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OXIDATION RESISTANT POROUS MATERIAL FOR TRANSPIRATION COOLED VANES

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Prepared by
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16. Abstract

Porous metal sheet with controlled permeability was made by space winding and diffusion bonding fine wire. Two iron-chromium-aluminum alloys and three nickel-chromium alloys were used: GE 1541 (Fe-Cr-Al-Y), H 875 (Fe-Cr-Al-Si), TD Ni Cr, DH 245 (Ni-Cr-Al-Si) and DH 242 (Ni-Cr-Si-Cb). GE 1541 and H 875 were shown in initial tests to have greater oxidation resistance than the other candidate alloys and were therefore tested more extensively. These two materials were cyclic furnace oxidation tested in air at 1800 and 2000° F (982 and 1093° C) for accumulated exposure times of 4, 16, 64, 100, 200, 300, 400, 500, and 600 hours. Oxidation weight gain, permeability change and mechanical properties were determined after exposure. Metallographic examination was performed to determine effects of exposure on the porous metal and electron beam weld joints of porous sheet to IN 100 strut material. Hundred hour stress rupture life and tensile tests were performed at 1800° F (982° C). Both alloys had excellent oxidation resistance and retention of mechanical properties and appear suitable for use as transpiration cooling materials in high temperature gas turbine engines. In particular, the GE 1541 alloy extends the useable temperature range by more than 300° F (149° C) as compared to the most common alloy in use today (DH 242).

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by Per Madsen[1] and Robert M. Rusnak[2]

THE BENDIX CORPORATION

1 SUMMARY

The objective of this work was to fabricate and test porous metal sheets of oxidation resistant alloys in order to determine their suitability for use in transpiration cooling of gas turbine vanes. Four alloys were evaluated and compared to an alloy commonly used in transpiration cooling — DH 242.

GE 1541	15Cr-4Al-1Y-Fe
H 875	23Cr-6Al-1Si-Fe
TD Ni Cr	20Cr-2ThO_2-Ni
DH 245	20Cr-1Si-5Al-Ni
DH 242	20Cr-1Si-1Cb-Ni

The first three alloys were selected on the basis of previous sheet and wire specimen screening tests as reported in references 1 and 2. The DH 245 alloy was selected as an improved version of DH 242 because of its aluminum addition. The alloys were obtained as 0.005 inch (0.13 mm) diameter wire and manufactured into Poroloy[3] porous metal with a wall thickness of 0.023 inch (0.58 mm).

In order to make the Poroloy, a new fabrication process was developed to overcome the difficulty in diffusion bonding the aluminum containing alloys. Also wire processing of the GE 1541 alloy was developed in cooperation with a commercial wire drawer. In preliminary static oxidation tests at 1800°F (982°C) DH 245 Poroloy exhibited less oxidation resistance than GE 1541 and H 875 Poroloy and was excluded from additional tests. The TD Ni Cr Poroloy showed a pronounced decrease in permeability following cyclic oxidation at 1800°F (982°C) for 600 hours. This coupled with the difficulty in processing fine wire caused the alloy to be dropped from the additional tests given the two most promising alloys — GE 1541 and H 875.

For the GE 1541 and H 875 alloys cyclic furnace oxidation tests were made on Poroloy sheet coupons at 1800 and 2000°F (982 and 1093°C) in air for accumulated exposure times of 4, 16, 64, 100, 200, 300, 400, 500 and 600 hours. The

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[3] Poroloy is a registered trade name describing space-wound wire porous metal material manufactured by The Bendix Corporation, Filter Division

specimens were cooled to room temperature after each exposure time. The specimens were measured to determine oxidation weight gain, permeability change, thickness change and room temperature mechanical properties after exposure. Metallographic examination of Poroloy and Poroloy/IN 100 weld samples was performed to show significant changes due to oxidation and heating.

Stress rupture and tensile testing at 1800°F (982°C) were performed on both 0.005 inch (0.13 mm) diameter wire and Poroloy.

Both alloys exhibited good oxidation resistance with no apparent spall. After 600 hours cyclic oxidation exposure, the GE 1541 Poroloy weight gain was 85 per cent higher at 1800°F (982°C) and 56 per cent higher at 2000°F (1093°C) than that of the H 875 Poroloy. However, the permeability decrease was the same for both alloys after 1800°F (982°C) exposure (20 per cent) and after 2000°F (1093°C) exposure, the GE 1541 Poroloy permeability decreased less than the H 875 Poroloy (22 per cent versus 44 per cent).

The GE 1541 oxidation resistance was found to be sensitive to processing conditions and contact with other alloys at elevated temperatures.

Retention of room temperature tensile properties following 600 hours of oxidation was good for Poroloy made from both alloys. In particular, the tensile elongation was equal to or greater than that prior to oxidation (GE 1541 — 10 per cent elongation and H 875 — 15 per cent elongation). The decrease in tensile and yield strength was less than 15 per cent. The 1800°F (982°C) tensile and stress rupture properties were comparable for both alloys with an ultimate tensile strength of about 2500 psi (17.2×10^6 N/m^2) and a 100 hour rupture stress of about 1500 psi (10.3×10^6 N/m^2).

Sound welds made between Poroloy and IN 100 strut alloy. The weld zones were stronger than the Poroloy and were not degraded by 1800°F (982°C) cyclic oxidation, however, exposure at 2000°F (1093°C) resulted in severe oxidation of the strut alloy and consequently the weld zone.

The good oxidation resistance, permeability retention and retention of mechanical properties at temperatures up to 2000°F (1093°C) makes the GE 1541 and H 875 alloys attractive for application as transpiration cooling materials for gas turbine vanes. In particular, the GE 1541 alloy at 2000°F (1093°C) has greater permeability retention after oxidation than the DH 242 standard alloy at 1700°F (927°C).

2 INTRODUCTION

This research and development work was conducted with the ultimate goal of producing porous metal sheet material with good high temperature oxidation resistance for application in the transpiration cooling of gas turbine engines.

2.1 Background

As reported in reference 3, the trend in both military and commercial gas turbine engines is towards turbofan engines having a compact, high temperature gas generator. To make such engines compact, lightweight, and with superior specific fuel consumption, there is the need to simultaneously increase both compressor pressure ratio and turbine inlet temperature. Turbine inlet temperatures may go to those corresponding to stoichiometric fuel-air mixture if materials and cooling designs can be developed that will tolerate such temperatures reliably.

Up to the present time, convection cooling has been the primary means of cooling gas turbine engines, with some film cooling augmentation in critical regions. At the severe cooling conditions expected in future engines, it is likely that convection cooling will be inadequate, and more advanced cooling schemes such as film and transpiration cooling will have to be utilized.

Theoretically transpiration cooling is superior to film cooling as shown in reference 4, provided the oxidation problems described below can be overcome. Also good creep life can be accomplished with transpiration cooled blade designs compared with other cooling designs as described in reference 5.

Transpiration cooling materials for turbine vanes, blades, shroud liners and similar components have been fabricated from fine wire about 0.005 inch (0.13 mm) in diameter, flattened, space wound in a geometric pattern and finally diffusion bonded to provide a porous structure — Poroloy[*] (See reference 6). The characteristics of high surface area to volume ratio and small pore size enhance cooling efficiency but aggravate oxidation problems. Oxidation corrosion which may be considered minor for solid or sheet metal components might seriously affect porous wall cooling air flow permeability and strength. Therefore, oxidation resistance is a primary design criterion for transpiration cooling material specification. Retention of good mechanical properties, especially ductility, after cyclic heating and aging is also important as well as the high temperature strength of the material. Accordingly, these properties are determined in this work.

Earlier work as described in references 1 and 2 was directed toward selection of alloys for testing in porous metal form. In reference 1, solid sheet specimens were evaluated on the basis of oxidation resistance and

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[*] Poroloy is a registered trade name describing space-wound wire porous metal material, manufactured by The Bendix Corporation, Filter Division.

mechanical properties. Four candidate alloys (N 155, TD nickel-chromium, DH 242 and Hastelloy X) were selected on the basis of the results of these tests for further evaluation. As reported in reference 2, these alloys were made into fine wire (0.005 inch (0.13 mm) diameter) and tested for oxidation resistance and mechanical properties. The testing of the alloys in wire form was a necessary prelude to fabrication of porous materials from these alloys because the oxidation behavior and mechanical properties could differ significantly between a given alloy in sheet and wire form. In addition, the difficulty in fabrication of good quality fine wire from each alloy was assessed. It was concluded that TD nickel-chromium showed the most promise and should be considered for evaluation in porous metal form. In addition, it was noted that alloy GE 1541 which had been tested in sheet but not wire form should be considered because of its superior oxidation properties.

2.2 Scope

Based on the earlier alloy selection work, this investigation was initiated to evaluate Poroloy made from GE 1541, TD Ni Cr and DH 242 and to supply NASA with sheets of the two best of these materials. Difficulty was encountered in procuring good quality TD Ni Cr wire in sufficient quantity for fabricating Poroloy and initial oxidation permeability test showed less permeability retention than GE 1541. Consequently this material was dropped from consideration in favor of two other candidate alloys, DH 245 and H 875. Both were selected for being readily available in fine wire form and for their expected good oxidation resistance; DH 245, a nickel-chromium type alloy similar to DH 242 except for the addition of aluminum and H 875, an iron chromium aluminum alloy which was shown in earlier work (reference 1) to have good oxidation resistance. Preliminary evaluation of the alloys in wire and Poroloy form led to the conclusion that only the GE 1541 and H 875 alloys should be evaluated extensively in porous metal form. The reason for this conclusion was that these alloys were the only ones that showed any promise of being able to operate at temperatures of 1800°F (982°C) and above, without oxidation sufficient to cause a significant loss of sheet permeability. However, the tenacious oxide layer formed by the alloys made diffusion bonding difficult and necessitated a process development for fabricating them into Poroloy. It was discovered during this process development that the GE 1541 alloy suffered accelerated oxidation at temperature levels where oxidation is normally slight in the bulk alloy. This poor oxidation resistance appeared to be introduced to the alloy during processing into wire form. An investigation was conducted to determine the cause of the loss of oxidation resistance and specifications were adopted for the alloy in wire and rod form to prevent such a loss from occurring in the future.

Evaluation of the GE 1541 and H 875 in porous metal form consisted of cyclic furnace oxidation tests at 1800 and 2000°F (982 and 1093°C) for exposure times of 4, 16, 64, 100, 200, 300, 400, 500 and 600 hours. After each exposure cycle, specimens were removed from the furnace and air cooled. Temperatures and times as well as general test procedures were the same as those reported in references 1 and 2. The oxidation effects were evaluated by measurement of weight gain, permeability change, thickness change and room temperature mechanical properties after exposure. Electron beam weld joints between Poroloy and IN 100 strut material were also evaluated in these tests. In addition, tensile and stress rupture properties were determined at 1800°F (982°C). Tests for permeability following oxidation at 1700 and 1800°F (927 and 982°C) and electron beam weld evaluations were made for the TD Ni Cr and DH 242 alloys. The results obtained for the DH 242 served as reference of comparison for the other alloys since DH 242 is the most commonly employed alloy for transpiration cooling today (reference 7).

2.3 Purpose

The purpose of this investigation was to evaluate the selected alloys in porous metal form in terms of potential application for transpiration cooling of gas turbine vanes. This purpose was to be accomplished by (1) developing procedures for producing oxidation resistant porous material from difficult-to-bond, oxidation resistant alloys, (2) evaluating electron beam weld joints of these materials to turbine blade strut alloys, and (3) experimentally determining the effects of oxidation on permeability and mechanical properties.

3 WIRE PROCUREMENT AND POROLOY PROCESS DEVELOPMENT

3.1 Wire Procurement

All wire for this program was made to a nominal diameter of 0.005 inch (0.13 mm) unless otherwise noted. The H 875, DH 242 and DH 245 alloy wires were purchased to commercial specifications from Hoskins Manufacturing Company and Driver Harris Company, respectively. A vacuum melted heat of GE 1541 was purchased from the General Electric Company and processed into fine wire by Hoskins Manufacturing Company. The chemical analysis of all alloy wires are presented in Table 1. The mechanical properties of the wires in the as-received, simulated sintered condition and after oxidation are given in Table 2.

3.1.1 Analysis of Accelerated Oxidation. — It was discovered during the development of bonding procedures for GE 1541 wire that bonded samples underwent accelerated oxidation after short exposure (24 hours) in air at 1800°F (982°C). The first impression was that some of the bonding techniques were contributing to the loss of oxidation resistance in the alloy since the bulk alloy oxidation resistance was excellent. However, it was quickly discovered upon oxidation testing of the GE 1541 wire prior to bonding that the poor oxidation resistance was inherent in the wire. Therefore an investigation was undertaken to determine the cause of the problem and to use this information to prepare a specification for the wire and the rod (from which the wire is drawn) which would prevent degradation of oxidation characteristics in the reduction of the alloy from bulk to wire form.

The first step in the analysis was metallographic examination. Photographs of the wire surface revealed the presence of transverse cracks along the wire which had poor oxidation resistance while the wire with good oxidation resistance had none (Figure 1). Examination of the 1/4 inch (6.4 mm) diameter rod from which the wire was drawn also revealed some surface cracks, but with a frequency much less than on the wire. Wires with poor oxidation resistance were found to regain oxidation resistance if, prior to oxidation, the wire was etched to remove all traces of the surface cracks. This indicated that the poor oxidation resistance was initiated by the surface layer containing the cracks. Samples with transverse surface cracks were found to oxidize more rapidly when they were exposed to hydrochloric acid and then oxidation-tested without cleaning the residue. Therefore, improper cleaning during wire processing could have contributed to its failure. However, this did not explain the presence of the cracks. It was thought that improper drawing procedures could be a cause (e.g., excessive reductions, improper lubrication).

The effect of annealing furnace atmosphere was investigated. Annealing the wire in hydrogen at 1900°F (1038°C) produced neither cracking nor greatly accelerated oxidation. However, the effect of nitrogen was pronounced. As shown in Figure 2, annealing in a nitrogen atmosphere for only 1/2 hour at 1900°F (1038°C) produced nitrides throughout the cross section of the wire. These nitrides embrittled the wire. Consequently, exposure to nitrogen was most probably the cause of surface cracking since the wires were annealed in cracked ammonia during processing. The exposure during processing was only the equivalent of about one minute static exposure and therefore, nitrides were not noted throughout the wire's cross section. However, formation in a thin surface layer would be sufficient to produce the cracking. Wires which were heated in the nitrogen atmosphere in the laboratory were then exposed to air at 1800°F (982°C) for 24 hours and found to suffer accelerated oxidation (Figure 3). Therefore, exposure of the wire during annealing to nitrogen-containing atmosphere was apparently the cause of the loss of oxidation resistance since wire processing performed in an inert (non-nitrogen) atmosphere yielded good quality wire. Examination of wire samples during various stages of wire drawing revealed that most of the transverse cracks which occurred in poor quality wire appeared after the final draw indicating this to be the most critical step in the process.

The loss of oxidation resistance after exposure to nitrogen appears to be a result of nitride formation with the alloying elements. General Electric has found that the GE 1541 alloy loses its good oxidation resistance when the aluminum level drops below 3.5 per cent. Apparently the formation of nitrides depletes the matrix aluminum to a concentration below this value.

3.1.2 Rod and Wire Specification. — Based on the results of this analysis, specifications were drawn up for the GE 1541 wire and rod (from which the wire is drawn) to prevent the loss of oxidation resistance during processing. The specifications were given Bendix Research Laboratories identification numbers and are designated as PS-985 (wire) and PS-997 (rod). The essence of the rod specification is that the aluminum and yttrium content of the rod be sufficiently high (4 per cent minimum aluminum and 0.5 per cent minimum yttrium) so that if some depletion occurs during wire drawing, it will not degrade the oxidation characteristics of the bulk alloy. In addition, the rod must be thoroughly inspected (visual and dye penetrant) for surface flaws and cracks prior to wire drawing. Radiographic examination for internal flaws is also required. The major requirements of the wire specifications are:

1. no nitrogen-containing, annealing atmospheres should be employed and reducing atmospheres should generally be avoided;

2. no phosphate wire drawing lubricants should be used since they can degrade oxidation resistance;

3. microscopic examination of the wire surface is required after various stages of wire drawing as a check for transverse surface cracks;

4. wire is oxidation-tested at 1800°F (982°C) for 24 hours after various stages of wire drawing in order to discover any loss of oxidation resistance;

5. wire must pass metallographic examination, oxidation testing and mechanical property requirements prior to final acceptance.

3.1.3 Wire Processing. — Starting stock was a 2 inch (51 mm) diameter rod, hot extruded at 1800°F (982°C) from a vacuum cast ingot. The rod was machined to remove surface flaws and then hot rolled at 2000°F (1093°C) to 1/4 inch (6.4 mm) diameter. After conditioning of the hot rolled rod in pickling solutions, wire drawing was done at room temperature using carbide dies through 0.032 inch (0.81 mm) diameter and diamond dies for the "fine wire" below 0.032 inch (0.81 mm) diameter. Area reduction between anneals ranged from 60 to 85 per cent for the fine wire. Intermediate anneals were carried out at 1400°F (760°C) in air for wire down to 0.069 inch (1.8 mm) diameter. Subsequent anneals down to fine wire size were performed at 1800°F (982°C) in air through a 12 foot long tube furnace at a wire speed of 90 feet per minute (0.46 m/sec). Fine wire was annealed at 1800°F (982°C) in an argon atmosphere. The finished wire was supplied in the as-drawn condition.

3.2 Poroloy Process Development

3.2.1 Poroloy Process. — Poroloy is a registered trade name describing space wound and diffusion bonded porous metal made by The Bendix Corporation, Filter Division.

The Poroloy process involves the wrapping of a flattened wire back and forth on a cylindrical or conical ceramic coated mandrel. Using the techniques of space winding, the mandrel becomes completely covered by a uniformly spaced pattern of wires accumulated to a desired thickness. The mandrel and winding are then sintered in a pure dry hydrogen or vacuum atmosphere for a period sufficient to accomplish complete bonding of all wires at contact points. After cooling, the winding is removed from the mandrel, rolled to desired density and resintered to further expand the bonded areas. If a flat sheet is desired, the cylinder is slit and flattened.

The pattern described on the mandrel by the initial point of contact between the mandrel and the wire during winding is a Lissajous figure. The winding is continuous. After a predetermined number of revolutions of the mandrel, the Lissajous figure is closed. This closure normally occurs after two, four or six thicknesses of wire have been accumulated. For convenience, the winding required to accomplish one closed figure is referred to as a layer; however, there are no true laminations since the process is continuous and uninterrupted.

The material used may be any alloy which can be drawn into wire and sintered. Certain alloys, notably those containing large quantities of aluminum, titanium and zirconium do not sinter readily.

The spacing of the wires, combined with rolling or cold working after sintering, controls both permeability and porosity. The wire size may be varied to control pore size and pore spacing.

The wind or pitch angle, i.e., the angle the wire helix makes with a plane normal to the axis of the mandrel, may be varied to control directional strength properties. Poroloy is an anisotropic material. The tensile strength of Poroloy in any one direction is dependent on the relationship of that direction to the bisector of the acute crossing angle and to the magnitude of the crossing angle. The most isotropic strength pattern is obtained with a wind angle of 45° (90° cross-over angle).

A photomacrograph of a typical Poroloy configuration for transpiration cooling applications is shown in Figure 4.

Figure 5 shows a Poroloy winding machine. As can be seen, the wire is taken from a spool and passed through a set of calender rolls where it is flattened into a ribbon. From there the ribbon passes over a pulley arrangement (where it is pre-stressed) and onto a shuttle that positions the ribbon on a rotating mandrel. The wind pattern is determined by the relative positions of the rotating mandrel and the oscillating shuttle.

<u>3.2.2 Diffusion Bonding Development</u>. — As mentioned earlier, alloys containing significant amounts of aluminum can not be diffusion bonded readily using conventional techniques. This is due to the formation of a tenacious aluminum oxide film which acts as a diffusion barrier. Therefore, considerable effort was directed toward the development of new techniques for bonding of GE 1541 and H 875 alloys which both contain approximately 5 per cent aluminum. The variables considered and investigated were:

1. Joint surface preparation.
2. Contact pressure.
3. Furnace atmosphere.
4. Bonding temperature.
5. Time at temperature.

Joint surface preparation: In all diffusion bonding processes it is important that the joint surfaces are clean and in intimate contact and remain so during the bonding cycle. In some cases a bonding aid in the form of the introduction of another metal into the joint is advantageous. This can serve several functions.

a. It can prevent the formation of oxide films prior to and during the bonding cycle.
b. It may form a liquid phase in the bond interzone thereby relaxing the requirements for contact pressure, temperature, and time.

A number of bonding aids were investigated: copper, chromium and carbon. A continuous cleaning and electroplating line was built to plate the 0.005 inch (0.13 mm) diameter wire and techniques and quality control methods were developed to ensure a dense and adherent plate. Carbon was applied using a gas carburizing method. The introduction of carbon was considered since thermodynamic calculations indicate that carbon can reduce aluminum oxide under vacuum diffusion bonding conditions.

In the final analysis it was found that the chromium was of no help and that carbon prevented bonding. Copper was of negligible help and was left out to prevent contamination of the alloy.

Contact pressure: The contact pressure between the wires is normally achieved by winding the wire under tension below the yield point. For the iron-chromium-aluminum alloys this proved to be insufficient. Therefore, an overwrapping technique was developed to increase the contact pressure during bonding. The overwrap consisted of a 0.015 inch (0.38 mm) diameter molybdenum wire which was wound over the Poroloy in a close helix.

The mandrel materials investigated were: Zirconia ceramic, alumina ceramic, stainless steel and mild steel. The ceramic mandrels were found to be inferior to the ferrous mandrels with respect to contact pressure because of their lower coefficient of thermal expansion.

When Poroloy was wound on mild steel (1010) mandrels some of the chromium from the wire diffused into the mandrel. This problem was eliminated when stainless steel mandrels were used because stainless steel has a chromium content comparable to that of both GE 1541 and H 875.

When mild steel mandrels were used they could be removed in nitric acid, but stainless steel mandrels could not be leached out. Therefore, the mandrels were coated with alumina before wrapping so that the bonded Poroloy would release.

In order to prevent the diffusion of molybdenum into the Poroloy a thin stainless steel foil was placed between the Poroloy and the wrapper. It was coated with alumina to prevent sticking. This wrapper has two other advantages. It prevents Cr and Al from vaporizing and being pumped out and it also limits the amount of oxygen coming into the vicinity of the Poroloy.

Furnace atmosphere: Attempts to use high quality dry hydrogen atmospheres (dew point less than -80°F) were unsuccessful because of discoloration and poor ductility bonds as measured qualitatively in bend tests. Successful bonding was achieved in a vacuum equal to or better than 10^{-4} Torr.

Bonding temperature: After the winding and overwrapping techniques were developed sample mandrels were made at bonding temperatures between 1900 and 2300°F (1038 and 1260°C) to determine an optimum bonding temperature. The optimum temperature was found to be 2150 to 2200°F (1178 to 1204°C). Lower temperatures did not produce adequate bond area and at higher temperatures grain coarsening takes effect.

Time at temperature: Two hours was sufficient, and one hour was found to be a minimum and acceptable only if a second bonding cycle is used after the Poroloy is densified.

Well-bonded Poroloy was produced using several of the investigated techniques. The optimized procedure used for diffusion bonding Poroloy made from aluminum containing alloys is summarized below:

1. Joint surface preparation: As-received wire in the hard drawn condition was used. It was found that bonding aids were not necessary if sufficient contact pressure was used during bonding.

2. Contact pressure: Sufficient contact pressure was achieved from differential thermal expansion when using molybdenum over-wrap and a type 304 stainless steel mandrel.

3. Furnace atmosphere: Vacuum 10^{-4} Torr or better.

4. Bonding temperature: 2175 to 2200°F (1190 to 1204°C).

5. Time at temperature: 1-1/2 hours.

4 EXPERIMENTAL PROCEDURE

4.1 Specimen Preparation

4.1.1 Wire Samples. — Wire samples of alloys GE 1541, H 875, DH 245 and TD Ni Cr were inspected and prepared for testing. All wires were 0.005 inch (0.13 mm) diameter within tolerances of ± 0.0003 inch (0.008 mm). The wire specimens were heat treated before testing to simulate annealing and grain growth effects expected in fabricated Poroloy structures. The heat treatment except for TD Ni Cr consisted of a 1-1/2 hour cycle at 2200°F (1204°C) in a vacuum of better than 10^{-4} Torr followed by cooling in argon. TD Ni Cr was simulated sintered in hydrogen for 4 hours at 2200°F (1204°C). Wires were fixtured in furnace racks to minimize contact and kinks to provide a straight, undistorted sample for stress rupture and tensile testing.

4.1.2 Poroloy. — Poroloy was made from 0.005 inch (0.13 mm) diameter wire flattened to a ribbon approximately 0.002 inch (0.051 mm) thick by 0.009 inch (0.23 mm) wide and wound with a spacing of 0.012 inch (0.31 mm) and a cross-over angle of 60 degrees. After diffusion bonding the resulting Poroloy tube was slit and opened into a sheet. The sheet was calendered to desired permeability and resintered to enhance diffusion bonds.

The fabricated thickness was 0.023 inch (0.58 mm) nominal. All Poroloy was made to have a permeability corresponding to an air flow rate of 0.005 lb/sec-in^2 (3.52 kg/sec-m^2) for an inlet pressure of 24.7 psia (170 x 10^3 N/m^2) and a discharge pressure of 14.7 psia (101 x 10^3 N/m^2) at an air temperature of 70°F (21°C).

4.1.3 Weld Samples. — Weld test specimens were made from Poroloy made of GE 1541, H 875, DH 242 and TD Ni Cr alloys electron beam welded to simulated strut specimens of IN 100 and Inconel 713C alloys. The strut specimens were machined to have four lands 0.060 inch (1.5 mm) deep to simulate a turbine blade or shroud configuration. The Poroloy was fixtured to the strut material and electron beam welded to the lands using the following weld parameters: 75 kilovolts, 2.5 milliampere and 30 inches per minute (1.3 cm/sec) weld speed with the beam focused on the surface of the Poroloy. The welds were evaluated by metallographic examination.

4.1.4 Oxidation Specimens. — Three permeability specimens, two by two inches, were made for each of the GE 1541, H 875, TD Ni Cr and DH 242 alloys. The following samples were fabricated only for the GE 1541 and H 875 alloys since these were the two most promising alloys for which more extensive evaluation was desired. For each alloy-time-temperature combination, three tensile and weight gain specimens 0.5 x 6.0 inches (13 x 152 mm) were cut with the 6 inch (152 mm) side parallel to the cross-over angle bisector. One of these had a small tab of IN 100 alloy attached by electron beam welding. Each of these specimens was measured to determine its actual size and area. Thickness was measured with a micrometer using a pin anvil. Measurements were made at three points along the specimen length to the nearest 0.0001 inch (0.003 mm). Specimen weight was determined to ± 0.1 mg with an analytical balance.

Joint tensile specimens were prepared by electron beam welding a 0.5 inch (13 mm) wide strip of Poroloy to an IN 100 strut material. Three specimens of each alloy were prepared for each of the following conditions: before exposure, after 100 and after 600 hours exposure. A sketch of all oxidation specimens is shown in Figure 6.

4.1.5 Poroloy Stress Rupture and Hot Tensile Specimen. — These specimens were sheared to 0.75 x 3.5 inches (19 x 89 mm) with the long side parallel to the cross-over angle bisector. Half of the hot tensile specimens were oxidized for 24 hours at 1800°F (982°C) in the air prior to testing. The machined specimens had a typical tensile specimen shape with a width of 0.375 inch (9.5 mm) and a gage length of 1.0 inch (25.4 mm).

4.2 Oxidation

4.2.1 Apparatus. — Specimens were contained in zircon ceramic thimbles during oxidation cycling to collect spall and avoid extraneous contamination. The thimbles were shaped like test tubes 0.88 O.D. x 0.75 I.D. x 7 inches (22 x 19 x 178 mm) long with a flat disc lid and having four 0.13 inch (3.3 mm) air-circulating holes drilled near the top and bottom as shown in Figure 7. Preliminary tests with mild steel showed that this arrangement allows sufficient air convection so that results are essentially equivalent to open air oxidation. Zircon base ceramic material ($ZrO_2 \cdot SiO_2$, Leco 528-125) was chosen to minimize fluxing or other interaction between metal oxides and the thimble. Specimens were placed in the thimbles with four-point contact at the corners of the specimens. Thimbles were vertically supported in a 2 x 7 array on a special rack made from 0.19 inch (4.8 mm) diameter type 330 stainless steel wire. Nine racks, one for each exposure time cycle, were arranged on a moveable skid-pan. Thermocouples of No. 14 gauge Chromel-Alumel were located several places on the skid. Figure 7 shows specimen and rack layout and thermocouple positions. An automatically controlled and recording electric furnace with 13 x 16 x 48 inch (330 x 407 x 1220 mm) chamber was used to maintain temperatures within ± 1 per cent of the nominal setting.

4.2.2 Schedule. — Oxidation cycling procedures consisted of adjusting the furnace to the required temperature with a proportional controller set to minimize temperature fluctuation. Prepared sample skid-pans were loaded with a forklift dolly into the furnace and power was increased and then backed-off to "meet" the temperature and minimize lag. Full furnace recovery time was less than one hour for all runs. After exposure for the required time, the skid-pan was removed and all sample sets were cooled to room temperature in about one hour in still air under ambient conditions. The assigned rack was then removed for later examination and the other samples were returned to the furnace. This procedure was repeated for each time interval 4, 16, 64, 100, 200, 300, 400, 500 and 600 hours and for each temperature of 1800 and 2000°F (982 and 1093°C) for the GE 1541 and H 875 alloys. The TD Ni Cr and DH 242 alloys were oxidized at 1700 and 1800°F (927 and 982°C).

4.3 Oxidation Specimen Examination and Testing

After oxidation exposure of the GE 1541 and H 875 Poroloy materials, the following characteristics were determined after each temperature-time period:

1. Total oxidation weight gain
2. Permeability
3. Thickness change
4. Mechanical properties at room temperature
5. Joint tensile strength
6. Metallographic examination of Poroloy and weld joints.

Only permeability was determined for the Td Ni Cr and DH 242 alloys.

4.3.1 Weight Gain. — The samples were weighed to 0.1 mg to determine total oxidation weight gain. Data was obtained as the difference between original weight and weight after oxidation. Oxide spalling was not observed in any of the tests.

4.3.2 Permeability. — The permeability of each specimen was measured in the as-fabricated condition and after each exposure time interval. In this work, permeability is defined as the air flow rate for an inlet pressure of 24.7 psia (170×10^3 N/m^2) and a discharge pressure of 14.7 psia (101×10^3 N/m^2) at an air temperature of 70°F (21°C). Probe diameter was 1.129 inch (28.7 mm) corresponding to one square inch of area. The air flowing through the specimen had a temperature of about 70°F (21°C) and was filtered through a five micron nominal filter. The volumetric flow was measured on the downstream side using a rotometer with an accuracy within 1 percent of full scale.

4.3.3 Thickness Change. — After weighing, the specimens were measured with the micrometer at the same reference points shown in Figure 6 to determine gross changes in thickness due to oxidation.

4.3.4 Mechanical Properties. — Each of the three oxidation specimens from each time-temperature-alloy combination were cut into two pieces of four and two inch lengths respectively. One of the two inch length specimens was bent around a radius three times the thickness (1/8 inch (3.2 mm) diameter was used) to determine the angle of bend at which fraying or fracturing would occur. All the four inch length specimens were tensile tested at a constant rate of 0.050 inch/minute (0.02 cm/sec) on an Instron testing machine to provide a continuous stress-strain curve. Ultimate tensile strength and yield strength (0.2 percent off-set) were determined on the basis of measured gross specimen thickness before oxidation. Elongation and modulus of elasticity were measured from the stress-stain plot. Identical tests were conducted on as-fabricated Poroloy.

The joint tensile tests were performed at the same crosshead speed to give a measure for the shear strength of the Poroloy/strut lap joint.

4.3.5 Metallographic Examination. — Poroloy and Poroloy/strut alloy weld samples were examined metallographically to determine the nature and extent of oxidation as well as microstructural changes which occurred as a result of the heat treatment. All metallographic samples were mounted with cold-mount material so as to avoid sample distortion and to preserve oxidized layers. Stainless steel shims were mounted with the Poroloy to aid edge retention in polishing. Standard metallurgical polishing procedures were employed with finish polishing performed with 0.3 μm alumina. Most of the oxidized specimens were unetched in order to highlight the degree of oxidation. Etched microstructures of all Poroloy weld samples in the unoxidized condition were prepared. The etchants used are summarized in Table 3. Photographs were made on either a macro camera or a metallograph with normal reflected light at appropriate magnifications to emphasize the features of interest.

4.4 Stress Rupture and Hot Tensile Tests

Stress rupture lives up to 100 hours were determined for GE 1541 and H 875 alloys both as 0.005 inch (0.13 mm) diameter wire and as Poroloy. The Poroloy tests were performed at 1800°F (982°C). Wire testing was performed at 1800 and 2000°F (982 and 1093°C). Wire tests were performed on specimens of about two feet in length with about six inches contained in the hot zone. Apparatus consisted of a horizontal tube furnace with six thermocouple stations within the hot zone. Test zone temperatures were continuously recorded. Wire specimens were strung through the split shell furnace and one wire end was fastened with split shot to a spring-loaded micro-switch which controlled an automatic interval timer. The other wire end was dead weight loaded over a ball bearing pulley. The pulley system was tested for drag and was found to contribute less than 10.0 mg error. Four wires were tested simultaneously with four parallel switch-pulley systems. A schematic of the test apparatus is shown in Figure 8. Tests were made to provide three to four data points for each alloy within the 100 hour time range. Tensile testing of both wire and Poroloy specimens of GE 1541 and H 875 alloys were performed at 1800°F (982°C).

4.5 Weld Examination

Electron beam welds of GE 1541, H 875, DH 242 and TD Ni Cr Poroloy to IN 100 and Inconel 713C strut alloys were examined metallographically. The basis of evaluation was freedom from cracks, porosity, undercutting, surface irregularities and detrimental microstructural changes.

5 RESULTS AND DISCUSSION

5.1 Static Oxidation Tests

Static oxidation tests, 24 hours at 1800°F (982°C), were carried out on wire and Poroloy samples to screen them for oxidation resistance. The poor oxidation resistance of DH 245 Poroloy material in this test was the reason it was dropped from consideration for cyclic oxidation testing. Photomicrographs of the oxidized wires and Poroloy are shown in Appendix F. Also included in this Appendix are the weight gain results from these tests.

5.2 Cyclic Oxidation

5.2.1 Weight Gain. — The specific oxidation weight gain for GE 1541 and H 875 Poroloy at 1800 and 2000°F (982 and 1093°C) is shown as a function of time in Figure 9 (specific weight gain is based on the apparent surface area and does not include the internal area of the porous structure). These results are tabulated in Appendix A and graphically illustrated with data points in Appendix B. The latter plots have some data points that are definitely lower than the straight line fit to the data. These low points correspond to samples that were oxidized at a lower temperature than those which fall on the curve. This is a consequence of the temperature gradient in the furnace relative to the specimen layout as shown in Figure 7. No oxide spalling was observed under any of the test conditions. At both temperatures the GE 1541 material gained significantly more weight than the H 875 material - 85% more at 1800°F (982°C) and 56% more at 2000°F (1093°C). The weight gain data should reflect the propensity of the Poroloy material for plugging. However, as will be discussed in the following section on permeability results, the oxidation weight gain data does not correlate with the decrease in permeability following exposure.

The straight line plot of weight gain versus time on log paper indicates a relationship of the form $\Delta w = kt^n$ when Δw is the weight gain, t the exposure time with k and n as constants at a given temperature. The exponent n was found to be approximately the same for GE 1541 and H 875 at a given temperature with a value of 0.27 at 1800°F (982°C) and 0.35 at 2000°F (1093°C). The fact that the exponent is less than 0.5 sometimes indicates that a single diffusion process is not controlling the growth. Other investigators have attributed this to internal oxidation of the yttrium in GE 1541. However, our results showing H 875 and GE 1541 as having the same exponent refute this claim and indicate that in both alloys oxidation is governed by the same physical process. Since H 875 does not contain yttrium, the low value of n may not be attributed to yttrium oxidation. Therefore, the mechanism and kinetics of oxidation appear little affected by the yttrium in GE 1541. As reported in reference 8 the oxide scale of both alloys is found to be predominantly α Al_2O_3. However, the yttrium may influence the kinetics of oxidation in a dynamic oxidation test (similar to conditions in a jet engine) in which the propensity for spalling is greater. In this case the GE 1541 kinetics may be slower relative to the H 875 alloy because yttrium promotes scale adherence (references 9 and 10).

5.2.2 Permeability. — The permeability, expressed as a percent of the air flow prior to oxidation, is presented in Figure 10 for the GE 1541 and H 875 alloys. These data are presented in tabular form in Appendix A and in graphical form with data points in Appendix B. After 600 hours total exposure at 1800°F (982°C) both alloys have approximately the same permeability - 80% of the pre-oxidized value. At 2000°F (1093°C), the permeability of the GE 1541 material is 78% of the pre-oxidized value while that of the H 875 is only 56%. Although both alloys have approximately the same retention of permeability following 1800°F (982°C) oxidation for 600 hours, the kinetics of change are not the same. The GE 1541 material decreases rapidly during the first 4 hours of oxidation exposure and then remains relatively constant up to test termination after 600 hours exposure. In contrast, the H 875 Poroloy exhibits a gradual but declining rate of decrease during the entire exposure period. The rapid decrease and then relatively constant permeability level of the GE 1541 Poroloy suggests that the permeability could be maintained approximately constant during application in vane cooling by pre-oxidation of the Poroloy prior to installation into the engine. This approach is also suggested in reference 11.

These results are not consistent with the weight gain results reported in Section 5.1.1 since the permeability would be expected to decrease as the oxidation increased. It was initially thought that the higher weight gain of the GE 1541 Poroloy without the attendant permeability drop could be attributed to internal oxidation. However, metallographic examination of the Poroloy (see Section 5.1) revealed that the surface oxide layer did increase with time and was in fact thicker than that of the H 875. The explanation for the relative constancy of the GE 1541 Poroloy is as yet unknown. The permeability results for the TD Ni Cr and DH 242 alloys, following oxidation at 1700 (927°C) and 1800°F (982°C), are presented in graphical form with data points in Appendix B. The permeability retention of these alloys is clearly less than that of the Fe-Cr-Al alloys. A graphical comparison of permeability versus oxidation exposure is made for the GE 1541 alloy at 2000°F (1093°C) and the DH 242 and TD Ni Cr alloys at 1700°F (927°C) in Figure 11. This graph illustrates that the GE 1541 alloy has more permeability retention at 2000°F (1093°C) than the nickel base alloys have at 1700°F (927°C).

5.2.3 Thickness Change. — Gross thickness changes are recorded as a function of oxidation exposure in Appendix A. The thickness generally increases with progressive oxidation. However, the results show considerable scatter because of the difficulty in measuring small thickness changes with a micrometer.

5.2.4 Bend Tests. — Bend test results are summarized in Appendix A. All samples, both as-fabricated and following oxidation under all conditions, could be bent 180° on a 3T (three times the thickness) radius without evidence of fracturing or fraying. The oxide appeared to adhere well.

5.2.5 Tensile Strength. — Room temperature tensile tests were conducted for as-fabricated and oxidized samples of GE 1541 and H 875 Poroloy to determine the ultimate tensile strength, the 0.2% offset yield strength, elongation and the modulus of elasticity. Results are summarized in Table 4 and tabulated in Appendix A. One of the most important properties in the application of these materials to transpiration cooling is ductility retention. As can be seen in Table 4, oxidation exposure does not decrease ductility and, in fact, may slightly increase it. The ultimate tensile strength is unaffected following 600 hours exposure at 1800°F (982°C) for both alloys. Oxidation at 2000°F (1093°C) decreases this property less than 15%. The yield strength decrease for both alloys is less than 10% after 1800°F (982°C) exposure and less than 15% after 2000°F (1093°C) exposure. Degradation of these properties then is of insufficient magnitude to hinder Poroloy implementation in transpiration cooling application. The room temperature tensile properties of the DH 242, DH 245 and TD Ni Cr Poroloy materials are also included in Table 4 for reference.

5.2.6 Joint Tensile Strength. — For all samples tested, the strength of the electron beam weld joint between the Poroloy and IN 100 strut alloy were higher than that of the Poroloy. That is the tensile sample always fractured in the Poroloy rather than in the weld zone. However, the samples exposed at 2000°F (1093°C) for 600 hours experienced severe oxidation of the strut material which degraded the weld to the extent that the strut and Poroloy separated. Therefore, no strength data was obtained after this exposure. This failure is not deemed serious in actual application since it was initiated by the strut material which was not designed to operate at 2000°F (1093°C).

5.2.7 Metallographic Examination. — The Poroloy and the Poroloy/IN 100 weld joints were examined metallographically for each time-temperature condition of the cyclic oxidation tests. Photomicrographs of the Poroloy samples are presented in Appendix C, Figures C-1, C-2, C-5, and C-6. Progressive oxidation with time of both GE 1541 and H 875 is evident at both temperatures. The GE 1541 oxide scale appears thicker than that of the H 875 which is consistent with the weight gain results. No spalling or plugging was noted. There was no significant oxidation of the bond joints which indicates good bond integrity.

Photomicrographs of the weld joints are shown in Appendix C, Figures C-3, C-4, C-7, and C-8. At 1800°F (982°C), although the weld zone oxidized to a greater extent than the Poroloy, the extent of oxidation was not great enough to seriously degrade the joint strength. This was confirmed in the joint tensile tests because the samples failed in the Poroloy rather than at the weld joint. At 2000°F (1093°C) extensive oxidation occurred at the weld zone of both GE 1541 and H 875 at the longer exposure time with the effect more pronounced in the GE 1541 material. GE 1541 weld joints completely oxidized at exposure times of 300 hours and longer while this occurred in the H 875 weld joints at 500 hours and longer exposure. This severe oxidation is attributed to the fact that the strut alloy IN 100 suffers rapid oxidation at this temperature and consequently initiated accelerated oxidation of the Poroloy. In addition, there may have been some degradation of the oxidation resistance in the weld zone during welding as a consequence of the melting and alloying of the two alloys. The extensive oxidation of the strut alloy slug is shown in Figure C-9. The extensive oxidation of the weld joint was confined mainly to that area in the H 875 material (Figure C-10). However, with GE 1541, once the severe oxidation was initiated, it propagated along the length of the sample, Figure C-11. The sensitivity of the oxidation of GE 1541 to contact with other alloys is also illustrated in Figure C-12. The regions of severe oxidation shown in this photograph were initiated at the point where the GE 1541 Poroloy contacted the basket holder (Type 330 stainless steel). This sensitivity of the GE 1541 dictates that care must be exercised in selecting alloys which may be in contact with GE 1541 at temperatures equal to or in excess of 2000°F (1093°C).

5.3 Stress Rupture

A comparison of GE 1541 and H 875 stress rupture curves is given in Figure 12 for 0.005 inch (0.13 mm) diameter wire at 1800°F and 2000°F (982 and 1093°C) and in Figure 13 for Poroloy at 1800°F (982°C). At 1800°F (982°C) the GE 1541 wire and Poroloy was slightly stronger than H 875 for lifetimes greater than 5 hours and slightly weaker for lifetimes less than 5 hours. At 2000°F (1093°C) the GE 1541 was clearly stronger than the H 875. Data is given in tabular form in Appendix D, along with stress rupture data for the DH 245 Poroloy. Stress rupture data for TD Ni Cr and DH 242 wire were given in reference 2. As reported in reference 2, TD Ni Cr wire was found to exhibit extensive internal oxidation above a critical applied stress level. Work was carried out in this investigation to determine the reasons for this stress sensitivity of oxidation. The results of this work are presented in Appendix D.

5.4 Hot Tensile Tests

Tabulated results of 1800°F (982°C) tensile tests for GE 1541, H 875 and DH 245 wire and Poroloy are presented in Appendix D. Considerable scatter is evident in the wire data, probably as a result of localized variation in properties stemming from inclusions or defects. Less scatter is found for the Poroloy samples in which localized wire deficiencies would be expected to average out. The tensile properties following a simulated sinter and preoxidation of the Poroloy at 1800°F (982°C) for 24 hours are summarized below:

Alloy	Ultimate Tensile Strength		0.2% Offset Yield Strength		Elongation %
	Ksi	$N/m^2 \times 10^{-6}$	Ksi	$N/m^2 \times 10^{-6}$	
GE 1541	2.80	19.6	2.65	18.5	49%
H 875	2.85	19.9	2.60	18.2	58%

These results are in agreement with the stress rupture results which showed comparable strengths for the two alloys at 1800°F (982°C).

5.5 Welding Tests

Weld samples tested in cyclic oxidation were GE 1541 and H 875 Poroloy welded to IN 100 strut alloy. Prior to cyclic oxidation testing, samples were made to determine the weldability of all the Poroloy alloys considered in this investigation to IN 100 and to an alternate strut alloy 713C. In addition to GE 1541 and H 875, Poroloy weld samples included TD Ni Cr and DH 242.

Photographs of representative weld samples are presented in Appendix E, Figure E-1. It should be noted that there is sufficient penetration for a sound joint and that there is no collapsing of the Poroloy between the strut supports. Figures E-2 through E-6 show etched microstructures of the weld zone. The TD Ni Cr welds had Thoria agglomeration and grain growth in the weld zone. These microstructural changes would certainly reduce the elevated temperature strength of the joint. All other welds appeared sound with consistent penetration and weld interface. No significant porosity, cracking, undercutting or surface irregularities were observed. Some slight cracking was observed primarily at the root of the GE 1541 and H 875 Poroloy welds. However, this degree of crack appear insufficient to significantly degrade the joint strength. This conclusion was confirmed in the cyclic oxidation tests in which the joint strength was always found to be greater than that of the Poroloy. In summary then, the electron beam welding technique appears to be a reliable method for joining porous metal sheets to struts in transpiration cooling applications except for the case of TD Ni Cr where the parent alloy properties are degraded.

6 CONCLUSIONS AND RECOMMENDATIONS

1. Process development performed in this program has shown that oxidation resistant alloys may be made into wire form and fabricated into porous metal sheet with retention of the alloys oxidation resistance.

2. Two iron-base alloys, GE 1541 and H 875, were found to offer a good combination in porous metal form (Poroloy) of oxidation resistance and room temperature tensile property retention following oxidation.

3. Cyclic oxidation behavior appears no more severe than that which is obtained during continuous oxidation since no spalling or cracking of the oxide was observed.

4. The GE 1541 Poroloy shows excellent permeability retention following cyclic oxidation exposure at 1800 and 2000°F (982 and 1093°C). The values after 600 hours exposure are 80% and 78% respectively of the as-fabricated values. Most of the decrease occurs during the first four hours of exposure (84% of as-fabricated value) so that almost constant permeability could be obtained during the design life by pre-oxidation of the Poroloy before service.

5. The H 875 Poroloy permeability retention after 600 hours exposure was the same as that of the GE 1541 alloy at 1800°F (982°C) but was only 56% of the as-fabricated value after exposure at 2000°F (1093°C). The initial change in permeability with time was more gradual than that of GE 1541 material.

6. GE 1541 and H 875 Poroloy may be reliably joined to strut alloys IN 100 and 713C by electron beam welding.

7. If in direct contact with another alloy of lower oxidation resistance, accelerated oxidation in the GE 1541 is initiated and once started, propagates into the alloy.

8. Exposure to pure nitrogen or a reducing atmosphere containing nitrogen will reduce oxidation resistance and embrittle the GE 1541 alloy.

9. GE 1541 and H 875 Poroloy appear suitable for application as transpiration cooling materials for gas turbine vanes at temperatures in excess of 1800°F (982°C) based on (1) good permeability retention after oxidation as a consequence of good oxidation resistance, (2) retention of room temperature ductility and strength, (3) ease of joining to strut alloys and (4) development of a reliable fabrication procedure.

10. GE 1541 Poroloy has greater permeability retention following oxidation at 2000°F (1093°C) than does DH 242 Poroloy following oxidation at 1700°F (927°C). Therefore, development of the GE 1541 material represents an advance of over 300°F (149°C) in the state-of-the-art operating temperature for transpiration cooling materials.

11. Additional work that should be performed prior to application of these materials to transpiration cooling in jet engines includes:

    a. Testing of transpiration cooled vanes for cooling effectiveness.

    b. Determination of hot corrosion resistance.

    c. Determination of thermal fatigue resistance of transpiration cooled vanes.

    d. Investigation of ways to increase alloy strength (dispersion strengthened Fe Cr Al alloys).

    e. Determination of the oxidation and thermal expansion compatibility of GE 1541 Poroloy vane shells with different strut alloys.

    f. Investigation of methods using GE 1541 Poroloy in vanes without a strut support.

TABLE 1 ALLOY CHEMICAL ANALYSIS

ALLOY	Fe	Cr	Al	Y	Si	Mn	C	S	P	Ni	OTHER
GE 1541	BAL	14.84	5.05	0.58	0.05		163 ppm	28 ppm	10 ppm		O 132 ppm N 37 ppm Mo 4 ppm
H 875	BAL	22.35	5.44		0.58	0.36	0.07				
DH 245	0.42	19.46	3.73		1.31		0.04			BAL	Mg 0.28
Td Ni-Cr		21.85					0.011	0.006		BAL	ThO_2 2.5
DH 242	0.66	19.18			1.08		0.03			BAL	Cb 0.95

TABLE 2 WIRE MECHANICAL PROPERTIES

WIRE 0.005-INCH (0.013 mm) DIAMETER		AS RECEIVED	AFTER SIMULATED SINTER	SINTERED & OXIDIZED*
GE 1541	US Ksi $(N/m^2 \times 10^{-6})$	130 (909)	69 (482)	62 (433)
Hard	YS Ksi $(N/m^2 \times 10^{-6})$	127 (888)	51 (357)	48 (336)
	%E	1.5	10	5.5
H 875	US Ksi $(N/m^2 \times 10^{-6})$	136 (951)	84 (587)	84 (587)
Annealed	YS Ksi $(N/m^2 \times 10^{-6})$	112 (783)	70 (489)	69 (482)
	%E	12	3.5	3.5
DH 245	US Ksi $(N/m^2 \times 10^{-6})$	184 (1290)	110 (769)	90 (630)
	YS Ksi $(N/m^2 \times 10^{-6})$	115 (803)	76 (532)	70 (489)
	%E	27	10	4
TD Ni-Cr	US Ksi $(N/m^2 \times 10^{-6})$	137 (958)	142 (993)	132 (923)
	YS Ksi $(N/m^2 \times 10^{-6})$	114 (797)	116 (812)	108 (755)
	%E	19	22	12
DH 242	US Ksi $(N/m^2 \times 10^{-6})$	177 (1240)	110 (769)	
	YS Ksi $(N/m^2 \times 10^{-6})$	173 (1210)	44 (308)	
	%E	7	23	

*Oxidized at 1800°F (982°C) in air for 24 hours.

TABLE 3 METALLOGRAPHIC ETCHANT SCHEDULE

Alloy	Etchant	Procedure
GE 1541	22% H_2SO_4 12% H_2O_2 (30% solution) 66% H_2O	Electrolytic: 6 volts, 5 to 20 seconds
H 875	25% HCl 25% HNO_3 50% H_2O	Direct application to surface for about 15 seconds
DH 242	10% Oxalic Acid	Electrolytic: 5 volts, 5 to 25 seconds
TD NiCr	10% Oxalic Acid	Electrolytic: 5 volts, 5 to 25 seconds

TABLE 4 SUMMARY OF POROLOY ROOM TEMPERATURE TENSILE PROPERTIES

Poroloy Alloy	Ultimate Tensile Strength* Ksi ($N/m^2 \times 10^{-6}$)			Yield Strength* Ksi ($N/m^2 \times 10^{-6}$)			Elongation* %		
	As Fabricated	Oxidized 600 hours at 1800°F	2000°F	As Fabricated	Oxidized 600 hours at 1800°F	2000°F	As Fabricated	Oxidized 600 hours at 1800°F	2000°F
GE 1541	42 (290)	42 (290)	38 (270)	35 (240)	32 (220)	30 (210)	9	10	10
H 875	56 (390)	56 (390)	49 (340)	42 (290)	40 (280)	36 (250)	15	18	16**
DH 245	84 (590)	--	--	62 (430)	--	--	12	--	--
TD Ni-CR	53 (370)	--	--	51 (360)	--	--	3	--	--
DH 242	56 (390)	--	--	34 (240)	--	--	14	--	--

* Values represent average of three samples

** Excluding one unrealistic low value

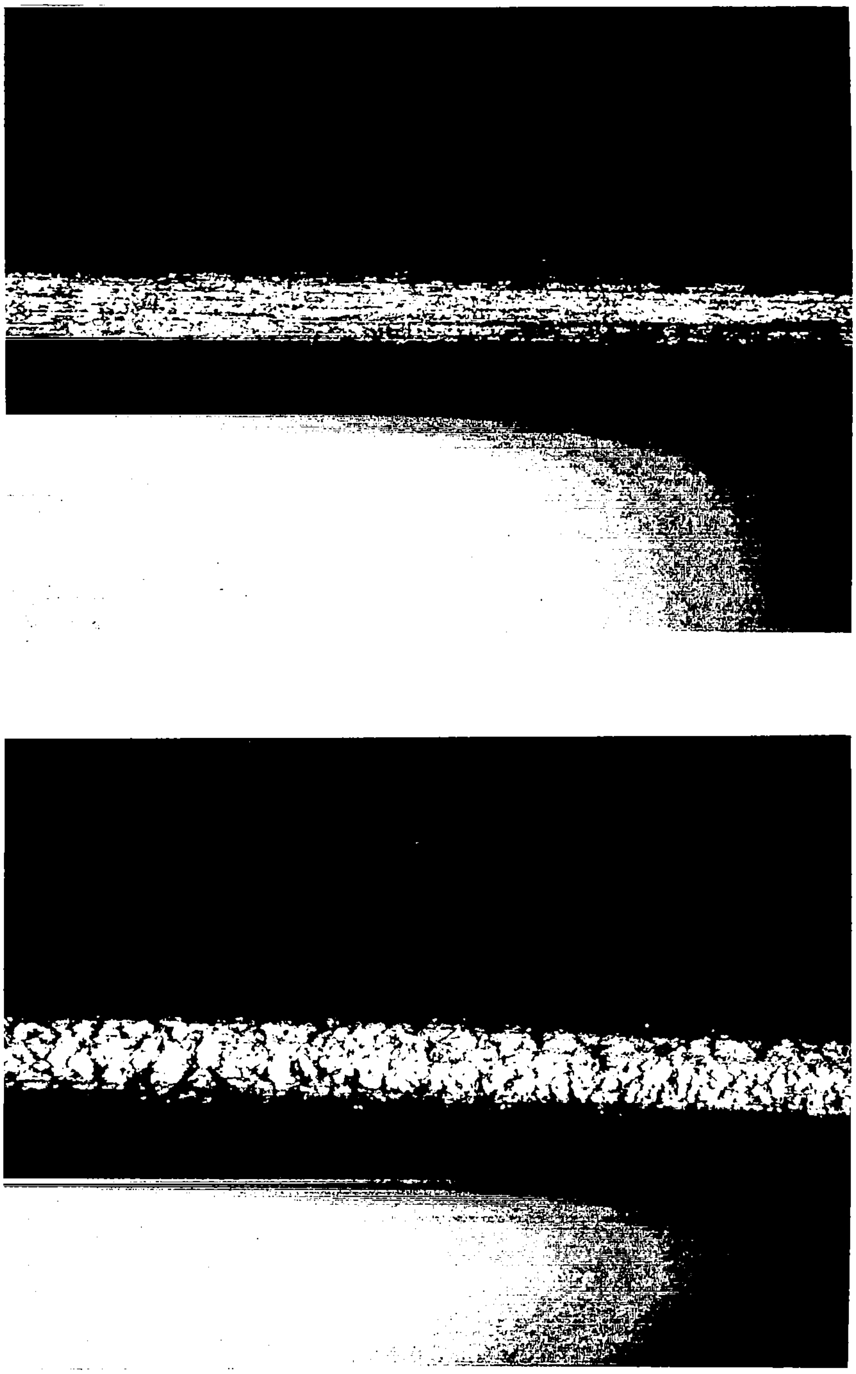


Figure 1 As-fabricated surface of GE 1541 wire, 0.005 inch (0.13 mm) diameter having good (above) and poor (below) oxidation resistance. X250

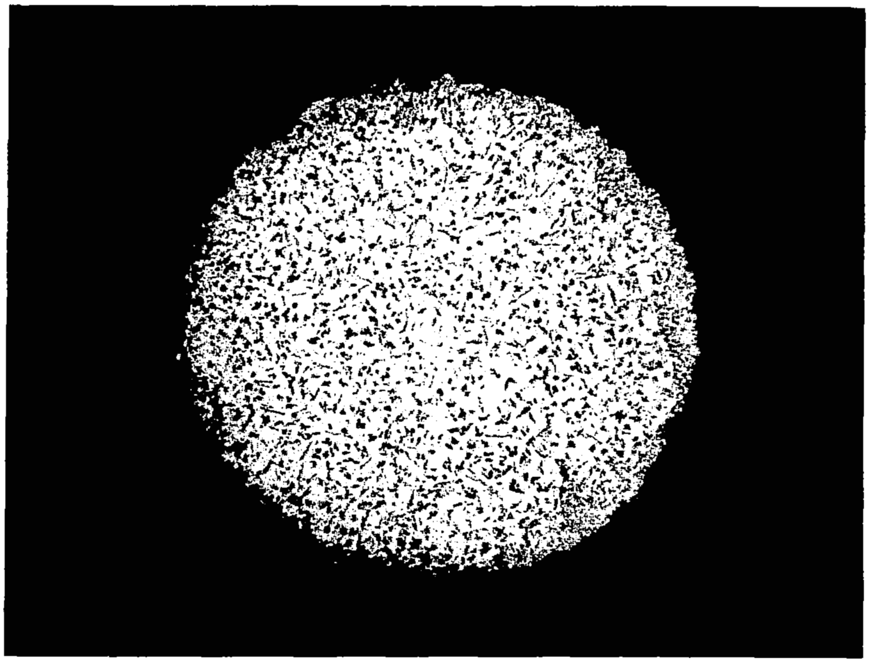


Figure 2 Microstructure of GE 1541 wire, 0.005 inch (0.13 mm) diameter after exposure to nitrogen atmosphere, 1/2 hour at 1900°F (1038°C). X500

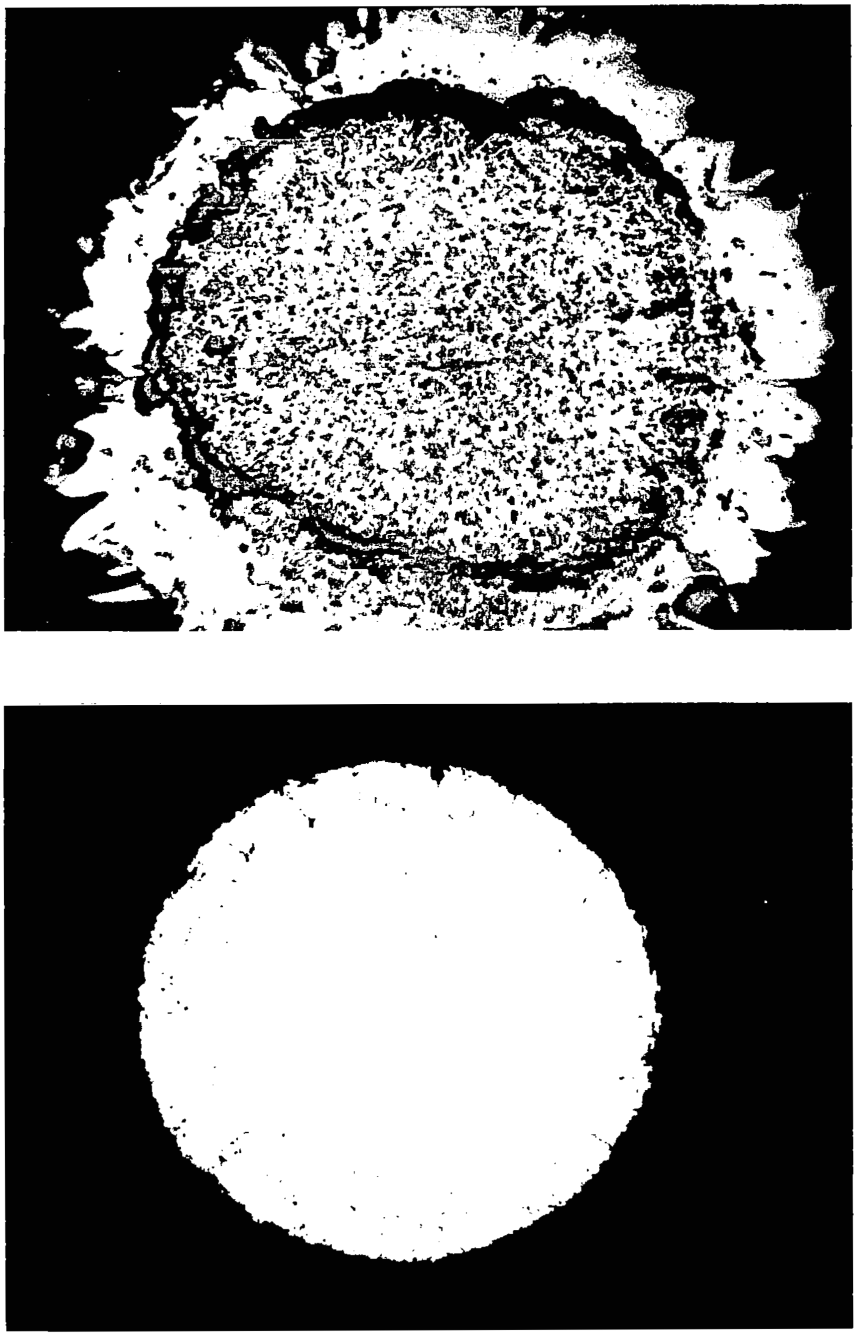


Figure 3 Cross sections of GE 1541 wires, 0.005 inch (0.13 mm) diameter following oxidation at 1800°F (982°C) for 24 hours. The top wire was heated in a nitrogen atmosphere for 1/2 hour at 1900°F (1038°C) prior to oxidation testing while the bottom wire was tested as-fabricated. X500

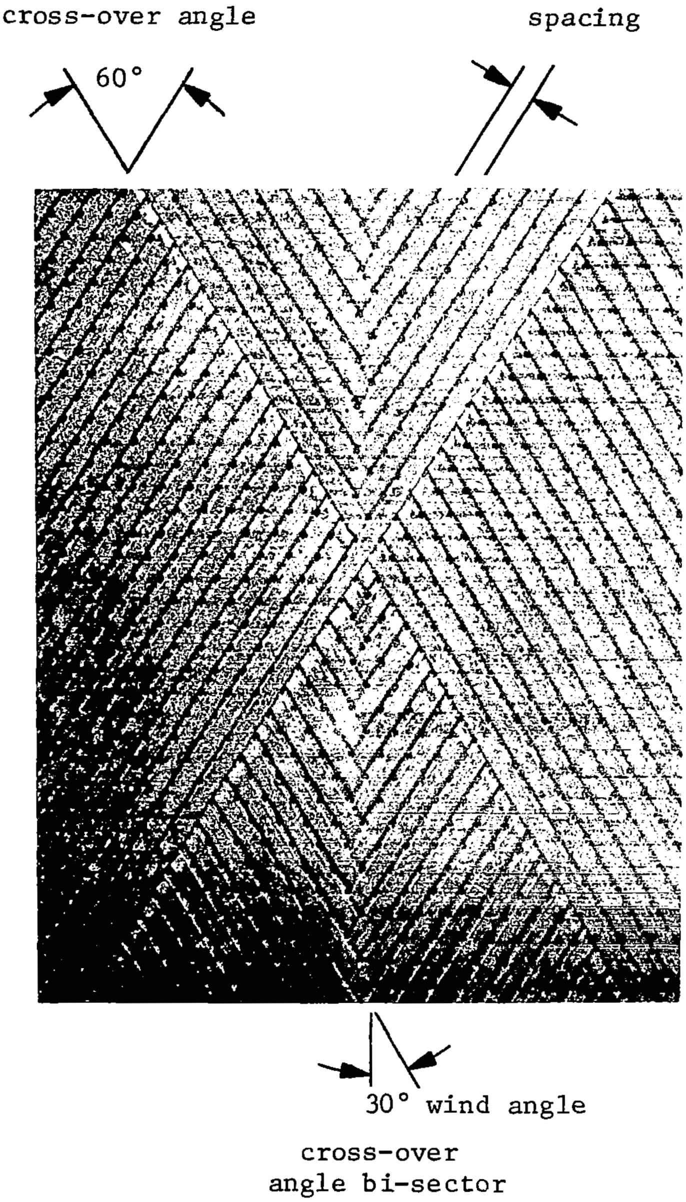


Figure 4 Photomacrograph of Poroloy.

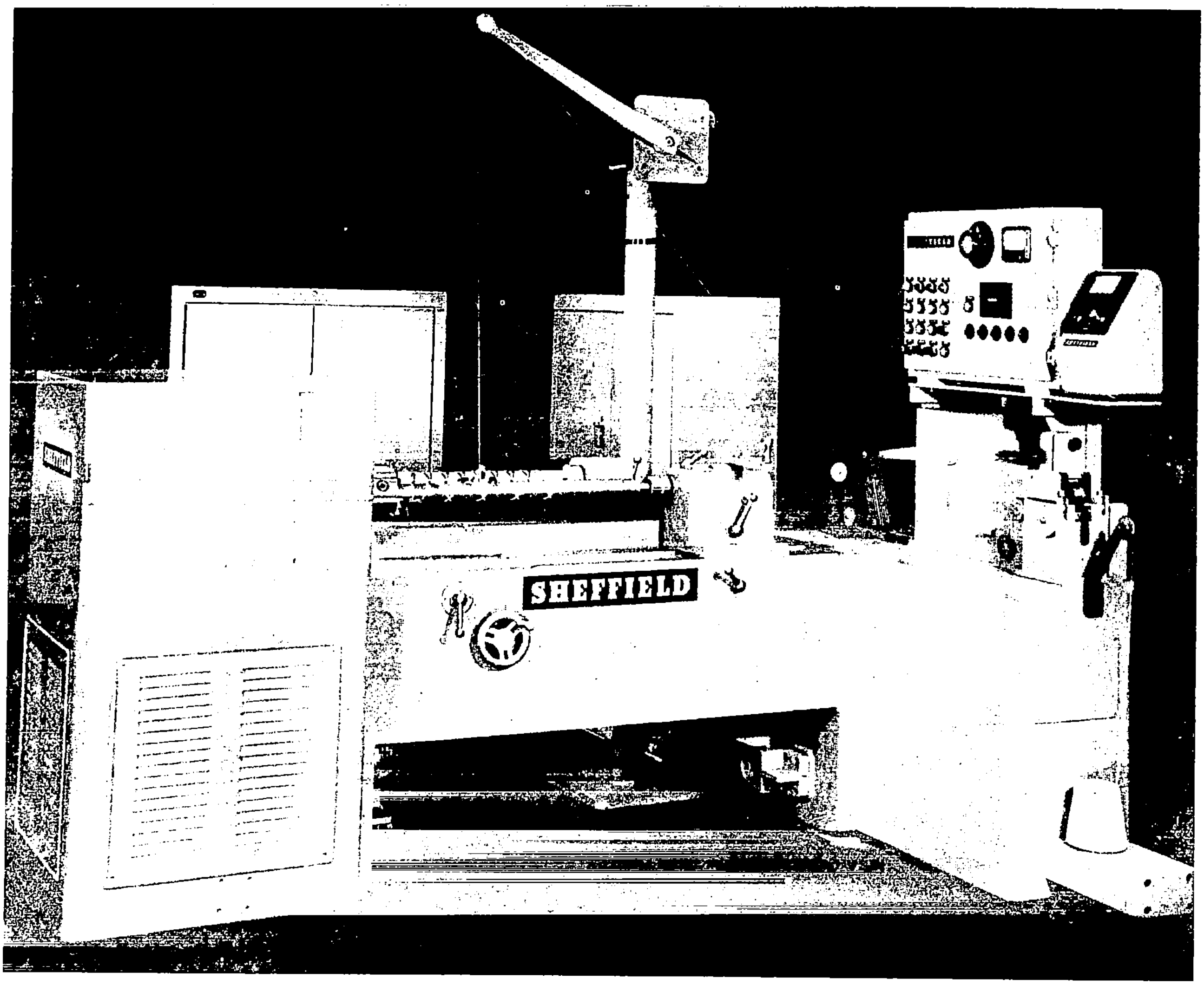
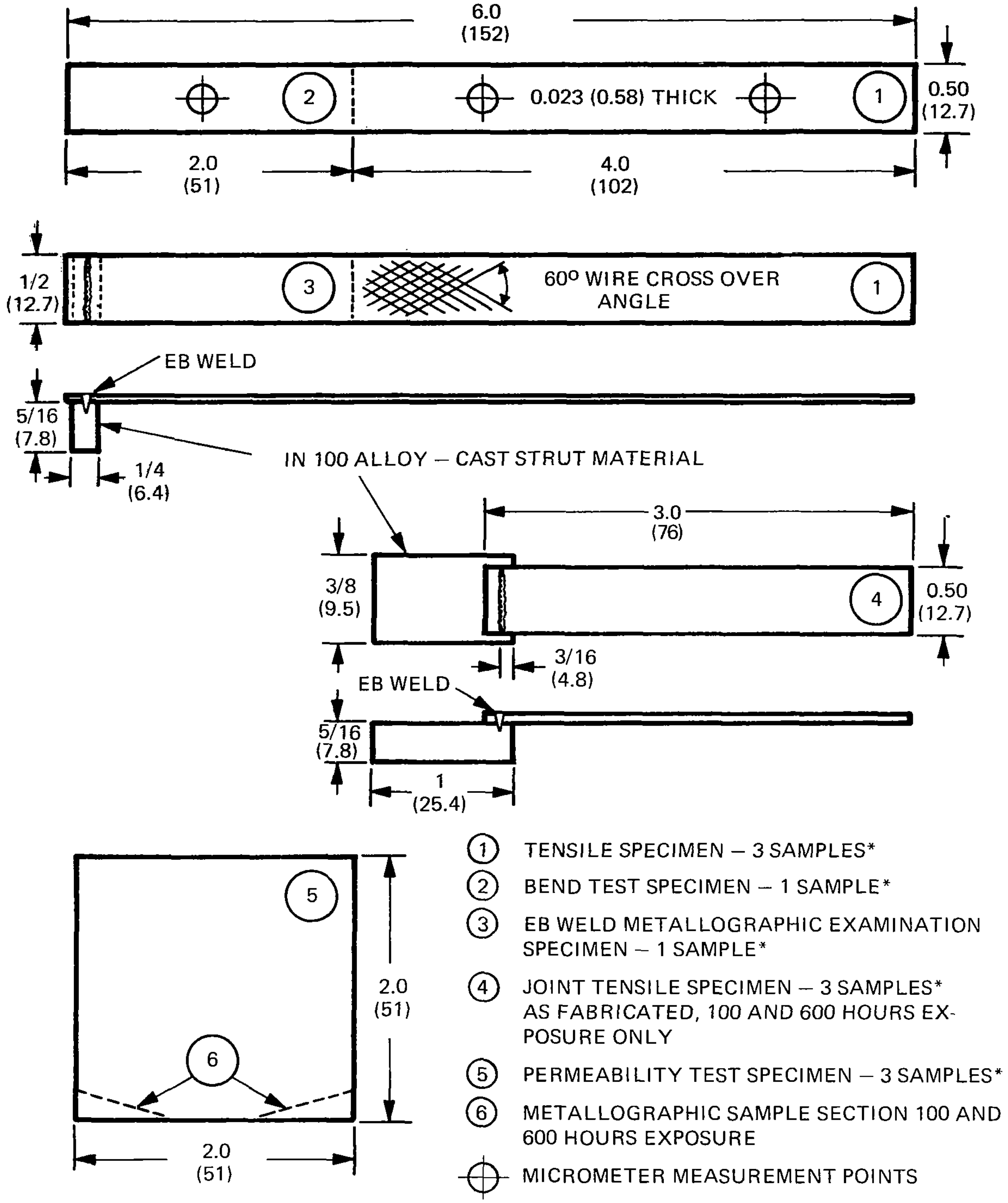


Figure 5 - Poroloy Winding Machine



① TENSILE SPECIMEN – 3 SAMPLES*

② BEND TEST SPECIMEN – 1 SAMPLE*

③ EB WELD METALLOGRAPHIC EXAMINATION SPECIMEN – 1 SAMPLE*

④ JOINT TENSILE SPECIMEN – 3 SAMPLES* AS FABRICATED, 100 AND 600 HOURS EXPOSURE ONLY

⑤ PERMEABILITY TEST SPECIMEN – 3 SAMPLES*

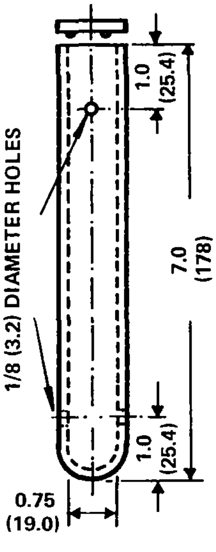
⑥ METALLOGRAPHIC SAMPLE SECTION 100 AND 600 HOURS EXPOSURE

⊕ MICROMETER MEASUREMENT POINTS

*NUMBER OF SAMPLES FOR EACH TIME TEMPERATURE CONDITION.

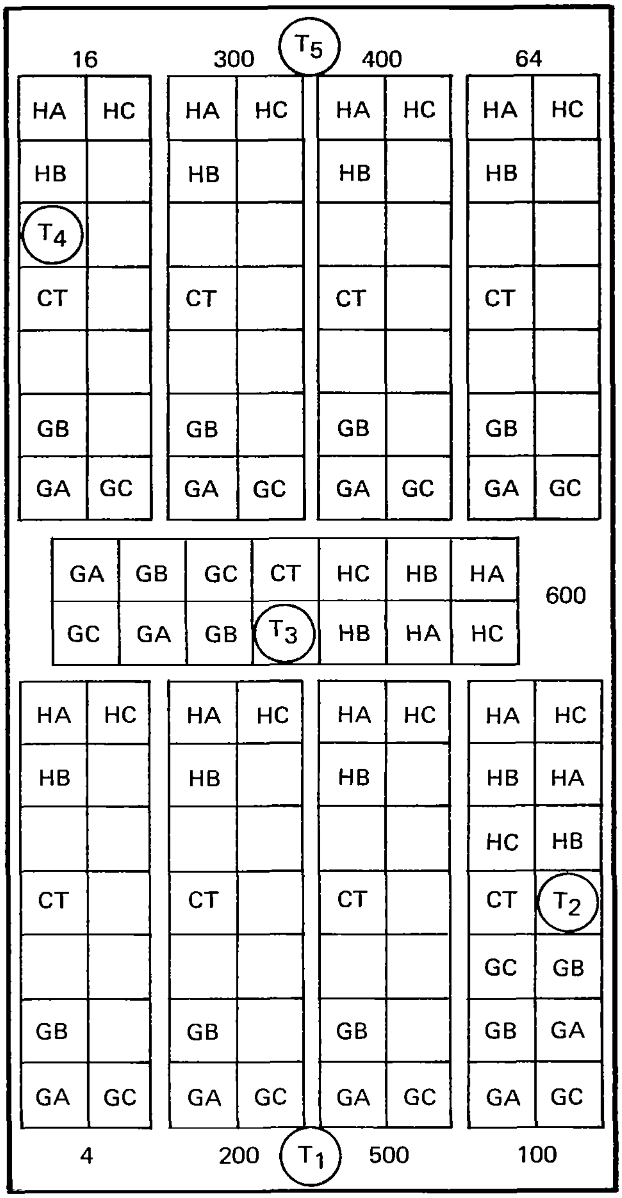
DIMENSIONS ARE IN INCHES WITH THOSE IN PARENTHESIS IN MILLIMETERS.

Figure 6 - Poroloy Oxidation Specimens



ZIRCON THIMBLE

T/C Location	Measured Average Temperature °F at Thermocouple Locations Nominal Temperature 1800°F (982°C)	 2000°F (1093°C)
T_1	—	1995 (1091)
T_2	1802 (983)	1995 (1091)
T_3	1825 (996)	2020 (1104)
T_4	1818 (992)	2015 (1102)
T_5	—	2010 (1099)



Furnace Rack Arrangement by Exposure Time – Hours. G and H signifies GE 1541 and H 875 alloy respectively; A, B and C refers to specimen identification as listed in Numerical Data Tabulation; CT is an empty control thimble and T indicates location of thermocouples.

Figure 7 - Specimen and Thermocouple Arrangement for Cyclic Oxidation Tests

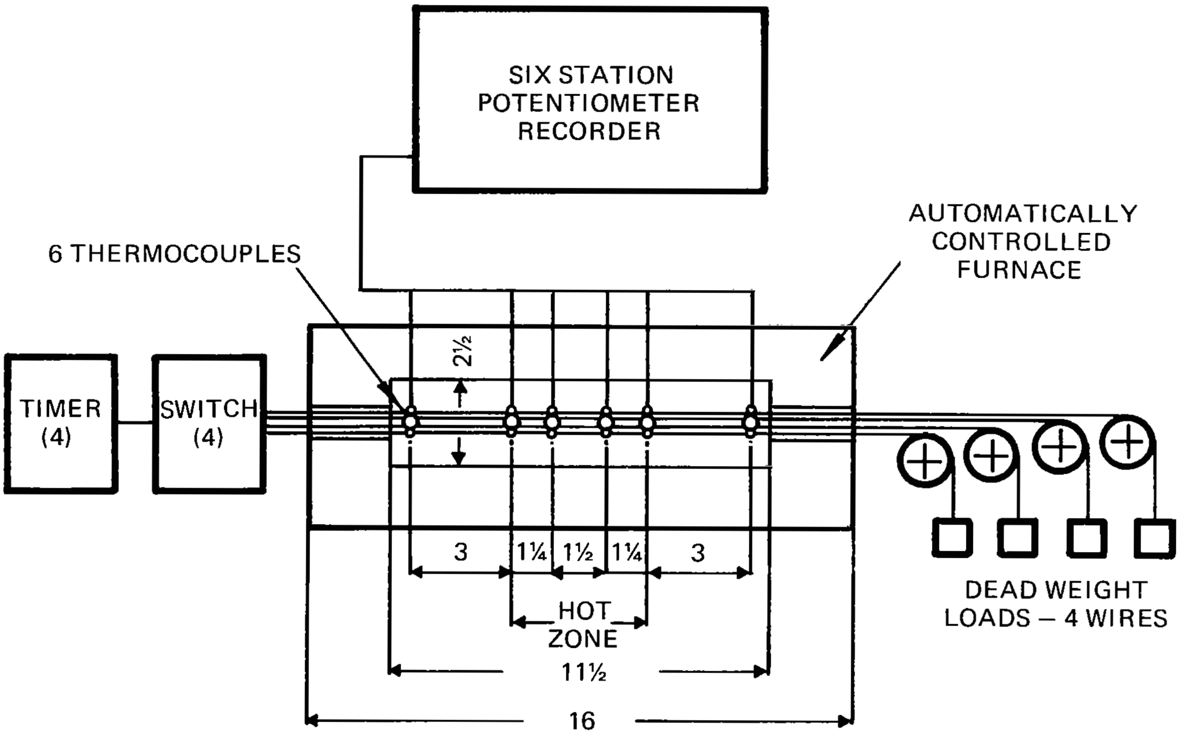


Figure 8 - Stress Rupture and Oxidation Apparatus

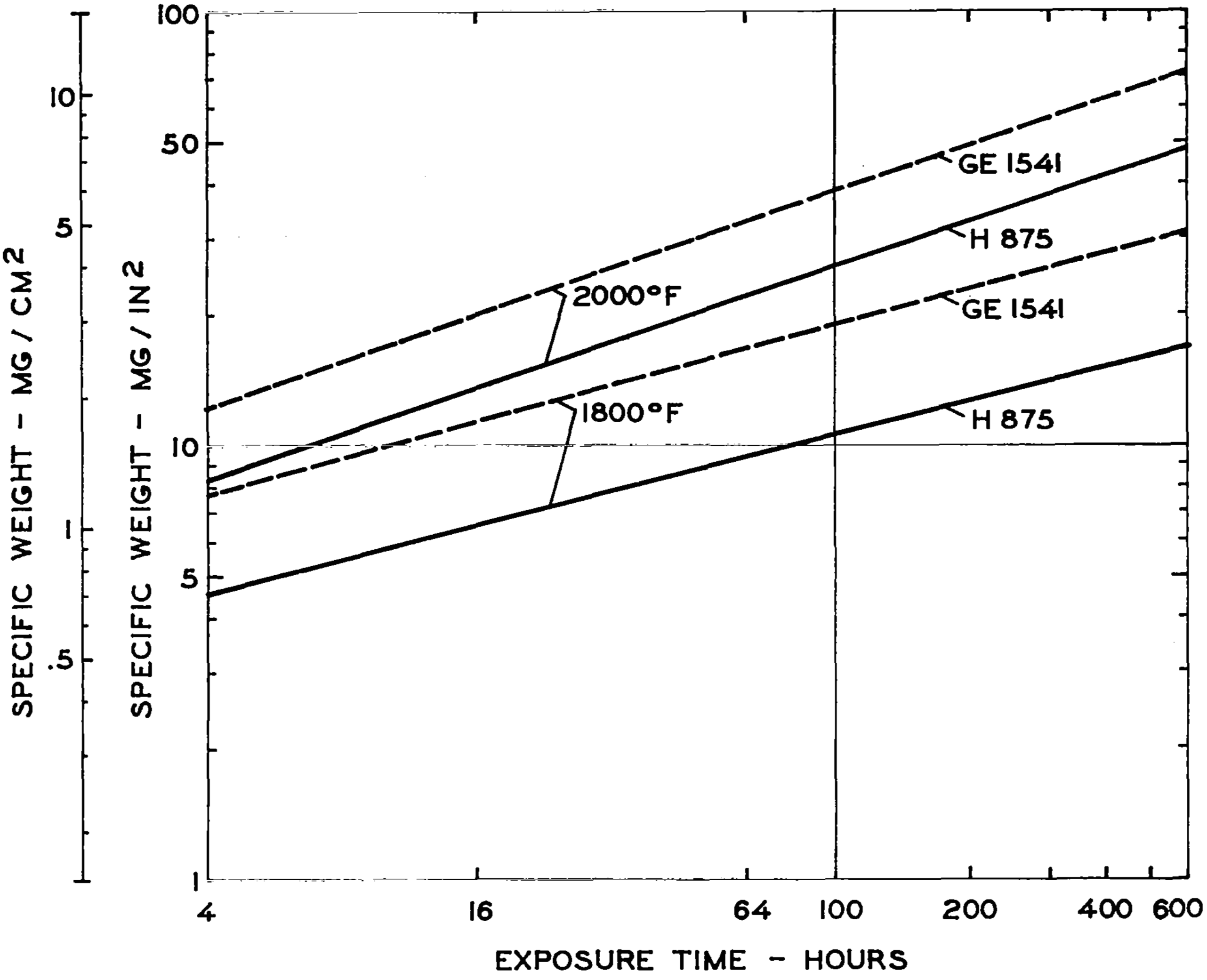


Figure 9 Specific Oxidation Weight Gain for Poroloy After Exposure at 1800 and 2000°F (982 and 1093°C) in Air.

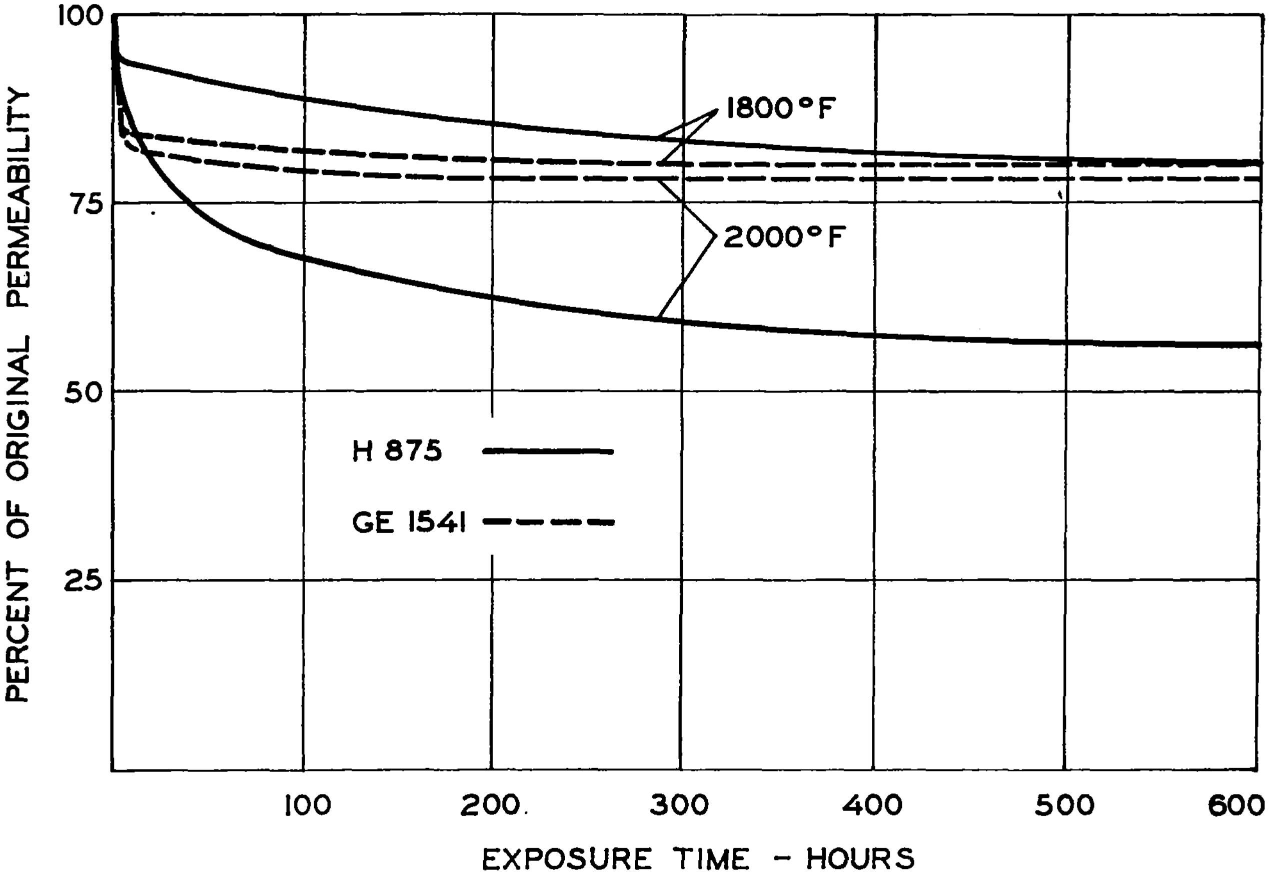


Figure 10 Poroloy Permeability Retention After Exposure at 1800 and 2000°F (982 and 1093°C) in Air. Alloys GE 1541 and H 875.

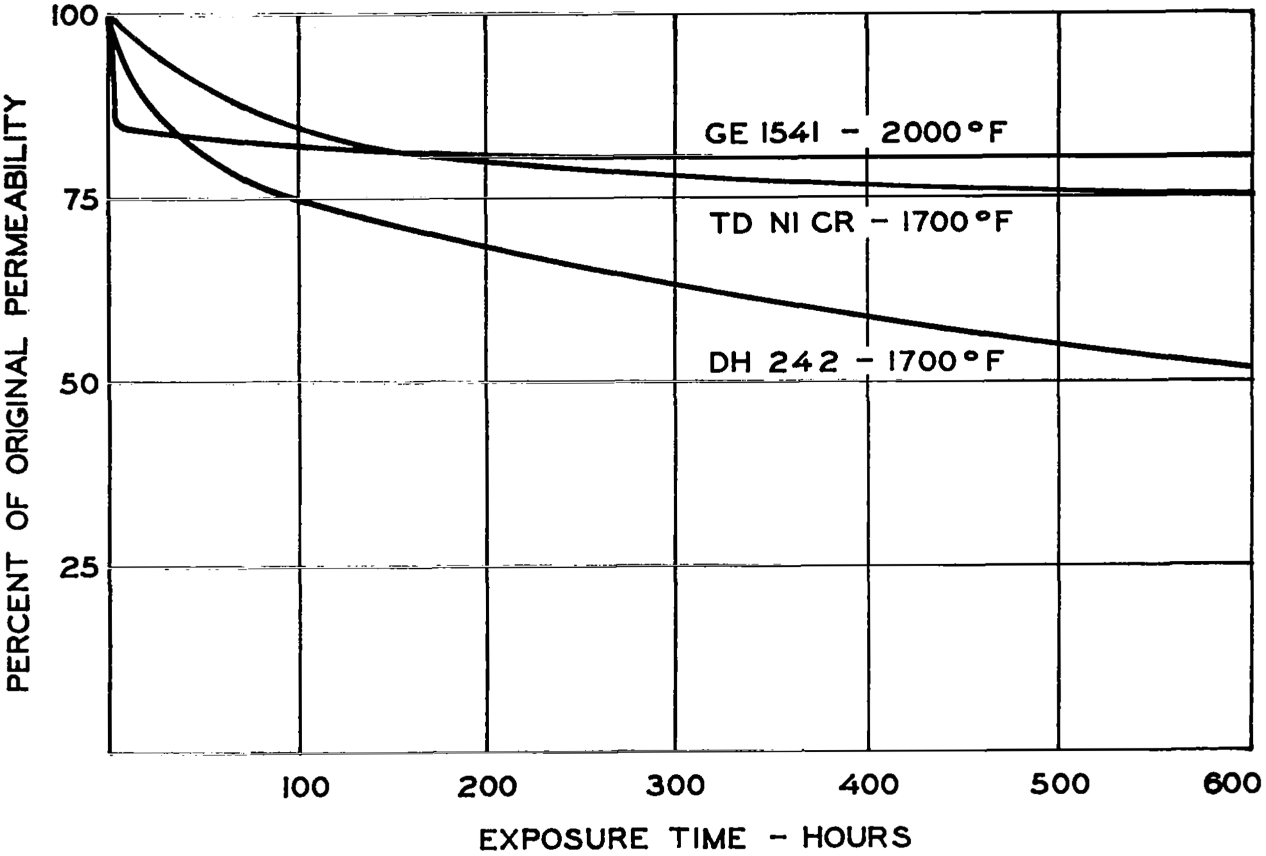


Figure 11 Poroloy Permeability Retention After Exposure in Air at 2000°F (1093°C) for the GE 1541 Alloy and at 1700°F (927°C) for the TDNiCr and DH 242 Alloys.

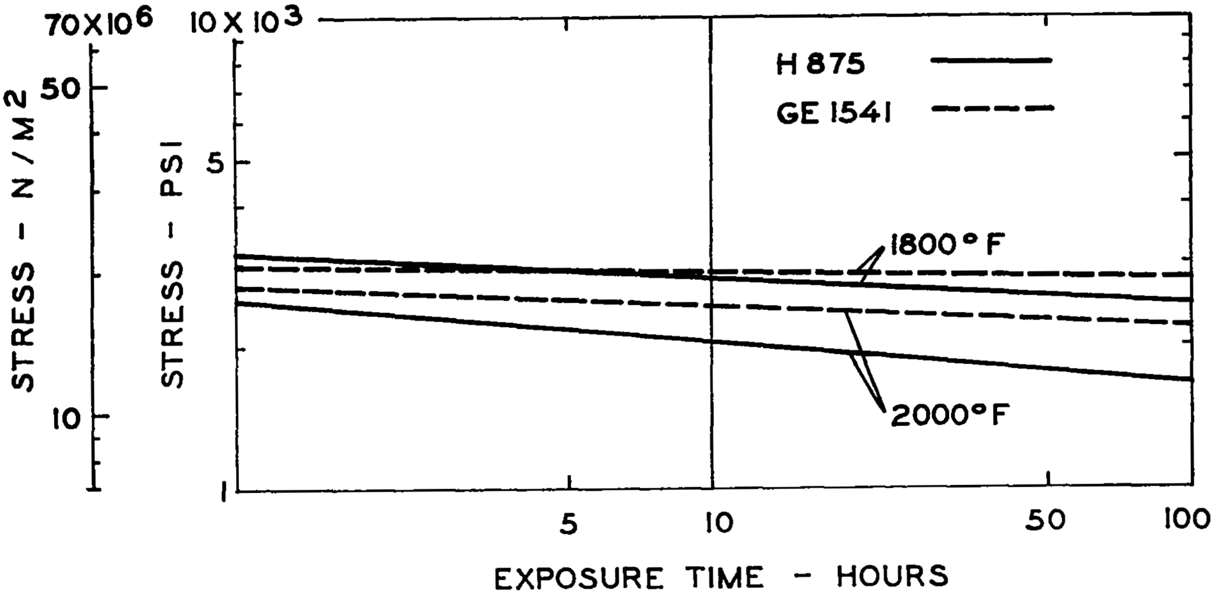


Figure 12 Stress Rupture Life at 1800 and 2000°F (982 and 1093°C) for GE 1541 and H 875 Alloy Wire, 0.005 Inch (0.13 mm) Diameter After Simulated Sinter Cycle.

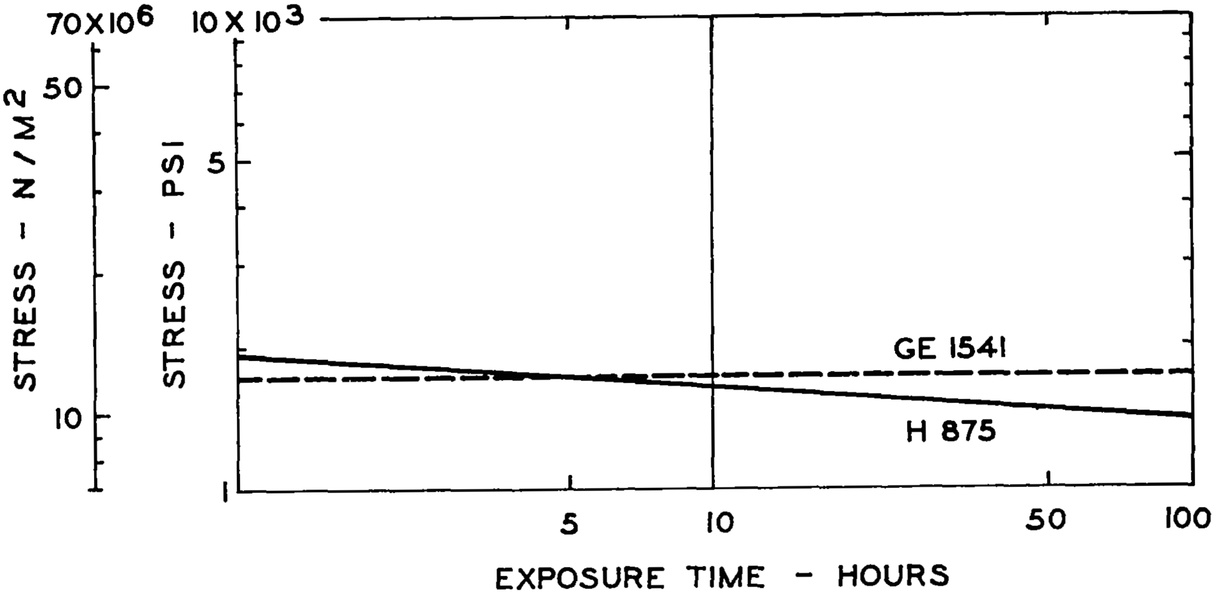


Figure 13 Stress Rupture Life at 1800°F (982°C) for GE 1541 and H 875 Alloy Poroloy.

APPENDIX A

NUMERICAL DATA TABULATION FOR CYCLIC OXIDATION TESTS

Alloy	Temperature	Table	Page
GE 1541	1800°F	A-1	40
GE 1541	1000°F	A-2	41
H 875	1800°F	A-3	42
H 875	2000°F	A-4	43

Reduced numerical data for the oxidation test is given according to the following legend:

A.	Exposure Time (hours)	All times are total hours at the specified temperature. 0 hours indicates nonoxidized control specimens. The specimens were air cooled to room temperature after each exposure interval.
B.	Specimen Identification	Specimens A and B were used to obtain oxidation weight gain data. Specimen C had a small tab of IN 100 alloy attached by electron beam welding. All specimens were tensile tested after exposure.
C.	Specific Oxidation Weight Gain (mg/in^2)	The specimen was weighed before and after exposure. The difference divided by original sample area was designated specific weight gain. Spalling of the oxide was not experienced.
D.	Permeability (SCFM/IN^2 at 10 PSID)	Air flow was measured for an inlet pressure of 24.7 psia and a discharge pressure of 14.7 psia at an air temperature of 70°F using a probe diameter of 1.129 in.
E.	Thickness Change (in x 10^3)	Based on the average of three (3) measurements with hand micrometer. Both ends and the center were measured.
F.	Bend Test (Degree bend)	Bend testing was performed over a 3T radius (a 1/8 in. diameter rod was used). All samples were bent 180° without evidence of any fraying or fracturing.
G.	Yield Strength (PSI x 10^{-3})	Determined using 0.2% offset. The higher of two distinct yield points is reported. Area based on measurements before exposure.
H.	Ultimate Strength (PSI x 10^{-3})	Area based on measurements before exposure.
I.	Elongation (Percent)	Measured on one (1) inch gage length.
J.	Modulus of Elasticity (PSI x 10^{-6})	Measured from trace of the tensile stress-strain curve.
K.	Joint Tensile Strength (PSI x 10^{-3})	In all the weld samples tested, breakage took place in the Poroloy at the indicated stress level. The samples exposed to 600 hours at 2000°F oxidized catastrophically in the strut material (IN 100) and could not be tensile tested. This condition is indicated by a "0".

TABLE A-1 - NUMERICAL DATA TABULATION: ALLOY GE 1541

EXPOSURE TIME - HOURS	SPECIMEN	SPECIFIC OXIDATION WEIGHT GAIN MG/IN2	PERMEABILITY AT 10 PSID SCFM/IN^2	THICKNESS CHANGE IN x 103	BEND TEST 3T RADIUS ° BEND	YIELD STRENGTH 0.2% OFFSET PSI x 10-3	ULTIMATE STRENGTH PSI x 10-3	ELONGATION IN 1 INCH %	MODULUS OF ELASTICITY PSI x 10-6	JOINT TENSILE STRENGTH PSI x 10-3
A	B	C	D	E	F	G	H	I	J	K
TEMPERATURE 1800°F										
	A		3.85		180	34.4	42.6	9.0	2.4	44.9
0	B		3.85			34.0	42.2	10.0	2.4	38.0
	C		3.96			35.4	41.2	7.3	2.4	44.8
	A	7.75	3.24	0.0	180	33.6	40.1	7.3	2.4	
4	B	8.18	3.24	0.0		34.1	44.3	14.3	2.4	
	C		3.34			33.6	42.6	10.5	2.4	
	A	11.60	3.19	0.2	180	33.9	45.4	17.5	2.5	
16	B	11.45	3.19	0.2		33.1	42.6	11.5	2.5	
	C		3.30			33.3	43.7	12.0	2.5	
	A	16.45	3.19	0.1	180	32.5	42.6	12.3	2.3	
64	B	16.75	3.19	0.1		32.6	42.9	14.3	2.3	
	C		3.30			32.8	43.3	14.3	2.3	
	A	13.90	3.11	0.2	180	32.7	42.5	12.0	2.7	42.7
100	B	14.90	3.11	0.2		32.5	43.5	14.8	2.7	43.4
	C		3.22			32.0	42.0	13.0	2.7	44.1
	A	15.85	3.11	0.2	180	32.2	42.3	12.0	2.7	
200	B	17.25	3.11	0.3		32.2	41.7	11.3	2.7	
	C		3.22			32.5	41.9	12.0	2.7	
	A	26.05	3.08	0.3	180	29.7	40.9	10.0	2.2	
300	B	25.80	3.08	0.4		30.3	42.7	13.5	2.2	
	C		3.19			29.9	41.2	12.5	2.2	
	A	28.60	3.10	0.4	180	30.1	41.6	12.5	2.1	
400	B	28.50	3.10	0.3		31.0	42.4	12.3	2.1	
	C		3.19			32.0	43.3	11.5	2.1	
	A	23.05	3.08	0.3	180	31.8	40.8	9.3	2.3	
500	B	24.90	3.08	0.3		31.6	41.7	10.0	2.3	
	C		3.19			33.1	43.3	11.8	2.3	
	A	31.40	3.08	0.5	180	31.1	41.4	10.8	2.1	42.1
600	B	32.65	3.08	0.3		31.1	41.7	10.8	2.1	45.7
	C		3.19			33.1	43.3	9.5	2.1	45.5

TABLE A-2 - NUMERICAL DATA TABULATION: ALLOY GE 1541

EXPOSURE TIME - HOURS	SPECIMEN	SPECIFIC OXIDATION WEIGHT GAIN MG/IN^2	PERMEABILITY AT 10 PSID SCFM/IN^2	THICKNESS CHANGE IN x 10^3	BEND TEST 3T RADIUS ° BEND	YIELD STRENGTH 0.2% OFFSET PSI x 10^{-3}	ULTIMATE STRENGTH PSI x 10^{-3}	ELONGATION IN 1 INCH %	MODULUS OF ELASTICITY PSI x 10^{-6}	JOINT TENSILE STRENGTH PSI x 10^{-3}
A	B	C	D	E	F	G	H	I	J	K
TEMPERATURE 2000°F										
0	A		3.88							
	B		3.71							
	C		3.68							
4	A	9.9	3.30	0.0	180	34.8	46.3	22.5	3.3	
	B	11.0	3.13	0.0		33.3	45.6	18.5	3.3	
	C		3.08			33.3	45.6	19.3	3.3	
16	A	20.4	3.19	0.3	180	31.4	44.0	14.3	2.6	
	B	20.6	3.02	0.2		31.5	44.3	14.5	2.6	
	C		3.00			30.4	41.9	13.5	2.6	
64	A	33.7	3.13	0.5	180	29.9	41.8	12.0	2.3	
	B	34.2	2.97	0.4		29.8	42.5	14.0	2.3	
	C		2.91			29.8	41.4	10.5	2.3	
100	A	26.3	3.11	0.3	180	30.0	43.1	14.0	2.2	41.4
	B	31.8	2.94	0.3		29.7	42.1	13.0	2.2	33.9
	C		2.89			31.2	42.4	12.0	2.2	36.2
200	A	42.0	3.08	0.2	180	30.1	41.7	11.8	2.1	
	B	52.2	2.86	0.2		30.0	40.0	10.3	2.1	
	C		2.86			28.7	40.6	10.5	2.1	
300	A	60.5	3.08	0.2	180	29.4	38.1	12.3	1.8	
	B	55.2	2.86	0.3		30.8	40.9	13.0	1.8	
	C		2.86			29.4	40.1	14.5	1.8	
400	A	59.2	3.08	0.4	180	30.7	40.6	14.5	1.9	
	B	61.3	2.86	0.4		31.0	40.8	13.5	1.9	
	C		2.86			28.0	36.9	12.0	1.9	
500	A	52.7	3.08	0.3	180	30.8	41.1	11.5	2.1	
	B	57.0	2.86	0.4		29.4	40.5	13.0	2.1	
	C		2.83			28.6	37.9	11.8	2.1	
600	A	76.2	3.08	0.3	180	29.6	38.5	9.0	2.1	0
	B	75.3	2.86	0.4		28.6	38.0	13.3	2.1	0
	C		2.83			30.2	37.5	7.5	2.1	0

TABLE A-3 - NUMERICAL DATA TABULATION: ALLOY H 875

EXPOSURE TIME - HOURS	SPECIMEN	SPECIFIC OXIDATION WEIGHT GAIN MG/IN^2	PERMEABILITY AT 10 PSID $SCFM/IN^2$	THICKNESS CHANGE IN x 10^3	BEND TEST 3T RADIUS ° BEND	YIELD STRENGTH 0.2% OFFSET PSI x 10^{-3}	ULTIMATE STRENGTH PSI x 10^{-3}	ELONGATION IN 1 INCH %	MODULUS OF ELASTICITY PSI x 10^{-6}	JOINT TENSILE STRENGTH PSI x 10^{-3}
A	B	C	D	E	F	G	H	I	J	K
TEMPERATURE 1800°F										
0	A		4.40		180	41.5	55.4	14.8	3.0	45.1
	B		4.19			42.8	57.0	15.3	3.0	45.6
	C		4.74			40.1	55.9	16.0	3.0	45.8
4	A	4.62	4.12	0.2	180	43.1	58.4	22.0	3.6	
	B	4.68	3.96	0.2		42.9	58.5	21.0	3.6	
	C		4.50			41.6	58.1	24.0	3.6	
16	A	6.31	4.07	0.3	180	43.9	58.6	21.3	2.8	
	B	7.47	3.88	0.4		43.1	58.8	22.8	2.8	
	C		4.45			42.2	57.1	19.8	2.8	
64	A	8.32	3.96	0.5	180	44.4	59.0	21.0	3.4	
	B	9.09	3.74	0.3		43.9	58.2	18.0	3.4	
	C		4.29			43.4	57.4	18.5	3.4	
100	A	9.62	3.85	0.4	180	44.1	59.0	21.5	3.0	58.4
	B	9.05	3.63	0.5		43.3	58.3	20.8	3.0	57.5
	C		4.18			40.6	56.4	19.8	3.0	55.0
200	A	11.28	3.77	0.6	180	39.4	57.7	20.3	2.7	
	B	10.20	3.55	0.6		44.0	57.7	20.5	2.7	
	C		4.13			41.9	56.2	19.5	2.7	
300	A	13.32	3.71	0.6	180	41.9	56.9	22.0	2.6	
	B	13.95	3.41	0.6		39.0	56.1	18.5	2.6	
	C		4.01			40.9	55.0	16.0	2.6	
400	A	14.30	3.63	0.5	180	42.6	56.0	15.3	3.3	
	B	15.65	3.35	0.7		41.9	56.7	15.5	3.3	
	C		3.95			42.4	55.6	16.8	3.3	
500	A	15.22	3.57	0.7	180	39.0	54.8	12.9	3.1	
	B	15.64	3.30	0.6		43.3	56.4	15.5	3.1	
	C		3.85			39.5	55.4	15.3	3.1	
600	A	16.75	3.52	0.5	180	42.4	56.1	16.5	3.3	59.0
	B	17.85	3.27	0.5		38.5	57.0	17.6	3.3	56.5
	C		3.85			38.1	55.3	21.0	3.3	57.6

TABLE A-4 - NUMERICAL DATA TABULATION: ALLOY H 875

EXPOSURE TIME - HOURS	SPECIMEN	SPECIFIC OXIDATION WEIGHT GAIN MG/IN^2	PERMEABILITY AT 10 PSID SCFM/IN2	THICKNESS CHANGE IN x 103	BEND TEST 3T RADIUS ° BEND	YIELD STRENGTH 0.2% OFFSET PSI x 10-3	ULTIMATE STRENGTH PSI x 10-3	ELONGATION IN 1 INCH %	MODULUS OF ELASTICITY PSI x 10-6	JOINT TENSILE STRENGTH PSI x 10-3
A	B	C	D	E	F	G	H	I	J	K
TEMPERATURE 2000°F										
	A		4.10							
0	B		4.06							
	C		4.24							
	A	7.6	3.68	0.2	180	42.4	58.5	20.0	3.0	
4	B	8.3	3.63	0.5		41.5	57.9	20.0	3.0	
	C		3.79			40.7	54.7	13.9	3.0	
	A	12.6	3.30	0.3	180	41.4	58.0	19.3	2.7	
16	B	14.5	3.30	0.3		40.9	57.5	20.5	2.7	
	C		3.41			40.0	56.7	15.5	2.7	
	A	21.2	2.86	0.2	180	40.4	56.5	24.5	2.8	
64	B	22.4	2.86	0.4		39.4	54.6	16.3	2.8	
	C		3.00			41.0	55.6	15.5	2.8	
	A	25.0	2.75	0.3	180	39.3	54.5	18.5	2.7	52.6
100	B	25.8	2.75	0.3		37.2	53.4	13.8	2.7	52.4
	C		2.86			41.3	55.5	20.5	2.7	50.1
	A	32.8	2.47	0.3	180	39.0	51.7	12.5	2.6	
200	B	35.1	2.53	0.2		35.8	50.9	11.8	2.6	
	C		2.65			37.9	53.7	18.5	2.6	
	A	35.7	2.39	0.4	180	38.7	53.2	12.8	2.3	
300	B	38.6	2.42	0.4		39.5	52.1	15.0	2.3	
	C		2.53			38.3	52.2	16.0	2.3	
	A	40.5	2.28	0.3	180	40.6	53.1	16.5	2.3	
400	B	42.3	2.34	0.4		39.9	51.0	11.5	2.3	
	C		2.47			37.9	51.5	18.5	2.3	
	A	44.5	2.20	0.3	180	39.0	50.6	14.5	2.4	
500	B	41.3	2.31	0.2		37.8	52.0	16.5	2.4	
	C		2.42			38.7	51.7	15.0	2.4	
	A	49.2	2.20	0.3	180	35.1	45.1	6.3	2.8	0
600	B	47.9	2.31	0.4		36.5	49.8	16.3	2.8	0
	C		2.42			36.9	51.0	14.8	2.8	0

APPENDIX B

POROLOY OXIDATION WEIGHT GAIN AND PERMEABILITY PLOTS

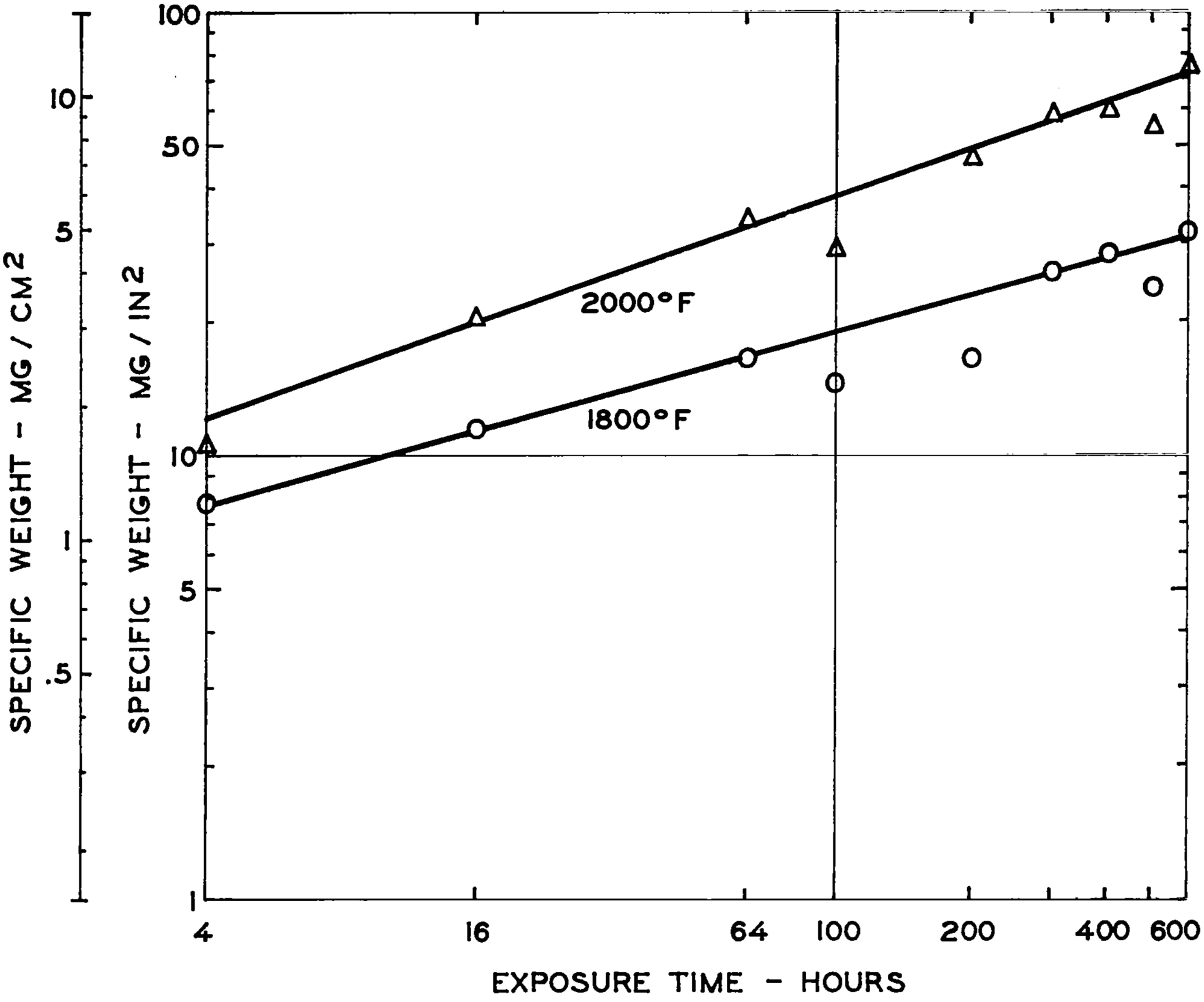


Figure B-1 Specific Oxidation Weight Gain for GE 1541 Poroloy After Exposure at 1800 and 2000°F in Air. Each point represents the average of two specimens.

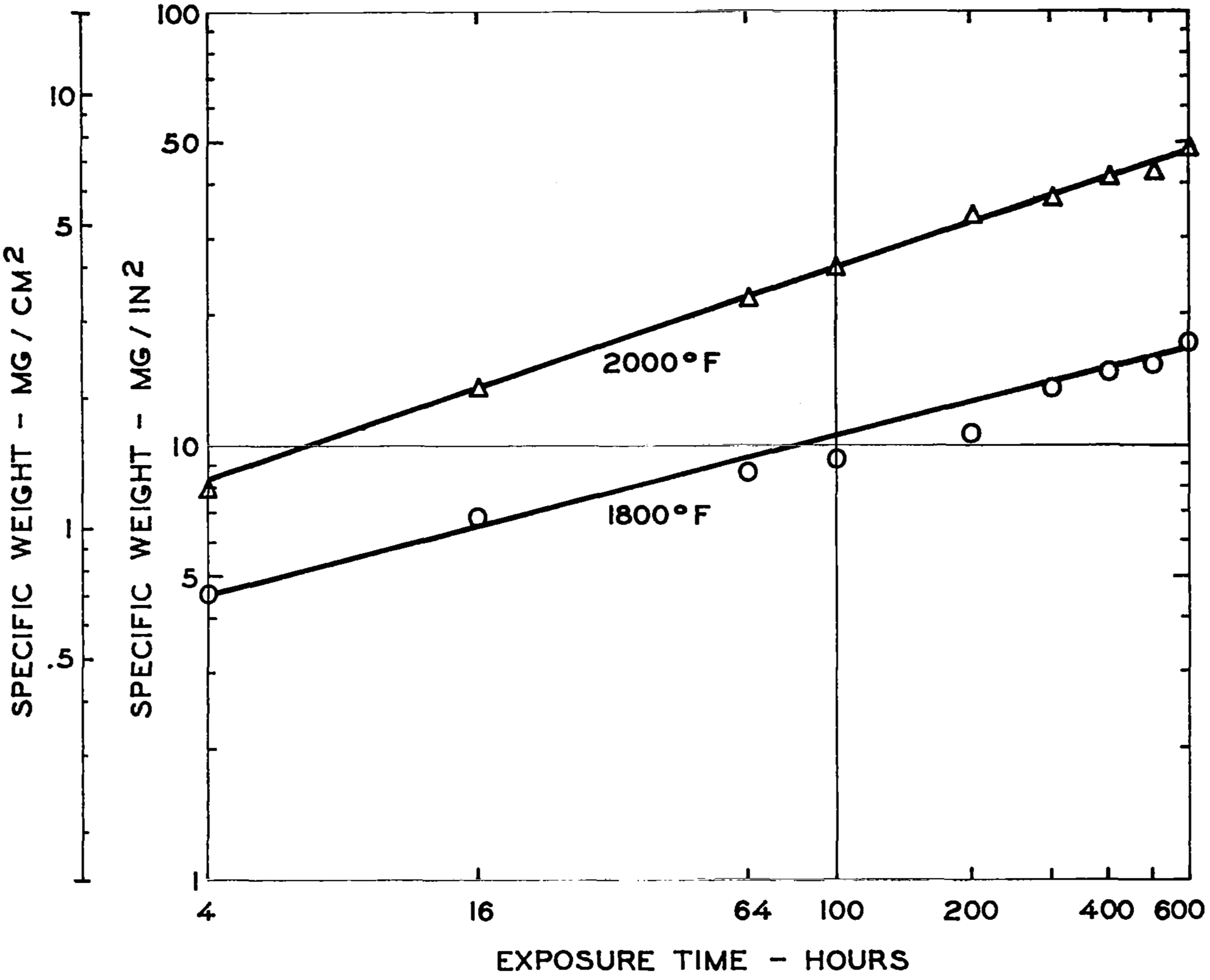


Figure B-2 Specific Oxidation Weight Gain for H 875 Poroloy After Exposure at 1800 and 2000°F in Air. Each point represents the average of two specimens.

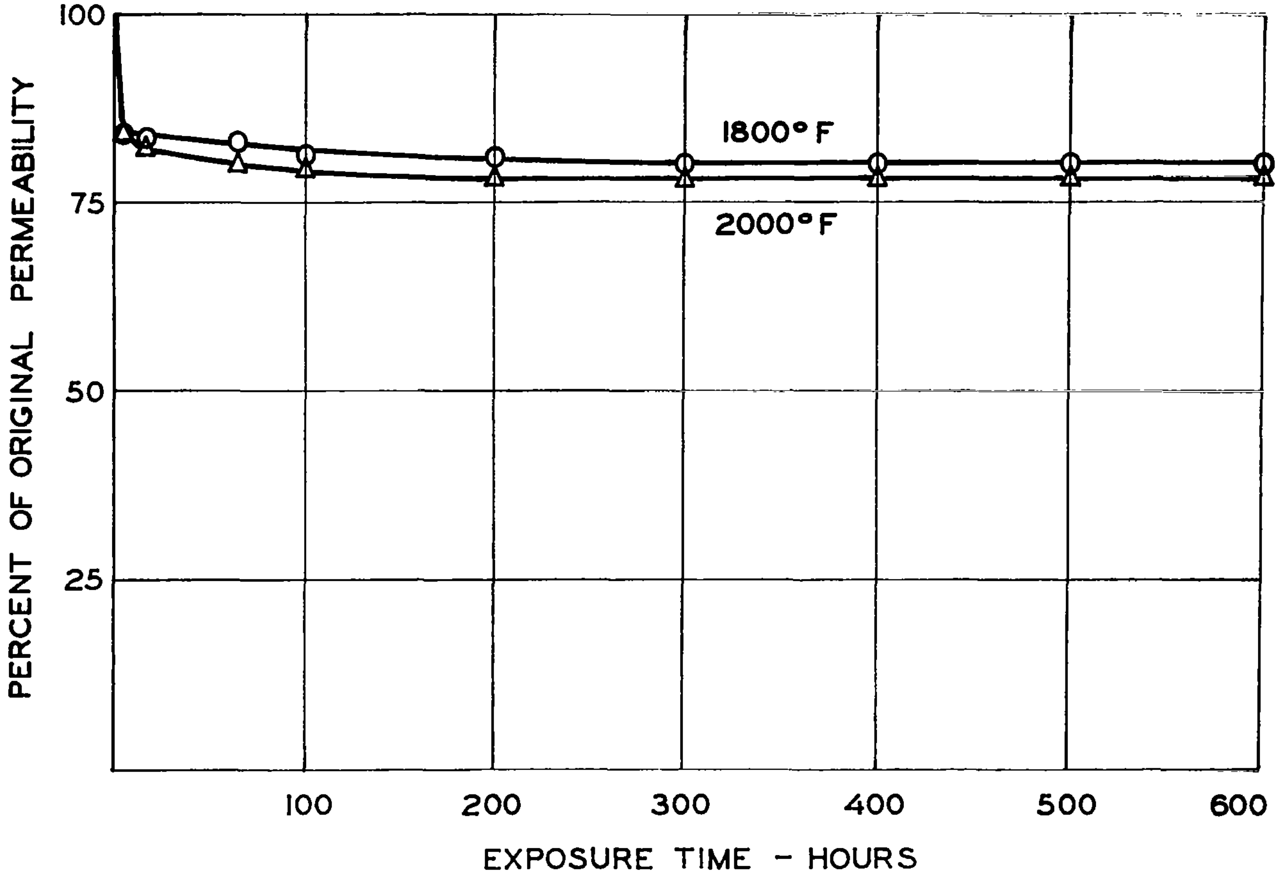


Figure B-3 GE 1541 Poroloy Permeability Change After Exposure at 1800 and 2000°F in Air. Each point represents the average of three specimens.

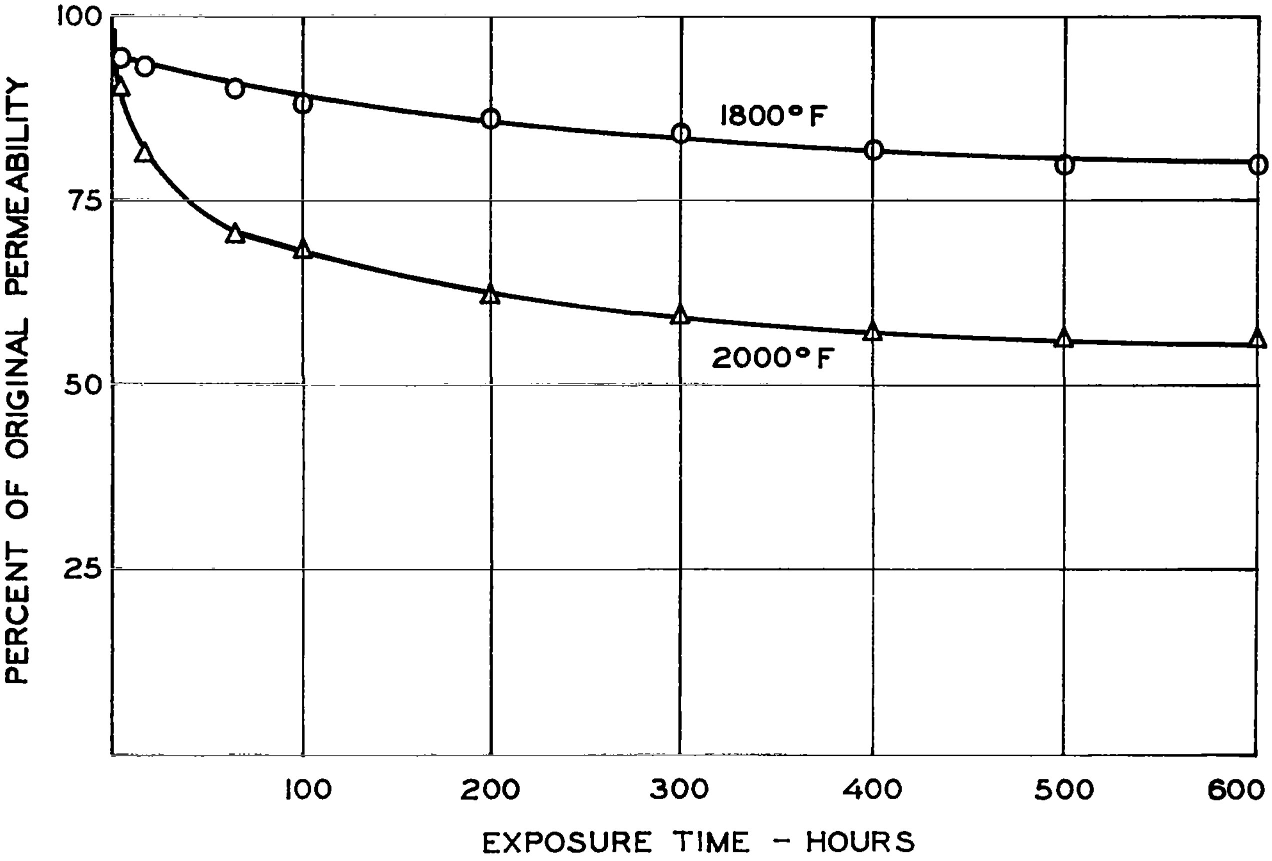


Figure B-4 H 875 Poroloy Permeability Change After Exposure at 1800 and 2000°F in Air. Each point represents the average of three specimens.

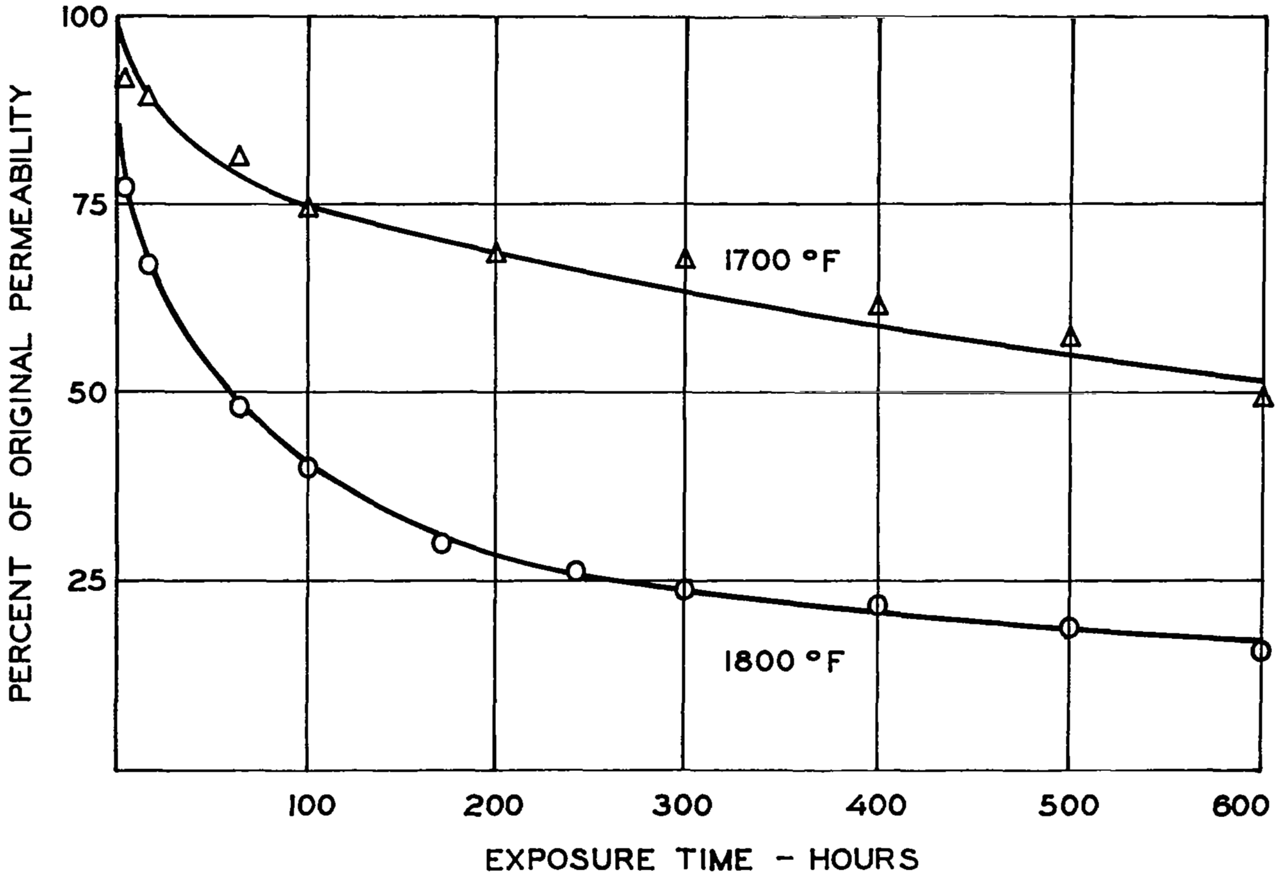


Figure B-5 - DH 242 Poroloy (0.005 Dia. Wire) Permeability Change
After Exposure at 1700 and 1800°F in Air.

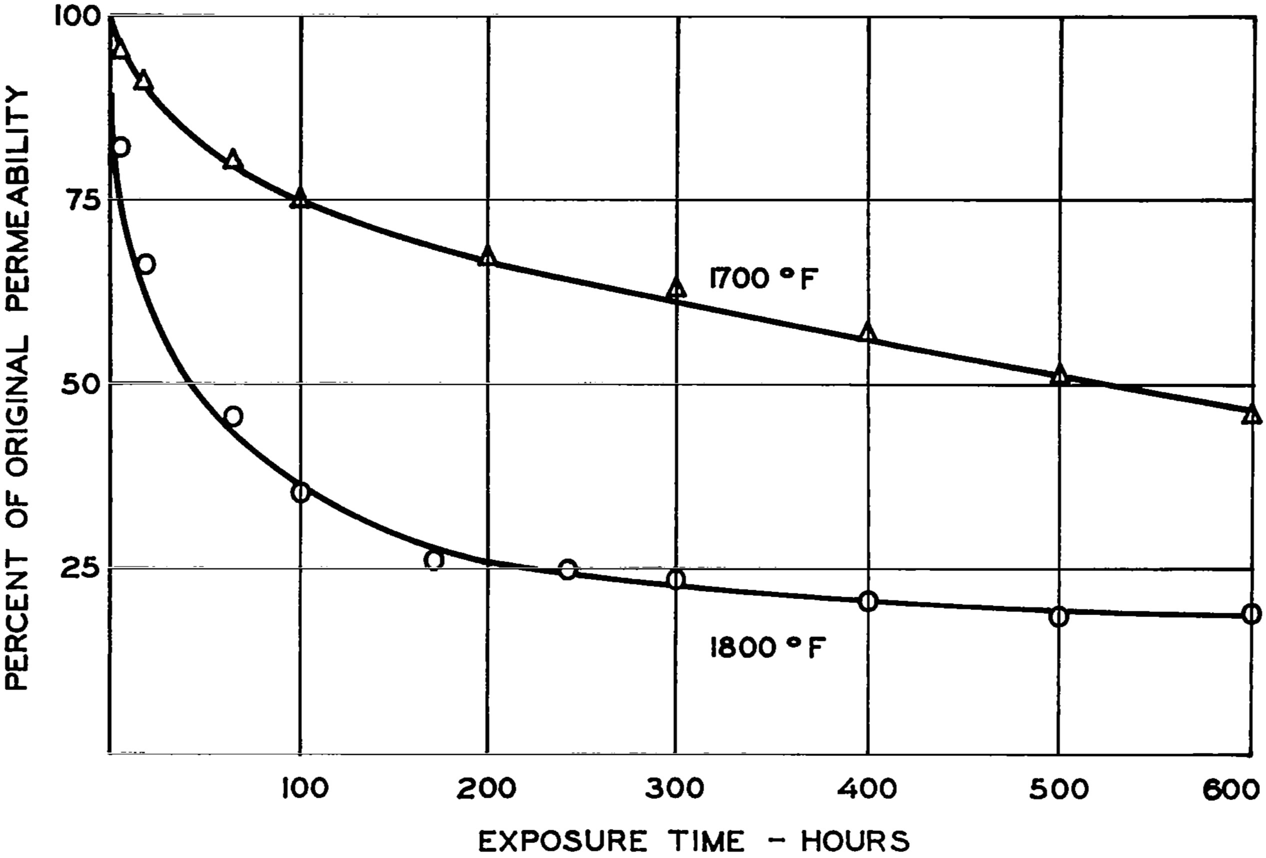


Figure B-6 DH 242 Poroloy (0.010 Dia. Wire) Permeability Change After Exposure at 1700 and 1800°F in Air.

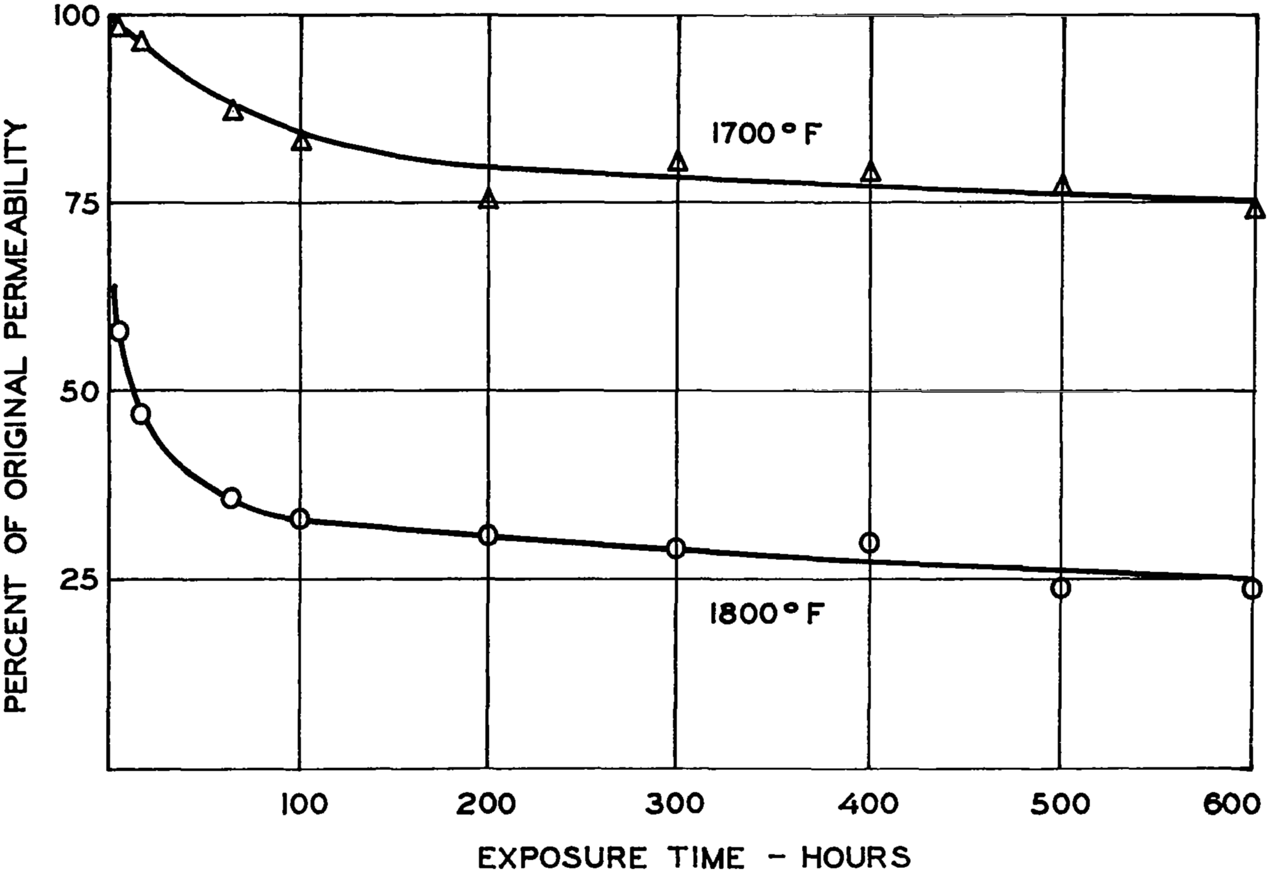


Figure B-7 - TD Ni Cr Poroloy Permeability Change After Exposure at 1700 and 1800°F in Air. (Note: Initial permeability of the 1800°F sample was 40 percent lower than the initial permeability of the 1700°F sample.

APPENDIX C

METALLOGRAPHIC EXAMINATION — CYCLIC OXIDATION

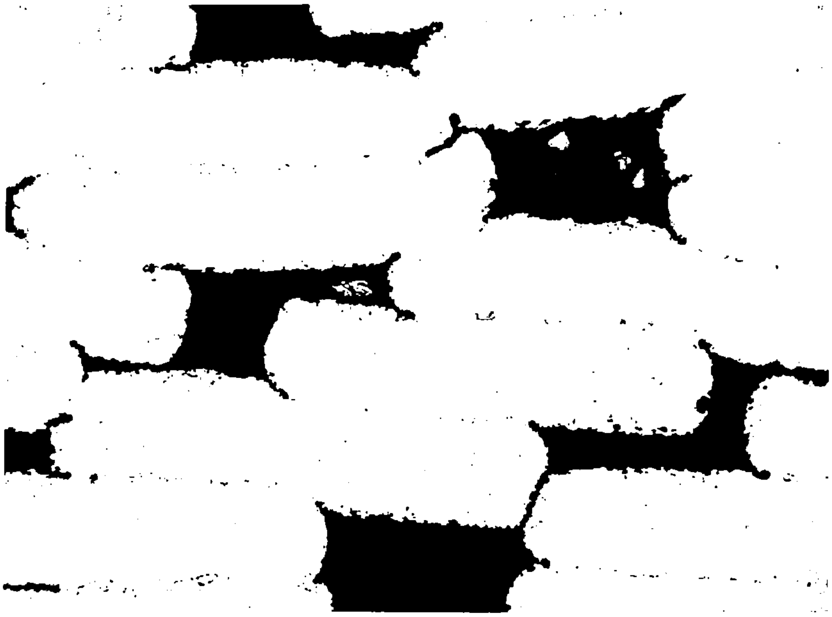
Sample	Oxidation Temperature	Figure	Page
GE 1541 Poroloy	1800°F	C-1	54
H 875 Poroloy	1800°F	C-2	57
GE 1541/IN 100 weld zone	1800°F	C-3	60
H 875/IN 100 weld zone	1800°F	C-4	63
GE 1541 Poroloy	2000°F	C-5	66
H 875 Poroloy	2000°F	C-6	69
GE 1541/IN 100 weld zone	2000°F	C-7	72
H 875/IN 100 weld zone	2000°F	C-8	74
IN 100 Slug	2000°F	C-9	76
H 875/IN 100 weld sample	2000°F	C-10	76
GE 1541/IN 100 weld sample	2000°F	C-11	77
GE 1541 Permeability sample	2000°F	C-12	77

0 Hours

4 Hours

Figure C-1 GE 1541 Poroloy following air oxidation at 1800°F for the indicated times. X250

64 Hours



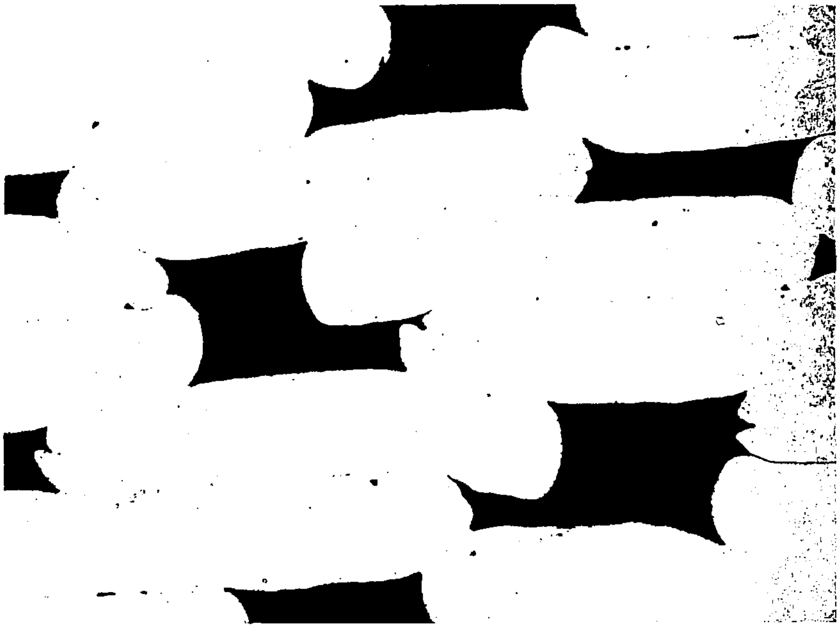
200 Hours

Figure C-1 Continued

400 Hours

600 Hours

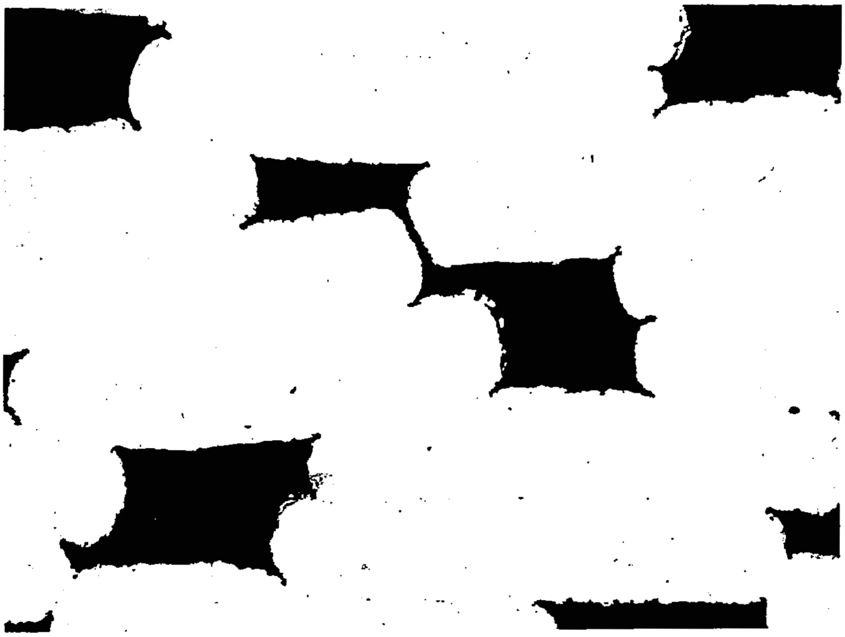
Figure C-1 Concluded



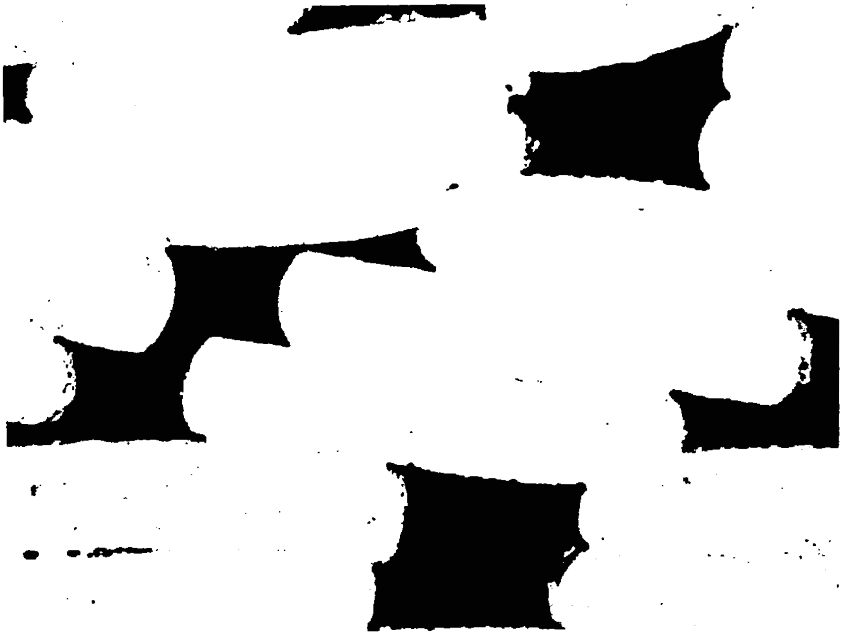
0 Hours

4 Hours

Figure C-2 H 875 Poroloy following air oxidation at 1800°F for the indicated times. X250



64 Hours



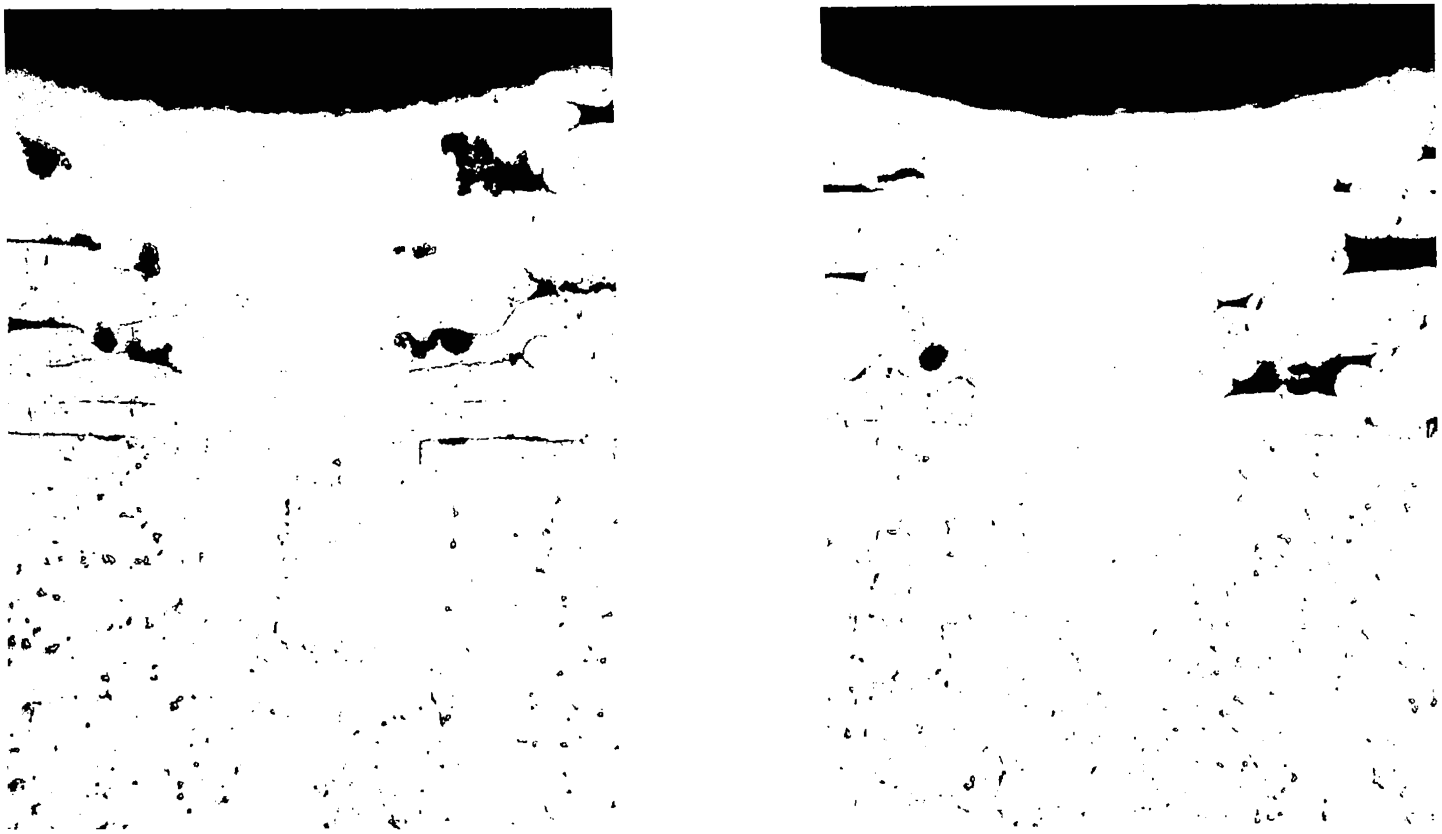
200 Hours

Figure C-2 Continued

400 Hours

600 Hours

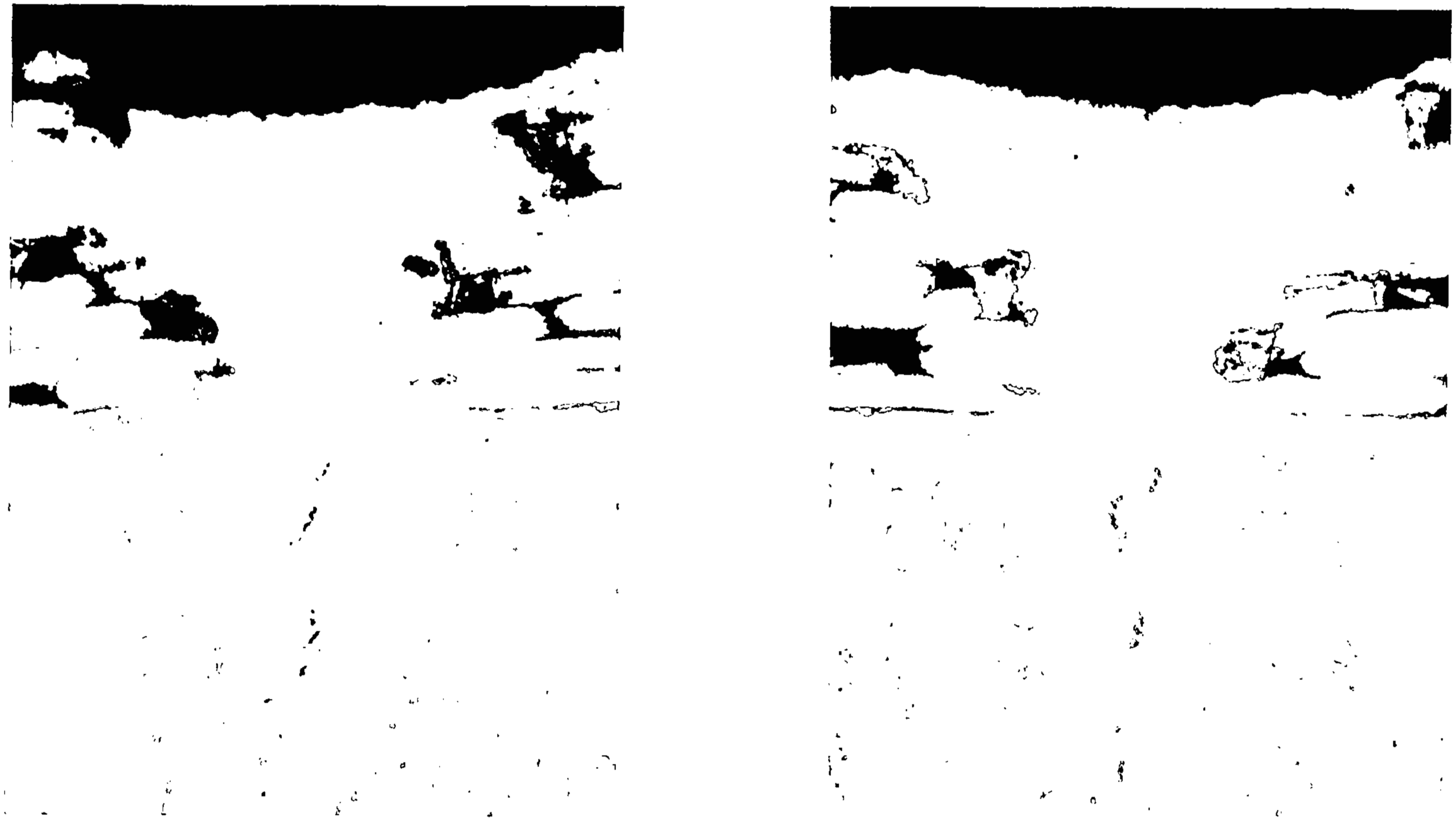
Figure C-2 Concluded



0 Hours

4 Hours

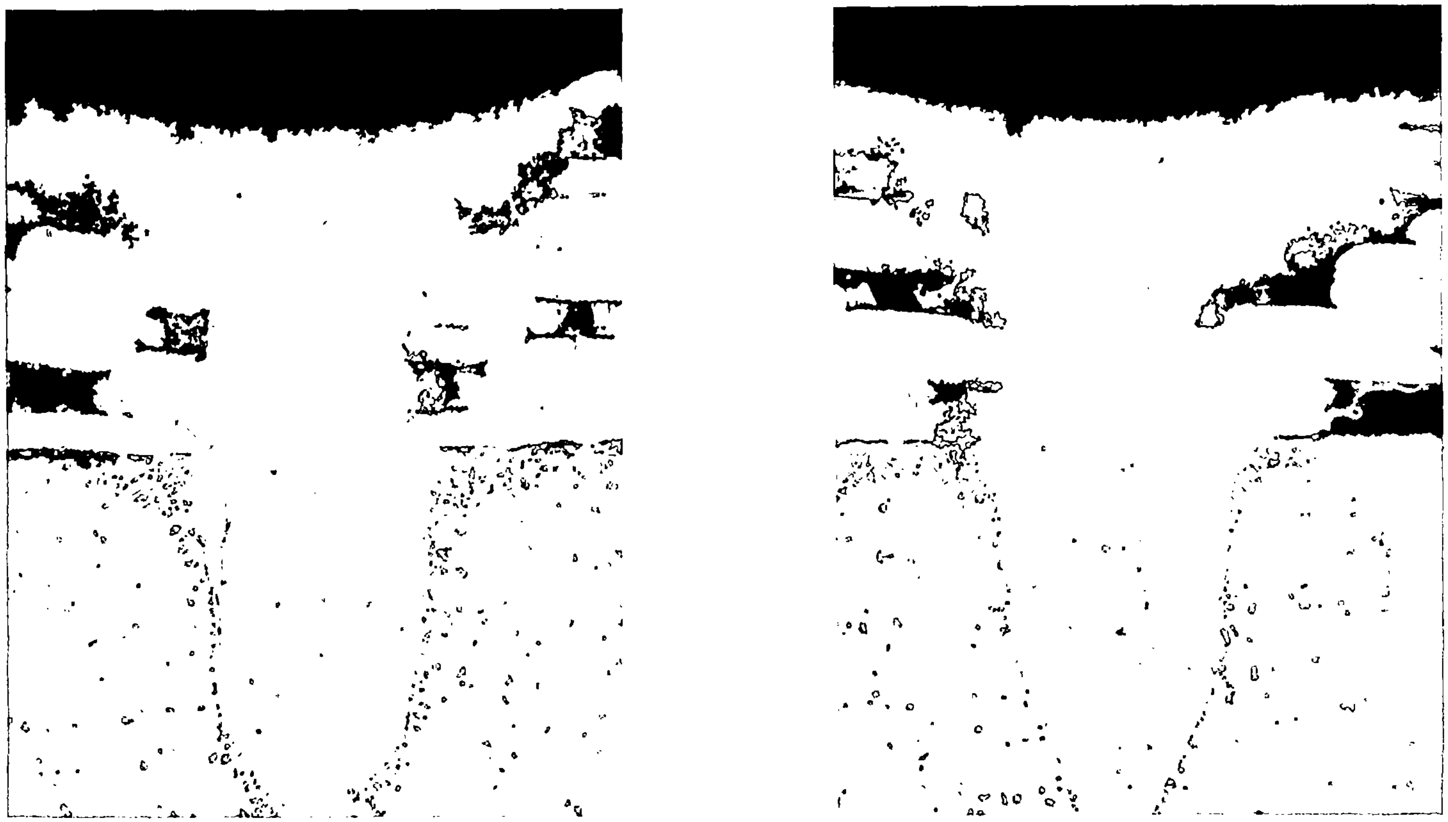
Figure C-3 Cross sections of GE 1541 Poroloy/IN 100 weld zones following air oxidation at 1800°F for the indicated times. X80



64 Hours

200 Hours

Figure C-3 Continued



400 Hours

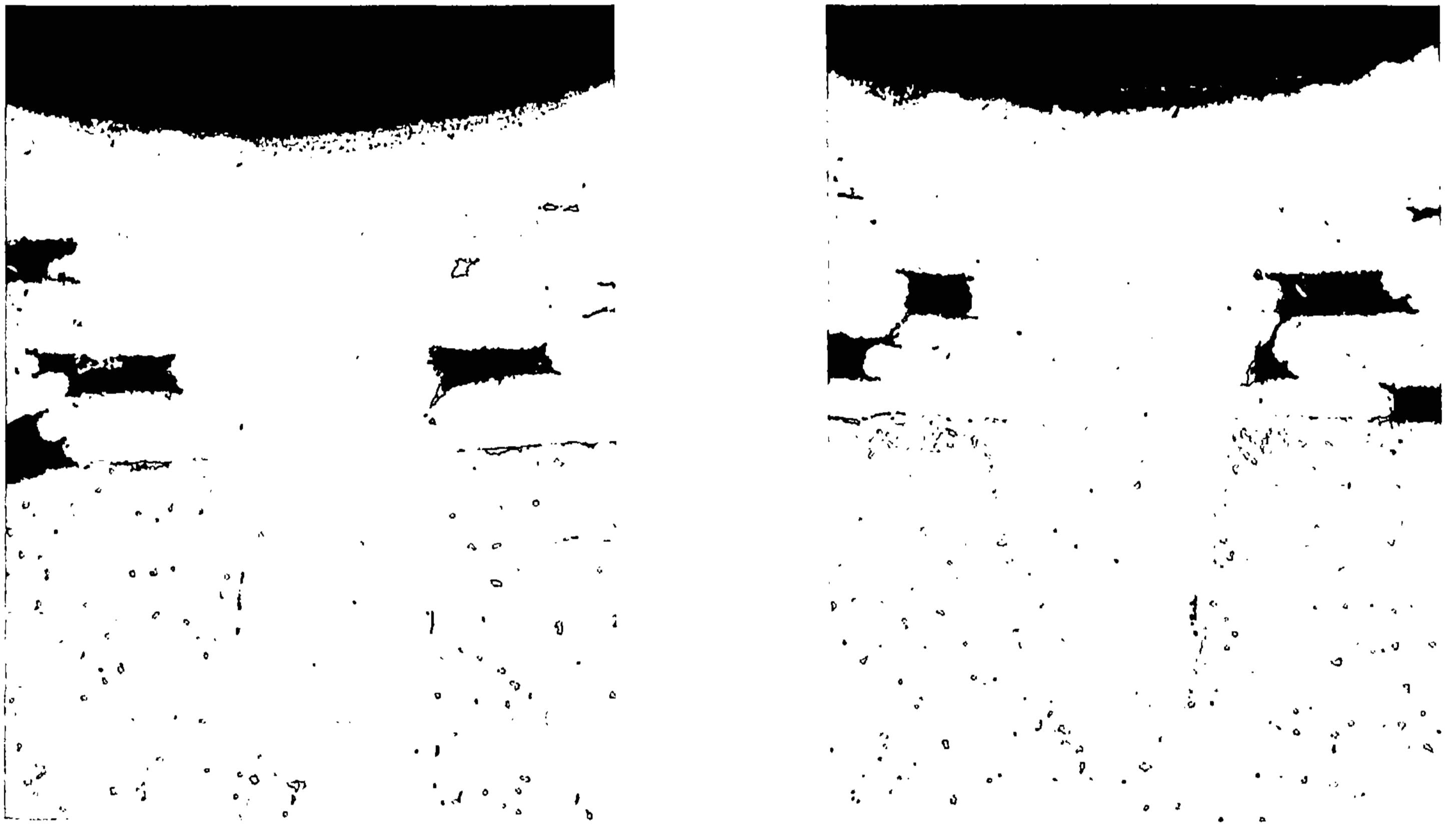
600 Hours

Figure C-3 Concluded

0 Hours

4 Hours

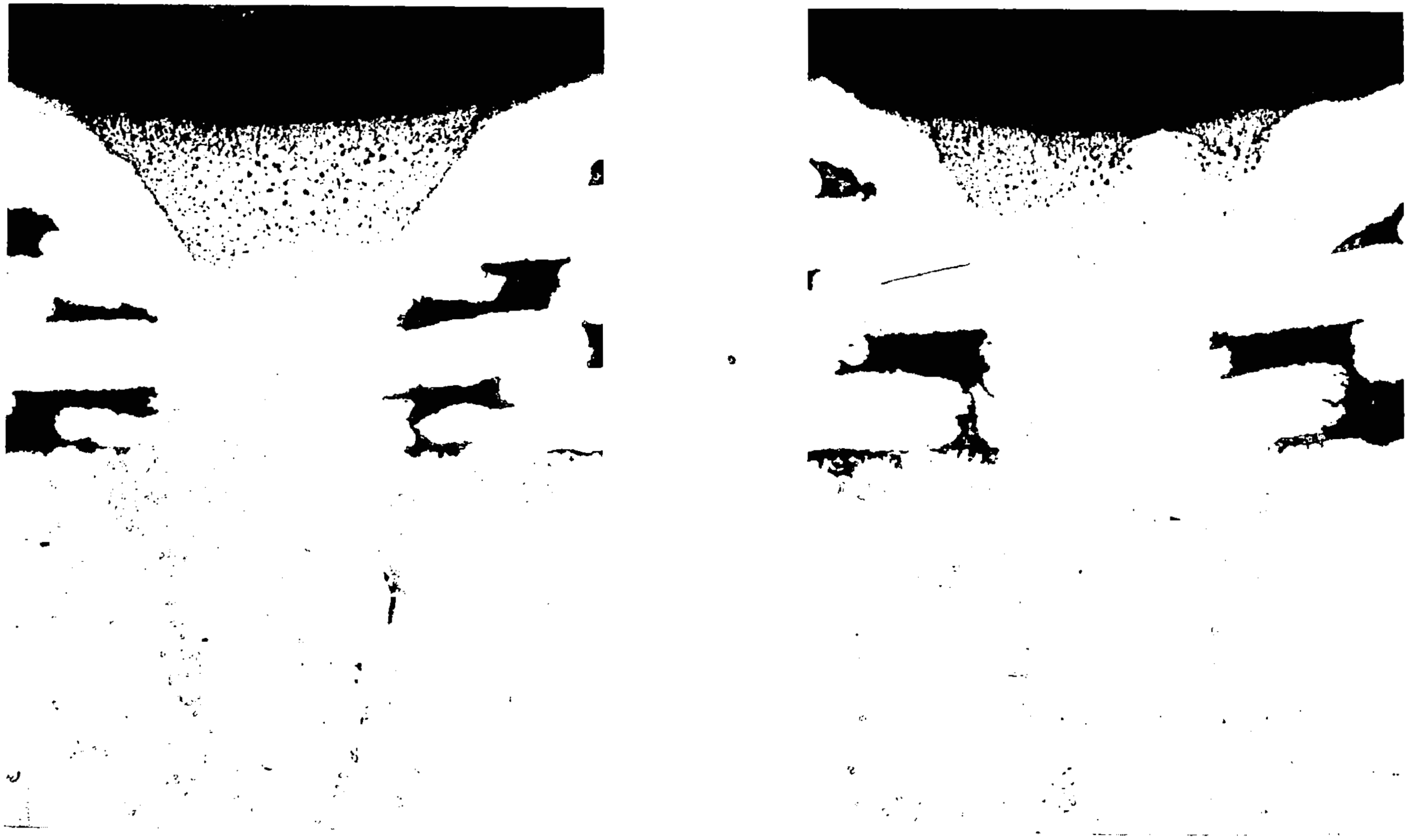
Figure C-4 Cross sections of H 875 Poroloy/IN 100 weld zones following air oxidation at 1800°F for the indicated times. X80



64 Hours

200 Hours

Figure C-4 Continued

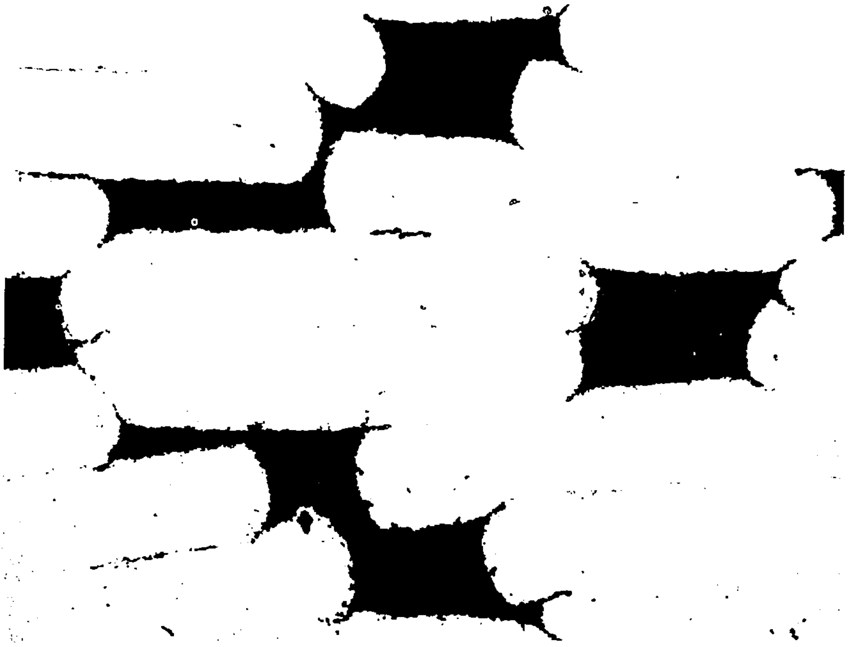


400 Hours

600 Hours

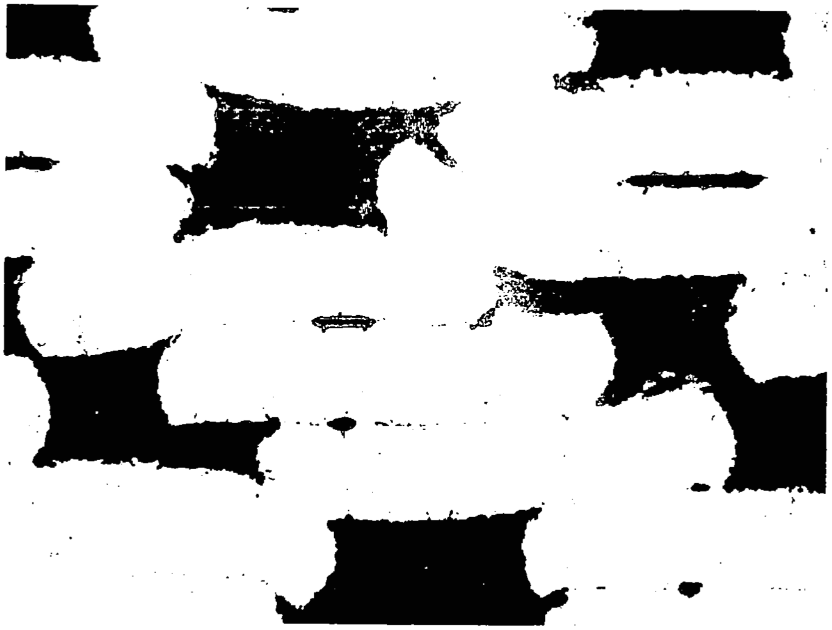
Figure C-4 Concluded

0 Hours

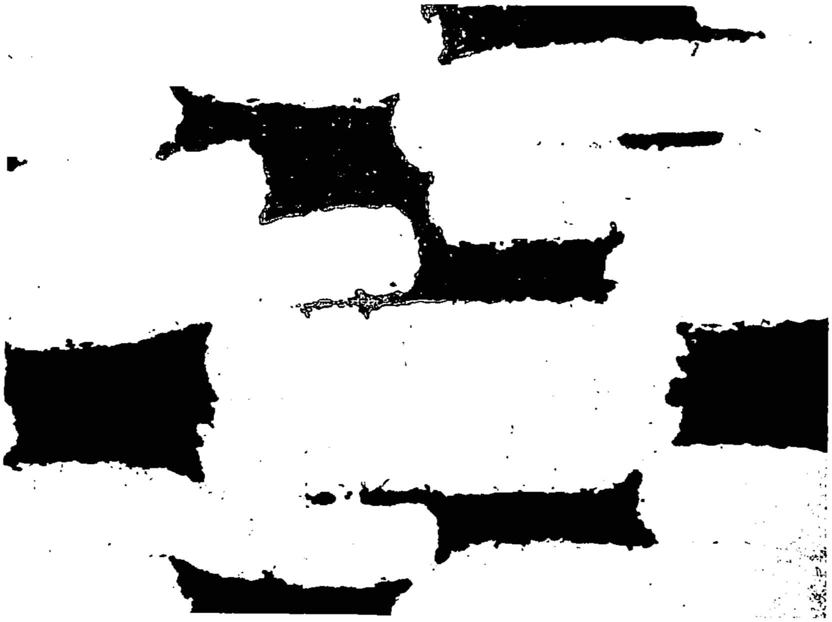


4 Hours

Figure C-5 GE 1541 Poroloy following air oxidation at 2000°F for the indicated times. X250

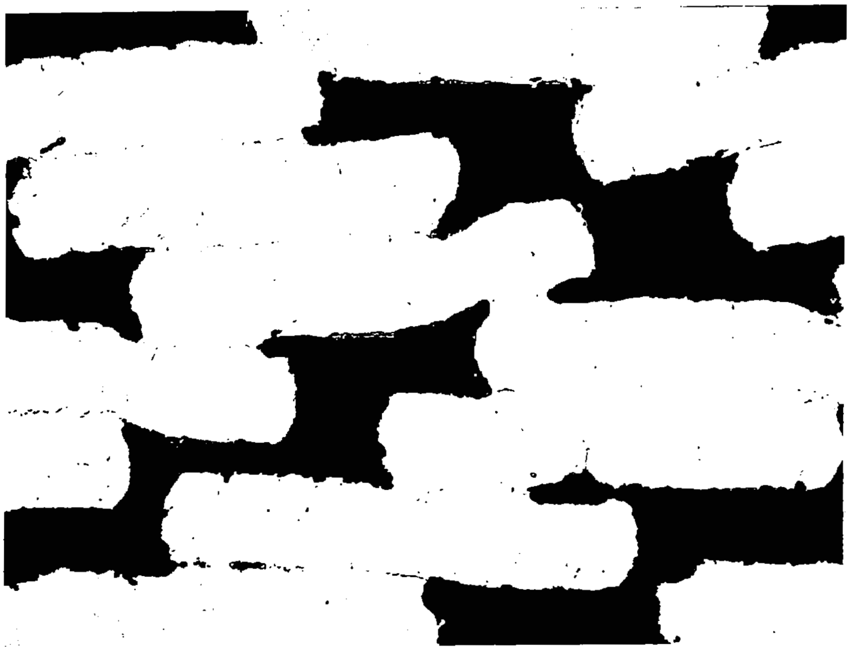


64 Hours



200 Hours

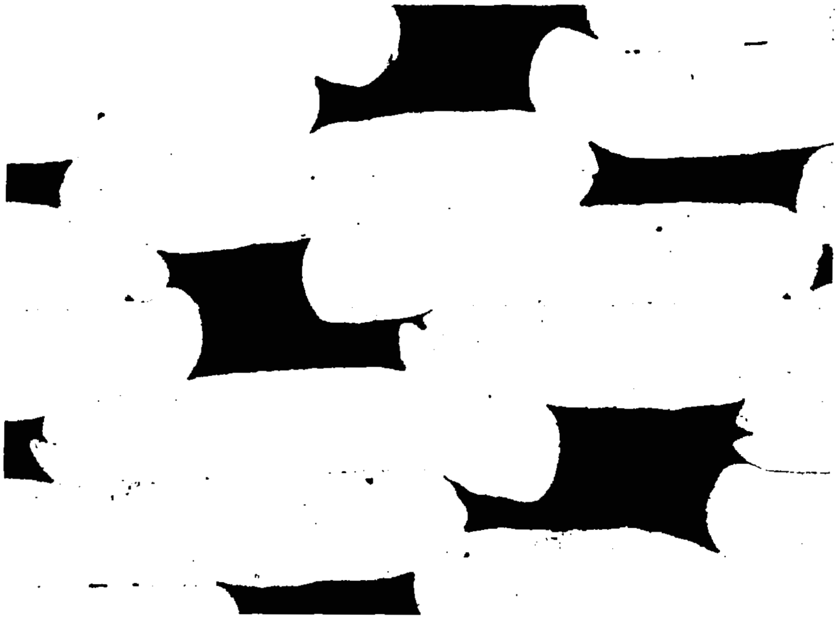
Figure C-5 Continued



400 Hours

600 Hours

Figure C-5 Concluded



0 Hours

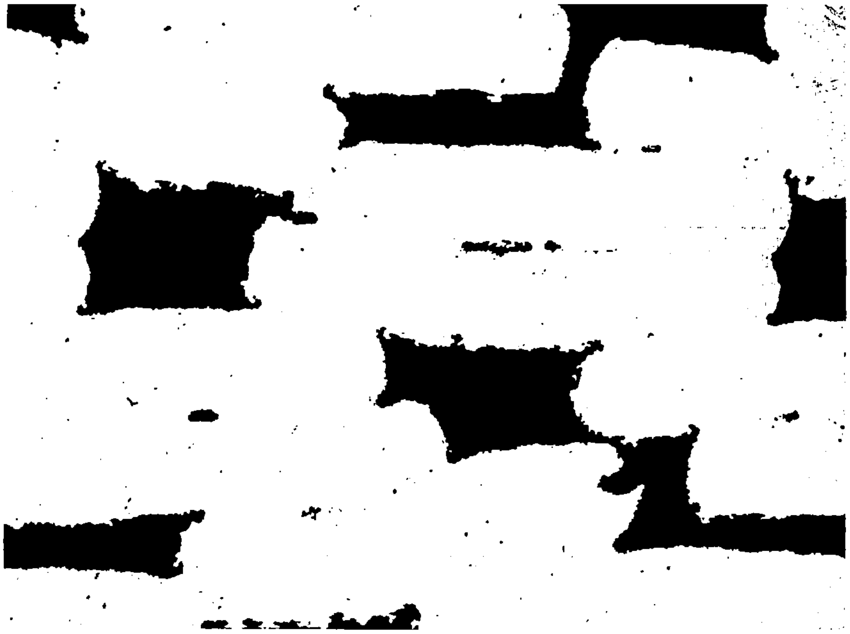
4 Hours

Figure C-6 H 875 Poroloy following air oxidation at 2000°F for the indicated times. X250

64 Hours

200 Hours

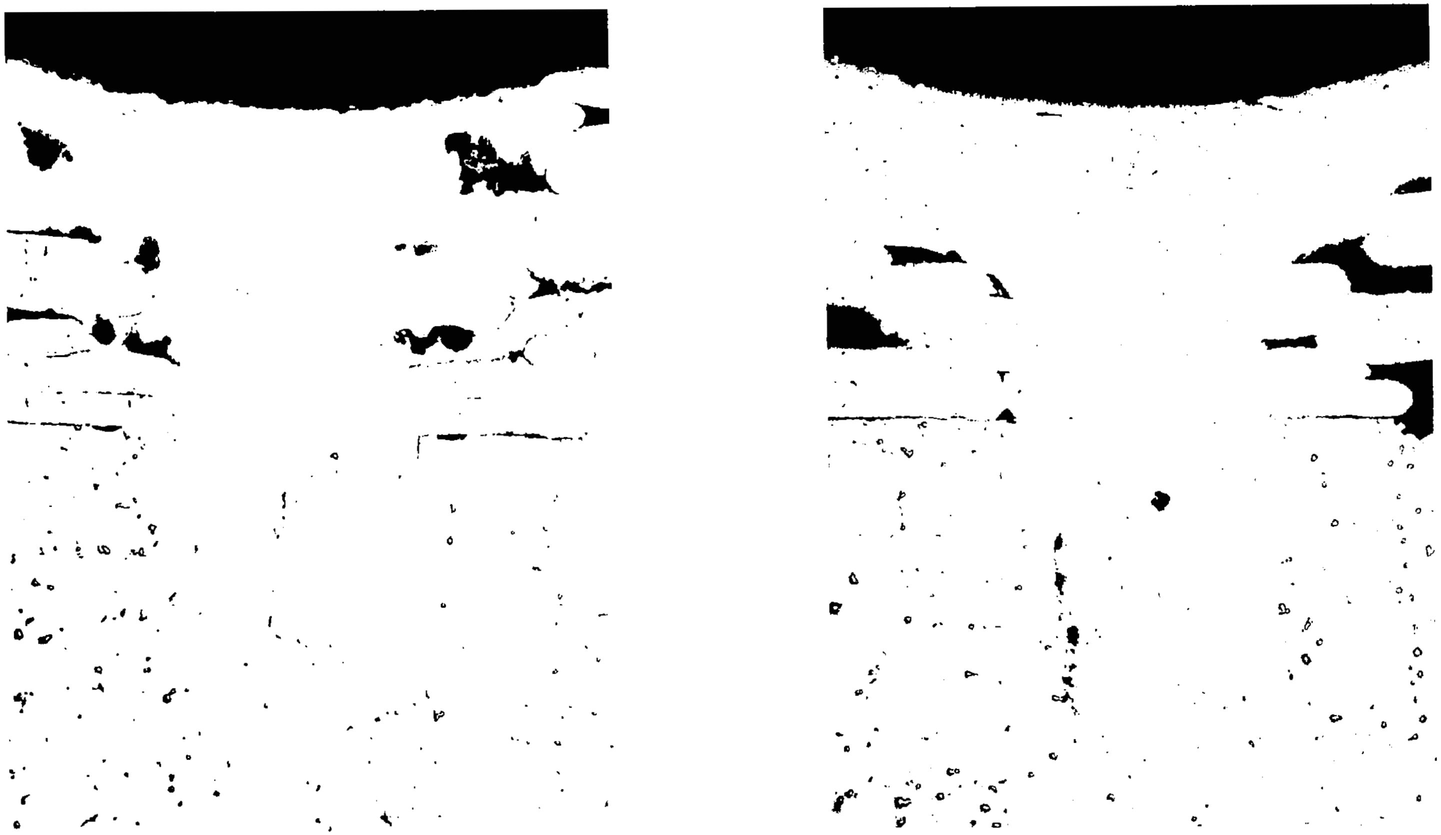
Figure C-6 Continued



400 Hours

600 Hours

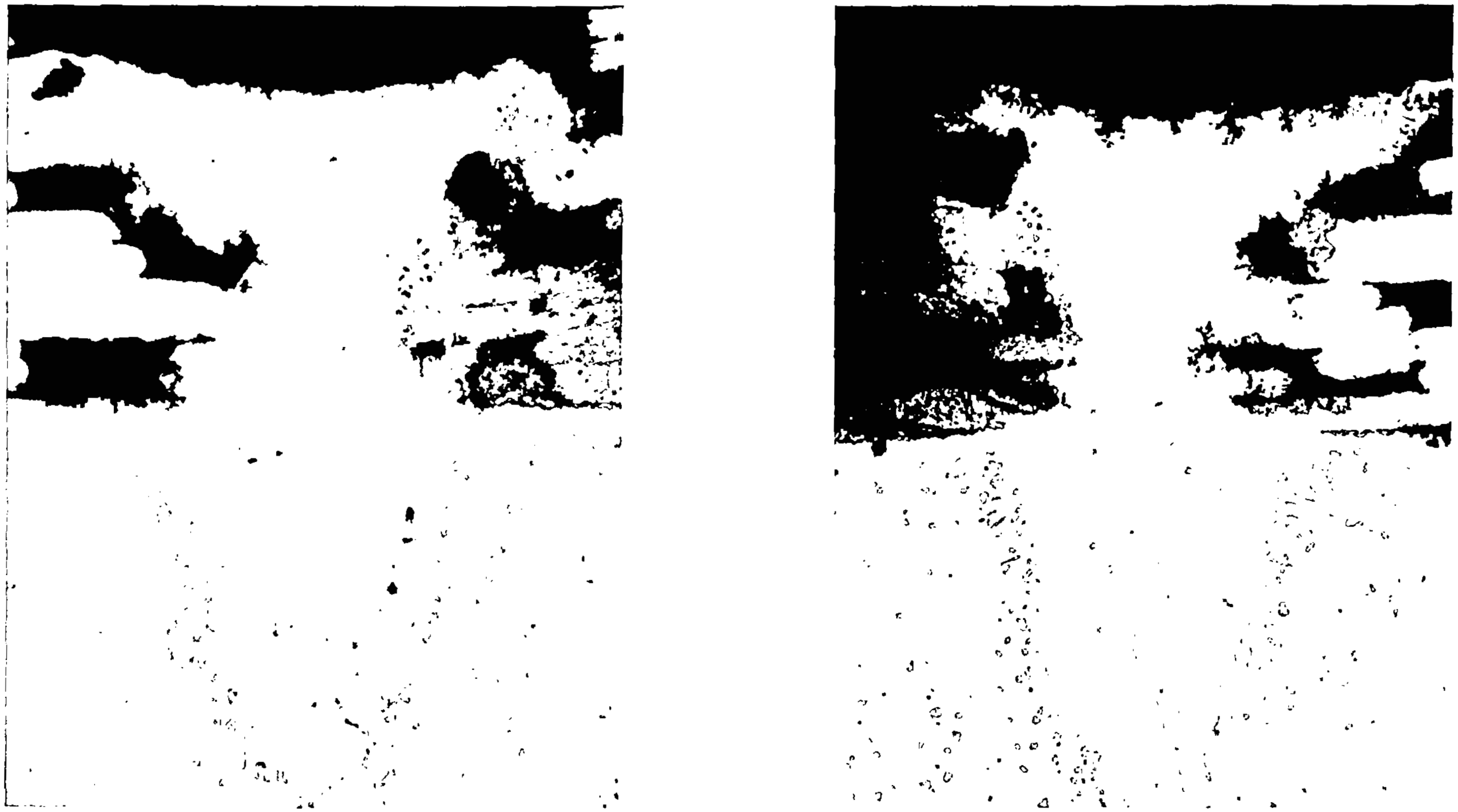
Figure C-6 Concluded



0 Hours

4 Hours

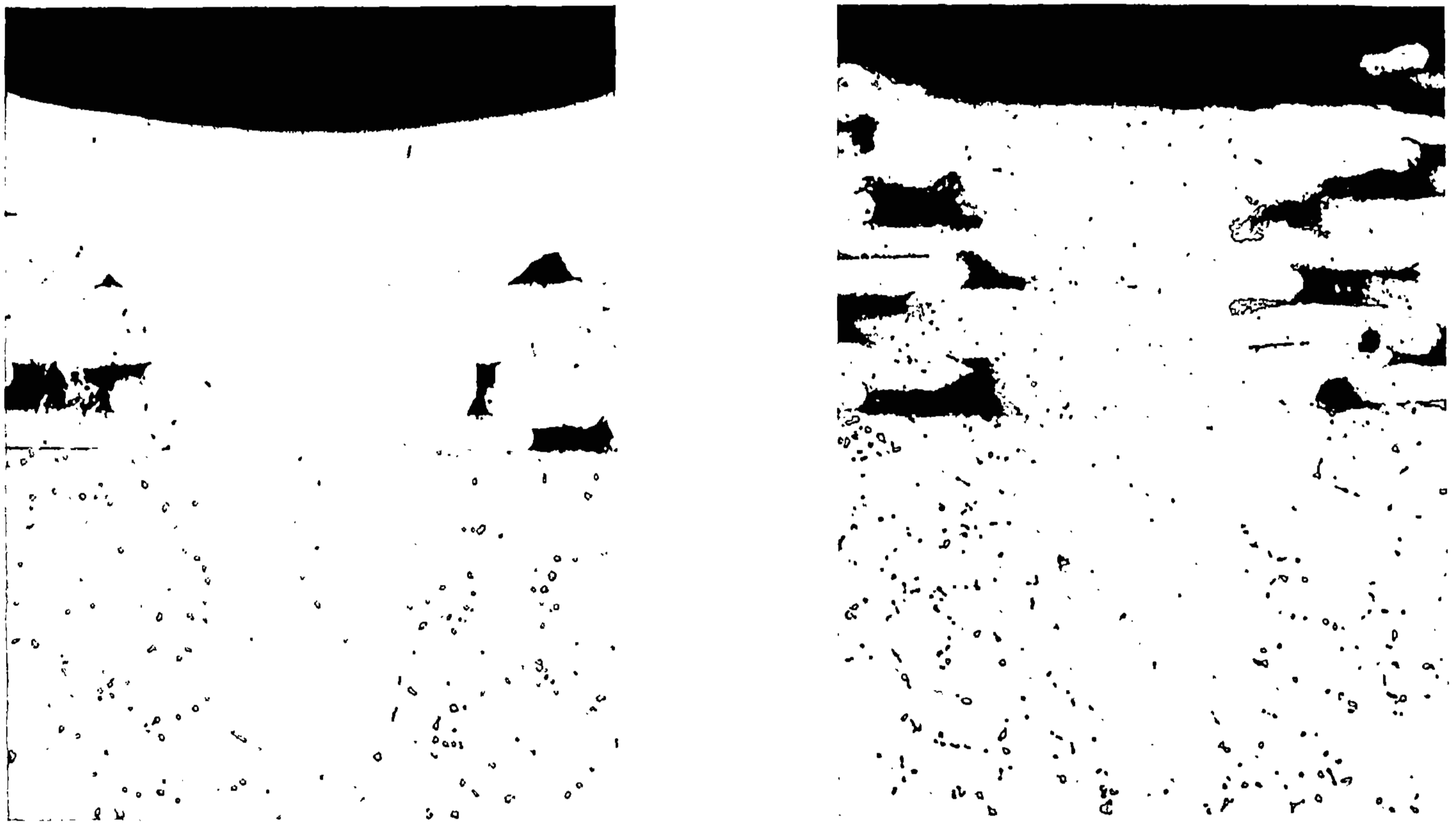
Figure C-7 Cross sections of GE 1541 Poroloy/IN 100 weld zones following air oxidation at 2000°F for the indicated times. X80



64 Hours

200 Hours

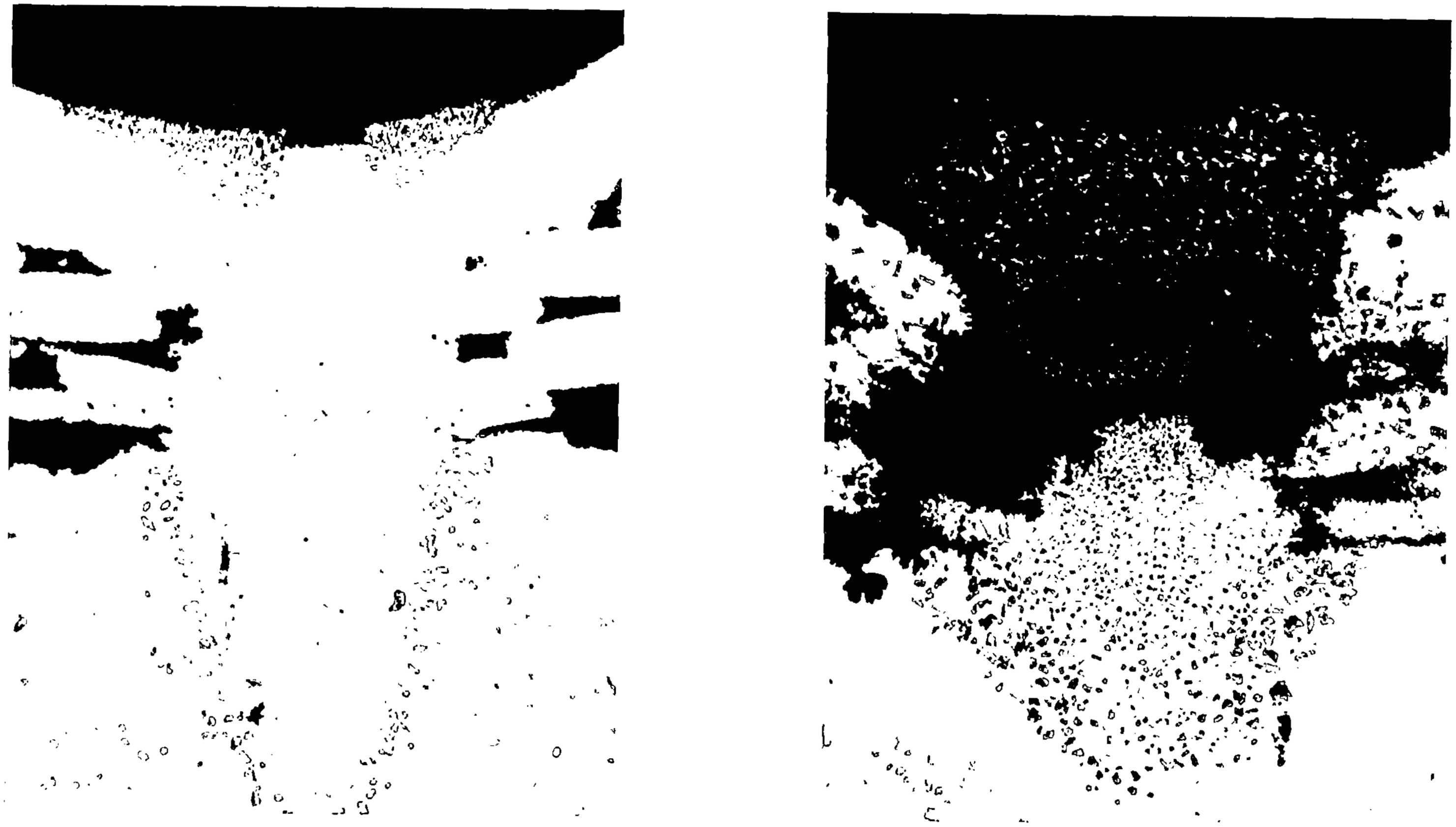
Figure C-7 Concluded



0 Hours

4 Hours

Figure C-8 Cross sections of H 875 Poroloy/IN 100 weld zones following air oxidation at 2000°F for the indicated times. X80



64 Hours

400 Hours

Figure C-8 Concluded

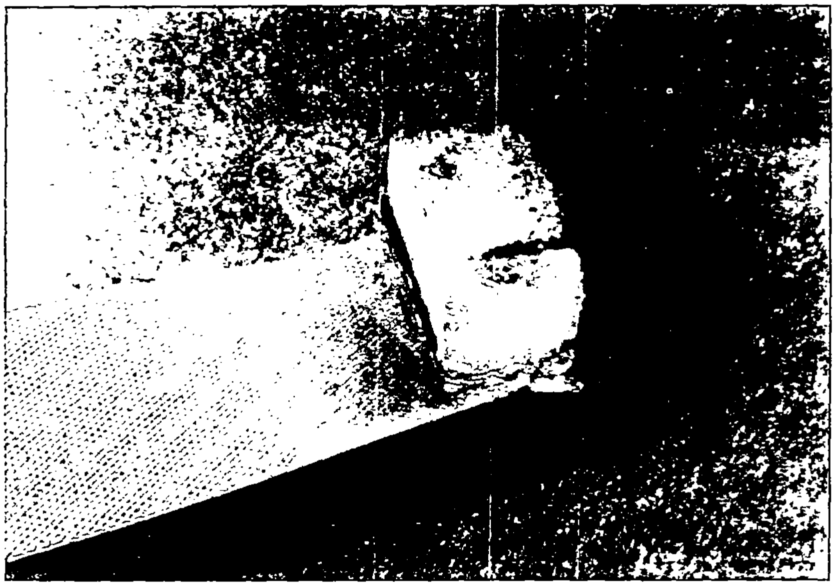


Figure C-9 Photograph of the underside of H 875/IN 100 weld sample showing extensive oxidation of the IN 100 slug following air oxidation at 2000°F for 600 hours.

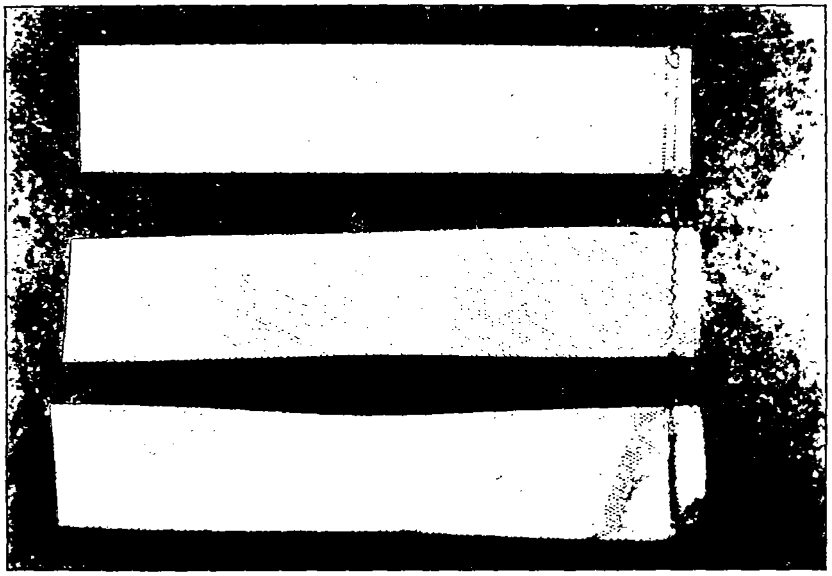


Figure C-10 H 875/IN 100 weld samples following air oxidation at 2000°F for 4 (above) 500 (center) and 600 (below) hours exposure.

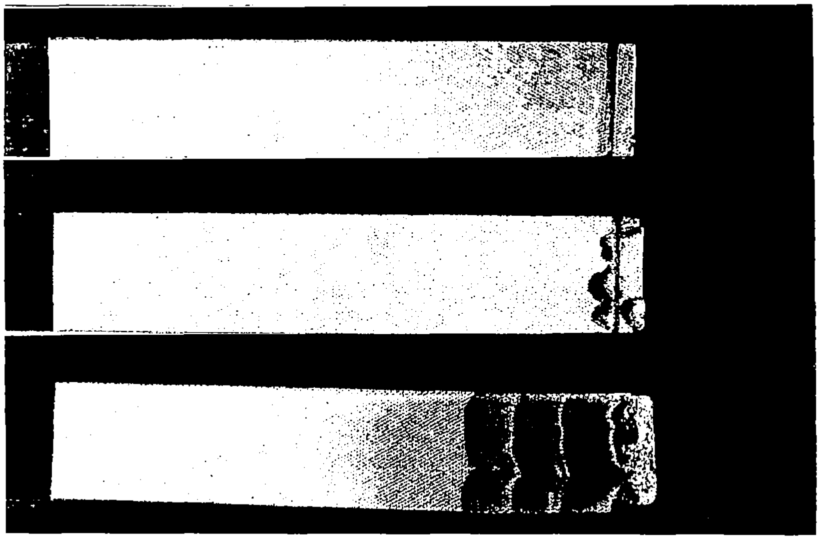


Figure C-11 GE 1541/IN 100 weld samples following air oxidation at 2000°F for 4 (above), 200 (center) and 400 (below) hours. Samples receiving longer exposure were no longer intact.

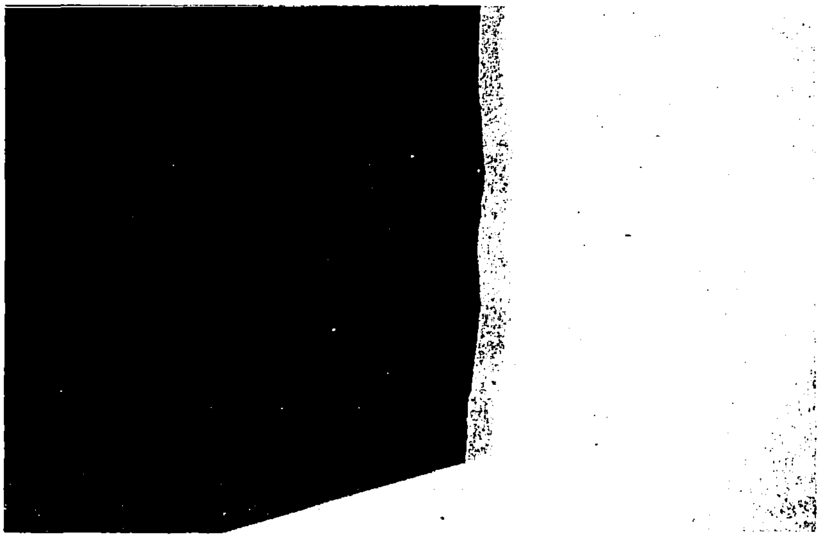


Figure C-12 Photograph of the edge of a GE 1541 permeability sample illustrating initiation of severe oxidation following contact with the basket after 600 hours exposure at 2000°F.

APPENDIX D

STRESS RUPTURE, HOT TENSILE AND STRESS OXIDATION TESTS

Tabulated Data
Stress Rupture and Hot Tensile Tests

Alloy	Test	Table	Page
GE 1541 Wire	Stress Rupture	D-1	80
H 875 Wire	Stress Rupture	D-2	80
GE 1541, H 875 and DH 245 Poroloy	Stress Rupture	D-3	81
GE 1541, H 875 Wire	Hot Tensile	D-4	82
GE 1541, H 875 and DH 245 Poroloy	Hot Tensile	D-5	83

Stress Rupture Plots

Alloy	Temperature	Figure	Page
GE 1541 Wire	1800°F	D-1	84
GE 1541 Wire	2000°F	D-2	84
H 875 Wire	1800°F	D-3	85
H 875 Wire	2000°F	D-4	85
GE 1541 Poroloy	1800°F	D-5	86
H 875 Poroloy	1800°F	D-6	86

TD Ni Cr Stress Oxidation

	Figure	Page
Wire Cross Section showing voids and internal oxidation	D-7	87
Wire longitudinal section showing voids and internal oxidation	D-8	89
Scanning electron micrograph showing large thoria particles	D-9	90

TABLE D-1 GE 1541 0.005 (0.13 MM) DIAMETER WIRE STRESS RUPTURE TEST

STRESS RUPTURE LIFE IN HOURS									
AT 1800°F (982°C)					AT 2000°F (1093°C)				
	SPECIMEN NO.					SPECIMEN NO.			
STRESS psi	1	2	3	4	STRESS psi	1	2	3	4
3035	1.79	1.04	1.45	1.08	2600	0.48	0.40	0.34	0.35
2860	3.25	3.91	3.97	2.24	2500	7.90	1.89	5.00	7.79
2800	6.50	19.40	42+	15.40	2400	20.55	25.42	31.49	18.90
2740	116+	18.51	104.79	53.11	2200	46.30	48.09	90.29	74.90

TABLE D-2 H 875 0.005 (0.13 MM) DIAMETER WIRE STRESS RUPTURE TEST

STRESS RUPTURE LIFE IN HOURS									
AT 1800°F (982°C)					AT 2000°F (1093°C)				
	SPECIMEN NO.					SPECIMEN NO.			
STRESS psi	1	2	3	4	STRESS psi	1	2	3	4
2900	3.50	2.30	10.4	6.40	2400	1.39	1.40	1.65	1.49
2800	17.60	12.10	9.20	11.69	2200	4.40	3.61	2.05	3.25
2685	---	17.89	22.20	21.59	2000	12.45	16.65	15.04	15.24
2450	72.30	41.56	83.75	63.00	1700	52.06	58.09	50.10	67.54

[+] Indicates that the test was terminated without wire rupture at this time.

TABLE D-3 POROLOY STRESS RUPTURE TEST AT 1800°F (982°C) IN AIR

ALLOY	LOAD psi	TIME HRS.	% ELONG.	% R.A.
DH 245	500-6800	777.2*		
	7500	8 mins.	34.5	21.9
	6500	193.9	106.9	31.5
	7000	8 mins.	21.2	15.1
	6750	6 mins.	17.5	14.0
	6500	21 mins.	28.3	19.4
	3500	160.8	61.9	18.1
	5500	7 mins.	23.2	18.3
	4500	30 mins.	25.3	20.5
H 875	2200	0.3	59.2	68.6
	2000	0.6	45.3	54.8
	1900	1.4	53.7	52.0
	1500	45.0	31.5	37.0
	1660	6.9	35.7	45.8
	1460	42.7	31.5	49.0
	1300	163.3*	22.2	20.0
	1375	108.7	25.2	30.0
	1575	19.5	25.0	37.8
GE 1541	1900	164.6*	5.9	3.6
	2200	136.0*	10.2	8.6
	2600	7 mins.	34.5	42.4
	2500	0.7	56.9	44.4
	2400	0.3	51.9	46.4
	2200	0.5	37.2	40.0
	1900	4.9	17.4	28.3
	1700	46.3	16.8	18.1
	1700	13.3	20.6	31.1
	1700	5.1	17.5	29.4

*Test Discontinued.

TABLE D-4 0.005 (0.13 MM) DIAMETER WIRE TENSILE STRENGTH AT 1800°F (982°C)

MATERIAL	CONDITION	ULTIMATE psi	YIELD 0.2% psi	ELONGATION % IN 1 in.
DH 245	Simulated Sintered	9100 5000 8900 8100	7700 -- 7000 6900	10.5 0.4 7.6 5.8
H 875	Simulated Sintered	1900 4000 4700 1800	1800 3200 4400 1800	10.8 20.1 8.7 12.1
GE 1541	Simulated Sintered	2900 3400 3900 3300	1800 3000 2200 2600	13.5 12.7 14.3 14.6
H 875	Simulated Sintered & Oxidized 24 hrs. at 1800°F	3600 1040 1900 5500	2800 -- 1800 3500	5.1 3.0 4.1 5.7
GE 1541	Simulated Sintered & Oxidized 24 hrs. at 1800°F	5600 3800 3600 3700	4400 2700 2800 2500	8.1 3.3 5.1 7.0

TABLE D-5 POROLOY TENSILE STRENGTH AT 1800°F (982°C)

MATERIAL	CONDITION	ULTIMATE psi	YIELD 0.2% psi	ELONGATION % IN 1 in.
DH 245	As Fabricated	7106 6805 7601	5639 5346 6053	13.7 28.4 20.5
H 875	As Fabricated	2489 2667 2655	1304 2429 2406	72.0 58.8 100.9
GE 1541	As Fabricated	2410 2570 2810	1610 1940 2230	57.5 48.2 43.4
DH 245	Oxidized 24 hrs. at 1800°F	7816 8115 8814	7451 7594 8054	8.3 6.9 11.1
H 875	Oxidized 24 hrs. at 1800°F	2755 2814 2993	2683 2524 2614	50.0 68.5 55.5
GE 1541	Oxidized 24 hrs. at 1800°F	2780 2910 2740	2710 2850 2380	53.1 45.5 47.3

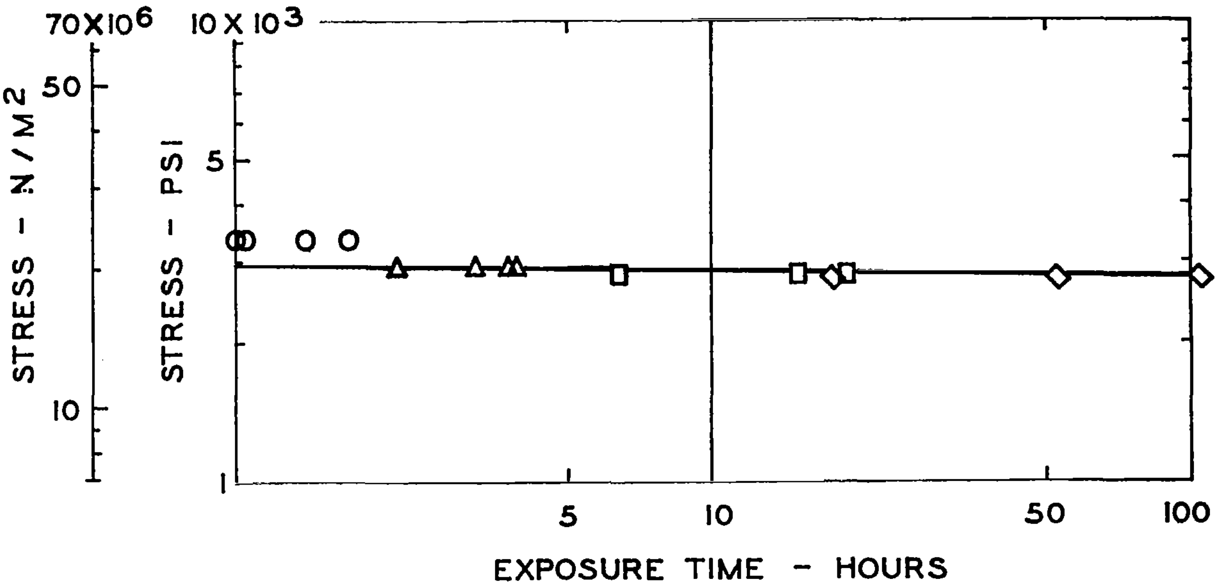


Figure D-1 Stress Rupture Life for GE 1541 0.005 Dia. Wire at 1800°F (Each data point symbol represents a different stress level).

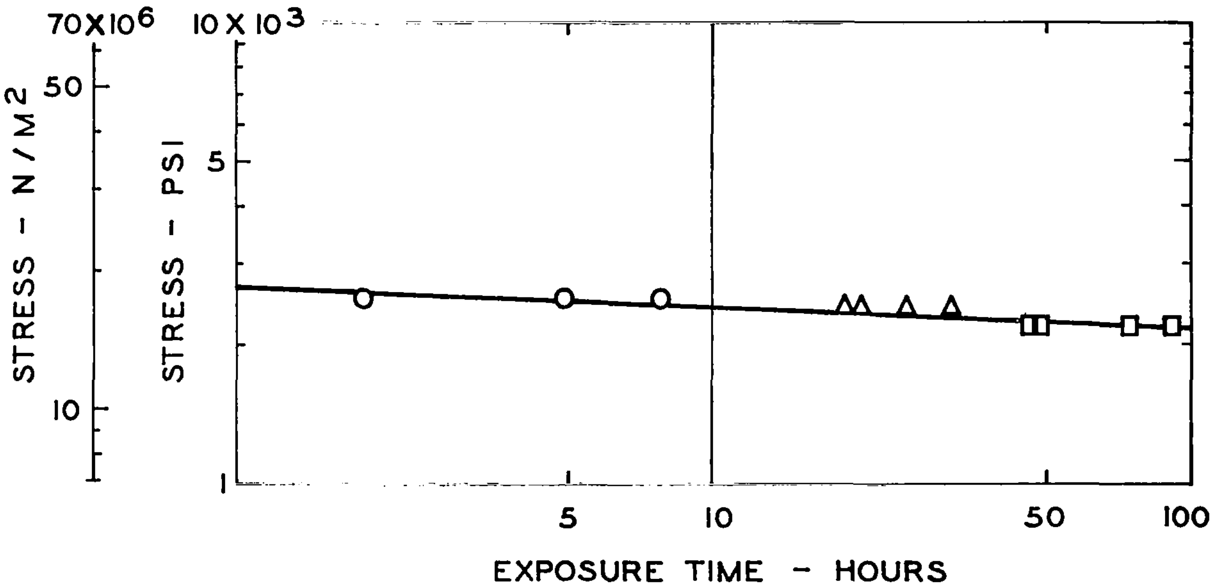


Figure D-2 Stress Rupture Life for GE 1541 0.005 Dia. Wire at 2000°F (Each data point symbol represents a different stress level).

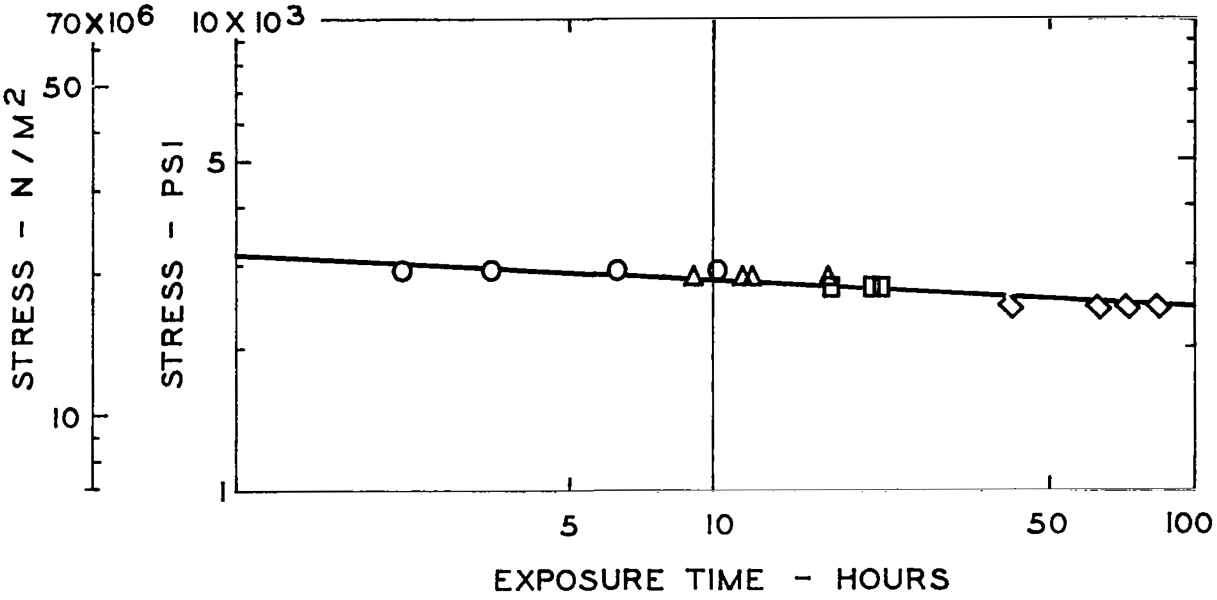


Figure D-3 Stress Rupture Life for H 875 0.005 Dia. Wire at 1800°F (Each data point symbol represents a different stress level).

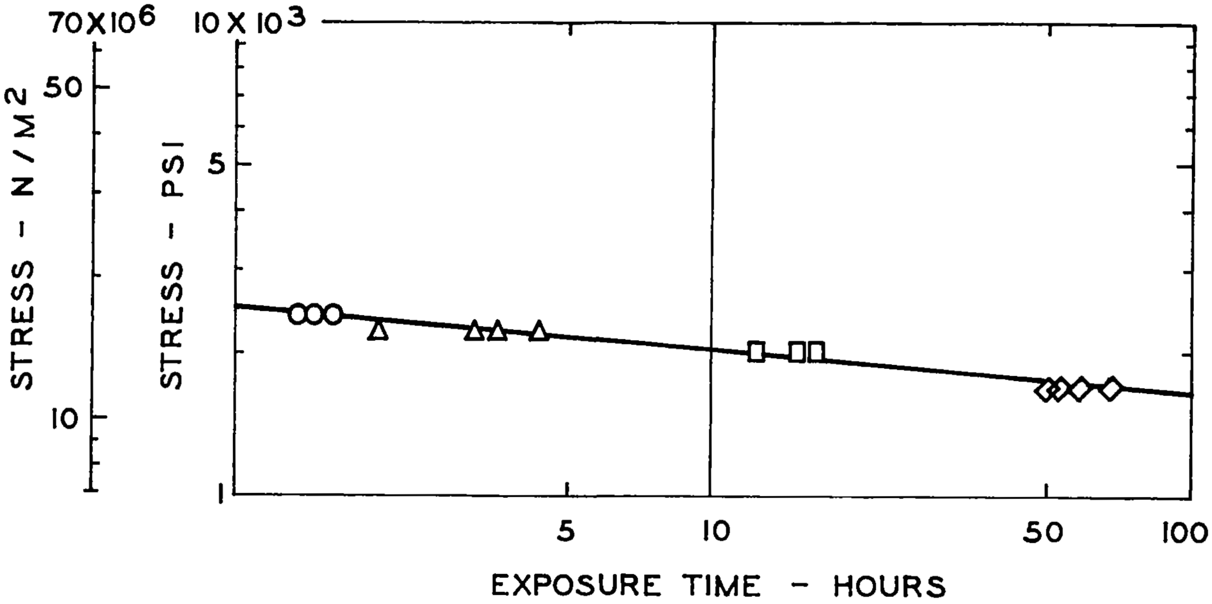


Figure D-4 Stress Rupture Life for H 875 0.005 Dia. Wire at 2000°F (Each data point symbol represents a different stress level).

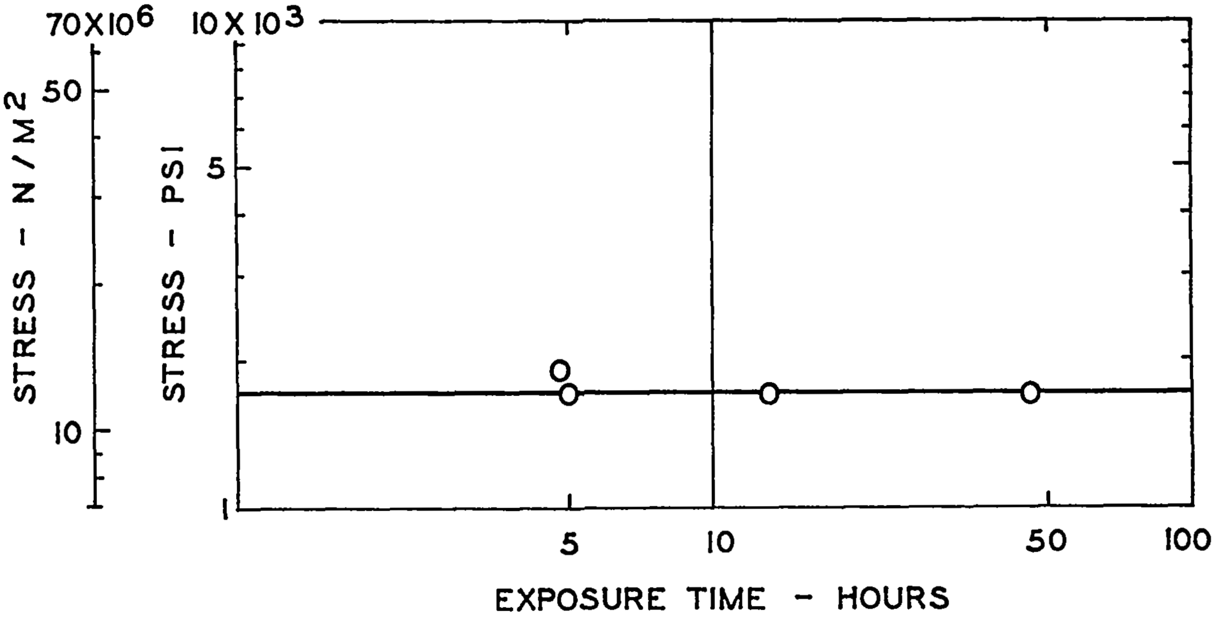


Figure D-5 Stress Rupture Life for GE 1541 Poroloy at 1800°F.

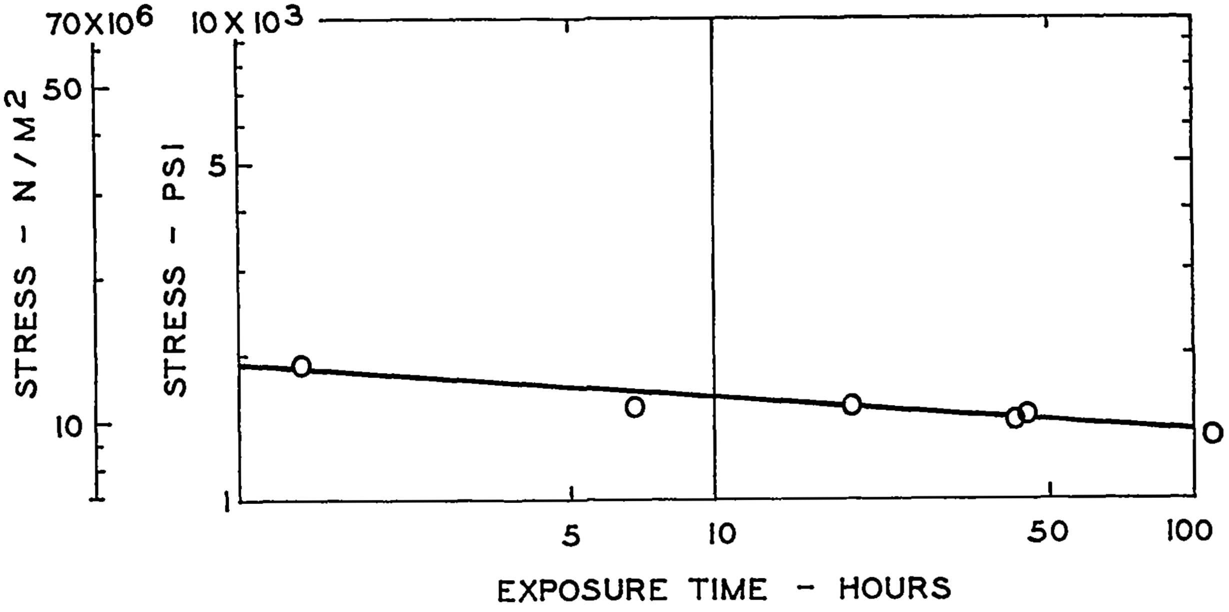


Figure D-6 Stress Rupture Life for H 875 Poroloy at 1800°F.

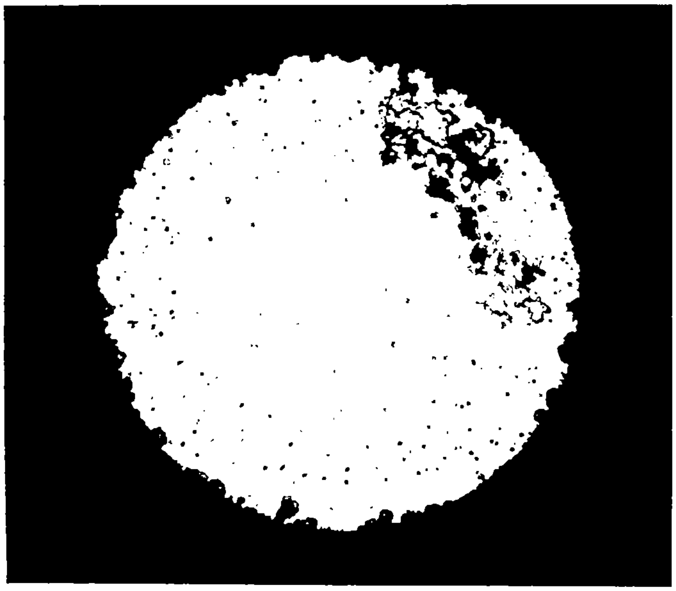
Stress Oxidation of TD Ni-Cr

Earlier work (reference 2) had shown that, although TD Ni-Cr had very good oxidation resistance under no load conditions, when a critical stress was applied, the alloy suffered extensive internal oxidation. Therefore, a portion of this investigation was directed toward determining the cause of this behavior. It was discovered that the internal oxidation was not caused by stress acceleration of the oxidation mechanism per se, but rather by structural degradation of the alloy. That is, at the critical stress level, extensive void and crack formation was initiated in the alloy with concurrent oxidation. Photomicrographs illustrating this behavior are shown in Figures D-7 and D-8.

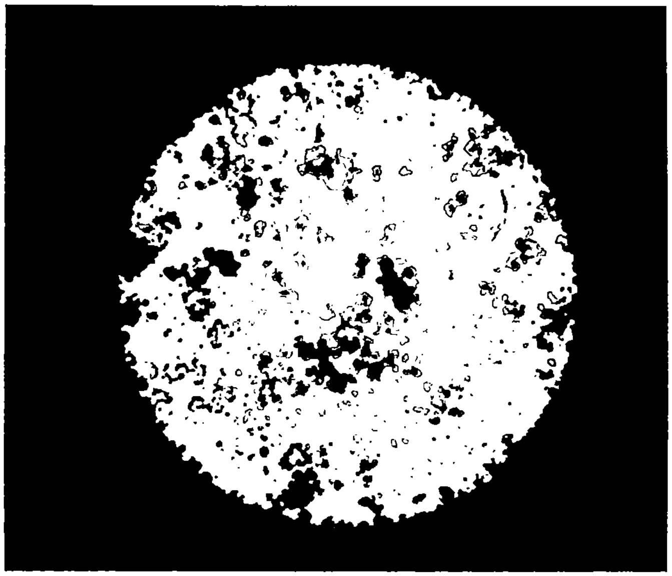
The formation of voids was attributed to the presence of a significant concentration of relatively large ThO_2 particles (Figure D-9). The average size of these large particles was about 5800A. This size particle makes vacancy condensation around the particle energetically favorable at the stress levels of this investigation. Consequently the thoria particles served as the nuclei for void formations throughout the sample. Subsequent growth of these voids provided internal surfaces for the observed internal oxidation. The critical stress levels (determined after 22 hours application of the load) as a function of temperature are

Temperature (°F)	Critical Stress (PSI)
1400	4800
1600	3900
1700	3500
1800	3200
2000	2800

The results of this work indicate that the stress sensitivity of internal oxidation could be eliminated in TD Ni-Cr wire. This would require inspection of the starting material to confirm the absence of large thoria particles. The commercial specification for TD Ni-Cr generally dictates ThO_2 particle sizes less than 1000A which would be sufficiently small to prevent void formation at stress levels up to at least 10,000 psi. In addition, wire processing should be designed to exclude steps which would tend to agglomerate the ThO_2 particles.

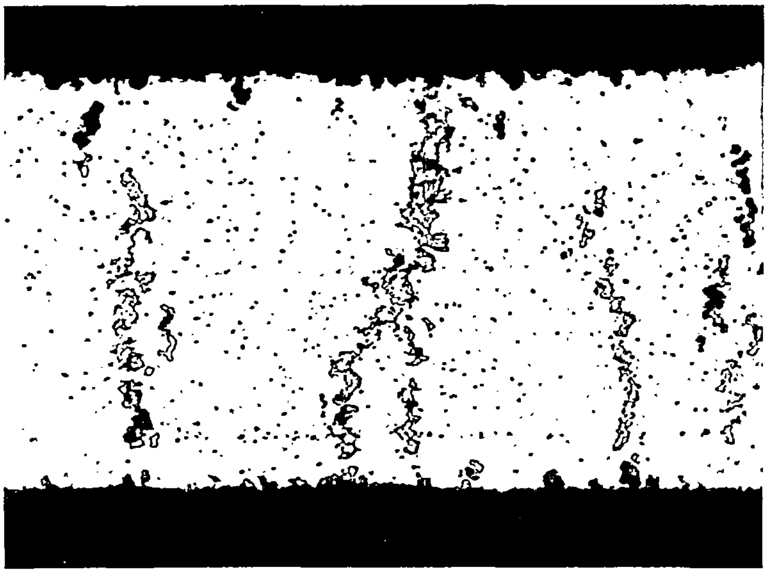


3200 PSI

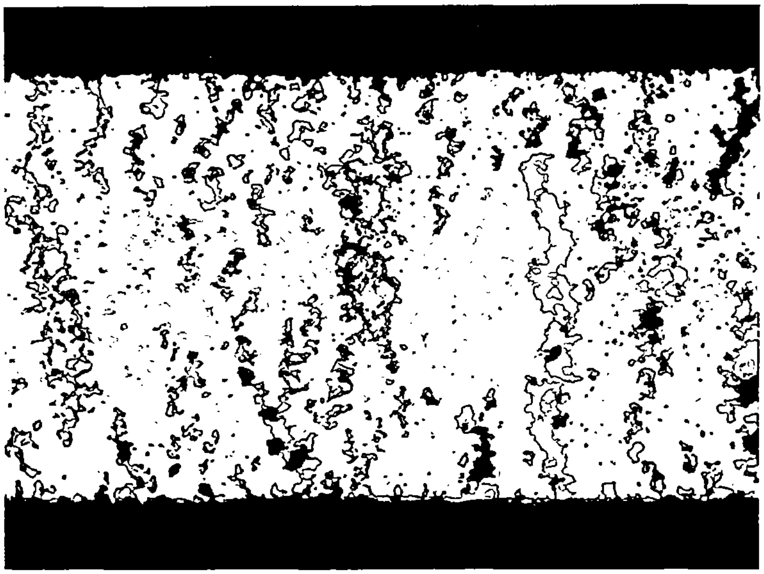


3600 PSI

Figure D-7 Cross sections of TD Ni-Cr wires (0.005 inch diameter) showing voids and internal oxidation after 1800°F exposure at the indicated stress level.



3200 PSI



3600 PSI

Figure D-8 Longitudinal sections of TD Ni-CR wires (0.005 inch diameter) showing voids and internal oxidation after 1800°F exposure at the indicated stress level.

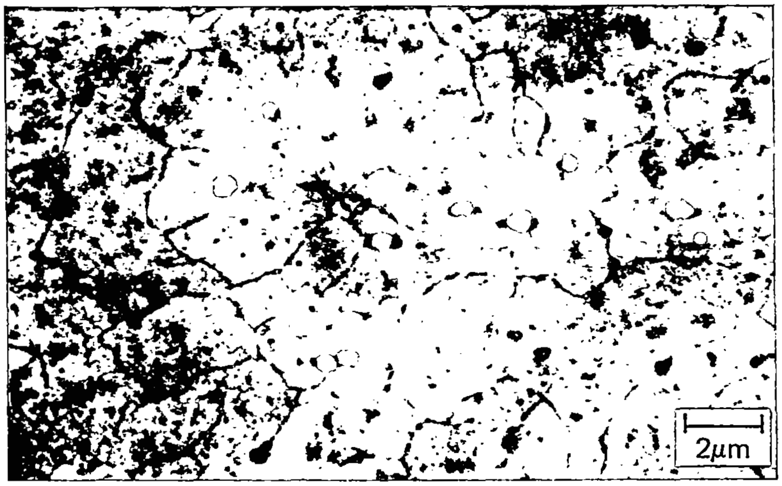
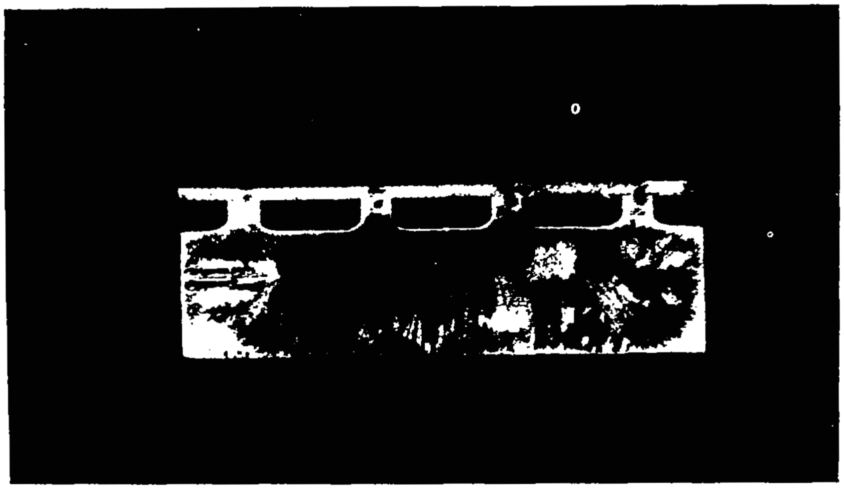


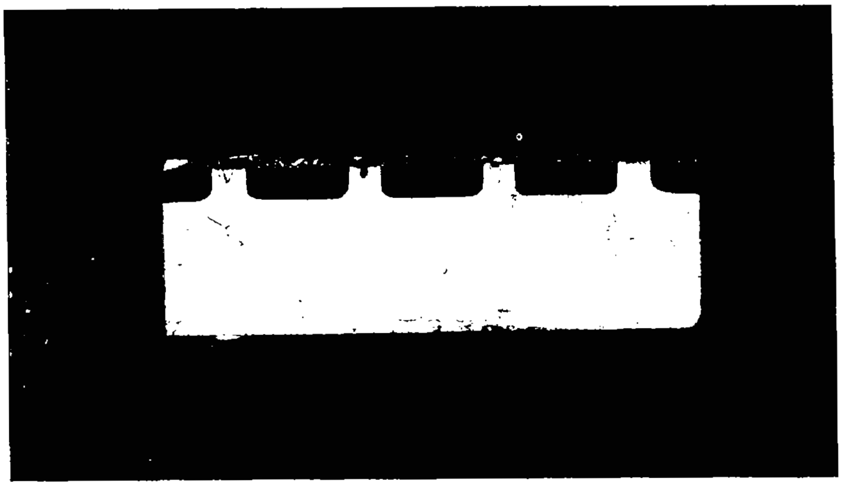
Figure D-9 Scanning electron micrograph of as-fabricated TD Ni-Cr wire (0.005 inch diameter) illustrating presence of large thoria particles.

APPENDIX E

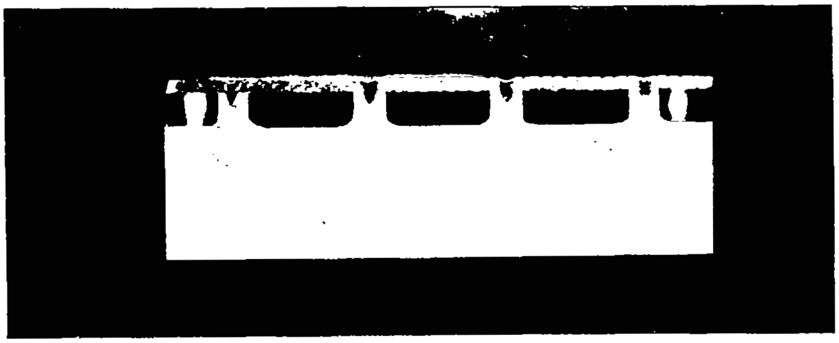
WELDING TESTS



GE 1541/IN 100



H 875/IN 100



TD Ni Cr/713C

Figure E-1 Cross sections of representative weld samples. Sample is etched to reveal weld penetration. X3

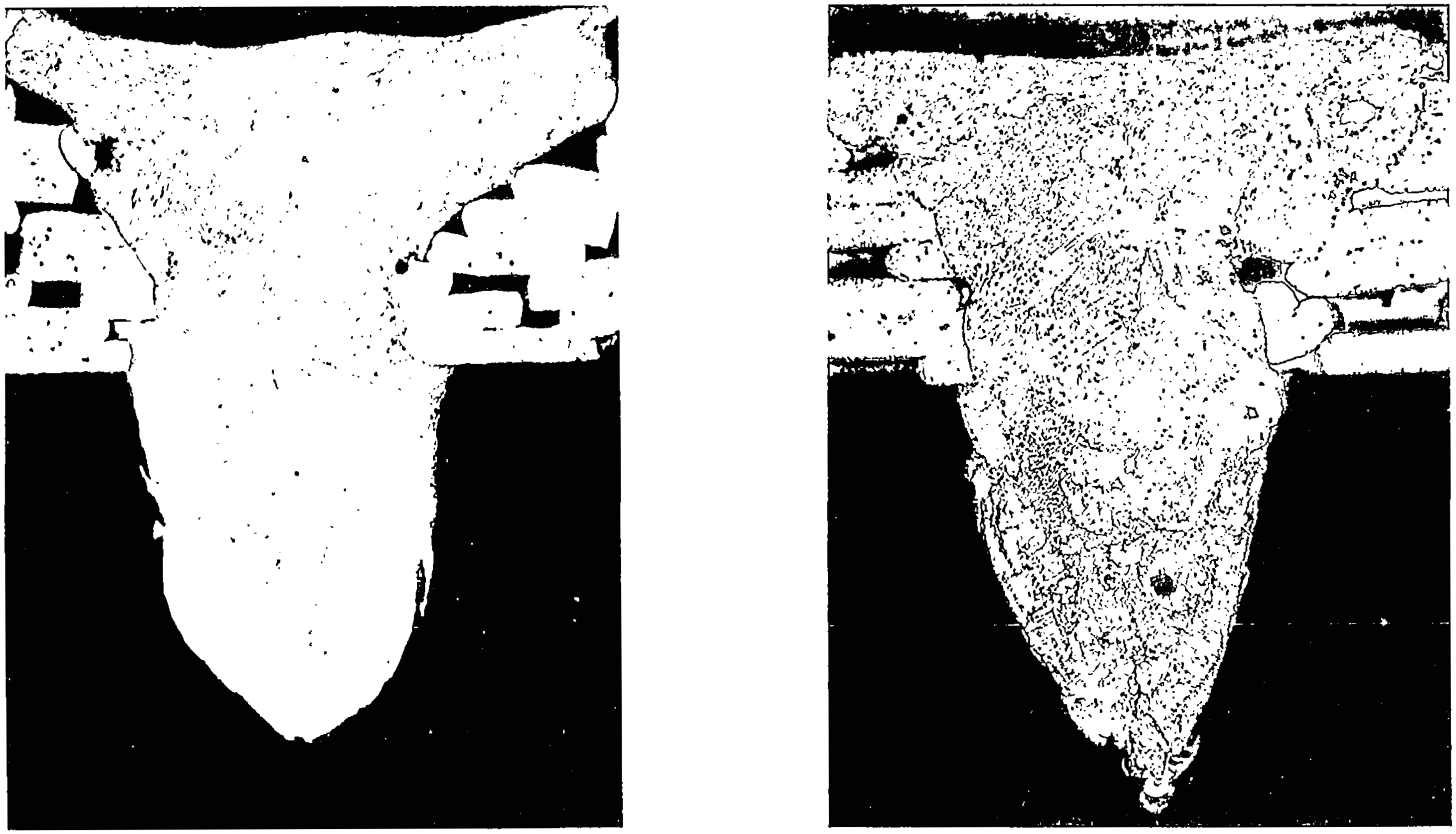


Figure E-2 Cross sections of GE 1541/IN 100 (right) and GE 1541/713C (left) welds etched to reveal microstructure. Strut alloy matrix appears black on photographs. X80

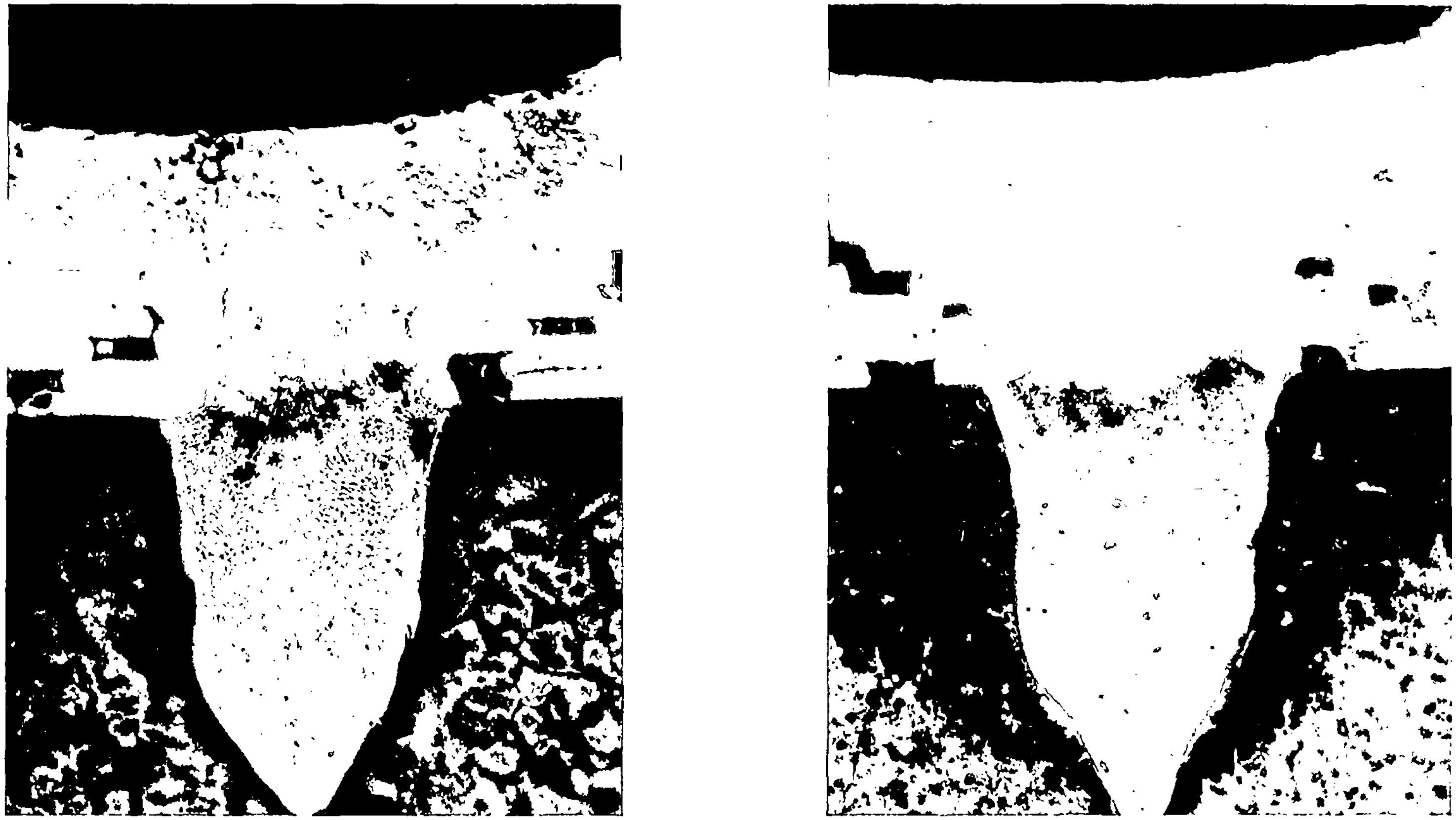


Figure E-3 Cross sections of H 875/IN 100 (right) and H 875/713C (left) welds etched to reveal microstructure. Strut alloy has etched black around molten zone tip. X80

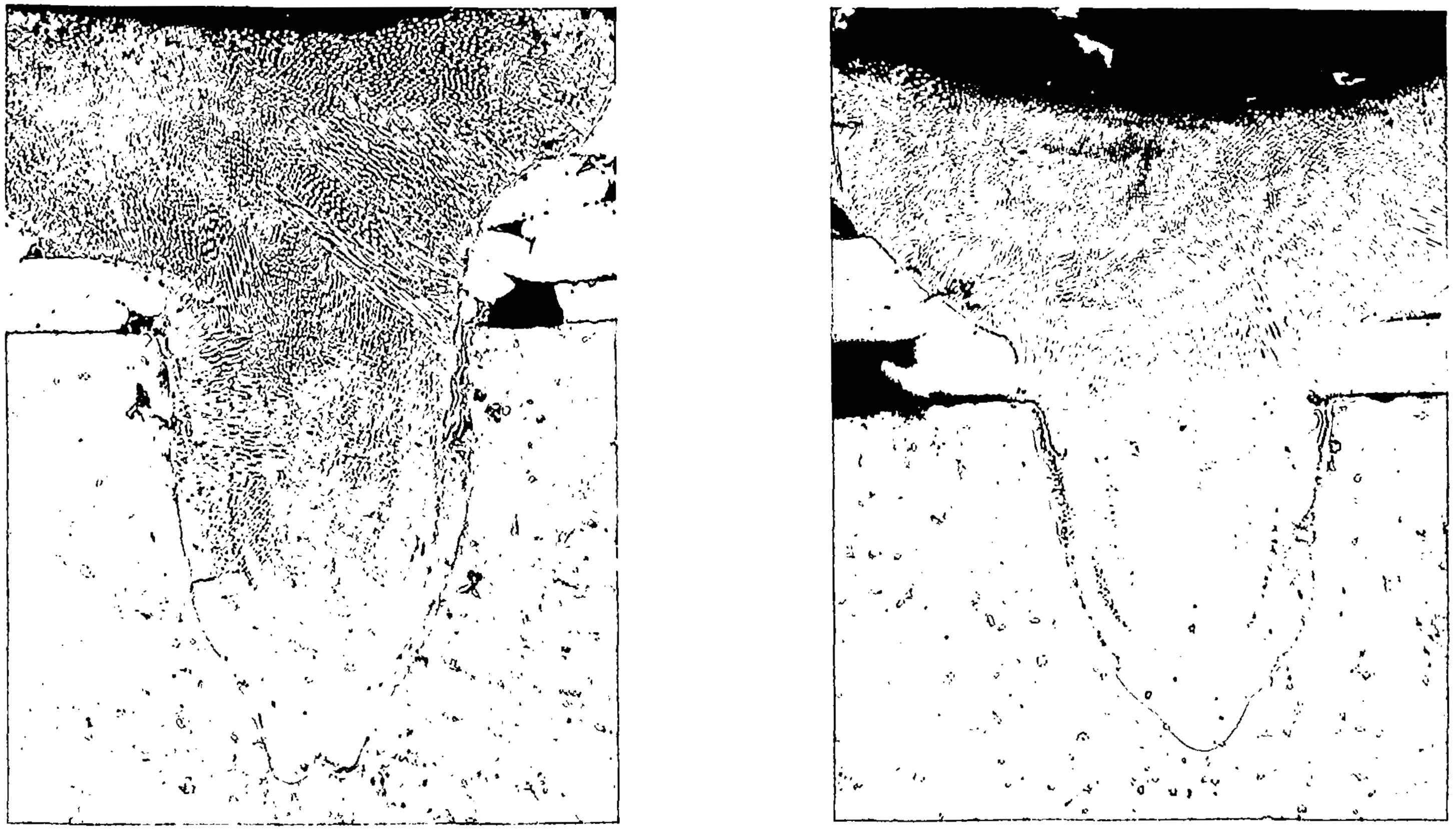


Figure E-4 Cross sections of DH 242/IN 100 (right) and DH 242/713C (left) welds etched to reveal microstructure. X80

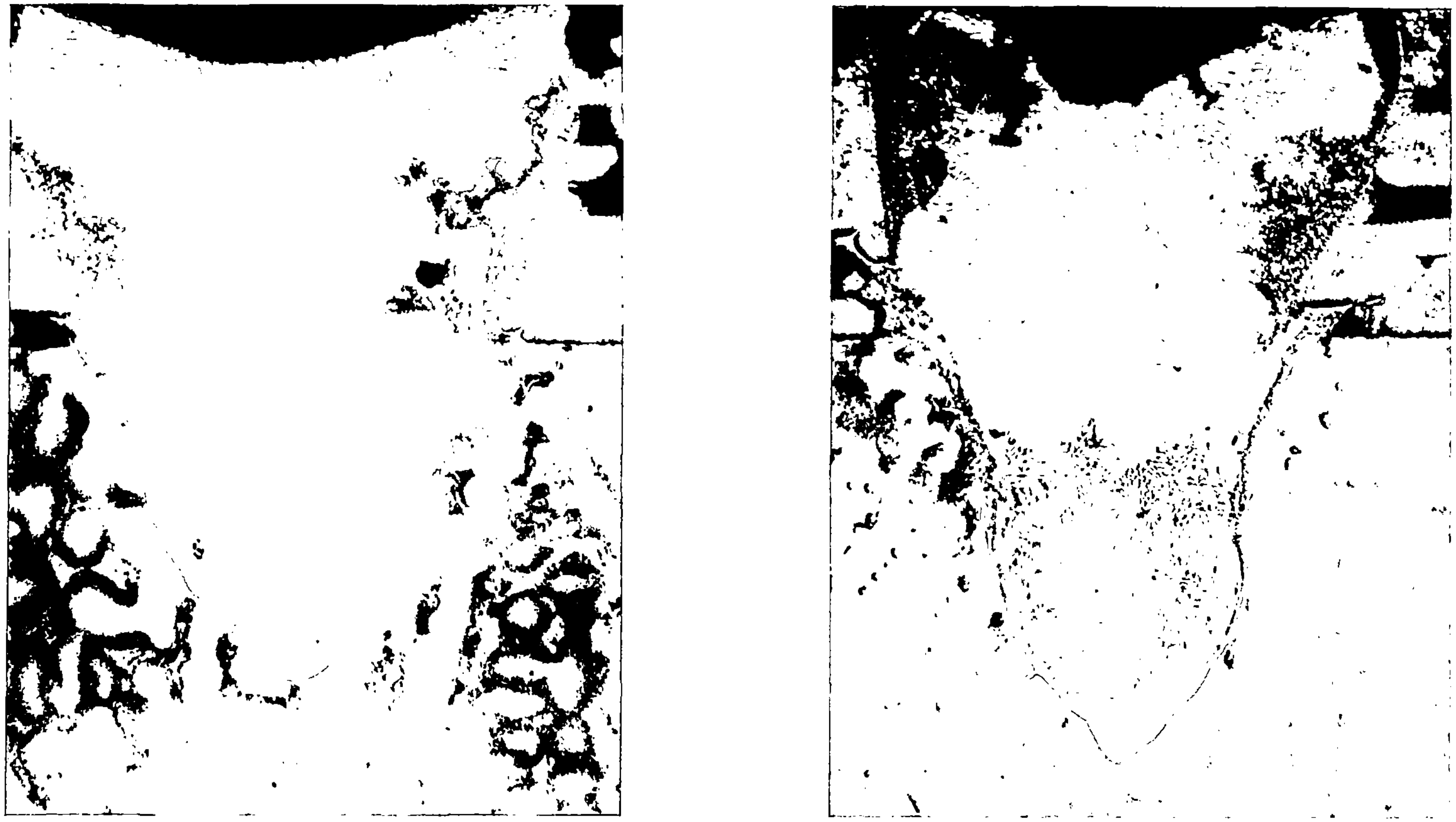


Figure E-5 Cross sections of TD Ni-Cr/IN 100 (right) and TD Ni-Cr/713C (left) weld etched to reveal microstructure. X80

APPENDIX F

STATIC OXIDATION TESTS

TABLE F-1 OXIDATION WEIGHT GAIN OF 0.005 (0.13 MM) DIAMETER WIRE EXPOSED AT 1800°F (982°C) FOR 24 HOURS IN AIR

MATERIAL	LENGTH in.	DIAMETER in.	AREA in^2	SAMPLE WEIGHT			SPECIFIC WEIGHT GAIN mg/in^2
				BEFORE mg	AFTER mg	WEIGHT GAIN mg	
DH 245	504	0.0049	7.77	1324.2	1353.1	28.9	3.72
DH 245	480	0.0049	7.40	1267.6	1295.9	28.3	3.83
H 875	480	0.0050	7.55	1159.2	1166.5	6.7	0.89
H 875	480	0.0050	7.55	1154.9	1161.9	7.0	0.93
GE 1541	480	0.0048	7.10	1094.4	1103.0	8.6	1.21
GE 1541	480	0.0048	7.10	1097.2	1107.3	10.1	1.42

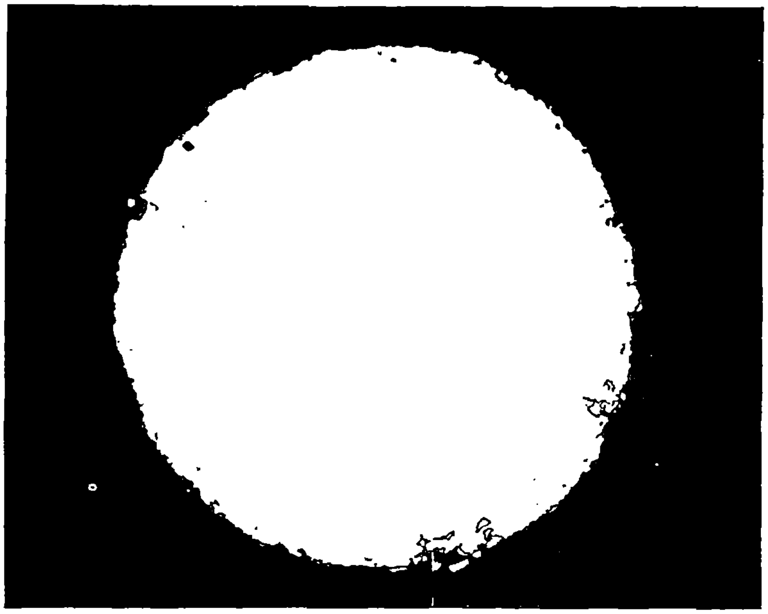


Figure F-1 Cross section of GE 1541 wire (0.005 inch dia.) air oxidized for 24 hours at 1800°F. X500

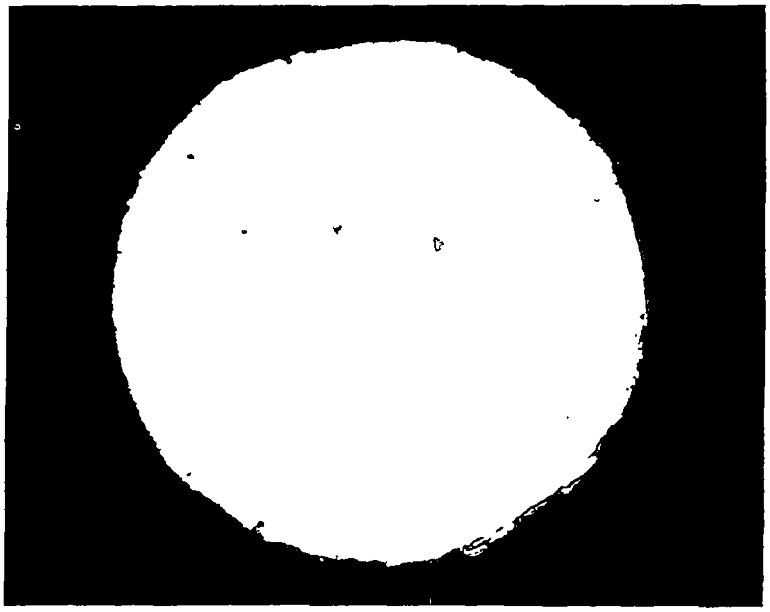


Figure F-2 Cross section of H 875 wire (0.005 inch dia.) air oxidized for 24 hours at 1800°F. X500

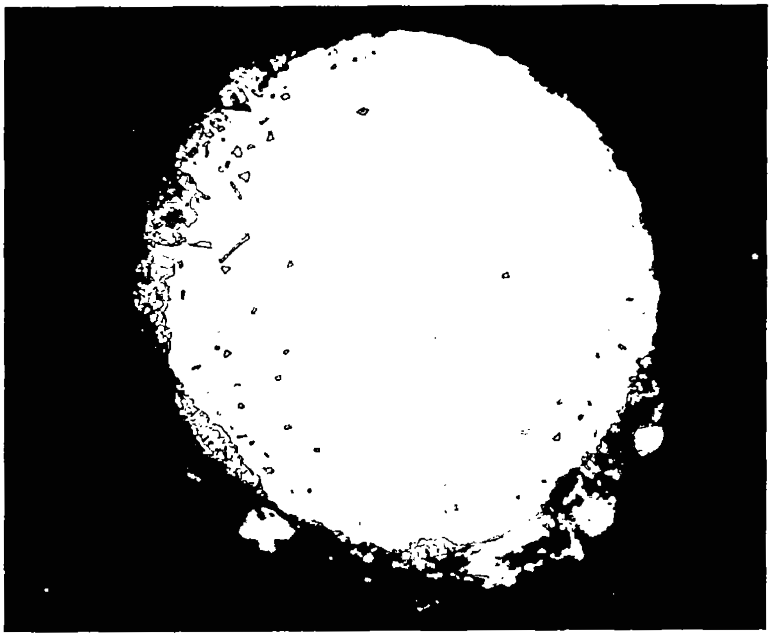


Figure F-3 Cross section of DH 245 wire (0.005 inch dia.) air oxidized for 24 hours at 1800°F. X500

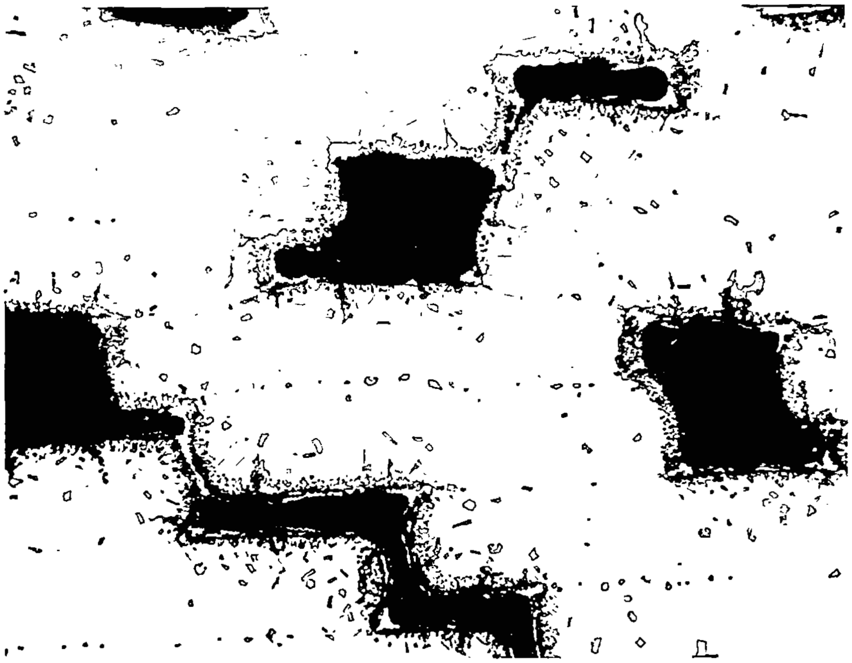


Figure F-4 Cross section of DH 245 Poroloy air oxidized for 24 hours at 1800°F. X250

REFERENCES

1. Cole, Fred W.; Padden, James B.; and Spencer, Andrew R.; Oxidation Resistant Materials for Transpiration Cooled Gas Turbine Blades. Part I Sheet Specimen Screening Tests NASA CR-930, 1968.

2. Cole, Fred W.; Padden, James B.; and Spencer, Andrew R.; Oxidation Resistant Materials for Transpiration Cooled Gas Turbine Blades. Part II Wire Specimen Tests. NASA CR-1184, 1968.

3. Esgar, Jack B.; Colladay, Raymond S.; and Kaufman, Albert.; An Analysis of the Capabilities and Limitations of Turbine Air Cooling Methods. NASA TN D-5992, 1970.

4. Eckert, E. R. G.; and Livingood, John, N. B.; Comparison of Effectiveness of Convection -, Transpiration -, and Film - Cooling Methods with Air as Coolant. NACATN 3010, 1953.

5. Kaufman, Albert; Steady State Stress Relaxation Analysis of Turbine Blade Cooling Designs. NASA TN D-5282, 1969.

6. Wheeler, Jr. H. L.; Transpiration Cooling in the High Temperature Gas Turbine. Bendix Corporation, Filter Division, 1964.

7. Moskowitz, S. L. and Schober, T. E.; Design and Test of a Small Turbine at 2500°F with Transpiration Cooled Blading, SAE Report 690035, 1969.

8. Grisaffe, Salvatore J.; and Lowell, Carl E.; Examination of Oxide Scales on Heat Resistant Alloys. NASA TN D-5019, 1969.

9. Perkins, R. J. and Wukusick, C. S.; Long Time Oxidation Resistance of Iron - Chromium-Aluminum-Yttrium Alloys at Temperatures of 450 to 1100°C, GE T M 67-2-3, 1966.

10. Stephan, H. R.; Accelerated Life Tests of an Iron-Chromium-Aluminum-Yttrium Alloy for Electrical Heating Elements, GE T M 66-6-1, 1966.

11. Kaufman, Albert; Analytical Study of Flow Reduction due to Oxidation of Wire-Form Porous Sheet for Transpiration Cooled Blades. NASA TN D-50001, 1969.