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TO

MATERIALS LABORATORY

WRIGHT AIR DEVELOPMENT CENTER

ON

AN INVESTIGATION OF THE RELATIONSHIP BETWEEN MICROSTRUCTURE AND CREEP-RUPTURE PROPERTIES OF HEAT-RESISTANT ALLOYS

by

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SUMMARY

This report covers progress made from March 15, 1957 to June 15, 1957 in an investigation concerning the relationship between microstructure and high-temperature properties of heat-resistant alloys.

The current phase of the investigation involves primarily a study of the effect of hot-working conditions on the creep-rupture properties of three types of alloys, including three ferritic steels (SAE 4340, "17-22-A"S, and "17-22-A"V), one precipitation-strengthened, austenitic alloy (A-286), and one commercially pure metal ("A" Nickel).

Rolling and creep-rupture testing conditions are reported for A-286 and "A" Nickel. Rupture data on "A" Nickel tested at 1100°F and 20,000 psi indicate a variation in rupture life from about 20 hours to somewhat greater than 1000 hours, depending on the rolling conditions. Partial data are presented for A-286 alloy rupture tested at 1200°F and 65,000 psi and at 1350°F and 40,000 psi.

The effect of austenitizing temperature (for normalizing) on the austenitic grain size is shown for "17-22-A"V steel.

INTRODUCTION

This report, the fifth progress report issued to the Materials Laboratory, WADC under Air Force Contract No. AF 33(616)-3239, covers work done from March 15, 1957 to June 15, 1957.

The purpose of this investigation is to establish basic relationships between microstructure and creep-rupture properties of heat-resistant alloys. The current objective is to establish the principles necessary to utilize conditions of hot working to produce maximum and predictable properties for service at high temperatures. The present phase of the investigation is designed to show the effect of varying the microstructure and thereby the properties by controlled hot working. The alloys under study include three ferritic materials (SAE 4340, "17-22-A"S, and "17-22-A"V), a precipitation-strengthened, austenitic alloy (A-286) and a commercially pure metal ("A" Nickel).

The interest in the relationship between working conditions and properties at high temperatures is based on two observed effects. It is now generally known that the creep-rupture properties of Ti + Al hardened heat-resistant alloys vary to a marked extent depending on hot-working conditions even after the usual standard solution and aging treatments are applied. This is an important subject because these types of alloys are practically universally used for the hot parts of aircraft power plants. In addition, the structures of alloys can be varied by hot-working conditions, in a manner not possible by any other means, offering the possibility of producing properties superior to those obtainable by simple heat treatment.

TEST MATERIALS

The alloys were supplied gratis by the following organizations: SAE 4340 from the Universal-Cyclops Steel Corporation, "17-22-A"S and "17-22-A"V from the Timken Roller Bearing Company, A-286 from the Allegheny-Ludlum Steel Corporation, and

"A" Nickel from the International Nickel Company. The chemical analyses supplied by the producers were as follows:

Alloy	C	Mn	Si	Cr	Ni	Mo	V	Fe	Other
SAE 4340 (Ht. D-14064)	0.40	0.80	0.27	0.82	1.67	0.32	---	Base	---
"17-22-A"S (Ht. 10420)	0.29	0.61	0.67	1.30	0.18	0.47	0.26	Base	---
"17-22-A"V (Ht. 11833)	0.29	0.70	0.71	1.43	0.31	0.51	0.81	Base	---
A-286 (Ht. 21030)	0.06	1.35	0.47	14.58	25.3	1.38	0.21	Base	2.00 Ti; 0.17 Al
"A" Nickel (Ht. N9500A)	0.06	0.27	0.06	---	99.46(Ni+Co)		---	0.09	0.03 Cu 0.008 S

PROCEDURE

The general procedure as outlined in the Fourth Progress Report (Ref. 1) consisted of the following sequence of operations: (1) hot rolling each material over a range of temperatures and reductions; (2) heat treating each heat treatable alloy with a standard treatment; (3) evaluating the creep and rupture properties of each alloy at appropriate temperatures for the objectives, and (4) correlating the properties with the structure as observed with electron and ordinary-light microscopy and x-ray studies.

The selection of the specific conditions of hot rolling, heat treating and creep-rupture testing depend upon the alloy. To aid in choosing rolling temperatures for A-286 and "A" Nickel, preliminary experiments were run to determine the minimum rolling temperatures for partial simultaneous recrystallization. For a 25-percent reduction in area these temperatures were found to be about 2150°-2200°F for A-286 and 1750°-1800°F for "A" Nickel. In order to obtain reductions both below and above temperatures where recrystallization would occur, rolling temperatures of 80°, 1700°, 1950°, and 2200°F were selected for A-286 and 80°, 1400°, 1600°, and 1800°F for "A" Nickel. The aimed reductions at each temperature with no reheats were set at 0,5, 10, 15, 20, and 40 percent and 0, 5, 10, 15, 20, 35, and 60 percent for A-286 and "A" Nickel, respectively. The A-286 bars were solution treated and aged after rolling while the "A" Nickel was left in the as-rolled condition.

Rolling temperatures at and below the minimum for simultaneous recrystallization are being emphasized. Previous experience has indicated that there may be a direct relationship between properties and amount of reduction whether or not recrystallization occurs during rolling. The first step is to determine if this behavior is general or the result of specific microstructural conditions which are a function of the type of alloy.

Rolling temperatures for the ferritic materials will be selected on the basis of the austenite grain size just prior to the austenite $\rightarrow$ banite transformation on cooling from rolling. Grain size versus normalizing temperature data have been obtained for "17-22-A"V and are presented in Figure 1. Grain size versus rolling temperature data will also be obtained before the final selection of rolling temperatures for testing purposes will be made for "17-22-A"V.

Tests are being run under rupture conditions (under high strain rates) and under creep conditions (slow strain rates) as a step in relating the structural conditions established by rolling which control behavior. For the same reason, tests are being conducted at relatively low and high temperatures. Prior experience indicates that the effects of rolling vary depending on these variations in creep conditions.

RESULTS AND DISCUSSION

To date, about 150 creep-rupture specimens have been prepared. Sixty tests have been completed, and 27 tests are in progress.

Commercially Pure Metal ("A" Nickel)

Rupture data have been obtained for "A" Nickel at 1100°F and 20,000 psi where successive reductions without reheating were used to obtain reductions up to about 60 percent. This defines the relative rupture times for a procedure approximating normal commercial reduction for rolling on a falling temperature. Figure 2 indicates the rupture life at 1100°F and 20,000 psi as a function of the percent reduction in area by rolling. Data on rolling conditions and ductility at rupture are presented in Table I. The structural analysis of this series of specimens has not yet been started. The first step will be to determine if the decrease in rupture life above a reduction of 20 to 35 percent for material rolled at room temperature and 1400°F was due to recrystallization during rupture testing. It is certain that recrystallization during rolling occurred only in the series rolled from 1800°F. One other important factor must be considered in interpreting these rupture data for "A" Nickel: no reheats were used during rolling, so for reductions exceeding 15-20 percent a considerable drop in temperature occurred during rolling. The introduction of strain hardening during the cooling portion of the rolling cycle is probably the explanation of the increase in strength for nickel reduced more than 20 percent at 1800°F (Fig. 2).

Inspection of the curves of Figure 2 shows that strengths started to fall off with percent reduction at about 15 to 20 percent and then increased again. This coincides with the introduction of more than two passes so that the material was being worked on a falling temperature. Previous experience on complex alloys has indicated that strengths tend to increase with percent reduction to large reductions if the material is worked on a falling temperature. If, however, it is worked at a relatively constant temperature, as is done in high production commercial equipment, the strengths fall off with reductions larger than 15 to 20 percent regardless of the temperature of reduction. The data to date suggest very strongly that if the "A" Nickel were rolled to obtain larger reductions at nearly constant temperature the strengths would also fall off at about 20-percent reduction. The next step will be to check this since it will be very important to the direction of the mechanism studies. If a relatively pure metal behaves in this manner it will establish a basic fact that such behavior is a function of the working and is not a result of the influence of working on the precipitation reactions of complex alloys.

About 30 creep tests have been completed or are in progress at 800° and 1100°F. The data from these tests have not yet, however, been correlated for report purposes.

Originally, it was planned to run both creep and rupture tests at both 800° and 1100°F. However, the proportional limit for nickel at 800°F is so low in relation to the rupture strength that excessive amounts (greater than 5 percent) of instantaneous plastic strain are introduced on loading to stresses high enough to cause rupture in 100 hours. The planned rupture tests at 800°F have, therefore, been held back because of the questionable significance of tests on specimens containing a variable mixture of the two types of prestrain.

Precipitation Strengthened, Austenitic Alloy (A-286)

The rupture testing of A-286 alloy is well under way. The tests evaluate the time for rupture at 1200° and 65,000 psi and at 1350°F and 40,000 psi. These conditions were expected to give a rupture time of about 40 hours for the weakest condition. The lower temperature, 1200°F, was selected as the temperature of widest interest for A-286 and 1350°F was added so that the properties could be evaluated for creep-rupture at more than one temperature. At 1200°F creep tests are also being conducted at 45,000 psi to establish the effect of rolling conditions on the lower-strain-rate type of creep. Initially, the tests at 1350°F are being restricted to rupture tests.

Rupture data for A-286 at 1200°F are presented in Figure 3. Table II gives all creep-rupture data obtained to date, along with the actual rolling conditions used. The data indicate a definite effect of heating to the rolling temperature which is not erased by the subsequent solution treatment at 1650° and aging at 1325°F. The effect of the amount of rolling will become clearer when more data are obtained.

Microscopic examination of the material rolled from 2200°F indicated that the amount of recrystallization during rolling varied from 0 to about 25 percent as the percent reduction was increased from 0 to 40 percent. No reheats were used during rolling.

FUTURE WORK

The next step will be to establish the behavior of "A" Nickel under wider conditions of isothermal rolling. The results of this work will be important in planning the structural studies.

The first attempt to observe the substructure in "A" Nickel will be with an etch pit technique which has been used successfully on higher-purity nickel. Electron miscroscope and x-ray studies will also be used if circumstances indicate that it would be profitable.

The use of electron microscopy is expected to be an important aid in understanding the behavior of A-286. The variation in the size and distribution of the $Ni_3(Al, Ti)$ precipitate is probably a large factor affecting the properties of A-286.

Hot rolling and testing of ferritic material will also be started immediately.

REFERENCES

1. Coldren, A. P. and Freeman, J. W. - "An Investigation of the Relationship Between Microstructure and Creep-Rupture Properties of Heat-Resistant Alloys," - Fourth Progress Report to Wright Air Development Center (December 15, 1956 to March 15, 1957).

TABLE I

ROLLING CONDITIONS AND CREEP-RUPTURE DATA FOR "A" NICKEL

Rolling Conditions			Creep-Rupture Data (a)				
emp (°F)*	Red. (%)	No. Passes	Temp(°F)	Stress (psi)	Life (hrs)	Elong. (%)	R. A. (%)
80	0	0	800	15,000	> 302.1	--	--
			800	20,000	> 982.0	--	--
			800	32,000	> 525.4	--	--
			800	35,000	> 525.6	--	--
			1100	5,000	> 934.3	--	--
			1100	8,000	>1079.6	--	--
			1100	11,000	In Progress 1850.0 hrs		--
			1100	14,000	359.0	29.1	30.7
			1100	17,000	102.1	51.8	52.8
			1100	20,000	25.1	61.6	74.8
			1200	15,000	10.2	81.5	67.3
	11.3	4	1100	11,000	I. P.	--	--
			1100	20,000	187.9	23.6	31.0
	16.5	6	1100	11,000	I. P.	--	--
			1100	20,000	269.9	27.8	31.5
	20.5	6	1100	20,000	470.5	20.9	29.8
	35.5	8	1100	20,000	442.1	30.0	42.1
	61.2	12	1100	20,000	56.3	53.7	86.5
1400	4.7	2	1100	11,000	> 861.1	--	--
			1100	20,000	43.9	40.0	58.1
	9.7	2	800	20,000	> 934.5	--	--
			1100	11,000	> 838.3	--	--
			1100	20,000	110.9	41.8	54.5
	14.7	2	1100	11,000	> 742.0	--	--
			1100	20,000	240.0	30.0	40.8
	19.5	3	1100	11,000	I. P.	--	--
			1100	20,000	265.0	30.0	40.8
	33.8	4	1100	11,000	I. P.	--	--
			1100	20,000	647.8	22.7	41.7
	58.7	6	1100	11,000	> 676.2	--	--
			1100	20,000	379.8	36.4	76.4
1600	0	0	800	16,000	I. P.	--	--
			1100	11,000	> 982.0	--	--
			1100	20,000	30.5$\pm$0.5	46.4	53.0

TABLE I (con'd.)

ROLLING CONDITIONS AND CREEP-RUPTURE DATA FOR "A" NICKEL

Rolling Conditions			Creep-Rupture Data (a)				
mp (°F)*	Red. (%)	No. Passes	Temp(°F)	Stress (psi)	Life (hrs)	Elong. (%)	R. A. (%)
1600	5.3	2	800	20,000	> 937.3	--	--
			1100	11,000	> 863.5	--	--
			1100	20,000	71.3	40.7	49.5
	9.9	2	800	24,000	I.P.	--	--
			1100	11,000	I.P.	--	--
			1100	20,000	125.2	38.2	51.0
	15.2	2	1100	11,000	I.P.	--	--
			1100	20,000	142.3	38.2	52.0
	20.3	3	800	28,000	I.P.	--	--
			1100	11,000	I.P.	--	--
			1100	20,000	180.9	28.2	56.7
	34.4	4	1100	11,000	I.P.	--	--
			1100	20,000	663.6	27.3	42.0
	59.1	6	1100	11,000	I.P.	--	--
			1100	20,000	1030.4	16.4	53.6
1800	0	0	1100	11,000	I.P.	--	--
			1100	20,000	19.5	50.9	62.9
	5.8	2	1100	20,000	72.8	43.4	49.1
	10.8	2	1100	20,000	69.9	47.3	64.3
	15.7	2	1100	11,000	I.P.	--	--
			1100	20,000	93.3	48.2	63.7
	20.7	3	1100	11,000	I.P.	--	--
			1100	20,000	75.2±1.5	53.6	63.9
	35.2	4	1100	20,000	214.3	27.3	48.6
	59.3	6	1100	11,000	I.P.	--	--
			1100	20,000	In Progress 860.0 hrs.		--

Initial temperature; no reheats were used during rolling.
Creep rates have not yet been calculated.
'.In Progress
Greater than; indicates time at which test was discontinued.

TABLE II

ROLLING CONDITIONS AND CREEP-RUPTURE DATA FOR A-286

Rolling Conditions			Creep-Rupture Data (a)				
Temp (°F)*	Red. (%)	No. Passes	Temp (°F)	Stress (psi)	Life (hrs)	Elong. (%)	R. A. (%)
80	0	0	1200	45,000	I. P.	--	--
			1200	65,000	19.1	13.6	17.6
			1350	40,000	14.1	49.1	48.4
	9.4	3	1200	65,000	27.1	10.9	15.2
			1350	40,000	17.2	41.8	52.0
	19.7	5	1200	65,000	29.9	9.1	13.8
			1350	40,000	16.3 ± 3.0		
	28.8	7	1200	65,000	31.0	13.6	16.0
			1350	40,000	19.9	49.1	52.5
	39.5	9	1200	65,000	20.7	10.0	13.8
			1200	45,000	I. P.	--	--
			1350	40,000	15.2		
1700	0	0	1200	65,000	30.5	10.0	9.8
			1350	40,000	18.9	39.1	47.5
	3.9	1	1200	65,000	38.5	16.3	13.3
			1350	40,000	21.0	37.2	43.9
	8.4	2	1200	65,000	(b)		
			1350	40,000			
	11.5	3	1200	65,000			
			1350	40,000	24.5		
	17.7	3	1200	65,000			
			1350	40,000			
	37.6	5	1200	65,000			
			1350	40,000			
1950	0	0	1200	65,000	127.2	6.4	4.8
			1350	40,000			
	3.9	1	1200	65,000	143.2		
			1350	40,000	66.3	23.6	24.9
	9.7	2	1200	65,000			
			1350	40,000	48.9		
	12.2	3	1200	65,000			
			1350	40,000			

TABLE II (con'd.)

ROLLING CONDITIONS AND CREEP-RUPTURE DATA FOR A-286

Rolling Conditions			Creep-Rupture Data (a)				
emp (°F)*	Red. (%)	No. Passes	Temp (°F)	Stress (psi)	Life (hrs)	Elong. (%)	R.A. (%)
	18.8	3	1200	65,000			
			1350	40,000			
	38.7	5	1200	65,000			
			1350	40,000			
2200	0	0	1200	65,000	41.2	2.7	5.6
			1350	40,000	128.1		
	5.4	1	1200	65,000	80.7	2.7	4.8
			1350	40,000	72.4	8.0	10.3
	10.4	2	1200	65,000	91.5	2.7	4.0
			1350	40,000	50.8	8.2	8.7
	14.5	3	1200	65,000	125.8	4.5	4.8
			1350	40,000	62.2	7.3	9.8
	20.8	3	1200	65,000	132.2	4.5	6.0
			1350	40,000	66.6		
	39.8	5	1200	65,000	186.2	4.5	5.2
			1350	40,000	94.4		

Initial temperature; no reheats were used during rolling.
Creep rates have not yet been calculated.
Blank spaces indicate data not yet available.

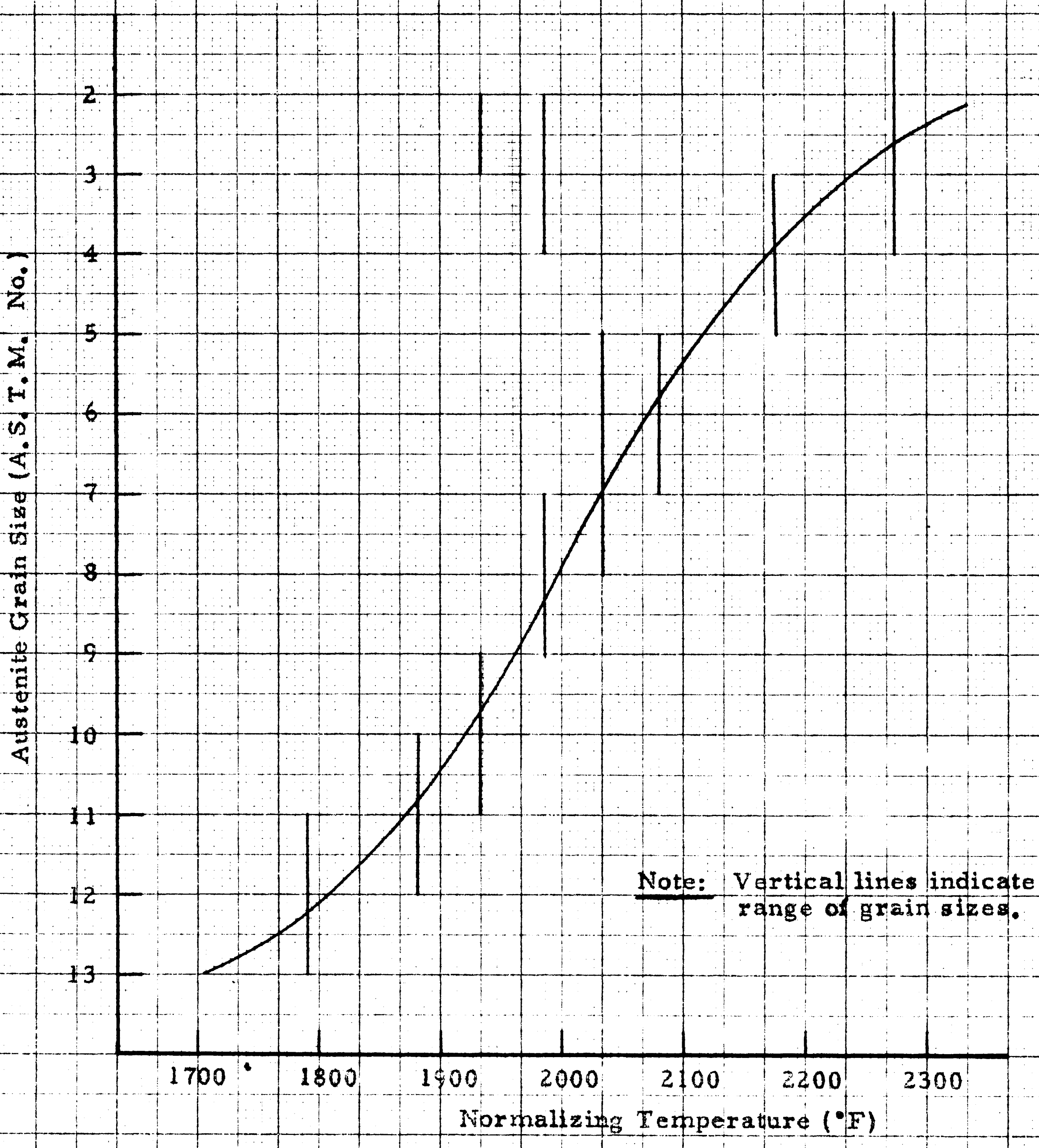

Figure 1. - Variation of Prior Austenite Grain Size with Normalizing Temperature for "17-22-A"V Steel. The specimens were air cooled after one hour at temperature and then tempered for one hour at 1200°F to reveal the prior austenite grain boundaries.

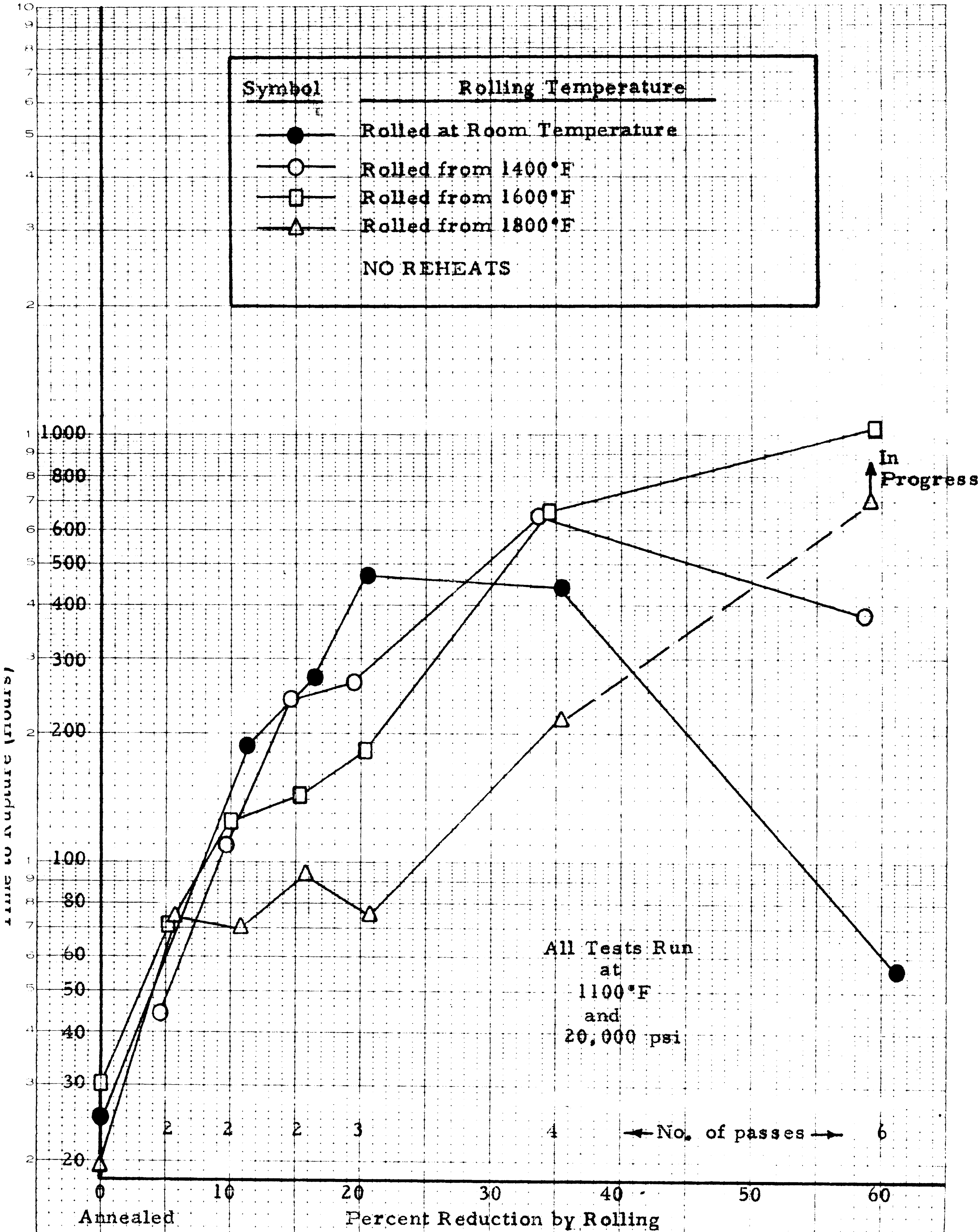

Figure 2. - Variation of Rupture Life with Rolling Conditions for "A" Nickel Tested at 1100°F and 20,000 psi.

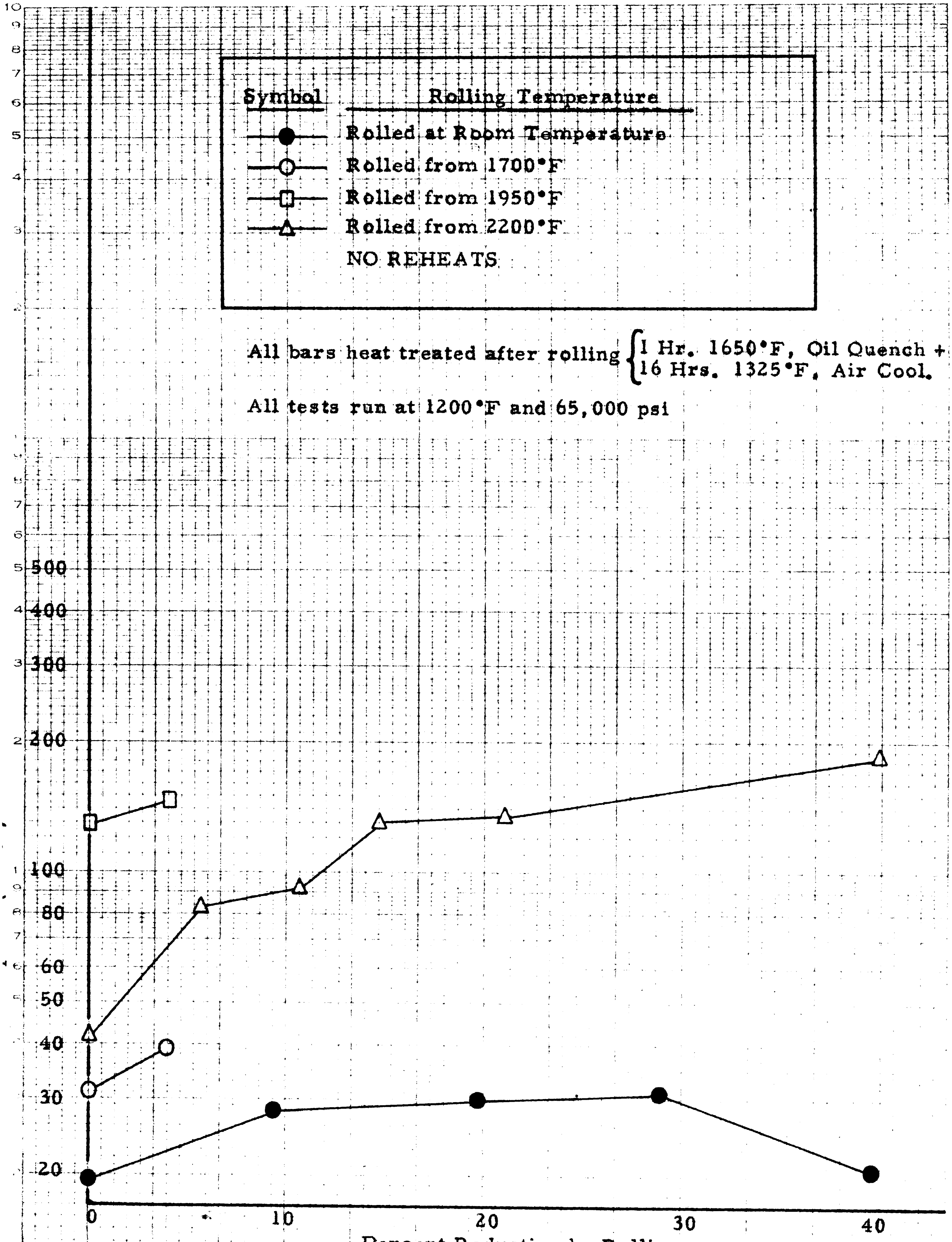

Figure 3. - Variation of Rupture Life with Rolling Conditions for A-286 Alloy Tested at 1200°F and 65,000 psi.