Component Reliability Testing of Long-life Sorption Cryocoolers

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This paper summarizes ongoing experiments characterizing the ability of critical sorption cryocooler components to achieve highly reliable operation for long-life space missions. Test data obtained over the past several years at the Jet Propulsion Laboratory (JPL) are entirely consistent with achieving ten year life for sorption compressors, electrical heat ers, container materials, valves, and various sorbent materials suitable for driving 8 to 180 K refrigeration stages. Test results reported include a praseodymium-cerium-oxide/oxygen ("PCO/O2") compressor system, for 65 to 90 K cryocoolers, that has accrued over 35,760 hours and 79,567 cycles of maintenance-free operation with no degradation through 1993, and a Saran carbon/krypton compressor system, for 120 to 140 K cryocoolers, that has demonstrated 16,600 hours and 33,200 cycles of operation. Similar extended cycling tests of $LaNi_{4.8}Sn_{0.2}$ and ZrNi hydride compressor systems for use in 8 to 30 K cryocoolers have been initiated.

Planned future tests necessary to gain a detailed understanding of the sensitivity of cooler performance and component life to operating constraints, design configurations, and fabrication, assembly and handling techniques, are also discussed. The basic technology base developed through this effort is enabling the design and manufacture of high-reliability sorption cryocoolers for future long-life, low-vibration, lightweight, spacecraft sensor cooling applications.

INTRODUCTION

One of the primary advantages of sorption cryocoolers are their potential to operate reliably for ten or more years. Ongoing reliability physics investigations at JPL have been directed at confirming this long life capability. These investigations consist of a thorough test and evaluation program at the materials, component, and system level to gain a detailed understanding of important degradation mechanisms. The knowledge thus obtained is leading to identification of design enhancements, operating constraints, and other means of eliminating failure mechanisms, as well as useful definitions of effective fabrication, assembly and handling techniques, By establishing the reliability physics technology base required to develop long-life sorption cryocooler systems, this research effort is enabling the early insertion of this novel refrigeration technology into future space missions. Ideally, life testing a statistically significant number of flight-qualified sorption cryocoolers is necessary to confirm the high reliability and long-life capability of these systems. The prohibitive cost and time required to complete the many years of necessary testing makes this approach impractical. Instead, the focus has been on reliability physics investigations at the component and compressor system-level. The accomplishments, status, and plans of this sorption cryocooler reliability physics research effort are described in this paper.

Sorption Cryocooler Description

In a typical sorption cryocooler system, a simple Joule-Thomson (J-T) expansion device is combined with a sorption compressor. The compressor contains solid sorbent material that either physically adsorbs or chemically absorbs the refrigerant gas at relatively low pressure when cooled to near ambient temperatures or above. When heated, the gas is liberated and pressurized, Because the compressor alternates between sorbing and desorbing as it is cooled and then heated, it produces an intermittent flow of gas. Thus, sorption cryocoolers inherently provide intermittent cooling, making them ideal for applications where periodic cooling is sufficient.[1-5] However, continuous cooling can be obtained by simply combining and sequentially heating and cooling a series of compressor elements, as shown in Fig. 1,

Different refrigeration temperatures can be achieved by use of various sorbent-gas combinations, as indicated in Fig. 2. Extreme low temperatures can be achieved by cascading several refrigeration stages together. For some systems, the refrigerant fluid is actually solidified using a specialized low pressure sorbent bed, such as the solid hydrogen 10 K sorption cryocooler described in Refs. 1-6.

Various laboratory sorption cryocooler systems have been successfully demonstrated. Coolers driven by physical adsorption compressors include a 185 K zeolite/ N_2O system,[7] a 120-140 K Saran carbon/ krypton system,[8] an 80-120 K charcoal/ N_2 system,[9] a 20-30 K charcoal/H_2 system and a C/He system for 4-5 K cooling. 10 Demonstrated chemisorption coolers include several hydride-based $LaNi_5/H_2$ systems for 20-30 K,[11-16] a periodic-operation solid hydrogen 10 K stage using ZrNi hydride with H_2,[3] and a praseodymium cerium oxide ($Pr_{1-n}Ce_nO_x$, or "PCO') system that provided cooling at 72-80 K.[17] In addition to these laboratory systems, a flight qualified periodic cooler combining a $LaNi_{4.8}Sn_{0.2}/H_2$ 20-30 K stage with a 10 K $ZrNi/H_2$ stage has been developed for a spaceflight experiment.[5,6]

Sorption Cryocooler Components

In a sorption cryocooler, compression is achieved thermally instead of mechanically, and refrigeration is produced by simple expansion through a passive orifice or capillary tube. There is

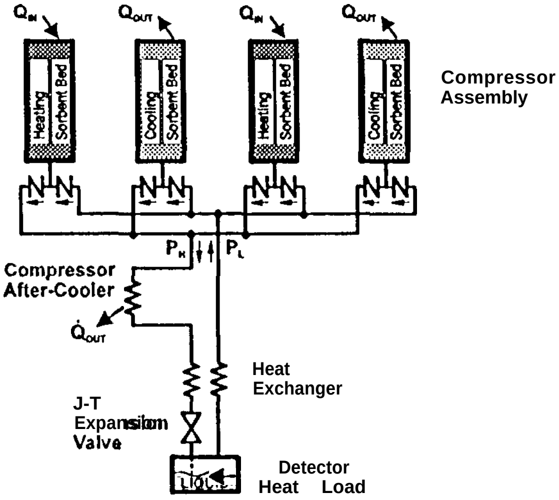


Figure 1. Single-stage sorption cryocooler cycle schematic.

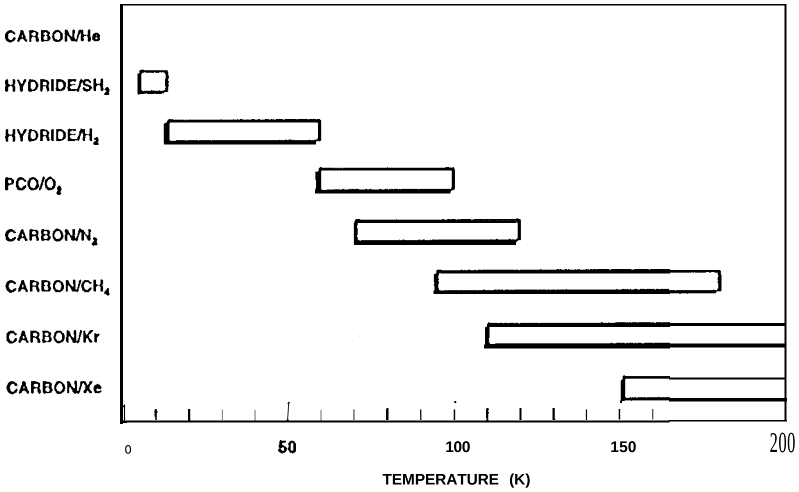


Figure 2. Temperature ranges of typical sorbent/ gas combinations.

no use of any high tolerance wear-related moving parts such as motors, flexure supports, gas bearings, or any cold end displacement devices. Because the only moving parts are highly reliable check valves or solenoid-operated valves that operate at very low frequencies (<< 0.1 Hz), sorption systems are inherently vibrationless, and do not experience many of the failure modes typically associated with mechanical cryocoolers. However, absence of moving parts dots not by itself insure high reliability. A number of important reliability issues must be considered, including those related to sorbent materials, electrical heaters, pressure containment vessels, valves, filters, and contamination, Degradation and failure mechanisms related to these areas must be understood, and eliminated or controlled, to achieve high reliability and long life. The results of ongoing experiments in these areas are described in the following sections.

EXPERIMENTS

Compressor Systcn~-Level Experiments

Experiments at the compressor system-level have enabled evaluation of long-term degradation trends and failure mechanisms of sorbent materials, pressure containment vessels, heaters, filters, and valves operating together in an integrated system. By studying integrated compressor systems, important system-level synergisms that may adversely affect long-term cryocooler performance are being identified.

Hydride Chemisorption Compressor Systems. Recent investigations have focused on long-term cycling of hydride sorption compressors for 10-30 K refrigeration stages.

Figure 3 shows the closed-cycle high pressure $LaNi_{4.8}Sn_{0.2}$ hydride compressor cycling apparatus. The compressor contains 574 g of $LaNi_{4.8}Sn_{0.2}$ hydride in a 1.1 cm thick, 19.7 cm long, annulus contained between a 1.3 cm O.D. sintered filter and a 4.22 cm O.D. pipe. All hydrogen-wetted surfaces are 3 16L stainless steel. A 0.15 mm orifice, located between the compressor and the 1.07 L supply tank, controls the flow rate during absorption to simulate a typical (J-T) valve.

A motor-driven tube furnace mounted on tracks provides programmed temperature cycling of the compressor between 21-275-21 ‘C over a 120 minute cycle, while the pressure response in the compressor and tank are monitored. First, the compressor is heated from ambient temperature to 270°C in about 35 minutes, thus pressurizing the hydrogen and charging the supply tank to 10.7 MPa (1550 psia). A 0.014 MPa (2 psi) check valve allows desorbed gas to flow back to the tank. The furnace is then removed, allowing the compressor to cool to ambient temperature in about 35 minutes. After completing the cooling phase, a pneumatically-actuated valve between the compressor and the 1 liter supply tank is periodically opened to allow eight 20-second absorption

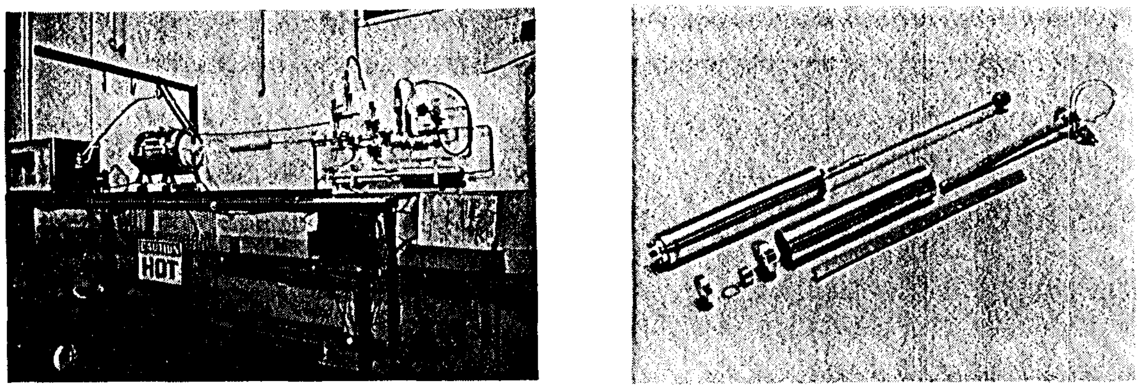
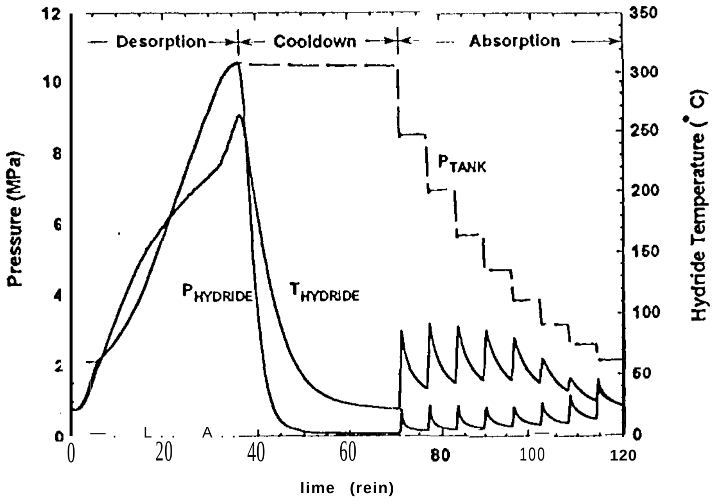


Figure 3. High pressure $LaNi_{4.8}Sn_{0.2}$ hydride sorbent bed cycling apparatus.

bursts of hydrogen, each followed by about a 6 minute cooling period. This cycle is representative of the range of flow rates, temperatures, pressures, and cycle times expected for the 20-30 K stage of typical periodic sorption cryocooler systems. However, the absorption is accomplished in eight separate small bursts instead of a single larger one *in* order to limit the temperature rise in the compressor, which has not been designed for efficient heat rejection. Figure 4 shows the pressure and temperature response to the imposed cycling conditions for the benchmark cycle.

A similar long-term cycling apparatus was developed for a low pressure ZrNi hydride compressor. The compressor contains 22.4 g of ZrNi hydride in a 4.75 mm thick annulus contained between a 2.67 cm O.D. pipe and a 1,3 cm O. D. filter tube, 19.7 cm in length, as shown in Fig. 5. The low pressure test apparatus contains a large supply tank (5 L), and uses two separate flow control orifices. By using two separate orifices, it is possible to simulate the absorption flow rates for the two distinct phases of a quick-cooldown 10 K sorption cryocooler.[3,4] First, a 0.28 mm orifice is opened for 30 seconds to obtain the high absorption flow rate that simulates hydrogen vaporization and sublimation during the quick cooldown of liquid hydrogen to solid hydrogen. Next, a 53 μm orifice is opened for 1 hour to simulate the lower absorption flow rate during a sustained cooling period below 11 K. The actual length of time that the compressor can maintain a pressure below about 0.69 kPa (5.2 torr) simulates the length of time that a cold-head can operate at < 11 K. Note that even lower pressures (and thus lower cold head temperatures) can be obtained by reducing the compressor temperature to below ambient, as is possible for typical space applications.

After completion of the absorption phase, the compressor is heated to 300°C in 20 minutes to desorb the hydrogen and recharge the supply tank to 0.1 MPa (15.5 psia). The compressor is



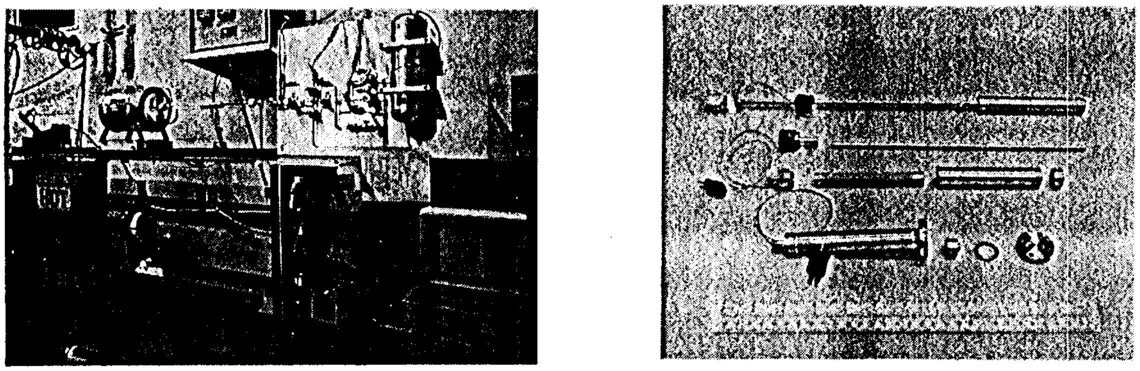


Figure 5. Low pressure ZrNi hydride sorbent bed cycling apparatus.

then allowed to cool to 21 ‘C in about 40 minutes. Figure 6 shows the pressure and temperature response to the imposed temperature cycling.

A decrease in reversible hydrogen capacity with increased cycling has been observed for both compressor systems. After about 3000 cycles, the maximum resorption pressure decreased by -30°A and 9% for the high and low pressure systems, respectively. Both apparatuses were then connected directly to a mass spectrometer to analyze desorbed gases and identify possible contaminants. The results indicated 450-600 ppm H_2O present in each system. The high pressure system also was found to contain -500 ppm methane and >1000 ppm argon. It is speculated that the argon was probably introduced during the initial hydrogen fill process, and the methane was formed from residual hydrocarbons that weren’t properly cleaned from the hydrogen fill line. A potential source of the water is the chemical reduction of oxide coatings present on filters, tubing, hydrides, and other components due to the presence of concentrated H_2. Further diagnostic tests are planned to test this theory, identify the source of H_2O, and gain a full understanding of the observed changes in hydrogen pumping capacity. These planned tests include thorough leak detection, addition of cold traps, determining effects of various reactivation techniques, and studies of cycled hydride materials to identify possible disproportionation, If the oxidation/ reduction theory can be confirmed, then techniques will be investigated to eliminate the oxide coatings with prolonged hydrogen bake-outs followed by high vacuum evacuation.

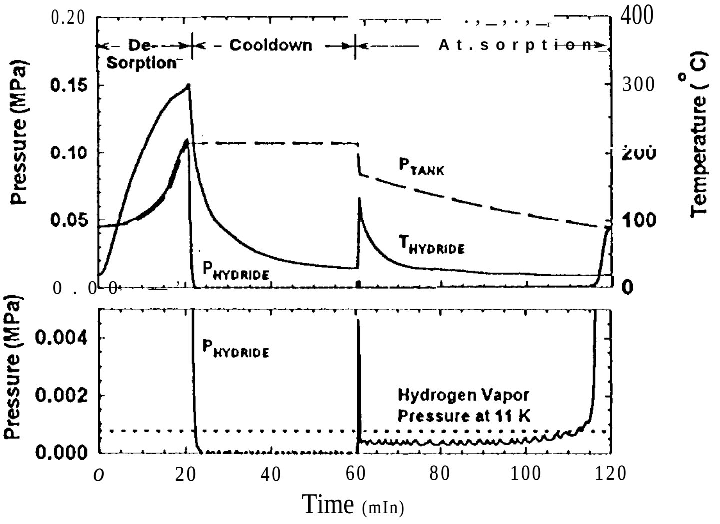


Figure 6. Low pressure ZrNi hydride sorbent bed endurance test benchmark cycle.

Fortunately, after thorough evacuation, bake-out, and reactivation the compressors were able to regain full reversible absorption capacity. If necessary, periodic reactivation can be made part of the normal operating procedure for hydride sorption cryocooler systems. However, the goal of these investigations is to determine the cause of the degradation so that it can be eliminated. If degradation is found to be inherent in the $LaNi_{4.8}Sn_{0.2}$ or ZrNi hydride materials, then it is important to separate and quantify its sensitivity to operating conditions, cycling, and long-term high-temperature exposure. This will enable definition of operating constraints, and allow incorporation of sufficient design margins (i.e. adding extra hydride material), or other means of recovery for future long-life sorbent beds. Follow-up investigations will focus on these key areas.

PCO/O_2 Chemisorption Compressor System. A praseodymium cerium oxide/ oxygen (PCO/O_2) sorption compressor system for use in 65 to 90 K cryocoolers has been undergoing continuous closed-cycle life testing since March 1989. The compressor was originally designed as part of a two-stage sorption cryocooler that provided 1/3 W cooling at 80 K, and achieved a minimum no-load temperature of 72 K.[8,17] After successfully demonstrating the required refrigeration capability, the system was reconfigured by upgrading the compressor stages for independent autonomous operation, and replacing the cryogenic J-T valve with a room temperature throttling valve.

After completing 12,526 hours and 27,921 cycles of continuous closed-cycle operation, comparisons of temperatures, pressures and flow rates indicated no observable change in compressor performance. At that point, one of the two PCO/O_2 compressor elements was removed from service, disassembled and analyzed in depth to identify and quantify any signs of internal degradation. See Fig. 7, Extensive physical and chemical analyses were conducted on the Inconel container, surrounding radiation shields, internal heater, check valves, filter, tubing manifold, and samples of removed PCO. The analyses, which included x-rays, Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), x-ray Diffraction (XRD), Diffuse Reflection Infrared Fourier Transform (DRIFT), and Thermogravimetric Analysis (TGA) indicated no significant degradation in any of the compressor components. No changes in geometry, metallurgical structure of the container, or migration of PCO particles were observed. The only significant change, noted by the XRD and DRIFT analyses, was the presence of carbonates, $Pr_2O_2CO_3$ and $C0_3^{-2}$, in both cycled and pristine PCO samples. This was attributed to prolonged exposure to ambient air, causing contamination by $C0_2$, which reacted to form carbonates. Fortunately, the carbonate layer seems to be permeable to oxygen, as the reversible oxygen absorption/ resorption capability was not inhibited. Furthermore, it was found that the carbonates can be essentially eliminated by prolonged vacuum bakeout (> 24 hours at > 800°C). Nevertheless, minimizing PCO exposure to air during the manufacture of future PCO/O_2 sorption cryocoolers is recommended.

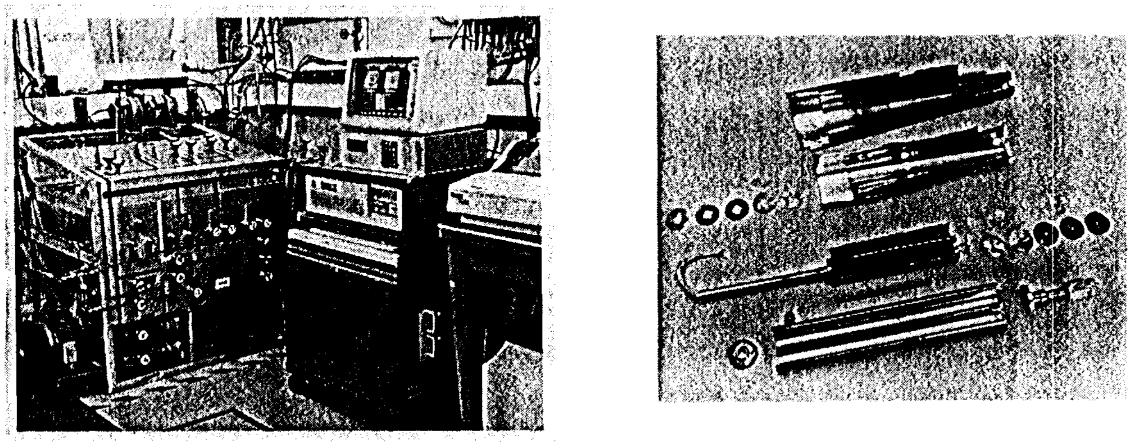


Figure 7. PCO/O_2 Oxide sorption compressor life test system Disassembled PCO/O_2 compressor showed no signs of internal degradation after 12,526 hours and 27,921 cycles, Remaining compressor completed 35,760 hours and 79,567 cycles with no observed performance changes.

Through 1993, the remaining compressor element has accrued over 35,760 hours and 79,567 cycles of continuous, maintenance-free, closed-cycle operation with the original oxygen load. Although a small amount of oxygen working fluid was lost when the other compressor element was removed, no new oxygen has ever been added. The PCO/O_2 compressor exhibits extremely stable and reliable performance, as no degradation in flow rate, pressures, temperatures, kinetics or capacity have been observed.

Note that the compressors in this life test have been subjected to significantly greater stress conditions than required for a PCO/O_2 sorption cryocooler. The compressors were operated at a maximum cycle temperature of 923 K, which is about 150 K higher than required.[8] Thus, the observed performance stability is especially impressive, as this has been a significantly accelerated aging test.

C/Kr Physisorption Compressor System. Upper stage cooling for the 80 K PCO/O_2 system described in the previous section was provided by a 140 K C/Kr refrigerator.[8] Subsequent to completing the successful cryogenic tests, the four C/Kr compressors were reconfigured for independent life testing in a similar manner to the PCO/O_2 compressors, One of the four compressors accrued over 16,600 hours and 33,200 cycles with no performance degradation, as no significant changes were observed in pressures, temperatures, or flow rates. Testing was stopped at that point and the system was disassembled due to lack of funding.

The other three compressors suffered from effects of HCl-caused corrosion, and were removed from the system after accruing between 1000 hours and 12,000 hours. The Saran carbon in the compressors was inadequately processed by the supplier, as 2.1 to 2.6°/0 residual Cl was present due to incomplete pyrolysis of the polyvinylidene chloride (PVDC) base material. In-place reprocessing by dehydrochlorination with a four-day 700”C bakeout, followed by two days at 900”C with a nitrogen purge, was conducted on the compressors, reducing the Cl level to O. 13%. However, this was not sufficiently low to prevent eventual evolution of HCl with continued cycling, thus causing the observed corrosion. Proper dehydrochlorination involves processing at 10OO°C to reduce the final chlorine content to less than 0.02%, as measured in JPL-manufactured Saran carbon. [19] Thus, HCl corrosion is not expected to be a problem if the Saran carbon is processed correctly, and extremely high long-term performance stability of carbon physical adsorption systems is expected,

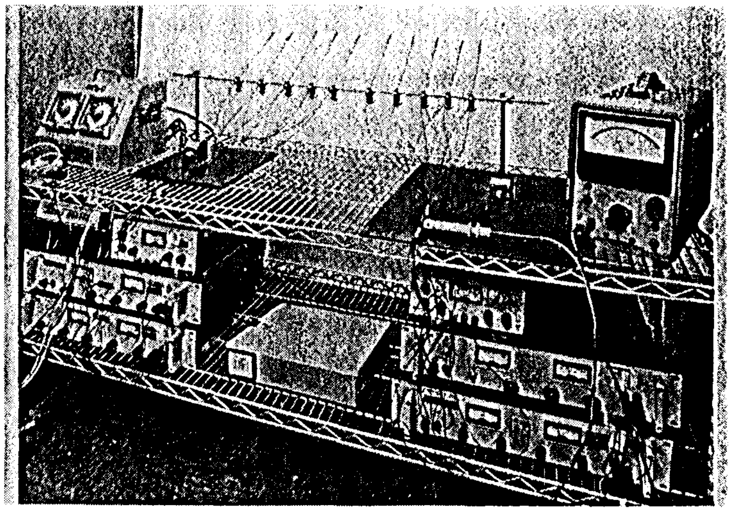
Sorbent Materials Studies

In addition to the compressor system-level reliability physics experiments described above, separate studies were performed at the sorbent materials level for candidate hydrides.

Hydride Materials. Recent materials studies involved detailed characterization of various hydrides, including measurements of chemisorption isotherms for ZrNi and $LaNi_{4.8}Sn_{0.2}$ hydrides, disproportionation studies of $ZrNiH_x$, tin substitution studies for $LaNi_{5-y}Sn_yH_{x}$., and studies of isotherm sensitivity to batch-to-batch variations in hydride materials. Detailed results were presented in Ref. 18.

A key result was that degradation of ZrNi hydride due to disproportionation may be eliminated by limiting resorption temperature to below 650 K. This causes resorption pressures to be below about 600 kPa (87 psia), which is completely compatible with the operating range of typical 10 K cryocooler systems. However, the sensitivity of disproportionation to cycling still needs to be determined.

Another important result was quantification of the effect of varying tin content in $LaNi_{5-y}Sn_yH_x$ on plateau pressure and extent, enabling optimization of sorbent bed designs for different applications and operating conditions. In a separate study, it was found that $LaNi_{4.8}Sn_{0.2}$ hydride performance is very sensitive to batch-to-batch variations. Large induction-melted batches were found to



Figure, 8. Heater life test apparatus.

produce more inefficient isotherms than smaller samples made from arc-melted ultra-high purity base metals. To achieve efficient, predictable, repeatable sorption compressor performance, it is important to use ≥ 99.90/0 pure base metals and have - 1°A over stoichiometry lanthanum to bind residual oxygen impurity. If chemical reduction of oxide coatings is determined to be the source of H_2O, as described earlier for the hydride compressor system-level tests, then it may be important to use even higher purity base metals and to prevent oxidation by handling only in inert atmospheres. Further testing is planned to resolve this issue.

Planned future experiments also include low temperature kinetics tests to determine the lowest compressor temperatures at which absorption is practical for both $LaNi_{4.8}Sn_{0.2}$ and ZrNi hydrides, and studies of disproportionation sensitivity to high temperature storage for $LaNi_{4.8}Sn_{0.2}$, and to cycling for both $LaNi_{4.8}Sn_{0.2}$ and ZrNi.

Electrical Heaters

Ironically, one of the major reliability considerations in sorption cryocoolers concerns the highest temperature component -- the electrical heater. Sorption compressors typically use cartridge-type heaters, consisting of a nichrome element surrounded by an electrically insulating material such as alumina or magnesia powder contained in a hermetically sealed metallic sheath. The heating element is the life limiting component, with failure mechanisms arising from stress effects, temperature variations, and material interactions such as corrosion and erosion.[20]

A program of accelerated aging tests, combined with analytical thermal models and scanning electron microscopy of failed elements was initiated, and the results were used to correlate with life prediction models that were developed.[20] The data confirmed that useful service life correlates with an Arrhenius temperature relationship. One interesting result was that heaters cycled between ambient temperature (-21 'C) and 1223 K (950°C) over 10 minute periods were found to perform better than heaters continuously maintained at 1223 K (950°C), as described in Ref. 20. This confirmed that fatigue is not the dominant failure mode at these high temperatures. SEM and EDS analyses indicated that a physical melting/ vaporization mechanism led to failure, possibly due to degraded heat transfer conductance between the heater element and case.

However, vaporization and melting, which are highly temperature dependent, may not dominate at the required operating temperatures for typical sorption compressors. Therefore, less accelerated tests were initiated in July 1990 using a set of five heaters operating continuously at 973 K (700°C), and another set of five heaters cycled between ambient (≈21°C) and 783 K (51O"C) over 3-minute periods. See Fig. 8. Although the electrical resistance increased up to 17% for some of these heaters, they all continued to operate for 11,232 hours (468 days). At that

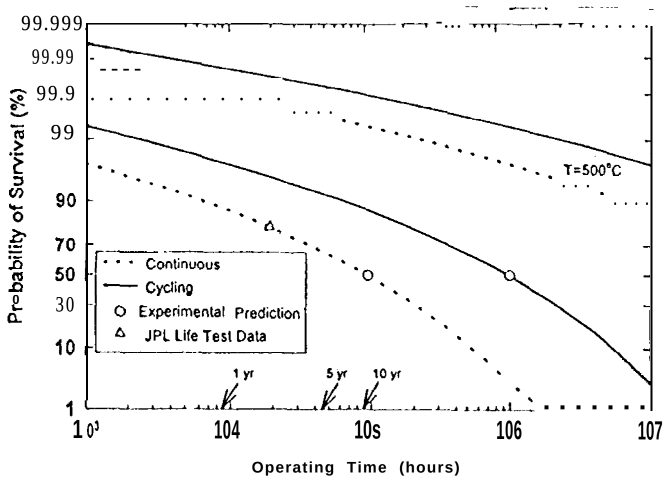


Figure 9. Heater life test predictions.

point, the test temperatures were increased by raising the input power levels. The continuous heaters were subsequently operated at temperatures between 1006 and 1033 K (733 and 760°C), while the average peak temperature of the cycled heaters was 855 K (582°C). One of the continuous heaters finally failed after 21,600 hours (900 days). As of May 1994, the five cycled heaters have continued to operate without failure for a total of 30,500 hours (1271 days) and 610,000 cycles, and the other four continuous heaters continue to operate after 30,500 hours.

The heater life prediction curves in Fig. 9 were produced by combining life test data from these ongoing tests with correlations between lifetime, temperature and cycling that were based on the earlier high temperature tests described above.[20] The Weibull reliability function shown indicates that a ~99.99% probability of survival over 10 years is achievable for a heater cycled up to a temperature of 500°C. Typical sorption cryocooler heaters require even lower temperatures, which will result in higher survival probability. Failure risk can be reduced even further by incorporating redundant heaters if desired.

These accelerated tests and life prediction models provide high confidence that > 10 year life is readily achievable by electrical heaters, especially when considering the less severe operating temperatures and longer cycle times required for typical sorption compressor systems. The > 4 year life accrued as of January 1994 by the heater in the PCO/O2 compressor system described earlier is also consistent with this result.

The accelerated high temperature tests and life prediction models suggest a simple screening test that should be performed on samples selected from the lot of heaters to be used in a long-life sorption compressor. Several test heaters should be continuously operated at highly elevated temperatures. At 1050- 1200°C, the predicted service life is between 10 and 100 hours. If a significant number fail before predicted, then further investigation should be conducted before a heater from that lot is used,

Container Materials

Because the efficiency of a sorption compressor decreases as its thermal mass increases, it is important to minimize the sorbent container mass. Therefore, sorption compressors are typically designed with relatively thin-walled pressure vessels. Degradation mechanisms of these thin-walled containers undergoing simultaneous pressure and temperature cycling include creep- and fatigue-induced fracture, creep ratcheting, scale formation and spalling, and crack formation and propagation.[20] A test program was initiated to study Inconel containers for PCO/O_2 compressors. Inconel was chosen because of its high strength at elevated temperatures and its high oxidation resistance, Combining analysis and experiment, accelerated testing was conducted on 6.99 cm long, 1.9 cm diameter Inconel 625 tubes with 0.2 to 0,3 mm (0.008 to 0.010 inch) walls.[20]

Experiments included static and cyclic temperature/ pressure tests both in oxygen-free and oxygen-rich environments. In the oxygen cyclic temperature/ pressure tests, samples cycled between ambient (-21 °C)/0. 1 MPa and 973 K (700°C)/5,51 MPa in 30 minute cycles accrued a total of 35,996 cycles and 17,998 hours without failure, before the test was finally terminated. Analytical life prediction algorithms were developed, that included the separate and combined effects of static creep, cyclic creep, and fatigue. Similarly to the high temperature heaters, the test results correlated well with Arrhenius temperature relationships. In summary, the results indicated that well over 10 year equivalent Inconel container life was achieved at the expected compressor design stress level (about 193 MPa, or 28 kpsi), and operating temperature (< 773 K).[20]

Consistent with this result, the thin-walled (0.245 mm, or 0.010 inch) Inconel container used in the previously described PCO/O_2 compressor system-level tests was cycled for over 4 years without failure, The removed compressor that was analyzed showed no signs of Inconel degradation, including no measurable loss in wall thickness due to oxidation and spalling.

For hydride-based sorption cryocoolers, hydrogen embrittlement of the container is of special concern, Highly hydrogen-compatible 316L stainless steel has been used to-date for hydride sorption systems, but higher strength materials such as Incoloy 903, A286 and JBK-75 are being considered. A similar test methodology as described above for Inconel is recommended for any container materials selected for use, with the additional goal of studying the effects of long-term high pressure and high temperature hydrogen exposure, Also, because of hydrogen's relatively high permeability, long-term leakage through thin-walled compressor vessels needs to be considered when designing long-life compressor systems.

Another issue uniquely associated with hydride-based systems is damage to container vessels due to stresses induced by expansion of the hydride during hydrogen absorption. Careful control of the container geometry, compartmentalizing the hydride within the container to minimize gross powder migration and compaction, and allowing significant vibration (including launch) only when the hydride is in the fully-expanded fully-absorbed state, has been found to prevent container damage.

Filters

Sorption compressors contain sintered filters, with typical filtration ratings between 0.5 and 5 microns, that are designed to prevent sorbent powder migration while allowing gas to permeate freely. Migration of sorbent material through the filter after launch vibration, or after prolonged cycling, is of concern because of potential degradation of downstream valves. In vibration tests simulating Shuttle launch loads, described in Ref. 4, no pre-cycled vanadium hydride particles penetrated filters as large as 20 microns. Also, absolutely no sorbent powder migration was observed in the previously described disassembled life-tested $PCO/0_2$ compressor, after the valve manifold was flushed with alcohol 'and precipitates were collected in a millipore filter. Microscopic examination of the downstream side of the internal compressor filter shown in Fig. 7 also indicated absolutely no signs of PCO powder. No evidence of degradation of valves or system performance due to sorbent migration has ever been noted in other sorption cryocooler systems.[6,9,12,13] The continually reversing flow during each cycle probably contributes to the high filter reliability that has been observed.

Valves

Major valve reliability issues that must be considered include compatibility with the working fluid, leakage caused by degradation of seat materials or particulate contamination, long term outgassing from polymeric seat materials, explosive decompression of seat materials, and failure of solenoid coils.

For hydrogen solenoid valves, it is sometimes necessary to insert a thin 316L sheath over non-hydrogen compatible magnetic window materials to prevent hydrogen embrittlement.[6] Viton, Neoprene, Kel-F, and Vespel valve seat materials have been used in various sorption systems without observation of outgassing-related problems after proper system cleaning and vacuum bakeout.[6,8,9,12,17] Similarly, explosive decompression of valve seats has not been observed in sorption systems. Insuring good thermal design to enable effective dissipation of heat generated by solenoids in space vacuum will prevent overheating and prevent coil failure. Use of strategically located particulate filters is common practice to prevent contamination-related valve degradation.

With typical sorption refrigerator cycle times ranging between 20 minutes and 12 hours, less than 263,000 cycles are accumulated in 10 years of continuous operation for sorption cryocooler valves, Tests have indicated that commercially available flight-qualified valve lifetimes can achieve this 10 year equivalent life.[21,22] Consistent with this result, the PCO/O_2 compressor system valves have accrued over 79,567 cycles and 35,760 hours of operation without any signs of leakage or other degradation, This includes several check valves and several two- and three-way solenoid valves.

As a screening test for specific valves used in a flight system, it is recommended that a number of qualification units be subjected to accelerated cycling well beyond the number of cycles required for flight, with periodic monitoring of leakage. Acceptance testing for flight units should be designed to screen out infant mortality.

Contamination

Contamination is the major sorption cryocooler reliability issue that has been somewhat neglected. The reason for this is not its lack of seriousness, but because of insufficient funding to properly invest igate this complex area with the attention needed. The two major concerns with contamination are freeze-out of gas-phase contaminants, causing J-T valve or cold heat exchanger plugging, and degradation of the sorbent material. Potential sources of gas-phase contamination include supply gas impurities, outgassing (e.g. from polymeric valve seat materials, tubing, fittings, high surface area sorbent bed filters, cold head wick materials, weld or “braze joint contaminants, and sorbent materials themselves), real leaks, virtual leaks from trapped volumes, and chemical reactions, especially within the compressors during their high temperature resorption operations. Effects of solid particulate contamination can be effectively controlled by particulate filters strategically placed within the refrigeration loop. Particulate filter reliability issues were discussed in a previous section.

The eventual goal of the planned contamination reliability physics investigations is to both minimize the level of contaminants in the final cryocooler system by developing detailed manufacture, assembly, handling, cleaning and fill procedures, and to develop designs that can tolerate reasonable contaminant levels. The latter includes incorporation of gas-purification filters and cold trap getters within closed-cycle sorption cryocooler systems.

Because it is impossible to completely eliminate contaminants, it is important to quantify the tolerance of sorption cryocoolers to specific quantities of different gas-phase impurities. Planned future tests include intentional introduction of controlled quantities of selected contaminants to determine both J-T refrigeration loop and sorbent bed tolerance to contamination. By connecting the sorption system directly to a mass spectrometer, the gas impurities can be monitored and controlled. Other tests include determining if periodic venting to space vacuum and reactivation of sorbent beds enables recovery from sorbent contamination, as described earlier.

A number of recommendations that minimize the risk of contamination-caused degradation can be made based on past experience with various sorption cryocooler systems. Supply gas purity can be controlled to better than 50 parts per billion using commercially available, semiconductor industry-standard, gas purification systems.[23] Use of electropolished tubing, fittings, pressure vessels, and containers will minimize surface outgassing. Use of organic materials (e.g. in valve seats) should be minimized. Completely welded or brazed systems will minimize permeation of air constituents through valves and fittings. Furthermore, outgassing by flux materials and trapped volumes can be reduced by welding instead of brazing wherever possible. Use of butt-welds instead of socket welds will reduce trapped volumes that are common sources of virtual leaks, Filter manufacturing processes should be carefully controlled, since their large surface area provides an ideal trap for contaminants that is extremely difficult to clean. The preferred approach is to prevent filter contamination during manufacture, and to develop detailed procedures for insuring cleanliness during handling and assembly into sorption compressors. Use of ultrasonic cleaning, followed by hot gas flushing and then vacuum bakeout, is recommended. For hydride coolers, hot bake-out in a hydrogen atmosphere followed by evacuation is recommended to chemically reduce oxide coatings on filters, plumbing components, as well as hydride materials, as discussed earlier, After integration, the entire system should be vacuum-baked to as high a temperature as can be tolerated. Because of the Ahrrenius temperature dependence of most outgassing sources, raising the bakeout temperature by 120”C can reduce the outgassing rate to the same level in 24 hours, as 11 days at the lower temperature level.[24] After completing the system bakeout, prolonged repeated hot fill/ flushes with high pressure refrigerant gas is recommended prior to the final fill.

Sorbent materials are ideal traps for many contaminants due to their high surface area, and in the case of chemisorption systems, their high reactivity. It is important to conduct repeated fill/ flushes with clean refrigerant gas followed by prolonged vacuum-bakeout to a temperature at least 50 K above the intended maximum cycling temperature to effectively desorb and outgas undesirable contaminants. Finally, the installation of gas purification filters or cold trap getters in the refrigeration loop plumbing is highly recommended to insure long-term system tolerance to residual contaminants.

SUMMARY AND CONCLUSIONS

Important progress has been made in experimental reliability physics investigations of various sorption cryocooler systems at the compressor, materials, and component-levels. Sorption cryocooler heaters have already demonstrated 10-year equivalent life in ongoing accelerated life tests, as have Inconel container materials. A closed-cycle PCO/O_2 compressor system for 65 to 90 K cryocoolers has logged over 35,760 hours and 79,567 cycles of maintenance-free operation with no degradation *in* oxygen pumping capability. Similarly, a Saran carbon/Kr compressor system for 120-140 K cryocoolers has accrued over 16,600 hours and 33,200 cycles. Similar hydride compressor system life tests have been initiated. Preliminary cycling data indicates some reduction in hydrogen pumping capability for both $LaNi_{4.8}Sn_{0.2}$ and ZrNi compressor systems, and the presence of 0.2 to 0.4% H_20 contamination, possibly due to chemical reduction of oxide coatings. However, full reversible absorption capacity was recovered after vacuum bakeout and reactivation. Future tests will focus on this area, as well as on developing a detailed understanding of important sorption cryocooler contamination issues. Although additional experiments are necessary to fully confirm long-life potential, testing to-date has been highly encouraging in demonstrating 10-year life capability of sorption cryocooler systems.

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