

# HIGH TEMPERATURE LIFE TESTING OF 80NI-20CR WIRE IN A SIMULATED MARS ATMOSPHERE FOR THE SAMPLE ANALYSIS AT MARS (SAM) INSTRUMENT SUITE GAS PROCESSING SYSTEM (GPS) CARBON DIOXIDE SCRUBBER

Christopher Hoffman<sup>1</sup>, Bruno Munoz<sup>1</sup>, Cynthia Gundersen<sup>2</sup>, Walter Thomas III<sup>3</sup> and Timothy Stephenson<sup>3</sup>

<sup>1</sup>Ball Aerospace & Technologies Corp., <sup>2</sup>AMU Engineering Inc., <sup>3</sup>NASA Goddard Space Flight Center (GSFC)

## ABSTRACT

In support of the GPS for the SAM instrument suite built by NASA/GSFC, a life test facility was developed to test the suitability of 80Ni-20Cr alloy wire, 0.0142 cm diameter, for use as a heater element for the carbon dioxide scrubber. The element would be required to operate at 1000°C in order to attain the 800°C required for regeneration of the getter. The element also would need to operate in the Mars atmosphere, which consists mostly of CO<sub>2</sub> at pressures between 4 and 12 torr. Data on the high temperature degradation mechanism of 80Ni-20Cr in low pressure CO<sub>2</sub>, coupled with the effects of thermal cycling, were unknown. In addition, the influence of work hardening of the wire during assembly and the potential for catastrophic grain growth also were unknown. Verification of the element reliability as defined by the mission goals required the construction of a test facility that would accurately simulate the duty cycles in a simulated Mars atmosphere. The experimental set-up, along with the test protocol and results will be described.

## INTRODUCTION

The Sample Analysis at Mars (SAM) instrument suite, being built and tested at Goddard Space Flight Center (GSFC) under direction of Principle Investigator, Dr. Paul Mahaffy, makes up over half of the science payload of the Mars Science Lab (MSL) rover that will launch at the end of 2009. SAM will address the past, present and future habitability of Mars using a suite of three primary instruments. These instruments include the Quadrupole Mass Spectrometer (QMS), provided by GSFC, the Gas Chromatograph (GC), provided by U. Paris/CNRS, and the Tunable Laser Spectrometer (TLS), provided by JPL. The major subsystems include the Chemical Separation and Processing Laboratory (CSPL), the Sample Manipulation System (SMS), and the Wide Range Pumps (WRP).

The five major science goals of SAM are as follows:

- 1) Survey carbon compound sources and evaluate their possible mechanism of formation and destruction.
- 2) Search for organic compounds of biotic and prebiotic importance. Of special interest is methane.
- 3) Reveal the chemical and isotopic state of elements (i.e., N, H, O, S and others) that are important for life as we know it.

- 4) Evaluate the habitability of Mars by studying its atmospheric chemistry and the composition of trace species that are evidence of interactions between the atmosphere and soil.
- 5) Understand atmospheric and climatic evolution through measurements of noble gas and light element isotopes.

The QMS analyzes the Mars atmosphere through direct gas sampling or through gases processed in the CSPL. It also functions as a detector for the GC. The TLS implements a search for methane and makes precision measurements of oxygen and carbon isotope ratios in carbon dioxide. The CSPL is the largest of all of the major subsystems and includes the Gas Processing System (GPS) that is comprised of microvalves, transfer and calibration gas reservoirs, enrichment cells, getter pumps, scrubbers and all associated manifolds.

The GPS component of interest is the CO<sub>2</sub> scrubber. Since CO<sub>2</sub> constitutes 95.32% of the Mars atmosphere it is important to remove this dominant species from the gas stream to provide enrichment of the trace gas molecules of most interest for analysis. This is the primary function of the CO<sub>2</sub> scrubber. After adsorption of the CO<sub>2</sub> the scrubber getter is fully regenerated during the mission by high temperature thermal processing which releases the CO<sub>2</sub> and prepares the getter for CO<sub>2</sub> adsorption during the next analysis cycle. Full regeneration of the getter requires the heating element, comprised of an 80Ni-20Cr alloy wire, 0.0142 cm diameter, to operate at 1000°C. The scrubber heater operates in the Martian atmosphere and is in a temperature-controlled environment ranging from -40°C to +50°C. The objective is to establish the reliability of the heater element operating cyclically to 76% of its melting point, based on the absolute temperature scale, in the low pressure CO<sub>2</sub> atmosphere of Mars and assure its suitability for achieving mission goals.

## **BACKGROUND**

There were three important facets to executing an accurate life test. First, ensuring the operating temperature of the element was as close as possible to the required 1000°C. Second, maintaining a relatively low pressure CO<sub>2</sub> environment that simulates exposure to the Mars atmosphere during the life test. And third, automating the life cycle test and monitoring the performance of the element.

To determine the electrical current required to achieve a temperature of 1000°C, preliminary testing was performed on a 53 cm length of 80Ni-20Cr alloy wire, 0.0142 cm in diameter. This closely approximated the length to be threaded into the CO<sub>2</sub> scrubber cartridge which was unavailable at the time. Based on the temperature coefficient of resistance of 1.07 for this alloy wire (ref. 1) calculations determined the wire resistance at 1000°C based on the room temperature resistance of the wire. The current required to achieve the wire temperature was 1.3 amperes. The wire temperature of 1000°C ± 30 °C was confirmed with an optical pyrometer.

Ensuring the correct operating temperature allowed evaluation of the effect of the potential downward shift in recrystallization temperature due to cold working of the thin wire as it is threaded into the scrubber cartridge. The amount of cold work is dependent on the initial condition of the wire, which was annealed in this instance, plus the bending required for threading the wire. The condition of the element after threading into the cartridge is therefore

difficult to estimate since it is dependent on workmanship and the geometry of the scrubber cartridge. These variables could directly affect the survivability of the wire. The cold work imparted to the wire during assembly, together with cyclic stresses during temperature cycling, could cause the metal grains to recrystallize and grow to such an extent that the microstructure of the wire evolves from polycrystalline to a chain of single grains. This condition reduces the creep strength of the wire by allowing strain to be accommodated by grain boundary sliding. Strain in the wire would occur during each cycle due to the thermal expansion mismatch with the alumina heater element. When two grains begin to slide past one another, the local cross section is reduced which increases local current density and joule heating causing metal vaporization and failure. Using a heat sink to cool the wire to  $-40^{\circ}\text{C}$  to simulate the worst-case low-temperature exposure was not necessary since this would add only an additional 6% to the thermal excursion between nominal  $20^{\circ}\text{C}$  room temperature and the  $1000^{\circ}\text{C}$  operating temperature experienced in a thermally passive terrestrial simulation.

Maintaining a relatively low pressure  $\text{CO}_2$  environment that simulates exposure to the Mars atmosphere during the life test was crucial to successfully evaluating the behavior of the element. The simulated Mars atmosphere was achieved with the use of a mechanical vacuum pump combined with a  $\text{CO}_2$  supply cylinder. The system used a metered flow of  $\text{CO}_2$  in combination with the mechanical vacuum pump to maintain a system pressure at approximately 7.5-8.0 torr. Figure 1 shows a schematic representation of the components used to maintain the required atmosphere.

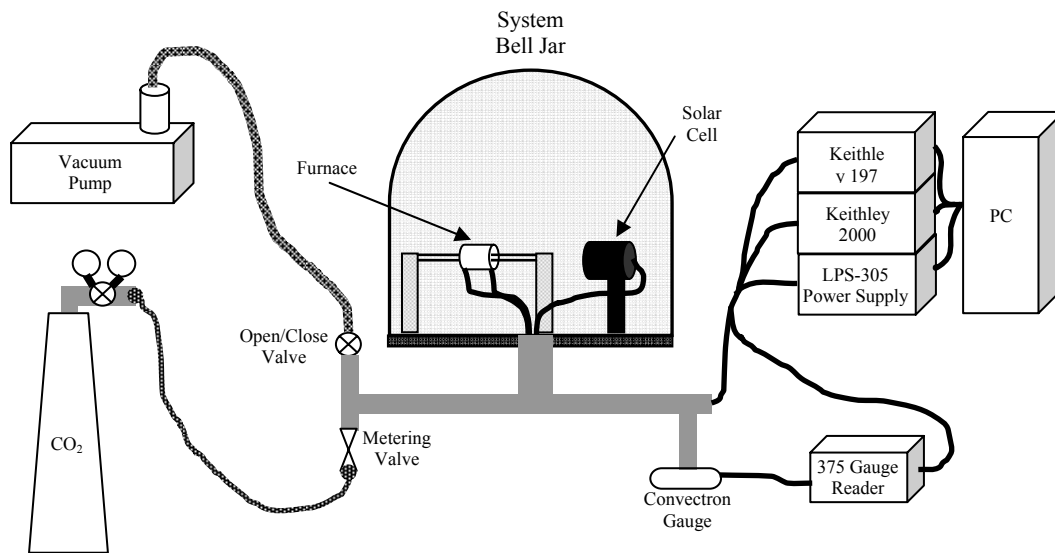


Figure 1. Scrubber element life test facility system diagram.

Metal alloys such as 80Ni-20Cr that form protective  $\text{Cr}_2\text{O}_3$  scale are susceptible to accelerated degradation at high temperature due to the evaporation of  $\text{CrO}_3$  (ref. 2). This condition thins the  $\text{Cr}_2\text{O}_3$  scale and depletes the matrix of Cr. In addition, temperature cycling can cause the scale to spall, exposing the underlying alloy to oxidation. Continued temperature cycling can therefore develop a reduced cross section. This condition can produce local hot

spots in a current-carrying element that further enhance degradation and cause early failure. The behavior of 80Ni-20Cr alloy wire at 1000°C in low pressure CO<sub>2</sub> was unknown.

Automation of the life cycle test and monitoring the performance of the element during testing required the development of custom software. This was crucial for continued, autonomous operation of the test allowed performance data to be downloaded for analysis at the conclusion of the experiment.

## **HARDWARE AND SOFTWARE COMPONENTS OF THE SAM CO<sub>2</sub> SCRUBBER ELEMENT LIFETEST FACILITY**

### **Data Acquisition and Control System**

The primary components of the data acquisition and control system are the Windows PC, National Instruments GPIB-USB Card, Keithley 2000 Digital Multimeter with 10 Channel Scanner Card, Keithley 197 Digital Multimeter, LPS305 Power Supply and LabVIEW™ software. A LabVIEW VI™ virtual instrument program was written to perform all of the data acquisition and storage functions as well as limit testing and control functions.

The primary control function performed by the VI, via a serial bus interface with the LPS305, was to cycle the furnace on and off at a constant current value selected by the user. The user also had control over the on and off duration of the power source. The user could select the on/off profile that best simulated in-flight instrument processes. The data system also provided a cycle counter and pressure limit. The pressure limit is a user selectable value that the program uses to prevent power cycling of the furnace in an atmosphere that does not meet test objectives.

The data from the test setup was stored in tab delimited files that are easily imported into spreadsheet programs. The data was stored at a user selectable rate. The parameters stored for all testing of the SAM scrubber cartridge heater element were: Date, Time, Elapsed Time, Cycles, Voltage, Current, Resistance, Solar Cell Current, Pressure, as well as temperature data from three Type-K thermocouples. The heater element electrical parameters along with the solar cell current were measured to characterize the change in performance of the 80Ni-20Cr alloy wire. This data verified that the power source was constant over the test duration to within  $\pm 2.5$  milliamperes and provided insight into the degradation of the element. In addition, the resistance of the element is used to estimate the element temperature to verify that the flight condition of 1000°C was achieved. This was confirmed using an optical pyrometer. The temperatures derived from the resistance were also compared with the thermocouple data. The pressure was monitored to verify stability of the simulated Mars atmosphere.

### **Pressure Limits**

The data acquisition and control software provided a user selectable range for system pressure. If the pressure exceeded the selected range, the program would disable the power to the furnace until the system pressure returned to the nominal operating range of ~7.5-8 torr.

## Life Test Facility

Figure 2 is a photograph of the as-built facility and shows the entire setup including all instrumentation that provide data on the status of the scrubber heater element under test along with the accompanying support hardware and computer.

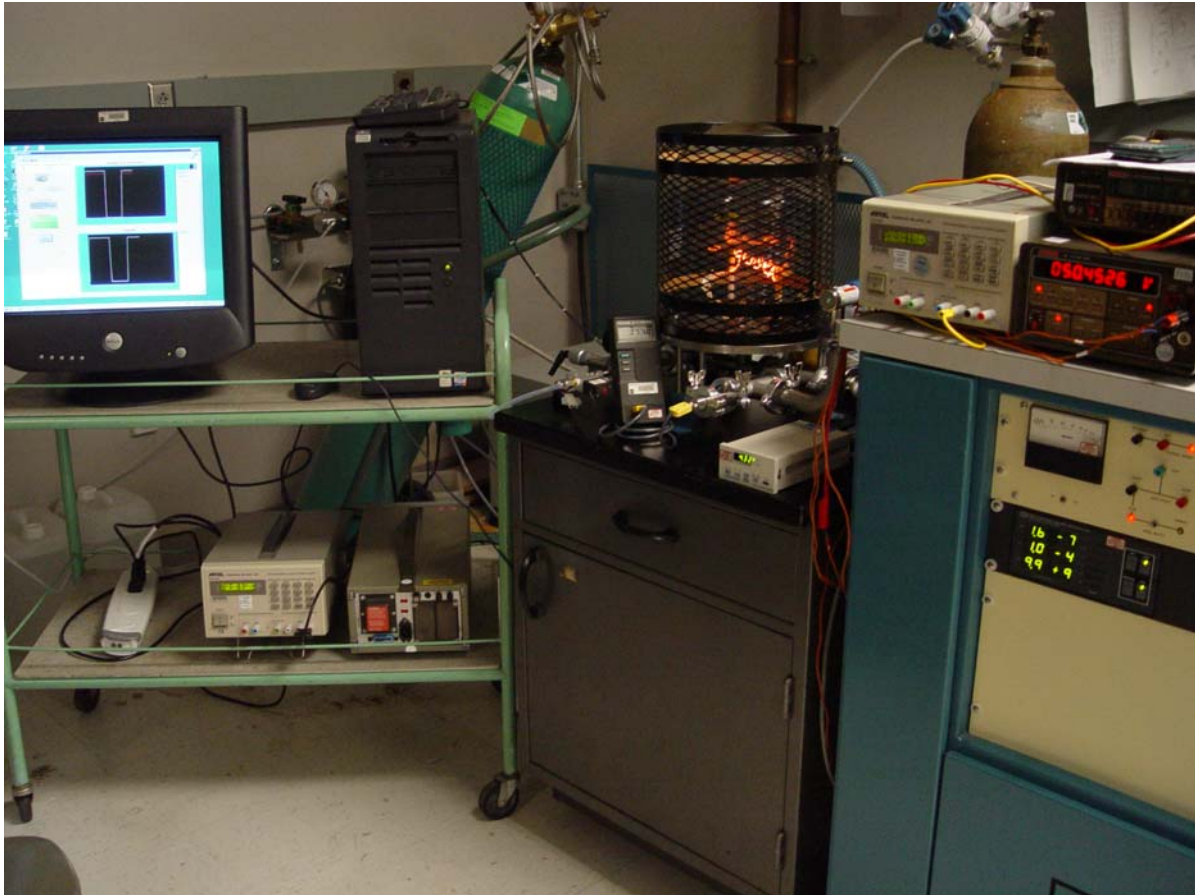


Figure 2. Scrubber heater element life test facility.

## TESTING SUMMARY

### Test Procedure

Figure 3 shows the wired furnace mounted and ready for testing. Current was applied to the heater element by the power supply and adjusted until the measured voltage across the element produced a resistance indicating an element temperature of  $1000^{\circ}\text{C}$  (as predicted by the temperature coefficient of resistance). Testing then proceeded with flight-like  $\text{CO}_2$  scrubber units which were powered by a current source programmed with a period of 20 minutes and a duty cycle of 50%, resulting in three complete on-off cycles per hour. The optical pyrometer was an

additional measurement technique used to confirm the element temperature throughout the life testing program.

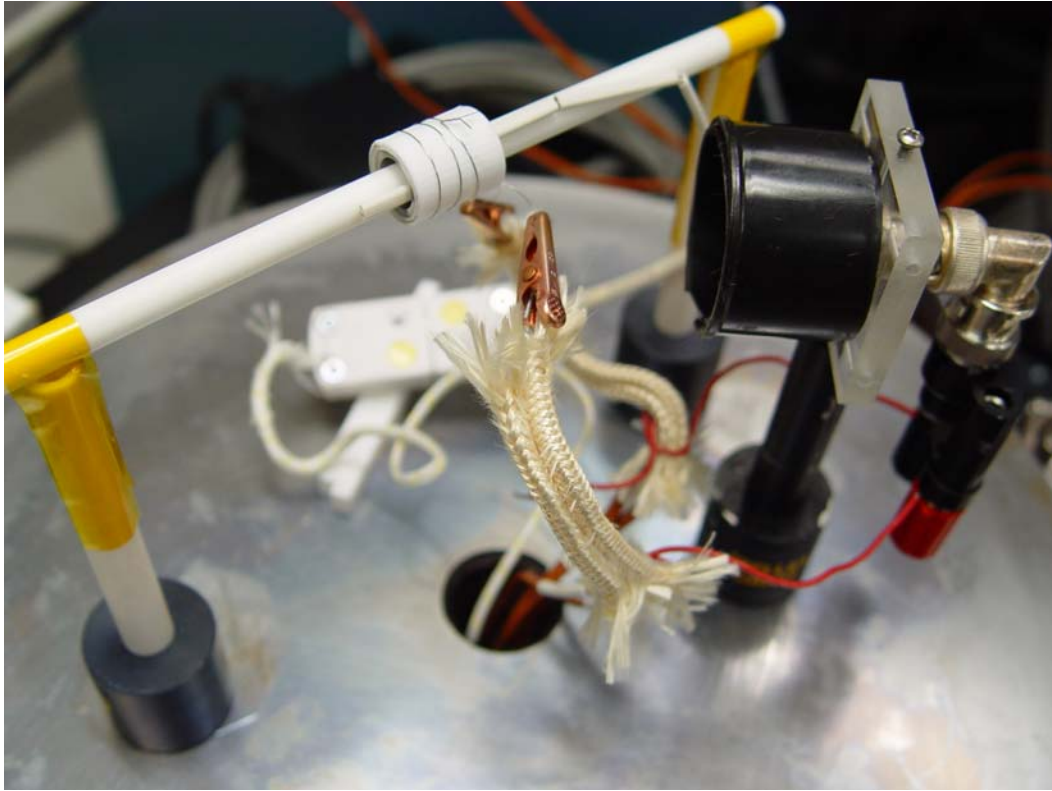


Figure 3. Mounted scrubber cartridge containing heater element ready for testing.

### Test Results

A total of twelve scrubber cartridges were tested in this facility. The unit 2 test was terminated before failure at 450 cycles when the SAM Project determined a mission life of 31 cycles. Under direction from the Project life testing resumed to a minimum 2x expected life which was 62 cycles. Seven of the scrubber cartridges were tested to 2x mission life. The remaining units were tested to failure in order to understand the failure mechanism and to calculate reliability. Table 1 summarizes the furnace cycling data.

Table 1. Scrubber cartridge cycling data.

Scrubber test no.	Cycles completed	Scrubber status at completion
1	437	Failed
2	450	OK
3-9	62	OK
10	720	Failed
11	864	Failed
12	762	Failed

A plot of element resistance at the end of each cycle before the current was switched off as a function of cycles is shown in Figure 4. Data for units 11 and 12 were lost due to computer hardware failure. For simplification the curves were normalized to the average of the first data point collected for each unit. A wire lead in scrubber unit 2 broke during setup so the resistance of the curve is lower by approximately  $0.8\Omega$  corresponding to the loss of about 13mm of wire. It is clear that the resistance of the element increases as the element is cycled. The data indicates that the resistance of the element at  $1000^{\circ}\text{C}$  increases on average  $1.9\% \pm 0.9\%$  from the first cycle to the 31<sup>st</sup> cycle. Power dissipation in the element during the first cycle was  $61 \pm 2$  watts. At a constant electrical current of 1.3 amperes the power dissipation also increased approximately 2% from the first cycle to the 31<sup>st</sup> cycle. These observations impacted the design of the heater circuit. It is important to point out that verification of the element temperature with optical pyrometry eliminated the possibility of using thermal shielding. Operating the heater element at  $1000^{\circ}\text{C}$  in this configuration required 45% more power than the maximum allotted for flight operation and insured a conservative test because the element would carry a higher current density and therefore would be more susceptible to failure if local reductions in cross section were to occur.

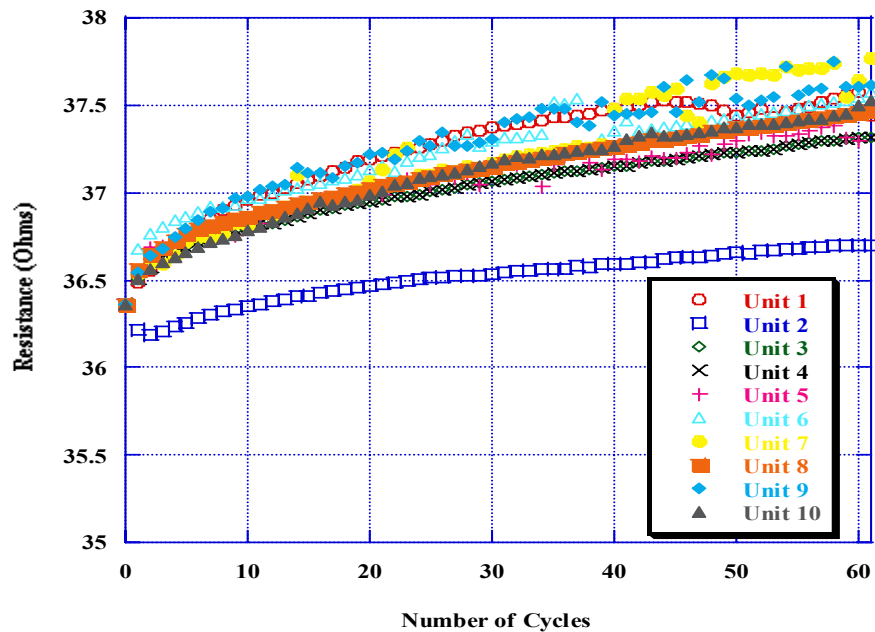
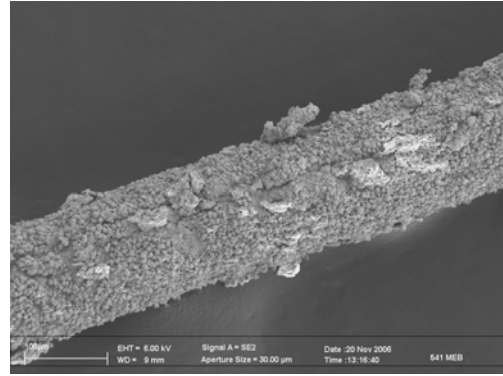


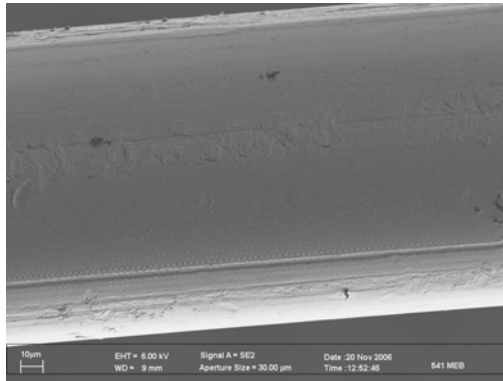
Figure 4. Element resistance for each cycle just before current is switched off.

The increase in resistance of the element with cycling probably results from a reduction in the metallic cross section of the wire due to oxidation of the wire surface. Figure 5 shows a comparison of the wire in the as-received condition and the test wire after 266 cycles when the test was terminated. Images (a) through (c) are secondary electron images (SEI) of the wire surface. Image (b) and (c) are as-received and after 266 cycles, respectively, both taken at the same magnification. It is clear that significant oxidation of the wire surface has occurred. Optical images (d) and (e) are longitudinal sections while images (f) and (g) are transverse sections.

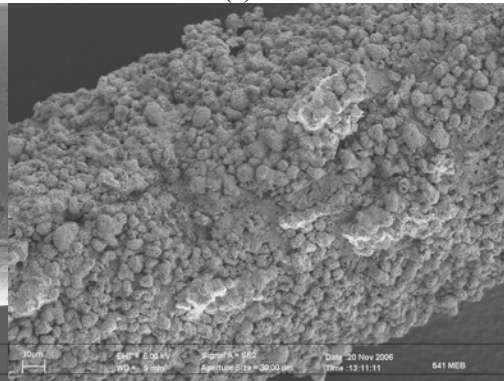
Figure 5. Comparison of as-received wire and test wire after 266 cycles. a) SEI of surface after 266 cycles, b) and c) SEI pre- and post test respectively, d) and e) optical image of longitudinal section, f) and g) optical image of transverse section. Optical images have same magnification.



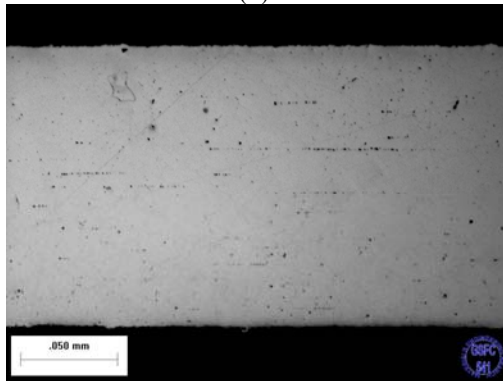
(a)



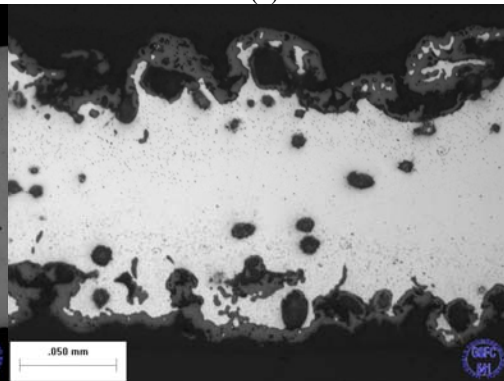
(b)



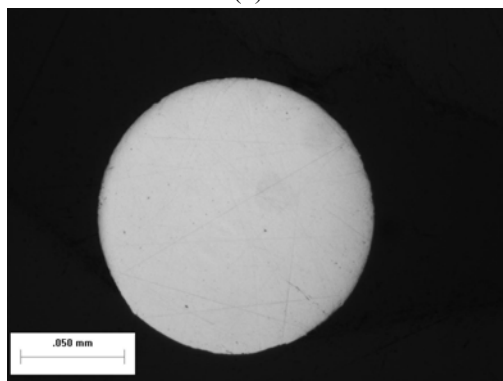
(c)



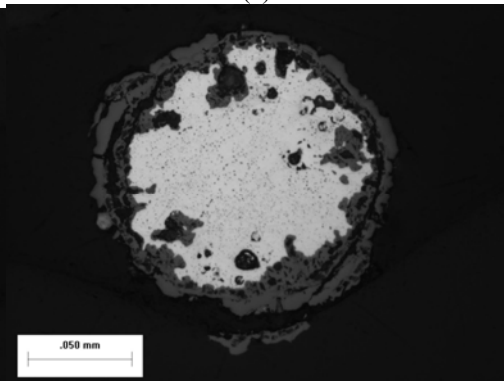
(d)



(e)



(f)



(g)



Both sets of images show the reduction in metallic volume as the surface oxidizes and the oxide spalls with thermal cycling. It is interesting to note that the room temperature length of the wire increases continuously by a factor of approximately 1.06 times its initial length of 50.8 cm  $\pm$  1.3 cm up to around 500 cycles where the growth appears to stop. This 6% increase in length is probably due to the stiff oxide restricting the wire from contracting as it cools when the current is switched off. The metallic part of the wire then creeps to relax the tensile stresses. When the current is switched on the wire heats up and expands cracking the oxide which exposes a fresh metallic surface for oxidation. In this way each cycle incrementally adds length to the wire.

Radiance from the heater element as monitored by the response of a solar cell was nearly constant after the first 15 cycles. Changes in the level of solar cell current during the first 15 cycles seems to correlate with vacuum level and is probably associated with changes in emissivity of the element under slightly different oxidation conditions. A plot of normalized solar cell response as a function of number of cycles is shown in Figure 6. The variations in absolute solar cell current did not correlate with thermocouple response and are due to slight differences in distance between the solar cell and the scrubber.

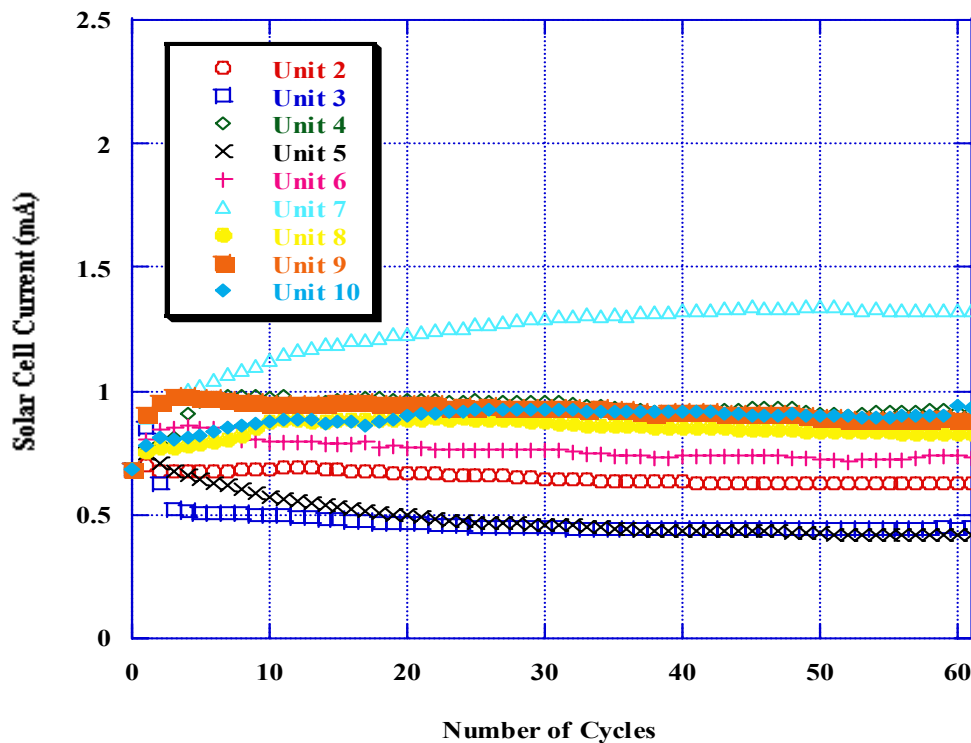


Figure 6. Solar cell current for each cycle just before current is switched off.

## RELIABILITY AND HEATER LIFETIMES

For the instrument system reliability assessment, heater lifetimes could be predicted using lifetime data from similar heaters or from component and materials physics of failure models. The SAM CO<sub>2</sub> scrubber heater element application presented several caveats to the usual

predictive methods: the heaters were designed uniquely for this space instrument application so no similar device existed for comparison and no test data existed for cold worked 0.0142 cm diameter 80Ni-20Cr alloy wire operated cyclically to high temperature in rarefied CO<sub>2</sub>. Eleven heaters were tested following the “Test Like You Fly – Fly Like You Test” approach (ref. 3), i.e., testing heaters resembling the flight articles in a simulated Martian atmosphere. These life tests were used to determine the *demonstrated* reliability.<sup>1</sup>

Seven units were tested to twice the expected 31-cycle mission life without failure, one to 450 cycles without failure, two to failure at 437 and 720 cycles, and two additional units to failure at 762 and 864 cycles. Weibull analysis<sup>2</sup>, useful for providing reasonably accurate lifetime predictions with small samples (ref. 4), was used to analyze the data. Analysis results, shown in Figure 7, indicated early wearout failures ( $\beta = 3.4$ ), consistent with the expected wire failure mode, and a characteristic life of 792 cycles. This characteristic life is the time, in cycles, when 63% of the population (samples) has failed<sup>3</sup>.

The resulting Weibull parameters allow a determination of heater reliability, that is, the probability of success for the 31-cycle mission requirement under the simulated Martian atmosphere. Life test data uncertainty is provided by a lower 95% confidence bound on fitted Weibull distribution.<sup>4</sup> Demonstrated heater reliability for the required thirty-one cycles is 0.9985 at a 95% lower confidence. This nearly “three nines” reliability for the CO<sub>2</sub> scrubber heater was considered sufficient for the SAM Instrument mission.

## CONCLUSION

Based on the principle that all GSFC missions shall follow a “Test Like You Fly – Fly Like You Test” approach, a test facility was constructed to determine the reliability of heater wire used for regeneration of the QMS CO<sub>2</sub> scrubbers. The heater wire, an 80Ni-20Cr alloy that was 0.0142 cm in diameter, was tested inside flight-like scrubber cartridges so that the effects of cold working in the wire due to assembly could be evaluated together with cycling to high temperature in a simulated Martian atmosphere. Testing twelve scrubber cartridges yielded a reliability of 0.9985 at a lower 95% confidence level for a 31 cycle (1x) mission life. This high reliability developed through testing faithful to the flight environment was sufficient to insure successful operation of the GPS for the SAM instrument suite.

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<sup>1</sup> *Demonstrated* reliability means that derived through either life tests (per this work) or in-service usage. It is so stated to distinguish it from a predicted reliability derived, for example, from *MIL-HDBK-217*. Demonstrated reliability is more credible than a predicted reliability if the test or service environment is the same as the intended usage environment.

<sup>2</sup> For those unfamiliar with Weibull analysis, failures are plotted on natural logarithmic scales,  $\ln t$  for the ordinate and  $\ln \ln [1/[1 - F(t)]]$  for the abscissa, where  $F(t)$  is the cumulative fraction of failures at time  $t$ . Un-failed units (called censored or suspensions) are accounted in the failure probability computations but not plotted. Data fitting linearly indicates the Weibull distribution is applicable. The slope ( $\beta$ ) characterizes failures as infant mortal, random (constant failure rate), or wearout.

<sup>3</sup> Characteristic life is *not* a mean-time-to-failure, except when  $\beta = 1.0$ .

<sup>4</sup> A lower bound (only) was chosen because the requirement is for a minimum lifetime.

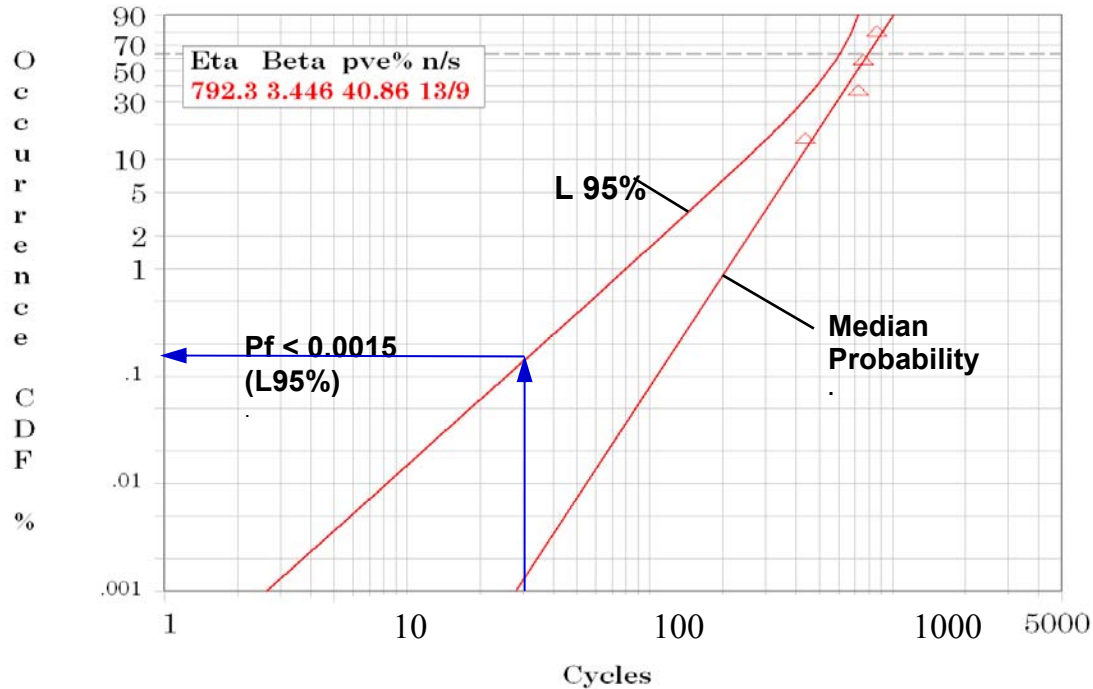


Figure 7. Weibull plot of SAM CO<sub>2</sub> scrubber heater life tests. The upper curve is a lower 95% confidence bound on the failure distribution. Blue arrows indicate how a heater failure probability estimate is determined; precise numbers are extracted from the statistical software.

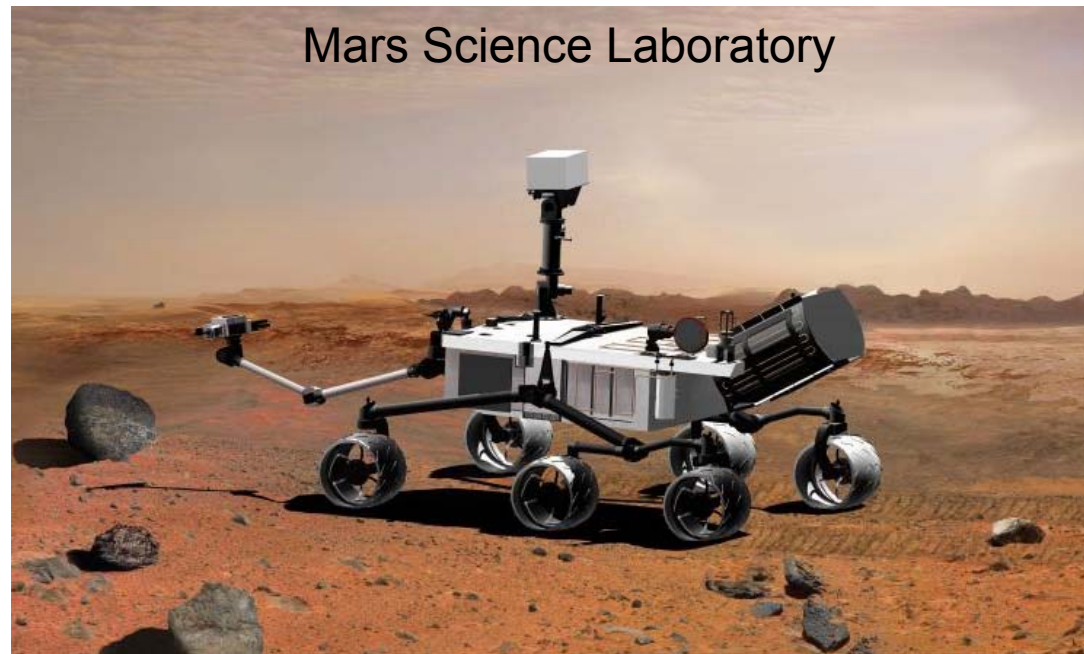
## ACKNOWLEDGEMENTS

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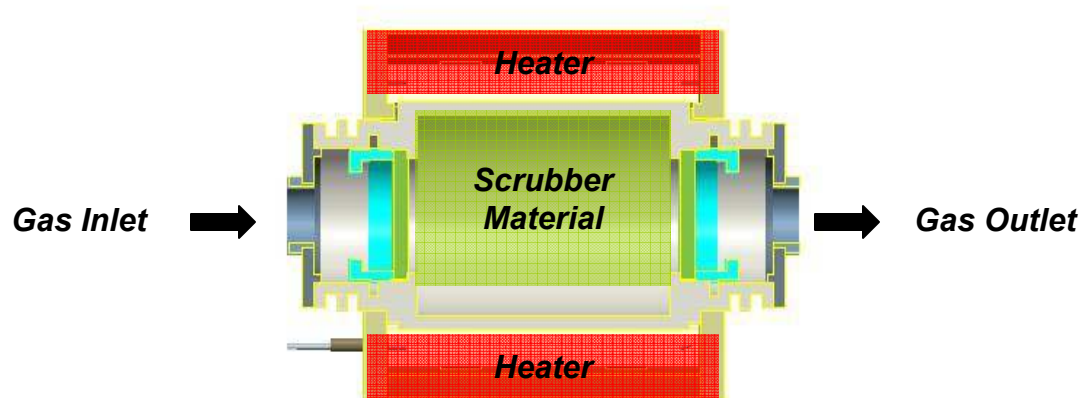


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Flight Center

# *Introduction*

- Test the suitability of 80Ni-20Cr wire, 0.0056 inches in diameter, for use as a heater element for the carbon dioxide scrubber in support of the GPS for the SAM instrument suite built by GSFC.
  - The heater element in the carbon dioxide scrubber is used to regenerate the scrubber material as part of the gas processing system. This figure is an illustration of the gas flow through the scrubber unit.



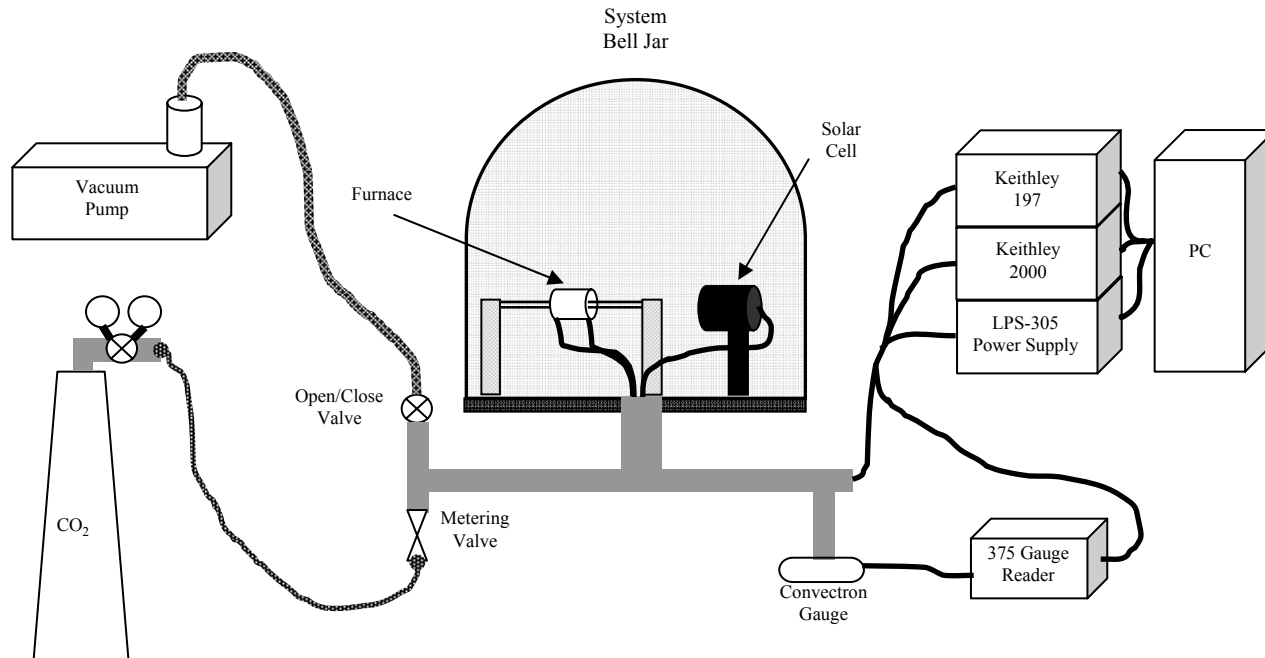
# ***Introduction (cont.)***

- The wire would be required to operate at 1000°C in order to attain the 800°C required for regeneration of the getter.
- The wire would need to operate in the Mars atmosphere, which consists mostly of CO<sub>2</sub> at pressures between 4 and 12 TORR.
  - Data on the high temperature degradation mechanism of 80Ni-20Cr in low pressure CO<sub>2</sub>, together with the effects of thermal cycling, were unknown.
  - The influence of work hardening of the wire during assembly and the potential for catastrophic grain growth also were unknown.
- Verification of the wire reliability as defined by the mission goals required the construction of a life test facility that would accurately simulate the duty cycles in a simulated Mars atmosphere.

# **Test Objectives**

- Keys to executing an accurate life test:
  - Ensure the operating temperature of the wire was as close as possible to the required 1000°C.
  - Maintain a relatively low pressure (7.5 – 8 TORR) CO<sub>2</sub> environment that simulates exposure to the Mars atmosphere during the life test.
  - Automate the life cycle test and monitor the performance of the wire.

# Life Test Facility

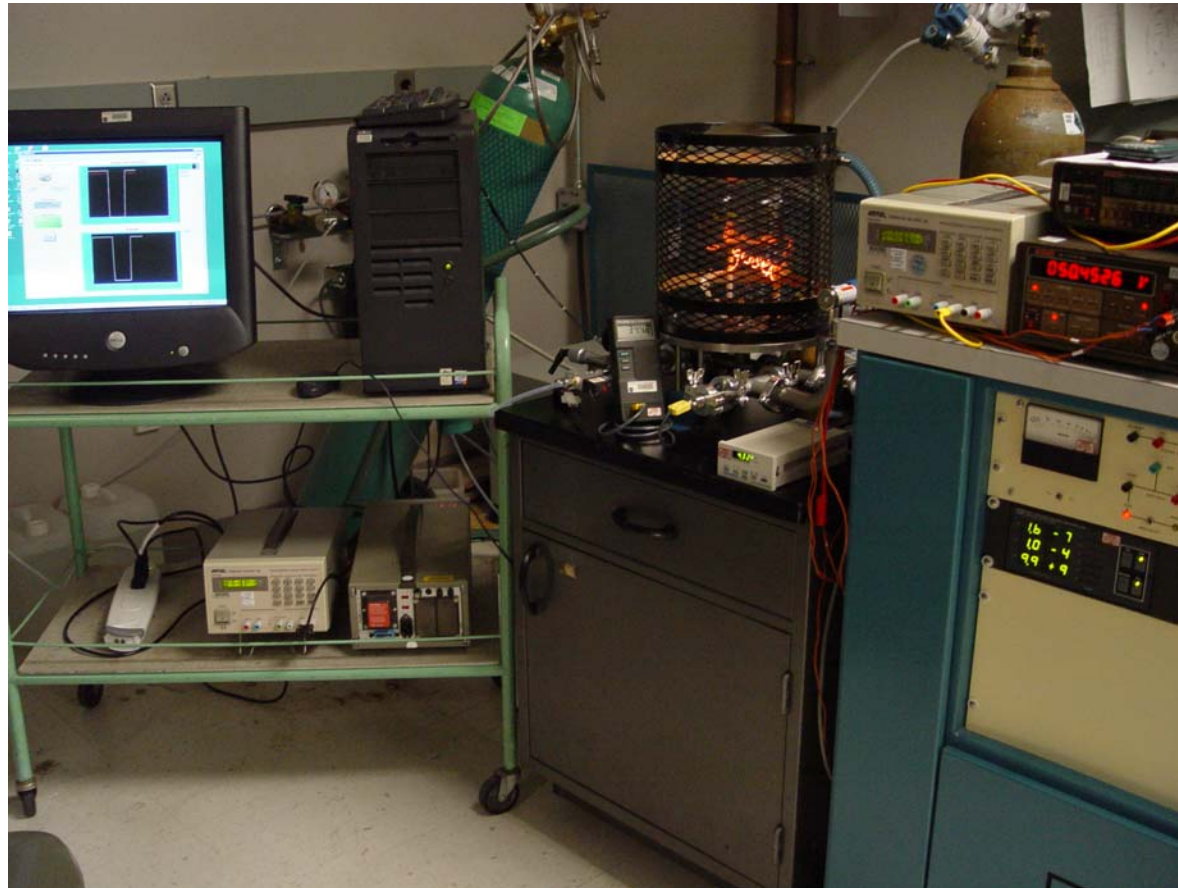


- Simulated Mars atmosphere was achieved with the use of a mechanical vacuum pump combined with a metered flow of CO<sub>2</sub>.
  - Metered flow of CO<sub>2</sub> in combination with the vacuum pump maintained a system pressure at approximately 7.5-8.0 TORR.
  - Software monitored simulated atmosphere performance and disabled wire testing if the system pressure exceeded test limits.



# Data and Control System

- LabVIEW VI performed all of the data acquisition and storage functions as well as limit testing and control functions
  - Control function cycled the furnace on and off at a constant current.
  - The pressure limit checking prevented power cycling of the furnace in an atmosphere that did not meet test objectives.
  - The data from the test setup consisted of: Date, Time, Elapsed Time, Cycles, Voltage, Current, Resistance, Solar Cell Current, Pressure, as well as temperature data from three thermocouples.
  - Resistance of the wire was used to estimate the wire temperature to verify that the flight condition of 1000°C was achieved. This was confirmed using an optical pyrometer.



# Test Procedure



- Current was applied to the heater wire and adjusted until the measured voltage across the wire produced a resistance indicating a wire temperature of 1000°C (as predicted by the temperature coefficient of resistance).
- Testing proceeded with flight-like CO<sub>2</sub> scrubber units which were powered by a current source programmed with a period of 20 minutes and a duty cycle of 50%, resulting in three complete on-off cycles per hour.

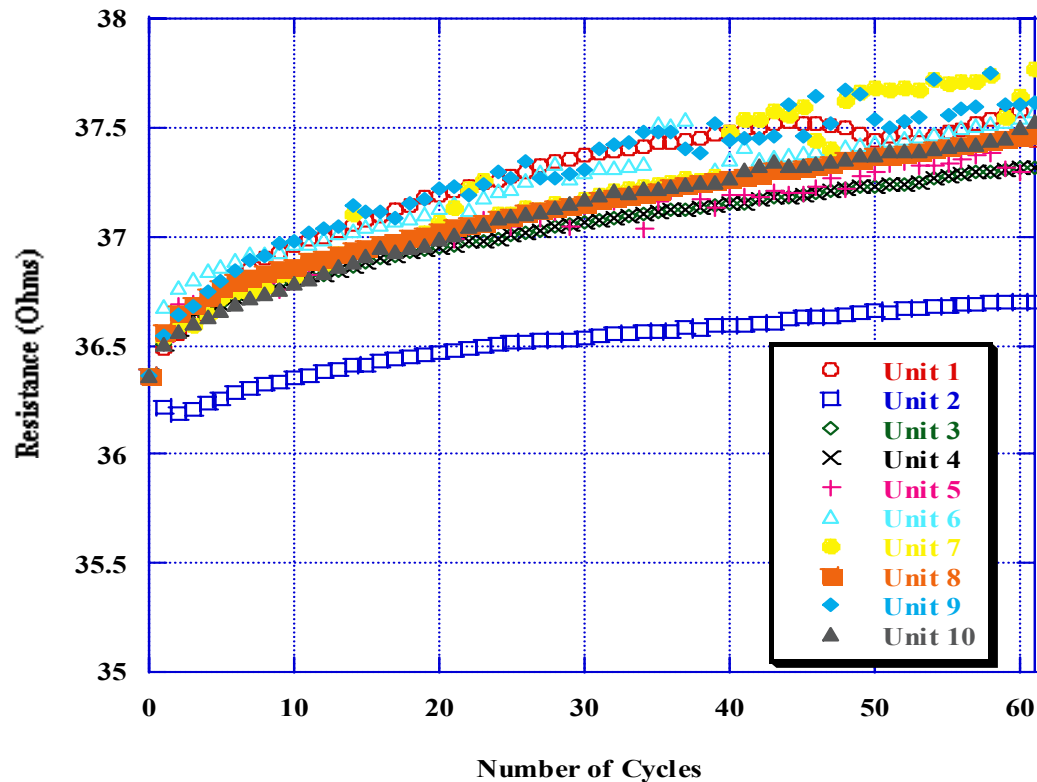
# Test Results

- Twelve scrubber cartridges were tested in this facility.
- Under direction from Project seven scrubber cartridges were tested to 2x mission life. Remaining units were tested to failure in order to understand the failure mechanism and to calculate reliability.

Scrubber Test Number	Cycles Completed	Scrubber Status at Completion
1	437	Failed
2	450	OK
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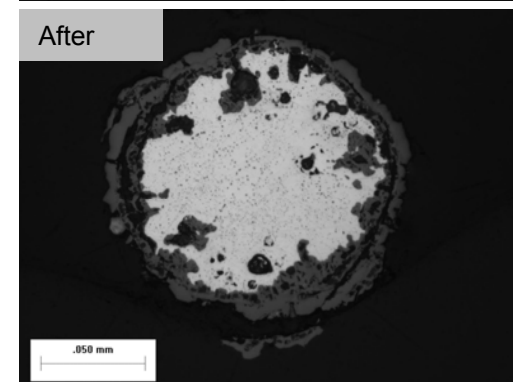
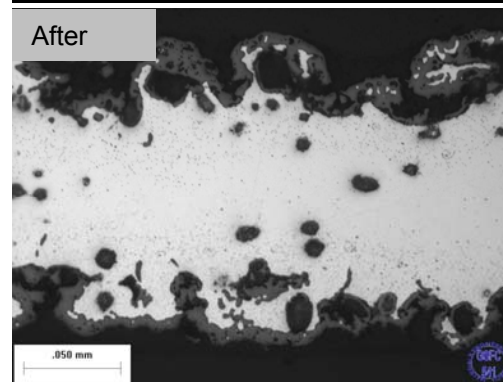
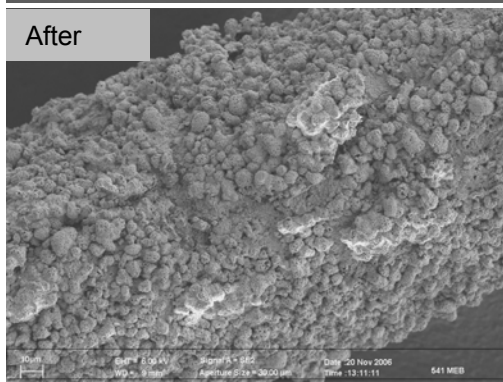
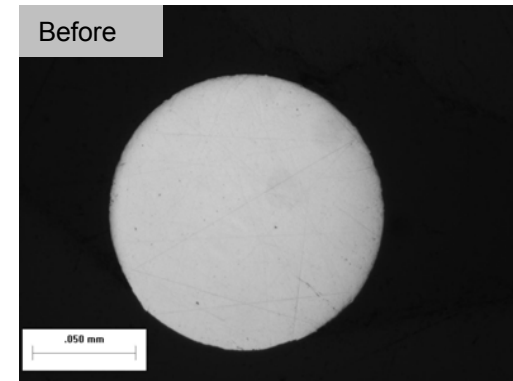
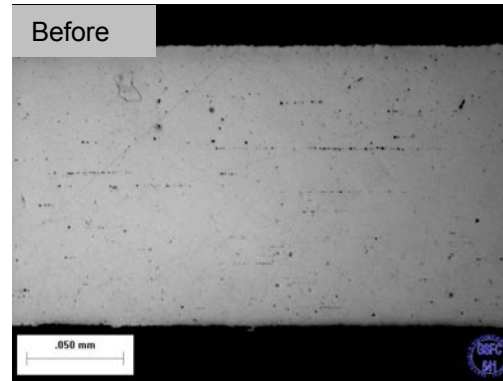
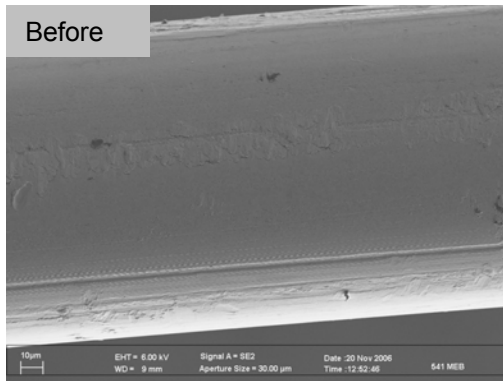
# Test Results (cont.)

- Resistance data indicates that the resistance of the wire at 1000°C increases on average 1.9%  $\pm$  0.9% from the first cycle to the 31st cycle. Power dissipation in the wire during the first cycle was 61  $\pm$  2 watts. At constant current the power dissipation would also increase approximately 2% from the first cycle to the 31st cycle. These observations impacted the thermal performance of the design.



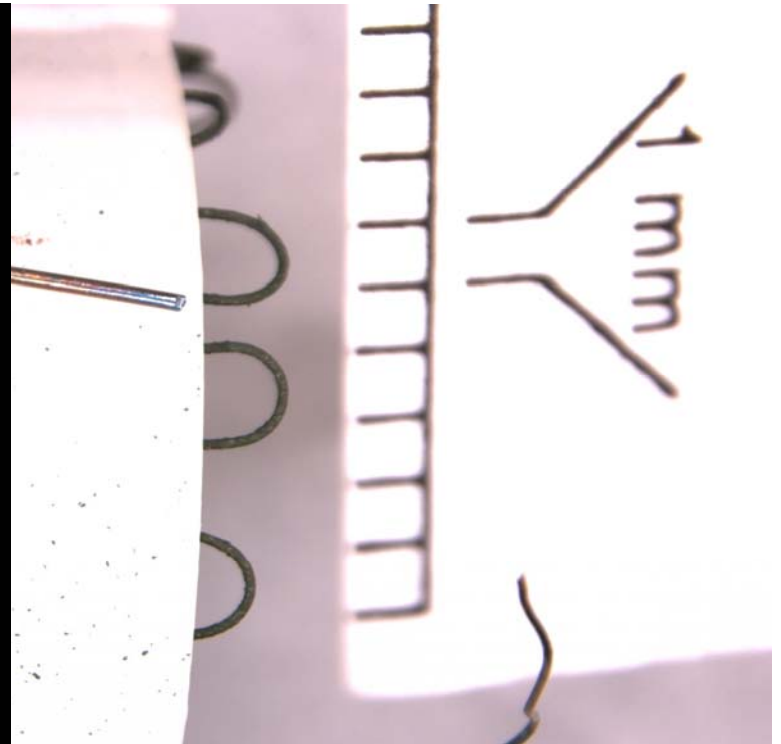
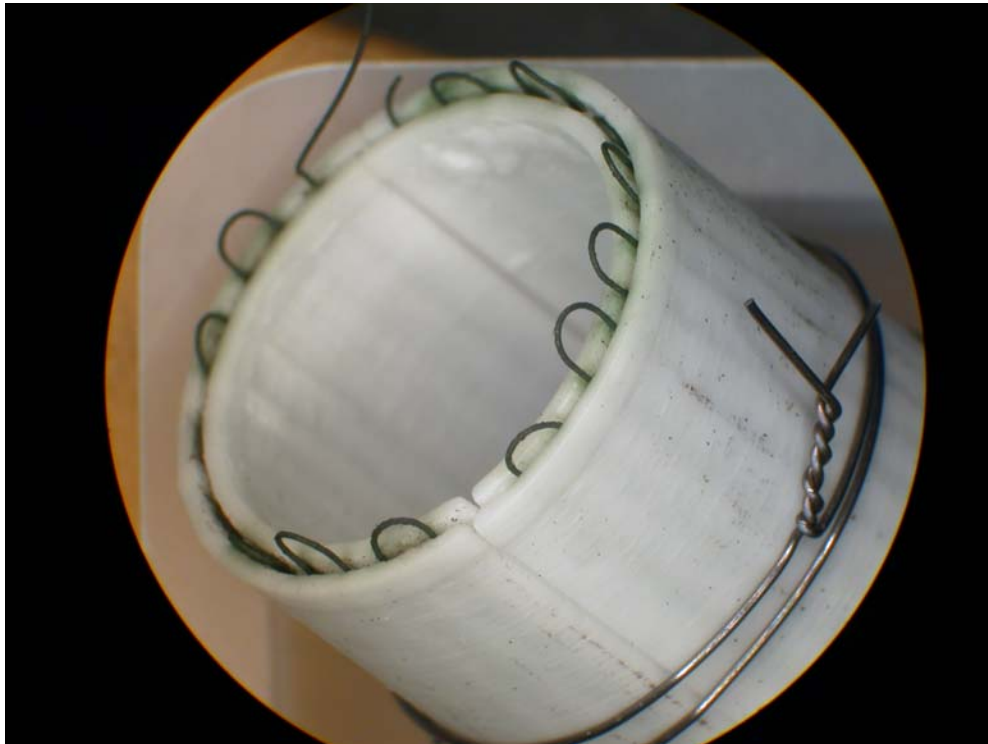
# Test Results (cont.)

- As cycling progressed, electrical resistance increased due to a reduction in cross section of the wire due to oxidation of the wire surface.
- Figures below show comparison of the wire in the as-received condition and after 266 cycles on a test wire sample.
  - Significant oxidation of the wire surface has occurred.
  - Images show the reduction in metallic volume as the surface oxidizes and the oxide spalls with thermal cycling.

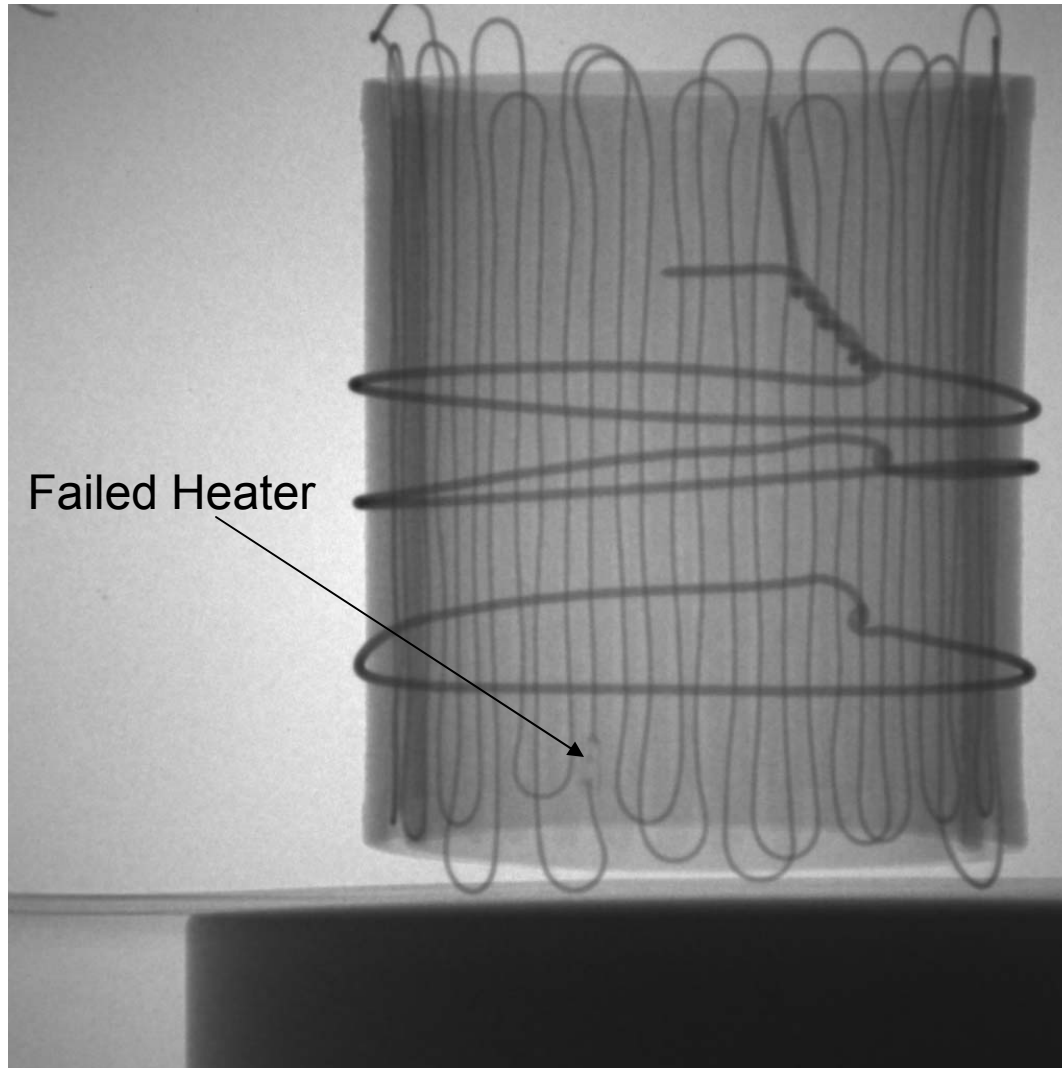


# Test Results (cont.)

- Room temperature length of wire increases continuously to approximately 6% of its initial length up to around 500 cycles where the growth appears to stop.
  - Growth probably due to the stiff oxide restricting the wire from contracting as it cools when the current is switched off. The metallic part of the wire then creeps to relax the tensile stresses. When the current is switched on the wire heats up and expands cracking the oxide which exposes a fresh metallic surface for oxidation. In this way each cycle incrementally adds length to the wire.



# *Results (cont.)*



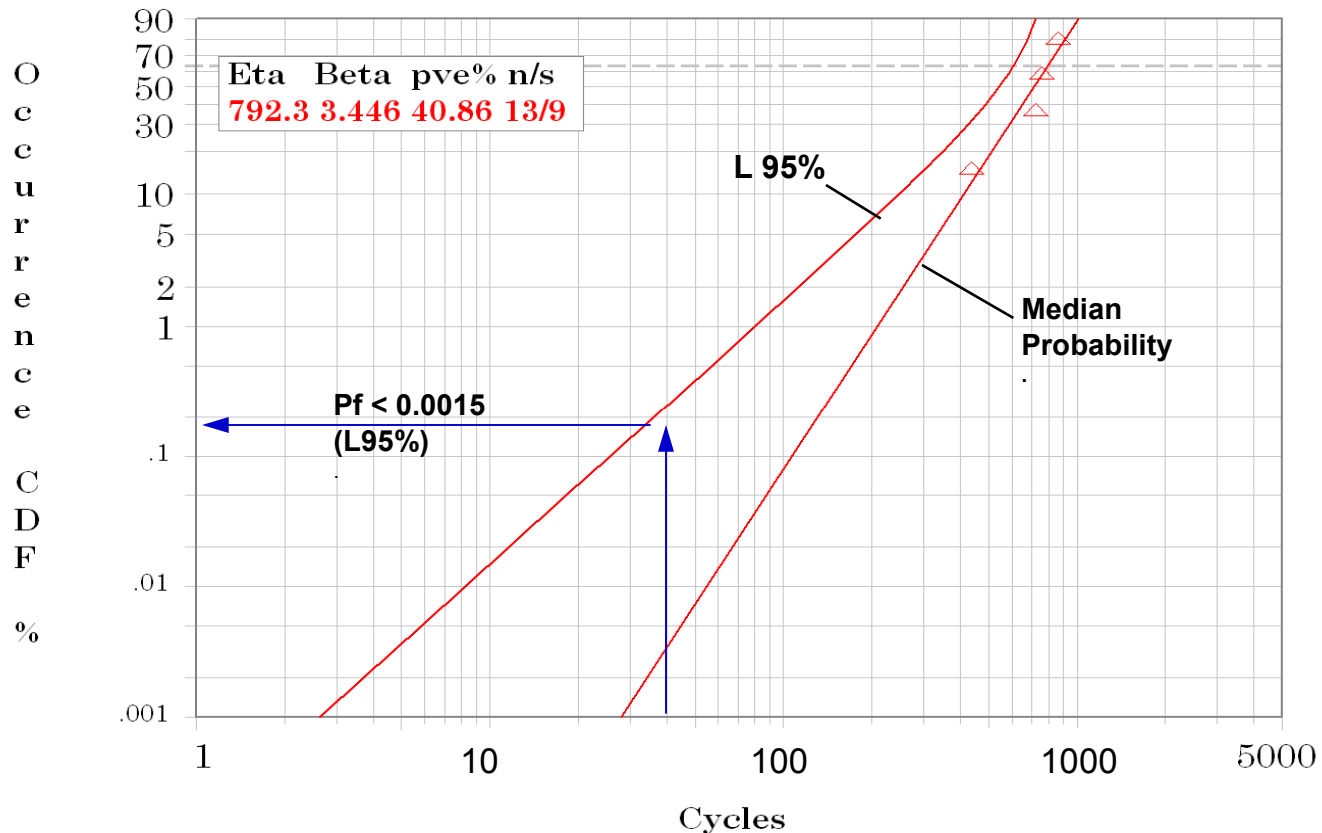
# **Reliability**

- Weibull analysis, useful for providing reasonably accurate and conservative lifetime predictions with extremely small samples, was used to analyze the data.
- Heater reliability derived from the life tests yielded a heater reliability of 0.9985 (at a lower 95% confidence) at 31 cycles.
- This nearly “three nines” reliability for the CO<sub>2</sub> scrubber heater wire was considered sufficient for the SAM Instrument mission.



# Reliability (cont.)

Below is a Weibull plot of SAM CO<sub>2</sub> scrubber heater life tests. The upper curve is a lower 95% confidence bound on the failure distribution. Blue arrows indicate how a heater failure probability estimate is determined; precise numbers are extracted from the statistical software.



# *Conclusions*

- Successful life testing in a simulated Mars atmosphere.
- Performance of alloy 80Ni-20Cr wire now documented in a simulated Martian atmosphere, metallurgical effects now better understood.
- All units passed 2x mission life.
- Reliability data analysis shows high confidence in reaching mission of 31 cycles to a probability of 0.9985.