

# OXIDATION RESISTANT MATERIALS FOR TRANSPIRATION COOLED GAS TURBINE BLADES

II. Wire Specimen Tests

by Fred W. Cole, James B. Padden, and Andrew R. Spencer

Prepared by BENDIX CORPORATION Madison Heights, Mich. for Lewis Research Center

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION . WASHINGTON, D. C. . SEPTEMBER 1968

## OXIDATION RESISTANT MATERIALS

## FOR TRANSPIRATION COOLED

## GAS TURBINE BLADES

## II. Wire Specimen Tests

## By Fred W. Cole, James B. Padden, and Andrew R. Spencer

Distribution of this report is provided in the interest of information exchange. Responsibility for the contents resides in the author or organization that prepared it.

## Prepared under Contract No. NAS 3-7269 by BENDIX CORPORATION Madison Heights, Mich.

## for Lewis Research Center

## NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

For sale by the Clearinghouse for Federal Scientific and Technical Information Springfield, Virginia 22151 - CFSTI price \$3.00

## PRECEDING PAGE BLANK NOT FILMED,

0

١.

## FOREWORD

The research described herein was conducted by the Bendix Corporation Filter Division under NASA contract NAS3-7269. Mr. Albert E. Anglin and Mr. Jack B. Esgar, both of the Airbreathing Engines Division, Lewis Research Center, were the Technical Manager and Research Advisor, respectively.

3

#### ABSTRACT

Four alloys were selected on the basis of sheet specimen tests, reported in Summary Report CR-930, and tested in 0.005 inch diameter wire form to determine their suitability for use as transpiration cooled gas turbine blade materials. The alloys were N 155, TD nickel-chromium, DH 242, and Hastelloy X. Tests consisted of cyclic oxidation, continuous oxidation, stress rupture life, and stress-oxidation at 1400, 1600, 1800, and 2000°F, and up to 2100 and 2200°F for some tests. Exposure times ranged up to 100 hours except for cyclic oxidation tests where times were 4, 16, 64, 100, 100, 300, 400, 500 and 600 hours. Total specific oxidation weight gain, oxide spall and penetration, and mechanical properties were determined for each alloy. Metallographic examination showed microstructure and oxidation effects. TD nickelchromium was superior, except for stress sensitivity resulting in internal oxidation. All alloys were limited to a useful service temperature near 1600-1700°F.

## SECTION

t,

V

$\mathbf{P}l$	\C	F.
. <b>т</b> . <b>с</b>	10	Ľ

_	1
1	
2	
3	wire Procurement,
4	
4.1	Specimen Preparation
4.2	Cyclic Oxidation Tests
4.3	Specimen Examination and Testing 9
4.3.1	Oxidation and Spall $\ldots$ 9
4.3.2	Thickness and Surface Examination 9
4.3.3	Metallographic Examination
4.3.4	Tensile Tests
4.4	Thermogravimetric Analyses
4.5	Stress Rupture Tests
4.6	Stress Oxidation Tests
5	Results and Discussion
5.1	Cyclic Oxidation and Spall
5.2	Thickness and Surface Examination
5.3	Metallographic Examination
5.4	Tensile Tests
5.5	Thermogravimetric Analysis
5.6	Stress Rupture
5.7	Stress Oxidation
6	Conclusions
÷	
TABLE	
3-1	Allov Analysis
<u> </u>	Metallographic Etchant Schedule
42	Mochanical Properties of Wire Specimens
4-2	Mechanical Hopercies of while opeciments
FTOUDE	
FIGURE	
4-1	Specimen and Equipment Schematic
4-2	Thermogravimetric Analysis Apparatus
5.1-1	Cyclic Oxidation Resistance Comparison, 1400°F 28
5.1-2	Cyclic Oxidation Resistance Comparison, 1600°F 29
5.1-3	Cyclic Oxidation Resistance Comparison, 1800°F 30
5.1-4	Cyclic Oxidation Resistance Comparison, 2000°F 31
5.1-5	Cyclic Oxidation Resistance Comparison, 2100°F
5.1-6	Cyclic Oxidation Resistance Comparison, 2200°F 33
5.1-7	Cyclic Spall Resistance Comparison, 1600°F
5.4-1	Mechanical Properties Comparison, 1400°F
5.4-2	Mechanical Properties Comparison, 1600°F
5.4-3	Mechanical Properties Comparison, 1800°F
5.4-4	Mechanical Properties Comparison, 2000°F
6-1	Extrapolated Oxidation Resistance Comparison
6-2	Extrapolated Penetration Resistance Comparison
1	JU

## APPENDIX

1	Numerical Data Tabulation .				•	٠	۰	٠	ø	٠	ø		.0		٠	٠	39
2	Alloy Oxidation Plots	•	•	9		•		•		•				•	٠	•	57
3	Metallographic Examination.		•	.0	•	ø	•	•			÷	•		.0	.0		71
4	Mechanical Properties		•		•	ø		•				.0	•		٠		101
5	Oxidation Penetration Plots	•		٠	•			٠		•		•	•	٠		٠	109

## NOTE

Tables and figures contained in the Appendixes are separately listed on the title page of the corresponding Appendix.

## OXIDATION RESISTANT MATERIALS FOR TRANSPIRATION

## COOLED GAS TURBINE BLADES

## **II. WIRE SPECIMEN TESTS**

by Fred W. Cole<sup>1</sup>, James B. Padden<sup>2</sup>, and Andrew R. Spencer<sup>3</sup>

THE BENDIX CORPORATION Filter Division

#### 1 SUMMARY

Four alloys were selected on the basis of previous sheet specimen screening tests and tested as 0.005-inch diameter wire specimens to determine their suitability for utilization in transpiration cooling materials such as Poroloy and Rigimesh or Poroplate. The alloys chosen from twelve sheet specimen alloys reported in the Part One Summary Report CR-930 were:

1.	N 155				•	٠	21Cr-20Ni-20Co-3Mo-2.5W-Fe
3.	TD nickel-chromium	٠	•	.0	•		2Th02-20Cr-Ni
6.	DH 242	•	•	•	•		20Cr-1Cb-Ni
10.	Hastelloy X		•		ė		22Cr-18.5Fe-9Mo-1.5Co-Ni

This selection was based on considerations of cyclic oxidation resistance and retention of good mechanical properties. Alloy N 155 was chosen as a base line for alloy comparison. Further tests were conducted on these four alloys in 0.005-inch diameter wire form.

All wire specimens were "sintered" in dry hydrogen for two fourhour cycles at 2100°F before testing to simulate fabrication practice. Cyclic oxidation tests were made on wire bundles, individually held in covered zircon ceramic "test tubes," as in the sheet specimen tests, with a separate specimen for each alloy-temperature-time combination. Test temperatures of 1400, 1600, 1800, 2000, and 2200°F and exposure times of 4, 16, 64, 100, 200, 300, 400, 500, and 600 hours were employed. Specimens were cooled to room temperature after each time cycle to simulate the cyclic, rather than steady-state, heat exposure expected in hot gas turbine service. Additional thermogravimetric analyses were conducted at 1400, 1600, 1800, 2000, 2100 and 2200°F to determine continuous weight gain as a function of time to 100 hours. Stress rupture lives to 100 hours were determined at 1400, 1600, 1800, and 2000°F and stress-oxidation tests of 100 hours were conducted at 60 percent of

<sup>&</sup>lt;sup>1</sup>Chief Project Engineer, Filter Division

<sup>&</sup>lt;sup>2</sup>Project Engineer, Filter Division

<sup>&</sup>lt;sup>3</sup>Staff Metallurgist, Research Laboratories

stress-to-rupture. Specimens were measured to determine specific weight gain, oxide spalling, and depth of oxide penetration, and samples were tensile tested at room temperature to determine changes in ultimate strength, yield strength, and elongation. Metallographic examination of wire sections was used to show significant changes due to oxidation and heating.

TD nickel-chromium proved to be superior in no-load oxidation tests, but stress-rupture and stress-oxidation testing showed a serious stress sensitivity which resulted in internal oxidation at temperatures above 1600°F. DH 242 and Hastelloy X were comparable in performance for this application. DH 242 is slightly more oxidation and spall resistant but much weaker than Hastelloy X in their useful temperature range below about 1600-1700°F. Cyclic oxidation proved to be generally more severe than other oxidation exposure. Wire specimens were more severely oxidized than comparable sheet specimens.

#### 2 INTRODUCTION

This experimental investigation was conducted to evaluate and select metal alloys which are particularly suitable for fabricating transpiration cooling materials used in high temperature gas turbine engines. Major emphasis was placed on measuring oxidation resistance and retention of mechanical properties of the selected alloys after cyclic furnace heating in air. Thermogravimetric analyses, stress rupture and stress oxidation tests were also performed. This report describes results and conclusions derived from testing 0.005-inch diameter wire specimens. A previous Summary Report CR-930\* describes a sheet specimen screening test program which provided the basis for alloy selection in the present investigation.

Transpiration cooling methods offer excellent potential for further increasing the operating temperature of hot gas turbines while minimizing cooling air consumption. Porous transpiration cooling material 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 strength and cooling air flow permeability. 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 necessary, but these characteristics are largely determined by metal oxidation resistance in porous materials.

Transpiration cooling materials for turbine blades, shroud liners, and similar components are typically fabricated from fine wire about 0.005 inch in diameter. The wire is space-wound in a geometric pattern (Poroloy\*\*) or woven into mesh and laminated (Rigimesh\*\*, Poroplate\*\*), and finally diffusion bonded to provide a porous structure. The properties of fine wires and fine wire structures are not easily extrapolated from data generated by testing bulk alloys. Oxidation resistance and mechanical properties may be substantially affected by the alloy's previous working history and grain structure. Differences in diffusion geometry may cause quantitative differences in oxidation resistance between wire and sheet or bar specimens. Therefore, these tests are de-

<sup>\*</sup>Investigation of Oxidation Resistant Materials for Transpiration Cooled Gas Turbine Blades. Part I - Sheet Specimen Screening Tests by F. W. Cole, J. B. Padden, and A. R Spencer, The Bendix Corporation, Filter Division. NACA CR-930, 1967.

<sup>\*\*</sup>Poroloy and Poroplate are registered trade-names describing spacewound wire and laminated woven wire mesh transpiration cooling materials, respectively, manufactured by The Bendix Corporation, Filter Division. Rigimesh is a registered trade-name describing laminated woven wire mesh materials manufactured by the Pall Corporation, Aircraft Porous Media Division.

signed to compare the selected metal alloys in fine wire form for application to transpiration cooling materials engineering. Much of the test data, however, should also be useful for other engineering applications.

All wires tested were heat treated in dry hydrogen at 2100°F to simulate the typical manufacturing diffusion bonding or sintering operation. Oxidation cycling tests were conducted at 1400, 1600, 1800, 2000, 2100 and 2200°F for exposure times of 4, 16, 64, 100, 200, 300, 400, 500 and 600 hours. Temperatures and times, as well as general test procedures, were the same as those used in the previous screening tests on sheet metal specimens, and represent the range of interest for application to high temperature gas turbines. Each specimen was tested as a bundle of wires held in a separate, covered zircon ceramic thimble or "test tube" which was designed to allow convective air circulation and catch oxide spall. After oxidation exposure, each wire specimen was examined and tested to determine total oxidation weight gain and spalling, depth of oxide penetration by metallographic section, and mechanical properties of yield strength, ultimate strength and percentage elongation. Separate wire specimens were tested for continuous oxidation weight gain by thermogravimetric analysis, stress rupture to 100 hours, and stress oxidation in the temperature range of 1400 through 2000°F.

This Final Report (Part Two, Wire Specimen Tests) is organized in parallel to the earlier Summary Report CR-930 (Part One, Sheet Specimen Screening Tests) which was the basis for wire alloy selection and which supplements this report. Tables and figures which directly illustrate the text and compare alloy specimen performance are collected at the end of the text and are labeled according to the relevant section number of the text. Additional tables and figures are presented in appendixes. The appendixes are supplementary in character and present additional data, photomicrographs, and plotted curves to further illustrate test results and conclusions.

## 3 WIRE PROCUREMENT

Specification of the four alloys tested in wire form was based on the results of previous sheet specimen screening tests conducted with twelve alloys. These alloys, which represent a cross-section chosen from the superalloy and resistance-heating alloy family, are listed below.

1.	N 155	•	•		21Cr-20Ni-20Co-3Mo-2.5W-Fe
2.	TD nickel	•	•	•	2Th02-Ni
3.	TD nickel-chromium	•	•	•	2Th02-20Cr-Ni
4.	Bendel 65-35	•	•	•	3 Spine1-35Cr-Ni
5.	Chromel A	•	•	•	20Cr-Ni
6.	DH 242	•	•	٠	20Cr-1Cb-Ni
7.	GE 1541		•	•	15Cr-4A1-1Y-Fe
8.	Hoskins 875	•	•	•	23Cr-6A1-Fe
9.	RA 333	•	•		18Cr-3Mo-3W-3Co-Fe
LO.	Hastelloy X		•		22Cr-18.5Fe-9Mo-1.5Co-Ni
11.	Udimet 500	•		•	19Cr-19.5Co-4Mo-3Ti-3A1-Ni
L2.	Haynes 25	•	•	•	20Cr-15W-10Ni-Co

The alloy numbers one through twelve assigned in this list are referenced throughout the previous summary report and will also be used in this report for alloy identification. The four alloys selected from this group for further testing in wire form were (1) N 155, (3) TD nickel-chromium, (6) DH 242, and (10) Hastelloy X. Selection was based on considerations of oxidation resistance and retention of good mechanical properties after cyclic heating of the sheet specimens. Alloy N 155 was included as a "base-line" material for comparison purposes. Alloy GE 1541 was not tested in wire form because it was not recognized to be a superior alloy until after wire tests were well under way.

Alloy wires N 155, DH 242, and Hastelloy X were procured from Bendix Filter Division stock. These alloy wires are commonly used for transpiration cooling materials fabrication and vendor certified stocks are normally kept for production requirements. Alloy TD nickel-chromium was purchased from Hoskins Manufacturing Company who were first to draw 0.005-inch diameter wire from this material. After receipt and inspection of the alloy wires to verify their documentation, each alloy was analyzed to verify its composition and tensile tested to determine its mechanical properties. These test results are shown in Table 3-1 which compares specified or certified composition for each alloy wire with the composition determined by wet chemical analysis. Wire suppliers, mill certifications, and similar data are also given. Yield strength, ultimate strength, and percentage elongation for "as received" and "as sintered" wire specimens are compared in Table 4-2. All wire lots were accepted for further testing in this program.

Wire drawing procedures were routine for all alloys except TD nickel-chromium. Typical procedure for N 155, DH 242, and Hastelloy X wire consisted of successive draws from 1/4-inch rod to about 0.050 wire, using sulfur-free calcium stearate powder, followed by nitric-hydrochloric acid pickling and further drawing in chlorinated oil lubricant to final 0.005-inch diameter size. About 50-70 percent reduction in cross section area was allowed between annealing cycles which were accomplished at 2000-2100°F in a dissociated ammonia atmosphere. TD nickel-chromium alloy was hot swaged from 1/2 inch to 0.090 inch diameter rod at 2000°F. The break-down stock was hot drawn with a graphite coating at 2000°F to 0.030 size, pickled in nitric-hydrochloric acid, and cold drawn to final 0.005-inch diameter using chlorinated oil lubricant. Only about 22 percent area reduction was allowed between annealing cycles at 1900°F. Wire breakage was comparatively frequent, probably because of voids, inclusions and thoria clumping in the wire. Continuous length wire spools of less than one pound were typical for TD nickel-chromium compared to five pound and larger spools for N 155, DH 242 and Hastelloy X.

#### 4 EXPERIMENTAL PROCEDURE

Test procedures were designed to simulate, within practicable limits, those conditions expected to arise in future utilization of the alloys. Wires were heat treated before testing to simulate typical production sintering or diffusion bonding processes and resultant annealing and grain growth expected in fabricated transpiration cooling materials. Furnace oxidation tests were conducted with cyclic heating and cooling of all specimens at each time interval to roughly simulate the on-off cycle of a hot gas turbine. All alloy wires were tested together to allow direct comparison of results.

#### 4.1 Specimen Preparation

Wire samples of each alloy, (1) N 155, (3) TD nickel-chromium, (6) DH 242, and (10) Hastelloy X, were inspected and selected for testing. Wire diameters were measured with a micrometer to the nearest 0.0001 inch and results were recorded. All wires were within normal tolerances (+0.0005 inch) of 0.005 inch in diameter. Continuous lengths of wire suitable for each test were sonic cleaned in hot trichlorethylene to remove residual drawing compounds or other extraneous surface contamination. Acetone rinsing was used to insure the absence of chloride residues. All wire specimens were heat treated before testing. The sintering cycle typically employed to bond space-wound or woven wiretype transpiration cooling materials was used to simulate annealing and grain growth effects expected in fabricated structures. Heat treatment consisted of two four-hour cycles at 2100°F in dry hydrogen (below -80°F dewpoint) with cooling to room temperature after each cycle. Wires were racked on fixtures to minimize contact or wire kinks and to provide a straight, undistorted sample for stress rupture tests. All wire specimens were clean and bright after simulated sintering. A comparison of wire microstructures showing the "as received" condition and the affect of subsequent simulated sintering is shown in the photomicrographs of Appendix 3, Metallographic Examination. Metallographic specimen preparation is outlined in Table 4-1, Metallographic Etchant Schedule. The effect of sintering on wire specimen mechanical properties 15mshown in Table 4-2, Mechanical Properties of Wire Specimens.

#### 4.2 Cyclic Oxidation Tests

All cyclic oxidation tests were conducted in a manner similar to that reported for sheet specimens in the previous Summary Report CR-930. Wire specimens consisted of loose bundles about six inches long and one half inch in diameter containing approximately 100 feet of 0.005-inch diameter wire which weighed about three grams. Samples were made as large as practical to minimize weighing errors. Each wire bundle was

7

contained in a separate zircon ceramic thimble during oxidation cycling to collect spall and avoid extraneous contamination. The thimbles are shaped like test tubes  $0.88 \ O.D. \times 0.75 \ I.D. \times 7$  inches long with a flat disc lid and having four 0.13-inch air-circulating holes drilled near the top and bottom as shown in Figure 4-1. Preliminary tests with mild steel sheet samples 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. All thimbles were hard fired at 2900°F and baked out at 1400°F to constant weight before using. Each wire bundle and thimble was weighed to 0.1 mg and stored in a dessicator pending oxidation testing.

Thimbles containing wire bundle specimens of each alloy were vertically supported in a 2 x 7 array in a special rack made from 0.19-inch diameter type 330 stainless steel rod to minimize contact area between racks and thimbles. Ten openings were used for thimbles containing alloy samples as shown by number in Figure 4-1. Two openings were used for empty control thimbles to check thimble weight changes due to heating. The last two openings in the rack center were used for dummy thimbles containing inserted thermocouples to simulate and monitor specimen temperature. Five racks containing ten alloy sample sets were arranged on a movable skid-pan, allowing one set for each time cycle and one spare or check set. Thermocouples were #24 Ga chromel-alumel for 1400 and 1600°F tests and #14 Ga chromel-alumel for the higher temperatures. Thermocouples from each rack were connected to a central potentiometer-recorder which continuously monitored the temperature of each specimen set. Automatically controlled and recording electric furnaces with 13 x 16 x 48 inch type 330 stainless steel muffles, loosely blocked with fire-brick, were used to maintain temperatures within +1.0 percent of the nominal setting. Two furnaces were used to expedite testing.

Oxidation cycling procedure consisted of adjusting the furnace to the required temperature with a proportional controller set to minimize temperature fluctuations. Prepared sample skid-pans were loaded with a fork-lift dolly into the furnace and power was first increased and then backed-off to "meet" the temperature to 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 to a dessicator for future examination and the other samples were returned to the furnace. This procedure was repeated for each time interval of 4, 16, 64, 100, 200, 300, 400, 500, and 600 hours and for each temperature of 1400, 1600, 1800, 2000, 2100, and 2200°F.

#### 4.3 Specimen Examination and Testing

After oxidation exposure, each sample set of four alloy wire bundles and thimbles was stored in a dessicator pending later weighing, examination and tensile testing. The following characteristics were determined for each alloy after each temperature-time oxidation period.

- (1) Total oxidation weight gain and oxide spall weight
- (2) Change in wire diameter and surface oxide characteristics
- (3) Oxide penetration and alloy microstructure
- (4) Mechanical properties at room temperature

<u>4.3.1</u> Oxidation and spall.-Each alloy wire-thimble combination was weighed to 0.1 mg to determine total oxidation weight gain and amount of oxide spalling. Data were determined by direct weighing although indirect or difference weighing was also used to provide a check against error. The weighing procedure followed this sequence:

- (1) thimble + spall + wire
- (2) wire
- (3) thimble + spall
- (4) spall
- (5) thimble

The thimble, spall and wire (1) were weighed together. Then the wire (2) was removed, along with adherent oxides, and weighed separately. The remaining spall and thimble (3) were weighed. The spall (4) was removed from the thimble with a soft brush and weighed. Finally, the empty thimble (5) was weighed alone. These redundant weighings provide for checking errors due to accident or oxide loss because of sticking to the thimble.

Total weight gain and spall weight were determined from these data. Direct weights were used throughout this series of tests. Total weight gain was considered equal to the increase in weight of thimble + spall + wire (1) compared to the original total weight of the thimble and wire bundle specimen. Spall was directly determined from spall weight (4) at the lower temperatures. At temperatures of 1800°F and higher, the wire bundle was partially broken by oxidation and wire pieces were difficult to separate from the spall for weighing. Spall weights were not separately determined for these runs.

4.3.2 Thickness and surface examination.-Unbroken wire bundles were remeasured with a micrometer to determine changes in diameter due to oxidation. Surface oxides were examined but were not considered to be suitable for photographic reproduction. Cross section photomicrographs were used to show oxide character. Wire specimens were selected for mechanical testing. 4.3.3 <u>Metallographic examination</u>.-Metallographic samples were selected from each wire bundle specimen. Wires were vertically mounted in lucite between stainless steel support shims. Liquid lucite was painted on each wire sample bundle to eliminate bubbles and to preserve the fragile oxide layer. Samples were segregated according to alloy. Grinding and polishing procedure was routine, finishing with Linde B. Specimens were unetched except for the longitudinal sections of "as received" and "as sintered" wires showing grain structure comparison. These etchants are given in Table 4-1.

Oxidized wires were observed and photographed at 500 X using normal, reflected light. Oxide layer thickness and residual metal cross section was measured directly from the photomicrographs by comparison with a stage micrometer photomicrograph at the same magnification. Metallographic examination of thermogravimetric analysis (TGA) specimens and stress oxidation wire samples, described in Sections 4.4 and 4.6, respectively, was conducted using the same procedure.

4.3.4 <u>Tensile tests</u>.-Wire samples from the cyclic oxidation tests were tensile tested to show changes in mechanical properties caused by oxidation and heat exposure. One inch gage lengths were held between rubber-padded plate jaws faced with fine emery paper to prevent wire slippage. Specimens were pulled at 0.050 inches/minute on an Instron testing machine to provide a continuous stress-strain curve for each specimen. Ultimate tensile strength and yield strength (0.2 percent offset) were determined on the basis of original wire diameter. Elongation was measured directly from the stress-strain curve. Additional tests were conducted on TGA, stress oxidation, "as received", and "as sintered" specimens for comparison purposes.

#### 4.4 Thermogravimetric Analyses

Thermogravimetric analyses (TGA) were conducted on sample wires of each alloy for the purpose of determining conventional oxidation rates in comparison to cyclic oxidation data. Specimen wires about 100 inches in length were coiled and suspended vertically in an electric tube furnace and connected with a platinum wire to a Cahn Electrobalance having 0.1 µg sensitivity. Each specimen was tested separately. Furnace and balance suspension were enclosed to minimize chimney effects in the ambient air atmosphere. The system was calibrated in place against the wire sample, which was previously weighed to 0.1 mg, in the cold furnace. After calibration and check out, the furnace was turned on. Heat-up to testing temperature required approximately 30 minutes. Furnace temperature in the specimen zone was automatically controlled and recorded as a function of time and data were read from the strip chart at 1, 2, 4, 8, 16, 32, 50, 75, and 100 hours at test temperatures of 1400, 1600, 1800, 2000, 2100, and 2200°F. After TGA tests were completed, specimen wires were examined metallographically and tensile tests were conducted. Apparatus is shown in Figure 4-2.

#### 4.5 Stress Rupture Tests

Stress rupture lives up to 100 hours were determined for each alloy. Specimen wires were about three feet in length with about six inches contained in the hot zone. Apparatus consisted of an automatically controlled 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 microswitch 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 at one time with four parallel switch-pulley systems to allow direct comparison of data. A schematic diagram of this equipment is shown in Figure 4-1. Tests were made to provide four data points for each alloy within the 100 hour time range at temperatures of 1400, 1600, 1800, and 2000°F.

#### 4.6 Stress Oxidation Tests

After determination of the stress to rupture at 100 hours for each of the alloy wires, additional tests, using the same apparatus, were made to investigate the effect of stress on oxidation rate. Three wire specimens of the same alloy were strung in the tube furnace as in the stress rupture tests and were loaded at 60 percent of the stress level determined for 100 hour life. Each alloy was tested for 100 hours at temperatures of 1400, 1600, 1800, and 2000°F. After stress oxidation exposure, each specimen was metallographically examined and tensile tested.

#### 5 RESULTS AND DISCUSSION

The purpose of this investigation was to provide supplementary engineering data to evaluate and compare four alloys, N 155, TD nickelchromium, DH 242, and Hastelloy X, which were chosen as "best-compromise" alloys for transpiration cooling material applications. These four alloys were selected from a group of twelve candidates on the basis of earlier tests, described in the previous Summary Report CR-930, which compared sheet specimen oxidation resistance and retention of good mechanical properties. The present tests were conducted with 0.005-inch diameter wire. Wire tests were considered to be necessary because fine wires were expected to have quantitatively different oxidation resistance and mechanical properties compared to the same alloy in sheet metal form. This difference is attributable to both diffusion geometry difference and alloy working history differences. Testing fine wires allows direct application of the resultant data to the problem of alloy selection and prediction for fabricated wire-type transpiration cooling materials such as Poroloy or Rigimesh and Poroplate, and may assist in the interpretation and extrapolation of existing conventional test data.

Oxidation resistance is a primary criterion for transpiration cooling material alloy selection. Maximum efficiency with minimum cooling air consumption requires operation at the highest possible temperature. Excessive oxidation limits service life, decreases air flow permeability, and causes mechanical degradation of the porous material. Retention of strength and ductility during oxidation and heating is more important than initially high mechanical properties alone. Good ductility retention is especially necessary to avoid cracking from cyclic heating and cooling or foreign object damage. Finally, manufacturing characteristics must be considered. Alloys must be drawn into fine wire, wound or woven, sintered, formed and fabricated into useful hardware at an economically feasible cost. This investigation was designed to test alloy properties related to these criteria which are essentially pertinent for transpiration cooling material alloy specification.

Test data comparisons of N 155 (base line alloy), TD nickel-chromium, DH 242, and Hastelloy X are given in the text. Detailed data and photographs for each alloy are presented in appendixes. All numerical data are reduced and tabulated in Appendix 1, "Numerical Data Tabulation". Total oxidation weight gain (cyclic and steady state) and oxide spall weight are plotted as a function of time for each alloy with temperature as a parameter, for both wire and previously tested sheet specimens, in Appendix 2, "Alloy Oxidation Plots". Photomicrographs of wire specimens showing "as received" and "as sintered" microstructure, cyclic oxidation and penetration, steady-state (TGA) oxidation, and stress-oxidation characteristics are shown in Appendix 3, "Metallographic Examination". Stress rupture life and tensile test data are given in Appendix 4, "Mechanical Properties". Oxide penetration is shown in Appendix 5, "Oxidation Penetration Plots".

#### 5.1 Cyclic Oxidation and Spall

Total oxidation weight gain (including spalled oxides) was determined for wire bundle specimens representing each alloy-temperature-time combination. Spall was separately determined for the 1600°F temperature only; at 1400°F spall was negligible, while at 1800°F and higher spall was difficult to separate from broken wire segments which occurred in most specimens. Specific weight gain or spall per unit area was calculated from direct weight data using the actual micrometer measurements (±0.0001 inch) as the basis for calculating area. Specific weight gain comparisons of each alloy wire are shown as a function of time for each temperature from 1400 to 2200°F in Figures 5.1-1 through 5.1-6. Specific oxide spall at 1600°F is shown for each alloy in Figure 5.1-7. All data are shown as faired power-function plots on log-log grids. The resultant linear relationship for most tests suggests that diffusion-controlled oxidation kinetics predominated, in which expected parabolic rate law models were modified by alloy complexity and test conditions such as oxide spalling from cyclic heating and cooling. Similar plots for each alloy with temperature as a parameter are shown in Appendix 2, "Alloy Oxidation Plots", Figures A2-1 through A2-4. Comparisons of both wire and sheet specimen forms are shown for each alloy with temperature as a parameter in Figures A2-5 through A2-8. The sheet data were determined and given in the earlier Summary Report CR-930.

The comparative oxidation and spall rates shown in Figures 5.1-1 through 5.1-7 are identified according to the alloy number sequence originally assigned in the Summary Report CR-930 and repeated in Section 3, "Wire Procurement": (1) N 155, (3) TD nickel-chromium, (6) DH 242, and (10) Hastelloy X. At 1400°F alloy oxidation resistance sequence is 3, 6, 10, 1 in order of decreasing performance. TD nickel-chromium is clearly superior at the longest oxidation times which are considered to be most significant for engineering purposes. Oxidation rate slopes of specific weight/cycle time are also significant since lower slopes indicate slower oxidation rates. The 1400°F ranking of alloys generally holds for the higher temperatures as well. TD nickel-chromium retains its comparative superiority through 2200°F. Hastelloy X and DH 242 are nearly equal at 1800°F, but the former alloy shows sharply increased oxidation rates after 64-100 hours at 2000-2100°F, probably because of increased spalling. The same degradation is shown by base line alloy N 155 beginning at 1600°F and 200 hours. All alloys except TD nickel-chromium "top-off" at the highest temperatures and longest times at an oxidation weight gain near 50-70 mg/in<sup>2</sup>. This limit indicates virtually complete oxidation of the metal wire. The spall data shown in Figure 5.1-7 for 1600°F indicates a comparable ranking of 3, 6, 1, 10, supporting these conclusions.

The cyclic oxidation rate plots of Appendix 2, Figures A2-5 through A2-8, show a comparison of wire and sheet specimens for each alloy at each test temperature. Oxidation rate slope and constant generally tend to increase with increasing temperature. Wire specimens generally oxidize

more rapidly than sheet specimens of the same alloy at the higher temperatures and longer times. With few exceptions, wire slopes are greater than sheet specimen slopes. Since oxidation of these nickel-base alloys is diffusion controlled, these characteristic differences are probably due to geometry differences rather than differences in test conditions or alloy make-up. Sheet specimens approximate an infinite slab model with a comparatively large thickness and mass which acts as a "sump" for outwardly diffusing elements such as chromium. Wire specimens approximate an infinite cylinder model with small diameter and mass which results in a larger concentration or activity gradient for diffusing species. This results in generally higher oxidation rates, especially for longer times and higher temperatures, which must be considered in the extrapolation of oxidation data for fine wires.

#### 5.2 Thickness and Surface Examination

Micrometer measurements of the wire diameter after oxidation were not considered to be significant. The fragile oxide skin was crushed by the micrometer anvil and readings were inconsistent. Therefore, thickness changes were determined by measurement of the metallographic sections shown in Appendix 3 only. Surface oxide appearance was not reproduced photographically because the large curvature of the 0.005-inch diameter wire made focusing of the image in one plane impossible. Visual examination showed textures and general appearance to be essentially similar to previous sheet specimen observations.

#### 5.3 Metallographic Examination

Photomicrographs are reproduced in Appendix 3, "Metallographic Examination", which show wire alloy microstructure in the "as received" condition and which show grain growth due to the "simulated sintering" cycle employed with all specimens. Additional photomicrographs demonstrate progressive attack and penetration of each alloy wire during cyclic oxidation. These photomicrographs were measured to obtain the data displayed in Appendix 5, "Oxidation Penetration Plots", which shows oxide penetration and growth as a function of time for each alloy and temperature. Photomicrographs of TGA and stress-oxidation samples are also shown.

Alloy 1, N 155, is shown in Figure series A3-1. The "as received" and "as sintered" specimens, shown in longitudinal section to demonstrate the wire microstructure, indicate the expected transition from an illdefined wrought structure containing carbides and other inclusions to a distinctly granular structure after sintering having discontinuous grain boundaries. Cyclic oxidation specimens are shown in transverse section to display oxide growth and penetration. At 1400°F oxide skin growth is steady and small with no visible penetration, internal oxidation, or significant microstructural changes in the unetched section. At 1600°F minor penetration begins after about 100 hours and considerable oxide growth is visible after 300 hours. At 1800°F oxide growth is immediate, internal oxidation and penetration is significant after 100 hours, and oxidation attack is virtually complete by 400 hours. At 2000 and 2100°F the wire is oxidized almost immediately. TGA specimens at 100 hours are generally less severely oxidized than cyclic oxidation specimens. At 1400 and 1600°F little attack is apparent, but 1800 and 2000°F specimens show heavy and severe oxidation. Stress oxidation specimens after 100 hours are generally comparable to the other specimens at the same time except that oxidation is visibly more severe at 1800 and 2000°F.

Alloy 3, TD nickel-chromium is shown in Figure series A3-2. The "as received" sample has an apparently homogeneous matrix at 500X with no discernable grain structure in the longitudinal section. Larger thoria particles are visible and elongated in the direction of the wire drawing. The sintered specimen, in contrast to the other alloy samples, shows little directionality and little grain growth. Cyclic oxidation transverse sections show little visible oxidation at 1400°F except for the longest times. At 1800°F external oxidation is more rapid and penetration begins after about 100 hours, but the wire bulk is still unaffec-At 2000°F behavior is essentially the same as at 1800°F. At 2100°F ted. external oxidation is more rapid but significant penetration or internal oxidation is not apparent. At 2200°F external oxidation is still more severe, the wire is less uniform in diameter, but major penetration into the wire bulk section or internal oxidation is still small. TGA specimens are virtually unaffected at 1400 and 1600°F after 100 hours. Oxidation progresses gradually at 1800 to 2100°F with comparatively little penetration and no internal attack. Stress oxidation specimens demonstrate significant internal oxidation. At 1400°F after 100 hours comparatively little oxidation is apparent and internal oxidation is absent or negligible. At 1600°F internal oxidation is substantial and becomes progressively more severe at 1800 and 2000°F. No comparable internal oxidation was observed in unstressed specimens at temperatures up to 2200°F and times up to 600 hours.

Alloy 6, DH 242, is shown in Figure series A3-3. The wrought, elongated grain structure of the "as received" longitudinal sample contains minor carbides which are retained after sintering. Grain growth occurs, but is smaller compared to alloys 1 and 10, and grain boundaries are comparatively continuous with fewer precipitates. Cyclic oxidation transverse sections at 1400°F show little attack except for minor penetration after about 300 hours. At 1600°F visible penetration begins after 100 hours and further penetration or internal oxidation progresses after 300-600 hours. At 1800°F major oxidation attack begins quickly and internal oxidation progresses significantly after 100 hours. At 2000°F oxidation is rapid and the wire has virtually disintegrated by 100-200 hours. At 2100 and 2200°F wire oxidation is nearly complete by 16-64 hours. TGA specimens after 100 hours show little attack at 1400 and 1600°F. Internal oxidation begins at 1800°F and is severe at 2000°F. Stress oxidation specimens are similar to TGA specimens. At 1400°F little oxidation is apparent, but at 1600°F internal oxidation begins and progresses at 1800 and 2000°F. Internal oxidation observed in chemically similar TD nickel-chromium is not apparent.

Alloy 10, Hastelloy X, is shown in Figure series A3-4. The long tudinal "as received" specimen shows heavy, directional inclusions or precipitates in a homogeneous matrix. After sintering, some precipitates have apparently dissolved and re-precipitated in the heavy, discontinuous boundaries of a comparatively large grain structure. Cyclic oxidation transverse sections at 1400°F show little oxidation attack before 300-400 hours. At 1600°F oxidation is more rapid with penetration and internal oxidation becoming apparent after 200-300 hours. At 1800°F internal oxidation becomes significant after 100 hours and progresses rapidly. At 2000°F disintegration is nearly complete by 200 hours. At 2100 and 2200°F oxidation has nearly destroyed the wire by 16-64 hours. TGA specimens after 100 hours are less severely attacked than cyclic oxidation specimens. Oxidation is relatively minor at 1400 and 1600°F, and is not severe at 1800°F. At 2000°F wire oxidation and disintegration is nearly complete. Stress oxidation specimens are generally similar in appearance to the TGA specimens after 100 hours. Substantial oxidation begins at 1800°F and progresses at 2000°F.

Examination of the oxide layers, penetration, and internal oxidation of these metallographic sections show progressive changes with increasing temperature and time which may be correlated with alloy performance. The most oxidation resistant alloys which retain good mechanical properties have comparatively thin, uniform oxide layers with little or no penetration and internal oxidation within the useful temperature-time range. The beginning of substantial oxide penetration or internal oxidation is associated with increased spalling and oxidation rates and results in a comparatively rapid deterioration in mechanical properties. Oxide penetration and thickness changes were measured from the cyclic oxidation sections and are plotted and explained in Appendix 5, "Oxidation Penetration Plots," as a function of time for each alloy and temperature.

#### 5.4 Tensile Tests

All tensile test data for cyclic oxidation, TGA, and stress-oxidation tests are tabulated in Appendix 1, "Numerical Data Tabulation", which gives ultimate strength, yield strength, and elongation for each alloy wire specimen. Selected data at 100 hours and 600 hours cyclic oxidation exposure time are plotted in Appendix 4, "Mechanical Properties", which shows progressive changes in alloy mechanical properties with increasing temperature. Mechanical properties of both wire and previously tested sheet specimens are shown in comparison in Figures A4-1 through A4-4. Wire tensile properties after cyclic oxidation, TGA, and stress oxidation are compared after 100 hours exposure time in Appendix 4, Figure A4-5 in a similar manner. Alloy comparisons at each test temperature are shown in the bar charts of Figures 5.4-1 and 5.4-2 for cyclic oxidation, TGA, and stress oxidation tests after 100 hours.

At 1400°F these bar chart alloy comparisons show that retained mechanical properties follow a similar pattern for each alloy. Cyclic (CYC) oxidation is generally more severe than steady-state TGA oxidation and stress-oxidation (S-0) tensile test results lie mid-way between these extremes. TD nickel-chromium (3) is superior, followed by alloys 10, 1, and 6 in order of strength. These general characteristics are retained at 1600°F with alloy 3 remaining conspicuously stronger after each test and retaining good ductility. Alloy 6, DH 242, begins to contend with the superalloys 1 and 10 in retained strength at room temperature and shows better ductility. At 1800°F the sense of these comparisons is sharply changed. TD nickel-chromium (3) is clearly strongest in CYC and TGA tests and retains good ductility, but no strength or ductility is retained after S-O tests because of severe internal oxidation induced by loading. DH 242 (6) is also oxidized but zero strength after S-O tests is due primarily to large elongation under load. Hastelloy X (10) is superior to N 155 (1), but both alloys are brittle. At 2000°F TD nickelchromium is the only "survivor"; all other alloys are oxidized to virtual destruction. Mechanical properties are good for both CYC and TGA tests. However, application of stress in S-O tests causes internal oxidation, as shown in Section 5.3, and structural integrity is completely lost.

The tensile test data plots of Appendix 4, Figures A4-1 through A4-4, show comparisons of wire and sheet properties as a function of temperature for each alloy. Wire properties are generally better than equivalent sheet properties at the lower temperatures of 1400 and 1600°F but fall off rapdily at 1800°F and higher because of oxidation attack. Alloy 1, N 155, has substantially higher mechanical properties in wire form for "as received", "as sintered", and 1400-1600°F cyclic oxidation tests. These properties fall off sharply after 100 hours at 1600°F and 600 hours at 1400°F. Alloy 3, TD nickel-chromium, has slightly better properties in wire form and retains better properties after 100 hours exposure time, falling off somewhat faster with increasing temperature at 600 hours. Alloy 6, DH 242, like alloy 1, has initially higher wire properties which rapidly decrease at temperatures over 1400-1600°F. Alloy 10, Hastelloy X, has similar behavior because of oxidation of the finer wire section.

Figure A4-5 provides a similar comparison of cyclic (CYC) oxidation, TGA, and stress-oxidation (S-O) specimens after 100 hours. These plots show that cyclic oxidation is generally more severe than steady-state TGA oxidation and stress-oxidation is intermediate in its effect on retained mechanical properties except for alloy 3, TD nickel-chromium, and at the highest temperatures. Alloy 1 drops off rapidly above 1600°F in mechanical properties and S-O exposure is most severe, probably because of increased creep and spalling. Alloy 3 is stress sensitive above 1600°F and no S-O specimens were tested because of extreme brittleness, while CYC and TGA specimens had good properties well above 2000°F. Alloy 6, like alloy 1, was good until 1600°F but oxidation at higher temperatures precluded further tensile testing. Alloy 10 also oxidized heavily beginning at 1800°F and higher temperature specimens were not tested.

### 5.5 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) data is tabulated in Appendix 1, "Numerical Data Tabulation," along with tensile test data for the wire specimens after 100 hours exposure time. Wire photomicrographs are shown in Appendix 3, "Metallographic Examination". Total specific weight gain from continuous, steady-state oxidation is plotted in Figures A2-9 through A2-12 of Appendix 2, "Alloy Oxidation Plots", for each alloy with testing temperature shown as a parameter. Tensile test data is plotted, as described in Section 5.4, in Appendix 4 and Figures 5.4-1 through 5.4-4 which also show comparisons with wires tested after cyclic oxidation and stress oxidation. Steady-state oxidation is less severe than cyclic oxidation. Comparison of the curves for each alloy shown in Appendix 2 indicates a generally lower slope and smaller rate constant or intercept for the TGA power function plots. The greater oxidation rates experienced in cyclic oxidation are probably due to greater oxide spalling caused by heating and cooling. The TGA plots for alloys 1, 6, and 10 are "well behaved" with little deivation from the expected orderly linear function. Alloy 3, TD nickel-chromium, deviates from this model at temperatures above 1800°F. Specific weight decreases at 2000, 2100, and 2200°F for times beyond eight hours and beyond 50 hours at 2200°F negative values are obtained. This anomalous behavior was originally attributed to spalling or chromium evaporation. Further analysis of the test method and data reduction shows that it is probably due to oxidation during the initial heat-up period. If corrections are estimated for this error, all higher temperature oxidation data falls in a narrow, horizontal band lying between 1.5 and 4.0  $mg/in^2$  total specific weight gain. This means that TD nickel-chromium's initial oxidation (during heat-up) is rapid but small in magnitude. Later oxidation is slow and small weight losses (due to spalling, etc.) have a large negative affect on the reduced data. The other alloys did not show this peculiarity because of their comparatively greater oxidation, especially at the higher temperatures.

#### 5.6 Stress Rupture

Stress rupture data for each time and load at temperature are listed in Appendix 1, "Numerical Data Tabulation". These data are plotted in Appendix 4, "Mechanical Properties", Figures A4-6 through A4-9 which show rupture stress as a function of exposure time for each alloy with temperature as a parameter. Interpolated stress to rupture at 100 hours is tabulated below in Table 5.6-1 for each alloy and temperature.

		TABLE	5.6	<u>5-1</u>		
STRESS	TO	RUPTURE	IN	100	HOURS,	PSI

Alloy		Temperature, °F								
		1400	1600	1800	2000					
(1)	N 155	32,000	15,000	7,800	2,100					
(3)	TD Ni-Cr	7,700	7,700	6,100	4,400					
(6)	DH 242	16,500	7,000	2,000	890					
(10)	Hastelloy X	15,700	7,500	4,200	2,520					

These stress rupture lives are generally comparable to literature values reported for bar specimens of the conventional superalloys N 155 and Hastelloy X. Wire stress rupture values are typically lower by about 5-20 percent, probably because of the much greater weakening effect of oxidation on the fine wire and weakening by small microstructural imperfections. DH 242 loses strength rapidly with increasing temperature, because it is a simple solid-solution alloy, compared to the precipitation strengthened superalloys. In contrast, the thoria dispersion strengthened TD nickel-chromium is weakest at lower temperatures but is comparatively strongest at higher temperatures. The linear extrapolation of 1400°F data for TD nickel-chromium has been assumed to become asymptotic to the 1600°F data extrapolation, giving the same stress-to-rupture value at 100 hours, since no data was generated for this time interval. The linear extrapolations are retained, however, in Figure A4-7 to illustrate this anomalous behavior. Id nickel-chromium, alone, exhibited nearly flat stress rupture curves at 1600°F and higher. Data points were difficult to obtain below 100 hours, especially at the higher temperatures, because of an unusual "stress sensitivity". For loads below a critical stress, wire life was indefinite and tests were discontinued after several hundred hours. At slightly greater loads wire life was shortened to a few minutes. Only about 100 psi difference in total stress was sufficient to cause this behavior which was attributed to catastrophic oxidation attack induced at the critical stress level. Further investigation is needed to define this phenomenon because of its importance for engineering design purposes.

#### 5.7 Stress Oxidation

Stress oxidation data and subsequent room temperature tensile test results are listed in Appendix 1, "Numerical Data Tabulation". Photomicrographs showing wire cross section and oxidation effects are reproduced in Appendix 3, "Metallographic Examination". Determination of specific weight gain due to oxidation was attempted and abandoned after experiments showed it to be impractical because of weighing error. The wire specimen in the six-inch hot-zone weighed less than 15 mg and weight increases were too small to provide significant data. Metallographic examination shows little stress sensitivity except at the highest temperature of 2000°F for alloys 1, 6, or 10. Above 1800°F these alloys show increased oxidation, probably because of spalling or elongation under load. Alloy 3, TD nickel-chromium shows marked stress sensitivity beginning at 1600°F. Internal oxidation, which was catastrophic for stress rupture specimens above a critical and reproducible stress level, and which does not occur in unstressed specimens below 600 hours at 2200°F, progresses significantly in the range of 1600-2000°F under the specified 60 percent of stress for 100 hour rupture life. This phenomenon is also confirmed by the tensile test data referenced in Section 5.4 of the text, which shows a sharp decrease in room temperature strength and ductility after stress-oxidation exposure in this high temperature range.

#### 6 CONCLUSIONS

Alloy specification for transpiration cooling materials requires selection of an optimum engineering compromise of properties which include oxidation resistance, strength and ductility retention and economic or manufacturing feasibility. Oxidation resistance is of paramount importance. The maximum operating temperature (engine cooling efficiency) and service life of a fine-wire transpiration cooling material are largely determined by the rate of oxidation attack and penetration. Fine wires are more susceptible to oxidation damage than thicker sheet metal. Uniform oxide penetration of only a few ten-thousandths of an inch will seriously affect the mechanical properties of 0.005-inch diameter wire. Similar oxide growth will reduce the transpiration cooling material's air flow permeability and may cause localized over-heating and increased oxidation attack. Non-uniform or internal oxidation may cause catastrophic failure of the wire and the material. Mechanical properties such as stress-rupture strength and ductility are also important, but these characteristics are largely determined by oxidation resistance in actual service. Manufacturing feasibility is the final consideration, but except for demonstration of wire drawing, it is beyond the scope of this report.

Alloy N 155 was chosen as the base line for comparing other alloys because of its previous utilization in many types of transpiration cooling materials which have been tested in a wide variety of applications. The maximum service temperature limit for N 155 is near 1600°F. Other alloys may be compared with base line N 155 by extrapolating oxidation test data to estimate the temperature at which their oxidation resistance is equivalent to N 155 at 1600°F and 600 hours exposure which is considered most significant for engineering applications. These extrapolations are made in the Arrhenius plots of Figures 6-1 and 6-2 for total specific weight gain and oxide penetration depth, respectively. These data may be reasonably approximated by a straight line or a smooth, upward-concave curve having one straight segment. Data fitting the former model indicate a simple exponential oxidation rate/reciprocal absolute temperature relationship while curved plots imply a temperature dependent change in oxidation activation energy. Specific weight gain and penetration are related to oxidation rate and the resultant model provides a numerical basis for alloy comparison. The more oxidation resistant alloys have a higher equivalent temperature than base line N 155 at 1600°F. These temperatures are tabulated below for each alloy in Table 6-1 in comparison with N 155 for which weight gain was 9.6 mg/in and penetration was 0.0016 inch at 1600°F for 600 hours exposure time. All data were read from the idealized curves of Figures 5.1-1 through 5.1-6 at 600 hours exposure time.

## TABLE 6-1COMPARISON OF EXTRAPOLATED ALLOY PROPERTIES

Equivalent Performance Temperature, °F

	Alloy	Weight Gain	Oxidation Penetration
1.	N 155	1600	1600
3.	TD nickel-chromium	2090	1970
6.	DH 242	1690	1700
10.	Hastelloy X	1710	1660

Comparisons are also shown by dashed lines for the previously tested sheet specimens for which data was given in the Summary Report CR-930, Sheet specimens are more oxidation resistant than wire specimens, because of their comparative geometry, and have higher equivalent temperature ratings when compared to wire. This characteristic is also shown in the wire-sheet comparisons of Section 5, and must be considered in the extrapolation of bulk specimen data for application to fine wires.

TD nickel-chromium is clearly superior in oxidation resistance and retention of mechanical properties as measured by the cyclic oxidation, TGA, and stress rupture tests at the higher temperatures and longer times which are most significant for further transpiration cooling development. However, stress-oxidation tests apparently show that service applications must be limited to low stress levels or temperatures below 1600°F to prevent stress-induced internal oxidation. Alternatively, the mechanism of this anomalous oxidation behavior should be determined to provide a basis for other corrective measures such as thoria particle modification, matrix alloy chemistry changes, or protective coatings. DH 242 and Hastelloy X are roughly equivalent in most of the measured characteristics and both have an apparent service temperature limitation near 1700°F. DH 242 is initially weaker than Hastelloy X, but slightly better oxidation resistance and better spall resistance tend to equalize mechanical properties, especially ductility retention, after oxidation exposure. All three alloys are feasible to use in the manufacture of transpiration cooling materials and components. Alloy GE 1541, which was tested as a sheet specimen but not in wire form, has since been recognized as a superior alloy, rivaling TD nickel-chromium in many of its properties, and should be further tested for transpiration cooling applications.

Based on these wire specimen tests, the major conclusions of this report are summarized.

1. TD nickel-chromium, while clearly superior in no-load oxidation resistance, is sensitive to stress above a sharply defined level and oxidizes internally at comparatively low temperatures above 1600°F.

22

- 2. DH 242 has good general oxidation resistance and ductility retention, but it is limited to small loads because of elongation at temperatures above 1600°F.
- 3. Hastelloy X has fair general oxidation resistance and ductility retention in spite of its comparatively high spall rate, and is strong at temperatures above 1600°F.
- 4. Cyclic oxidation exposure is substantially more severe than steady-state oxidation, especially for wire specimens.
- 5. Wire specimens are more severely oxidized than equivalent sheet specimens, especially at higher temperatures.

Other		2.5 ThO2 2.23ThO2			
р.	0.018	0.005	0.004	0.016 0.004	
s	0.013	0.006	0.002	0.004 0.003	
Сu			0.02		
Fe	Bal. Bal.		0.87 0.88	18.07 18.00	
Cb∔Ta	1.13 1.18		1.27 1.33		
м	2.41 2.26			0.52 0.61	
Мо	2.72 2.69			9.08 8.77	
Co	19.92 19.85			1.45 0.75	
ŢΝ	19.16 19.33	Bal. Bal.	Bal. Bal.	Bal. Bal.	
Сr	21.07 20.88	21.85 21.52	19.26 19.23	21.57 21.53	
Si	0.31 0.34		0.95 1.04	0.57 0.71	
Mn	1.35 1.24		0.08 0.02	0.58 0.50	
c	0.06 0.092	0.011 <0.01	0.03 0.035	0.09 0.08	
Refer Below	-1 <sup>-</sup> 2	0.12	юю	4	
ALLOY	(1) N 155	(3) TD nickel- chromium	(6) DH 242	(10) Hastelloy X	

ALLOY ANALYSIS TABLE 3-1

Wire Supplier

Mill Certification

National Standard Co. Hoskins Manufacturing Co. Driver-Harris Co. Hoskins Manufacturing Co. 4.9.2.4

AMS 5794 Proprietary Alloy-DuPont Proprietary Alloy-Driver-Harris AMS 5798

Heat Number M4-5905 1895 1826 X3-4844

Chemical Analysis

5. Bendix Filter Division Laboratory

ALLOY	ETCHANT	PROCEDURE				
(1) N155	2% Chromic Acid> - 4 ml Hydrochloric Acid - 96 ml	Let solution set for one-half hour or until solution light- ens. Using a carbon cathode electrolytic etch with 5 volts until a blue stain appears. Turn the current off and-swirl the sample in the etchant until the blue stain is removed.				
(3) TD Ni-Cr	10% Oxalic Acid	Electrolytic: 5 volts, 3-10 seconds.				
(6) DH 242	10% Oxalic Acid	Electrolytic: 5 volts, 5-25 seconds.				
(10)	Ferric Chloride - 5 ml Hydrochloric Acid - 50 ml Water - 100 ml	Swab.				
Hast X	ALTERNATE ETCHANT 10% Oxalic Acid	Electrolytic: 5 volts, 2-5 seconds.				

## TABLE 4-1 METALLOGRAPHIC ETCHANT SCHEDULE

TABLE 4-2 MECHANICAL PROPERTIES OF WIRE SPECIMENS

		AS RECEIVE	D	AS SINTERED			
ALLOY	Y.S. 10 <sup>3</sup> psi	U.S. 10 <sup>3</sup> psi	%E. in 1.0 in.	Y.S. 10 <sup>3</sup> psi	U.S. 10 <sup>3</sup> psi	%E. in 1.0 in.	
(1) N 155	186.0	196.0	6.3	79.2	138.5	14.0	
(3) TD Ni-Cr	117.0	144.0	22.4	104.0	140.0	18.5	
(6) DH 242	173.0	177.0	7.3	44.3	110.5	22.7	
(10) Hast X	162.0	200.0	5.5	72.1	123.9	12.0	

25







Figure 4-1 Specimen and equipment schematic.

26



Figure 4-2 Thermogravimetric analysis apparatus.


Figure 5.1-1 Cyclic oxidation resistance comparison, 1400°F.



Figure 5.1-2 Cyclic oxidation resistance comparison, 1600°F.



Figure 5.1-3 Cyclic oxidation resistance comparison, 1800°F.



Figure 5.1-4 Cyclic oxidation resistance comparison, 2000°F.



Figure 5.1-5 Cyclic oxidation resistance comparison, 2100°F.



Figure 5.1-6 Cyclic oxidation resistance comparison, 2200°F.



Figure 5.1-7 Cyclic spall resistance comparison, 1600°F.





ħ.



Figure 6-1 Extrapolated oxidation resistance comparison at 600 hours exposure time.



Figure 6-2 Extrapolated penetration resistance comparison at 600 hours exposure time.

### APPENDIX 1 NUMERICAL DATA TABULATION

#### CONTENTS

ALLO	Y				1	TABLE								·								PAGE
1.	N 155		•	•		A1-1	٠		•	•		•		•		.•	•			•	•	40
3.	TD nickel-chromium	•	•	•	.•	A1-2	•		•								٠	•		÷	•	44
6.	DH 242	•	•		•	A1-3		•					•			•			٠		•	48
10.	Hastelloy X					A1-4					•				•							52

Reduced numerical data for all tests are tabulated for each alloy wire. <u>Cyclic oxidation</u> data is listed for exposure time (A), specific total weight gain (B), specific spall weight (C), change in wire diameter (D), uniform oxide thickness (E), depth of penetration (F), yield strength (G), ultimate strength (H), and percentage elongation (I) for each alloy and test emperature. <u>Thermogravimetric analysis</u> data is listed for exposure time (J), specific weight gain (K), change in wire diameter (L), uniform oxide thickness (M), depth of penetration (N), yield strength (O), ultimate strength (P), and percentage elongation (Q). <u>Stress rupture</u> data is listed for exposure time (R), and ultimate or stress-to-rupture strength (S). <u>Stress oxidation</u> data is listed for change in wire diameter (T), uniform oxide thickness (U), depth of penetration (V), yield strength (W), ultimate strength (X), and percentage elongation (Y). Alloy identification and test temperatures applicable to each data series are noted on each table.

Asterisks (\*) in data columns indicate that significant measurements were not obtained for the following column items.

	$\int$	No Correct Contents	ALL		CYCLIC	OXIDATIC	DN			7	THERMOGRA	AVIMETRIC Z
Et Open	HOURS TINE	SPECIFIC TOTAL	Relicing Statut	10-3 ELANE WIRE	10-3 01 10E	10-34 0110 11-34 0110 11-10 10-34	HIJIN FOR	103° 577EWCTH PS1 DS1 PERCHENT	1.00 111 2.00 111 E.100011	SPECIFIC TIME	CHANCE IN WEIGHT	in the second
A	В	C	D	E	F	G	Н	Γ I	J	K	Γ L	ſ
TEMPER	ATURE 14	00°F			1							
4	0.7	0.0	0.0	0.04	0.0	42.1	91.8	20.5	1	0.04		1
16	1.1	0.0	0.0	0.08	0.0	45.8	59.9	5.5	2	0.05		1
64	1.4	0.0	0.0	0.10	0.0	48.4	99.0	15.0	4	0.07		1
100	1.5	0.0	0.0	0.10	0.0	56.1	94.5	14.5	8	0.10		1
200	1.5	0.0	0.0	0.12	0.0	58.6	107.1	11.5	16	0.13		]
300	1.5	0.0	0.0	0.12	0.0	63.8	105.8	8.8	30	0.19		]
400	1.8	0.0	0.0	0.12	0.0	71.3	116.0	14.0	50	0.22		
500	1.7	0.0	0.0	0.14	0.0	68.8	118.2	12.0	75	0.25		
600	1.8	0.0	0.0	0.16	0.0	68.8	112.8	9.8	100	0.26	0.0	
600c	1.8	0.0			1	66,3	111.0	11.2				
TEMPER	ATURE 16	00°F	1	1		<b></b>		<u> </u>				1
4	0.9	0.0	0.0	0.04	0.0	52.3	74.1	6.8		0.13		
16	1.5	0.0	0.0	0.08	0.2	51.7	96.4	15.0	2	0.19		
64	2.2	0.1	0.0	0.10	0.2	59.7	103.0	12.5	4	0.28		
100	2.6	0.2	0.0	0.12	0.24	63.8	99.5	11.5	8	0.36		
200	2.4	0.5	0.0	0.14	0.24	52.3	96.8	10.3	16	0.45		
300	4.3	1.5	0.0	0.34	0.40	58.6	94.3	8.5	30	0.56		
400	6.4	2.7	0.2	0.60		53.6	62.6	4.8	50	0.59		
500	8.0	2.7	0.2	0.80	0.90	48.0	58.6	4.5	75	0.81	1	
600	8.9	2.9	0.3	0.70	0.90	31.9	31.9	0.8	100	0.90	0.10	
600c	8.4	2.5	L		L	55.1	61.2	2.5				
TEMPER	ATUKE 180			0.00	0.01						1	
16	3.1	*	0.1	0.06	0.04	61.3	94.3	113.0	1	0.33		
4/	67		0.2	0.20	0.30	53.6	85.4	9.8	2	0.52	+	
100	15.2		0.2	0.24	0.24	48.5	66.8	5.8	4	0.91	+	
200	29 /	-	0.2	0.80	0.70	43.8	43.8	2.5	8	1.32	+	
300	25 5		1 2	2.0			<u> </u>	*	16	1./9		
400	50.7		2.8	2.0	*	÷			30	2.39		
500	59.7		*	*					50	3.23		
600	62.4	_	_	_			<u> </u>		100	3.43	0.00	
600c	62.4	_	1			_	_		100	5.90	0.20	

TABLE A1-1 NUMERICAL DATA TABULATION: ALLOY 1, N 155.

Image: Second			THERMO	GRAVIMET	RIC ANAI	YSIS	STRE	SS RUPTU	RE		STRESS	OXIDATI	ON
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	THICONECC	10-3 04 04 20-3 04 10E	0-374 704 705 21, 01 01 01 01 01 01 01 01 01 01 01 01 01	The Dest Merin	Par Relicity	1.0 - 11. 2051 - 11. 2051 - 11.	Star 111E	103-255 18-1 18-1 19-1 19-1 19-1 19-1 19-1 19-1	0-3 ETER MIRE HICKNESS	10-3 0, 00 211.05 211.05	0-3 Pan 1011 21. 011 ELD	Marty Contracting	ELONGACITA
MN0PQRSTUVWXYTEMPERATURE 160°FIIIIIS4.00.00.140.1686.0113.09.8IIIIIS5.00.00.140.1686.0113.09.8IIIIIS5.00.00.140.1686.0113.09.8IIIIIS5.00.00.140.1686.0113.09.8IIIIS6.334.5IIIIIIIII82.8032.3IIIIIIIIII94.0031.8IIIIIIIIIII93.031.8II <td>1.5</td> <td></td> <td></td> <td>1 in</td> <td>/ ~</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	1.5			1 in	/ ~								
TEMPERATURE 1400°F        I      I      I      I      1.00      54.0      0.0      0.14      0.16      86.0      113.0      9.8        I      I      I      1.95      50.6      I <tdi< td=""><td>М</td><td>N</td><td>0</td><td>P</td><td>Q</td><td>R</td><td>S</td><td>Т</td><td>U</td><td>V</td><td>W</td><td>X</td><td>Y</td></tdi<>	М	N	0	P	Q	R	S	Т	U	V	W	X	Y
	TEMPER	ATURE 14	00°F			8		<b>.</b>	1				
Image: state of the state	· · · · · · · · · · · · · · · · · · ·					1.00	54.0	0.0	0.14	0.16	86.0	113.0	9.8
Image: state of the state						1.95	50.6						
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$						11.55	40.9	<b>[</b>					
Image: state of the state						36.35	34.5	[					
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$						82.80	32.3						
Image: state of the s						94.00	31.8	ļ					
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$						98.30	33.4						
0.02    0.04    78.0    115.0    5.3			· · · · · · · · ·					<b>_</b>			ļ		
TEMPERATURE 1600°F      Image: strate	0.02	0.04	78.0	115.0	5.3			ļ					
TEMPERATURE 1600°F      I.6      32.3      0.0      0.12      0.16      51.0      99.0      9.8        Image: Ima		<u> </u>	I					L			L	ļ	
1.6    32.3    0.0    0.12    0.16    51.0    99.0    9.8      1.65    27.0    6.46    21.6    1.65    27.0    1.65    1.65    1.65      1.65    27.0    16.2    1.65    27.0    1.65	TEMPE	RATURE 16	500°F	1		1		<del></del>	r		<b>r</b>	r	
Image: state of the state		-				1.6	32.3	0.0	0.12	0.16	51.0	99.0	9.8
-    6.46    21.6    - </td <td></td> <td></td> <td></td> <td></td> <td></td> <td>1.65</td> <td>27.0</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>						1.65	27.0						
						6.46	21.6				· · · · ·		
		+				37.50	16.2						
96.21    15.3    1    1      113.91    15.1    1    1    1      0.6    0.8    67.0    113.0    7.4    1    1    1    1      TEMPERATURE 1800°F      1    7.20    10.8    0.10    0.50    0.80    42.0    42.0    0.6      1    22.88    9.7    1    1    1    1    1    1      1    73.74    7.6    1    1    1    1    1    1      1    1    1    1    1    1    1    1    1    1      1 </td <td></td> <td></td> <td></td> <td><u> </u></td> <td></td> <td>57.40</td> <td>15.5</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>				<u> </u>		57.40	15.5						
113.91    15.1    1			-			96.21	15.3						
0.6    0.8    67.0    113.0    7.4 <t< td=""><td>-</td><td></td><td></td><td></td><td></td><td>113.91</td><td>15.1</td><td></td><td></td><td></td><td></td><td></td><td></td></t<>	-					113.91	15.1						
0.6    0.8    67.0    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    7.4    113.0    113.0    7.4    113.0    113.0    7.4    113.0    113.0    7.4    113.0    113.0    7.4    113.0    113.0    7.4    113.0    113.													
TEMPERATURE 1800°F      1    7.20    10.8    0.10    0.50    0.80    42.0    42.0    0.6      22.88    9.7    27.95    8.6    1    1    1    1      1    27.95    8.6    1    1    1    1    1    1      1    73.74    7.6    1	0.6	0,8	67.0	113.0	7.4			<u> </u>					
1111 Extracted 1000 F    7.20    10.8    0.10    0.50    0.80    42.0    42.0    0.6      22.88    9.7    27.95    8.6    1 </td <td>TEMPE</td> <td>DATURE 11</td> <td>1 800°ឆ</td> <td><b>i</b></td> <td>l</td> <td>J</td> <td></td> <td></td> <td></td> <td></td> <td>1</td> <td>l,_</td> <td></td>	TEMPE	DATURE 11	1 800°ឆ	<b>i</b>	l	J					1	l,_	
22.88  9.7			<u> </u>	Ţ		7.20	10.8	0.10	0.50	0.80	42.0	42.0	0.6
27.95  8.6    73.74  7.6    0.20  0.60    51.0  75.0    4.7	, <u>, , , , , , , , , , , , , , , , , , </u>			[		22.88	9.7	1			1		
73.74  7.6    73.74  7.6    0.20  0.60    51.0  75.0    4.7		1	h		1	27.95	8.6	1					
0.20 0.60 51.0 75.0 4.7		1	Ľ			73.74	7.6						
0.20 0.60 51.0 75.0 4.7	·	1		1		<u> </u>			ł		1		
0.20 0.60 51.0 75.0 4.7		1		1	1	1 I		1	1				
0.20 0.60 51.0 75.0 4.7	······································	1	1	1					1			1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	
0.20 0.60 51.0 75.0 4.7	·····	1					1		1		l · · · · · · · · · · · · · · · · · · ·		·,
	0,20	0.60	51_0	75.0	4.7	1	· · · · ·					ţ.	
		1 0.00	1 21.0	1	1	1						<u> </u>	<u> </u>

TABLE A1-1 NUMERICAL DATA TABULATION: ALLOY 1, N 155. (Continued)

	$\square$	<u></u>			CYCLIC	OXIDATIO	N	<u></u>		Т	HERMOGRAVIMETRIC ANALYSIS	<sup>5</sup> 7
	DURS TINE	12 CALINAL 12 CALINAL	10% 10% 10% 10% 10% 10% 10% 10% 10% 10%	Street With	10 10 10 10 10 10 10 10 10 10 10 10 10 1	Starton Strate	Post WCTH	Portencer	10- 10- 101	The The	The second se	7
	A SPEC	17 - 53 18 18 18 18 18 18 18 18 18 18 18 18 18		OT THE	07 12 12 1427		0., 12 11 11	9   II <u>8</u>			and the second	
A	В	С	D	Ę	F	G	H	I	J	ĸ	L	
TEMP	ERATURE 2	2000°F							-			
4	8.4	*	0.2	0.3	0.08	51.0	62.5	8.8	1	1.08		
16	22.6	. <del>.</del>	0.6	1.0	1.0	*	*	*	2	1.67		
64	63.6	_	*	*	*	-				2.30		
100	62.3	_	-	-	_	-	-	-	8	3.27		
200	61.0	-	-	-	-		-	-	16	4.69		
300	61.7	-	_	_ `		-		-	30	6.70		
400	60.2	-	-	-	-	-	-	_	50	9.28		
500	61.2	-	-		-	_	<u>.</u>		75	12.33		
600	61.6	_				_			100	17.10	0.40	
600c	60.9		_	-	-	-	_	÷	L			
TEMP	ERATURE 2	210 <b>0°</b> F										
4	46.8	*	2.0	*	*	*	*	*	1	1.7		
16	66.3		*	_	~		-	-	2	2.5		
64	63.0	_	-	-	-	-	-	-	4	3.7		
100	60.7		– .	-	-		-	_	8	5.4		
200	60.8		-	-		-	_	-	16	7.9		
300	*				-	-		-	30	16.4		
400	-			-	-	-	-	_	50	37.6		
500				-	-	~		-	75	56.0		
600		-			-		÷		100	57.2	*	
600c		L			-	-						
TEMP	ERATURE 2	2200°F	p		<u></u>							
4	66.6	*	*	*	*	*	*	*	1	*		
16	64.4			_	-		-	-	2	-		
64	59.2		-	-	-	-	-	-	4	_		
100	59.3	-	-	-	-	-	-	-	8	-		
200	58.3		-	-	-	_	-	-	16	-		
300	*	-	-	_	-	-	-	-	30	-		
400	-	-	-			-	-	-	50	-		
500	-	-	_	-	-	-	-	_	75	-		
600	-	-	-	-	-	-		-	100	-	*	
600c	-	-	-	-	-	-						

TABLE A1-1 NUMERICAL DATA TABULATION: ALLOY 1, N 155.

â



TABLE A1-1 NUMERICAL DATA TABULATION: ALLOY 1, N 155. (Continued)

		<u>en ander opper</u> te de la constant de la c	ainstanoiteinteinen non seuren se		CYCLIC	OXIDATIO	Ň	Карад Каланда (колонала) <b>26</b> 4		7	THERMOGRAVIMETRIC ANALYSIS
Z.PO.OST.	HOURS TINE SPECIFIC T	The Ling all all	Me Land Drall	10-3 ETER WIRE	10-201 00 04	10-5741 0410E	LTINGTE	PERCENCERT	1.0 - 11.01	SPECIFIC LINE	atry William
<u>⊢</u>	В	C C	D	Е	F	G		/ I	J	ĸ	f I
TEMPERA	L	ן איד					<b></b>	<u> </u>	- -		······
4	0.2	0.0	0.0	0.0	0.0	87.2	150.5	20.8	1	0.05	
16	0.2	0.0	0.0	0.0	0.0	118.3	148.2	13.7	2	0.06	
64	1.3	0.0	0.0	0.0	0.0	104.5	140.2	14.3	4	0.08	
100	0.5	0.0	0.0	0.02	0.0	108.0	133.6	8.8	8	0.09	
200	0.5	0.0	0.0	0.02	0.0	107.8	137.4	11.8	16	0.11	
300	0.7	0.0	0.0	0.02	0.0	105.9	147.5	16.3	30	0.11	
400	0.9	0.0	0.1	0.04	0.0	115.1	147.1	19.7	50	0.11	
500	0.9	0.0	0.1	0.04	0.12	115.0	144.5	14.0	75	0.15	
600	0.9	0.0	0.1	0.10	0.10	105.8	148.3	21.3	100	0.15	0.0
600c	0.9	0.0				95.3	143.0	19.3			
TEMPERA	TURE 160	0°F		_		g=======					
4	0.4	0.0	0.0	0.02	0.0	105.2	143.3	19.8	1	0.04	
16	1.0	0.1	0.0	0.04	0.08	106.0	143.0	16.3	2	0.06	
64	2.0	0.1	0.1	0.06	0.06	111.5	143.6	12.3	4	0.63	
100	2.0	0.1	0.1	0.08	0.08	121.5	147.2	17.2	8	0.14	
200	2.3	0.1	0.1	0.08	0.16	109.8	136.1	11.7	16	0.20	
300	2.4	0.2	0.1	0.12	0.12	84.1	137.3	14.5	.30	0.28	
400	3.0	0.1	0.1	0.12	0.12	111.1	141.4	15.5	50	0.36	
500	3.3	0.2	0.1	0.12	0.30	111.1	132.2	12.5	75	0.45	
600	3.4	0.2	0.2	0.14	0.12	109.8	138.7	13.7	100	0.53	0.10
<u>600c</u>	3.5	0.3	L	Ļ		101.5	135.0	14.8	L	L	
TEMPERA	TURE 180	0°F	1	r	n	r	<del></del>	·····			
4	1.3	*	0.0	0.0	0.04	116.4	149.5	19.3	1	0.18	
16	1.9		0.1	0.0	0.07	107.9	140.2	11.2	2	0.29	
64	2.9		0.2	0.08	0.04	123.2	138.5	10.5	4	0.49	
100	2.7		0.2	0.08	0.04	110.0	135.0	9.0	8	0.71	
200	2.9		0.2	0.08	0.02	105.7	137.4	11.5	16	1.09	
	3.7		0.3	0.20	0.05	103.1	135.0	9.5	30	1.59	
400	3.3	-	0.3	0.20	0.07	97.8	124.4	5.5	50	1.98	1
500	3.7		0.3	0.08	0.09	103.1	124.4	4.0	75	2.33	
600	3.8		0.3	0.20	0.09	103.1	118.0	4.8	100	2.73	0.20
600c	4.1	-				100.5	124.4	6.0			

### TABLE A1-2 NUMERICAL DATA TABULATION: ALLOY 3, TD NICKEL-CHROMIUM.

-74

ť



## TABLE A1-2 NUMERICAL DATA TABULATION: ALLOY 3, TD NICKEL-CHROMIUM. (Continued)

		<u> </u>		<u></u>	CYCLIC	OXIDATIO	N	in the state of the second		7	THERMOGRAVIMETRIC ANALYSIS	7
	OUR TINE	7 CALIN TAL	TIN SEAL	Ling Wiles	10 00 00 00 00 00 00 00 00 00 00 00 00 0	The Party of The	Pot Wein	Por Trencin	in the second	The The	111 WELGHT	7
	H COLEM	T CHI	Sal La	TOT IN THE	107 1217 1217	107 107			0.7	OF IJAG	CHARTER CONTROL	
A	В	С	Ð	E	F	G	н	I	J	K	L	
TEMPER	ATURE 20	00°F										
4	2.1	*	0.1	0.08	0.08	116.2	145.2	11.4	1	0.75		
16	2.4	-	0.1	0.08	0.08	106.2	128.5	3.7	2	1.00		
64	3.9	-	0.2	0.12	0.08	95.4	121.3	2.8	4	1.25		
100	2.7	<u> </u>	0.2	0.12	0.04	100.5	131.2	12.0	8	1.38		
200	3.6		0.2	0.06	0.0	92.6	118.9	10.0	16	1.55		
300	4.1	-	0.2	0.08	0.0	103.1	130.5	7.9	30	1.66	<u></u>	
400	3.6		0.3	0.08	0.0	84.5	113.8	9.7	50	1.74		
500	9.0	-	0.3	0.08	0.0	95.3	116.2	4.5	75	1.63		
600	-		0.3	0.08	0.0	82.2	108.3	5.0	100	1.37	0.30	
600 c	3.8	-		L		95.3	108.3	2.3				
TEMPER	ATURE 21	00°F				· · · · · ·				r		
4	2.4	*	0.1	0.12	0.12	105.8	118.9	3.3	1	0.76		
16	3.1	-	0.2	0.14	0.12	100.5	109.0	1.5	2	1.03	<u> </u>	
64	3.1		0.2	0.14	0.12	100.5	122.5	5.5	4	1.23		
100	3.0	-	0.2	0.06	0.04	100.5	115.2	2.7	8	1.65		
200	3.8	-	0.2	0.12	0.04	103.1	124.4	6.0	16	1.77		
300	3.6	-	0.3	0.24	0.08	97.8	113.8	2.0	30	1.46		
400	4.0	-	0.4	0.14	0.12	97.8	116.8	2.0	50	1.24	+	
500	4.4	-	0.4	0.10	0.08	87.2	96.3	2.5	75	0.92	+	
600	9.2		0.3	0.10	0.06	95.3	113.8	5.0	100	0.31	0.30	
600 c	7.4			L;	Ļ:	105.8	127.0	3.3			1	
LEMPER		JO-F	[									
16	5./	*	0.2	0.12	0.08	107.2	129.8	8.3	1	0.41		
10	0.3		0.4	0.30	0.08	99.0	113.8	3.2	2	0.50	+	
100	12 5		0.5	0.40	0.08	75.5	87.2	5.5	4	0.59	+	
200	20.6		0.6	0.40	0.08	97.8	105.8	3.5	8	0.64	<b>--</b>	
300	29.0		0.6	0.40	0.10	*	*	*	16	0.59	<u> </u>	
400	42.2		0.6	0.28	0.04			-	30	0.29	+	
500	33 5		0.6	0.28	0.04	·-		-	50	0.27	+	
600	*		0.5	0.28	0.02		-	-	/5	-0.53		
600° c			*	*		-		;- 	100	-1.20	+ <u>*</u>	
000 0						_	<u></u>					

### TABLE A1-2 NUMERICAL DATA TABULATION: ALLOY 3, TD NICKEL-CHROMIUM.

÷ŧ.

	$\square$	THERMO	GRAVIMET	RIC ANAL	YSIS	STRE	SS RUPTU	RE	<u></u>	STRESS	OXIDATI	ON	
INTECONECC	10-30 02 02 20-3 02 02 20-10-10-10-10-10-10-10-10-10-10-10-10-10	To 3 ran arise	HIJHT Fod	Hichitar Co	1.0217401	STR.	103 0555 Cature 141	10-3 PERMITE	20-20-20 02 22771 - 20-20 22771 - 20-20	10-3 64 1701 18	II Thurst Co.	Partic Cot	1.0 - 11.0
M	N	0	P	Q	R	S	T	U	V	W	X	Y	
TEMPERA	TURE 200	0°F	1						<u> </u>		Γ.	· · ·	
	1				0.10	4.7	0.40	0.20	*	*	*	*	
					<u>80.34</u>	4.3	1	···· · ·		····			
	1				171+	4.4	1					<u> </u>	
		L											
	<u> </u>									·		[	
0.12	0.10	114.0	140.0	6.6	<u> </u>							· · · · · · · · · · · · · · · · · · ·	
TEMPER		1	1				1	<b>i</b>				·]	
I CMP EKA			1		<b></b>		*	*	*	*	*	•	
	1	1					1						
							1						
			:						:				
··			<u> </u>				<u> </u>						
		<u> </u>			<u> </u>								
0.20	0.20	96.0	122.0	3.5			1	· · · ·					
TEMPER	L ATURE 220	1 )0°F	1	<u>I</u>	<u>I.,</u>	I	1	<b>I</b>	1	L	L	L	
			1		T		*	*	*	*	*	*	
	1		1		1	1			1				
		<b>_</b>	<b> </b>		1								
		ļ			<u> </u>		ļ						
	1	<b> </b>			ļ		<b>.</b>				ļ		
*	*	*	*	*	<u> </u>		<b>.</b>						
				I		1						1	

# TABLE A1-2 NUMERICAL DATA TABULATION: ALLOY 3, TD NICKEL-CHROMIUM. (Continued)

	$\square$			and the second	CYCLIC	OXIDATIC	)N			7	THERMOGRA ANALY	VIMETRIC /
ETD OG	HOURS TIME	SPECIFIC CALINAL	The Line of the Li	10-3 WILLER WILLE	10-3 0410E	10-374 OKIDE I.E.D. ON	PST PST CIA	PERCONCELL	1.0 211401	PPECIFIC TIME	Cally WE ICHT	10.3000
-	<u>/</u>	<u> </u>	$\frac{1}{1}$	<u> </u>	<u> </u>	<u> </u>		<u> </u>	<u> </u>	<u> </u>	/	/
A		0097	D	L E	F	L G	<u> </u>			K		
1 EFIPER	ATURE 14			0.04		44.2	112 1	24.7	<u>г</u> ,	0.02	T	
16	0.4	0.0	0.0	0.04	0.0	44.2	113 1	24.7	2	0.05		
64	0.6	0.0	0.0	0.08	0.06	44.2	116.1	28.5	4	0.12		
100	0.7	0.0	0.0	0.4	0.26	46.4	111.5	23.2	8	0.15		
200	0.7	0.0	0.0	0.08	0.10	42.6	110.5	23.7	16	0.17		
300	0.7	0.0	0.0	0.04	0.16	43.1	114.9	27.5	30	0.20		
400	0.9	0.0	0.0	0.08	0.12	42.0	99.5	17.8	50	0.23		
500	1.1	0.0	0.0	0.12	0.28	52,0	112.1	15.3	75	0.27		
600	0.9	0.0	0.0	0.12	0.28	45.2	101.0	17.5	100	0.30	0.0	
600c	1.0	0.0				44.7	108.2	21.5				
TEMPER	ATURE 16	00°F										
44	0.8	0.0	0.0	0.10	0.10	47.0	121.7	29.0	1	0.20		
16	1.1	0.0	0.0	0.08	0.10	48.2	116.1	26.5	2	0.35		
64	1.4	0.1	0.0	0.08	0.16	44.7	103.2	20.7	4	0.45		
100	2.0	0.1	0.0	0.14	0.12	44.2	104.8	21.4	. 8	0.49	1	
200	2.5	0.1	0.0	0.14	0.36	42.6	74.5	8.0	16	0.66		
300	3.5	0.4	0.0	0.16	0.40	38.7	56.3	4.5	30	0.87		
400	4.1	0.9	0.0	0.20	0.40	35.9	46.4	1.5	50	1.07		
500	4.4	1.1	0,0	0,20	0,60	35.9	35.9	0.7	75	1.28	·	
600	5.6	2.6	0.0	0,24	0.80	34.8	39.8	1.0	100	1.43	0.20	0
600c	4.7	1.4	L	<u> </u>	l	35.9	46.4	1_1.5			<u>}</u>	
TEMPER	ATURE 18	00°F			0.10						1	
44	1.9	*	0.0	0.10	0.12	44.2	107.5	21.5	1	0.62		
	2.9		0.1	0.24	0.36	39.2	71.9	9.3	2	1.04		
100	5.0		0.1	0.40	0.10	32.0	34.3	0.7	4	1.48		
100	7.2		0.1	0.40	1.0	20.0	26.0	0.5	8	2.03		
200	12.3			0.40	1.0	*	*	*	16	2.77	+	
300	14 =		0.0	0.40	*				30	3.79		
500	17.2		0.2	0.60	-	-			50	4.95		
600	17.6		0.2	0.60			1		100	0.18	0.00	
600c	16.9	_	0.2	0.00		_	-		100	0.10	0.30	

TABLE A	A1-3	NUMERICAL	DATA	TABULATION:	ALLOY	6,	DH	242.
						-		

	/	THERMOO	GRAVIMETI	RIC ANAL	YSIS	STRE	SS RUPTU	re/		STRESS	OXIDATIO	ON
THICKNESS C	DEPTH DE OF OF	10-3 CAT 2012	INTINUE De L'UCTH	PSA TENCTH	1. William	anti Sunor	111 1117 180 507 1807 1017	10-3 ETER WIRE	DEPTH OF OF	10-3 4411410 10-3 4411010 10-3 411010 10-5 500	LITHURS COL	Per Contract
М	N	0	P	Q	R	S	Т	U	V	W	X	Y
TEMPERA	ATURE 14	00°F	<u> </u>		· · · · ·			· · · · ·	r	<u>, i na ku in i</u>		
					2.70	31.6	0.0	0.04	0.04	55.0	110.0	19.0
					3,90	30.4				· · · · · · · · · · · · · · · · · · ·		
				••••••••	4.25	29.2						
					14.10	24.3						
		· · · · · · · · · · · · · · · · · · ·			18.25	23.1						
					50.48	18.9						
					64.30	18.3						
					89.10	16.4						
0.02	0.20	75.0	112.0	12.8								
		600°=			· · · · · · · · · · · · · · · · · · ·				1		l	
TEMPE	KATURE 1	000 F			0.76	14.4	0.10	0.00	0.60	50.0	06.0	1/ 1
					0.76	16.4	0.10	0.06	0.60	50.0	90.0	14•1
					2.60	12.0						
					20.60	12.2						
					51 00	6.0	· · ·			· · · .		
				, , ,	57 25	6.0						
					1424	6.8						·····
					1014	6.6	· · ·					ł
0.04	0.24	64.0	99.0	12.8	+247					· · · · · · · · · · · · · · · · · · ·		
	¥147	<u>, , , , , , , , , , , , , , , , , , , </u>										
TEMPE	RATURE 1	.800°F										
					5.00	4.9	0.30	0.40	0.70	*	*	*
					5.70	3.0						
					13.48	3.7						
					24.70	2.4						
					37.70	1.8						
					140+	1.6						
0.20	0.80	*	*	*								

## TABLE A1-3 NUMERICAL DATA TABULATION: ALLOY 6, DH 242. (Continued)

					CYCLIC (	DXIDATIO	N			1	THERMOGRA ANALÝ:	VIMETRIC SIS
T.POOSLIT.	HOURS TIME	SECTAL CALVAL	121 - 121 -	10-3 ETER WIRE	10-30 02 02 DEPTH 05 0	10-3 Ray WIDE TELD Star 10W	LTINGTE Des WCTH	PERCENCENT	1.0 - 11-01 2.10-11-01 2.10-01/2-	SPECIFIC LINE	CALIN PEICHT CALINE CAL	ALL REAL CON
<u> </u>	/ 	$\frac{1}{c}$		/ 	F	с с	/ ~ 	(		K K	$\int \mathbf{T}$	/
TEMDED	ATTURE 200	0°₽								<u> </u>	1	
4	3 4	*	0.1	0.20	0.60	41 5	85.2	15.0	1	1 66		
16	9.4		0.1	0.20	0.00	23.8	22.8	10.0	2	2 15		
64	15 4		0.2	0.40	*	*	*			2.11		
100	23.1		0.6	0.60	_	_			8	4 70		
200	43.2		1.4	1,50		4	-	_	16	7.62		
300	57.8		*	*				-	30	10.2		
400			-	_	_	_	. <u></u>		50	12.6		
500	54.8		-		_		_		75	15 1		
600	59.0	_	_	_	_	-	_		100	17.2	0.5	
600c	59.2		_	-			_	_	100	1.1.2		
TEMPER	ATURE 210	00°F			L <u></u>		•		· · · · · · · · · · · · · · · · · · ·	A		
4	4.4	*	0.0	0.08	0.24	42.6	59.6	4.3	1	*	1	
16	9.6	-	0.4	0.36	0.80	35.9	37.0	0.8	2	-		
64	35.4		1.0	1.50	*	*	*	*	4	_		
100	50.7	-	*	*	_	_	<u> </u>	-	8	_		
200	57.3	-		_	_	_	_		16	_		
300	*	-	_		_	_		_	30	-		
400	_	-		_		-	-		50	_		
500	_	_	_	-	_	-	-	-	75	-		
600	-	-	-	_	_	-	_	-	100	_	*	
600c	-	-	_		_		-	_				
TEMPER	ATURE 220	00°F										
4	9.3	*	0.2	0.60	0.60	*	*	*	1	*		
16	38.1	-	1.0	1.50	*	-	_	_	2	_		
64	60.0	-	*	*	-	_	_	_	4	_		
100	60.5	-	-	_	-			-	8			
200	58.4	-	_	_		_	-	-	16	-		
300	*	-	-		-		-		30	-		
400	-	-	-	-	-	-	-	-	50	-		
500	-	-	-	-	-	-	-	-	75	-		
600	-	-	-	÷	-	-	-	-	100	-	*	
600c	-	-	-	-	_	-	-	-				

### TABLE A1-3 NUMERICAL DATA TABULATION: ALLOY 6, DH 242

						7				000000		
		THERM	JGRAVIMET	KIC ANAL	JYSIS		SS RUPTU			STRESS		
THOME	10-35 0210E	0-374 PLDE	103 STRENGTH	LOJ STRENCTH	1.0 1.1.1.0 W	HOURS TINE	103 055 103 055 101 131	7-3 miles Wites	0-30 02 02 211.02 212.05 213.05 210.05 20.05	7-374 110W	to3 offer	LOJ STRENGTH
1.5		~~/ <del>~</del>	Time 1				18 ~		~ ~ ~			
м	N	0	P	Q	R	S	Т	U	v	W	X	Y
TEMPERA	TURE 200	0°F			<u>,</u>							
	1	ļ			0.92	3.7	0.0	0.40	*	*	*	*
			<b>.</b>	L	2.00	3.0		· · · · · ·			ļ	
		<b>_</b>			4.20	2.4					ļ	
<u> </u>	ļ				7.60	1.8	ļ				<u> </u>	
				L	15.01	1.2					<u> </u>	
	ļ	ļ		<u> </u>	39.31	0.9					ļ	
					144+	0.6					ļ	
	ļ											
0.60	1.20	*	*	*		<u> </u>		ļ				
	<u> </u>				<u>I</u>	L					<u> </u>	
TEMPERA	ATURE 210	00°F 1	1	r	T	1	T	1	1	[·····	1	T
				<b>_</b>	1	<u> </u>	*	*	*	*	*	*
		<u> </u>			1		<b> </b>			<u> </u>	<u> </u>	
		<u> </u>					· · · · · · · · · · · · · · · · · · ·		· · · · · · · · · · · · · · · · · · ·			
				<u> </u>		<u> </u>			· · ·			
											<u> </u>	
and the states of			+	<u> </u>		<u> </u>	:					
· · · · · · · · · · · · · · · · · · ·								- · · · -				
		<u>                                      </u>	1.	<u> </u>				<u> </u>				
*	*	*	*	*	1						<u></u>	
me) men			1	L	1	<u> </u>	1			<u>.</u>	<u> </u>	<u>i</u>
I GPIF GRA	LUKE 220		-	<u> </u>	1	[	1			T	Ι.	Ι.
		<u>                                     </u>		<u> </u> `			×	*	*	*	*	*
••••••••••••••••••••••••••••••••••••••				· · · · · · · · · · · · · · · · · · ·			·			<u> </u>	<u> </u>	
L	+	<u> </u>	+	<u> </u>	1		1	<u> </u>		<u> </u>	+	<u> </u>
	ł		+			<b> </b>	<u> </u>				┢────	<u> </u>
		<u> </u>		<u> </u>				<del> </del> -		+		<u> </u>
	·	·					<u> </u>	+		<u> </u>		1
			+	<u> </u>		<u> </u>	<u> </u>			<u> </u>	+	+
	<u></u>		+	<u> </u>	<u> </u>			+	<u> </u>		<u> </u>	<u> </u>
*	*	*	*	*		<u> </u>	<u> </u>					<u> </u>
		1	1	<u>i</u>	1		1	1				

TABLE A1-3 NUMERICAL DATA TABULATION: ALLOY 6, DH 242. (Continued)

		THERMOGRAVIMETRIC										
TIDOOSIT	HOURS TIME	IPECIFIC COTAL	Territor In Contraction	10-3 ETER WIRE INTERNIESS	10-30 04 10-3 04102 11.102 11.102 11.102	10-3741 2412E	LITHITE COL	PS1 DS1	1.0 - 11-01 Eteosita	SPECIFIC TIME	10-12-12-12-12-12-12-12-12-12-12-12-12-12-	
$\bigwedge$	<u>/</u>	<u> </u>		/				/			(	
		00°E						<u> </u>			L	
LEPIPER 4	ATURE 14		0.0	0.02	0.0	45.9	106.0	25.0		0.00		
16	0.1	0.0	0.0	0.02	0.0	42.0 50.0	11/ 0	22.0	2	0.09		
<u> </u>	0.5	0.0	0.0	0.12	0.0	58.7	122 3	18 5	4	0.13		
100	0.6	0.0	0.0	0.10	0.10	61.2	127.5	19.7	8	0.14		
200	0.8	0.0	0.0	0.04	0.10	67.8	132.8	15.2	16	0.18		
300	1.1	0.0	0.0	0.08	0.20	65.8	130.0	15.5	30	0.24		
400	1.3	0.0	0.0	0.08	0.12	61.3	125.0	12.7	50	0.30		
500	1.7	0.0	0.0	0.14	0.10	61.4	113.2	10.7	75	0.37		
600	1.3	0.0	0.0	0.08	0.10	56.2	124.4	15.7	100	0.41	0.0	
600c	1.7	0.0				59.2	123.0	12.5				
TEMPERATURE 1600°F												
4	0.5	0.0	0.0	0.04	0.06	54.5	120.0	23.5	1	0.16		
16	1.1	0.0	0.0	0.04	0.14	61.2	122.3	17.3	2	0.22		
64	2.3	0.0	0.0	0.12	0.30	53.5	105.0	9.0	. 4	0.35		
100	2.7	0.1	0.0	0.14	0.60	54.1	94.5	8.3	8	0.45		
200	3.2	0.6	0.0	0.10	0.32	56.2	87.7	5.5	16	0.75		
300	4.1	2.0	0.0	0.16	0.60	53.5	74.0	3.2	30	1.02		
400	5.4	2.2	0.1	0.20	0.40	48.4	53.5	1.0	50	1.33		
500	2.6	3.2	0.1	0.20	0.40	51.0	69.4	2.2	75	1.66		
600	5.7	2.5	0.1	0.20	0.80	43.8	48.8	0.7	100	1.93	0.10	
<u>600c</u>	6.2	2.4	L			46.8	46.8	1.2				
TEMPER	RATURE 18	00°F	r	r	1	<u> </u>	r	r		r	·····	
4	1.5	*	0.0	0.08	0.20	48.4	108.6	15.8	1	0.33		
16	2.8		0.0	0.10	0.40	46.0	97.0	13.3	2	0.55		
64	4.8	-	0.1	0.16	0.40	48.4	68.8	5.0	4	0.87		
100	6.6		0.1	0.40	1.0	31.1	31.1	0.5	8	1.44		
200	8.4		0.2	0.40	1.0	24.5	24.5	0.5	16	2.05		
300	9.8		0.2	0.40	1.0	*	*	*	30	2.87		
400	11.8		0.2	0.50	*	-	-	-	50	3.68	<b></b>	
500	12.5		0.2	*	-			-	75	3.95		
600	14.0	-	0.2	-		-	-	-	100	4.18	0.30	
600c	13.9	-						-		1 ×		

## TABLE A1-4 NUMERICAL DATA TABULATION: ALLOY 10, HASTELLOY X.

×

-1



## TABLE A1-4 NUMERICAL DATA TABULATION: ALLOY 10, HASTELLOY X. (Continued)

	$\square$		THERMOGRAVIMETR ANALYSIS										
Et Oliv	HOURS LIVE SPECIFIC T	PECTAZAIN SPECTAZAIN MATIC OF	The Line of the Li	10-3 ETA VIAE	10-20 02 02 10-3 02102 11. 11. 12 12. 11. 02 12. 11. 12 12. 12 12. 12 12. 12 12 12 12 12 12 12 12 12 12 12 12 12 1	LO-STANTON LIEL CONTRACTION	LITHER PST WCTH	Part Part Part Part Part Part Part Part	1.0 - 1.01 2.00012 2.00012	SPECIFIC LINE	101 101 101 101 101 101 101 101 101 101		
	/	(		F		/	/ <sup>5</sup>	/		K K			
			, P			L~	L	L -	<u> </u>		<u> </u>		
	3.5	*	0.1	0.12	0.50	43.3	81.7	1.4	1	1.22			
16	6.6	-	0.2	0.20	0.80	38.3	43.3	1.0	2	2.11			
64	12.3	_	0.2	0.40	1.00	*	*	*	4	3.43			
100	15.4	-	0.3	0.50	*	-	_	_	8	5.21			
200	43.2		0.6	1.00		_	_	_	16	8.39			
300	52.6		*	*	-	-	_	-	30	10.9			
400	47.6			-	-	_	-	·	50	14.8			
500	47.7	-		_	_	_			75	22.8			
600	42.3	-				-			100	31.6	0.60		
600c	39.7	L						_					
TEMPER	TEMPERATURE 2100°F												
4	5.8	*	0.2	0.20	0.28	74.0	108.6	14.8	1	*			
16	7.6	<u> </u>	0.2	0.20	0.60	61.3	81.7	9.0	2				
64	18.4	<u> </u>	0.2	0.60	1.20	*	*	*	4	-			
100	47.8	-	*	*	*			-	8	-			
200	43.8			-	-	-		-	16				
300	*		-		-		÷	-	30	-			
400	_		-	_		-			50	-			
500	<u> </u>		-						75	` <del>_</del>			
600				-				-	100	:-	*		
600c	-	L		<u> </u>	L	<u> </u>	<u> </u>	-	<b></b>				
TEMPER	ATURE 22	00°F		<u> </u>		1	T		T				
4	10.0	*	0.5	0.40	0.40	51.0	58.7	1.5	1	*			
16	33.3		1.4	1.00	1.00	*	*	*	2				
64	39.6		+ *	*	*		<u>↓ -</u>	<b> </b> -	4				
100	39.3								8	-			
200	40.0			-				-	16	-			
300	*	<u> </u>					<u> </u>	<u>                                     </u>	30	:+ 			
400					-		-	-	50	-			
500		-		-		-	+	1	75				
600					-				100		*		
600c	-	-	1			. +		-					

TABLE A1-4 NUMERICAL DATA TABULATION: ALLOY 10, HASTELLOY X.

-9

بہ

		THERMO	OGRAVIMET	RIC ANAL	YSIS	STRI	SS RUPTU	JRE	STRESS OXIDATION						
Introduced	10.3 02100 10.3 02100 12.0 02 10.0 02	10-3740 PKIDE	INTINATE COS	Peronear Press	1.0 2114 LON	BILL SHID	203 655 Chuller DT., Th	10-3 MIR WIRE	10-3 02102 DEPT 01.02	10-3 RATION 11. 10W	ULTINGTS OF CIT	Part Contraction			
М	N	0	Р	Q	R	S	T	U	V	W	x	Y			
TEMPERA	TURE 200	0°F	T	r	r	r	<b>r</b>	<u>.</u>	r						
<u>.</u>	<u> </u>		<u> </u>		3.55	3.4	0.50	0.40	1.00	*	*	*			
					12.50	2.8									
<u>k</u>					69.47	2.6		·							
••••••••				<b>†</b>	100+	2.7			· · · · · · · · · · · · · · · · · · ·						
			1	<u>†</u>			l.				1				
				L											
1.00	*	*	*	*				<u> </u>	<u> </u>		<u> -,-,-</u>				
		<u> </u>	<u> </u>		I				I		<u> </u>	<u> </u>			
TEMPERA	TURE 210	I I	I	<u>r</u>	J						Γ.				
	<u> </u>	-	1	<u> </u>			<u> </u> <sup>*</sup> −−	<u> </u>	*		*	*			
			1						·····						
			ļ								L				
	ļ	ļ	<u> </u>	ļ			ļ		<u> </u>		ļ				
	<u> </u>	<u> </u>	<u> </u>	<u> </u>						· · ·	<u> </u>				
*	*	*		*		·			· · ·		+				
TEMPERA	TURE 220	0°F	1	<b>.</b>	1	L	L		1	l					
							*	*	*	*	*	*			
÷.	<u> </u>	ļ	1	 				ļ	ļ		<u> </u>				
		ļ		<u> </u>		·····					L				
	<u> </u>	<b> </b>		<u> </u>	· · · · · · · · ·		<b> </b>				<u> </u>	ļ			
	<u> </u>	· · · ·	<u> </u>	<u> </u>	<b> </b>			<u> </u>			<u> </u>	<b></b>			
	<u> </u>	<u> </u>	1.		<u> </u>						<u> </u>	<b>  </b>			
*	<u>  *</u>	*	*	<u>    *                                </u>	ļ	<b>├</b> ──	ļ	ļ	<u> </u>	ļ	J	1			

•

u.

# TABLE A1-4 NUMERICAL DATA TABULATION: ALLOY 10, HASTELLOY X. (Continued)

i.

ъ.

### APPENDIX 2 ALLOY OXIDATION PLOTS

#### CONTENTS

### SPECIFIC TOTAL OXIDATION WEIGHT GAIN

ALLO	Y	FIGURE PAG	PAGE			
1.	N 155	A2-1	3			
3.	TD nickel-chromium	• • A2-2 • • • • • • • • • • • • • • • • • 59	•			
6.	DH 242	A2-3	)			
10.	Hastelloy X	A2-4	•			

Specific total oxidation weight gain is plotted for each alloy as a function of exposure time with temperature as a parameter. Power function plots on a log-log grid were essentially linear for most data.

#### WIRE AND SHEET OXIDATION RESISTANCE COMPARISON

ALLO	FIGURE												PAGE									
1.	N 155	.0	.•	•	•	.A2-5		•		•	•	•	•	•		•	•	•		.0	•	62
3.	TD nickel-chromium			•	•	.A2-6		•	٠	•			•			٠	•	•	•	•	•	63
6.	DH 242		•		٠	.A2-7	•			•			•	•			•			•		64
10.	Hastelloy X	.0		•		.A2-8			•	•			•		٠		•	٠	•			65

Faired power function plots of total specific weight gain as a function of exposure time are shown for wire (solid line) and sheet (dashed line) specimens of each alloy with temperature as a parameter.

### CONTINUOUS (TGA) SPECIFIC OXIDATION WEIGHT GAIN

ALLO	Y					FIGURE														<u>]</u>	PAGE
1.	N 155	•	•		•	.A2-9 .	•	•	,•	•	•	•	•	•	•	•	.•	.•	٠	•	66
3.	TD nickel-chromium	•	•	•	٠	.A2-10.	•	•		•	•		•	٠	•	•	•			.0	67
6.	DH 242	•	.•			.A2-11.	٠	•	•	•	•	•	٠	٠	•	•	•	•	•	•	68
10.	Hastelloy X	•	•	•	•	.A1-12.	•	•	٠	•	٠	•	•	•	٠		•	•	•	•	69

Continuous specific oxidation weight gain is shown as a function of steady-state exposure time for each alloy with thermogravimetric analysis (TGA) testing temperature as a parameter.



Figure A2-1 Specific total oxidation weight gain: Alloy 1, N 155 wire. 58



Figure A2-2 Specific total oxidation weight gain: Alloy 3, TD nickel chromium wire.



Figure A2-3 Specific total oxidation weight gain: Alloy 6, DH 242 wire.



Figure A2-4 Specific total oxidation weight gain: Alloy 10, Hastelloy X wire.



Figure A2-5 Wire and sheet oxidation resistance comparison: Alloy 1, N 155.



Figure A2-6 Wire and sheet oxidation resistance comparison: Alloy 3, TD nickel-chromium.


Figure A2-7 Wire and sheet oxidation resistance comparison: Alloy 6, DH 242.



Figure A2-8 Wire and sheet oxidation resistance comparison: Alloy 10, Hastelloy X.



Figure A2-9 Continuous (TGA) specific oxidation weight gain: Alloy 1, N 155.



Figure A2-10 Continuous (TGA) specific oxidation weight gain: Alloy 3, TD nickel-chromium.



Figure A2-11 Continuous (TGA) specific oxidation weight gain: Alloy 6, DH 242.



Figure A2-12 Continuous (TGA) specific oxidation weight gain: Alloy 10, Hastelloy X.

сê

## PRECEDING, PAGE BLANK NOT FILMED.

.

## APPENDIX 3 METALLOGRAPHIC EXAMINATION

## CONTENTS

ALLOY				FIGURE										PAGE
1.	N 155	•	•	A3-1	•	• •	 •	•	•	•	•	•	• .	72
3.	TD nickel-chromium .			A3-2	•			•	•	•	•	•	•	78
6.	DH 242	•		A3-3	٠		 ٠	•	•	•	•	.•	•	86
10.	Hastelloy X	•	•	A3-4	•			•		•	•		•	93

Photomicrographs of each alloy are reproduced to demonstrate morphology changes and oxidation effects resulting from each test series as indicated:

- 1. As received microstructure.
- 2. Sintered microstructure.
- 3. Continuous (TGA) oxidation tests showing oxidation effects at each test temperature.
- 4. Stress-oxidation showing oxidation effects at each test temperature.
- 5. Cyclic oxidation tests showing oxidation progression with increasing time at each temperature.



Figure A3-1.1 Alloy 1, N 155, As Received. 285X



Figure A3-1.2 Alloy 1, N 155, As Sintered. 285X



(1) 1400°F.



(2) 1600°F.



(3) 1800°F.



(4) 2000°F.

Figure A3-1.3 Alloy 1, N 155, Continuous Oxidation 100 Hours. 285X





(3) 1800°F.



(4) 2000°F.

Figure A3-1.4 Alloy 1, N 155, Stress Oxidation 100 Hours. 285X





(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



(7) 400 Hours

(8) 500 Hours

(9) 600 Hours

Figure A3-1.5 Alloy 1, N 155, Cyclic Oxidation at 1400°F. 285X





(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



- (7) 400 Hours
- (8) 500 Hours
- (9) 600 Hours

Figure A3-1.5 Alloy 1, N 155, Cyclic Oxidation at 1600°F. 285X





(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



(7) 400 Hours

(8) 500 Hours

(9) 600 Hours

ł

Figure A3-1.5 Alloy 1, N 155, Cyclic Oxidation at 1800°F. 285X



(1) 4 Hours









Figure A3-1.5 Alloy 1, N 155, Cyclic Oxidation at 2000°F. 285X



## (1) 4 Hours

Figure A3-1.5 Alloy 1, N 155, Cyclic Oxidation at 2100°F. 285X



Figure A3-2.1 Alloy 3, TD Nickel-Chromium, As Received. 285X



Figure A3-2.2 Alloy 3, TD Nickel-Chromium, As Sintered. 285X



(1) 1600°F



(2) 1800°F



(3) 2000°F











(3) 1800°F



(4) 2000°F

Figure A3-2.4 Alloy 3, TD Nickel-Chromium, Stress Oxidation 100 Hours. 285X



Figure A3-2.5 Alloy 3, TD Nickel-Chromium, Cyclic Oxidation at 1400°F. 285X



(1) 4 Hours



(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



- (7) 400 Hours
- (8) 500 Hours

(9) 600 Hours

Figure A3-2.5 Alloy 3, TD Nickel-Chromium, Cyclic Oxidation at 1600°F. 285X



(1) 4 Hours



(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



(7) 400 Hours



(8) 500 Hours



(9) 600 Hours

Figure A3-2.5 Alloy 3, TD Nickel-Chromium, Cyclic Oxidation at 1800°F. 285X



(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



- (7) 400 Hours
- 500 Hours (8)

(9) 600 Hours

Figure A3-2.5 Alloy 3, TD Nickel-Chromium, Cyclic Oxidation at 2000°F. 285X



(1) 4 Hours



(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



(7) 400 Hours



(8) 500 Hours



(9) 600 Hours

Figure A3-2.5 Alloy 3, TD Nickel-Chromium, Cyclic Oxidation at 2100°F. 285X



(1) 4 Hours



(2) 16 Hours



- (6) 300 Hours
- (7) 400 Hours



Figure A3-2.5 Alloy 3, TD Nickel-Chromium, Cyclic Oxidation at 2200°F. 285X



Figure A3-3.1 Alloy 6, DH 242, As Received. 285X



Figure A3-3.2 Alloy 6, DH 242, As Sintered. 285X



(1) 1400°F



(2) 1600°F



(3) 1800°F



(4) 2000°F

Figure A3-3.3 Alloy 6, DH 242, Continuous Osidation 100 Hours. 285X





(3) 1800°F



(4) 2000°F

• Figure A3-3.4 Alloy 6, DH 242, Stress Oxidation 100 Hours. 285X





(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



(7) 400 Hours



(8) 500 Hours



(9) 600 Hours

Figure A3-3.5 Alloy 6, DH 242, Cyclic Oxidation at 1400°F. 285X



(1) 4 Hours



(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



200 Hours

(5)



(6) 300 Hours



- (7) 400 Hours
- (8) 500 Hours
- (9) 600 Hours

Figure A3-3.5 Alloy 6, DH 242, Cyclic Oxidation at 1600°F. 285X





(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



(7) 400 Hours



(8) 500 Hours



(9) 600 Hours

Figure A3-3.5 Alloy 6, DH 242, Cyclic Oxidation at 1800°F. 285X



(1) 4 Hours



(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours

Figure A3-3.5 Alloy 6, DH 242, Cyclic Oxidation at 2000°F. 285X



 (1) 4 Hours
 (2) 16 Hours
 (3) 64 Hours

 Figure A3-3.5
 Alloy 6, DH 242, Cyclic Oxidation at 2100°F. 285X





(1) 4 Hours (2) 16 Hours (3) 64 Hours Figure A3-3.5 Alloy 6, DH 242, Cyclic Oxidation at 2200°F. 285X



Figure A3-4.1 Alloy 10, Hastelloy X, As Received. 285X



Figure A3-4.2 Alloy 10, Hastelloy X, As Sintered. 285X



(1) 1400°F.



(2) 1600°F.







(4) 2000°F.







1800°F. (3)



(4) 2000°F.





(7) 400 Hours

(8) 500 Hours

(9) 600 Hours

Figure A3-4.5 Alloy 10, Hastelloy X, Cyclic Oxidation at 1400°F. 285X





(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



300 Hours (6)



(9) 600 Hours

Figure A3-4.5 Alloy 10, Hastelloy X, Cyclic Oxidation at 1600°F. 285X



(1) 4 Hours



(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours



(6) 300 Hours



(7) 400 Hours

(8) 500 Hours

(9) 600 Hours

Figure A3-4.5 Alloy 10, Hastelloy X, Cyclic Oxidation at 1800°F. 285X



(1) 4 Hours



(2) 16 Hours



(3) 64 Hours



(4) 100 Hours



(5) 200 Hours







(2) 16 Hours



(3) 64 Hours

Figure A3-4.3 Alloy 10, Hastelloy X, Cyclic Oxidation at 2100°F. 285X



(1) 4 Hours

(2) 16 Hours

Figure A3-4.5 Alloy 10, Hastelloy X, Cyclic Oxidation at 2200°F. 285X
\*

# PRECEDING PAGE BLANK NOT FILMED.

۷

## APPENDIX 4 MECHANICAL PROPERTIES

#### CONTENTS

#### WIRE AND SHEET TENSILE TEST DATA COMPARISON

ALLOY				1	FIGURE														PAGE
1.	N 155		.•	•	A4-1 .	.•	•	•	•	•		•	•			•			. 102
3.	TD nickel-chromium	•	•	÷	A4-2 .	,				•	•	•	•	:.		•	•	•	. 102
6.	DH 242		•		A4-3 .		•	•	•		•	÷	•	•	•	•			• 103
10.	Hastelloy X	•	.•		A4-4 .				•	•				•	•		•	•	. 103

Wire (solid line) and sheet (dashed line) specimen tensile test data from cyclic oxidation test samples are compared at 100 and 600 hours for each alloy as a function of temperature, showing yield strength, ultimate strength, and percentage elongation.

#### OXIDATION EXPOSURE TENSILE TEST DATA COMPARISON

FIGURE																			PAGE
A4-5 .	•		•	•	•		 ٠	.•					•		•	•	•	•	104

Tensile test data from each test series of cyclic oxidation (CYC), thermogravimetric analysis (TGA), and stress-oxidation (S-O) is shown for each alloy at 100 hours exposure time as a function of test temperature.

#### STRESS RUPTURE LIFE

ALLOY	FIGURE	PAGE
1.	N 155 A4-6	105
3.	TD nickel-chromium A4-7	106
6.	DH 242	107
10.	Hastelloy X	108

Stress rupture lives for each alloy are shown with stress as a power function of exposure time with temperature as a parameter.



Figure A4-1 Wire and sheet tensile test data comparison at room temperature: Alloy 1, N 155.



Figure A4-2 Wire and sheet tensile test data comparison at room temperature: Alloy 3, TD nickel-chromium.



Figure A4-3 Wire and sheet tensile test data comparison at room temperature: Alloy 6, DH 242.



Figure A4-4 Wire and sheet tensile test data comparison at room temperature: Alloy 10, Hastelloy X.





a,

-19



Figure A4-6 Stress rupture life: Alloy 1, N 155.



Figure A4-7 Stress rupture life: Alloy 3, TD nickel-chromium.



Figure A4-8 Stress rupture life: Alloy 6, DH 242.



Figure A4-9 Stress rupture life: Alloy 10, Hastelloy X.

## APPENDIX 5

## OXIDATION PENETRATION PLOTS

#### CONTENTS

ALLO	Y		]	FIGURE														PAGE
1.	N 155			A5-1 .	•	•	•	•	.•			•	•			•	•	110
3.	TD nickel-chromium .	 •	•	A5-2.	.•	•		•	•	٠	•			•	•			112
6.	DH 242		•	A5-3.	.•	•		•	•		•			٠	•	•	•	114
10.	Hastelloy X			A5-4 .					•	•			•	•			•	116

Wire specimen thickness change and oxide penetration depth are shown in relationship to the original wire surface, measured on its radius, as a function of exposure time for each alloy and temperature. Progressive increase in thickness (upper solid line) indicates specimen oxidation and growth. Decrease in thickness shows oxide spalling. Oxide penetration (lower solid line) below the final surface represents uniform oxide layer growth plus oxide penetration into the metal.

Oxide penetration may or may not extend below the initial surface depending on the relative thickness change and surface oxidation rates. Corresponding curves are shown for equivalent sheet specimens (dashed lines) to show wire and sheet specimen comparative oxidation characteristics. Oxidation and penetration curves shown for the higher temperatures and longer times may be sharply divergent, indicating that oxidation attack has become catastrophic and marking the useful temperaturetime limit for the alloy.



Figure A5-1 Oxidation penetration plot: Alloy 1, N 155 at 1400, 1600, 1800°F.



Figure A5-1 Oxidation penetration plot: Alloy 1, N 155 at 2000°F.



Figure A5-2 Oxidation penetration plot: Alloy 3, TD nickel-chromium at 1400, 1600, 1800°F.



Figure A5-2 Oxidation penetration plot: Alloy 3, TD nickel-chromium at 2000, 2100, 2200°F.



Figure A5-3 Oxidation penetration plot: Alloy 6, DH 242 at 1400, 1600, 1800°F.



Figure A5-3 Oxidation penetration plot: Alloy 6, DH 242 at 2000, 2100, 2200°F.



Figure A5-4 Oxidation penetration plot: Alloy 10, Hastelloy X at 1400, 1600, 1800°F.



Figure A5-4 Oxidation penetration plot: Alloy 10, Hastelloy X at 2000, 2100, 2200°F.

CR-1184 NASA-Langley, 1968 — 17

## NATIONAL AERONAUTICS AND SPACE ADMINISTRATION Washington, D. C. 20546 OFFICIAL BUSINESS

FIRST CLASS MAIL

POSTAGE AND FEES PAID NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

POSTMASTER:

If Undeliverable (Section 158 Postal Manual) Do Not Return

"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."

-NATIONAL AFRONAUTICS AND SPACE ACT OF 1958

## NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

TECHNICAL REPORTS: Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

TECHNICAL NOTES: Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

TECHNICAL MEMORANDUMS: Information receiving limited distribution because of preliminary data, security classifica-

tion, or other reasons. CONTRACTOR REPORTS: Scientific and technical information generated under a NASA contract or grant and considered an important

contribution to existing knowledge.

TECHNICAL TRANSLATIONS: Information published in a foreign language considered to merit NASA distribution in English.

SPECIAL PUBLICATIONS: Information derived from or of value to NASA activities. Publications include conference proceedings, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

TECHNOLOGY UTILIZATION

PUBLICATIONS: Information on technology used by NASA that may be of particular interest in commercial and other non-aerospace applications. Publications include Tech Briefs, Technology Utilization Reports and Notes, and Technology Surveys.

Details on the availability of these publications may be obtained from:

SCIENTIFIC AND TECHNICAL INFORMATION DIVISION NATIONAL AERONAUTICS AND SPACE ADMINISTRATION Washington, D.C. 20546