1

AEC
RESEARCH
and
DEVELOPMENT
REPORT

BNWL-167
52-

HIGH TEMPERATURE CREEP-RUPTURE PROPERTIES OF HASTELLOY X IN VACUUM

D. H. NYMAN

PACIFIC NORTHWEST LABORATORY operated by BATTELLE MEMORIAL INSTITUTE

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

PACIFIC NORTHWEST LABORATORY

RICHLAND, WASHINGTON

operated by

BATTELLE MEMORIAL INSTITUTE

for the

UNITED STATES ATOMIC ENERGY COMMISSION UNDER CONTRACT AT(45-1)-1830

PRINTED BY/ FOR THE U. S. ATOMIC ENERGY COMMISSION

BNWL-167
UC-25, Metals, Ceramics,
and Materials

HIGH TEMPERATURE CREEP-RUPTURE
PROPERTIES OF HASTELLOY X IN VACUUM

By
D. H. Nyman
Reactor Metals Research Section
Reactor and Materials Technology Department

November, 1965

PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON

HIGH TEMPERATURE CREEP-RUPTURE PROPERTIES OF HASTELLOY X IN VACUUM

INTRODUCTION

Hastelloy X is being considered as a structural material for the ATR* Gas Loop for service in the test section and associated piping at temperatures to 2100 °F. Creep strength is one of the principal design criteria. A review of the literature of Hastelloy X showed that its creep-rupture properties had been determined only at temperatures to 1800 °F.⁽¹⁾ Although such methods as the Larson-Miller parameter⁽²⁾ can extrapolate these data to higher temperatures, experimental verification is the only assurance of complete validity. Therefore, a program was initiated to determine the actual creep-rupture behavior of Hastelloy X at temperatures to 2100 °F.

SUMMARY

- The creep-rupture properties of Hastelloy X in vacuum have been determined at 1950 °F and 2100 °F.
- Grain growth occurred at 2100 °F for times greater than 20 hr and caused a marked reduction in both ductility and time to rupture from expected values.
- The data from this investigation are compared with lower temperature data obtained by other investigators.

EXPERIMENTAL PROCEDURE

The creep-rupture tests were conducted in the type of machine shown in Figure 1. The apparatus and procedures developed for this investigation have been described in detail elsewhere.⁽³⁾ The machine was designed for either vacuum or gaseous environments, and is capable of operating at 2400 °F with an internal dynamic pressure of 5×10^{-5} torr.

The specimens used in this investigation were fabricated from a 0.375 in. sheet having a specific composition (Table I). Buttonhead specimens with a 1 in. long and a 0.160 in. diameter gage section were machined from the rolling direction.

* Advanced Test Reactor, National Reactor Testing Site, Arco, Idaho.

FIGURE 1

High Temperature Creep-Rupture Apparatus

TABLE I
CHEMICAL ANALYSIS, wt%
OF HASTELLOY X USED FOR TEST SPECIMENS
(HEAT NUMBER X-4882)

C	0.12	Al	--	Mo	9.13
Si	0.81	Cr	22.04	Ni	Bal
Mn	0.56	Co	1.88	Ti	--
P	0.005	Cu	--	V	--
S	0.007	Fe	17.46	W	0.68

RESULTS

A total of 13 creep-rupture tests were conducted at temperatures of 1950 and 2100 °F. Minimum creep rates, rupture life, and elongation at rupture for these tests are given in Table II, and the test results are plotted in Figures 2 through 6. Figures 2 and 3 are log-log plots of stress versus rupture life and stress versus minimum creep rate, respectively.

TABLE II
CREEP-RUPTURE DATA OF HASTELLOY X

<u>Temp,</u> <u>°F</u>	<u>Stress,</u> <u>psi</u>	<u>Minimum</u> <u>Creep Rate,</u> <u>/hr</u>	<u>Elongation,</u> <u>%</u>	<u>Time to Rupture,</u> <u>hr</u>
2100	5000	4.8×10^{-1}	24.7	0.4
	4000	3.0×10^{-1}	23.5	0.6
	3000	1.1×10^{-1}	23.4	2.0
	2500	3.1×10^{-2}	19.8	4.3
	1300	7.7×10^{-3}	23.0	21.0
	900	1.4×10^{-3}	10.8	65.0
	500	7.2×10^{-4}	14.0	114.5
	300	2.0×10^{-4}	14.2	340.0
1950	8000	3.0×10^{-1}	27.0	0.7
	6000	1.5×10^{-1}	32.9	3.9
	4000	3.2×10^{-2}	26.5	7.3
	2500	4.8×10^{-3}	19.0	36.0
	1500	1.3×10^{-3}	24.1	250.0

Lower temperature data from the literature⁽¹⁾ are also plotted in these two figures. At low stress and high temperature, both rupture life and minimum creep rate suffer in comparison with other test conditions (Figures 2 and 3).

FIGURE 2

Stress-Rupture Data for Hastelloy X

FIGURE 3

Stress Versus Minimum Creep Rate for Hastelloy X

FIGURE 4

Creep of Hastelloy X at 1950 F

FIGURE 5

Creep of Hastelloy X at 2100 F

FIGURE 6

Correlation of Rupture Data for Hastelloy X

Rupture life is much shorter and minimum creep rate is higher than would be predicted from extrapolation of the higher-stress data. For example, at 2100 °F and a stress of 500 psi, a rupture life of 450 hr and a minimum creep rate of 2.5×10^{-4} /hr would be predicted; but, in actuality, the rupture life was 115 hr with a minimum creep rate of 7.2×10^{-4} /hr. Some typical creep curves at various stresses and temperatures are drawn in Figures 4 and 5. Elongation values ranged from 24.7% to 10.8% at 2100 °F and from 32.9% to 19% at 1950 °F. Ductility shows a trend toward lower values below a threshold of 1300 psi at 2100 °F and exhibits comparable values above 1300 psi at both 1950 and 2100 °F.

Larson-Miller plots of the experimental data and the lower-temperature data from the literature are shown in Figure 6. Two curves are shown: one for the literature data extrapolated to higher temperatures and the second for the experimental higher-temperature data. With this combined construction, the rupture life from 1350 to 2100 °F can be predicted for a range of stresses.

Metallographic examination of the specimens after they were tested showed many intergranular voids and cracks near the fracture with a few cracks extending to the surface. Figure 7 is a macroscopic view of a broken specimen. Figure 8 shows the intergranular mode of fracture initiation at 2100 °F for various stresses. The specimens tested at 1300 psi, 900 psi, 500 psi and 300 psi (Figures 8c, 8d, 8e, and 8f, respectively) show a considerable amount of grain growth compared to the specimens tested at higher stresses (Figures 8a and 8b). This grain growth is caused by the longer test time and is considered responsible for the reduced ductility at the lower stresses. Figures 8d, 8e, and 8f show subsurface voids. Bourgette⁽⁸⁾ has also noted these voids in nickel-, cobalt-, and iron-base alloys held at temperatures to 1800 °F and pressures of 10^{-9} torr for periods up to 3500 hr. He attributes these voids to the selective evaporation of high-vapor-pressure alloy ingredients, mainly chromium and manganese.

DISCUSSION

The structural components in the ATR Gas Loop will be exposed to an atmosphere of high-purity helium. The tests in this investigation were carried out in vacuum, which simulates a high-purity, inert atmosphere.

FIGURE 7

Macroscopic View of Fractured Specimen
(2100 °F and 2500 psc stress)
5X

Negative 5652137

(a)

4000 psi Stress, Rupture at 0.6 hr

Fracture

(c)

1300 psi, Stress, Rupture at 21 hr

Fracture

(e)

500 psi Stress, Rupture at 65 hr

(b)

2500 psi Stress, Rupture at 4.3 hr

Fracture

(d)

900 psi Stress, Rupture at 65 hr

Fracture

(f)

300 psi Stress, Rupture at 65 hr

FIGURE 8

Mode of Fracture Initiation in Vacuum at 2100 °F
50X

Negatives 46J2059; 46J2166

Negatives 46J2168; 46J2167

Negatives 46J2059; 46J2166

Fracture

(a)

ess, Rupture at 0.6 hr

Fracture

(c)

1300 psi, Stress, Rupture at 21 hr

Fracture

(e)

500 psi Stress, Rupture at 114.5 hr

Fracture

(b)

ess, Rupture at 4.3 hr

Fracture

(d)

900 psi Stress, Rupture at 65 hr

Fracture

(f)

300 psi Stress, Rupture at 340 hr

FIGURE 8

Mode of Fracture Initiation in Vacuum at 2100 °F
50X

Negatives 46J2168; 46J2167

Negatives 46J2059; 46J2166

In a creep test of nickel-base alloys, oxygen will influence the mechanical strength by forming a protective oxide film and by blunting the crack tip, thereby decreasing the stress concentration.

The low-stress, high-temperature, creep-rupture behavior of Hastelloy X in this study agrees with results reported by other investigators.^(1, 4-7) Reduced rupture life in vacuum compared to air has been noted by Shahinian and Achter.^(4, 5) Their work on Nichrome V and nickel showed better strength in air than in vacuum at low stresses, but a higher strength in vacuum than in air at high stresses. They explained their results in terms of two competing processes: oxidation strengthening and surface energy effects on crack propagation. Air strengthening can be caused by one or more processes. Surface oxide films act as a barrier to dislocations while internal oxidation reduces the stress concentration by blunting a crack tip. Neither oxidation nor gas adsorption should play a major role in this investigation, since the tests were carried out in vacuum.

At low stresses and high temperatures, a definite deviation from a power law relationship with stress appeared for both rupture life and minimum creep rate (Figures 2 and 3). Similar downward deviations have also been noted by Donachie and Shephard.⁽¹⁾ They assumed that vacuum data represent the intrinsic strength of the alloy; therefore, the downward break in vacuum data results from a lack of air strengthening.

In this investigation, however, the reduced rupture life and increased creep rate at low stresses can be explained by grain growth and its influence on intergranular crack propagation. Referring to Table II and Figure 8, it can be seen that for stresses above 1300 psi at 2100 °F, the rupture times are relatively short, i. e., less than 21 hr, and values of total elongation at rupture vary from 19.8% to 24.7%. The microstructures are similar in that there is very little grain growth and many intergranular cavities. At stresses below 1300 psi the rupture times are much longer, and values of total elongation are reduced almost 50%. The microstructure shows considerable grain growth, a lack of second phase, large cracks extending to the surface, and subsurface voids.

The subsurface voids are attributed to the volatilization of manganese and chromium, which have a vapor pressure of 5×10^{-1} torr and 1.2×10^{-4} torr, at 2100 °F respectively. The formation of these voids is time-dependent (Figures 8d, 8e, and 8f). At longer times, the voids are present at greater depths below the surface.

The larger grains resulting from grain growth have much less grain boundary area for supporting plastic deformation of the specimen by grain boundary shear, and rupture occurs prematurely. Although measured minimum creep rates appear to be greater than extrapolated values for the large-grained material, the lack of structural stability during the test renders these values nonrepresentative of the final grain size.

Correlation of the rupture data is shown in Figure 6. The dotted line is extrapolated from lower-temperature data from the literature. A second line is drawn through the higher-temperature values from the present study. With a nominal stress of 1000 psi at 2100 °F, a rupture life of about 450 hrs would be predicted from the extrapolated curve, while in actuality, rupture occurred in only 35 hr. This exemplifies the need to determine creep-rupture properties of a structural material under actual service conditions, and the limitations of extrapolating lower-temperature data in predicting long-term mechanical behavior at temperatures where structural instabilities may have a controlling role.

ACKNOWLEDGEMENTS

The author wishes to express his appreciation to G. L. Hood and N. E. Harding for assisting in the creep-rupture tests. Thanks are also extended to Dr. A. L. Bement for his helpful discussions in analyzing the results.

REFERENCES

1. M. J. Donachie and R. G. Shephard. "Creep-Rupture Behavior of Hastelloy-X in Vacuum," Mater. Res. Std., p. 495. September, 1964. ^{Vol. 4}
2. F. R. Larson and J. Miller. "A Time-Temperature Relationship for Rupture and Creep Stresses," Trans. ASME, vol. 74, p. 765. 1952.
3. D. H. Nyman, M. R. Keegan, and J. A. Williams. "A High Temperature Creep Machine for In-Cell Operation," BNWL-137. September, 1965.
4. P. Shahinian and M. Achter. "Temperature and Stress Dependence of the Atmosphere Effect on Nichrome V," Trans. ASM, vol. 51, p. 244. 1959.
5. P. Shahinian and M. Achter. "A Comparison of the Creep-Rupture Properties of Nickel in Air and in Vacuum," Trans. AIME, vol. 215, p. 37. 1959.
6. P. Shahinian and M. Achter. "Effect of Environment on Creep-Rupture Properties of Some Commercial Alloys," Trans. ASM, vol. 49, p. 862. 1957.
7. R. Widmer and N. Grant. "The Role of Atmosphere on 80 Nickel-20 Chromium Alloys," Trans. ASME, vol. 82, p. 882. 1960.
8. D. T. Bourgette. Evaporation of Iron, Nickel, and Cobalt Base Alloys at 760-980 C in High Vacuums, ORNL-3677, Oak Ridge National Laboratory. November, 1964.

ONSITE DISTRIBUTIONCopy NumberPacific Northwest Laboratory

1	F. W. Albaugh
2	R. P. Allen
3	R. C. Aungst
4	J. A. Ayres
5	A. L. Bement
6	W. A. Burns
7	S. H. Bush
8	J. J. Cadwell
9	T. T. Claudson
10	G. M. Dalen
11	L. J. Defferding
12	D. R. deHalas
13	R. F. Dickerson
14-15	R. L. Dillon
16	S. L. Fawcett
17	N. E. Harding
18	R. G. Hoagland
19	G. L. Hood
20	J. E. Irvin
21	J. H. Johnson
22	D. C. Kaulitz
23	R. S. Kemper
24	H. E. Kissinger
25	G. L. Kulcinski
26	G. A. Last
27	R. D. Leggett
28	I. S. Levy
29	J. E. Minor
30	E. L. Moore
31-40	D. H. Nyman
41	D. P. O'Keefe
42	R. E. Olson
43	H. M. Parker
44	F. A. Smidt, Jr.
45	F. W. Van Wormer
46	K. R. Wheeler
47-48	R. G. Wheeler
49	O. J. Wick
50	J. A. Williams
51	H. H. Yoshikawa
52-56	Technical Information Files
57	300 Area Technical Publications
58-59	700 Area Technical Publications

ONSITE DISTRIBUTION (Contd)Copy NumberRichland Operations Office

60	P. G. Holsted
61	L. R. Lucas
62	R. K. Sharp
63	Technical Information Library

OFFSITE DISTRIBUTION (SPECIAL)No. of Copies

7	Argonne National Laboratory
	R. J. Armani
	T. H. Blewitt
	L. R. Kelman
	J. H. Kittel
	F. D. McGinnis-EBR-1, Idaho Falls
	A. D. Rossin
	D. K. Youngdahl
1	Battelle Memorial Institute
	F. R. Shober
3	Brookhaven National Laboratory
	J. G. Y. Chow
	D. H. Gurinsky
	S. B. McRickard
2	General Atomic Division
	T. A. Trozera
	W. P. Wallace
1	Material Research Laboratory
	E. J. Ripling
4	Naval Research Laboratory
	J. R. Hawthorne
	J. M. Krafft
	L. E. Steele
	A. M. Sullivan
7	Nuclear Materials and Propulsion Operation
	H. C. Brassfield
	F. D. Kingsbury
	J. McGurty
	J. Moteff
	R. C. Rau
	F. C. Robertshaw
	J. P. Smith

OFFSITE DISTRIBUTION (SPECIAL)No. of Copies

11	Oak Ridge National Laboratory R. C. Berggren W. E. Brundage N. E. Hinkle E. C. Miller S. M. Ohr J. T. Stanley D. B. Trauger - Nuclear Division, UCC P. O. Box Y, Oak Ridge M. S. Wechsler J. R. Weir J. M. Williams F. W. Young
3	Phillips Petroleum Company J. M. Beeston W. C. Francis C. H. Hogg
1	USAEC - Chicago Operations Office Claude Pursel
1	USAEC - Idaho Operations Office John F. Kaufmann
1	USAEC - Oak Ridge Operations Office D. F. Cope
4	USAEC - Washington, D. C. Division of Licensing and Regulation M. Bolotsky E. G. Case J. J. DiNunno A. B. Holt
7	Division of Reactor Development R. C. Cilimberg I. H. Mandil J. L. Mershon W. L. R. Rice J. M. Simmons R. H. Steele S. A. Szawlewics
1	Division of Research D. K. Stevens
1	J. L. Ault Metallurgy Division Lewis Research Center, NASA Cleveland, Ohio

OFFSITE DISTRIBUTION (SPECIAL)No. of Copies

1	L. P. Bupp, Manager Vallecitos Atomic Laboratory General Electric Company P. O. Box 846 Pleaston, California
1	Dr. M. B. Biles Acting Senior AEC Representative, Brussels Office U. S. Mission to the European Communities APO 667 New York, N. Y.
1	L. M. Bonnefond Reactor Division U. S. Atomic Energy Commission San Francisco Operations Office 2111 Bancroft Way Berkeley, California 94704
1	W. P. Chernock Nuclear Division Combustion Engineering Corporation Prospect Hill Road Windsor, Connecticut
1	R. H. Fillnow Westinghouse Bettis Atomic Power Laboratory P. O. Box 1468 Pittsburgh, Pennsylvania
1	A. E. Focke University of Cincinnati Dept. of Chemical Engineering and Metallurgical Engineering Cincinnati 21, Ohio
15	R. F. Fraley (15) Executive Secretary Advisory Committee on Reactor Safeguards U. S. Atomic Energy Commission Washington, D. C. 20545
1	J. F. Haines Nuclear Power Department Atomic Energy Division Allis-Chalmers Manufacturing Company 508 Kennedy Street, N.W. Washington 11, D.C.

OFFSITE DISTRIBUTION (SPECIAL)No. of Copies

1	E. T. Hayes Special Assistant for Materials DDr&E The Pentagon Washington 25, D.C.
1	George Knighten Nuclear Power Field Office Engineer Research and Development Center Fort Belvoir, Virginia 22060
1	M. Krasner Nuclear Reactor Division Lewis Research Center 21000 Brookpark Road Cleveland 35, Ohio
1	E. Landerman Westinghouse Electric Corporation Atomic Power Division P. O. Box 355 Pittsburgh 30, Pennsylvania
1	J. R. Lane Materials Advisory Board National Academy of Sciences 2101 Constitution Avenue, N.W. Washington 25, D.C.
1	A. T. Lovell Nuclear Materials and Propulsion Operation National Reactor Test Station P. O. Box 2147 Idaho Falls, Idaho
1	S. G. Nordlinger AEC Scientific Representative USAEC London Office U. S. Embassy London, England
20	John W. Norris (20) Chief, Cataloging Branch U. S. Atomic Energy Commission Division of Technical Information Extension P. O. Box 62 Oak Ridge, Tennessee 37831

OFFSITE DISTRIBUTION (SPECIAL)No. of Copies

1	L. M. Raring Pratt & Whitney Aircraft Division CANEL Operation P. O. Box 611 Middletown, Connecticut
1	Headquarters Directorate of Materials and Processes Aeronautical Systems Division Air Force Systems Command U. S. Air Force Attn: Col. L. J. Standifer Wright-Patterson Air Force Base, Ohio
1	G. W. Titus Aerojet-General Nucleonics P. O. Box 77 San Ramon, California
1	J. Van Ullen Knolls Atomic Power Laboratory P. O. Box 1072 Schenectady, New York
1	L. R. Weisert Babcock & Wilcox 1201 Kemper Street Lynchburg, Virginia