The University of Michigan • Office of Research Administration
Ann Arbor, Michigan

A SURVEY OF CREEP-RUPTURE PROPERTIES AT 1100°F OF 2 1/4 Cr 1 Mo STEEL TUBES MADE BY MICHIGAN SEAMLESS TUBE COMPANY

by

R. Jackowski
J. W. Freeman

Project 05007-30-T

July 31, 1964

MICHIGAN SEAMLESS TUBE COMPANY
400 WEST AVENUE
SOUTH LYON, MICHIGAN

A SURVEY OF CREEP-RUPTURE PROPERTIES AT 1100°F OF 2 1/4 Cr1Mo STEEL TUBES MADE BY MICHIGAN SEAMLESS TUBE COMPANY

The Michigan Seamless Tube Company produces 2 1/4 Cr 1Mo steel tubes for use in superheaters and other high temperature applications. This report presents the results of a limited number of creep-rupture tests at 1100°F designed to survey the high temperature strength of the tubing. The data obtained were to be used to select tubes for the 5000 to 10,000 hour test now required for evaluation of superheater tubing.

The samples were taken from production processed tubing so that the data would be representative of the tubes as supplied to customers. This insured that effects of prior processing, if any, unique to Michigan Seamless Tubing Company production would thereby be included in the investigation.

Samples of 14 tubes were supplied. The production of all the tubing involved a cold working process prior to a final heat treatment. Of the 14 tubes, nine were annealed at 1450°F, two were normalized and tempered, and two were annealed at 1750°F. Tests were not carried out on a quenched and tempered tube.

The steel for manufacturing the tube is purchased from steel producers. The investigation accordingly included tubes from different suppliers.

A major objective of the investigation was to determine if the tubing as produced had the properties expected by the Table P7 stresses of the Power Boiler (Section I) Code of the ASME Boiler and Pressure Vessel Committee. While prolonged rupture tests were not included, the data were extrapolated in accordance with the general trend of data for 2 1/4 Cr 1Mo steel to obtain approximate values for comparison with the Code values. The heat treatment preferred by Michigan Seamless Tube Company was an anneal at 1450°F. During the course of the investigation, the heat treatment clause in ASTM Specification A213 was changed, to cast doubt on the treatment at 1450°F meeting the requirements. When this became known, prolonged tests were postponed pending clarification of the heat treatment.

SUMMARY AND CONCLUSIONS

Survey type rupture tests at 1100°F were carried out on 14 samples of production superheater tubing from Michigan Seamless Tube Company. Extrapolation of the stress-rupture time curves to 100,000 hours indicated the following approximate rupture strengths (psi):

	Maximum	Average	Minimum	Number of Tubes Tested
Annealed 1450°F	9600	7080	6000	9
Normalized and Tempered	---	7400-6600	---	2
Annealed 1750°F	---	7000-6800	---	2
ASTM STP No. 151	8500	7700	6350	Limited tests on barstock
ASME Boiler Code Stress	---	4200	---	Based on "average" creep strength

The present Code stress of 4200 psi was based on an average stress of 4200 psi for 0.01 percent per 1000 hours creep rate. This was the controlling factor in that 60 percent of the average and 80 percent of the minimum stress for rupture in 100,000 hours were both higher than 4200 psi. The above tabulation shows that 80 percent of minimum rupture strengths were above 4200 psi. The average strengths were somewhat below the average used in evaluating data for the present Code stress but certainly well within the range of strengths.

One prolonged creep test at 4200 psi was conducted on a sample from Tube 1 (Ann. 1450°F). This test indicated a stress of 3800 psi for a minimum creep rate of 0.01 percent per 1000 hours. This is slightly below but certainly within the range of rates in which the Code stress of 4200 psi was set. It is, however, "low" in that prolonged testing usually results in creep strengths considerably higher than those of the short time tests on which the present Code stress was set.

The tubes annealed at 1450°F had a wide range in rupture strengths including high values for Tubes 12, 13 and 14 which were specially processed to produce high rupture strengths. It also resulted in the lowest strength measured. Normalizing and tempering or annealing at 1750°F resulted in near average values.

Microstructures varied sufficiently to preclude their use as a means of identifying high or low strength material. The source of steel showed no significant effect although the data were too few to be certain.

DESCRIPTION OF TUBES

The information supplied describing the samples of tubes submitted is summarized in Table 1. Most of the tubes were finished by cold working and "annealing" at 1450°F. Two tubes (No.'s 6 and 9), however, were given a "full" anneal at 1750°F and two tubes (No's. 2 and 10) were normalized and tempered. Tube No. 11 was submitted after water quenching from 1700°F and tempering at 1450°F, although no testing was carried out in view of the general concensus that tempered martensite would have poor properties.

Tube 2 was made by normalizing and tempering Tube 1 stock which had been prior annealed at 1450°F. Tubes 4 and 5 were tested to compare material for tubes from two sources of supply.

Tube 6 was included to provide comparative data for full annealing using the same material as Tube 5.

Tubes 7 through 11 were submitted to evaluate manufacturing variables using one heat of steel. They included tube reducing versus cold drawing, as well as heat treatment variables.

Tube 7 had both an unusual microstructure and an unusual stress-rupture time curve. Tube 12 was submitted to check the unusual characteristics of Tube 7.

Tube 12 had unusually high rupture strength. Tubes 13 and 14 were submitted to determine if the high strength of Tube 12 could be duplicated.

RESULTS

The data from the rupture tests at 1100°F are given in Table 2. The stress-rupture time curves defined by the data are shown as Figures 1 through 3. Plates 1 through 14 show the microstructures prior to testing.

Boiler Code stresses at temperatures where creep controls strength are based on the following properties:

(1) The stress for rupture in 100,000 hours.

(2) The stress for a creep rate of 0.00001 percent per hour (usually extrapolated as the stress for a creep rate of 0.01 percent per 1000 hours and sometimes as the stress for 1 percent creep in 100,000 hours).

The Code stress is the lowest of the following:

(a) 60 percent of the average stress for rupture in 100,000 hours.

(b) 80 percent of the minimum stress for rupture in 100,000 hours.

(c) 100 percent of the average creep strength for 0.01 percent per 1000 hours.

Accordingly, the stress-rupture time curves obtained were extrapolated to 100,000 hours, recognizing that the testing was less than that considered necessary for reliable extrapolation to 100,000 hours. The extrapolations were based on the curves defined by the test points, a reasonably reliable procedure because 2 1/4 Cr.1Mo steel usually has stress-rupture time curves which do not change slope.

The rupture strengths for 100,000 hours derived by extrapolation are given in the following tabulation:

Tube No.	Heat Treatment	Extrapolated 100,000-hour Rupture Strength (psi)
1	Anneal at 1450°F	6400
3	Anneal at 1450°F	7500
4	Anneal at 1450°F	6000
5	Anneal at 1450°F	6400
7	Anneal at 1450°F	6700
8	Anneal at 1450°F	6200
12	Anneal at 1450°F	9600
13	Anneal at 1450°F	7000
14	Anneal at 1450°F	8000
Average	Anneal at 1450°F	7080
2	Normalize 1650° - temper 1375°F	6600
10	Normalize 1750° - temper 1375°F	7400
6	Annealed 1750°F	7000
9	Annealed 1750°F	6800
11	Water quench 1700°F + temper at 1450°F	No tests.

The comments regarding these rupture strengths are as follows:

1. The range in strengths for rupture in 100,000 hours obtained by extrapolation are as follows:

Annealed at 1450°F	-----	6000 to 9600 psi	(9 tubes)
Full annealed 1750°F	-----	6800 to 7000 psi	(2 tubes)
Normalized and tempered	-----	6600 to 7500 psi	(2 tubes)

These data would seem to indicate that annealing at 1450°F can produce as high or higher rupture strength as a full anneal or a normalize and temper as well as very low strengths.

2. The range of rupture strengths for an anneal at 1450°F apparently is not a "statistical" scatter but involves changes in prior processing to alter properties when heat treated at 1450°F.

3. Tubes 13 and 14 were submitted to verify that the high strength of Tube 12 could be reproduced. They were higher than average but below the

high value for Tube 12.

4. A full anneal of Tube 6 at 1750°F compared to Tube 5 with a 1450°F anneal only raised the strength from 6400 to 7000 psi. This is hardly a significant change. In the case of Tube 9, the anneal again resulted in only slightly higher strength than for Tubes 7 and 8 from the same heat. There is only a slight superiority indicated for the full anneal but it should be noted that in both cases it led to a higher strength than the 1450°F treatment.

5. Normalizing and tempering of Tube 1 material after the original treatment at 1450°F to produce Tube 2 material hardly changed the strength at long time periods. It did, however, raise strengths at short time periods (See Fig. 3). Tube 10 had a 100,000 hour strength of 7400 psi while Tube 7 and 8 annealed at 1450°F or Tube 9 annealed at 1750°F had slightly lower strengths. Again short time strengths were high for normalizing and tempering.

6. Tubes 4 and 5 did not show a significant difference between suppliers of steel. Tube 14 also did not show a significant difference which could be attributed to the steel producer.

7. Tube 7 had a microstructure (Plate 5) of fine pearlite in coarse grains. This was a considerably different structure than the other tubes heat treated at 1450°F. The stress-rupture time curve was also high at short times with a relatively steep slope reducing long time strength.

8. Tube 12 made as a check on Tube 7 had the usual microstructure expected (Plate 7) for annealing at 1450°F after cold work. The available data showed less slope and extrapolated to the highest stress for rupture in 100,000 hours of all tubes tested.

9. Tubes 13 and 14 were used to check the reproducibility of the high strength of Tube 12 using steel from two suppliers. The results of the tests resembled those of Tube 7 more than they did those for Tube 12 - i.e., higher strengths at short time periods and steeper curves than the other curves for tubes heat treated at 1450°F. The duplication was not too good although the extrapolated stresses for rupture in 100,000 hours were higher than the other tubes heat treated at 1450°F.

Creep Test

A creep test on a specimen from Tube 1 was run for 4242 hours (Fig. 4) at 1100°F under 4200 psi, the present Code stress. At this time the creep rate had become essentially constant (Fig. 5) at 0.000015 percent per hour (0.015 percent per 1000 hours). The creep rates used in Fig. 5 were obtained from a working curve drawn to a scale more consistent with the sensitivity of the creep measuring equipment than could be drawn in the condensed scale of Fig. 4. Extrapolation of this rate to 0.01 percent per 1000 hours by using a curve with a slope usually exhibited by this steel (Fig. 6) indicates a creep strength of 3800 psi. The error in this procedure should be small because the rate of the actual test was so close to 0.01 percent per 1000 hours. Because the creep rate had not changed for a considerable period of time, continuing the test to longer times would not have changed the strength significantly.

Microstructures

The microstructures of the tubes (Plates 1 through 14) indicate the following:

1. The material annealed at 1650°F prior to cold working and annealing at 1450°F had a structure of fine ferrite grains slightly elongated along the axis of the tube with spheroidized carbides (Plate 1).

2. When annealed at 1450°F prior to cold working and then again annealed at 1450°F after cold working resulted in considerable variation in microstructures. Tube 3 (Plate 2) had a structure which was a combination of coarse ferrite grains and what appeared to be tempered bainite. Tubes 4 and 5 had structures (Plates 3 and 4) similar to Tube 3. Tube 7, however, had a structure (Plate 5) that appeared to be bainite or extremely fine pearlite in ferrite. Tube 8 (Plate 6) had a structure of ferrite plus highly spheroidized carbide particles. The only reported difference between Tubes 7 and 8 was cold drawing rather than tube reducing prior to the final heat treatment at 1450°F. Tube 12 (Plate 7) was more like Tube 8 than Tube 7 although there were a few small areas of the "bainite like" structure in Tube 12. Tubes 13 and 14 (Plates 8 and 9), however, had structures similar to Tube 7.

3. Normalizing Tube 2 at 1650°F and tempering at 1375°F produced a structure (Plate 10) of fine ferrite grains with small areas of spheroidized

carbides which apparently had formed in the austenite transformed during treatment at 1650°F. Evidently 1650°F was below the upper critical temperature and transformation was only partially complete. Increasing the normalizing temperature to 1750°F (Tube 10 and Plate 11) evidently caused complete transformation to austenite. On air cooling this produced a structure of bainite or very fine pearlite which was not appreciably changed by the temper at 1375°F.

4. A full anneal of Tube 6 at 1750°F resulted in fine ferrite, some imperfect pearlite grains and extensive carbide spheroidization (Plate 12). Tube 9 (Plate 13) had a considerably coarser grain size and no evidence of pearlite. For reasons not understood from the available information, there was a marked difference in microstructure for these two full annealed tubes.

5. As expected the structure of Tube 11 (Plate 14) was tempered martensite. The structures seem to indicate that the 1700°F treatment caused complete transformation before quenching to martensite. The treatment at 1450°F tempered the martensite and caused precipitation and some agglomeration of carbides.

The number of variations in the microstructures of the tubes for a heat treatment at 1450°F indicates prior history effects.

The variation in structure for the two normalized and tempered tubes was not unexpected due to the difference in normalizing temperature. Also the normalize was applied to tubing which had primarily been annealed 1450°F for Tube 2 while it was applied to a cold worked structure for Tube 10.

The difference in microstructure for the two fully annealed tubes was unexpected. It must reflect a difference in response to heat treatment between heats if the conditions of heat treatment were the same as the data indicate.

Reviewing these microstructures in relation to the results of the rupture tests seemingly leads to two conclusions:

(1) Rupture strengths could not be predicted from the microstructures.

(2) Microstructures would not identify the heat treatments. It would only be fair, however, to point out that the reported conditions of heat treatment also did not define the rupture strengths.

DISCUSSION

The investigation was undertaken with the objective of defining the creep-rupture strength of tubes made for superheater service by Michigan Seamless Tube Company under ASTM Specification A213. Fourteen samples of tubing were submitted. All were hot pierced and hot rolled, annealed, cold worked to final size and then finally heat treated. Nine of the samples were annealed at 1450°F as a final heat treatment; two tubes were normalized and tempered; two tubes were full annealed at 1750°F; and one was quenched* and "tempered" at 1450°F.

The survey rupture tests at 1100°F gave data which can be evaluated as follows:

	Extrapolated 100,000-Hour Rupture Strengths (psi)			
	Maximum	Average	Minimum	
Annealed 1450°F	9600	7080	6000	9 tubes
Normalized + Tempered		7400-6600		2 tubes
Full annealed 1750°F		7000-6800		2 tubes
ASTM STP No. 151	8500	7700	6350	Limited tests on barstock
Present Code Stress		4200	(based on "average" creep strength)	

The 100,000-hour rupture strengths at 1100°F for the tubes annealed at 1450°F cover the range for the steel. The data for Tubes 12, 13 and 14 suggest that prior history control might keep strengths at or above average. The low values are within the range which the present Code stress would accept (i.e., 60 percent of the "average" for tubes treated at 1450°F was 4248 psi and 80 percent of the minimum was 4800 psi. From this viewpoint the tubes treated at 1450°F had rupture strength meeting the Code stress minimum criteria.

*No tests were conducted on the quenched tube due to the general opinion that tempered martensite would have low strength.

The tubes which were full annealed or normalized and tempered had near average and well above minimum rupture strengths.

The 3800 psi creep strength after annealing at 1450°F for the only tube tested was on the low side of the range for creep strength in STP No. 151 but within the range of data. One creep test does not define the creep strength for treatment at 1450°F and therefore the significance of the test is uncertain.

Another objective of the tests was to survey the product and select tubes for the 10,000-hour rupture and creep tests generally considered necessary to properly determine superheater tube strengths. The alteration in the heat treatment clause of the ASTM A213 specification after the investigation was started and the subsequent disagreements in interpretation had been a major factor in not proceeding with the prolonged tests. It should be recognized, however, that for T22 material the justification for requiring an anneal from near or above the upper critical temperature was largely based on creep strengths (not rupture strengths). In this report the relatively low strength of the one creep test on Tube 1 supports the probability of creep strength on the low side of the range for sub critical temperature heat treatments after cold work. This again is subject to the qualification that Tubes 12, 13 and 14 suggest that there are conditions of prior treatment which could if properly controlled yield average or better strengths.

The lack of a correlation of microstructure with either heat treatment of creep-rupture properties suggests that factors other than heat treatment were influencing properties. This would not be surprising for annealing at 1450°F. It was, however, surprising for the other two heat treatments.

Examination of the data show no significant differences in tubes made from steel from two sources.

Table 1

DESCRIPTION OF SAMPLES OF TUBES SUBMITTED

Tube No.	Heat No.	Tube Size Dia. (in.)	Tube Size Wall (in.)	Heat Treatment	C (%)	Mn (%)	Si (%)	Cr (%)	Mo (%)	P (%)	S (%)
					Chemical Composition Reported						
1	3341384	2	0.404	Annealed 1450°F[(a)]	.13	.44	.28	2.22	1.02	.010	.017
2	3341384	2	0.404	Norm. 1650°+Temp. 1375°F[(b)]	.13	.44	.28	2.22	1.02	.010	.017
3	?	2 3/8	0.404	Annealed 1450°F[(a)]				?			
4	3320643	2 1/8	0.471	Annealed 1450°F[(a)]	.11	.44	.31	2.25	1.02	.010	.008
5	27218[(e)]	2 1/8	0.471	Annealed 1450°F[(a)]	.11	.46	.30	2.31	0.94	.013	.025
6	27218[(e)]	2 1/8	0.471	Annealed 1750°F[(c)]	Same as Tube No 5.						
7	3341339	2 1/8	0.450	Cold drawn+1450°F[(a)]	.11	.42	.29	2.23	0.95	.008	.013
8	3341339	2 1/8	0.450	Tube reduced+1450°F[(a)]	.11	.42	.29	2.23	0.95	.008	.013
9	3341339	2 1/8	0.440	Cold drawn+Ann. 1750°F[(c)]	.11	.42	.29	2.23	0.95	.008	.013
10	3341339	2 1/8	0.450	Cold drawn+Norm. and Temper.[(d)]	.11	.42	.29	2 23	0.95	008	.013
11	3341339	2 1/8	0.450	Cold drawn+Water quenched 1700°F+ temper. 1450°F	11	.42	.29	2.23	0.95	.008	.013
12	3342219	2 1/8	0.452	Duplicates No. 7	.105	.47	.30	2.27	0.96	.008	.012
13	332108	2 1/8	0.452	Duplicates No. 12	.105	.45	.33	2.29	0.98	.008	.016
14	30558[(e)]	2 1/8	0.452	Duplicates No. 12	.11	.45	.32	2.25	0.90	.013	.025

(a) Cold worked tube was heated to 1450°F, 1 hour at heat and air cooled.
(b) Tubing heat treated at 1450°F from the same lot as Tube No. 1 was reheated to 1650°F for 1 hour and air cooled plus tempered at 1375°F for one hour, air cooled.
(c) Cooled at 50°F per hour from 1750°F.
(d) Fast cooled from 1750°F+tempered at 1375°F for one hour.
(e) Tubes 5, 6 and 14 were made from steel from a different steel producer than the other tubes.

Table 1 (continued)

Tube No.	Ultimate Strength (psi)	Yield Strength (psi)	Elongation (% in 2")	Hardness Rockwell "B"	Piercing and Rolling	Heat Treatment Prior to Cold Working to Size (°F)	(hours)	(cooling)
1	67,600	34,700	57	72	>1800°F	1650	3	50°F/hr. to 1100°F
2	69,300	46,600	65.6	76/77	Same as 1 except reheat treated.			
3	68,700	40,100	64.8	75/77	>1800°F	1450	1	Air cooled
4[a]	69,000	50,800	57	74/75	to 2 7/8" x 9/16"	1450	1	Air cooled
5[a]	70,000	48,700	54.6	77/78	to 2 7/8" x 9/16"	1450	1	Air cooled
6					Same as Tube No. 5 except final heat treatment.			
7-10	84,400[b]	58,700	53.1	72/76				
11	78,200	63,200	53.9	85				
12	72,100	41,000	56.2	72/74				
13	84,200	61,600	48.4	85	Same as No. 12			
14	84,000	56,600	53.9	85	Same as No. 12			

(a) To compare tubes of steel, from two suppliers, made the same way.
(b) May be heat certification properties.

Table 2

STRESS-RUPTURE TIME DATA AT 1100°F FOR
MICHIGAN SEAMLESS TUBE COMPANY 2 1/4 Cr - 1 Mo STEEL TUBING

Tube No.	Stress(psi)	Rupture Time(hours)	Elongation (% in 2 inches)	Reduction of Area (%)
		Heated for one hour at 1450°F, air cooled.		
1	20,000	25.9	63.5	87.0
	17,000	63	72.0	84.5
	15,000	155	71.0	84.5
	13,000	560	74.5	81.0
	11,000	1664	62.5	77.0
3	17,000	86.2	47.0	83.0
	15,500	174	71.0	84.0
	14,000	562	52.5	81.0
	12,500	1187	69.5	83.5
4	16,000	106	54.0	59.0
	14,800	151	45.5	60.0
	13,000	401	39.0	55.5
5	16,000	187	38.5	53.5
	14,500	280	33.5	46.5
	13,500	410	35.5	49.5
	12,000	974	38.5	50.0
7	19,000	238	49.0	80.0
	17,000	384	68.5	81.0
	15,500	814	55.5	77.5
8	17,000	79.4	67.0	87.5
	14,000	265	59.0	86.5
	12,000	1005	68.5	86.5
12	16,000	123	51.0	55.5
	14,500	449	50.0	70.0
	13,500	1063	56.0	62.5
13	16,500	369	59.5	65.5
	14,500	905	41.0	66.0
	13,500	1516	45.0	61.5
14	16,500	864	43.5	63.5
	14,500	1575	43.5	67.5
	13,500	3225	36.5	56.0

Table 2 (continued)

Tube No.	Stress(psi)	Rupture Time(hours)	Elongation (% in 2 inches)	Reduction of Area (%)
Heated for one hour at 1650°F, air cooled and tempered for one hour at 1375°F, air cooled.				
2	17,000	180	55.0	81.0
	15,000	393	58.0	82.5
	12,000	1924	55.0	74.0
Heated for one hour at 1750°F, air cooled and tempered for one hour at 1375°F				
10	17,000	299	60.5	85.0
	15,000	726	49.0	82.5
	14,000	1181	50.5	83.0
Furnace cooled 50°/hour from 1750°F				
6	17,000	70.5	85.0	76.5
	14,000	335	59.5	70.5
	12,500	799	60.0	61.0
9	17,000	62.2	76.5	91.5
	14,000	305	72.0	89.0
	12,500	697	62.5	90.0

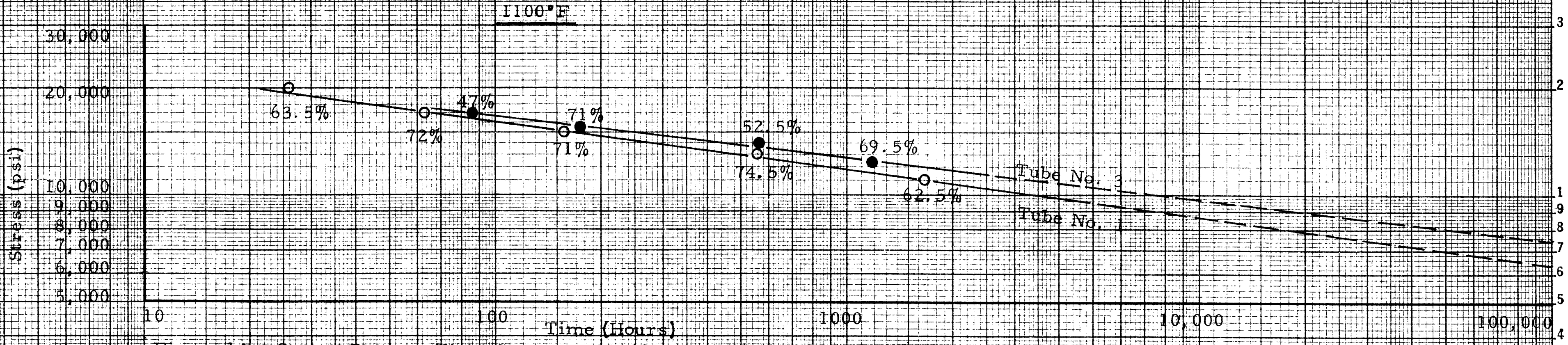

Figure 1A. Stress-Rupture Time Curves at 1100°F for 2 1/4 Cr 1 Mo Tubes Annealed at 1450°F.

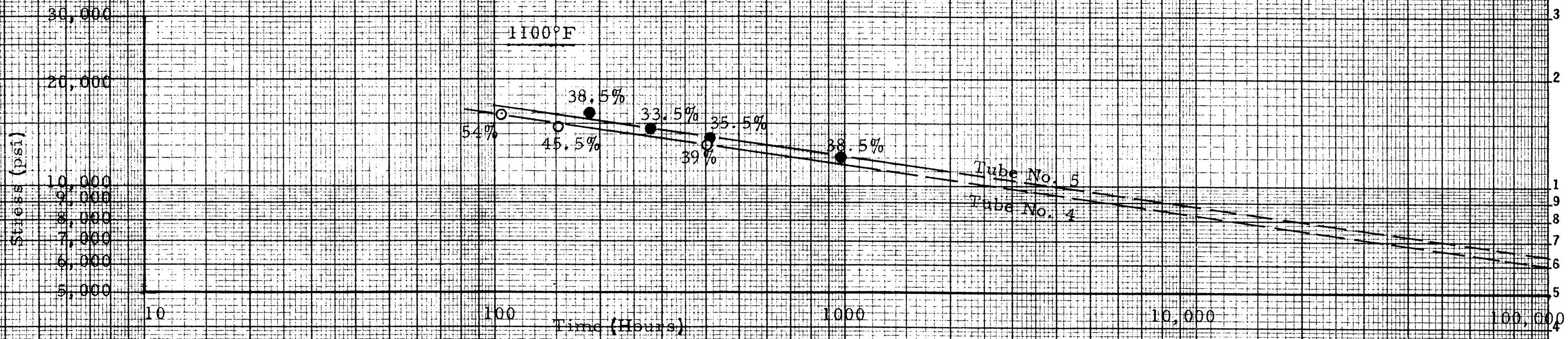

Figure 1B. Stress-Rupture Time Curves at 1100°F for 2 1/4 Cr 1 Mo Tubes Annealed at 1450°F.

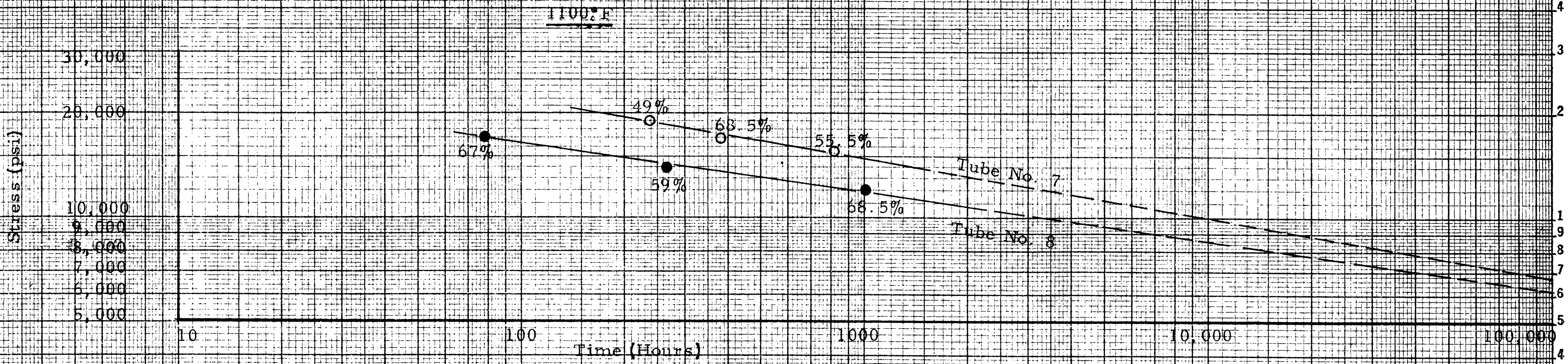

Figure 1C. Stress-Rupture Time Curves at 1100°F for 2 1/4 Cr 1 Mo Tubes Annealed at 1450°F.

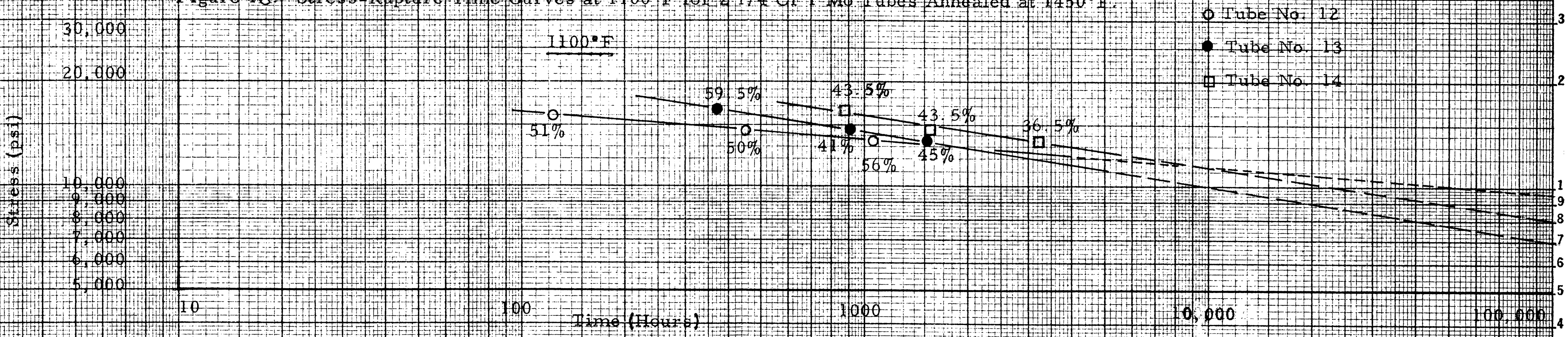

Figure 1D. Stress-Rupture Time Curves at 1100°F fpr 2 1/4 Cr 1 Mo Tubes Annealed at 1450°F.

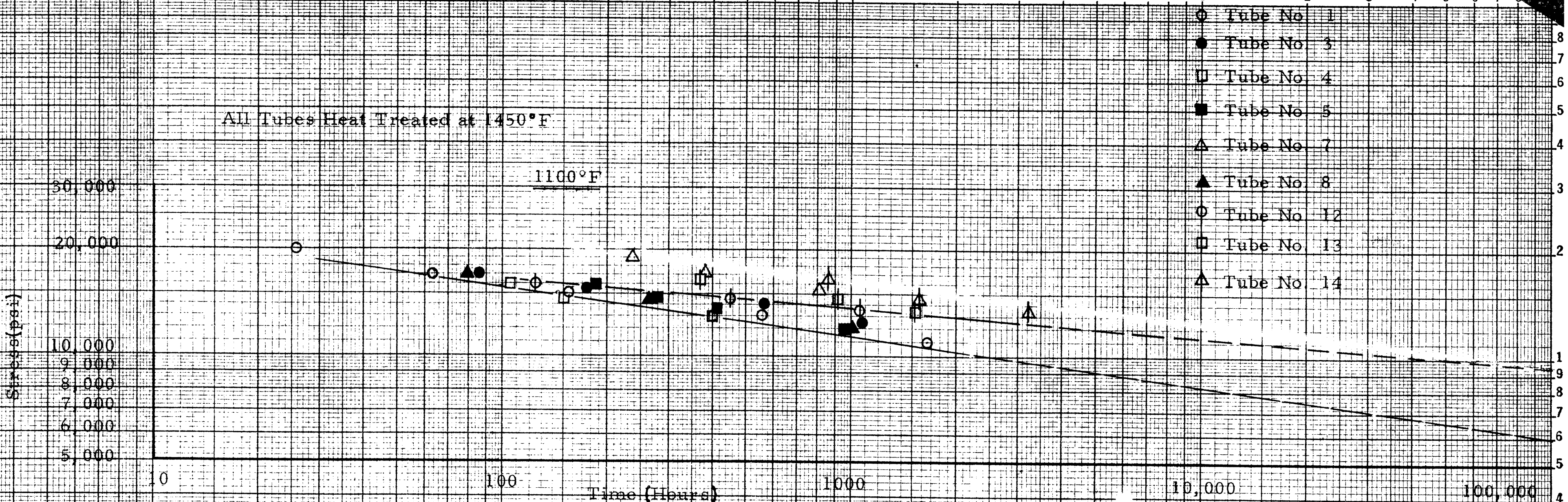

Figure 2. Comparative Stress-Rupture Time Data at 1100°F for the Nine 2 1/4 Cr 1 Mo Steel Tubes Heat Treated for One Hour at 1450°F.

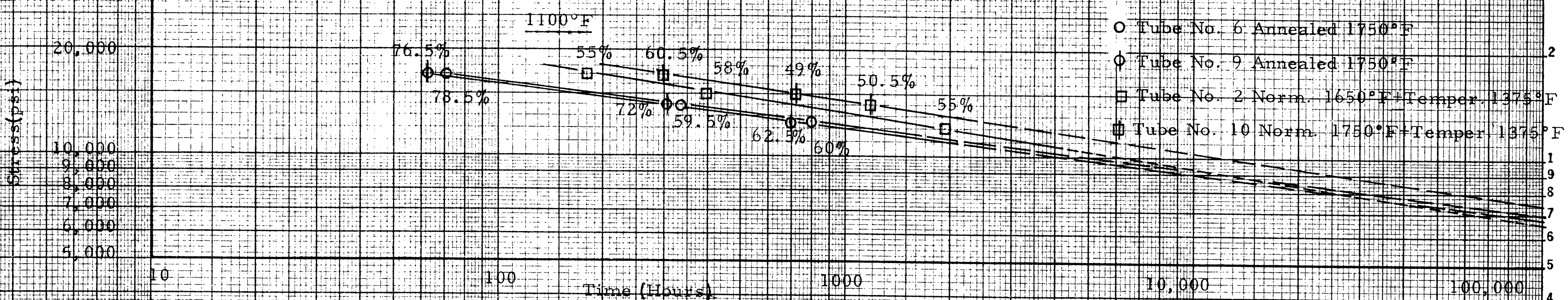

Figure 3. Stress-Rupture Time Data at 1100°F for 2 1/4 Cr 1 Mo Steel Tubes Full Annealed at 1750°F or Normalized and Tempered.

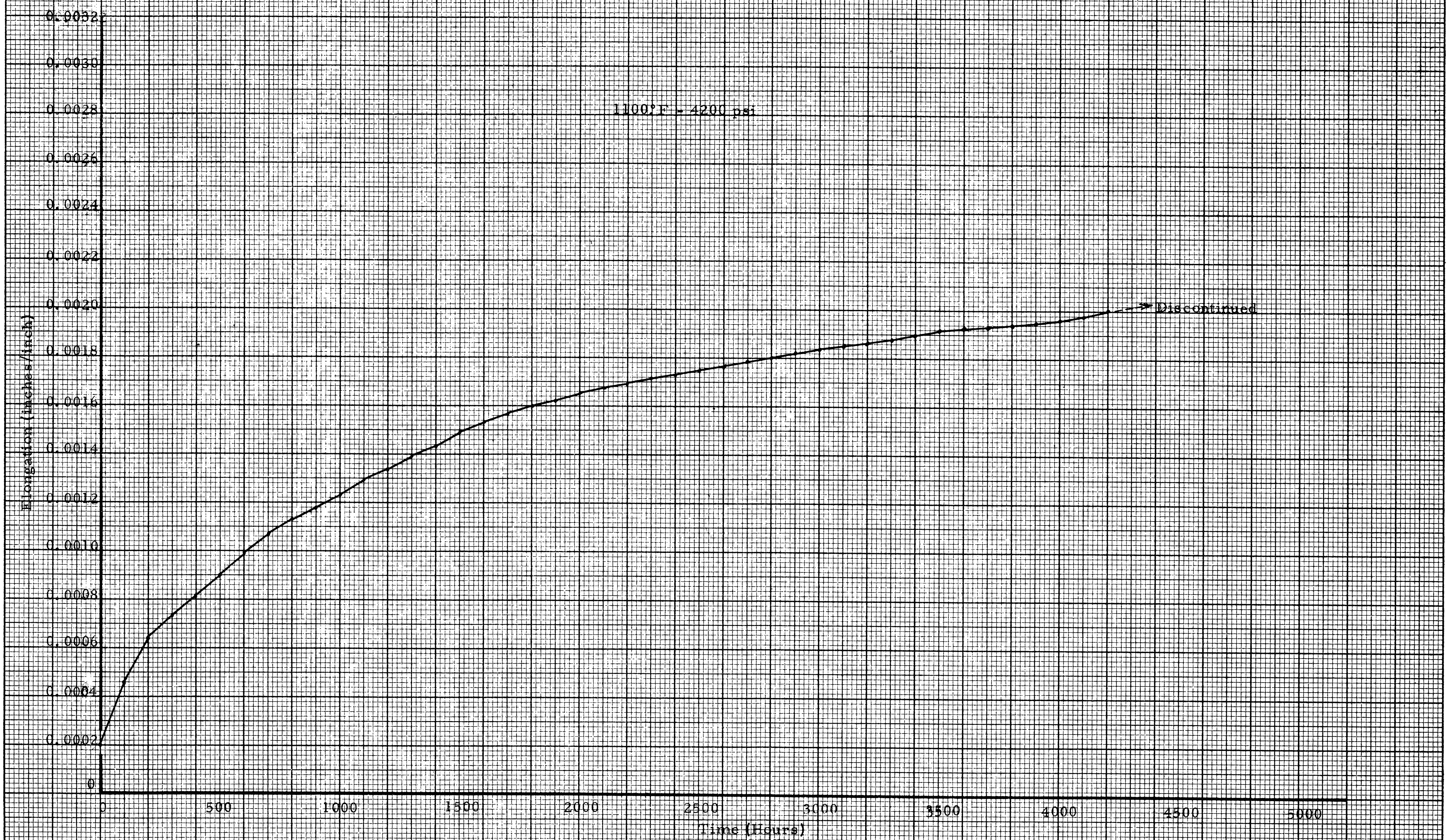

Figure 4 Time Elongation Curve from a Creep Test at 1100°F for 2 ¼ Cr 1 Mo Steel Tube No 1 (Heat 3341384) Annealed at 1450°F

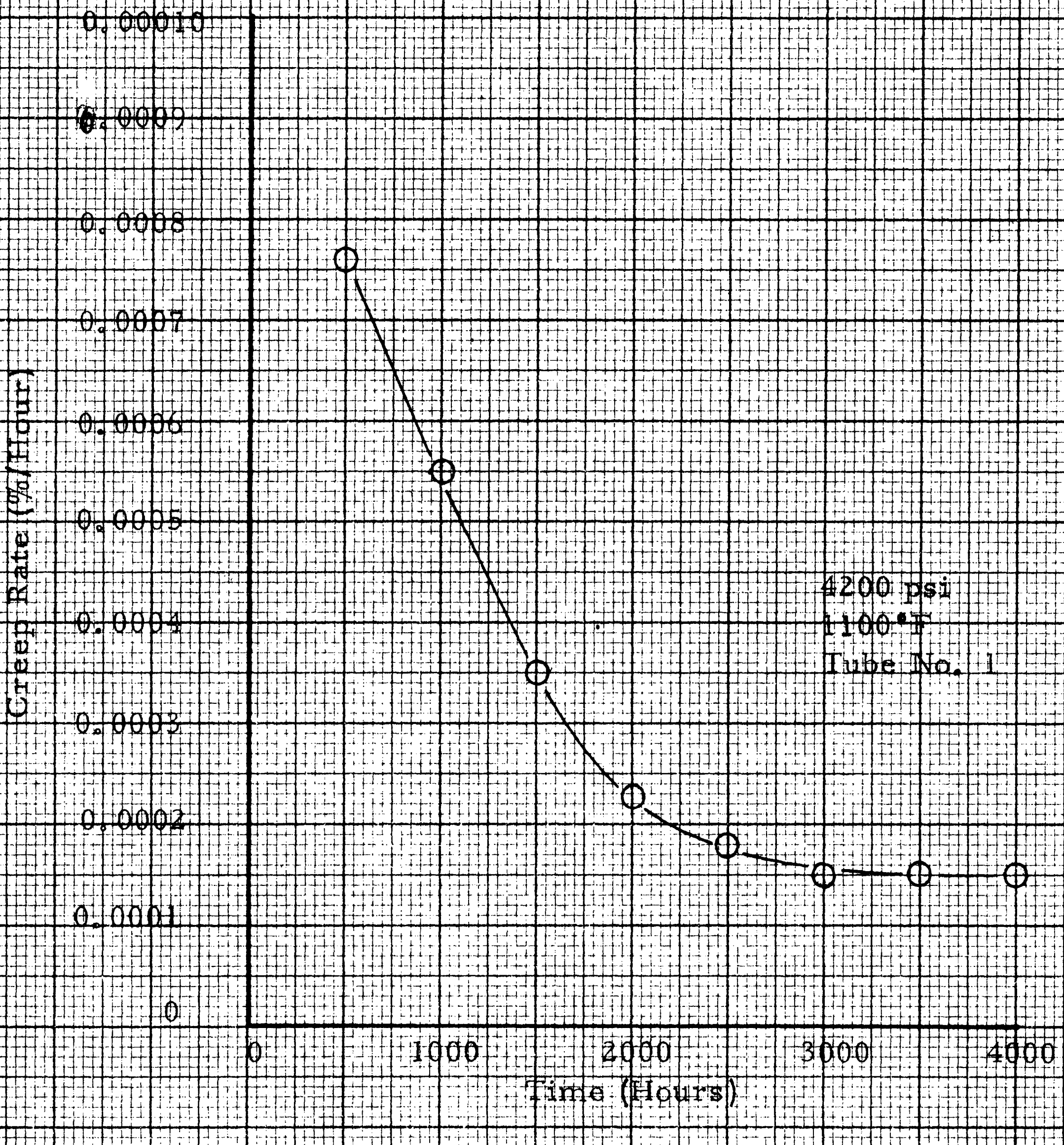

Figure 5. Creep Rate-Time Curve from a Creep Test at 1100°F on 2 ¼Cr 1 Mo Steel Tube No. 1 (Heat 3341384) Annealed at 1450°F.

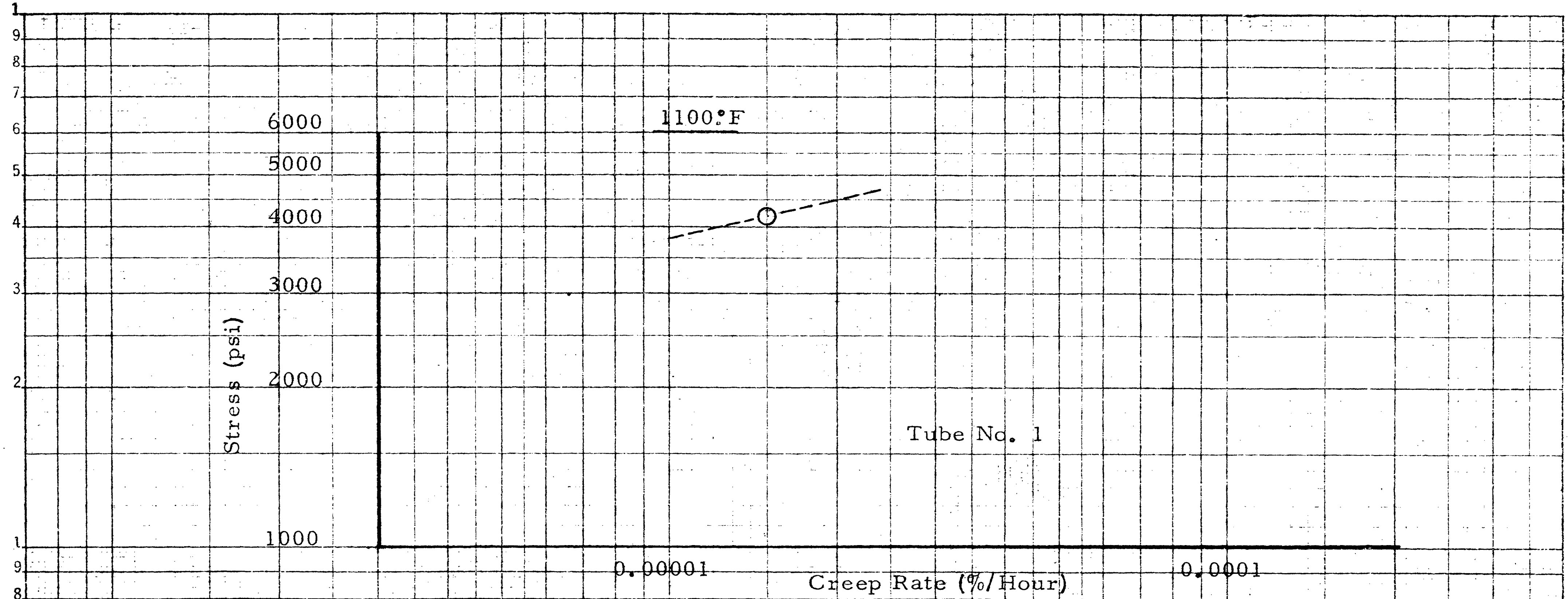

Figure 6. Stress-Creep Rate Curve at 1100°F for 2 1/4 Cr 1 Mo Steel Tube No. 1 (Heat 3341384) Annealed at 1450°F.