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Characterization of Filter Elements for Service in a Coal Gasification Environment

Key Words: Hot gas filters, Metal filter elements, Ceramic filter elements

Introduction

The Power Systems Development Facility (PSDF) is a joint Department of Energy/Industry sponsored engineering-scale facility for testing advanced coal-based power generation technologies. High temperature, high pressure gas cleaning is critical to many of these advanced technologies. Barrier filter elements that can operate continuously for nearly 9000 hours are required for a successful gas cleaning system for use in commercial power generation. Since late 1999, the Kellogg Brown & Root Transport reactor at the PSDF has been operated in gasification mode. This paper describes the test results for filter elements operating in the Siemens-Westinghouse particle collection device (PCD) with the Transport reactor in gasification mode. Operating conditions in the PCD have varied during gasification operation as described elsewhere in these proceedings (Martin et al, 2002). Nominal operating conditions during the most recent gasification runs were:

PCD Inlet Temperature	371 – 427 Deg. C (700 – 800 Deg. F)
System Pressure	1.10 – 1.65 MPa (160 – 240 psig)
Face Velocity	0.015 – 0.026 m/s (3 – 5 ft/min)
Baseline Pressure Drop	126 – 302 mbar (50 – 120 inH_2O)

In addition to these normal operating conditions, the elements are subjected to system upsets that result in more severe, but transient conditions. Thermal transients are sometimes caused by oxygen breakthrough to the PCD causing ignition of char on the surface of the elements. During several of these transients, temperature measurements on the surface of some elements have indicated a temperature increase of approximately 300°F in one minute. There was evidence, such as the response of some elements during thermal transients, that the transients were more severe on other elements that were not instrumented with thermocouples.

Twenty-two different types of filter elements have been tested in gasification operation at the PSDF. The types of elements tested are shown in Table 1. The hours of operation accumulated on each type of element are shown in Figure 1. The value plotted on the y-axis of Figure 1 is the total hours accumulated on all elements of a particular type. The maximum hours accumulated on any individual element is shown above the bar for each element type. Pall iron aluminide (Fe_3Al) and Hastelloy X metals, Pall 326 ceramic, and

Pall/Schumacher ceramics with the "T" binder, T05-20, T10-20, and TF20, have seen the most operation and are the subject of this paper. A brief description of each of these element types is given below.

Pall Fe_3Al filter elements were manufactured of sintered metal powder with a composition of 2 at.% Cr,-15.9% Al,-balance Fe. The elements were seamless cylinders with nominal dimensions of 60 mm (2.36 in) O.D. and 56 mm (2.22 in) I.D. The elements were manufactured in sections of approximately 0.5 m (20 in) length with individual sections joined by welding the porous Fe_3Al to solid 310 SS support rings. Most elements tested at the PSDF consisted of three sections for an overall length of 1.5 m (60 in) but some elements had four sections for an overall length of 2 m (80 in). All elements were preoxidized by Pall to form a protective layer of alumina over the particles.

Pall Hastelloy X filter elements were manufactured of sintered metal powder to nominal dimensions of 60 mm (2.36 in) O.D. and 53 - 56 mm (2.1 - 2.2 in) I.D. The elements had an axial seam weld. These elements were manufactured in sections of approximately 0.5 m (19.5 in) length with individual sections joined by welding the porous Hastelloy X to solid Hastelloy C-276 support rings. All elements tested at the PSDF consisted of three sections for an overall length of 1.5 m (59 in).

Pall/Schumacher TF20, T10-20, and T05-20 were membrane-coated, clay-bonded SiC particle filter elements. The structural support wall of these materials consisted of individual SiC particles connected by clay or glass bridges. The elements had a nominal I.D. of 40 mm (1.58 in) and a nominal O.D. of 60 mm (2.36 in). Mechanical and thermal properties of the elements were controlled by the structural walls and the thin (~100 μm) membrane layer provided filtration. The structural walls of TF20, T10-20, and T05-20 were the same but the filtration membranes were different in chemical composition and pore size. Since mechanical and thermal properties of these materials were controlled by the structural walls, the properties are presented together on one graph. Pall/Schumacher N10-20 is similar to T10-20 except that the binder has a lower cristobalite content and improved bonding between the binder and SiC particles.

Pall 326 was a membrane-coated, bonded SiC particle filter element. The structural support wall consisted of SiC particles in an alumino-silicate binder. The elements had a nominal I.D. of 40 mm (1.58 in) and a nominal O.D. of 60 mm (2.36 in). Mechanical and thermal properties of the elements were controlled by the structural walls and the relatively thin membrane layer provided filtration.

Objective

The objectives of this work were to understand the performance of filter elements in the coal gasification environment and identify filter materials with the best chance of meeting the demand of 9000 hours of continuous operation under the conditions given above.

Approach

The approach used in this effort has been to measure the physical, mechanical, and thermal properties of the filter materials and relate those properties to performance in the coal gasification environment. Based on operating conditions measured at the PSDF, in-service performance of filter elements tested so far, and anticipated requirements in future systems, several material issues have been identified that are critical to the performance of filter elements. Test matrices used for testing as-manufactured elements and elements after gasification operation are shown in Tables 2 and 3. Tensile tests were conducted in both the axial and hoop direction. Axial tests were conducted on dog-bone shaped specimens cut from the wall of the filter elements. Load was applied through pins in the grips. Hoop tensile tests were conducted on 50.8 mm (2 inch) long rings loaded internally by hydrostatic pressure. Tensile testing was used to determine if a material had sufficient strength to withstand all operating conditions including handling, installation, and backpulse cleaning. Tensile testing was also conducted on filter elements removed after operation. The tensile strength and tensile strain-to-failure, an indication of ductility, were compared to those measured on as-manufactured elements to assess degradation during operation. Unit thermal expansion (unit thermal expansion refers to length change divided by the original length) and thermal conductivity were measured on as-manufactured materials to predict performance during thermal transient conditions. Thermal conductivity testing was not feasible on the metal filter materials because of their thin walls and relatively high thermal conductivity; however, these same characteristics also made thermal stress failure unlikely in these elements. Optical microscopy at low magnifications, <200X, was also conducted as needed.

Results

As of June, 2003 Pall iron aluminide (Fe_3Al) elements have been in gasification operation at the PSDF for a total of ~150,000 hours with some individual elements in operation for over 2000 hours. The operational results with Fe_3Al elements have been very good with no element failures during normal operation. The elements have survived several thermal transients where the temperature measured at the element surface increased ~167°C (300°F) in 60 seconds and at least one thermal transient where the surface temperature increased ~333°C (600°F) in 180 seconds during off-coal operation. However, there have been three element failures, all of which occurred under severe circumstances during off-coal operation. The first was during gasification run TC06A when char in the PCD, apparently bridged material, ignited because of oxygen breakthrough to the PCD. The temperature on one side of the element most likely exceeded 982°C (1800°F), the temperature where a phase change accompanied by a permanent length decrease occurs in Fe_3Al. Since only one side of the element reached that temperature, the element tended to bow toward the side with the decreased length. Since the bottom support rods prevented the element from bowing enough to accommodate the length decrease, it cracked. The effects of this thermal transient were localized. Inspection of the elements indicated that only 5 Fe_3Al elements were affected. Two elements failed during an off-coal period of gasification run TC07 and the cause of these failures was not determined. There were some anomalous conditions when the failures occurred. There was severe char bridging and oxygen detected downstream of the PCD, so char ignition may have occurred; however, the thermocouples on the filter elements did not detect any temperature spikes. There was also a period of erratic backpulse system performance preceeding the element failures. It is not known if these anomalous conditions caused the element failures in run TC07.

Tensile strength measured at room temperature and 399°C (750°F) is plotted versus hours in operation in Figure 2. There was some element-to-element variability in strength, but nearly all values fell in the range of 90 to 138 MPa (13 to 20 ksi) with no decrease after up to 1450 hours in operation. There was also no strength difference between room temperature and 399°C (750°F). One advantage of Fe_3Al elements over monolithic ceramics is greater ductility. Strain-to-failure is plotted versus hours in operation in Figure 3. The strain-to-failure appeared stable, in the range of 0.5 – 1.0% at room temperature and 0.8 – 1.8% at 399°C (750°F), for up to 1450 hours in gasification operation. Earlier results on elements from combustion operation at the PSDF indicated that the tensile strength and strain-to-failure remained constant through at least 2780 hours in operation.

The flow resistance of the elements was measured using pressure drop as a function of flowrate measurements. All flow-tests were conducted using air at ambient temperature and pressure flowing from the outside to the inside of the elements. Corrections must be made for the viscosity of syngas at the operating temperature to obtain the pressure drop for the same flowrate during operation. Elements were tested in as-manufactured condition and after operation. The elements flow-tested after operation were cleaned before testing either by high-pressure water or by chemical cleaning with a mild caustic solution. For as-manufactured elements, the average pressure drop was 10.3 mbar (4.1 inH_2O) at a face velocity of 91.4 m/hr (5 ft/min). For elements tested after operation, the average pressure drop increased slightly to 17.1 mbar (6.9 inH_2O) at a face velocity of 91.4 m/hr (5 ft/min). This is still well within the acceptable range for re-installation into the PCD. The flow resistance does not appear to be increasing with longer operation. The flow resistance was similar for either cleaning method.

Pall Hastelloy X elements have been in gasification operation for a total of approximately 9000 hours with some individual elements in operation for over 1800 hours. These elements were in operation during the same thermal transients as the Fe_3Al and have survived with no failures. One Hastelloy X element was in the area of the PCD where the temperature of the Fe_3Al elements apparently exceeded 982°C (1800°F) during TC06A and survived with no apparent affects.

Tensile testing has been conducted at room temperature on both as-manufactured elements and elements after 1025 hours in gasification operation at the PSDF. Tensile strength and strain-to-failure are plotted versus hours in operation in Figures 4 and 5. The measured ultimate tensile was slightly greater after 1025 hours in operation than as-manufactured. This probably represents material variability and not an increase with operation. The measured tensile strength for all specimens, as-manufactured or after 1025 hours in operation, ranged from 193 – 269 MPa (28 – 39 ksi). Ductility was also unaffected by 1025 hours in operation with strain-to-failure of >8% measured on as-manufactured material or after operation.

One concern with the use of Hastelloy X elements in coal gasification is the formation of nickel sulfide resulting in blinded pores and increased pressure drop. This has been reported (Nieminen, et. al., 1996) in laboratory tests at 300 ppmw H_2S, 500°C (932°F). Flow, porosity, and x-ray diffraction tests have been conducted to try and determine if nickel sulfide is forming and blinding the pores at the PSDF operating conditions of 100 – 200 ppmw H_2S and 371 – 427°C (700 – 800°F). Flow-test results showed an average pressure drop of 6.3 mbar (2.5 inH_2O) at a face velocity of 91.4 m/hr (5 ft/min) using air at ambient temperature and pressure for as-manufactured elements. For elements removed after operation and then cleaned by pressure washing with water, the average pressure drop increased slightly to 14.2 mbar (5.7 inH_2O) at a face velocity of 91.4 m/hr (5 ft/min). This is still well within the acceptable range for re-installation into the PCD. These results showed similar flow resistance for the Hastelloy X and Fe_3Al elements both as-manufactured and after removal from operation and cleaning. Porosity measurements showed that the porosity after TC06B was 21.7% compared to 23.3% for virgin material. After ultrasonic cleaning, the porosity of the sample from TC06B returned to 22.3%, indicating that at least some of the decreased porosity was because of char trapped in the pores that was removed by ultrasonic cleaning. No nickel sulfide was detected by X-ray diffraction; however, X-ray diffraction will not detect a compound with a concentration of less than ~3% by volume.

Pall 326 elements have been tested in gasification operation at the PSDF for a total of ~17,000 hours with a maximum of 218 hours on an individual element with no failures. Room temperature tensile strength was measured as-manufactured, after 183 hours, and after 218 hours in operation and the results are plotted in Figure 6. The average strength was 14.3 mPa (2100 psi) with no change after 218 hours in gasification. Earlier test results indicated that there was no strength change during combustion operation for up to 2830 hours.

Pall/Schumacher elements with the “T” binder, TF20, T10-20, and T05-20, have been tested in gasification operation for a total of ~23,000 hours with a maximum of 218 hours on an individual element. There have been no failures during normal operation; however, one element failed in the flange region during sand circulation before coal feed started for run GCT3. Room temperature tensile strength was measured as-manufactured, after 183 hours, and after 218 hours in operation and the results are plotted in Figure 7. The strength decreased ~30% during the first 180 hours, from 13 MPa (1875 psi) to 8.6 MPa (1250 psi); however, earlier results from combustion elements showed a similar strength decrease during the first 500 hours of operation with no further decrease for up to at least 1800 hours. The initial strength decrease may have occurred faster but no elements were tested with fewer than 180 hours in combustion or 500 hours in gasification.

Pall/Schumacher N10-20 elements have been tested in gasification operation at the PSDF for a total of 654 hours with a maximum of 218 hours on an individual element with no failures. Room temperature tensile strength was measured as-manufactured and after 218 hours in operation and the results are plotted in Figure 8. The average strength was 16.8 MPa (2430 psi) with no change after 218 hours in gasification.

Conclusions

Both the operational results and material property measurements obtained so far indicate that in the absence of major thermal transients Pall Fe_3Al and Hastelloy X elements can survive for at least 2000 hours in the gasification operating environment at the PSDF. However, successful operation in a commercial system will require filter elements to operate continuously for nearly 9000 hours. Iron aluminide elements have failed under extreme off-coal conditions on two different occasions during gasification operation. One of these occasions was a severe thermal transient with the temperature on several elements probably reaching at least 982°C (1800°F). A small number of Pall Hastelloy X elements were also in operation during these same gasification runs and survived. While the iron aluminide elements failed under these severe conditions, they have survived some others, including one measured temperature increase of 333°C (600°F) in three minutes and another of 222°C (400°F) in 1-1/2 minute, that would have likely caused failure of Pall 326 or Pall/Schumacher elements with the "T" binder. It is important to note that no Pall 181 or Pall/Schumacher N10-20 elements have failed during operation, but they were not installed during these most severe thermal transients. Because of better performance during thermal transients, most or all operation at the PSDF in the near future will be with metal elements. Property measurements will continue as operational experience with these types of filter elements increases to address the affect of long-term exposure to this operating environment. Future testing will also address other issues that effect long-term operation including integrity of joints in the metal elements, corrosion or chemical degradation, the effect of cyclic thermal and mechanical loading, and manufacturing consistency. While the SiC ceramic elements have not been tested recently because of susceptibility to thermal stress failure, test results indicated that in the absence of severe thermal transients, these elements could survive in the gasification environment at the PSDF.

New types of filter materials are also being introduced into the system. Pall/Fluid Dynamics sintered metal fiber elements, manufactured using Haynes alloy HR160, are now being tested in operation. Three HR160 elements have been tested so far with 800 hours on one. No property measurements have been conducted on this material yet. Materials characterization will begin and operation will continue with these materials.

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Table 1. Filter Elements Tested in Gasification at the PSDF

Monolithic Ceramics	**Ceramic Composites**	**Metals**
Pall 326	3M Oxide/Oxide	Pall Fe_3Al
Pall 442T	McDermott	Pall Hastelloy X
Pall 181	PRD-66C	Pall 310 SS
Pall/Schumacher TF20	AIT N610/Mullite	Fairey 316L SS
Pall/Schumacher T10-20		Fairey Inconel 601
Pall/Schumacher T05-20		Fairey Hastelloy HR
Pall/Schumacher N10-20		Pall/Fluid Dynamics Haynes HR160
IF&P REECER™		Pall/Fluid Dynamics Fecraloy
		Pall/Fluid Dynamics Haynes 214
		Pall/Fluid Dynamics Haynes 230

Table 2. Test Matrix for As-Manufactured Filter Elements

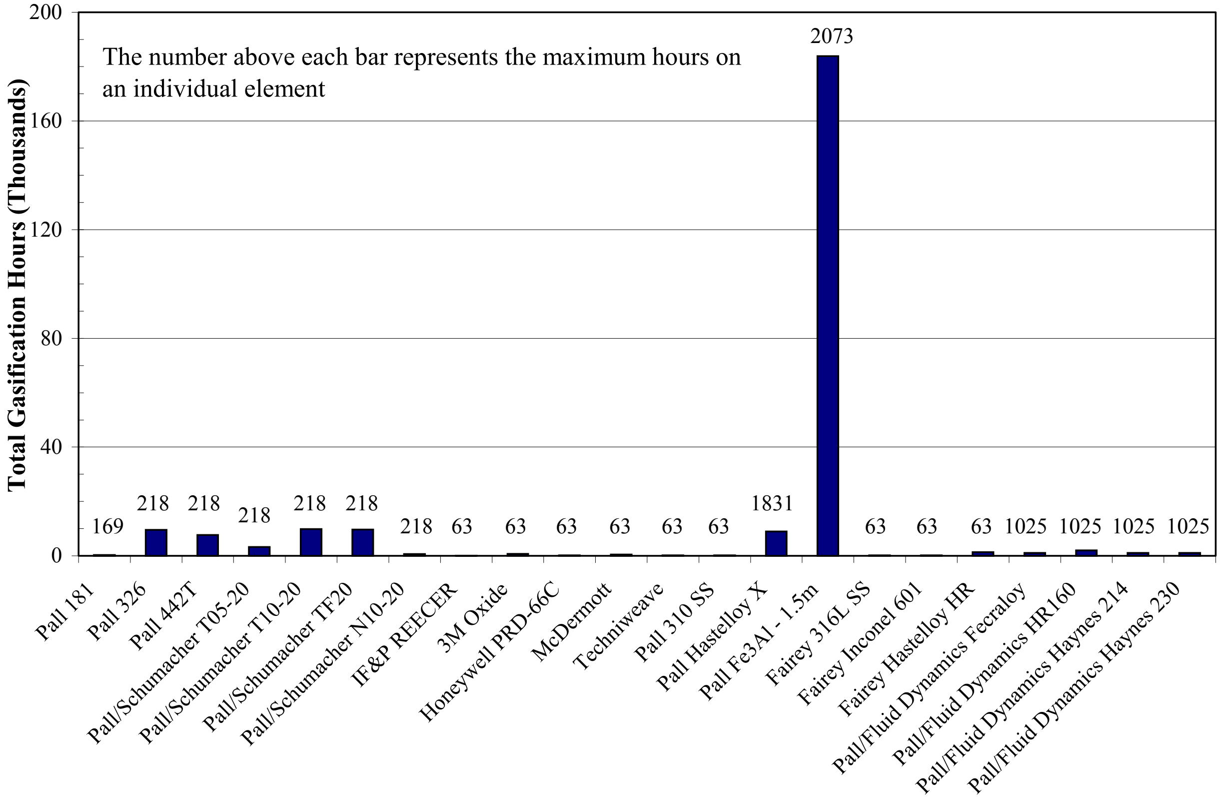
		Temperature in Deg. C (Deg. F)		
Test	**Room**	**399 (750)**	**760 (1400)**	**982 (1800)**
Hoop Tension	6			
Axial Tension	6	3	3	3
Thermal Expansion	2--→			
Thermal Conductivity	2---→			
Microscopy	As-req'd			

Notes: 1. Hoop tension by hydrostatic pressure.
2. Thermal conductivity as-required based on wall thickness and material type.

Table 3. Test Matrix for Filter Elements After Gasification Operation at the PSDF

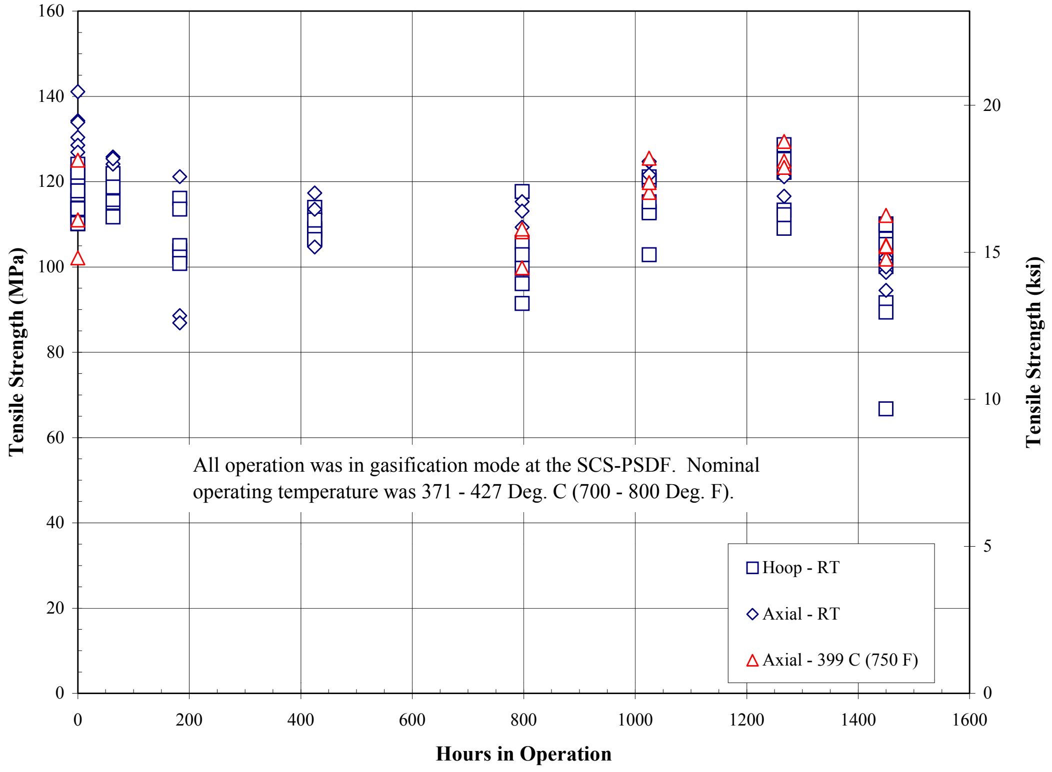
		Temperature in Deg. C (Deg. F)		
Test	**Room**	**399 (750)**	**760 (1400)**	**982 (1800)**
Hoop Tension	6			
Axial Tension	6	3		
Microscopy	As-req'd			

Notes: 1. Hoop tension by hydrostatic pressure.



The number above each bar represents the maximum hours on an individual element

Figure 1. Hours of Gasification Operation for Filter Elements Tested at the PSDF



All operation was in gasification mode at the SCS-PSDF. Nominal operating temperature was 371 - 427 Deg. C (700 - 800 Deg. F).

Figure 2. Tensile Strength of Pall Fe_3Al

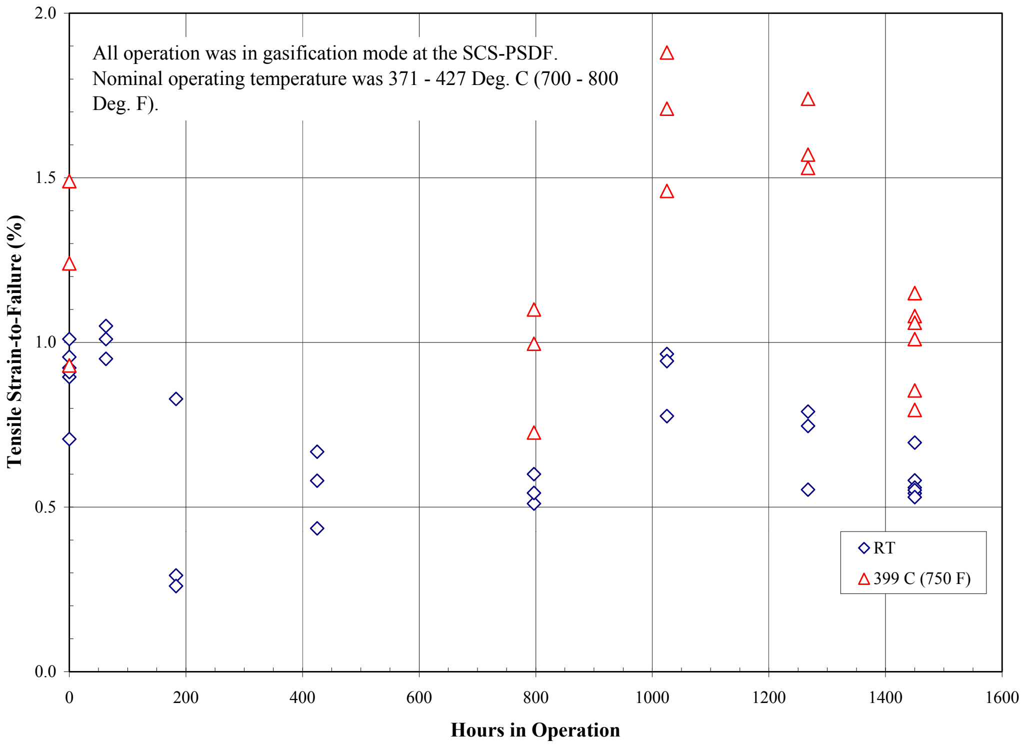


Figure 3. Strain-to-Failure of Pall Fe_3Al

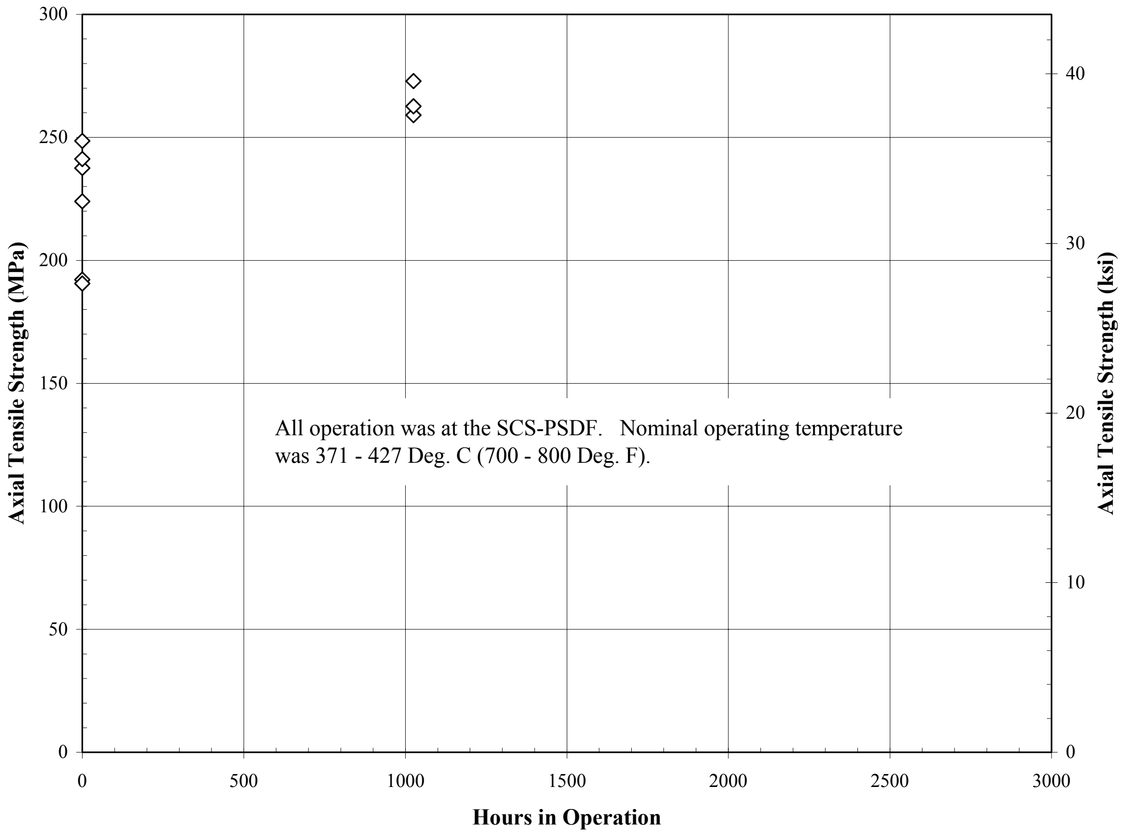


Figure 4. Room Temperature Tensile Strength of Pall HastelloyX

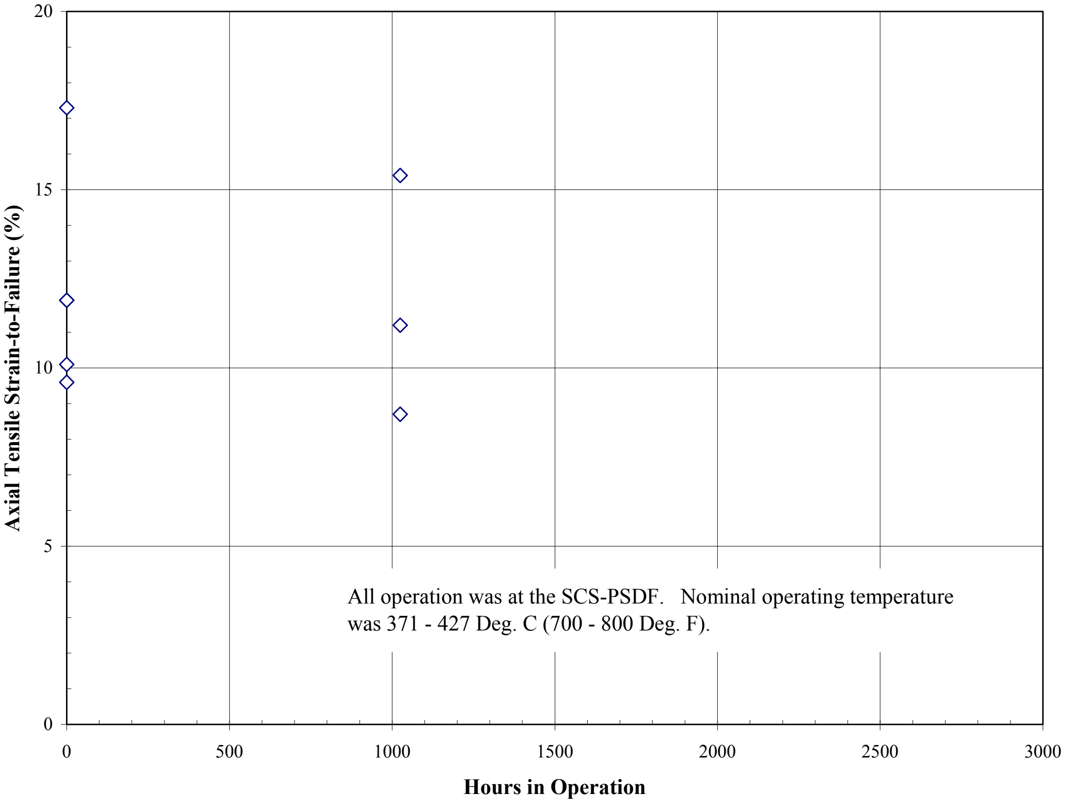


Figure 5. Room Temperature Tensile Strain-to-Failure of Pall Hastelloy X

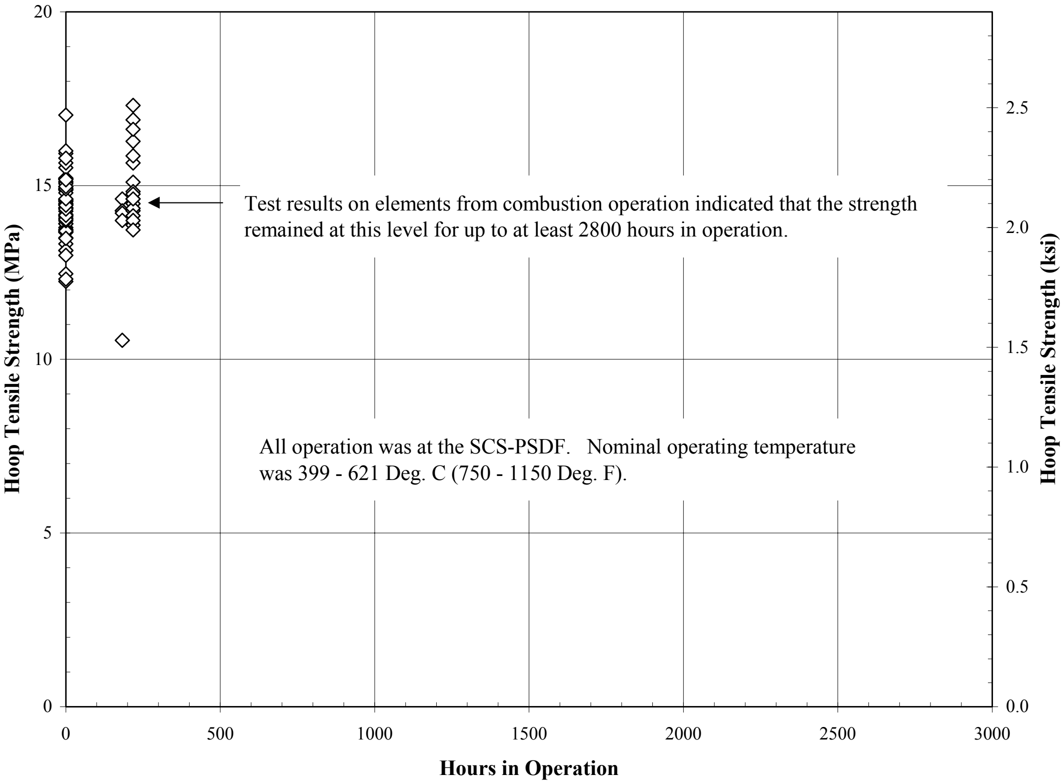


Figure 6. Room Temperature Hoop Tensile Strength of Pall 326

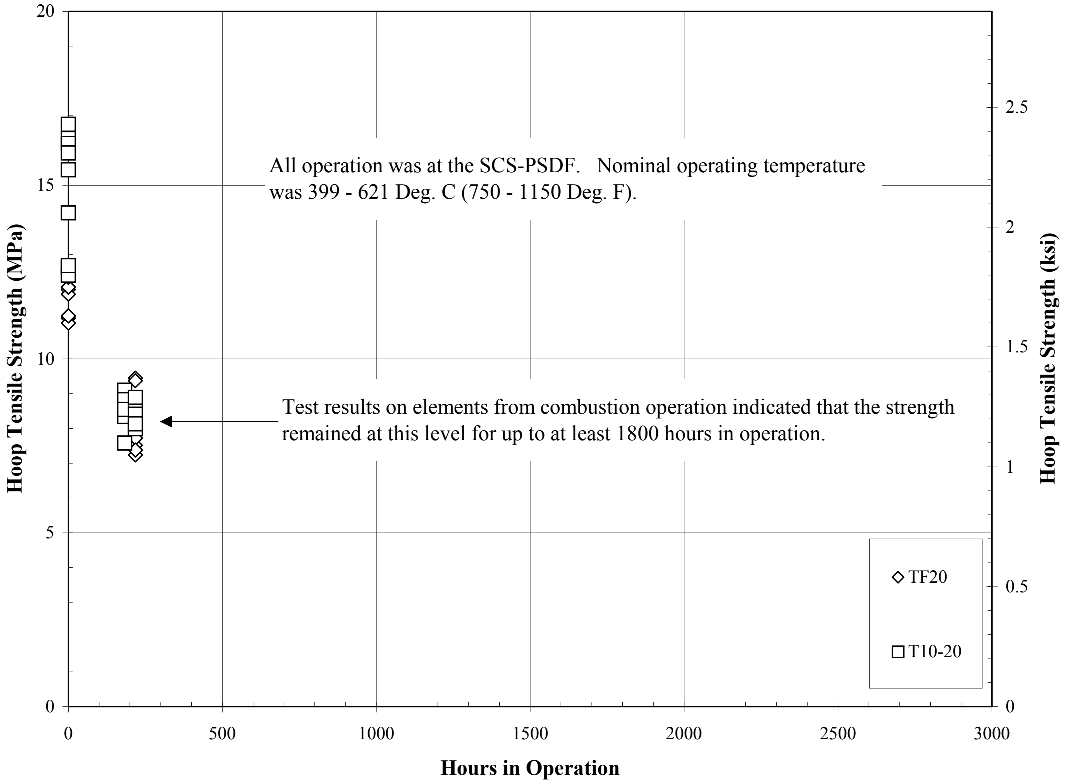


Figure 7. Room Temp. Hoop Tensile Strength of Pall/Schumacher TF20 and T10-20

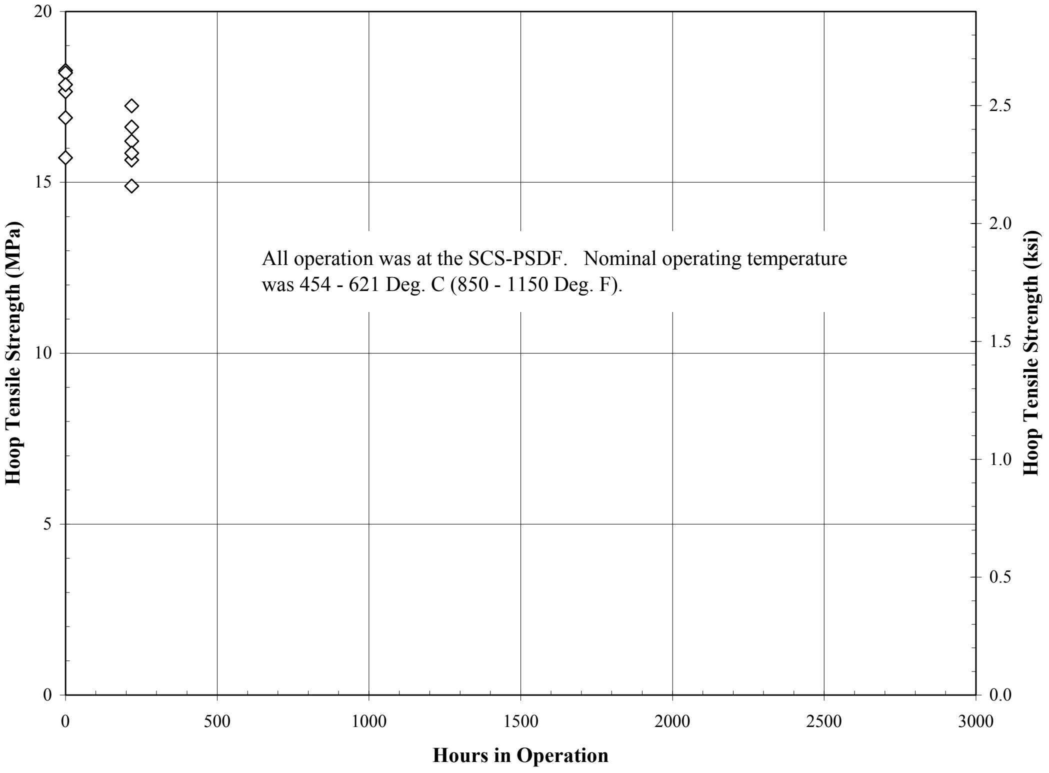


Figure 8. Room Temperature Hoop Tensile Strength of Pall/Schumacher N10-20