RELIABILITY OF HIGH-TEMPERATURE METALLIC COMPONENTS IN SORPTION CRYOCOOLERS[1]

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ABSTRACT

Dependable long-term performance of sorption cryocoolers necessitates using sorbent materials, heaters, and pressure vessels designed to retard or eliminate identified failure mechanisms. During a 10-year mission, heaters and container materials experience approximately 175,000 thermal and pressure cycles [260K to 773K, $2x10^3$ N/m^2 to $2.6x10^6$ N/m^2 (-13°C to 500°C, 0.3 psia to 382 psia)]. The high temperature creep and fatigue requirements associated with these environments are being successfully met through the use of high temperature nickel-chromium alloys -- namely, Nichrome for the cartridge heaters and Inconel for the container materials. This paper describes details of ongoing research at JPL to understand and confirm the reliability of these nickel-chromium alloys for sorption compressor applications.

An understanding of degradation mechanisms for both container vessels and heaters is derived from interpreting data from static and cyclic accelerated temperature/pressure tests both in oxygen-free and oxygen-rich environments, and from developing and studying structural and thermal computer verification models and life-prediction algorithms. High temperature cyclical operation appears to result in longer service life than does continuous operation. Service life correlates well with an Arrhenius temperature dependency -- and appears entirely consistent with 10-year-life sorption refrigerators.

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1. INTRODUCTION

1.1. BACKGROUND

Sorption refrigeration systems exploit temperature-dependent adsorption/desorption properties of a gas by a solid sorbent. The sorbent, contained in a cylindrical canister, is alternately heated and cooled to desorb and readsorb the working fluid. During the compressor heating phase, the pressure of the evolved sorbate gas increases until it exceeds the system high pressure, at which time an outlet check valve opens, allowing the high pressure gas to flow into the high pressure lines. The high pressure gas flows through a conventional Joule-Thomson (J-T) expansion cycle. Meanwhile, another sorbent bed is being cooled causing the canister pressure to drop below the system low pressure, at which time an inlet check valve opens, allowing the low pressure gas to enter the canister and be readsorbed by the sorbent.

Because sorption refrigeration systems feature few moving parts and hence minimum vibration, they are expected to possess a longer service life than typical mechanical refrigeration systems. However the need to operate the compressors at relatively high temperatures [typically 523K to 773K (250°C to 500°C)] imposes certain reliability requirements on the system, particularly on the heat source (usually an electrical cartridge heater) and the containment vessel.

Fig. 1 illustrates a representative three-stage carbon/krypton (C/Kr), carbon/xenon (C/Xe) and praseodymium-cerium-oxide/oxygen (PCO/O_2) compressor configuration currently under investigation at the Jet Propulsion Laboratory. The three stage compressor is designed to drive a three stage 65K to 120K to 165K cryocooler. The C/Xe and C/Kr compressors each undergo temperature cycles between 260K and 523K, and pressure cycles between 1.01×10^5

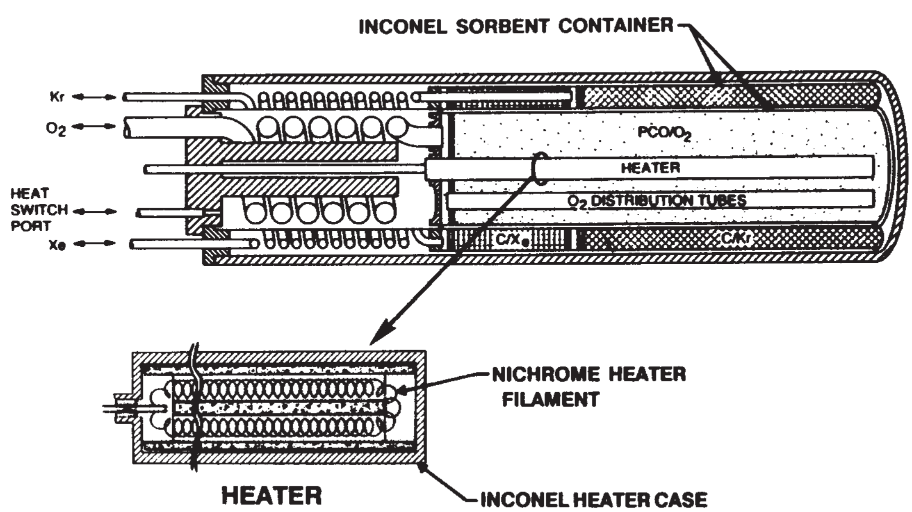


Fig. 1. Concentric regenerative compressor configuration

N/m^2 and 25.9×10^5 N/m^2 and between 1.03×10^5 N/m^2 and 14.3×10^5 N/m^2, respectively.

The heater and PCO containment vessel undergo temperature cycles ranging from 553K to 773K and pressure (stress and/or strain) cycles ranging from about 2.3×10^3 N/m^2 to 11.8×10^5 N/m^2. The cycle time is typically 30 minutes.

1.2 RELIABILITY ISSUES

Oddly enough, some of the major reliability considerations in sorption cryocoolers must focus on high temperature and high pressure components: the compressor walls and the cartridge heater. A conventional strategy of high temperature, high pressure component design is to provide ample safety margin. For example, thick compressor walls and heavy gauge heater elements will significantly alleviate most reliability concerns regarding these components. However, as pointed out in a sorption refrigeration system study, Ref. [1], the efficiency of a sorption compressor decreases as its thermal mass increases. An ideal sorption compressor would operate at relatively high temperature, would have minimum container and heater mass, and would yet be capable of providing sufficiently long service lifetime (5 to 10 years).

Other reliability issues in sorption cryocooler design concern sorbent performance degradation, contaminant corrosion, clogged check valves and J-T valves, etc. These and other issues are addressed in Refs. [1] and [2].

Identifying and describing the degradation mechanisms of metal containment materials undergoing simultaneous pressure and temperature cycling is challenging. This is especially true in a corrosive environment (e.g. high pressure oxygen). Creep, fatigue, scale formation and spalling, and crack formation have been identified as potential degradation mechanisms.

On the other hand, the mechanisms underlying heater element failure are much less apparent, as the final melting of the damaged element obscures the details of transitional metallurgical changes. Heater elements are known to be susceptible to fatigue, corrosion, and erosion damage that is accelerated by high temperature.

Although the failure mechanisms of heaters and containers may differ, the study of their reliability reveals recognizable parallels. Firstly, the high temperature alloys for both applications exhibit similar response characteristics to stress and corrosive environments. Secondly, for both components, the useful service life correlates with operating temperature through the well-known Arrhenius law. And again, the system requirements imposed on these components and the logical strategies for addressing the reliability issues are virtually identical.

Life testing at the systems level is useful to reveal systems-related failure modes and mechanisms and long-term wear. However, system reliability can not be assured by life-testing, because demonstration of full service life would take 10 years. A more practical approach is to also utilize accelerated testing of components and the development of analytic life-prediction techniques.

The main emphasis in the present investigation is thus focused on the development, based upon accelerated tests, of quantitative engineering correlations that can be used for accurate service-life prediction. A set of relevant experiments designed to validate the correlations and the resultant life predictions are described.

2. CHARACTERISTICS OF HIGH TEMPERATURE MATERIALS

The thermo-mechanical behavior of various metallic materials exhibits similar characteristics at temperatures T that are in fixed proportion to their respective melting temperatures, T_m. This ratio, $T_h = T/T_m$, is called the homologous temperature. For example, when a metal is heated above $0.4T_m$, residual stress relaxation and recrystallization rapidly occur. Elevated temperature operation is said to occur for $T_h > 0.5$. Elevated temperature behavior is noted at approximately 478K (205°C) for aluminum alloys, 588K (315°C) for titanium alloys, 643K (370°C) for low-alloy steels, 813K (540°C) for austenitic, iron-based alloys, 923K (650°C) for nickel based and cobalt-based alloys, and 1253K to 1813K (980°C to 1540°C) for refractory metals and alloys.

High temperature alloys such as Inconel (container vessel) and Nichrome (heater element) share many similarities in both metallurgical and thermo-mechanical behaviors. The melting temperatures of Inconel and Nichrome alloys range from 1623K to 1673K (1350°C to 1400°C). A heater operating temperature of 773K (500°C) corresponds to a homologous temperature around 0.5. Similarly, the Inconel container vessel at 773K corresponds to a homologous temperature of about 0.5.

The tensile properties of most engineering metals at low temperatures are time-independent. When the applied stress exceeds yield strength, the total strain consists of an elastic component and a plastic component. When the homologous temperature exceeds 0.4, the material becomes ductile in the so-called "hot working" range and creep can occur under external load or displacement as a time-dependent process. In other words, the strain rate is now governed by stress and temperature, with additional dependencies on grain structure and other metallurgical considerations. Fig. 2 illustrates the reported creep rates, Ref. [3], for annealed Inconel 625 as a function of applied stress level at 1088K ($T_h = 0.67$). The steady-state creep rates can be expressed by the so called "strain-rate hardening" equation, which exhibits a power law sensitivity to stress

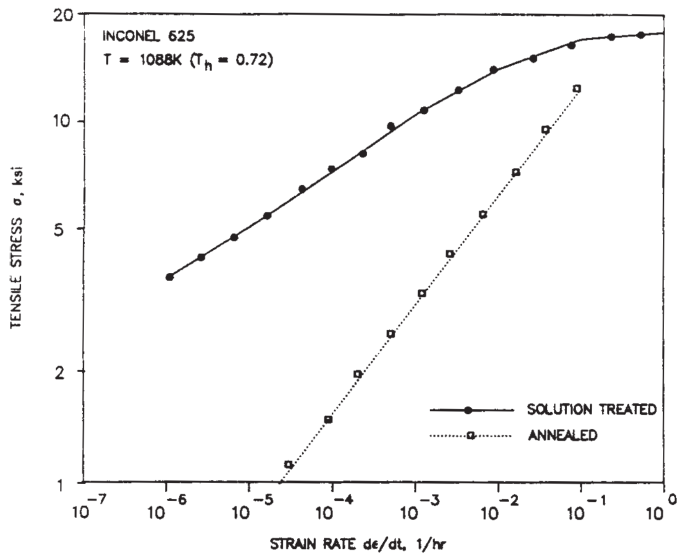


Fig. 2. Tensile strain-rate hardening for Inconel 625 Ref. [3]

level and an Arrhenius dependency on operating temperature:

$$d\epsilon/dt = B\,\sigma^n \exp(-Q/kT) \qquad (1)$$

where

- ϵ is the strain
- B is a proportionality constant
- Q is the activation energy for creep
- σ is the applied stress
- k is the Boltzmann constant
- T is the operating temperature, K

3. COMPRESSOR RELIABILITY

Two concerns dominate compressor vessel reliability -- (1) chemical interactions between the PCO/O_2 and the thin (< 10 mil) Inconel 625 wall that can pit and/or scale it, and (2) structural/metallurgical changes in the Inconel 625 that can be induced by the cyclic temperature/pressure, pure oxygen environment. Temperature/pressure cycling in the compressor ranges between 553K/2.3×10^3 N/m^2 and 773K/1.18×10^4 N/m^2 (285°C/0.3 psia and 500°C/175 psia). Active failure mechanisms may include creep- and/or fatigue-induced fracture and creep racheting.

The test apparatus is shown in Fig. 3. The compressor wall test specimens are 6.99 cm (2.75 inch)-long cylindrical Inconel tubes with the central 1.9 cm (0.75 inch) "test section" ground to an 0.2-0.3 mm (8-12 mil) wall thickness. The individual samples are welded together end-to-end and attached to a gas feeder and support tube. This entire assembly is horizontally mounted to a vertical support wall.

Gas bottles, valves, and plumbing allow the cantilevered sample assembly to be internally pressurized or evacuated (pressure cycling). Likewise, a tube furnace can be moved onto and off of the assembly to provide temperature cycling. Pressure and temperature cycling are controlled by a Minarik Micro-Master controller, which permits both static and cyclic testing.

The actual experiments that were performed are listed in the accelerated test matrix, Table 1.

Combining analysis and experiment, the current effort to understand compressor degradation mechanisms aims at interpreting accelerated test data from static and cyclic temperature/pressure tests both in oxygen-free and oxygen-rich environments, and at developing and studying structural and thermal

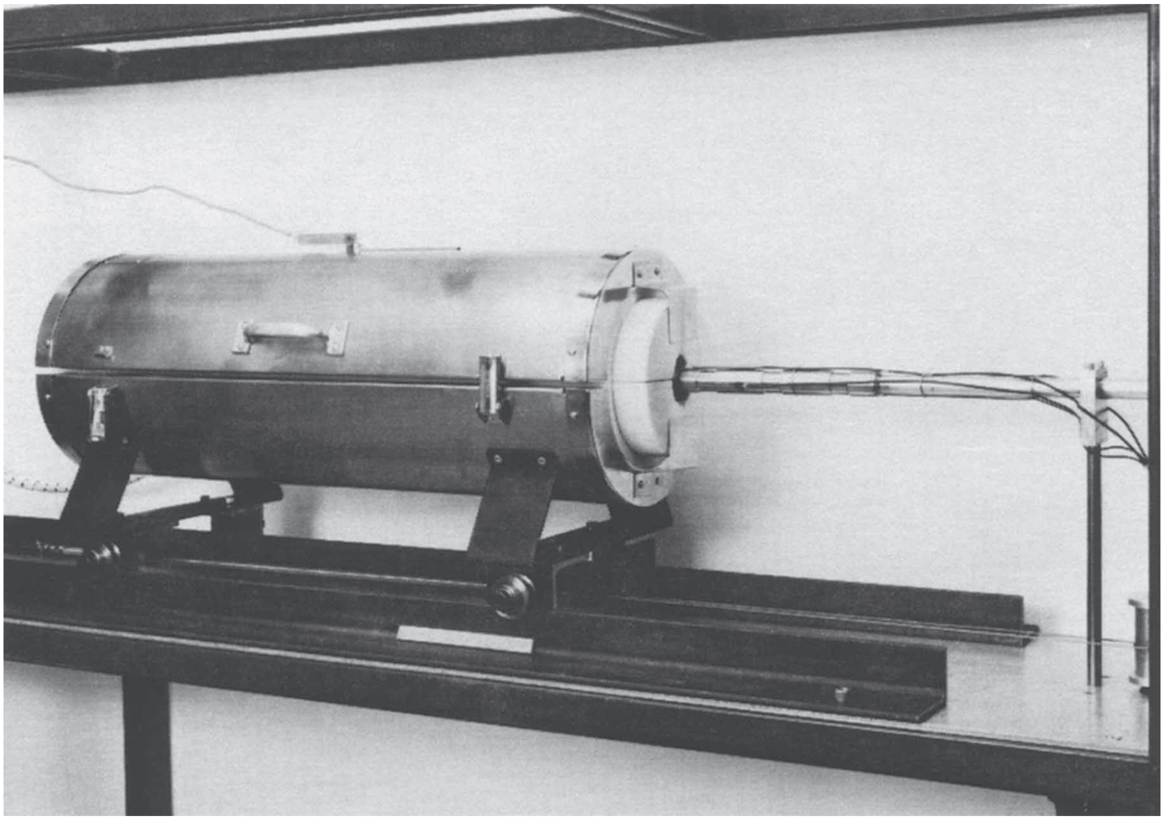


Fig. 3. Test apparatus for Inconel 625 cylindrical samples

TABLE 1.

INCONEL CONTAINER MATERIAL
ACCELERATED TEST MATRIX

(0.010" Wall x 1" Diameter Specimens)

Static Creep			
923K	5.5×10^6 N/m^2	Argon	constant stress
973K	5.5×10^6 N/m^2	Argon	constant stress
1023K	5.5×10^6 N/m^2	Argon	constant stress
Combined Creep, Fatigue, Oxidation			
973K	5.5×10^6 N/m^2	Oxygen	30 minute cycles
973K	6.2×10^6 N/m^2	Oxygen	30 minute cycles
1023K	6.9×10^6 N/m^2	Oxygen	30 minute cycles

computer verification models and life-prediction algorithms. In what follows, creep and fatigue are examined individually and interactively.

3.1. STATIC CREEP

Two pressure-vessel damage concerns arise from high temperature creep. The first is rupture and the second is racheting (successive changes in diameter). Test data for creep rupture under constant stress and temperature are abundantly available in the literature, Refs. [4], [5] and [6], and can be modeled by an Arrhenius-type relationship:

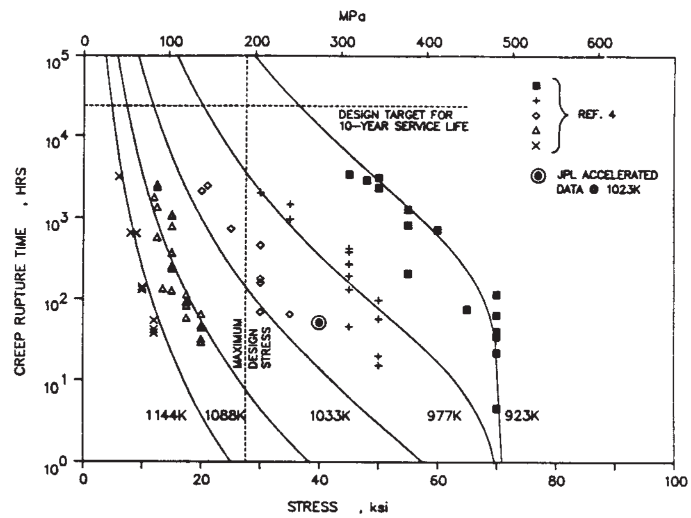


Fig. 4. Isothermal static creep-rupture data for Inconel 625

$$r = [(A/\sigma^2)/\{(C/\sigma)^4 - 1\}] \exp(Q/kT) \qquad (2)$$

where

r = creep rupture time, hours
σ = stress, N/m^2
A = 2.38×10^{-8} N^2-hr/m^4
C = 4.896×10^8 N/m^2
Q/k = 58771K
T = operating temperature, K

Fig. 4 compares the analytical Eqn. (2) and the test data. Despite the scatter, the analytical expression fits the data reasonably well. For an isothermal, low stress operation the creep rupture time is proportional to σ^6. When the stress level approaches the tensile strength (constant C in the equation), rupture time decreases sharply. A static creep test of the container specimen at 1023K/5.52×10^6 N/m^2 (5.52×10^6 N/m^2 corresponds to approximately 40 ksi hoop stress) results in a rupture time of 69 hours, which correlates reasonably well with the prediction and provides some credence to the applicability of the analytical expression. If static creep were the only active mechanism, then at the system design point stress and operating temperature level (773K), Fig. 4 predicts that the design life of the Inconel container vessel will amply exceed the service life target. Thus static creep rupture is not a significant issue.

3.2. CYCLICAL CREEP

The rachet tolerance for the sorbent container wall is around 1% before it contacts the far wall of the gas-gap heat switch. For a cyclic load, a portion of the creep occurring during the on-load phase of the cycle period (the so-called "anelastic strain") is recovered during the off-load phase of the cycle period, Ref. [7]. Fig. 5 illustrates a comparison between constant load deformation and that with cyclic loading. Cyclic loading significantly reduces the total elongation over that which would have occurred under static creep conditions.

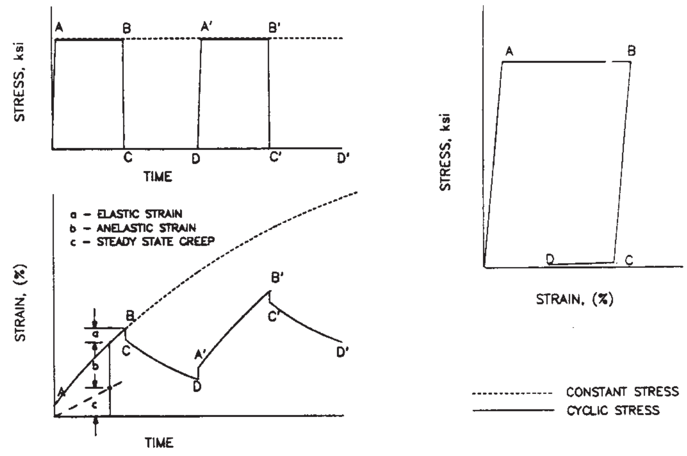


Fig. 5. Schematic representation of cyclic stress loading

In addition to reducing the total creep deformation, pressure cycling also increases the effective creep rupture time. An experimental study, Ref. [8], has shown that for a cycling period of around 30 minutes, the effective time-to-rupture is approximately four times longer than if the period is 12 hours or more. These considerations imply that, insofar as the creep mechanisms are concerned, the Inconel container walls can easily meet their requirements over the ten year service life.

3.3. FATIGUE

Fatigue endurance is a basic material property that is typically measured at high cycle rates. Fig. 6 shows fatigue data, Ref. [9], for Inconel 625 in the temperature range of interest. The fatigue endurance limit can be modelled as an Arrhenius relationship similar to Eqn. (2):

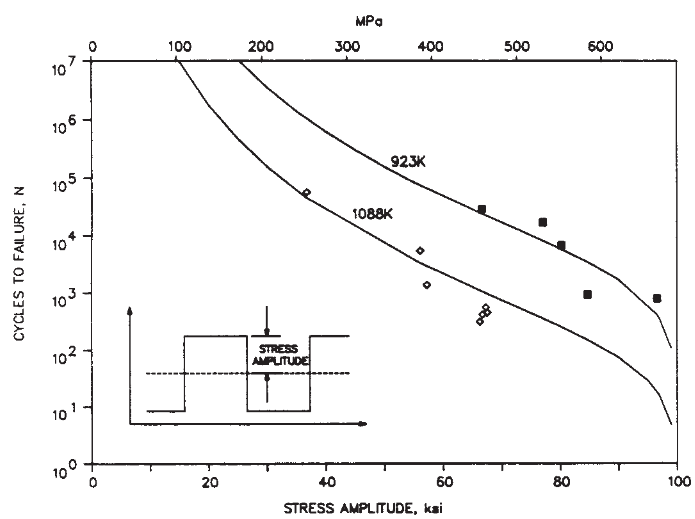


Fig. 6. Isothermal fatigue failure data for Inconel 625

$$N^f = [(B/\sigma)^2/((C'/\sigma)^4 - 1)] \exp(Q'/kT) \quad (3)$$

where

N_f = cycles to failure
B = 0.03
Q'/k = 19000K
C' = 6.895×10^8 N/m^2
σ = applied stress
T = operating temperature, K

3.4. COMBINED CREEP-FATIGUE DAMAGE

Stress cycling at elevated temperatures involves both creep and fatigue. Although there are many theories to assess the interactive damage contributions of cyclic creep and fatigue, the most straight-forward technique is Miner's rule, Ref. [10], that linearly accumulates the individual fractional damage rate, as follows:

$$1/\tau = 1/(G\Gamma) + 1/(N_f\lambda) \quad (4)$$

where

τ = useful service life considering both cyclic creep and fatigue damages
Γ = the creep rupture time under constant stress (static creep)
G = modifier for cyclic creep (= 4, e.g., for a 30 minute cycle period)
N_f = fatigue endurance limit
λ = cycling period, hr

Fig. 7 shows the expected material service life prediction, based upon Eqn. (4), for a cyclic period of 0.5 hours in the temperature range between 823K and 1023K (550°C and 750°C). In the high temperature regime, service life is dominated by creep; at lower temperatures, fatigue damage contributions become more predominant. Based upon these data, we conclude that at the compressor design stress and operating temperatures (< 773K), predicted operating life of the Inconel compressor sorbent container greatly exceeds the 10-year service life goal (8.76×10^4 hrs).

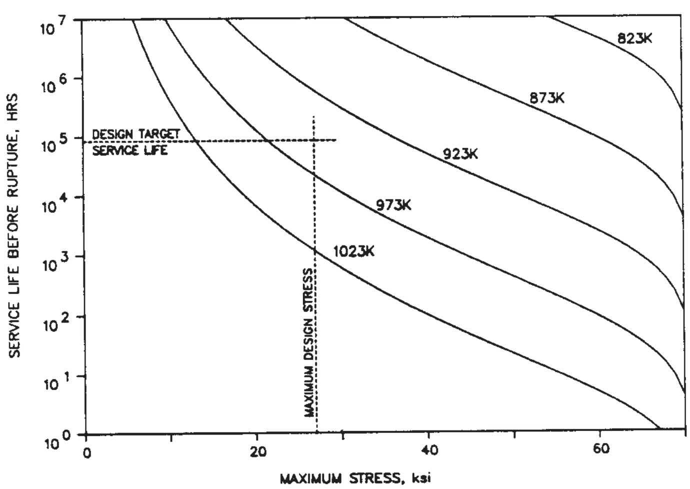


Fig. 7. Isothermal creep-fatigue service life prediction for Inconel 625.

4. RELIABILITY OF ELECTRICAL CARTRIDGE HEATERS

A typical high-temperature electrical cartridge heater consists of a conductor that generates ohmic heat in response to an impressed voltage across its length. As shown in Fig. 1, the heating element is surrounded by an insulating material such as alumina or magnesia powder, and is then packaged in a protective sheath, usually a superalloy such as Inconel. The heating element is the life-limiting component. Similar to pressure vessel reliability, heater element failure mechanisms arise from stress effects, temperature variations, and material interactions such as corrosion and erosion. For a homologous temperature lower than 0.5, the dominant failure mechanism for cyclically operated heaters is fatigue fracture; heaters designed for this lower temperature range focus on arrangements to relieve excessive thermal stresses. Failure at high temperature operation can result from corrosion and from metallurgical changes that erode material endurance properties.

Two considerations guide sorption compressor cartridge heater design: (1) small thermal inertia; and (2) long service life. The service life for low temperature service operation is dictated by material fatigue properties. As temperature increases, damage contributions from fatigue and creep combine to limit service life.

The main objective of the first phase of JPL's test program has been to understand and quantify the degradation mechanisms likely to limit sorption heater life, and in particular to evaluate accelerated test techniques for heater qualification and acceptance testing.

Because heaters contain a number of complex interactive stressing environments, it is important to carry out tests on heater constructions as representative as possible of those likely to be used in the actual sorption application. To this end, Tayco Engineering, Inc., an experienced and respected manufacturer of space-qualified high temperature heaters, supplied sixty small, custom designed cartridge heaters for the test program. This heater incorporated coils of fine (0.1 mm diameter) nichrome wire embedded in ceramic tubes (4-holed straws) that are packed and filled with finely-powdered alumina to provide a compact heat conductive path. The unit is sheathed and hermetically sealed in Inconel to exclude oxygen. A wide variety of characterization and accelerated aging tests have been conducted on the heaters.

Fig. 8 shows the test setup. The infrared thermography equipment monitors the temperature level lengthwise along the heater sheath. Sheath temperature profiles and heater electrical resistance are monitored periodically. Fig. 9 shows that significant temperature gradients exist along the heater sheath length. Maximum temperature is located at the central section of the sheath. A typical profile exhibits a central peak temperature of about 1223K (950°C) and endpoint

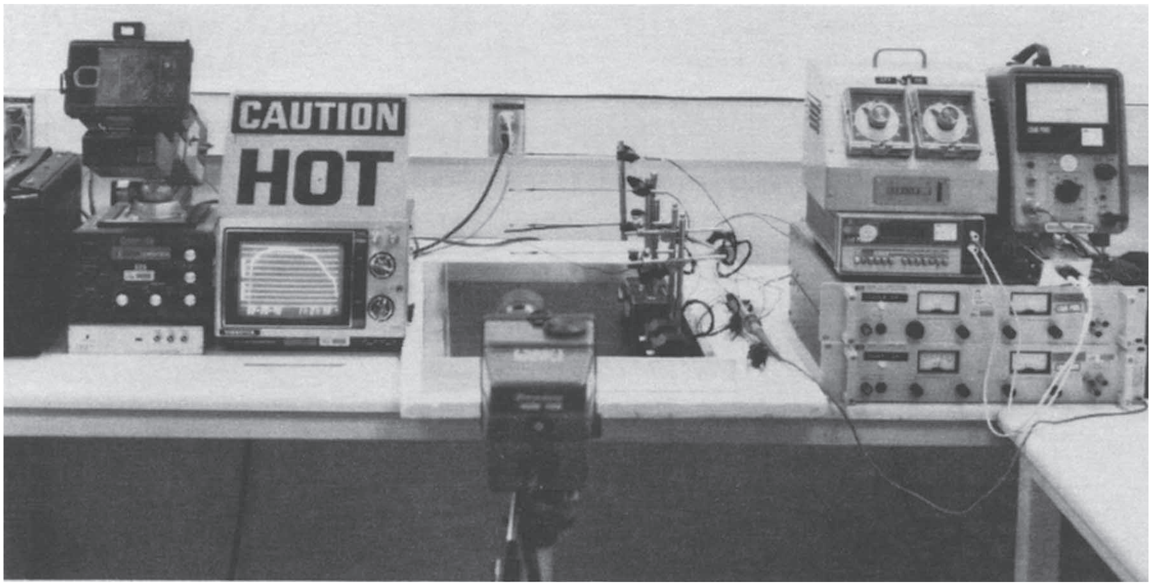


Fig. 8. Heater reliability test with infrared thermography apparatus

temperatures of from 973K to 1073K (700°C to 800°C). Accelerated aging tests have been conducted parametrically, using both sheath temperature level [1223K and 1323K (950°C and 1050°C)] and degree of on-off cycling (none or every 10 minutes) to vary the imposed stress. Several short-duration precursor experiments were also conducted to understand possible problem areas. These included purposely breaching the hermetic seal to understand the visual manifestations of failure by oxidation, and special experiments to evaluate the conductance between the heating element and the insulating powders that provide heat transfer to the outer sheath.

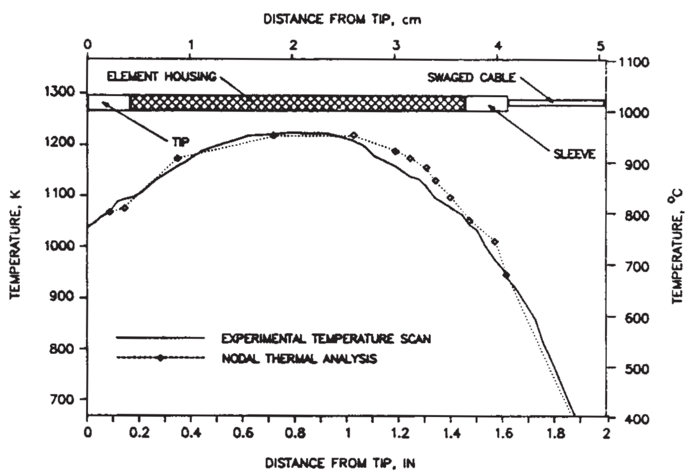


Fig. 9. Temperature distribution along a cartridge heater

During both continuous heating tests and on-off cycling, the heater electrical resistance increases steadily as operating hours are accrued. Fig. 10 shows typical percentage variation in resistance, displaying a monotonic, and for the most part linear, increase with test time. At constant voltage, electrical resistance increase implies a decrease in total power dissipation level (Ohm's law). The temperature of the central section of the heater cartridge does not change, but the endpoint temperatures drop. Thus the temperature gradient along the sheath increases over time.

Maximum filament temperature is the most important parameter governing heater element service life. Heater element coils with fine wires can effectively relieve thermal stress concentration and thus improve low temperature operation service. However, element useful life is shortened for high temperature operation, Ref. [11]. Fig. 11 illustrates the useful life for heater on-time-before-failure as a function of maximum temperature for two filament diameters. The literature data for a 0.75 mm diameter heater filament, Ref. [12], correlates well with an Arrhenius-type function (dotted line). The prediction for 0.1 mm filament diameter heaters, represented by the solid curve, shows a significant decrease (around an order of magnitude) in useful life as compared to the 0.75 mm case.

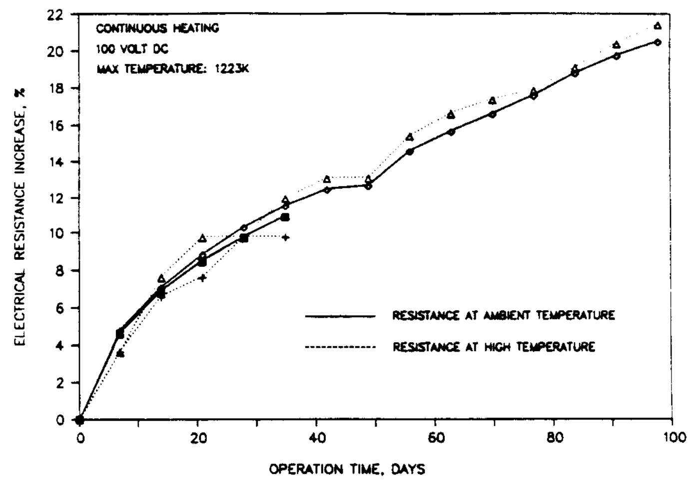


Fig. 10. Electrical resistance increase as a function of operating time

As shown in Fig. 11, the JPL test data (Tayco heaters with 0.1 mm filaments) exhibit considerable scatter. At the lower temperature, 1223K (950°C), the failure times varied by a factor of 30; at the upper temperature 1323K (1050°C), the spread was similar. Surprisingly, the heaters that were cycled on and off (10-minute periods, 50% duty cycle) performed somewhat better than the continuously-on heaters in terms of total accrued on-time before failure. This confirms that fatigue is not a driving issue at the high temperatures.

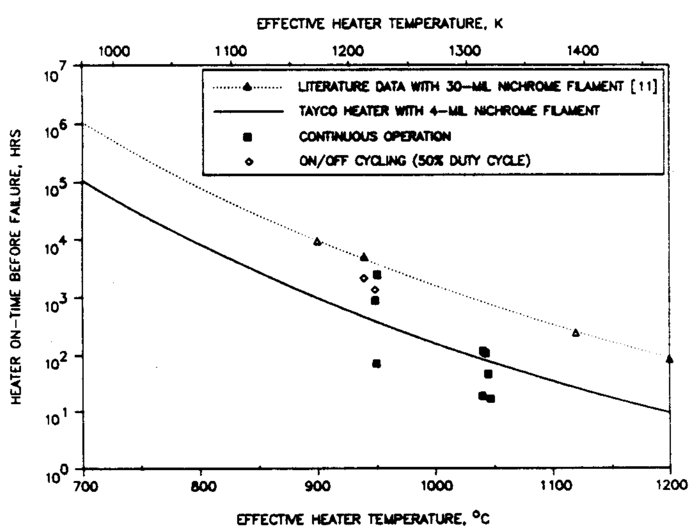
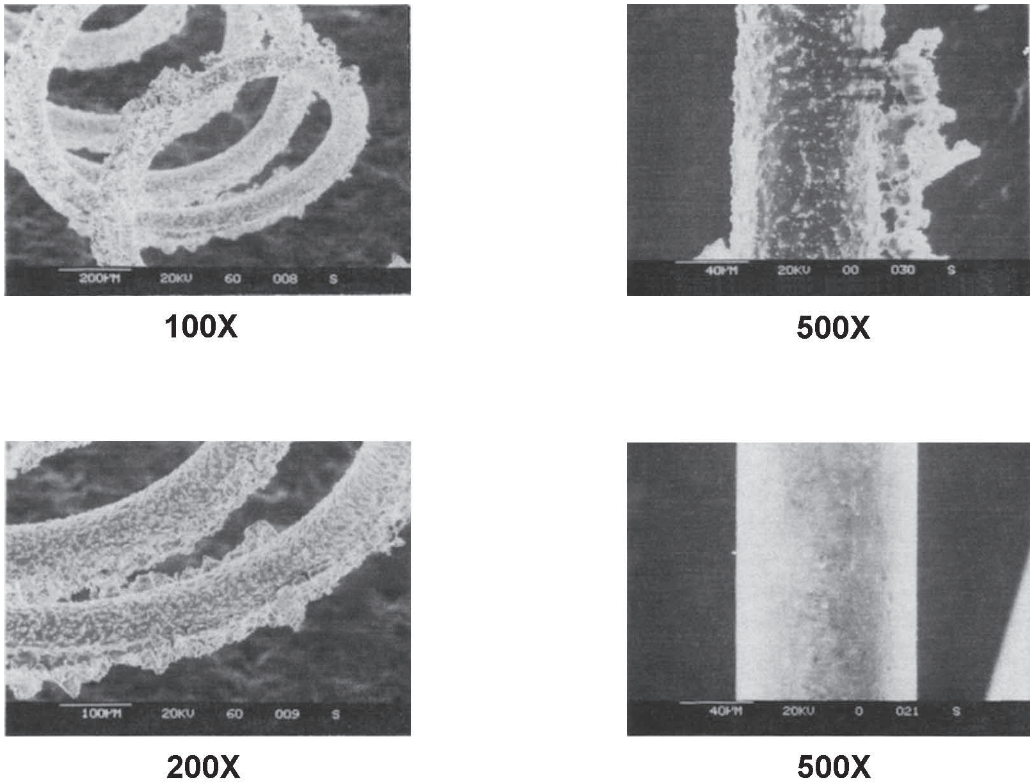


Fig. 11. Heater service life predictions as a function of maximum temperature for Tayco P/N 55-5088 heaters and literature hearters



(a) Erosion of heater element

(b) Diameter reduction of heater element

Fig. 12. SEM pictures of heater elements

In an effort to understand the failure mechanisms revealed by the changes in resistance and temperature, extensive failure analyses were performed, including scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). Results shown in Fig. 12 illustrate a typical cross-section near the failure point. The damage features a decrease in wire diameter and many "dendritic" formations; the reduction in wire diameter correlates with the observed electrical resistance increase. Although the exact cause of the observed heater filament erosion is not apparent, it is suspected to be a physical melting/vaporization mechanism -- possibly brought on by degraded heat transfer conductance between the filament and case. No evidence of foreign materials or oxides was found in the SEM studies.

Because evaporation and melting are highly temperature dependent, it is unlikely that these mechanisms will predominate at the planned operating temperatures in the sorption compressors. This implies that less-accelerated tests, using temperatures closer to the expected operating temperatures, should be conducted also. In this regard, longer term tests are presently underway to further validate the life prediction correlations; two additional heater experiments, each consisting of 5 specimens, are underway. One set of heaters is operating in a continuous mode at 973K (700°C), the other set is being cycled at 3-minute periods, 50% duty cycle, with a maximum temperature of 823K (550°C). These experiments are designed to provide a projected failure time of around 1 year. None of the specimens have failed to date; the electrical resistance measurements show a typical 5% increase.

Since nearly all mechanisms experience Arrhenius temperature acceleration similar to that noted in Fig. 11, it is unlikely that future performance will fall below the indicated projection. This provides high confidence that heaters should be easily compatible with high-reliability 10-year life sorption refrigerators.

5. CONCLUSIONS

The accelerated heater and container material tests indicate that over ten-year life can be achieved for these critical components under the much less severe sorption cooler conditions. Future work must also focus on developing non-destructive acceptance tests to screen out defective components.

6. ACKNOWLEDGEMENTS

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