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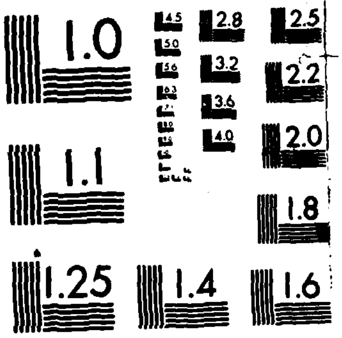

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Failure Analysis of a Sodium, Inconel 617 Heat Pipe

D. L. Jacobson and J. H. Wang

MECHANICAL AND AEROSPACE ENGINEERING
ARIZONA STATE UNIVERSITY
TEMPE, ARIZONA 85287

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Valerie J. Van Griethuysen
VALERIE J. VAN GRIETHUYSEN
Power Technology Branch
Aerospace Power Division
Aero Propulsion Laboratory

Hugh C Briggs
HUGH C. BRIGGS, Major, USAF
Chief, Power Technology Branch
Aerospace Power Division
Aero Propulsion Laboratory

FOR THE COMMANDER

JAMES D. REAMS
Chief, Aerospace Power Division
Aero Propulsion Laboratory

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A 4.57-meter (15-foot) sodium heat pipe, Inconel 617, was examined following failure. The sodium was exposed to the atmosphere following initial testing. Testing was resumed following attempts to reprocess the sodium by high-temperature outgassing. The pipe failed with intergranular cracks which completely penetrated the heat pipe wall leaking sodium to the atmosphere. Carbide precipitates and some grain boundary impurities were found in the cracked regions, which, along with the oxygen-contaminated sodium, embrittled the material. Thermal gradient-produced stresses during start-up coupled with embrittled grain boundaries appears to have resulted in the failure of the Inconel 617 sodium heat pipe.

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SECTION I

INTRODUCTION

A thermal power train was developed and tested by the Xerox, Electro Optical Systems Corp. for the Air Force Wright Aeronautical Laboratories Aero Propulsion Laboratory starting in 1971 and ending in 1974.[1] The thermal train consisted of a 4.57 meter sodium heat pipe mated to a secondary sodium heat pipe which was integral to a lithium fluoride thermal energy storage system. Mission considerations dictated that the thermal train be capable of energy storage and transmission. The system was designed to operate between 980K and 1200K and provide 3 kilowatts continuous and six kilowatts maximum thermal energy to a load between 810K and 980K. Initial performance and acceptance testing was accomplished by Xerox and the unit was put into storage in 1974.

In 1980 the system was transferred to ASU.[2] Sometime between the end of testing in 1974 and the transfer, all of the sodium fill valves were removed and brass swagelock caps were installed. It was estimated by Xerox personnel that the valves were removed just prior to shipping. During the removal, the primary heat pipe and the sodium joint were exposed to the atmosphere. The primary heat pipe is the subject of this investigation. It was noted by Xerox[1] that during fabrication the primary heat pipe was also exposed to the atmosphere through a fill tube weld crack which was repaired by brazing while the pipe was placed under an argon purge.

The heat pipe was 4.57 meters long with a 50.8 cm long evaporator, a 311 cm long adiabatic section and a 95 cm condenser. The Inconel 617 pipe had an outside diameter of 5.72 cm with an 0.165 cm wall thickness. The diametral wick consisted of 22 layers of 50-mesh Inconel 600 bolding cloth. A circumferential wick was made of 60-mesh Nickel 200 screen. Wick connector and retainer were made from Inconel 600. An 0.0025cm-thick Zirconium foil getter was installed. A schematic of the pipe cross section is shown in Fig. 1.

Extensive efforts were made to decontaminate the sodium by repeated evacuation by vac-sorb and vac-ion pumping and heating. The pipe was evacuated, then heated to 370K for 24 hours, cooled and evacuated to 10^{-6} torr. This process was repeated several times a week for over 5 months as the temperature was gradually increased and the internal pressure reduced. Eventually, 10^{-8} torr was maintained with molten sodium. Subsequent testing achieved the same performance obtained by Xerox 6 to 7 years earlier.[3]

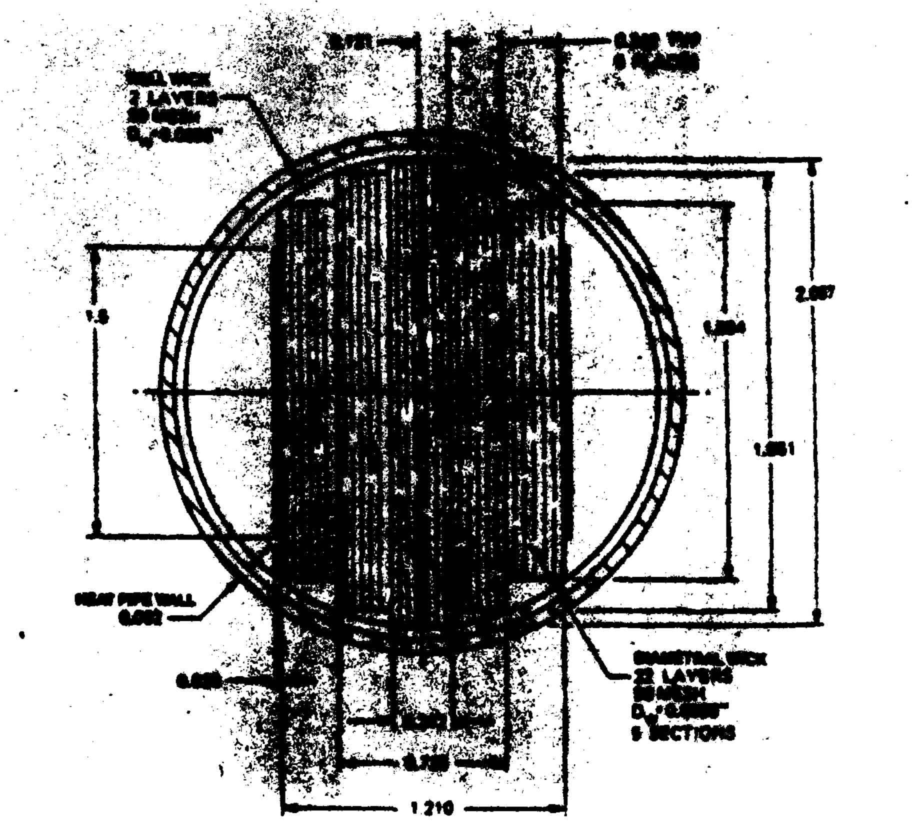

Figure 1. Cross-Sectional View of Primary Wick Structure.

During testing at ASU the heat pipe failed catastrophically with leaking sodium. Cracks extending through the wall were observed primarily in the evaporator section which allowed the leakage. Metallographic, Auger and Microprobe analyses were performed on post-failure test samples.

SECTION II

PRELIMINARY INVESTIGATION

The failure investigation was begun with the removal of the insulation and sheathed heater which was wound around the pipe. Figure 2 shows the evaporator section with heater wires around the evaporator section with leakage residue. The heater wire was removed and the pipe was then carefully cut into five sections, followed by immersion of the sections in Dowanol EB (Ethylene Glycol Butyl Ether) in order to dissolve the sodium remaining in the pipe. The Dowanol EB was changed every 2 days for 4 weeks until complete dissolution of sodium was accomplished. Figure 3 shows the cleaned evaporator section and the exposed cracks. Figure 4 shows a crack which severed the wall. The gap at the top is a lengthwise cut. Figure 5 shows a similar crack terminating at an apparent pit.

The cracks occurred within the 20 cm-long evaporator where the measured temperature was approximately 1035K. The actual maximum temperatures reached at the heater wire, pipe surface is not known.

The heat pipe sections were further sectioned to produce metallographic samples. Figure 6 shows the evaporator sectioned lengthwise and shows one part where the wall is cracked into two pieces. Corrosion products are visible on the internal surfaces. Figure 7 shows typical internal wall corrosion while Fig. 8 shows corrosion of the circumferential wick which was fused to the pipe wall in many areas of the evaporator. The diametral wick did not exhibit noticeable corrosion.

Figure 9 shows the edge of a cracked section which has the appearance of a brittle fracture.

Metallographic samples were prepared by polishing and etching with a mixture of one part HCI and one part HNO_3 with a 5 to 10 second etch. Microprobe samples were not etched. Figure 10 is a metallograph of a cracked region showing intergranular cracking and wall penetration. Severe corrosion on the inside walls of the tube accompanied deep cracks in several regions.

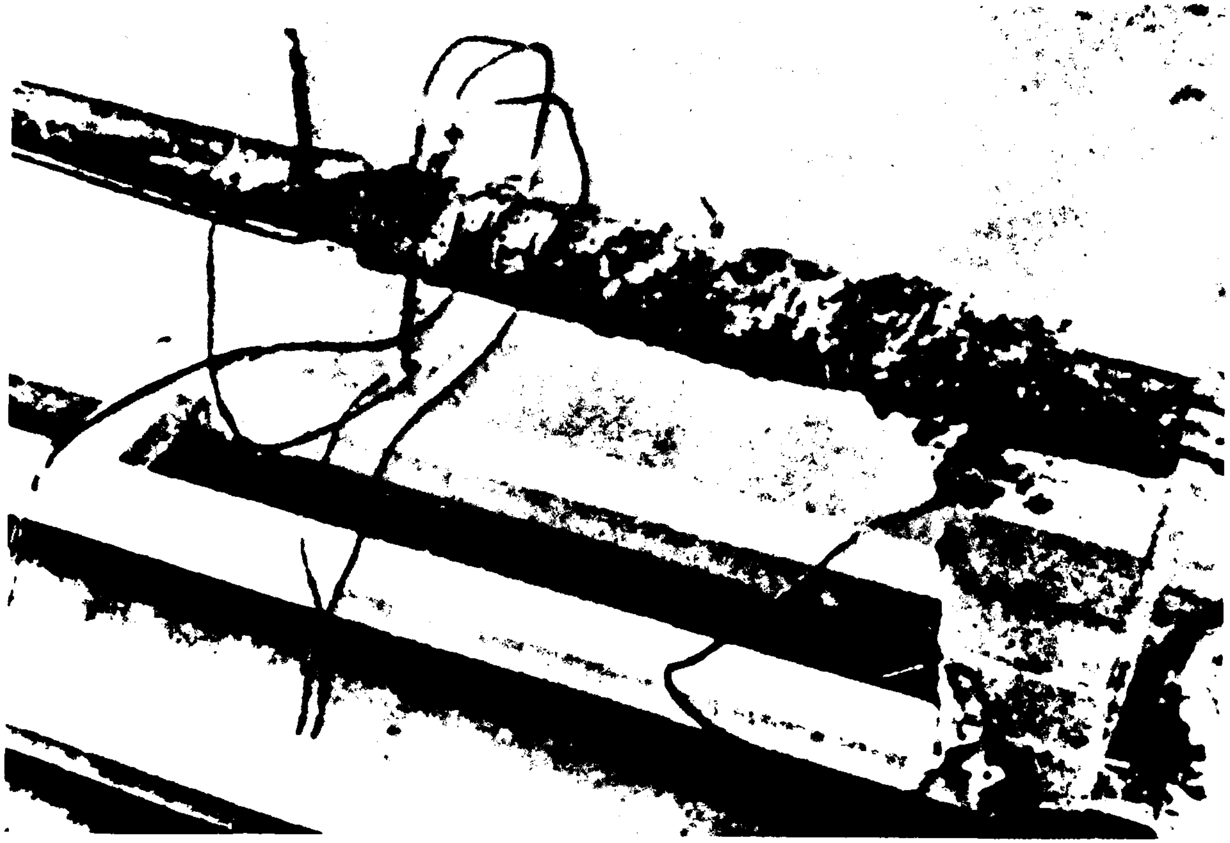

Figure 2. Failed Pipe with Heater and Residue.

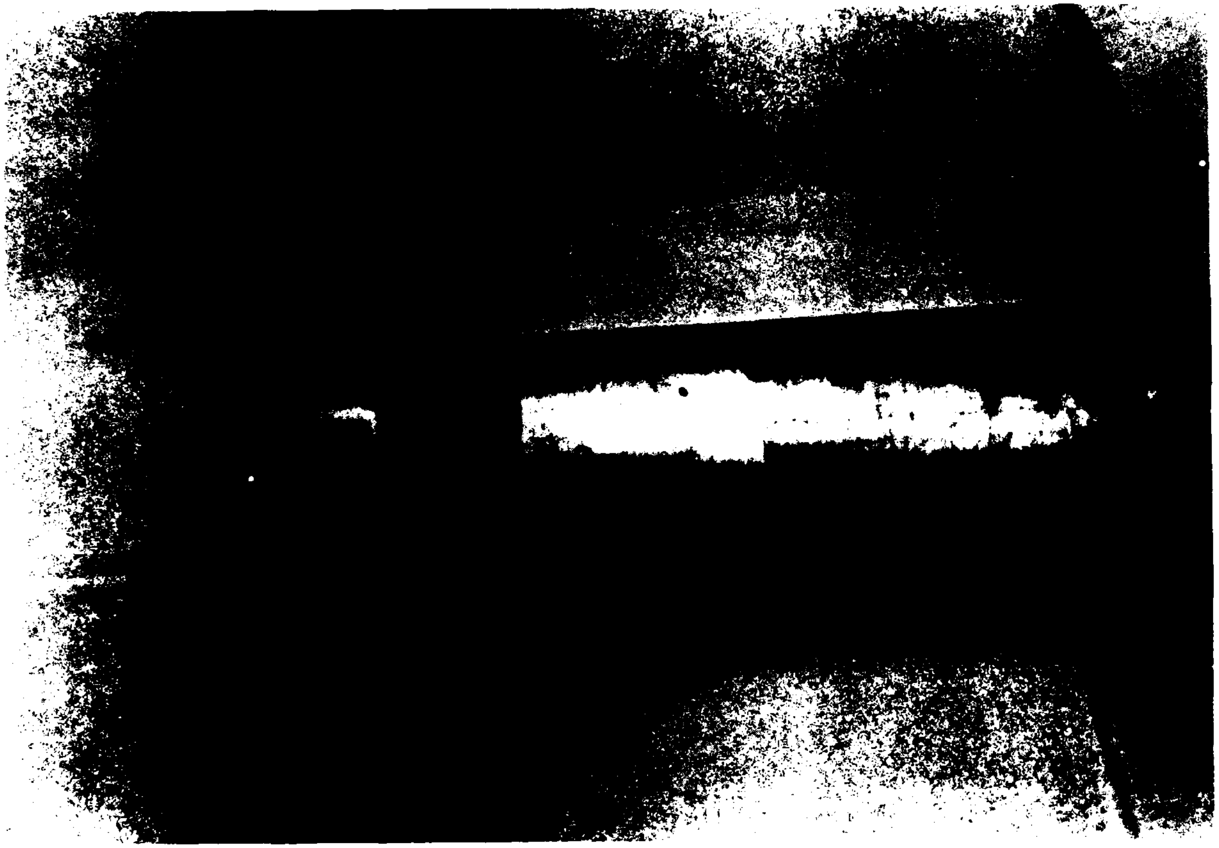

Figure 3. Post-Test Cleaned Evaporator Section.

Figure 4. Crack Penetrating Evaporator Wall.

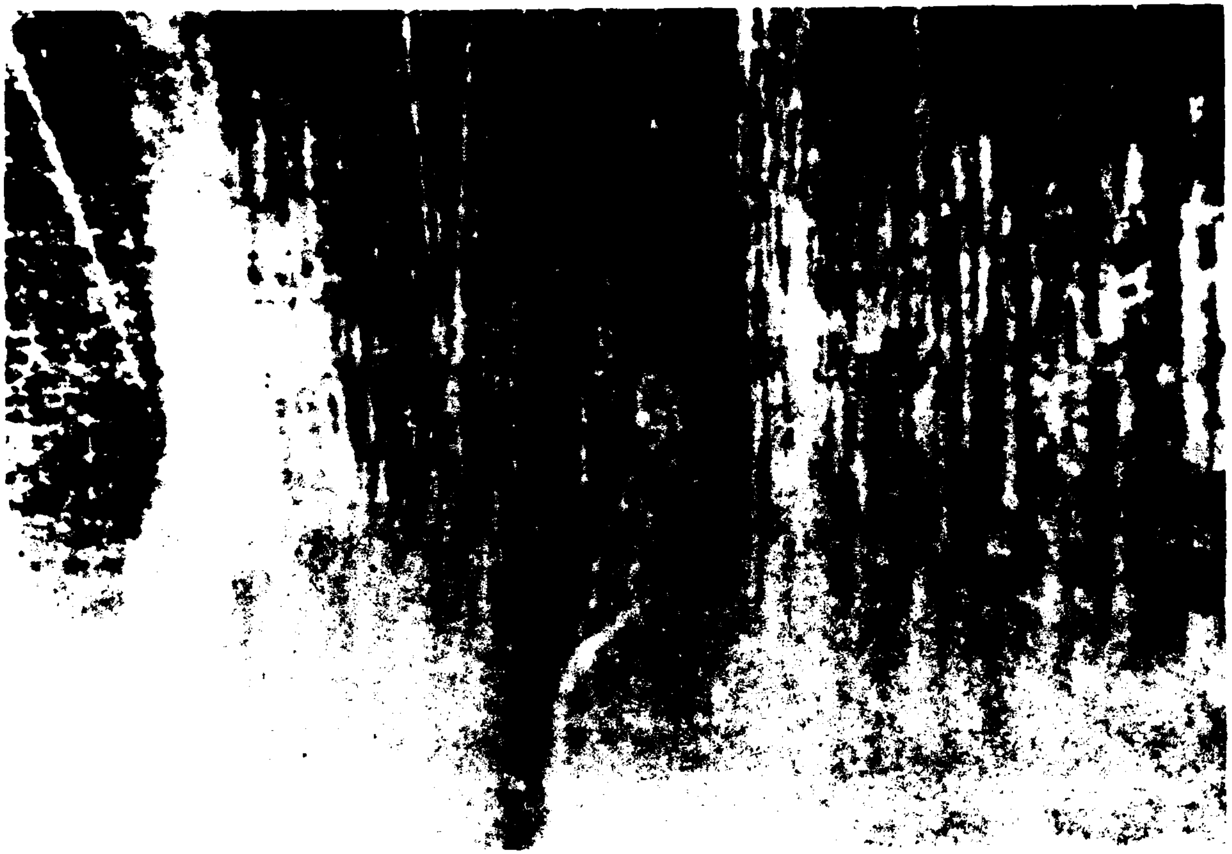

Figure 5. Crack Terminating at Apparent Pit.

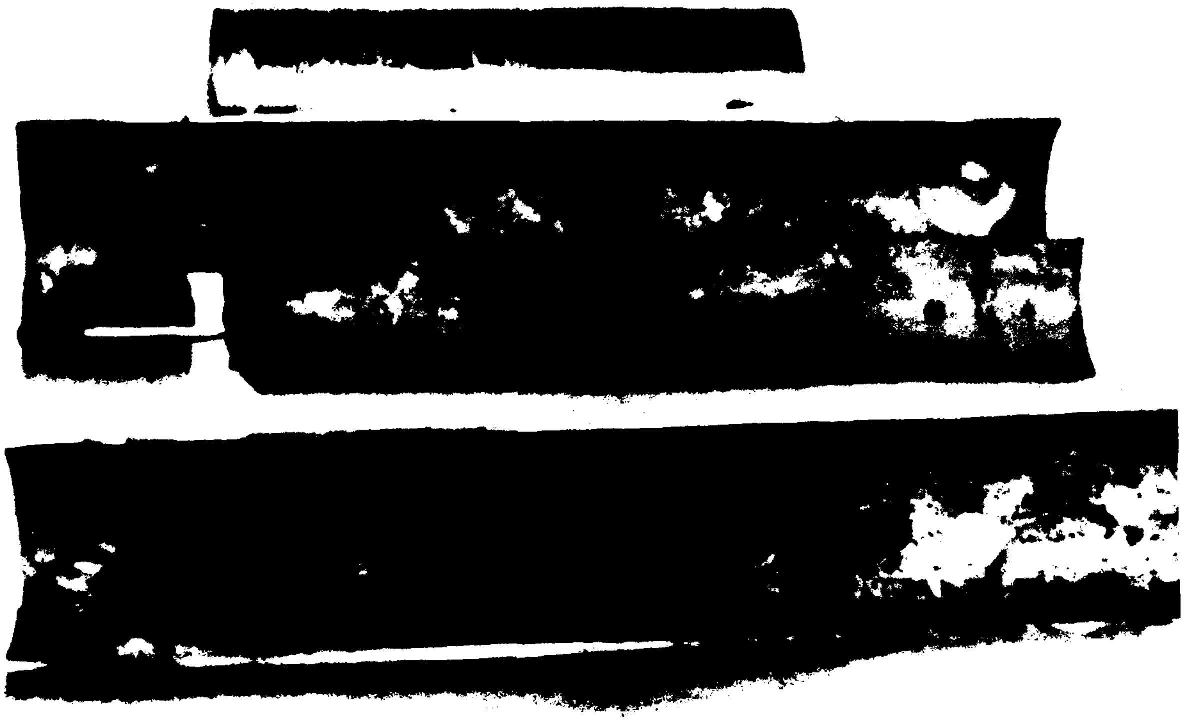

Figure 6. Evaporator Sections with Corrosion Products.

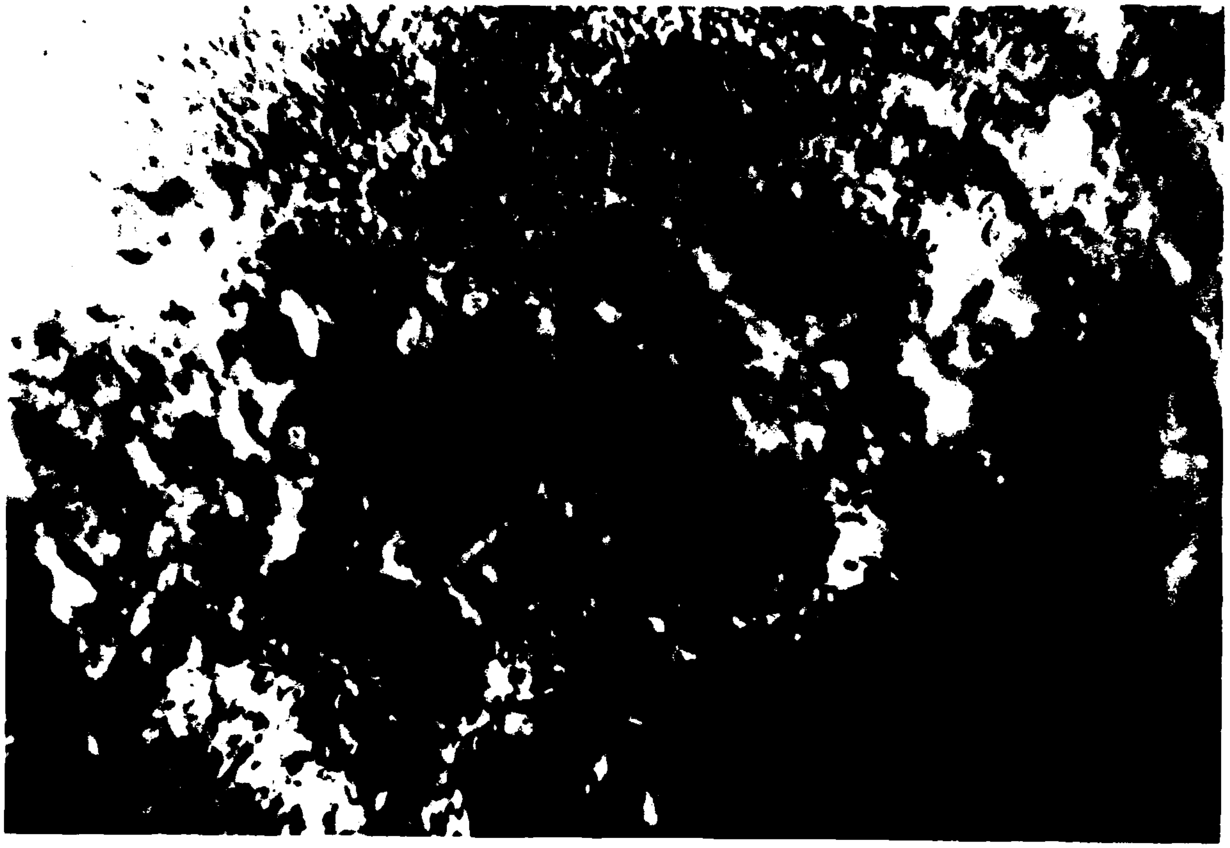

Figure 7. Internal Wall Corrosion.

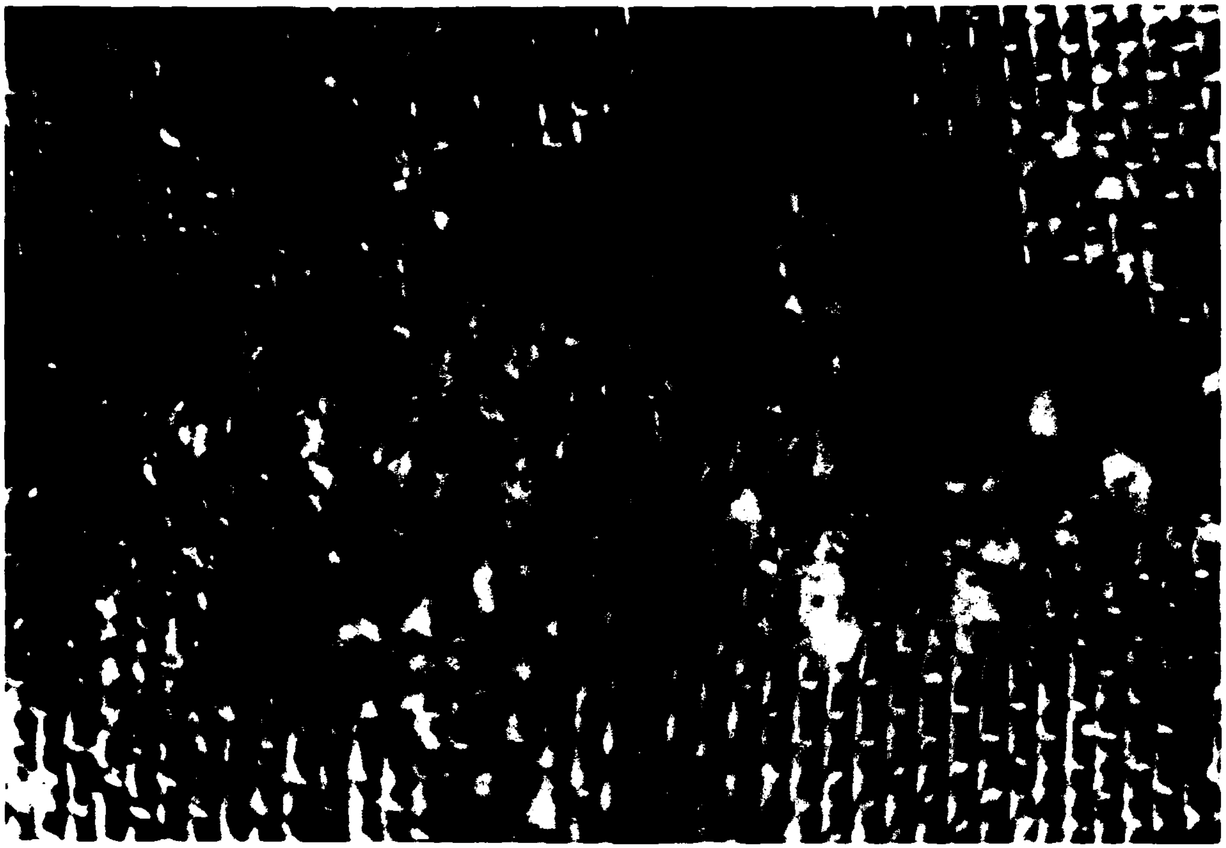

Figure 8. Circumferential Wick Corrosion.

Figure 9. Pipe Wall Crack Edge.

Figure 10. Intergranular Attack and Complete Wall Crack Penetration (100X).

SECTION III

MICROSTRUCTURE AND MICROPROBE EXAMINATIONS

Table 1 gives the nominal composition of I617 which is a face-centered-cubic solid-solution strengthening alloy with a nickel-rich austenitic matrix phase. The microstructure in the cracked region shown in Fig. 11 showed relatively large precipitates at some grain intersections.

Table 1

I617 Nominal Chemical Composition[4]
w/%

Nickel	52.0
Chromium	22.0
Cobalt	12.5
Molybdenum	9.0
Iron	1.5
Aluminum	1.2
manganese	0.5
Silicon	0.5
Titanium	0.3
Copper	0.2
Carbon	0.1

Microprobe analyses indicated that the composition was $Cr_{23}C_6$ which like sigma phase is brittle and will shorten the rupture life of the material. The phase is formed due to improper heat treatment whereby carbides migrate and accumulate at grain boundaries. Grain boundary cracks were found to coincide with these precipitates in many areas. Near the cracked areas microprobe analyses showed elevated levels of chromium and molybdenum. Small black particles in these areas were found to form continuous chains in the grain boundaries. The chains were estimated to be a combination of $Cr_{23}C_6$ and MO_6C. $Cr_{23}C_6$ will form as a precipitate in the grain boundaries during heat treatment in the 1033K to 1253K range. The continuous chain results in fracture paths and low impact properties. Distributed or non continuous chains on the other hand are beneficial in hindering grain boundary cracking.[5] Titanium carbide chains found in the material were the same throughout. Figure 12 shows precipitation from carbide accumulation.

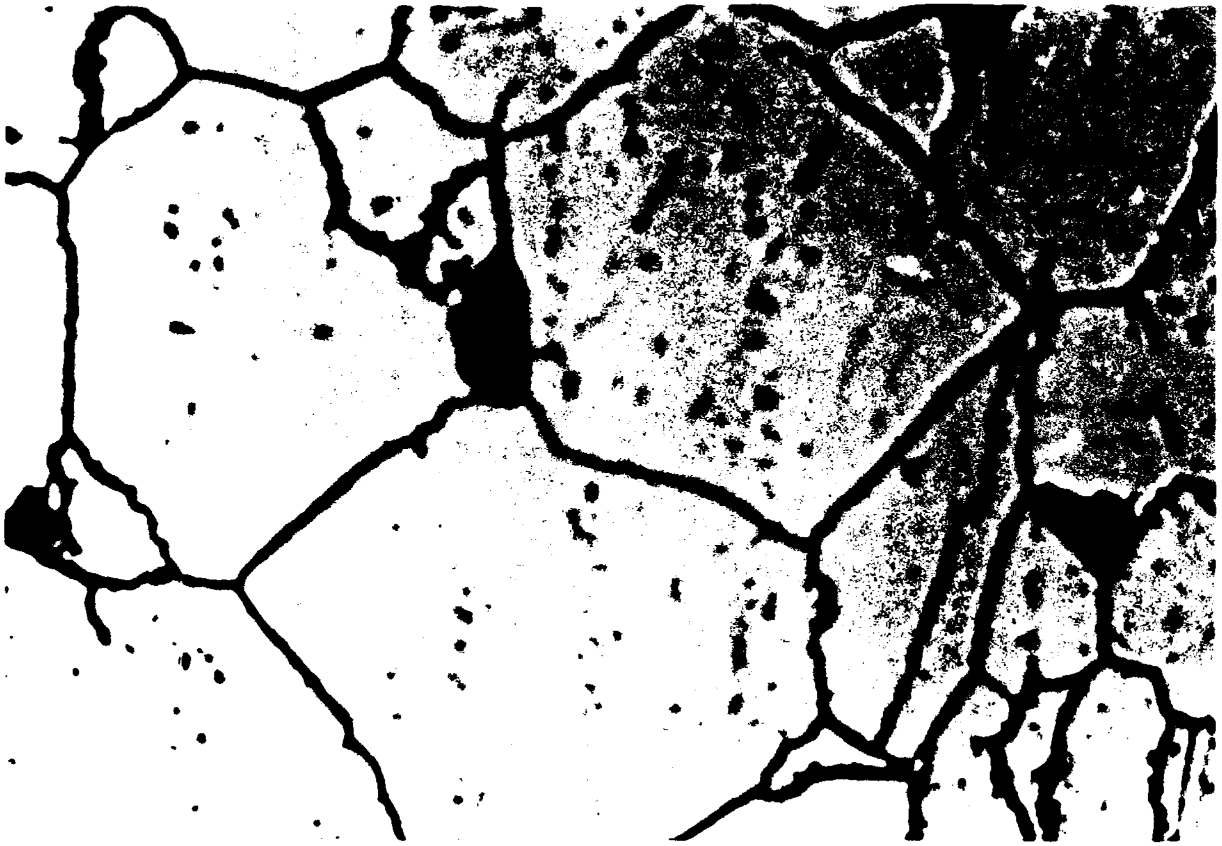

Figure 11. Evaporator Region Precipitates (400X).

Figure 12. Formation of Precipitates by Carbide Accumulation.

Metallography and SEM micrographs showed extensive intergranular attack. The sodium with its apparent high oxygen content produced the grain boundary penetration. Intergranular attack is shown in Figs. 10, 13 and 14. Part of the metallography and Scanning Emission Microscope (SEM) results were provided by the AFWAL Materials Laboratory.[6] In some areas, entire grains were found to be separated from the matrix. The associated surfaces were consequently very rough from the corrosion. As seen in the above micrographs the cracks were generated in an intergranular fashion.

Regions away from the evaporator did not exhibit noticeable grain boundary corrosion.

Microprobe analyses showed no difference in composition of the matrices between the evaporator and other areas of the heat pipe. However, the grain boundaries in the evaporator were rich in chromium and molybdenum, as discussed, in relation to the precipitate carbides. Iron was also enriched in the grain boundaries, again probably as a carbide. (Carbon was not able to be detected in the SEM).

Figure 15 is a microprobe picture of the grain boundary corrosion with the corrosion products. Phosphorous, sulphur and copper were found to be rich in the intergranular corrosion products. The phosphorous and sulphur can embrittle the structure. The phosphorous may have come from fabrication of the I617, or possibly from the brazing alloy, along with the copper, when the fill tube was repaired at Xerox. Figure 16 is a microprobe[6] picture showing the progress of the intergranular corrosion from the interior surface on the left side.

Aluminum and titanium were not detected on the exposed grain boundaries and the titanium carbides observed elsewhere remained unchanged in the corroded areas.

Sodium was not detected since it was dissolved in the Dowanol EB. Sodium was detected on a separated fracture surface where the Dowanol had not penetrated.[6]

The corrosion products external to the heat pipe were examined by microprobe and found to be rich in sodium, silicon, aluminum and titanium. The silicon came from insulation packed around the pipe. The pipe aluminum and titanium could have been oxidized by the sodium oxide and swept from the heat pipe by the leaking sodium.

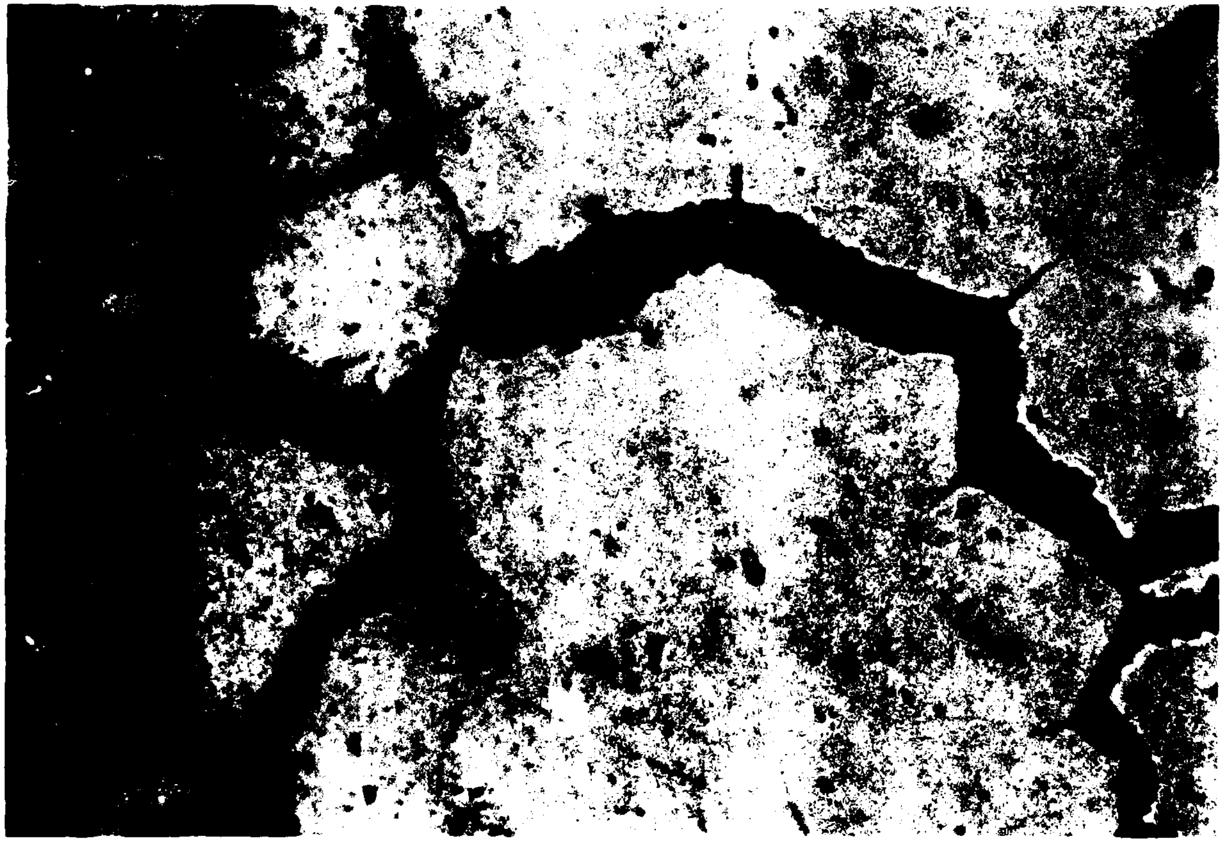

Figure 13. Grain Boundary Attack (500X)[6].

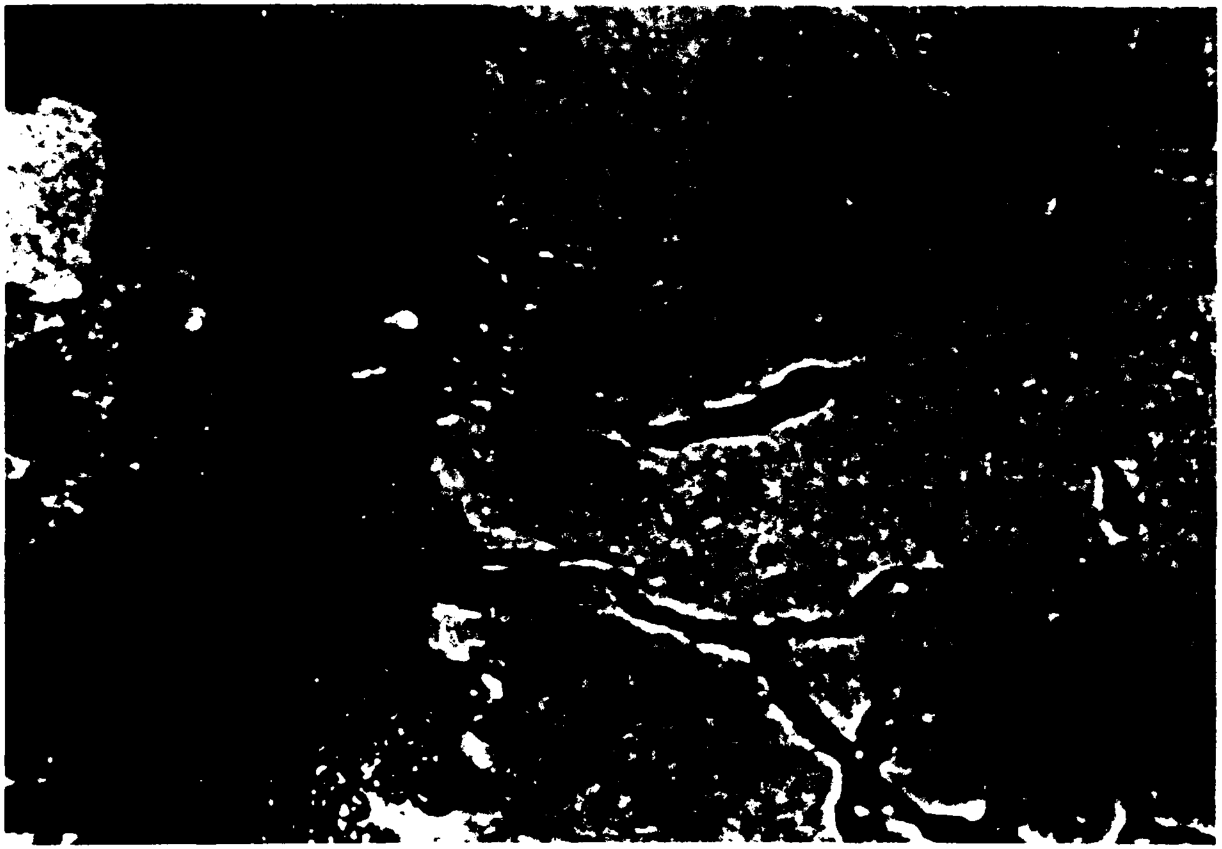

Figure 14. Grain Boundary Attack (400X).

Figure 15. SEM Micrograph of Intergranular Corrosion.

Figure 16. SEM Micrograph of Intergranular Corrosion (200X)[6].

SECTION IV

EVALUATION RESULTS AND CONCLUSIONS

Significant temperature gradients may have taken place during heat pipe start-up, producing stresses which could have contributed to wall cracking.[7,8] The heater wires in contact with the outer heat pipe walls forming the evaporator may also have been significantly higher than the measured wall temperature, also contributing to thermal stresses. The thermally produced stresses could have contributed to propagation of microcracks produced by sodium-oxygen attack[9] and precipitate embrittlement.[10,11,12,13]

The long term exposure to the high evaporator temperatures caused precipitation of cellular $CR_{23}C_6$ and other carbides forming continuous carbide chains which result in embrittled grain boundaries. Operation in the sensitizing temperature range resulting in such precipitate chains must be avoided. Microprobe analyses revealed that phosphorous and copper were present in the corroded intergranular material but were not present in the matrix grains. Sulphur and iron were found in elevated amounts compared to the base metal. Analysis of a separated fracture surface also revealed elevated amounts of copper and sulphur with trace amounts of phosphorous and sodium. The phosphorous probably came from original I617 alloy preparation although some phosphorous plus copper may have arisen from brazing materials. Phosphorous and sulphur embrittle the structure and should be avoided.

The sodium in the pipe was contaminated when exposed to the atmosphere. Although extensive efforts were made to reprocess the contaminated sodium, considerable impurities remained in the sodium. Only sodium oxide (Na_2O) was considered since other compounds like sodium peroxide (Na_2O_2) and sodium hydroxide (NaOH) would react with sodium to produce sodium oxide.[14] With a melting point of 1150K, the sodium oxide in the pipe would not melt at operating temperatures, and is thus swept to the evaporator, with grain boundary corrosion and penetration resulting.[14,15]

Pure sodium, according to the literature should not present a corrosion problem in I617 in this heat pipe application. However, if the sodium is contaminated by as little as 10 to 20 ppm oxygen it is known to produce liquid metal embrittlement and the corrosion can be appreciable. Therefore, oxygen in the sodium heat pipe system should be reduced to the lowest possible level.

It is concluded that the failure was caused by the combination of sodium-oxygen corrosion or liquid metal embrittlement and thermal stress corrosion aided by precipitates and impurity embrittlement.

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