FT20 and Refractron 326 materials. The test matrix used to evaluate these two materials is given in Table 3.

Table 3

Test Matrix for Refractron 326 and Schumacher FT20 Candle Filter Materials

Test Type	Orientation	RT	1600 °F	1700 °F	1800 °F
Tensile	Hoop	9			
	Axial	4	4	4	4
Tensile Creep	Axial		4	4	
Thermal Expansion	Hoop	2	------------------------>		
	Axial	2	------------------------>		

Results

Schumacher FT20

Tensile results measured for Schumacher FT20 are plotted versus temperature in Figures 1 - 3. Tensile properties previously reported for Schumacher F40 are included in these figures for comparison. Figure 1 shows an average axial tensile strength for Schumacher FT20 of ~600 psi at room temperature increasing to a maximum value of ~1340 psi at 1600 °F. Schumacher F40 had an average axial tensile strength of ~1120 psi at room temperature increasing to a maximum value of ~1360 psi at 1500 °F. These results indicate that Schumacher FT20 has a reduced room temperature tensile strength; however, in the operating range the strength is near the same for FT20 and F40 materials.   Schumacher FT20 had an average room temperature hoop tensile strength of ~1690 psi. Figure 2 shows that the average value of Young's modulus of FT20 decreased with temperature from ~4.0 x 10^6 psi at room temperature to ~2.2 x 10^6 psi at 1600°F. Figure 3 shows that the tensile strain-to-failure of FT20 increased with temperature from ~0.00016 in/in at room temperature to ~0.00073 in/in at 1600 °F and ~0.0013 in/in at 1700 °F. Only one value of strain-to-failure was obtained at 1600 °F and 1700 °F. Additional results are needed at these test temperatures to confirm that these results are typical. Strain-to-failure results were near the same for FT20 and F40.

Unit thermal expansion of Schumacher FT20 and F40 are plotted in Figure 4. The thermal expansion curve of FT20 had a "kink" at ~400 °F which was not seen for F40. This kink in thermal expansion has been seen in other ceramics tested at SRI in the past and is probably due to the new binder used. From ~500 °F up, the shape of the

thermal expansion curves for FT20 and F40 was the same. Therefore, the coefficient of thermal expansion (CTE) in the operating range is the same. Figure 4 shows graphically the relationship between strain-to-failure and thermal expansion. The strain-to-failure measured at 1600 °F for FT20 is shown along with the temperature drop on the I.D. of a candle filter operating at this temperature which would cause this strain. This temperature drop does not take the cylindrical geometry into account. Based on the measured properties and the dimensions of Schumacher candle filters, a temperature drop of ~250 °F was calculated to cause microcracking in FT20 filters. For F40 filters, a temperature drop on the I.D. of the candle of ~180 °F was calculated to cause microcracking.

Room temperature hoop tensile strength was measured on nine Schumacher FT20 specimens after ~540 hours in-service at Karhula. Hoop tensile strength values of as-manufactured and post-exposure FT20 specimens are compared in Figure 5. This graph compared both the tensile strengths and the strength distributions. After ~540 hours in-service at Karhula, the average hoop tensile strength was decreased by ~9% from 1690 psi to 1530 psi. Post-exposure testing of previous clay-bonded SiC materials indicated that most of the strength degradation occurred rapidly and then the strength leveled off. Testing of the FT20 material after different exposure durations is needed to determine if the tensile strength would degrade further with longer exposure.

Creep testing of Schumacher FT20 is summarized in Figure 6. At 1600 °F and an axial tensile stress of 500 psi, the secondary creep rate was ~6.4 x 10^{-9} in/in/sec. Figure 6 shows the initial creep rates of specimens Creep-ax-2 and Creep-ax-7 were different; however, the secondary creep rates appear near the same. After ~117 hours at 1600 °F, specimen Creep-ax-7 was heated to 1700 °F with the same 500 psi tensile stress applied. The specimen then broke after ~17 hours. Creep rates of FT20 and F40 are compared graphically, along with creep rates for the Refractron materials, in Figure 7. Creep rates are compared by comparing glass/binder viscosity. Glass/binder viscosity is calculated from

$$\mu = \sigma_{binder}/3\varepsilon_{binder}$$

where, μ = glass/binder viscosity

σ_{binder} = stress in glass/binder

ε_{binder} = strain rate in glass/binder

From microstructural models developed for Schumacher F40, stress and strain in the glass are related to average body stress and strain by

$$\sigma_{binder} = 17\sigma_{avg.}$$

and $\varepsilon_{binder} = 50\varepsilon_{avg.}$

Microstructural models have not yet been developed for FT20 so the models for F40 were applied to this material also. Although this calculation does not give an accurate value for binder viscosity in Schumacher FT20, it does serve to provide a comparison of creep rates for FT20 and F40 materials. Figure 7 indicates that the creep rate of Schumacher FT20 is 1 to 2 orders of magnitude less than the creep rate of F40.
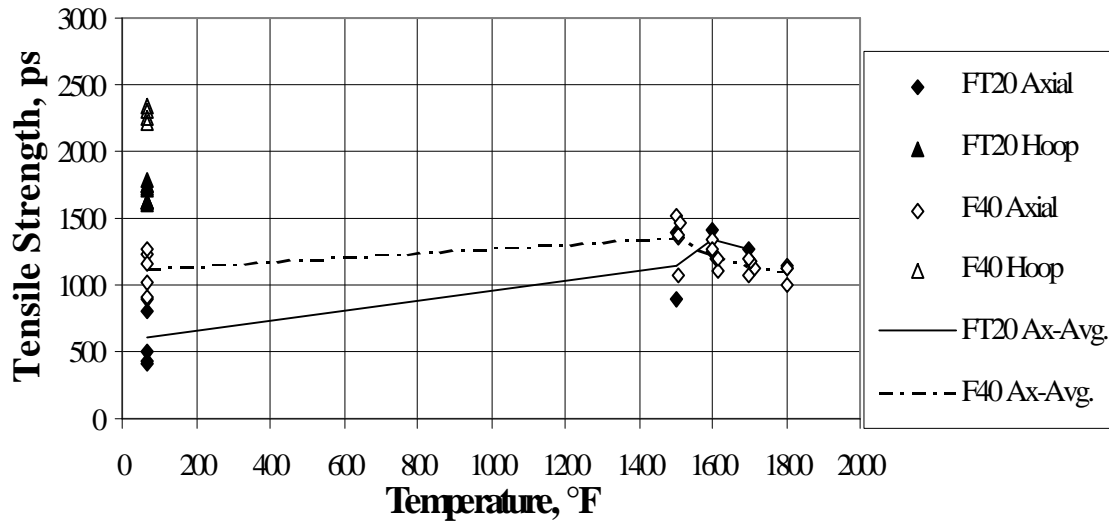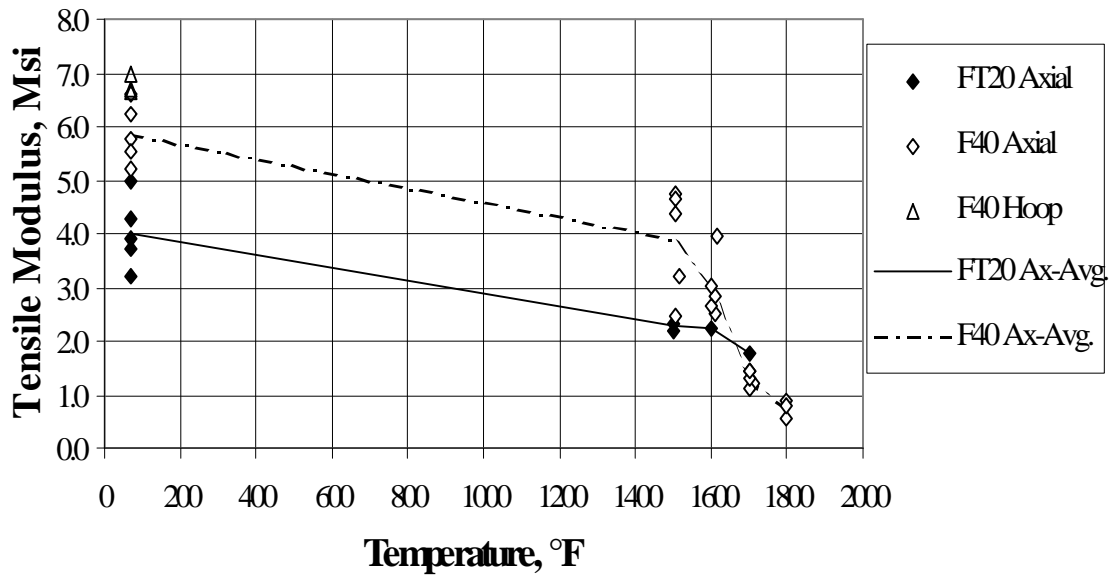
Figure 1. Ultimate Tensile Strength Versus Temperature for Schumacher FT20 and F40 Materials

Figure 2. Young's Modulus Versus Temperature for Schumacher FT20 and F40 Materials
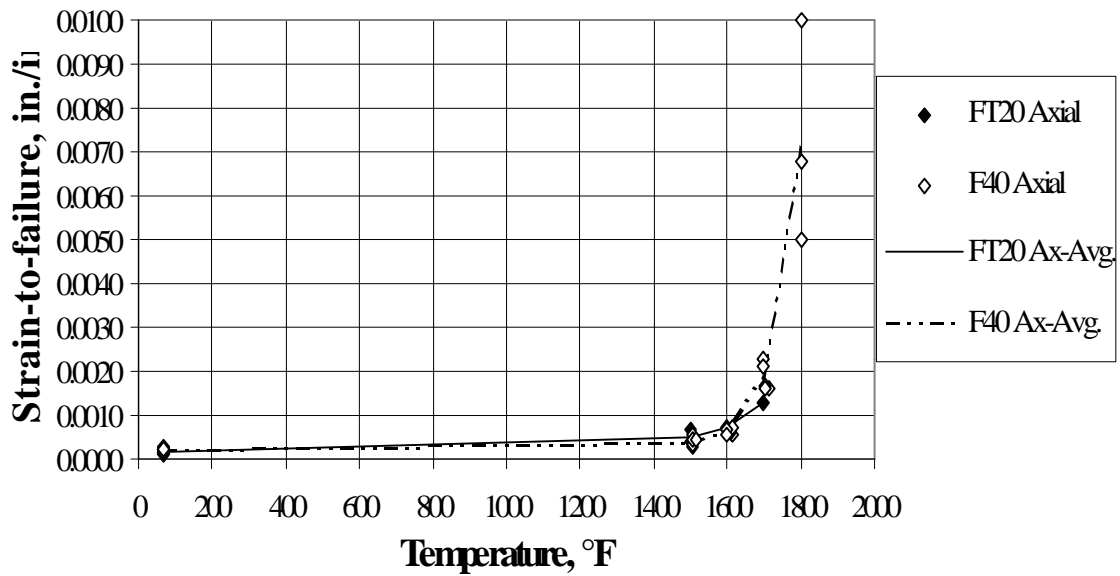
Figure 3. Tensile Strain-to-Failure Versus Temperature for Schumacher FT20 and F40 Materials
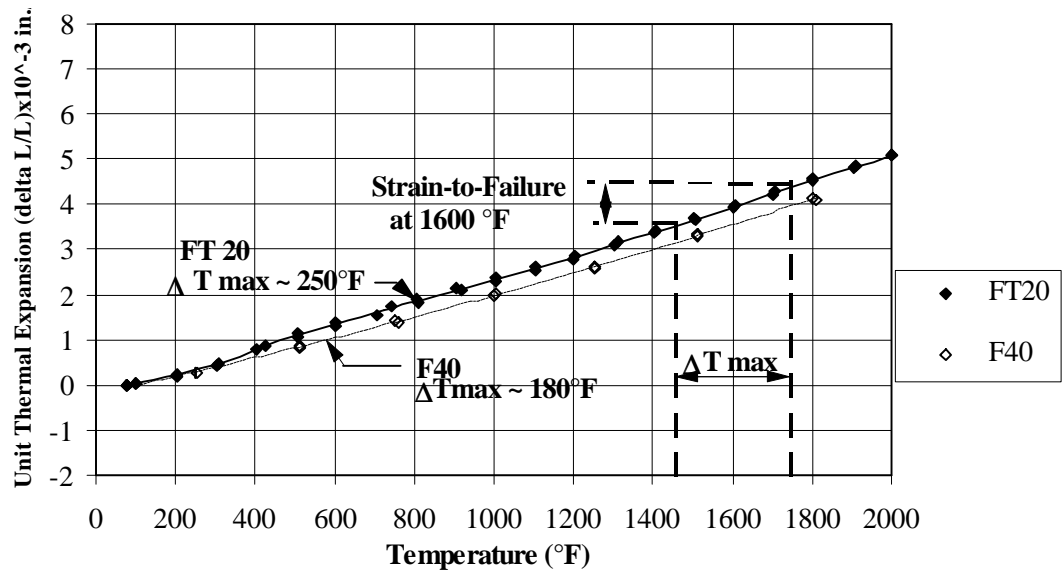
Figure 4. Unit Thermal Expansion of Schumacher FT20 and F40 Materials
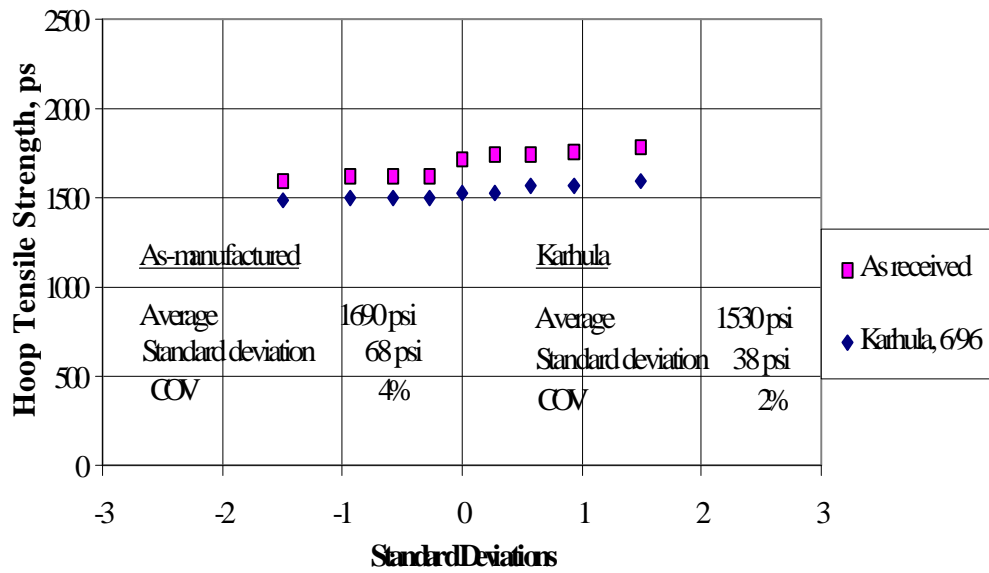
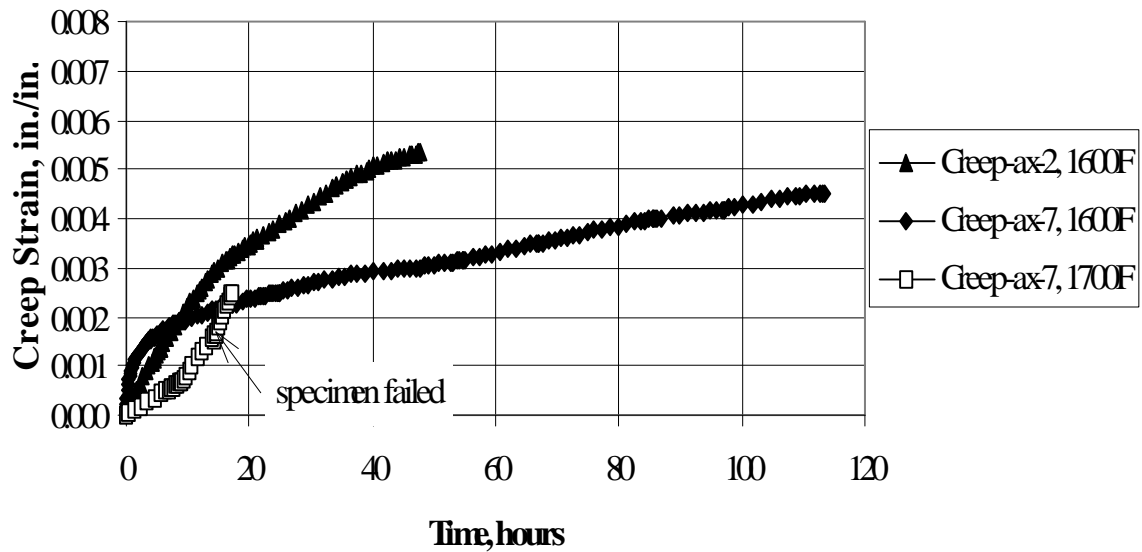Figure 5. Hoop Tensile Strength Distribution for As-Manufactured and Used Schumacher FT20

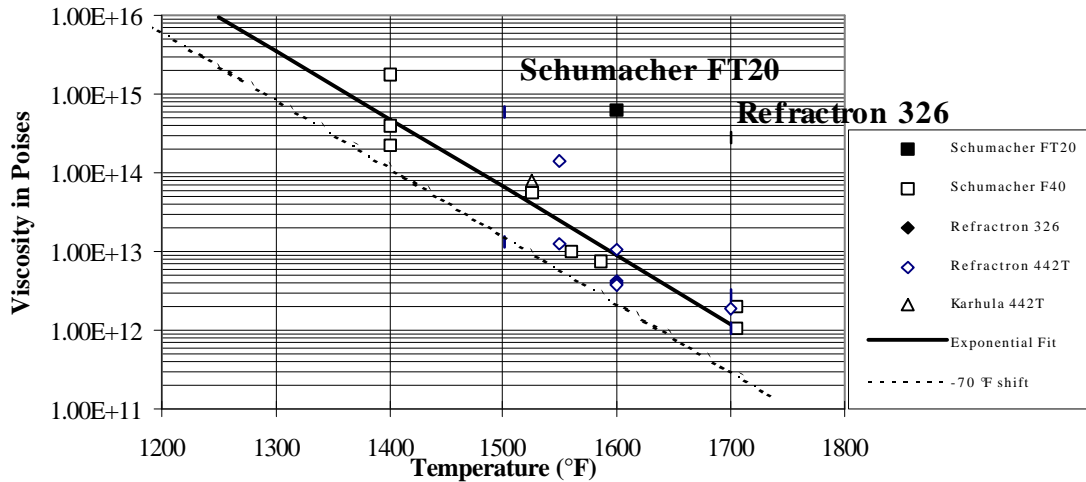Figure 6. Creep Strain Versus Time for Schumacher FT20 at 1600 °F and 1700 °F, 500 psi

Figure 7. Glass Viscosity Versus Temperature for Schumacher and
Refractron Candle Filter Materials

Refractron 326

Tensile results measured for Refractron 326 are plotted versus temperature in Figures
8 - 10. Tensile properties previously for Refractron 442T are included in these figures
for comparison. Figure 8 shows an average axial tensile strength for Refractron 326 of
~1200 psi at room temperature increasing to a maximum value of ~1600 psi at 1600
°F. Additional testing at 1400 °F or 1500 °F is needed to determine whether the
strength values obtained at 1600 °F represent the maximum strength of this material or
if the maximum strength is at some lower temperature. Refractron 442T had an
average axial tensile strength of ~2000 psi at room temperature decreasing to ~1430
psi at 1600 °F. These results indicate that Refractron 326 has a reduced room
temperature tensile strength; however, in the operating range the strength is near the
same for 326 and 442T materials.   Refractron 326 had an average room temperature
hoop tensile strength of ~2130 psi. Figure 9 shows that the average value of Young's
modulus of Refractron 326 decreased with temperature from ~5.6 x 10^6 psi at room
temperature to ~2.4 x 10^6 psi at 1600 °F. Figure 10 shows that the tensile strain-to-
failure of Refractron 326 increased with temperature from ~0.00020 in/in at room
temperature to ~0.00165 in/in at 1600 °F and then decreased to ~0.00050 in/in at 1800
°F. The decrease in strain-to-failure from 1600 °F to 1800 °F is different from any
other clay-bonded SiC materials tested thus far. No reason for this decrease has been
determined. This figure again shows the need for tensile measurements at ~1400 °F or
1500 °F to determine whether the strain-to-failure of 0.00165 in/in at 1600 °F is the
maximum value or if the maximum value occurs at some lower temperature.

Unit thermal expansion of Refractron 326 and 442T are plotted in Figure 11. The
thermal expansion curve of 326 had a "kink" at ~400 °F, similar to Schumacher FT20.
This kink is probably due to the new binder used. From ~500 °F up, the shape of the
thermal expansion curves for 326 and 442T was the same. Therefore, the CTE of

Refractron 326 and 442T at the operating temperature are near the same. Figure 4 shows graphically the relationship between strain-to-failure and thermal expansion. The strain-to-failure measured at 1600 °F for 326 is shown along with the temperature drop on the I.D. of a candle filter operating at this temperature which would cause this strain. This temperature drop does not take the cylindrical geometry into account. Based on the measured properties and the dimensions of Refractron candle filters, a temperature drop of ~320 °F was calculated to cause microcracking in 326 filters. For 442T filters, a temperature drop on the I.D. of the candle of ~180 °F was calculated to cause microcracking. Note that the temperature drop to cause microcracking was calculated based on the measured thermal expansion at 1600 °F. Because strain-to-failure is changing rapidly with temperature in this range, the temperature drop to cause microcracking is also changing rapidly. When strain-to-failure is measured at some intermediate temperature, probably 1500 °F, the temperature drop calculated to cause microcracking may be more than or less than 320 °F.

Room temperature hoop tensile strength was measured on nine Refractron 326 specimens after ~540 hours in-service at Karhula. Hoop tensile strength values of as-manufactured and post-exposure 326 specimens are compared in Figure 12. This graph compares both the tensile strengths and the strength distributions. After ~540 hours in-service at Karhula, the average hoop tensile strength decreased by ~33% from 2130 psi to 1430 psi. Testing of the 326 material after different exposure durations is needed to determine if the tensile strength would degrade further with longer exposure.

Creep testing of Refractron 326 is summarized in Figure 13. One specimen tested at 1600 °F and an axial tensile stress of 500 psi had a secondary creep rate was ~8.6 x 10^{-9} in/in/sec. Testing of a second specimen at 1700 °F and 500 psi was stopped after ~17 hours because of heater failure. Creep strain measured over 17 hours for this specimen is plotted in Figure 12 and is similar to the initial creep strain rate measured at 1600 °F. Re-testing of this specimen has begun and is still in progress. Creep rates measured for Refractron 326 and 442T are compared graphically, along with creep rates for the Schumacher materials, in Figure 7. As discussed previously, creep rates are compared by comparing glass/binder viscosity where binder viscosity is calculated from

$$\mu = \sigma_{binder}/3\varepsilon_{binder}$$

From microstructural models developed for Refractron 442T, stress and strain in the glass/binder are related to average body stress and strain by

$$\sigma_{binder} = 14\sigma_{avg.}$$

and $\qquad \varepsilon_{binder} = 65\varepsilon_{avg.}$

Microstructural models have not yet been developed for 326 so the models for 442T were applied to this material also. Although this calculation does not give an accurate

value for binder viscosity in Refractron 326, it does serve to provide a comparison of creep rates for 326 and 442T materials. Figure 7 indicates that the creep rate of Refractron 326 is ~2 orders of magnitude less than the creep rate of 442T.
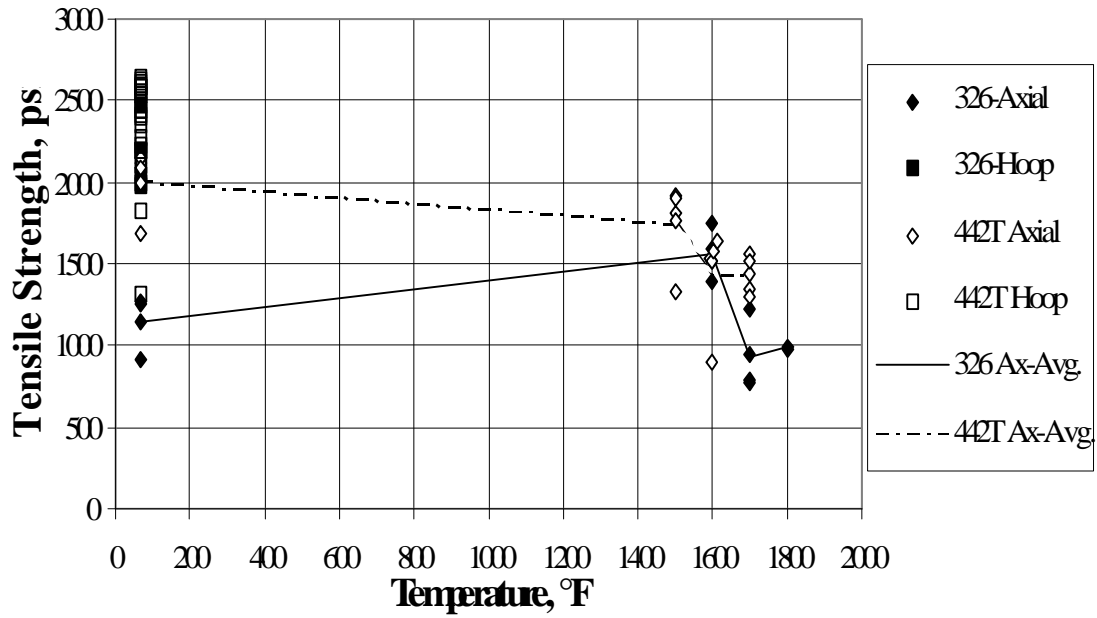

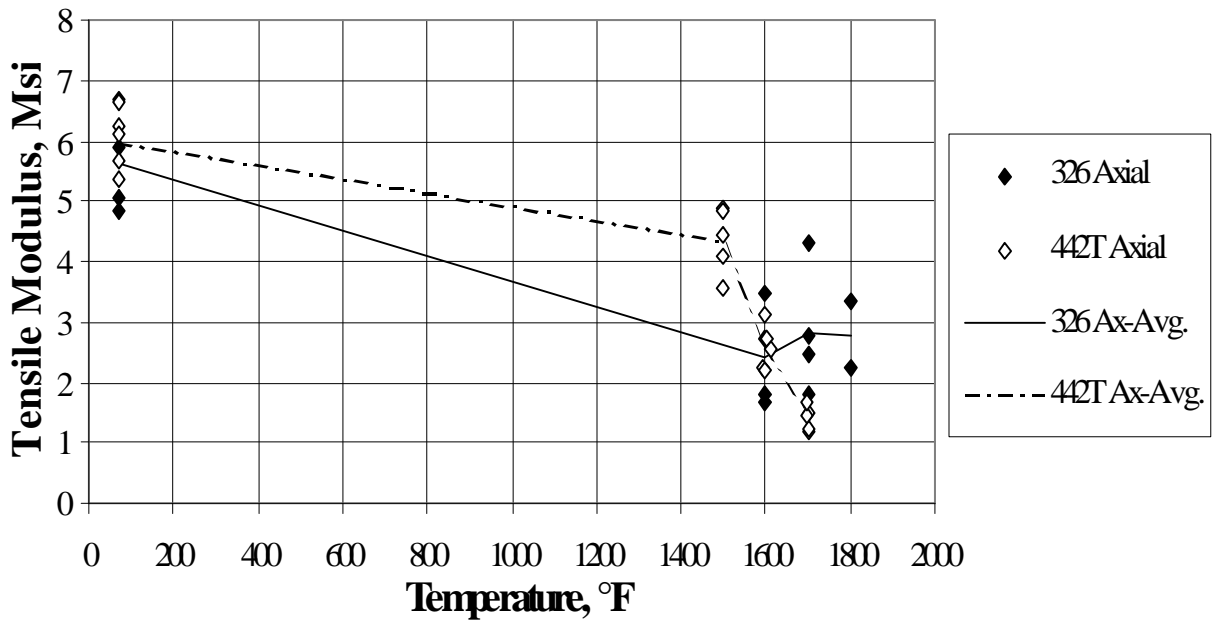Figure 8. Ultimate Tensile Strength Versus Temperature for Refractron 326 and 442T Materials

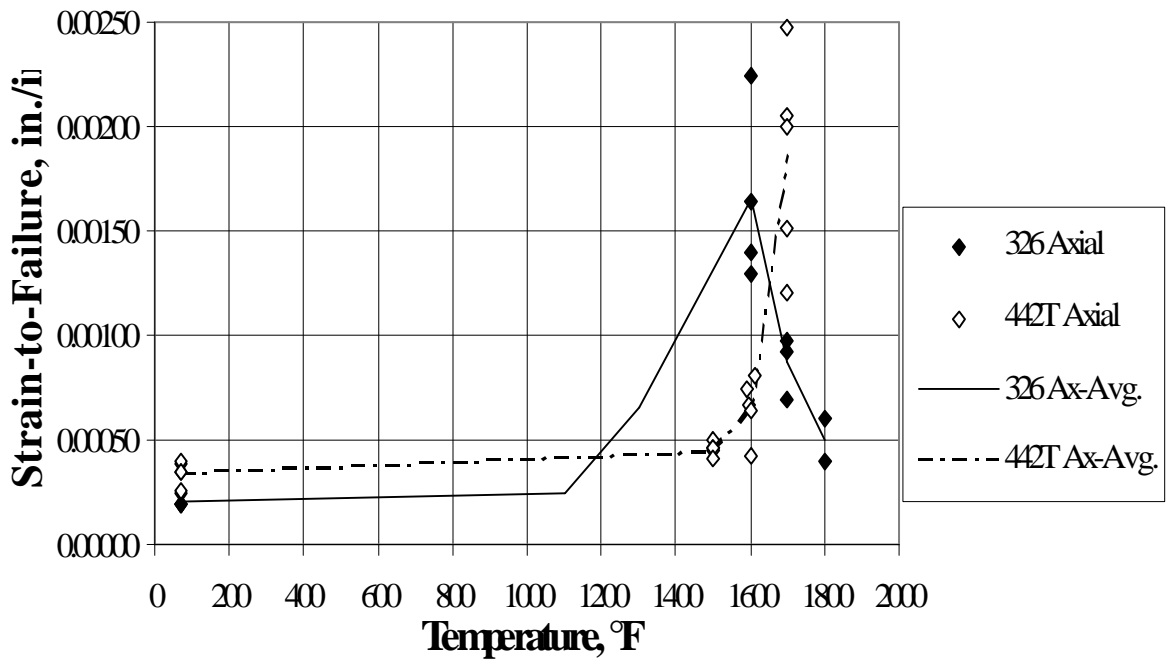Figure 9. Young's Modulus Versus Temperature for Refractron 326 and 442T Materials

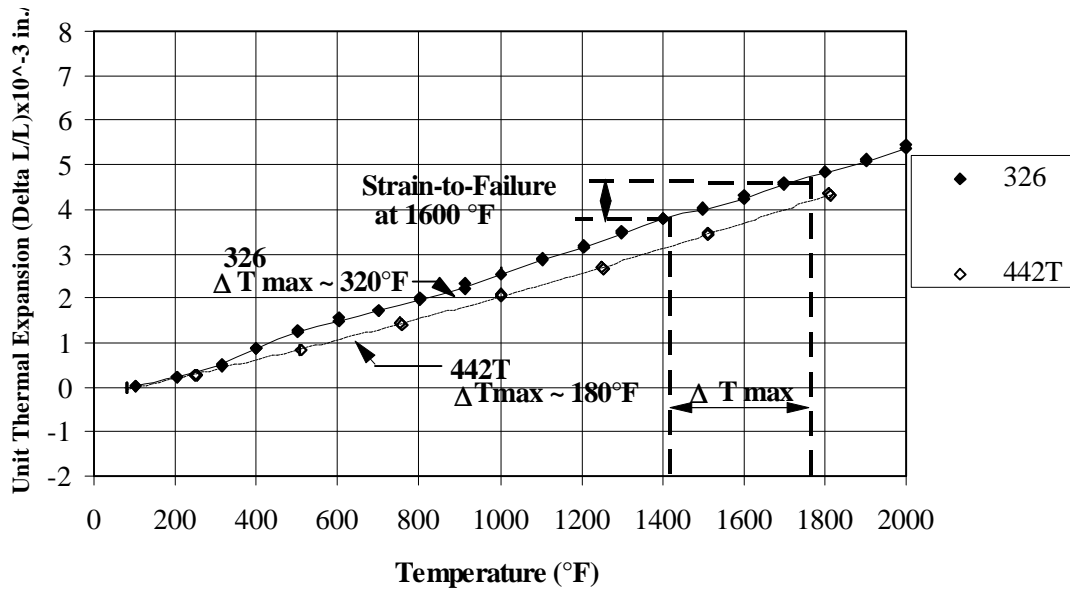Figure 10. Strain-to-Failure Versus Temperature for Refractron 326 and 442T Materials

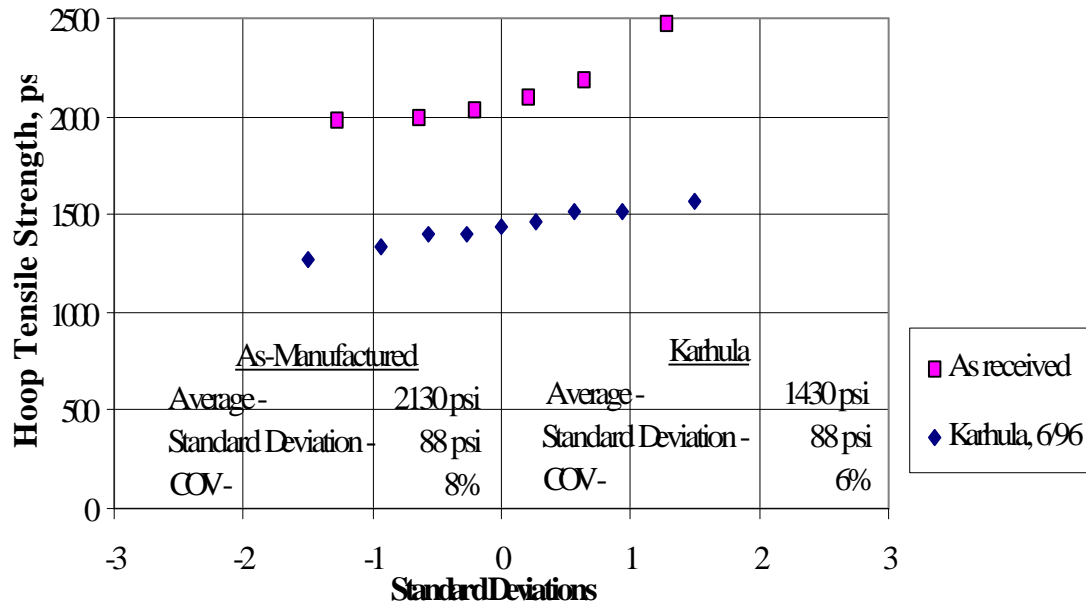Figure 11. Unit Thermal Expansion of Refractron 326 and 442T Materials

Figure 12. Room Temperature Hoop Tensile Strength Distribution for As-Manufactured
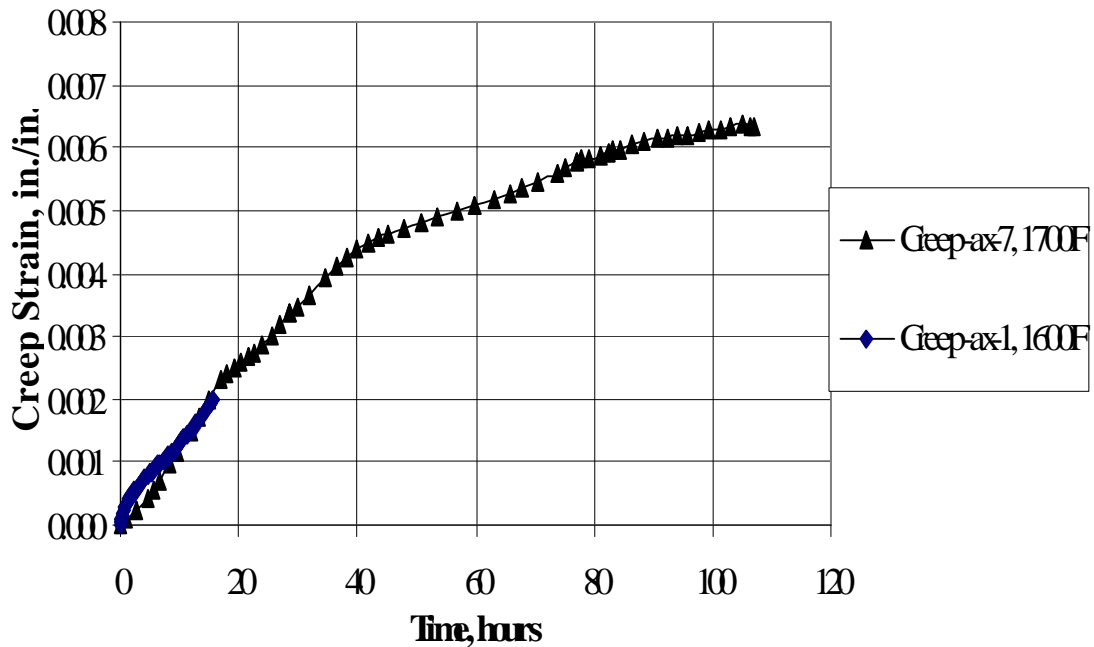and Post-Exposure Refractron 326 Material

Figure 13. Creep Strain Versus Time for Refractron 326 at 1600 °F and 1700 °F, 500 psi

Based on the properties measured, the following conclusions were obtained :

1. Schumacher FT20 and Refractron 326 materials have lower room temperature strengths than Schumacher F40 and Refractron 442T. In the operating range, the strength difference is small.

2. For Schumacher FT20 at an operating temperature of 1600 °F, thermal stress microcracking is likely to occur for $\Delta T \cong 250$ °F. For Schumacher F40, $\Delta T \cong 180$ °F would likely cause microcracking.

3. For Refractron 326 at an operating temperature of 1600 °F, thermal stress microcracking is likely to occur for $\Delta T \cong 320$ °F. For Refractron 442T, $\Delta T \cong 180$ °F would likely cause microcracking.

4. The average room temperature hoop tensile strength of Schumacher FT20 decreased ~9% from 1690 psi to 1530 psi after ~540 hours in-service at Karhula.

5. The average room temperature hoop tensile strength of Refractron 326 decreased ~33% from 2130 psi to 1430 psi after ~540 hours in-service at Karhula.

Future Activities

Microstructural evaluations of Schumacher FT20 and Refractron 326 are needed to model the creep response of these materials. Microstructural evaluations of Schumacher F40 and Refractron 442T provided the models of creep in these materials as flow of the binder and allowed calculations of glass viscosities and prediction of creep rates for various temperature and stress levels. Similar studies of FT20 and 442T materials are in progress. Microstructures of as-manufactured material and post-exposure material will be compared to determine the mechanisms and degree of degradation suffered in-service.

Post-exposure hoop tensile testing after some service times other than 540 hours are needed to determine whether the degradation suffered at Karhula would continue with longer service durations. For Schumacher F40 and Refractron 442T, most of the property degradation occurred in the first few hours. The properties of Schumacher F40 and Refractron may or may not degrade further with service durations greater than 540 hours.

Axial tensile testing of Refractron 326 is needed at ~1500 °F because the properties are changing rapidly with temperature in this range. The maximum tensile strength and strain-to-failure measured were at 1600 °F; however, the maximum values for may occur at a lower temperature. Testing at ~1500 °F is needed to define the properties within the operating range.