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REPORT NO.
TDR-169(3240-11)TN-1

Correlation and Interpretation of High-Temperature Mechanical Properties of Certain Superalloys

11 MARCH 1963

Prepared by H. CONRAD and J. WHITE
Materials Sciences Laboratory
Aerospace Corporation

and

E. BERNETT
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Prepared for COMMANDER SPACE SYSTEMS DIVISION
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LABORATORIES DIVISION • AEROSPACE CORPORATION
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Report No.
TDR-169(3240-11)TN-1

CORRELATION AND INTERPRETATION OF HIGH-TEMPERATURE MECHANICAL PROPERTIES OF CERTAIN SUPERALLOYS

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Contract No. AF 04(695)-169

11 March 1963

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ABSTRACT

High-temperature mechanical property data (tensile and compressive proportional limit, 0.2 percent yield strength, flow stress, tensile strength, creep rate and time to rupture) for an iron-base (N-155), a nickel-base (René 41) and a cobalt-base (L-605) superalloy were analysed to evaluate the equation which correlates the data and to identify the dislocation mechanism which is rate controlling. The data correlated through the relationship

$$\dot{\epsilon} = \frac{A}{T} \exp-\left(\frac{H_c}{RT}\right) f(\sigma) \cong kt_r \qquad ,$$

where $\dot{\epsilon}$ is the strain rate, t_r is the time to rupture, H_c is the activation energy for deformation, A and k are constants, and $f(\sigma)$ is a stress function which was $f(\sigma) = \sigma^n$ with $n = 4$ to 5 at stress below about 10 kg/mm^2 (15,000 psi). The values of H_c are 100, 215, and 120 kcal/mole, respectively, for N-155, René 41, and L-605, values higher than those for self-diffusion of the base metals of these alloys.

The form of the above equation, especially in regard to the stress law, is in agreement with Weertman's dislocation climb model of high-temperature creep. It is suggested that the higher values of H_c may represent either changes in structure with temperature or the interaction of solute atoms or precipitates with dislocations during climb.

CONTENTS

ABSTRACT ii

I. INTRODUCTION 1

II. MATERIAL AND EXPERIMENTAL PROCEDURE 4

III. RESULTS 5

IV. DISCUSSION 7

V. TABLES AND FIGURES 11

REFERENCES 24

TABLES

I. Nominal composition of alloys 12

II. Melting temperature of alloys and activation energy for self-diffusion of base metal 13

III. Values of the activation energy H_c and the constants A and n derived from the experimental data 13

FIGURES

1. 0.2 percent yield stress versus strain rate for N-155 alloy [data from Bernett (Ref. 13)]. 14

2. Stress versus tensile creep rate for N-155 alloy [data from Bernett (Ref. 13)]. 15

3. Stress versus strain rate for program-controlled strain rate in tension on René 41 [data from Dioguardo and Lloyd (Ref. 15)]. 16

4. Stress versus time to obtain 0.5 percent creep strain from L-605 alloy [data from Dioguardo and Lloyd (Ref. 15)]. 17

5. Logarithm of temperature compensated deformation rate versus logarithm of stress for N-155. 18

6. Logarithm of temperature compensated deformation rate versus logarithm of stress for René 41. 19

7. Logarithm of temperature compensated deformation rate versus logarithm of stress for L-605. 20

8. Log average creep rate to 4 percent strain versus log rupture time. 21

9. Microstructures of L-605 as annealed and tested at 1800°C (982°C) and 2200°F (1204°C). Etch: 92 percent HCl, 5 percent H_2SO_4, 3 percent HNO_3 22

10. Microstructures of René 41 as heat treated and after compression testing at 1600°F (871°C), 1800°F (982°C), and 2000°F (1093°C). Etch: electrolytic 55 percent HCl, 15 percent HNO_3, 15 percent acetic acid, 15 percent H_2O . . . 23

SECTION I

INTRODUCTION

There is now considerable evidence[1-3] that the activation energy H_c for deformation of relatively pure metals at high temperatures (i.e., at temperatures above $0.5\ T_m$, where T_m is the melting temperature in °K) is the same as that for self-diffusion H_{SD} and, hence, that the deformation is diffusion-controlled. This agreement between H_c and H_{SD} has also been observed for simple alloys.[4-6] Some diffusion-controlled mechanisms which might be rate-controlling at high temperatures are:

1. The climb of dislocations;
2. The nonconservative motion of jogs in screw dislocations;
3. The dragging of a solute atmosphere;
4. The healing of the disruption of a cluster of solute atoms or of short-range order as a dislocation moves through the lattice;
5. The dissolution or agglomeration of precipitates opposing the motion of dislocations;
6. Nabarro-Herring diffusion creep.

In more complex alloys, the value of H_c is often somewhat higher than H_{SD} of the solvent; for example in iron- and nickel-base alloys it has been found that $H_c = 90$ to 120 kcal/mole,[5-7] whereas $H_{SD} = 60$ to 80 kcal/mole.[1] The reason for this difference is not yet known.

Of considerable technological importance is the ability to interpolate or extrapolate mechanical property data from a minimum number of tests. A review and evaluation of the various equations or parameters used for this purpose was made by Conrad,[8] who found that they all possessed certain limitations. He suggested that correlations be based on latest theoretical as well as experimental information and that differential-type tests, where stress and temperature are changed during the test, be considered as a rapid and economical method for evaluating the constants of any correlating parameter.

Deformation equations most generally relate strain rate with temperature and stress. Often from a practical standpoint it is more important to know the interrelationship among time to rupture, stress, and temperature. Of significance in this regard is the experimentally observed relationship between the time to rupture t_r and the secondary creep rate $\dot{\epsilon}_s$:[9-12]

$$t_r = \left(\frac{K}{\dot{\epsilon}}\right)^m \quad , \tag{1}$$

where m is a constant approximately equal to one.

In the present paper, high-temperature mechanical property data obtained at the Marquardt Corporation[13-15] for three complex superalloys -- N-155, L-605 and René 41 -- are analyzed with the objective of providing additional information regarding the points discussed above, especially in regard to first, establishing the mechanism which is rate controlling, and second, correlating the data for use in interpolating and extrapolating. The Marquardt data is especially significant for such an analysis because:

1. A variety of tests were conducted: constant strain rate tensile tests, constant strain rate compression tests, programmed constant strain rate tests (differential tests), tensile creep, compressive creep;
2. The alloys considered represent three base metals: iron-base (N-155), nickel-base (René 41), and cobalt-base (L-605);
3. The data are sufficiently extensive: strain rate range of 10^{-5} to 1.0 sec^{-1}, temperature range 1600° to 2200°F (871° to 1204°C);

4. Heating of the specimen was accomplished in a very short time ($200^{\circ}F$ per second heating rate) by self-resistance heating; consequently, the structural changes associated with heating of the specimen were kept to a minimum.

SECTION II

MATERIAL AND EXPERIMENTAL PROCEDURE

The nominal compositions of the three alloys considered are given in Table I. Their minimum melting temperatures and the activation energy for self-diffusion of the base metal are given in Table II. Mechanical property tests were conducted on sheet 0.05 to 0.07 inch in thickness. (Detailed history of the particular heats tested and of the mechanical testing conditions can be found in Refs. 13-15, as can also the detailed mechanical test data.) The data considered here are for temperatures of 1600°F (871°C) and above (0.7 to 0.95 T_m), where the proportional limit and yield stress are sensitively dependent on strain rate.

SECTION III

RESULTS

Typical mechanical property data are shown in the log-stress versus log-strain rate plots of Figs. 1-4. These suggest a relationship between strain rate $\dot{\epsilon}$ and stress σ of the form

$$\dot{\epsilon} = a\sigma^n \quad . \qquad (2)$$

From many such plots it was found that n decreased with temperature, approaching a constant value between 4.0 and 5.0 at the higher temperatures, generally at 1800°F (982°C) or higher.

The apparent activation energy H_c for the high-temperature deformation of these alloys was obtained by the conventional techniques.[16] The values so obtained were found to be independent of stress and the same for all deformation phenomena (proportional limit, yield stress, tensile stress, flow stress, and creep) and types of testing (tension, compression, programmed strain rate) within the experimental error associated with their determination, which was ±15 percent. The average values of H_c are presented in Table III.

Since H_c was independent of stress and deformation phenomena, the deformation rate may be expressed by an equation of the form proposed by Dorn[1] and Weertman[2, 17] for high-temperature creep:

$$\dot{\epsilon} = \frac{A}{T} \exp\left(\frac{-H_c}{RT}\right) f(\sigma) \quad , \qquad (3)$$

where A is a constant and $f(\sigma)$ is a stress function. Figures 5-7 show how well the mechanical test data for the present alloys correlate using Eq. (3).

At low stress, <10 kg/mm^2 (<15,000 psi), as is seen here $f(\sigma) \approx \sigma^{4.0-5.0}$, while at higher stresses n increases as stress increases, similar to the observations of Weertman[2, 17-19] on relatively pure metals.

From Fig. 8 it is seen that the relationship between strain rate and time to rupture given by Eq. (1) is approximated by the present alloys with $m \simeq 1.0$.

SECTION IV

DISCUSSION

The manner in which the data correlate in Figs. 5-7 and the fact that the stress exponent at low stresses is between four and five suggest that Weertman's dislocation climb mechanism[2, 17] applies to the high-temperature deformation of the presently considered superalloys. However, the values of H_c are significantly higher than rather than equal to H_{SD}, as required by the theory. These higher activation energies may be due to one or more of the following possibilities:

1. The values of H_c obtained are only apparent activation energies and include changes in structure with temperature (solute distribution, precipitates, etc.);
2. The activation energy for deformation includes the energy H_j to form the jogs required for dislocation climb[20] so that $H_c = H_{SD} + H_j$;
3. The activation energy for climb includes the interaction energy between a dislocation and a solute atom[21];
4. The activation energy for climb includes the surface energy associated with precipitates on dislocation lines[22];
5. Alloying increases the value of H_{SD} through the interaction energy between a vacancy and a solute atom;
6. Deformation is controlled by the diffusion of an alloying element whose activation energy for diffusion H_{AD} is larger than that for self-diffusion of the base metal.

These possibilities will now be considered in the order in which they are listed.

Very little, if any, change in structure is expected to occur in N-155 and L-605 during testing in the temperature range under consideration, namely, 1600° to 2200°F (871° to 1204°C). Although definite carbide precipitation is known to occur in the cobalt-base L-605 alloy from 1400° to 1800°F (760° to 982°C), the strength in the testing temperature range for this alloy, 1800° to 2200°F (982° to 1204°C), is primarily due to solid solution strengthening. Wagner and Hall[23] report that the strength of this material above 1800°F (982°C) is essentially the same for the aged and annealed conditions. Microstructure and hardness values of annealed L-605 both previous to testing and after testing are given in Fig. 9 and support the contention that practically no changes in structure occurred during testing. Although test specimens of the iron-base N-155 alloy were not available for study, it, too, is primarily a solid solution strengthened alloy in the temperature range under consideration, and no changes in structure are expected.

The nickel-base alloy René 41 is not simply a solid solution strengthened alloy, but is dependent upon the precipitation of Ni_3 (Al, Ti) called γ' during aging at 1400°F (760°C) for its superior strength. Testing conducted from 1600° to 2000°F (871° to 1093°C) is accompanied by very definite agglomeration of this precipitate, and at higher temperatures re-solution occurs. Microstructures and hardness values of annealed and tested samples of René 41, Fig. 10, show definite evidence of changes in precipitate distribution. It is seen in this figure that the microstructure and hardness did not change much during short times at 1600°F (871°C); however, at 1800°F (982°C) and 2000°F (1093°C) agglomeration and some re-solution of the precipitate did occur, accompanied by significant loss in hardness. The higher hardness for testing at 2000°F (1093°C) than at 1800°F (982°C) is due to the rapid reprecipitation of γ' upon cooling from the solution temperature, which cannot be inhibited even by an ice brine quench. This characteristic is discussed by Decker et al.[24] for a similar nickel-base alloy.

In conclusion, it appears that structural changes could account for the extremely high value of H_c obtained for René 41, but that this does not seem to be the case for N-155 and L-605. However, even for René 41 it is somewhat puzzling that the same high value for the activation energy was obtained for the proportional limit as for the tensile strength and for changes in strain rate after appreciable plastic flow, indicating that the relative changes in structure were constant throughout a test and for all testing conditions.

The values for H_c of 100 to 120 kcal/mole for N-155 and L-605 are similar to those obtained by others for various steels[5] and for the Nimonic alloys.[7] These higher values do not seem to be related to the stacking-fault energy, because approximately the same values are obtained for ordinary carbon steels as for stainless steels, nickel alloys and cobalt alloys, which have a considerably lower stacking-fault energy. This observation suggests rather strongly that if dislocation climb is the rate-controlling mechanism, the jogs are formed mechanically rather than thermally, and, hence, that the activation energy for deformation does not include the energy to form jogs.

There is insufficient information to evaluate the possibility that the higher H_c is due to the fact that the activation energy for climb includes the interaction energy between a dislocation and a solute atom H_{AD}. If this were the case, the present data on N-155 and L-605 indicate that $H_{AD} \approx 1$ to 2 ev, which is reasonable. Likewise, there is insufficient information to evaluate whether the interaction between precipitates and dislocations is responsible for the higher values of H_c, but this is also a possibility.

Measurements on the interaction energy between solutes and vacancies E_{Av} in aluminum give values of the order of 0.1 ev. On this basis, one expects that E_{Av} should not exceed 0.5 ev for iron, nickel, and cobalt alloys. This assumption is in agreement with the fact that the activation energy for self-diffusion in these metals is not significantly affected by alloying elements. Consequently, the high values of H_c probably are not due to an increase in H_{SD} through the interaction energy between a vacancy and a solute atom.

That the higher H_c represents the activation energy for the diffusion of an alloying element does not seem reasonable either. Values for diffusion of molybdenum in iron by Sherby and Simnad[26] and for the diffusion of tungsten in nickel by Swalin and Martin[27] indicate that diffusion involves an energy only about 15 percent greater than that for self-diffusion in iron or nickel.

In summary, the above discussion leads one to the conclusion that the higher values of H_c may be due to:

1. Changes in structure associated with changes in temperature;
2. An increase in the activation energy for climb due to the interaction energy between a solute atom or a precipitate and a dislocation.

The very large value of H_c obtained for René 41 and the fact that actual structural changes were observed suggest that changes in structure are responsible for the high apparent activation energy for deformation of this alloy. For N-155 and L-605, as well as for steels in general and for the Nimonic alloys, the higher value of H_c is probably associated with the interaction energy of a dislocation line with a solute atom, with an agglomeration of solute atoms, or with a precipitate.

SECTION V

TABLES AND FIGURES

Table I. Nominal composition of alloys.

Base	N-155 Fe	René 41 Ni	Haynes 25 (L-605) Co
Fe	Bal	5.0 max	3.0 max
Ni	19.0-21.0	Bal	9.0-11.0
Co	18.5-21.0	10.0-12.0	Bal
Cr	20.0-22.5	18.0-20.0	19.0-21.0
Mo	2.50-3.50	9.0-10.50	---
W	2.0-3.0	---	14.0-16.0
Mn	1.0-2.0	0.10 max	1.0-2.0
Al	---	1.4-1.6	---
Ti	---	3.0-3.3	---
C	0.08-0.16	0.12 max	0.005-0.15
Si	1.0 max	0.50 max	1.0 max
S	0.03 max	0.15 max	0.040 max
P	0.04 max	---	0.0300
Cb + Ta	0.75-1.25	---	---
N_2	0.10-0.20	---	---
B	---	0.003-0.010	---

Table II. Melting temperature of alloys and activation energy for self-diffusion of base metal.

Alloy	Base	T_m °F	T_m °K	H_{SD}(1) kcal/mole
N-155	Fe	2350	1561	48-74.2
René 41	Ni	2425	1602	63.0-66.8
Haynes 25 (L-605)	Co	2420	1599	61.9-67.0

Table III. Values of the activation energy H_c and the constants A and n derived from the experimental data.

Alloy	H_c kcal/mole	n	A °K-sec^{-1}
N-155	100	4.1	4×10^{12}
René 41	215	5.0	5×10^{30}
Haynes 25 (L-605)	120	4.8	6×10^{14}

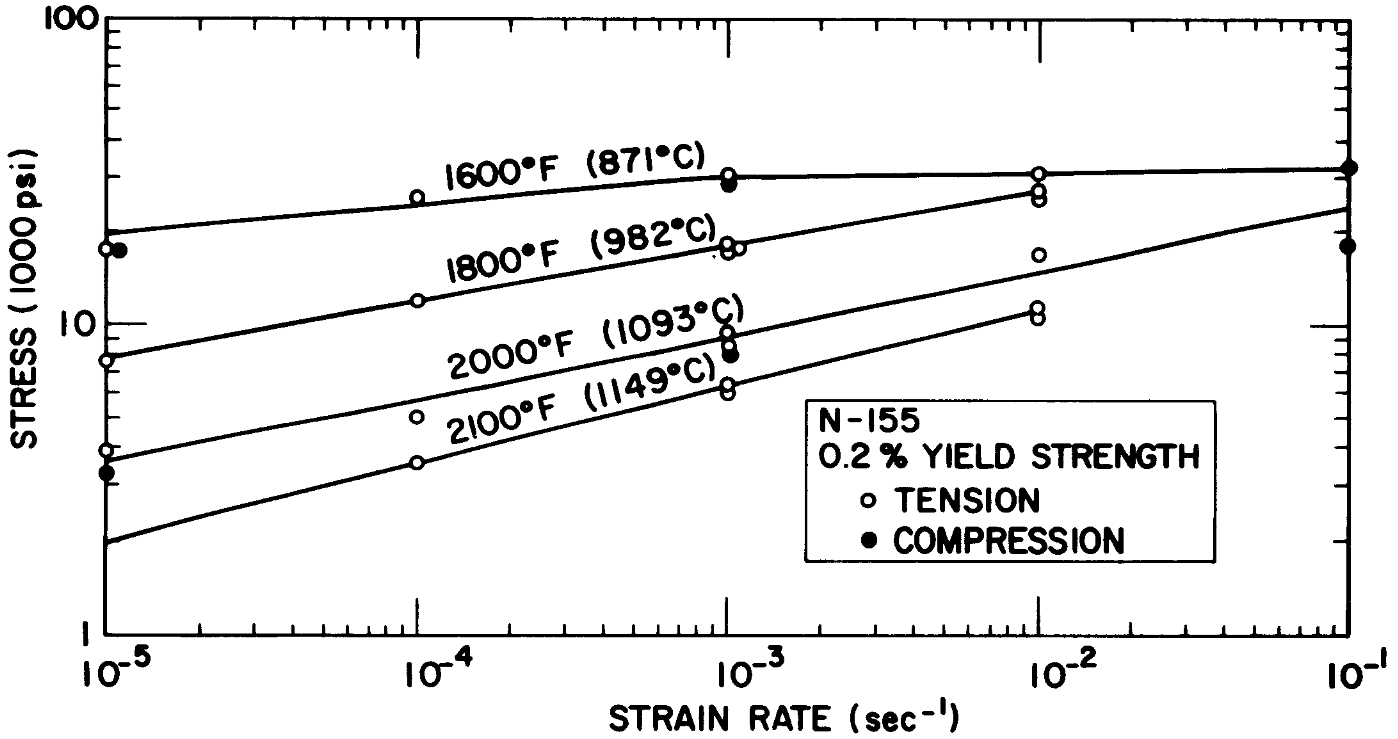


Fig. 1. 0.2 percent yield stress versus strain rate for N-155 alloy [data from Bernett (Ref. 13)].

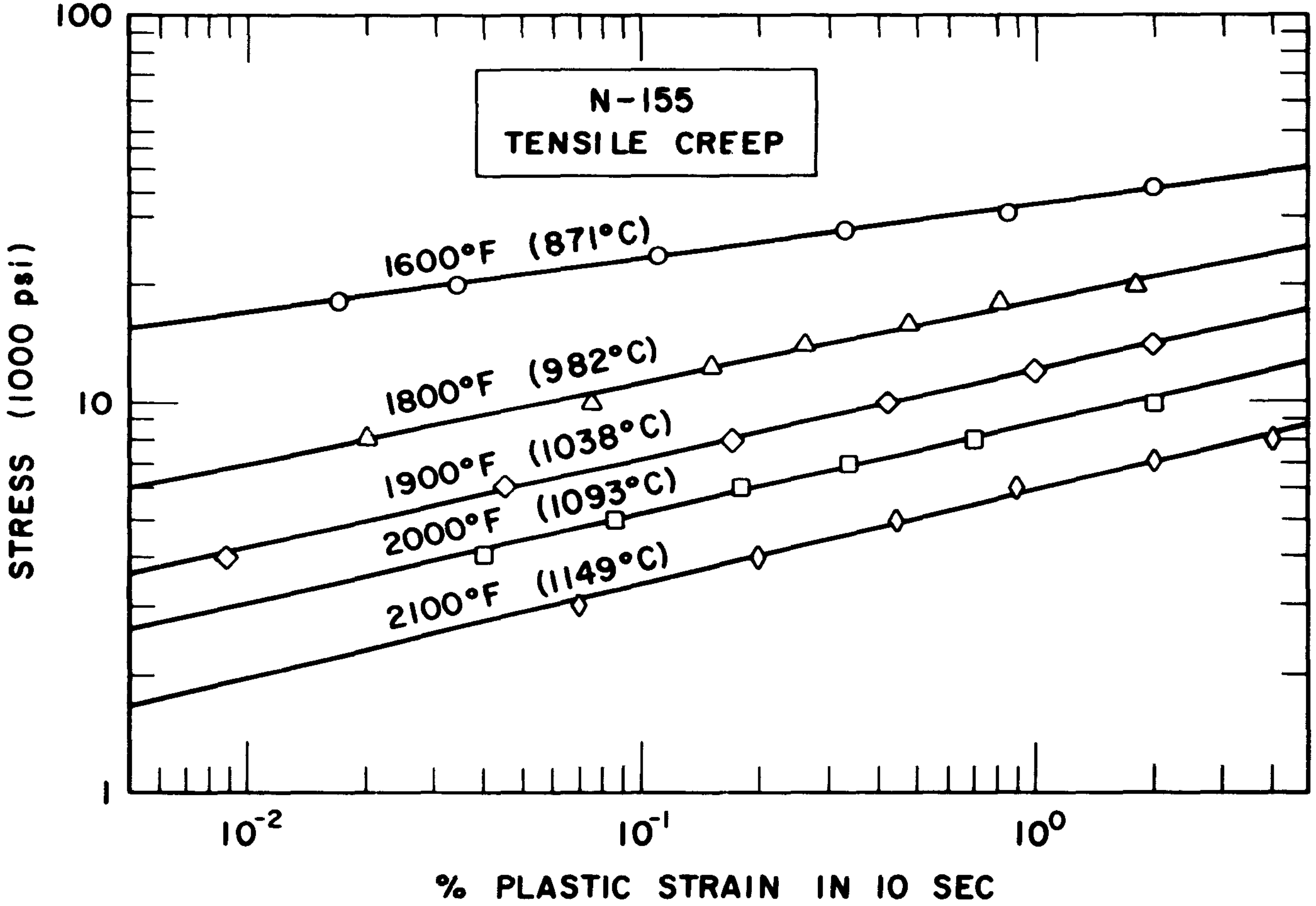


Fig. 2. Stress versus tensile creep rate for N-155 alloy [data from Bernett (Ref. 13)].

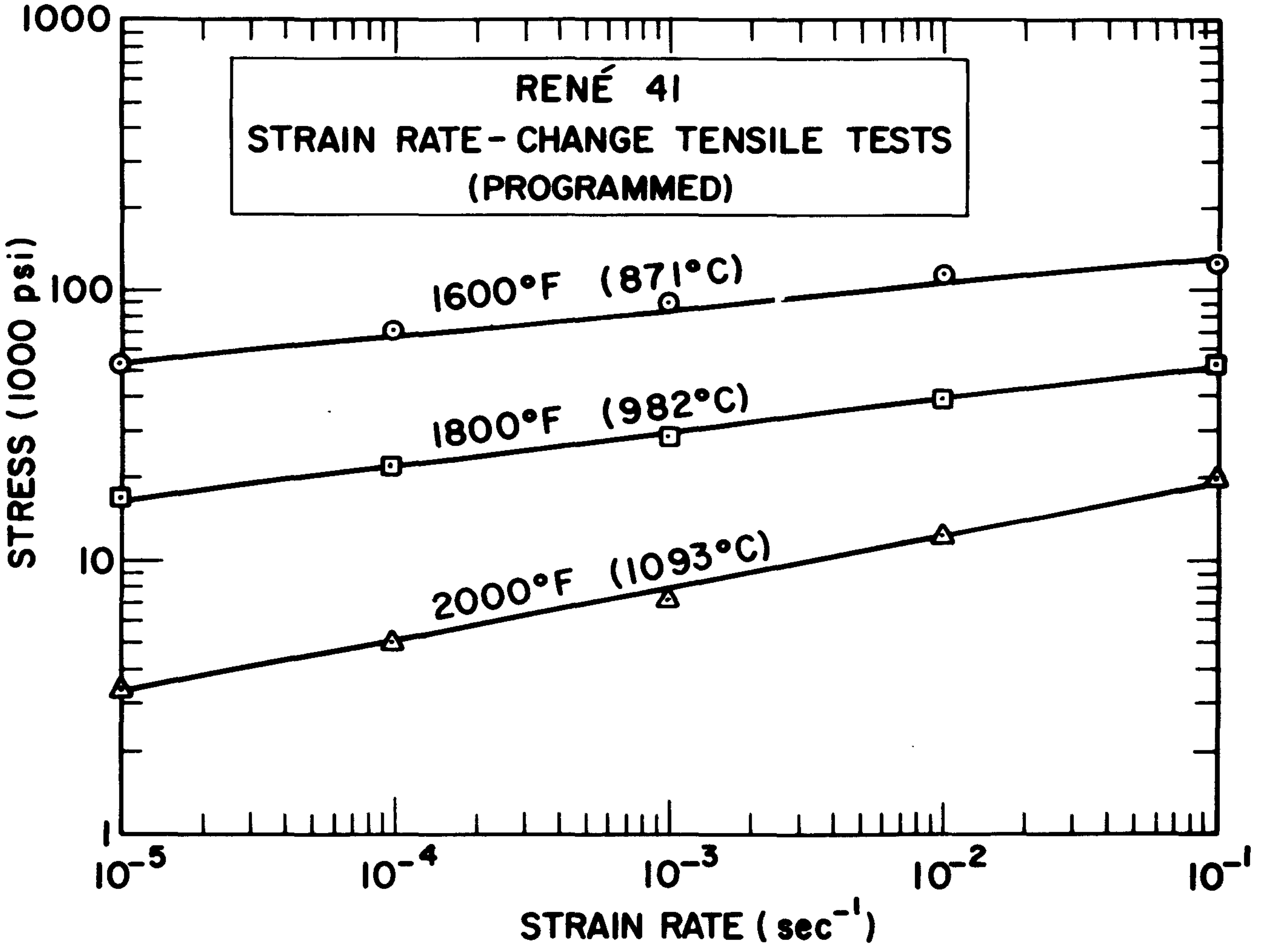


Fig. 3. Stress versus strain rate for program-controlled strain rate in tension on René 41 [data from Dioguardo and Lloyd (Ref. 15)].

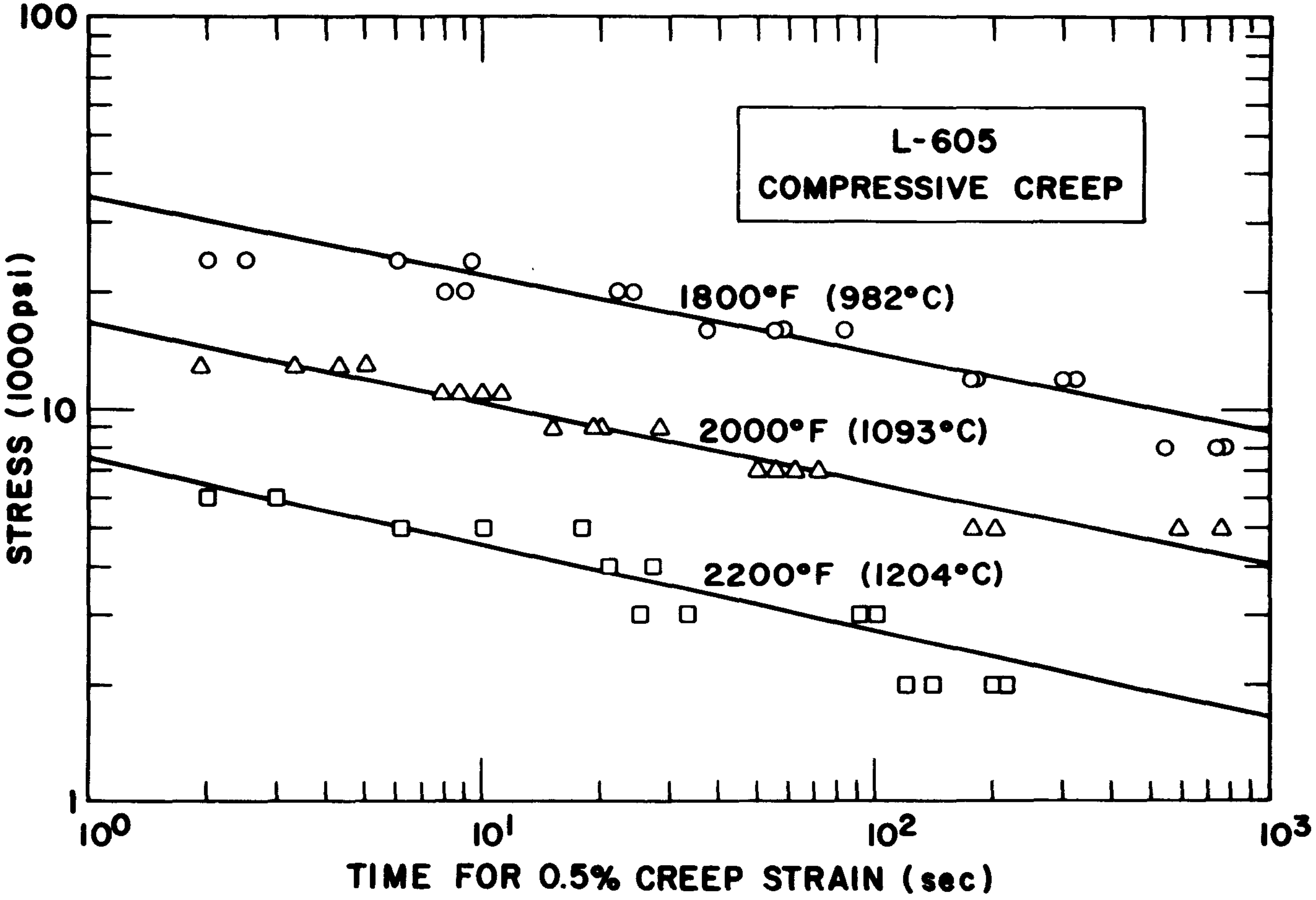


Fig. 4. Stress versus time to obtain 0.5 percent creep strain from L-605 alloy [data from Dioguardo and Lloyd (Ref. 15)].

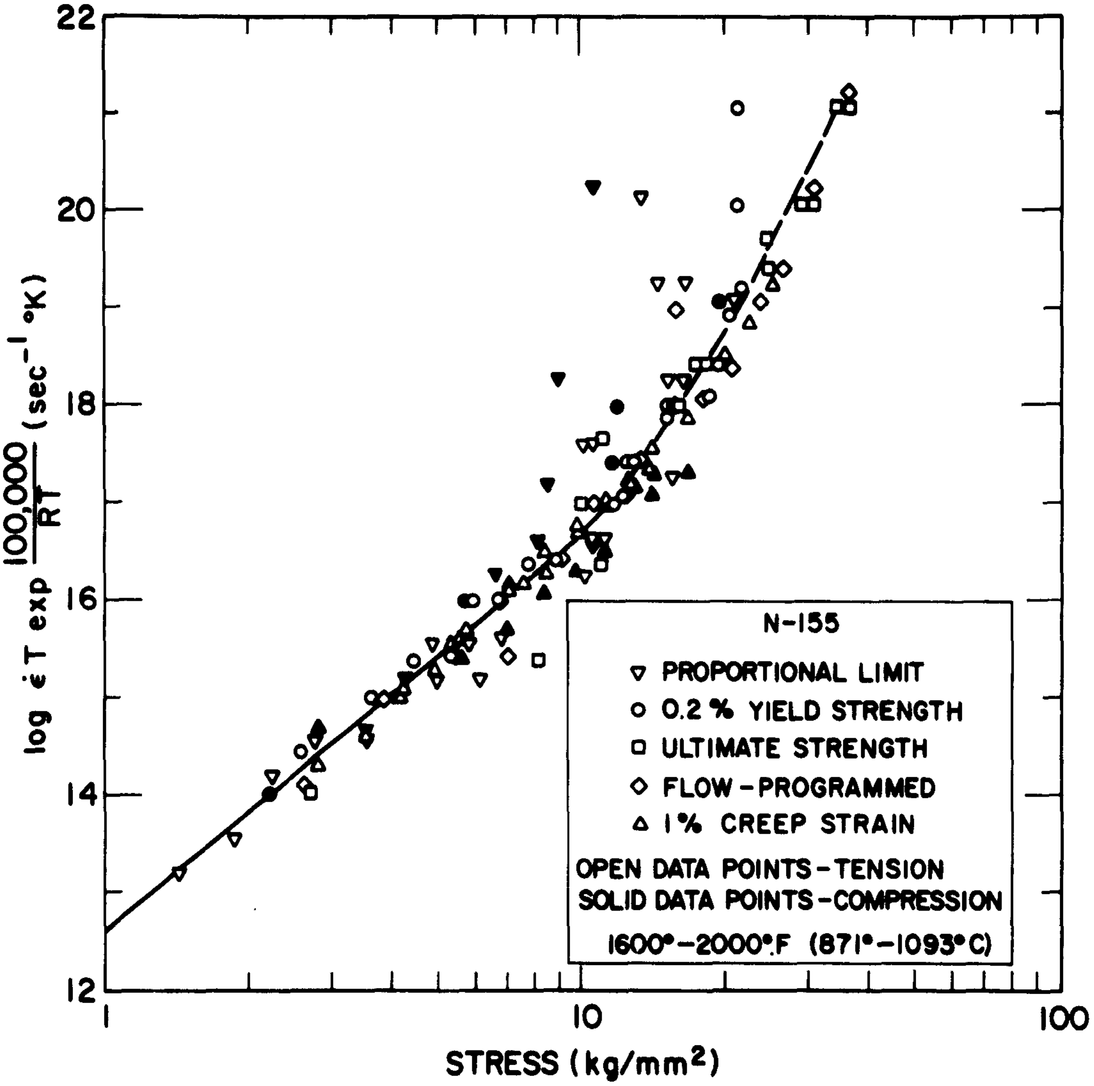


Fig. 5. Logarithm of temperature compensated deformation rate versus logarithm of stress for N-155.

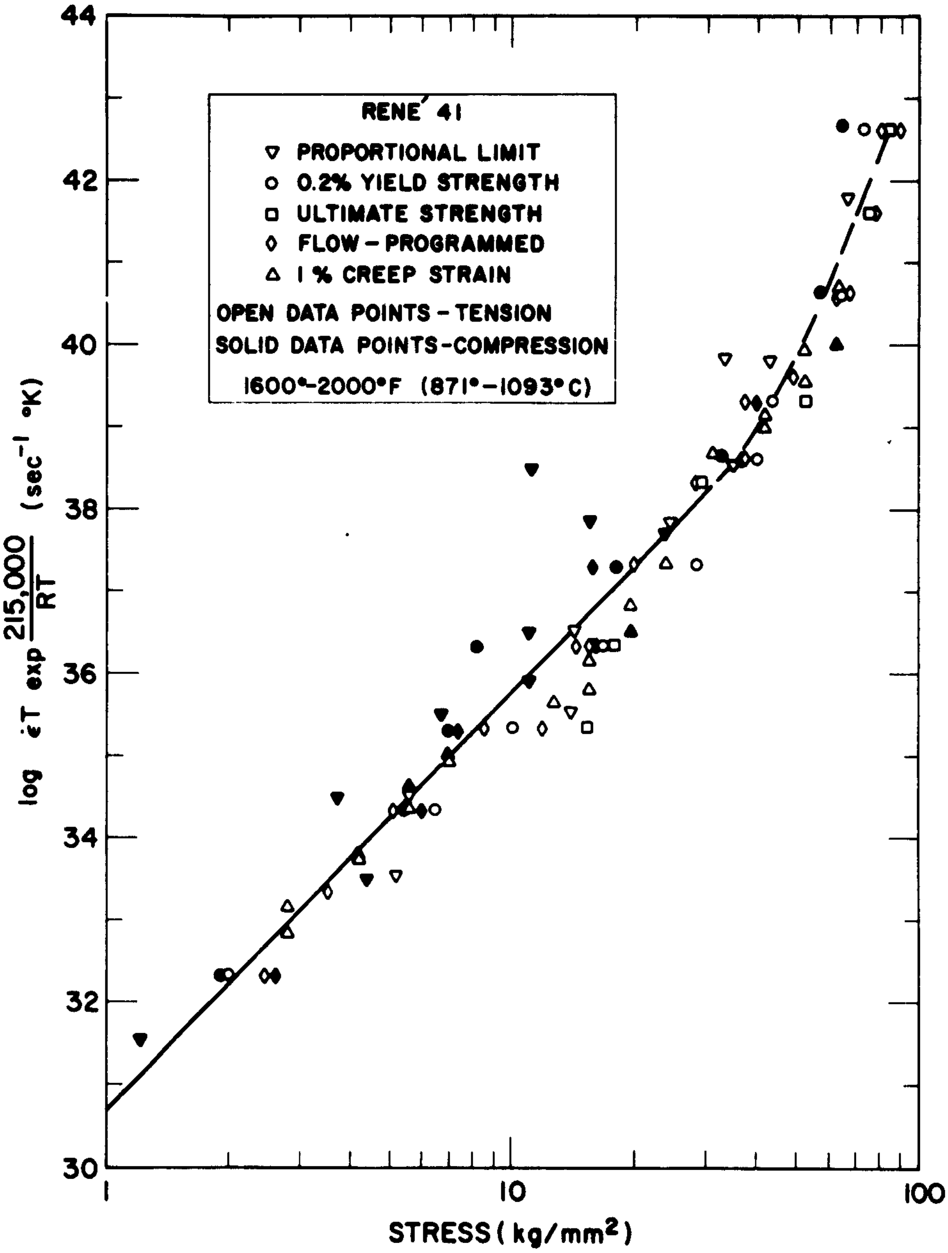


Fig. 6. Logarithm of temperature compensated deformation rate versus logarithm of stress for Rene' 41.

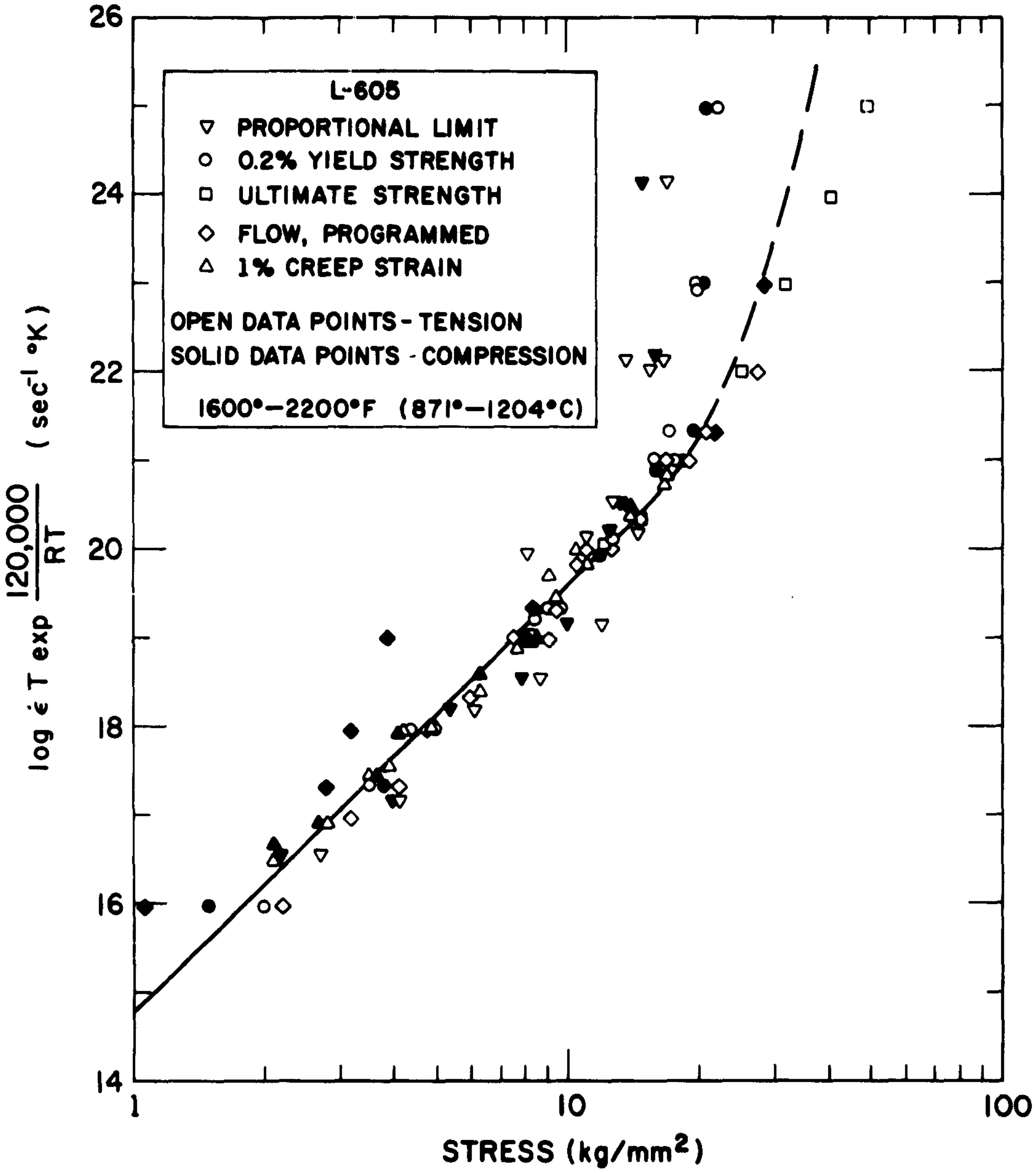


Fig. 7. Logarithm of temperature compensated deformation rate versus logarithm of stress for L-605.

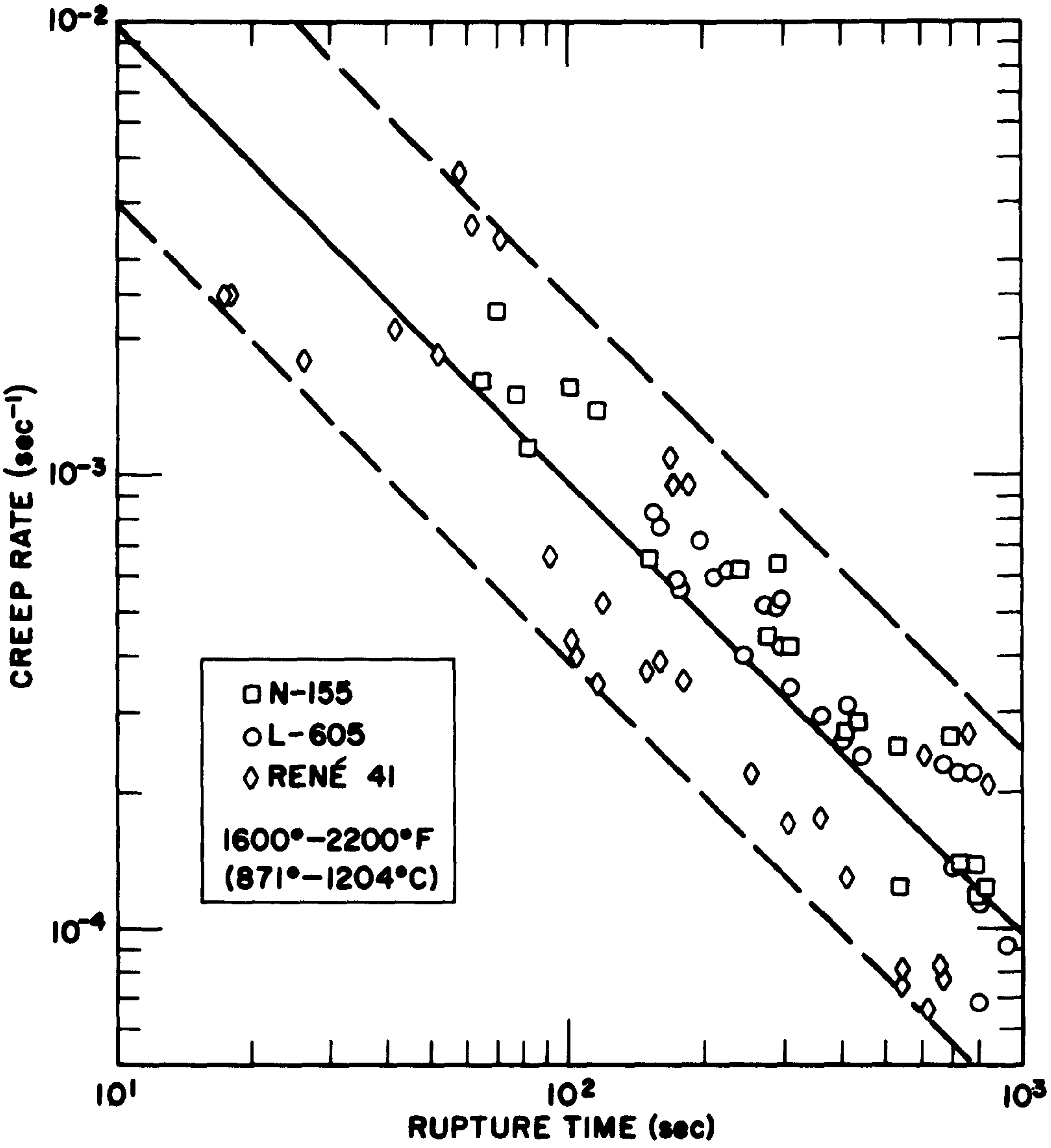
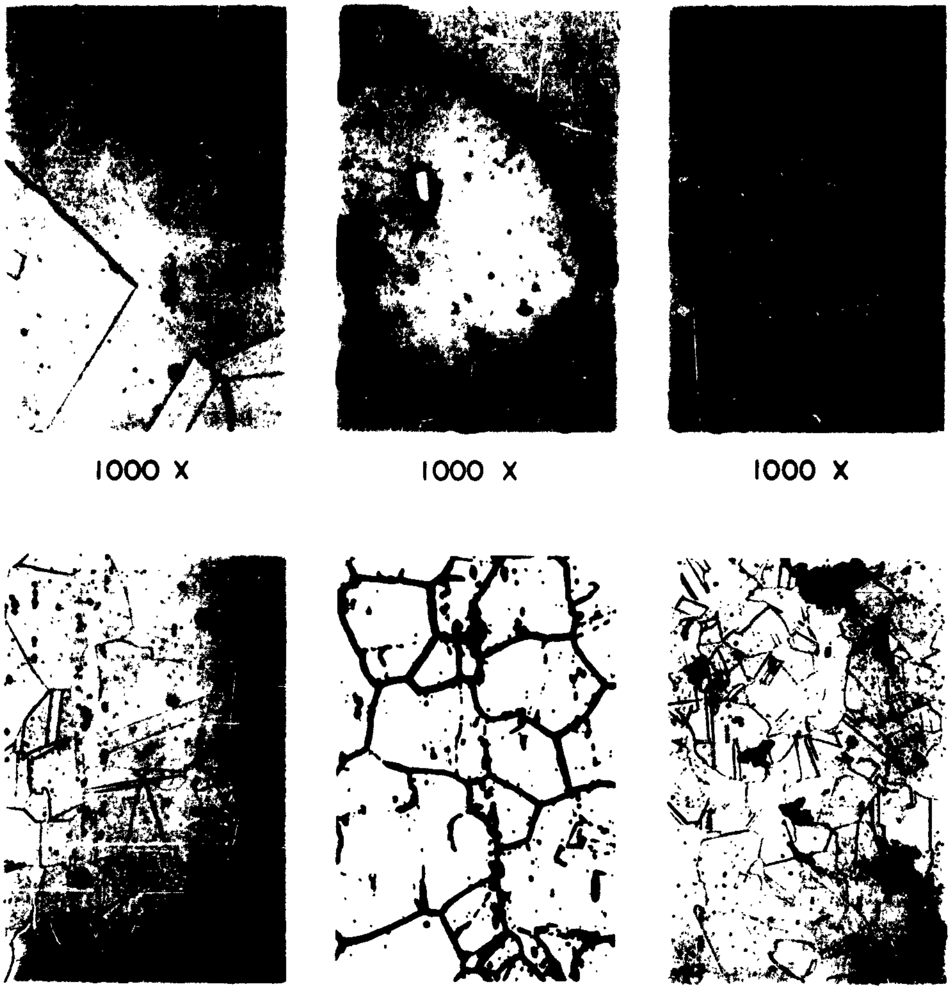


Fig. 8. Log average creep rate to 4 percent strain versus log rupture time.



AS MILL ANNEALED AT 2250°F (1232°C) AC

DPH-255

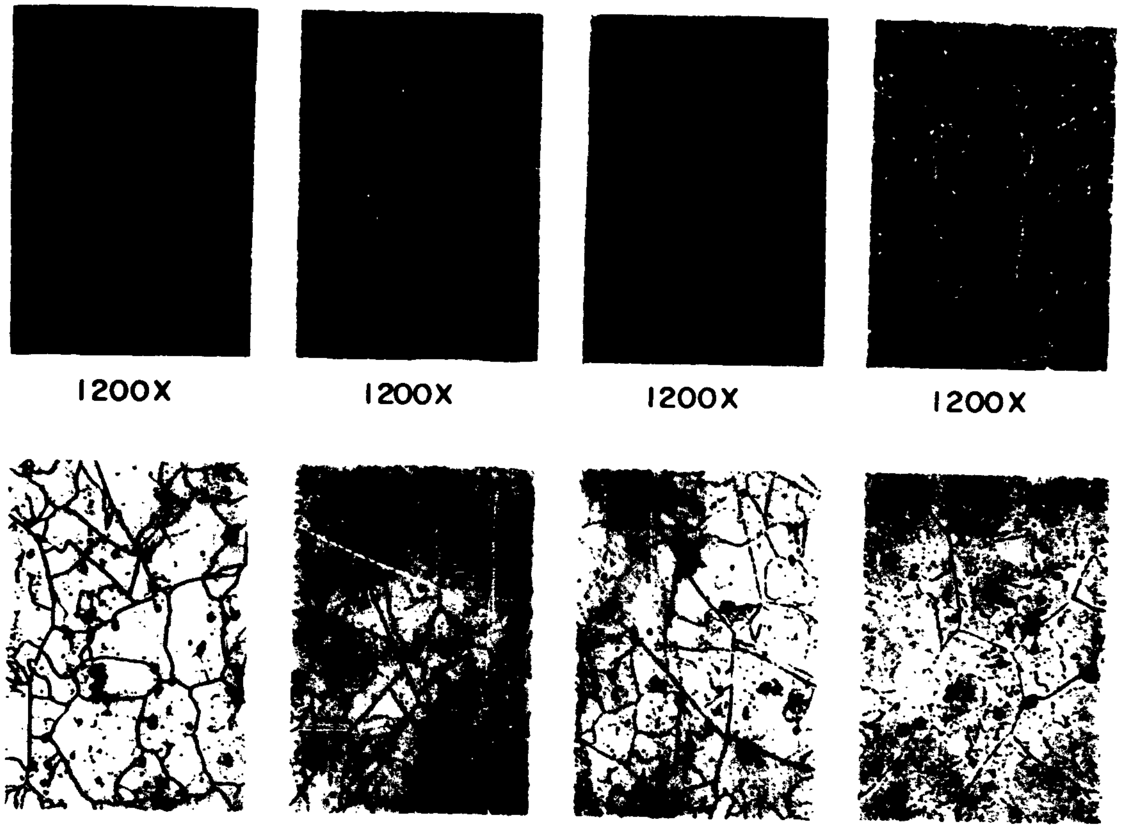
COMPRESSION TESTED 1800°F (982°C) STRAIN RATE 0.00001 sec^{-1}

DPH-250

TENSILE TESTED 2200°F (1204°C) STRAIN RATE 0.00001 sec^{-1}

DPH-255

Fig. 9. Microstructures of L-605 as annealed and tested at 1800°F (982°C) and 2200°F (1204°C).
Etch: 92 percent HCl, 5 percent H_2SO_4, 3 percent HNO_3.



AS HEAT TREATED
1/2 HR 1975°F (1079°C)
WQ 16 HR AT 1400°F
(760°C)
DPH–430

COMPRESSION CREEP
TESTED FOR 900 SEC
AT 1600°F (871°C)
DPH–420

COMPRESSION TESTED
AT 1800°F (982°C)
STRAIN RATE =
0.00001 sec^{-1}
DPH–312

COMPRESSION TESTED
AT 2000°F (1093°C)
STRAIN RATE =
0.00001 sec^{-1}
DPH–342

Fig. 10. Microstructures of René 41 as heat treated and after compression testing at 1600°F (871°C), 1800°F (982°C), and 2000°F (1093°C). Etch: electrolytic 55 percent HCl, 15 percent HNO_3, 15 percent acetic acid, 15 percent H_2O.

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