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A STUDY OF CREEP OF TITANIUM AND TWO OF ITS ALLOYS

by

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SUMMARY

During the period covered by this report, April 1, 1953 to June 30, 1953, creep testing has been confined to RC 130A and Ti 150A.

In the case of RC 130A, test temperatures have been extended to 210° and 400°F. In addition, transverse testing of as-received and heat treated conditions is reported. The appearance of omega phase, as reported by Battelle, is believed to account for embrittlement of aged beta-quenched samples and the appearance of negative creep phenomena.

For Ti 150A, transverse testing is reported at 76° and 600°F. In addition, the occurence of strain aging in the as-received material is discussed.

INTRODUCTION

This report, the eleventh under Air Force Contract AF 33(038)-14111 (Expenditure Order: R 463-BR1, Supplemental Agreement No. S6 (53-748) covers the study of the creep of titanium and two of its alloys for the period from April 1, 1953, to June 30, 1953. The materials investigated, in the form of commercially produced 0.060-inch sheet, were commercially pure titanium (Ti 75A), a manganese binary alloy (RC 130A) and a ferro-chrome ternary alloy (Ti 150A). The object of the investigation continues to be a study of fundamental structural factors affecting creep properties of the above materials. Cold working and recovery treatments were the variable of investigation for Ti 75A, while heat treatments alone were used in the cases of RC 130A and Ti 150A.

RESULTS AND DISCUSSION

Commercially Pure Titanium

No experimental results are available from work done on Ti 75A during the period covered by this report.

A sheet of new stock has been received from Titanium Metals Corporation. It is of greater thickness, 0.125-inch versus 0.060-inch, than the stock previously studied in order that reductions greater than 40% be obtained by rolling. The end product with the original stock would be too thin for practical testing of reductions over 40%. Annealing and cold working treatments are in progress on samples from both this sheet and the original stock.

Several creep tests are now in progress at room temperature in order to study the effect of a higher annealing temperature and variable cooling rates. In addition, elevated temperature tests will shortly be continued on samples prepared transverse to the rolling direction. This is a continuation of work at 75° and 210°F previously reported[(1)].

Manganese Alloyed Titanium (RC 130A)

Major effort of testing in the time period covered by this report has been devoted to RC 130A, a binary alloy containing 7% manganese.

The conditions under investigation represent two general structural types. These are variations in $\alpha + \beta$ proportions through direct cooling from temperatures in the $\alpha + \beta$ region and water-quenched or furnace cooled structures from the all β field. Air cooling from the β region produces brittleness for reasons that will be discussed later.

In addition to the continuation of tests at temperatures in the range from 76° to 600°F on these conditions, additional work has been started on two aspects of creep of this material. They are the testing of samples in the transverse direction and investigation of lower temperature aging effects on quenched structures.

Creep Testing of As-Received RC 130A

Testing of as-received RC 130A (hot rolled and annealed) is virtually complete at 76°, 210°, 400° and 600°F. All data are presented in figure 1. Results from only one test are presently available at 400°F.

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(1) Ninth Progress Report, December 31, 1952, page 2 and figure 1.

In general, it can be said that little temperature dependency of creep exists in the range from 76° to 400°F. In addition, relative insensitivity to stress seems to be the case in this temperature range. At 600°F, the material is considerably less creep resistant than at lower temperatures, and the creep itself is more stress dependent.

Creep rates were taken at both 500 and 1000 hours. They show an appreciable difference in rates at 76° and 400°F and less difference at 600° and 210°F. Subsequent results at 400°F may reduce the variation between the 500 and 1000 hour rates. It will be noted that the 1000 hour rate is the lower one at 76°, 210° and 400°F. At 600°F, it is greater than the 500 hour rate at the higher stresses. Examination of the creep curves suggests that the onset of third stage creep may take place before 1000 hours. If so, it is only slightly attained, since creep curves run out to 1300 hours show no sharp increases in slope.

In order to check aging effects that may occur during testing at 400°F, several samples have been aged at 400°F for various time periods and then tested at 76°F. The result of only one of these tests is available at present and is plotted on figure 1. A sample aged 25 hours at 400°F and tested at 76°F and 90,000 psi shows a slightly greater creep rate at 1000 hours than the as-received stock. In exhibiting this behavior, it acts similarly to material tested unaged at 210°F. Other tests representing aging at 400°F for both longer and shorter time periods are now in progress.

Creep Testing of Heat Treated RC 130A

Complete results from creep testing of heat treated conditions of RC 130A are now available at 76° and 600°F. These results are presented in figures 2 through 10. Summary plots for comparison purposes are given in figure 12 for 76°F tests and figure 13 for 600°F tests. In addition, limited data at 210°F and 400°F are presented in figures 2 through 10.

Heat treatments studied were the following:

800°F	Air Cooled	figure 2
1100°F	Air Cooled	figure 3
1100°F	Water Quenched	figure 4
1250°F	Air Cooled	figure 5
1250°F	Water Quenched	figure 5
1325°F	Air Cooled	figure 6
1400°F	Air Cooled	figure 7
1400°F	Water Quenched	figure 8
1625°F	Furnace Cooled	figure 9
1625°F	Water Quenched	figure 10

The first eight conditions, from 800° to 1400°F, represent temperatures in or below the $\alpha + \beta$ region The condition representing maximum room temperature tensile ductility is air cooling from 800°F. Furnace cooling and water quenching from 1625°F are the only treatments from the all β region resulting in sufficient ductility for creep testing.

The general feature of the data, figure 2 through 10, is the apparent "strengthening" in the range of 210° and 400°F. As mentioned before, the results are still incomplete; however, at 210° and 400°F creep resistance appears to be about the same as or even greater than at 76°F.

In general, creep resistance of structures having a structure of spheroidal α in a β matrix is a direct function of the relative amount of β. Thus, a hot-rolled and annealed material reheated only in the $\alpha + \beta$ region before quenching will exhibit this characteristic, and such is the case in the present work[(2)]. This linear relation has been developed in the present investigation. It appears to hold true both for room temperature tensile tests and creep tests at room temperature and 600°F. Data

(2) Tenth Progress Report, March 31, 1953, figure 4.

at 210° and 400°F are not yet complete to test the validity of this relation over the entire temperature range. However, at 76° and 600°F, it can be said that an increased amount of beta increases the creep resistance (lowers creep rate at a given stress) in a linear fashion, when the amount of β is plotted against the log of the creep rate.

Examination of figures 2 through 4 (heat treatment at 800° and 1100°F) indicates nothing unexpected. There is evidence to show that the 210° and 400°F creep rates will be the same or even a bit less than the 76°F rates in the same stress range. Such "strengthening" may be due to a strain aging-type reaction. The existence of such an effect in titanium alloys will be discussed subsequently in the section on Ti 150A. The data on RC 130A are as yet too incomplete to permit full extension of the strain aging hypothesis. It will be noted again that 1000-hour creep rates at 600°F are greater than 500-hour rates. This was mentioned previously concerning the as-received material. The effect, therefore, carries over to heat treated material.

Figure 5, 1250°F treatments, shows a somewhat different relation at 76°F. Since a considerable and unexplainable change in slope seems to be the case, the data at the lower stress are being re-checked and, consequently, these points are being marked as "in doubt." Figure 12 summarizes much of these results and in relation to data from other heat treatments, shows that the data at 1250°F may be out of line.

The only condition showing a break in the stress - creep rate curve was that of the material air cooled from 1325°F, figure 6. Careful re-checking of the data appears to confirm the effect and when compared in figure 12, there is some reason to suspect this heat treatment temperature as representing a transition in the slope of creep curves between the lower and higher treatment temperatures.

Thus, at 1400°F, both air cooling, figure 7, and water quenching, figure 8, show at 76°F a considerable increase in creep resistance with a lowering of stress dependency (that is, the slope increases). Figure 8 also shows the first results from the study of lower-temperature aging of heat treated material. A sample originally water quenched from 1400°F and then aged 10 hours at 400°F shows a substantial increase in creep resistance over as-quenched material when tested at 76°F and 115,000 psi.

Upon going into the all β region, several interesting effects are noted. Figure 9 for material furnace cooled from 1625°F, shows a very definite reversal of creep resistance with temperature between 76° and 400°F. Thus, at 210° and 400°F, this material displays increased creep resistance over that at 76°F. These results are analogous to those obtained with annealed conditions of both Ti 75A and Ti 150A.

Figure 10, water quenching from 1625°F, shows even more striking results. Creep resistance at 76°F is high and fairly independent of stress. Subsequent aging of the as-quenched material results in considerable embrittlement. Thus, aging at 600°F, which occurred while merely bringing up a test for loading, embrittled the material to the point that failure occurred before 90,000 psi was reached on loading. Aging at 400°F for various times before testing at room temperature produced similar results. For instance, aging for one-half hour at 400°F produces what appears to be improved creep resistance at 70,000 psi; however, another specimen failed in loading before 105,000 psi was reached. In contrast, the as-quenched material was successfully tested at 130,000 psi. Similarly, a specimen aged two hours at 400°F also failed before reaching 105,000 psi, and another specimen aged ten hours at 400°F failed before even 70,000 psi was reached.

Such embrittling is believed due to formation of the omega phase, a pseudo-cubic transition phase from beta to alpha. The occurrence of this phase in several alloys, including a manganese alloy whose composition approximates that of RC 130A, has been demonstrated by recent work reported by Battelle[3]. The exact mechanism of hardening (and embrittling) has not yet been established by them. X-ray data shows that omega tends to disappear on long-time over-aging, hence the transitional nature of the phase. It is "apparently nucleated sooner than α, but disappears as α forms"[4].

Little is yet known about the crystal structure of omega. Battelle reports the lattice constant to be about 8 Å; however, its exact structure and composition are unknown. Apparently it has some crystallographic relation to β through similar interplanar spacings.

Another effect that may be due to omega formation is also reported in figure 10. Here it will be seen that material as-quenched from 1625°F and then tested at 400°F shows a definite negative creep phenomenon. On figure 11 is plotted the time - elongation data for this test. This shows the beginnings of first - stage creep up to about 20 hours, followed by a very definite shrinkage through the remainder of the test. This effect is confirmed through dilatometric studies also plotted in figure 11[5]. In these tests, RC 130A slugs, brine quenched from 1600°F, were heated to 400°, 700° or 1000°F and held isothermally up to 100 hours. The greatest shrinkage occurred in holding at 400°F. The slope of dilatometric curve was computed and extrapolated to 500 hours. Over the "steady state" portion of the creep curve a fairly good correlation was found between dilatometric shrinkage rate and shrinkage rate in the "creep" test.

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(3) Battelle Memorial Institute, "Development of Titanium Base Alloys," WADC Tech. Report, 52-334 (Contract No. AF 33(038)-3736), December 31, 1952, pp. 80-6, 90-112.
(4) Ibid., p. 85.
(5) Private Communication, C. W. Phillips, U. of M.

"Transverse" Creep Testing of RC 130A

Creep testing at 76°F and 600°F has been performed on samples transverse to the rolling direction for the as-received condition (hot rolled and annealed). Available results are presented in figure 14. In addition, tests are in progress at 210° and 400°F in as-received material, and at 76° and 600°F on representative heat treated conditions.

Preliminary results of transverse testing (figure 14) show somewhat higher creep resistance at 76°F and a considerably higher resistance at 600°F than was found for longitudinal specimens. The results at 76°F are not clear since a different slope appears for transverse testing than for longitudinal testing. Results obtained on Ti 75A and Ti 150A suggest retention of the same slope to be the case, at least over identical stress ranges.

Complete interpretation of these results will be deferred, therefore, until more data are obtained.

Ferro-Chrome Alloyed Titanium (Ti 150A)

Tests are now in progress on Ti 150A to check three effects either noted previously or believed present. They are as follows:

1. Effect of testing transverse to the rolling direction;
2. Strain aging effects believed present in as-received and annealed material--this is analogous to the case of Ti 75A;
3. Long time eutectoid decomposition of quenched structures.

In addition, testing on stock from another heat was approved in order to check the effects of heat-to-heat variations, if any. Unfortunately, Ti 150A in the form of sheet has been withdrawn from the market by the producer. Accordingly, such check tests will be run on a bar stock lot that has recently been received. Results from concurrent work on another project suggest a fairly good agreement between sheet and bar stock (both taken the direction of rolling) at least in the case of tensile properties.

Limited data are now available in the case of the first two points mentioned above.

"Transverse" Creep Testing of As-Received Ti 150A

Creep testing of Ti 150A in the as-received condition (hot rolled and annealed) in the direction transverse to the rolling direction has been initiated at 76° and 600°F. Limited results are presented in figure 15, In all cases, creep rates were taken at 500 and 1000 hours.

At 76°F it was found that the transverse direction exhibited higher creep resistance than the longitudinal direction in the range studied. This is in agreement with results obtained for both as-received and annealed Ti 75A(6) (commercially pure titanium). In all cases, transverse creep rates at 76°F were one-half to one-third the longitudinal creep rates at corresponding stresses. Put another way, the stress for a given creep rate was 3000 to 5000 psi higher.

Limited data at 600°F suggests this effect is somewhat greater at the higher temperature. Thus, the 500 hour rate at 55,000 psi and 600°F for the transverse sample was one-fifth that of the longitudinal sample. In this case, a seeming divergence exists in the behavior of Ti 75A and Ti 150A. At 210°F, it was indicated that for Ti 75A(7) the difference between the two directions was minimized. Further testing of both Ti 75A and Ti 150A at 600°F and intermediate temperatures is planned. Until these results are available, no comment can be offered to explain the effect.

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(6) Eighth Progress Report, September 30, 1952, p. 3 and figure 1; Ninth Progress Report, p. 2 and figure 1.
(7) Ninth Progress Report, p. 3 and figure 1.

Strain Aging of Ti 150A

Results obtained from previous elevated temperature creep testing of Ti 150A suggested the presence of a strain aging phenomenon, that is, reversal of creep resistance with increasing temperature. Thus, creep resistance at 400°F was greater than at 210°F; however, at 600°F it was poorer than either lower temperature[8]. Explanations of these results in terms of eutectoid decomposition was not feasible, so it was decided to check for possible strain aging phenomena.

The existence of strain aging in pure titanium has been confirmed both under this project[9] and by other investigators[10]. One criterion of strain aging, so-called "strengthening," i.e., decrease in slope of tensile strength vs. temperature curves, was noted in Ti 150A in the range from 600° to 800°F through work on a concurrent project at the University of Michigan[11] and also from information published by the Titanium Metals Corporation[12]. Qualitatively, appearance of a definite yield point and serration of the stress - strain curve were noted by the operator in the running of tensile tests on bar stock in connection with the concurrent work mentioned before[11]. Strain rate sensitivity was not checked for Ti 150A.

In the case of Ti 75A, the effect of strain aging was found to cause a reversal of second stage creep rates with increasing temperature[9]. It was also found that long time aging at the "reversed" temperature before

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(8) Tenth Progress Report, figure 11.
(9) Sixth Progress Report, March 31, 1952, pp. 7-8 and figures 2-7; Seventh Progress Report, June 30, 1953, p. 4
(10) Rosi and Perkins, Trans. ASM, 45, p. 972 (1953).
(11) Gluck and Freeman, "Intermediate Temperature Creep and Rupture Behavior of Titanium Alloys," Third Progress Report, April 15, 1953, figure 7, (under Air Force Contract AF 33(616)-244).
(12) Titanium Metals Corporation, Handbook on Titanium Metal (6th ed.), pp. 30-1 (1952).

testing resulted in improved creep resistance at room temperature.

Both these effects have been found in Ti 150A. The "reversal" was mentioned previously. In addition, a sample of as-received Ti 150A was aged for 100 hours at 400°F and then tested at 76°F and 70,000 psi. The point for this test is plotted in figure 15 and shows a substantial increase in creep resistance as a result of the aging treatment. Thus, the same reactions causing strengthening during testing at 400°F also increase creep resistance at 76°F. Other tests are being run to confirm and extend these results.

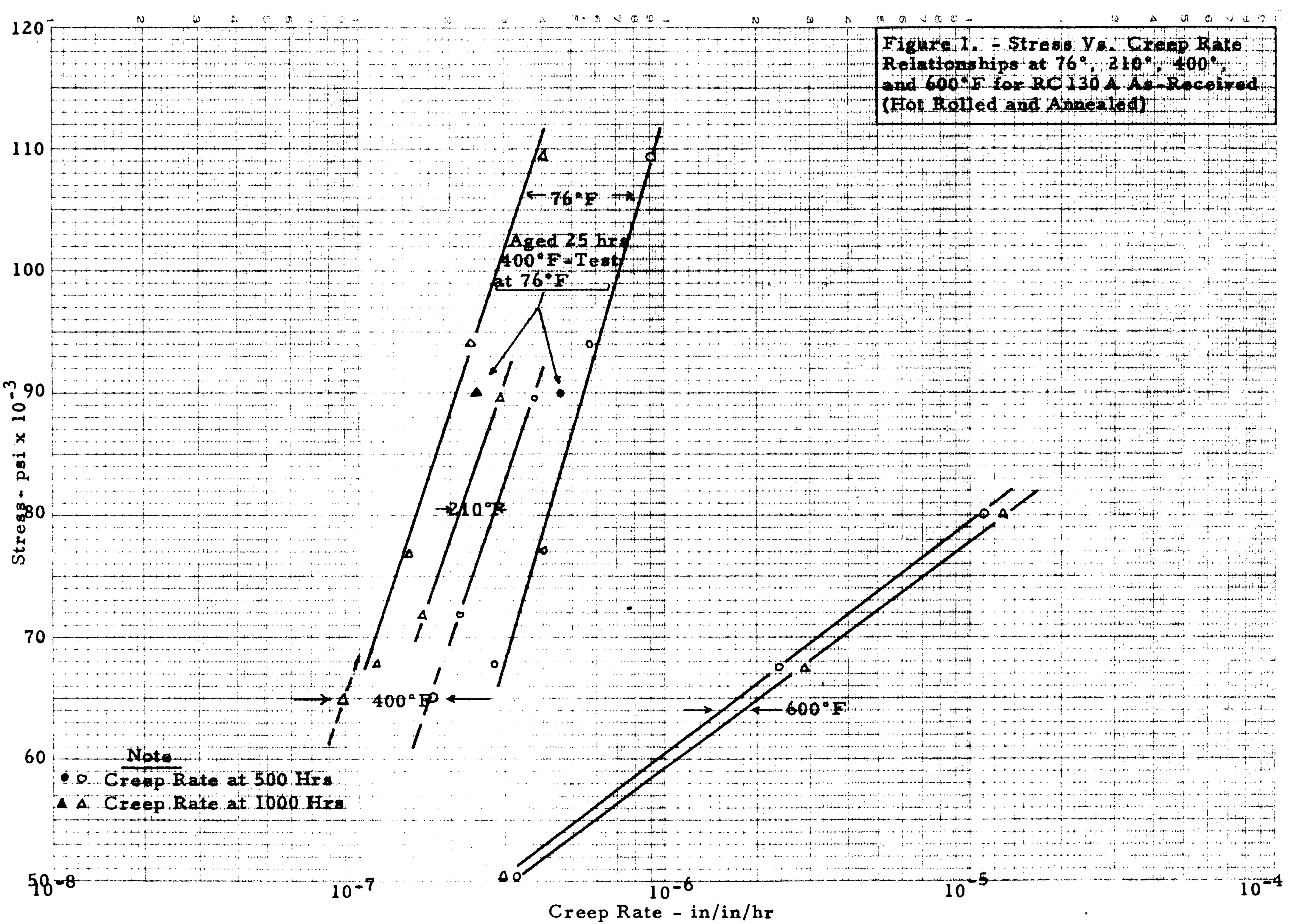

Figure I. - Stress Vs. Creep Rate Relationships at 76°, 210°, 400°, and 600°F for RC 130 A As-Received (Hot Rolled and Annealed)

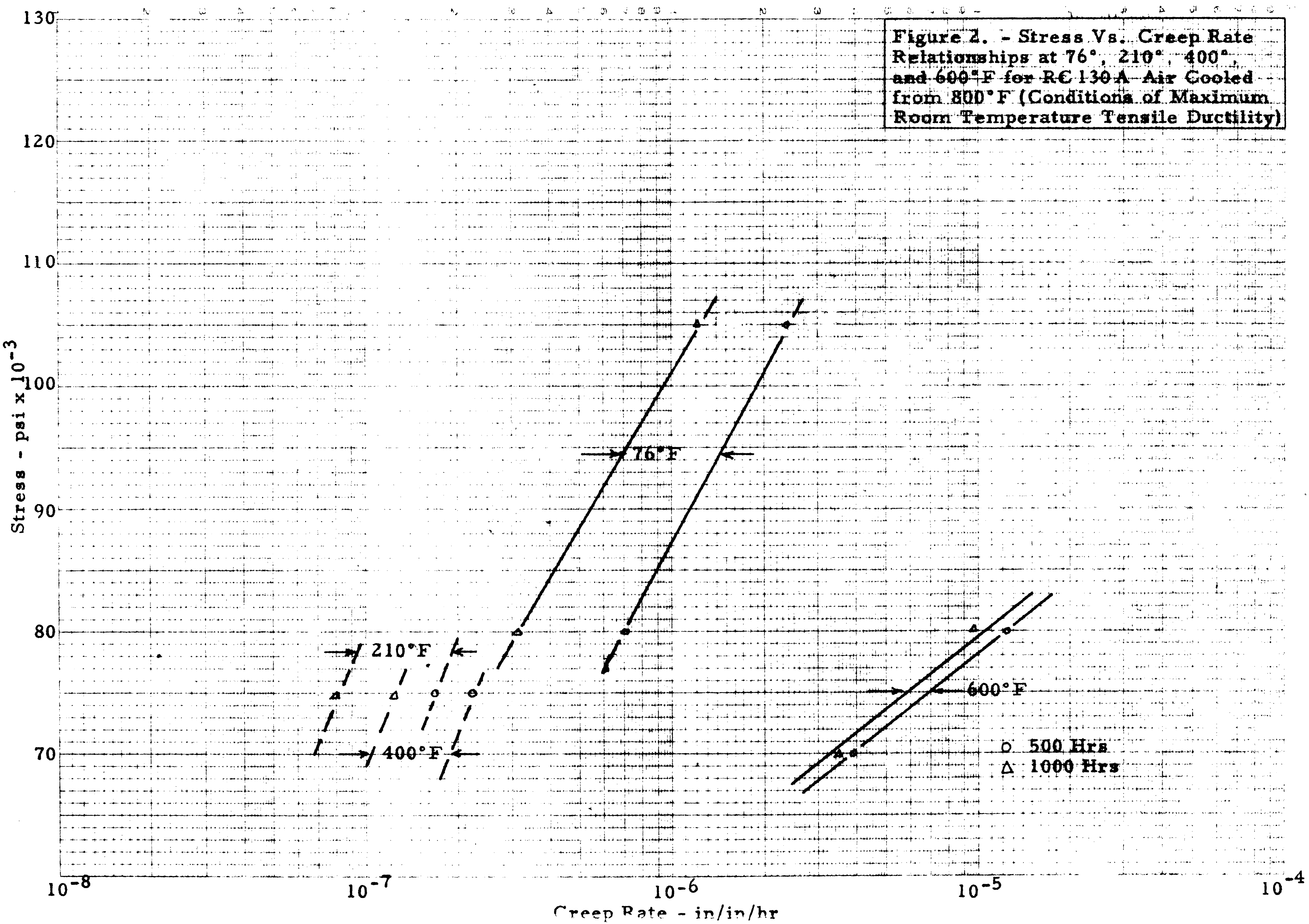

Figure 2. - Stress Vs. Creep Rate Relationships at 76°, 210°, 400°, and 600°F for RC 130 A Air Cooled from 800°F (Conditions of Maximum Room Temperature Tensile Ductility)

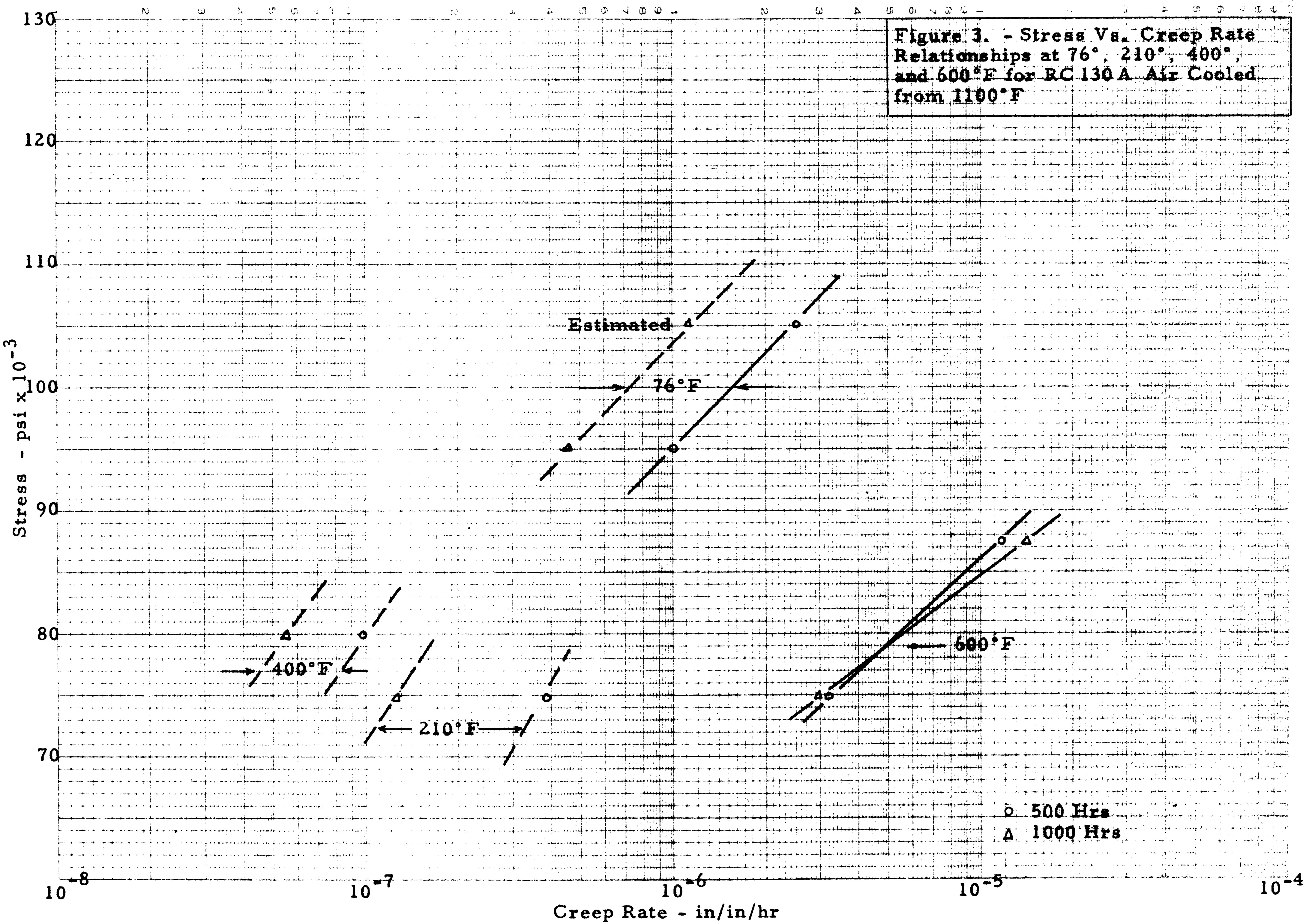

Figure 3. - Stress Vs. Creep Rate Relationships at 76°, 210°, 400°, and 600°F for RC 130 A Air Cooled from 1100°F

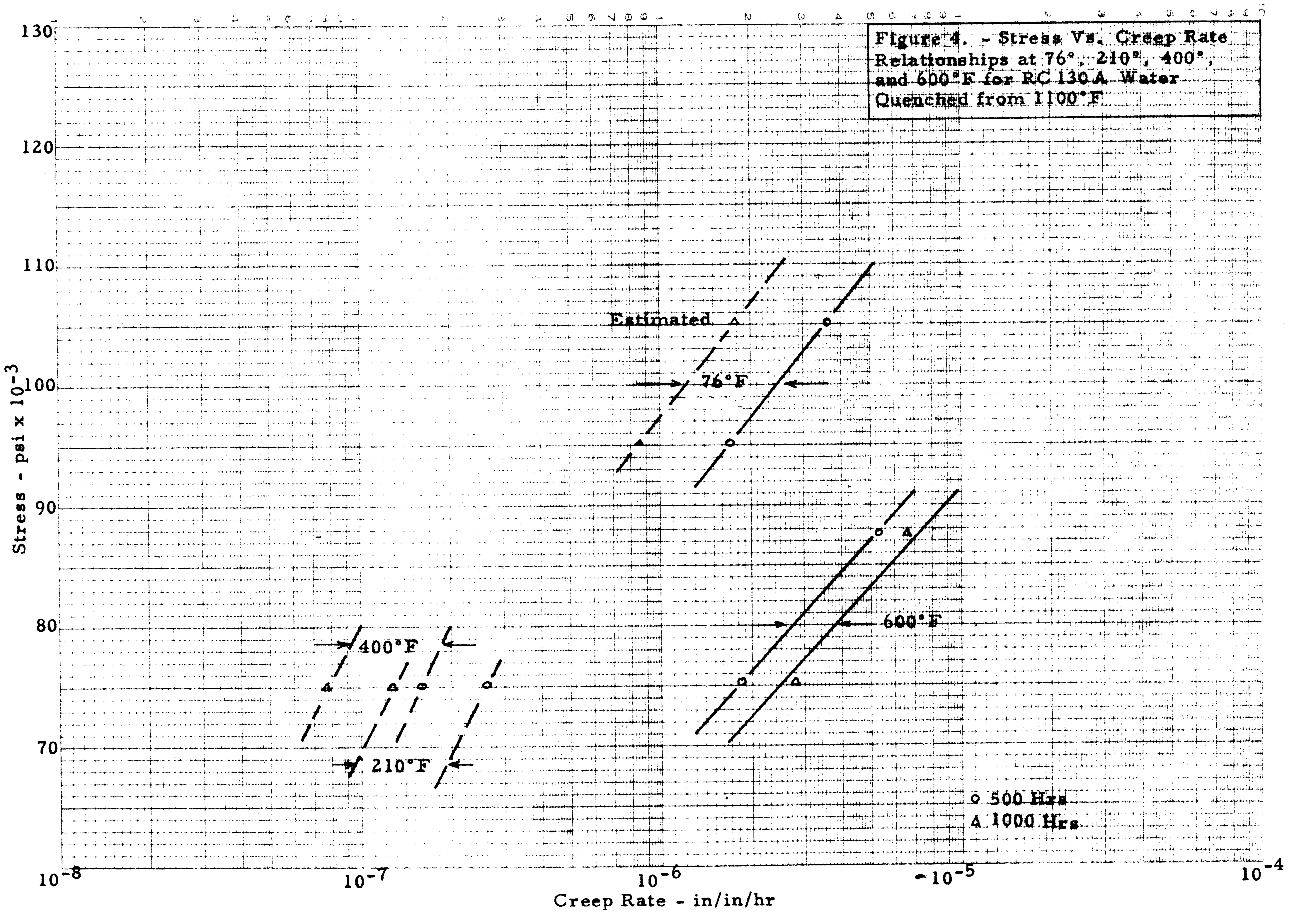

Figure 4. - Stress Vs. Creep Rate Relationships at 76°, 210°, 400°, and 600°F for RC 130 A Water Quenched from 1100°F

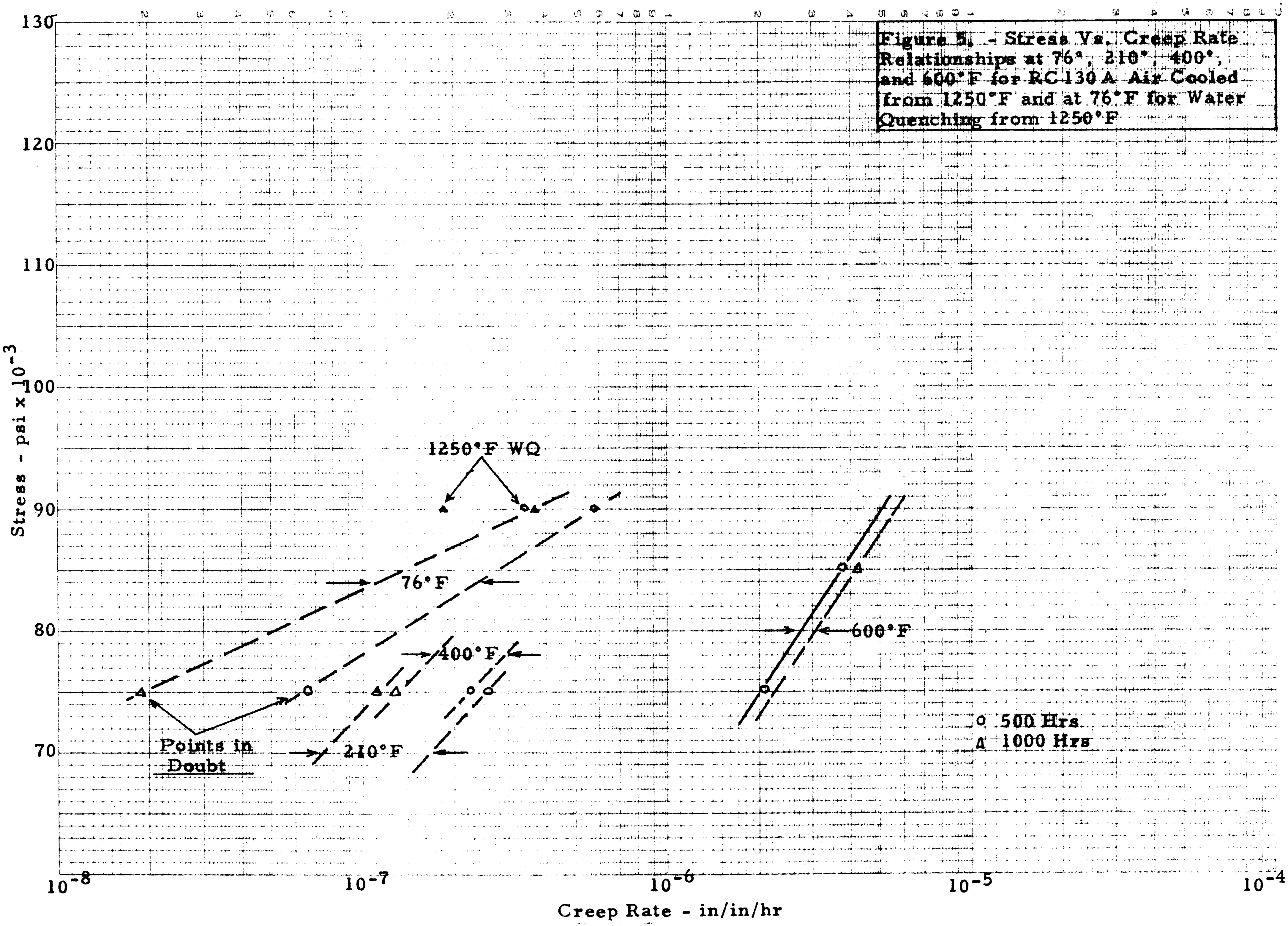

Figure 5. - Stress Vs. Creep Rate Relationships at 76°, 210°, 400°, and 600°F for RC-130 A Air Cooled from 1250°F and at 76°F for Water Quenching from 1250°F

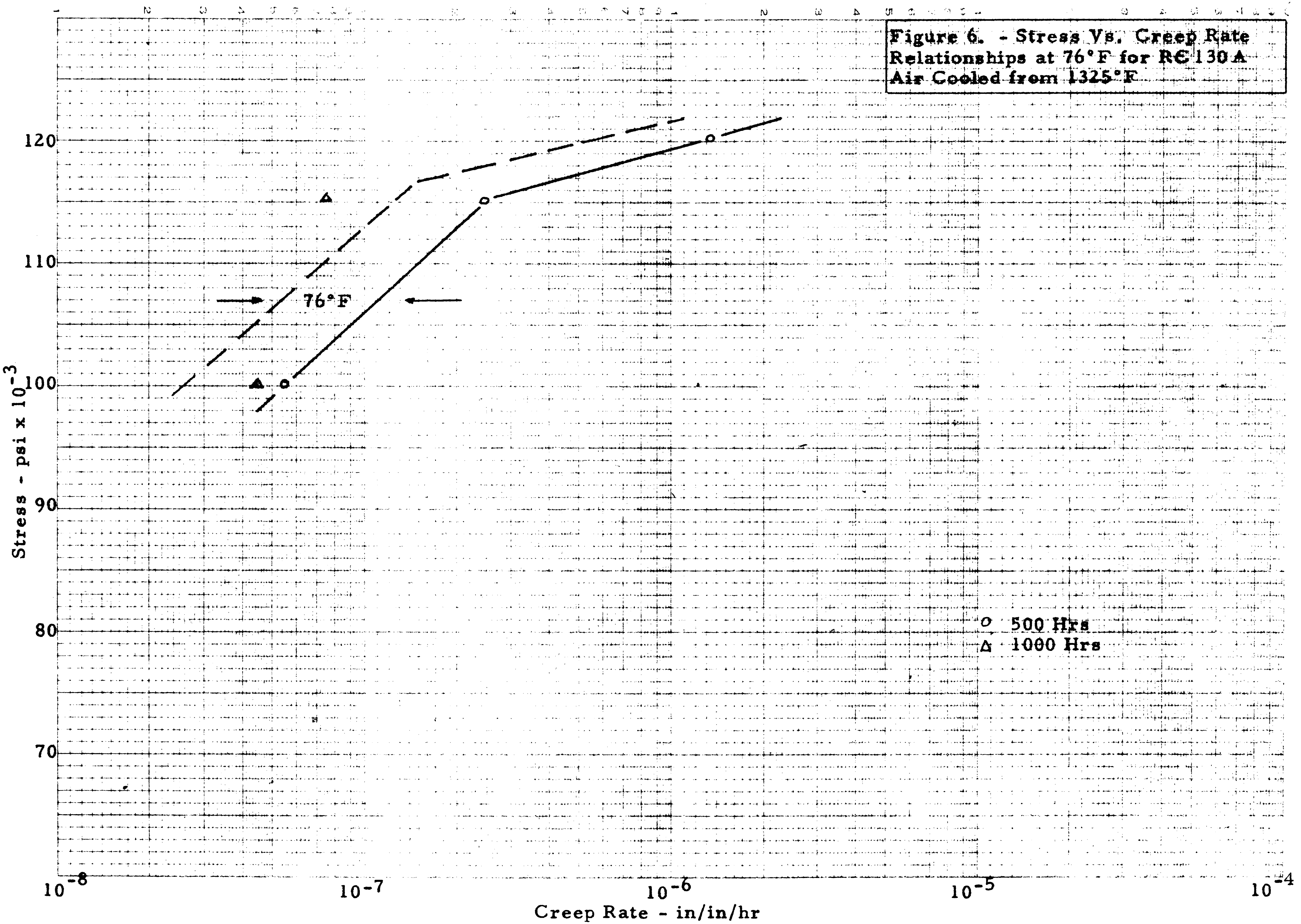

Figure 6. - Stress Vs. Creep Rate Relationships at 76°F for RC 130 A Air Cooled from 1325°F

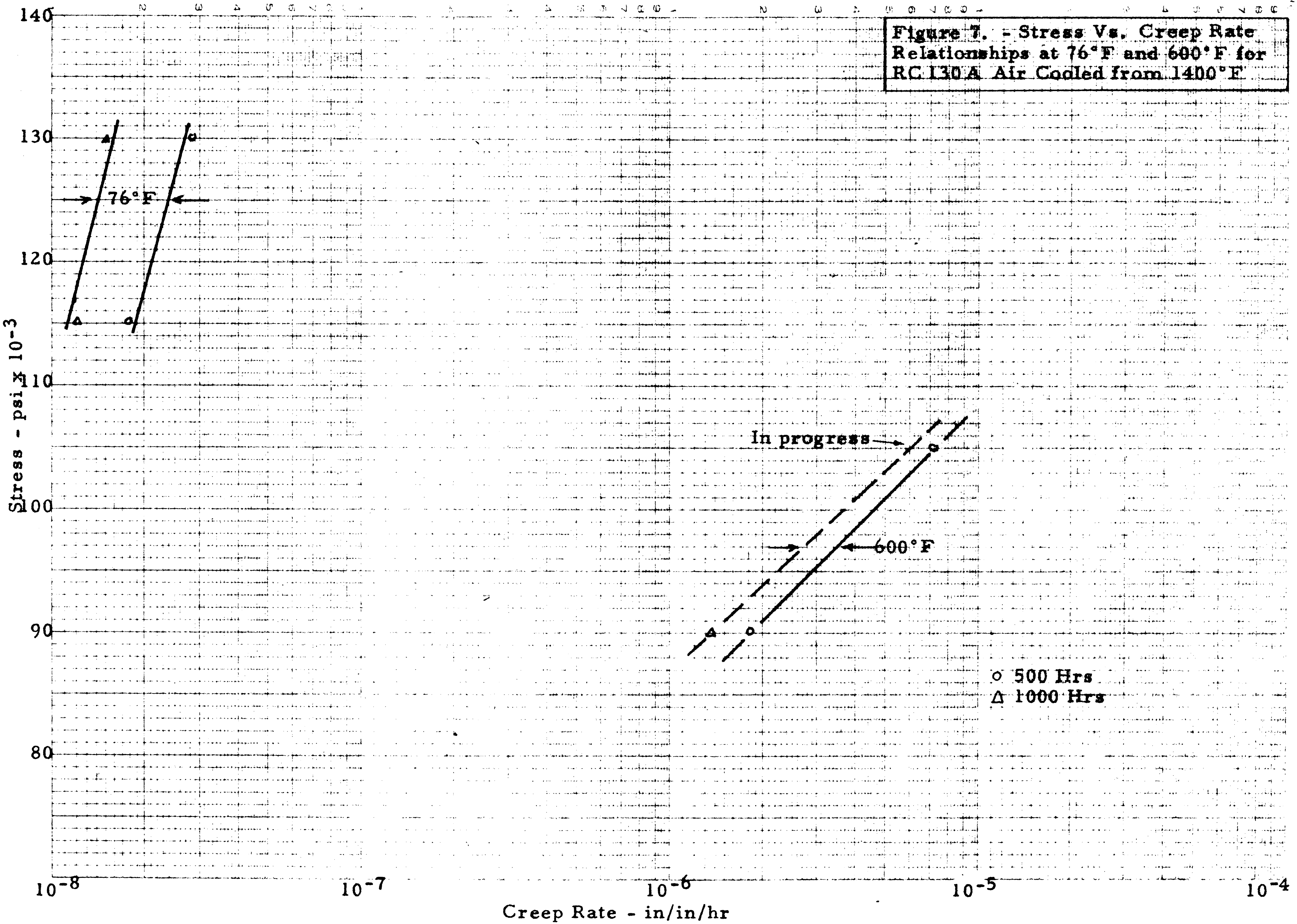

Figure 7. - Stress Vs. Creep Rate Relationships at 76°F and 600°F for RC130A Air Cooled from 1400°F

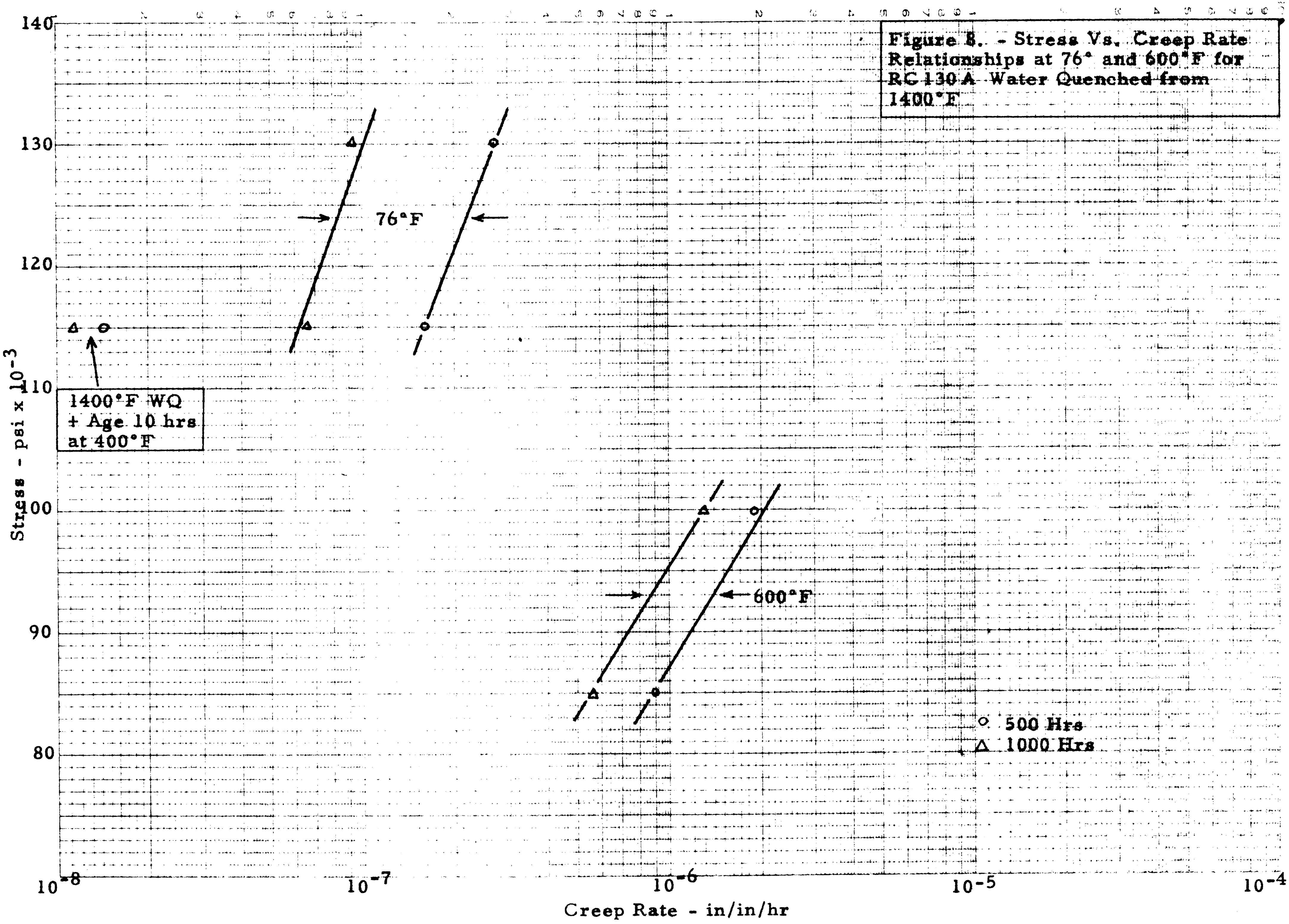

Figure 8. - Stress Vs. Creep Rate Relationships at 76° and 600°F for RC 130 A Water Quenched from 1400°F
140
130
120
110
100
90
80
76°F
600°F
1400°F WQ + Age 10 hrs at 400°F
○ 500 Hrs
△ 1000 Hrs
Stress - psi x 10-3
10^{-8}
10^{-7}
10^{-6}
10^{-5}
10^{-4}
Creep Rate - in/in/hr

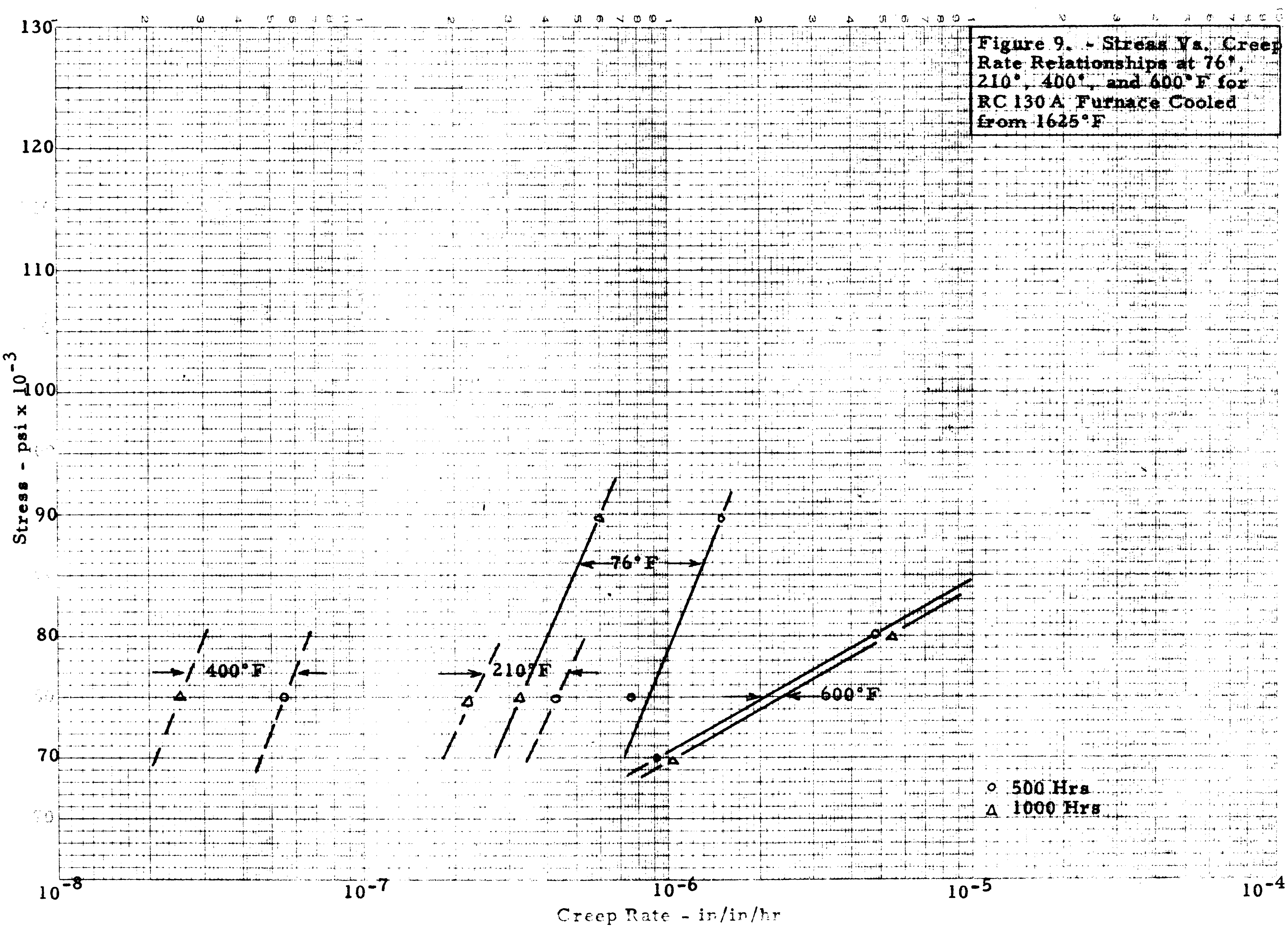

Figure 9. - Stress Vs. Creep Rate Relationships at 76°, 210°, 400°, and 600°F for RC 130 A Furnace Cooled from 1625°F

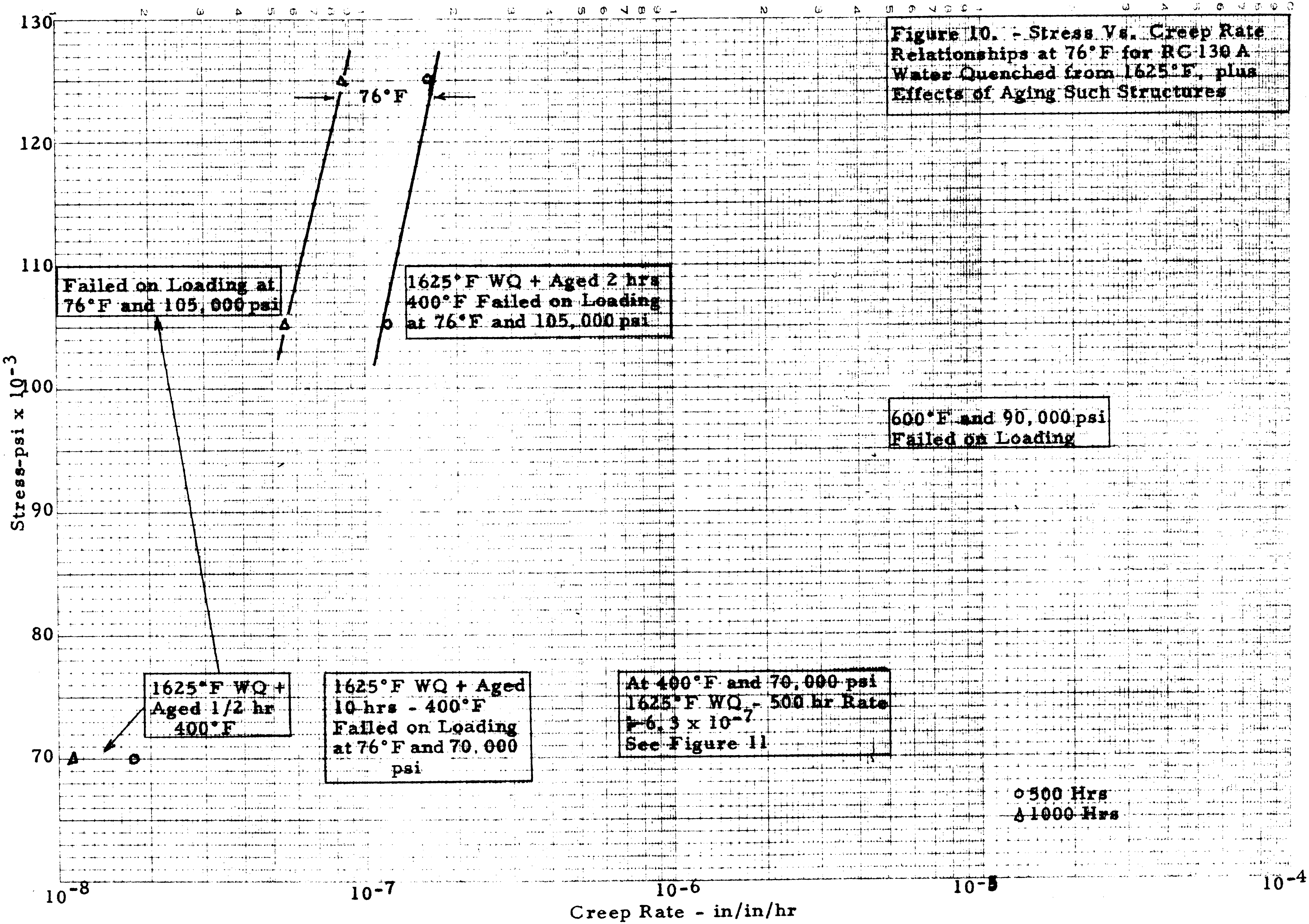

Figure 10. - Stress Vs. Creep Rate Relationships at 76°F for RC-130 A Water Quenched from 1625°F, plus Effects of Aging Such Structures
76°F
Failed on Loading at 76°F and 105,000 psi
1625°F WQ + Aged 2 hrs 400°F Failed on Loading at 76°F and 105,000 psi
600°F and 90,000 psi Failed on Loading
1625°F WQ + Aged 1/2 hr 400°F
1625°F WQ + Aged 10 hrs - 400°F Failed on Loading at 76°F and 70,000 psi
At 400°F and 70,000 psi 1625°F WQ - 500 hr Rate = 6.3 x 10-7 See Figure 11
o 500 Hrs
Δ 1000 Hrs
Stress-psi x 10-3
130
120
110
100
90
80
70
10-8
10-7
10-6
10-5
10-4
Creep Rate - in/in/hr

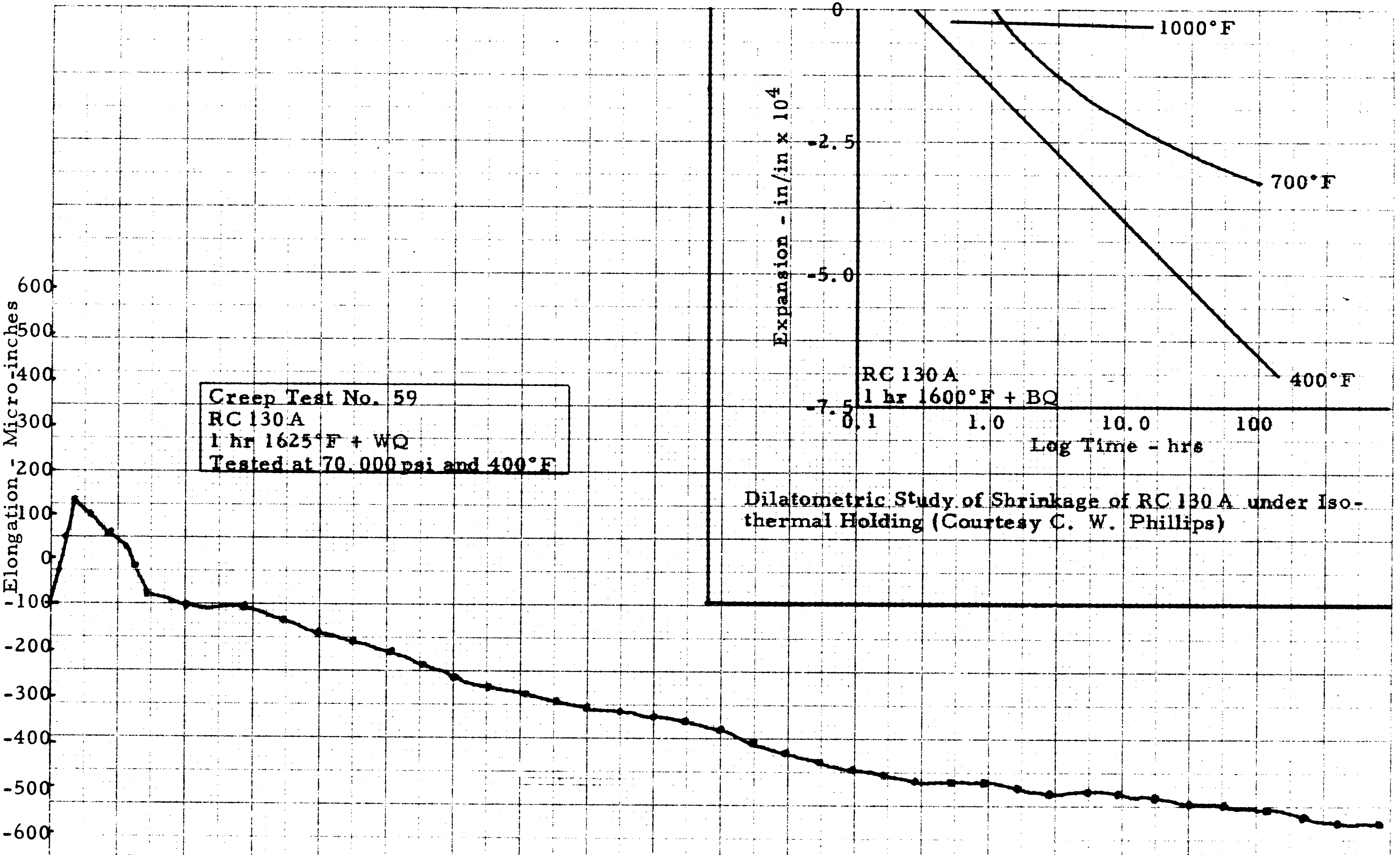

Figure 11. - Time - Elongation Curve for RC 130 A Sheet (Treatment Above) Showing Shrinkage as an Effect of Temperature and Time and Correlated with Dilatometric Tests Run at No Stress -- Other Conditions Equivalent

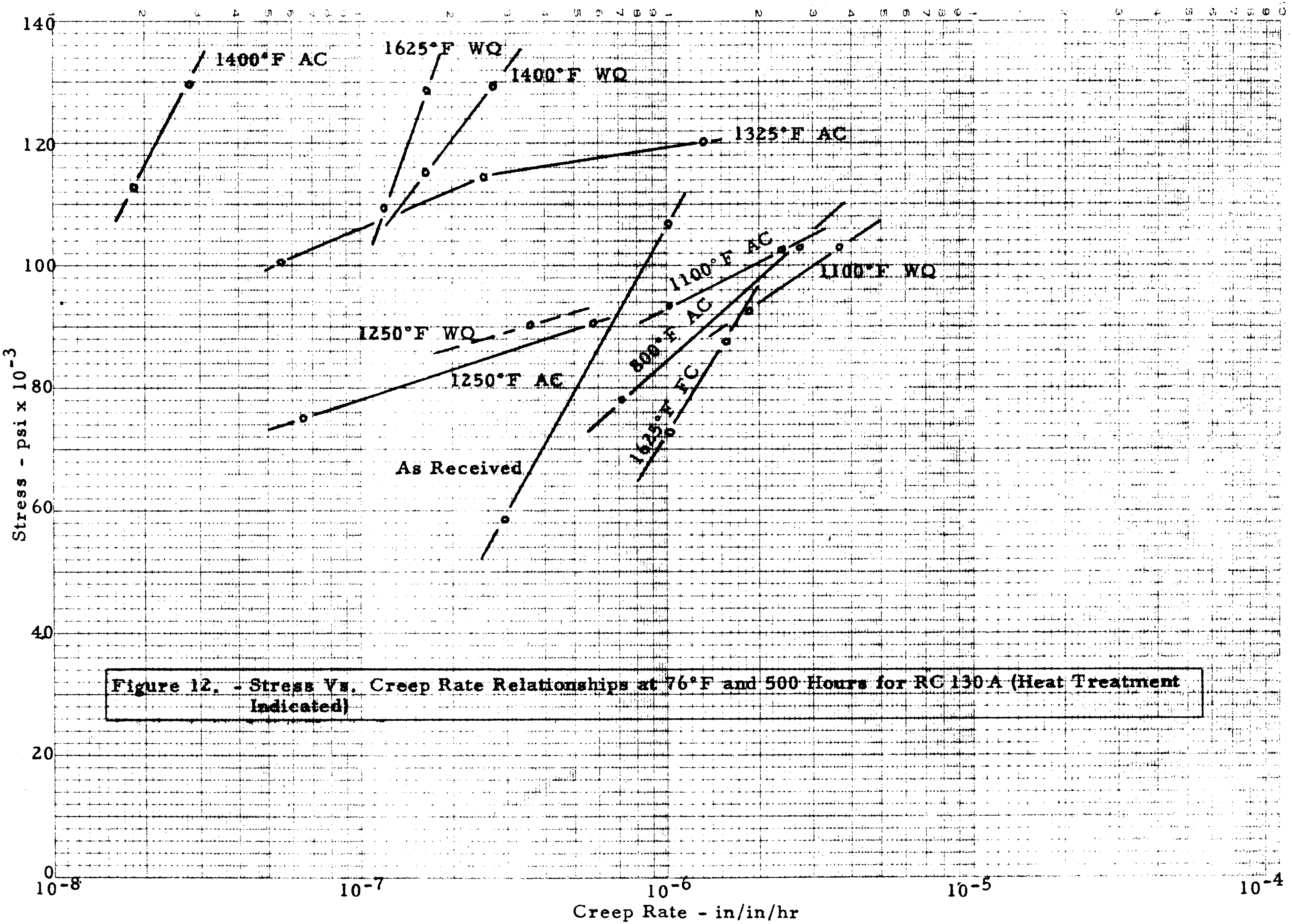

Figure 12. - Stress Vs. Creep Rate Relationships at 76°F and 500 Hours for RC 130 A (Heat Treatment Indicated)

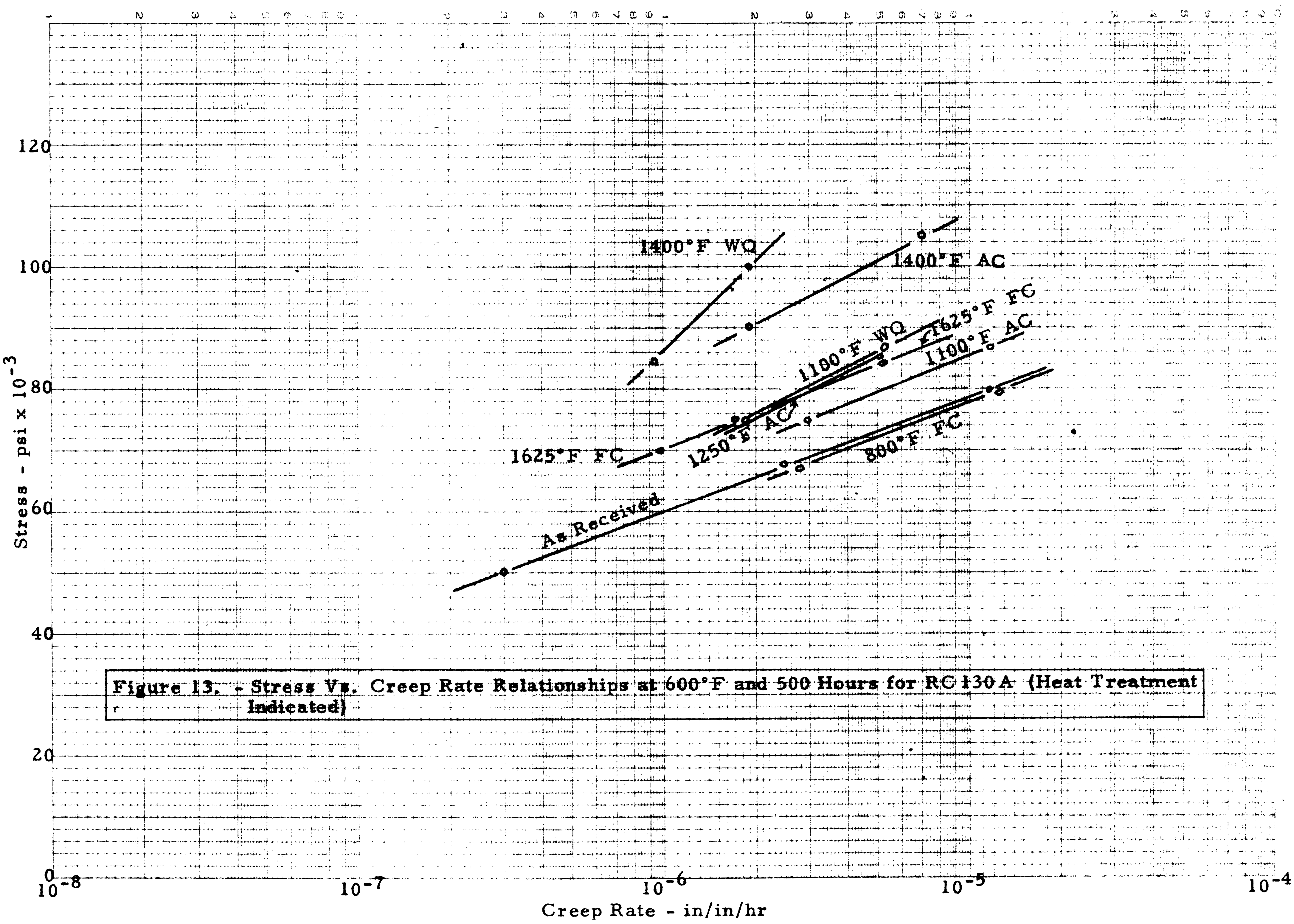

Figure 13. - Stress Vs. Creep Rate Relationships at 600°F and 500 Hours for RC 130 A (Heat Treatment Indicated)

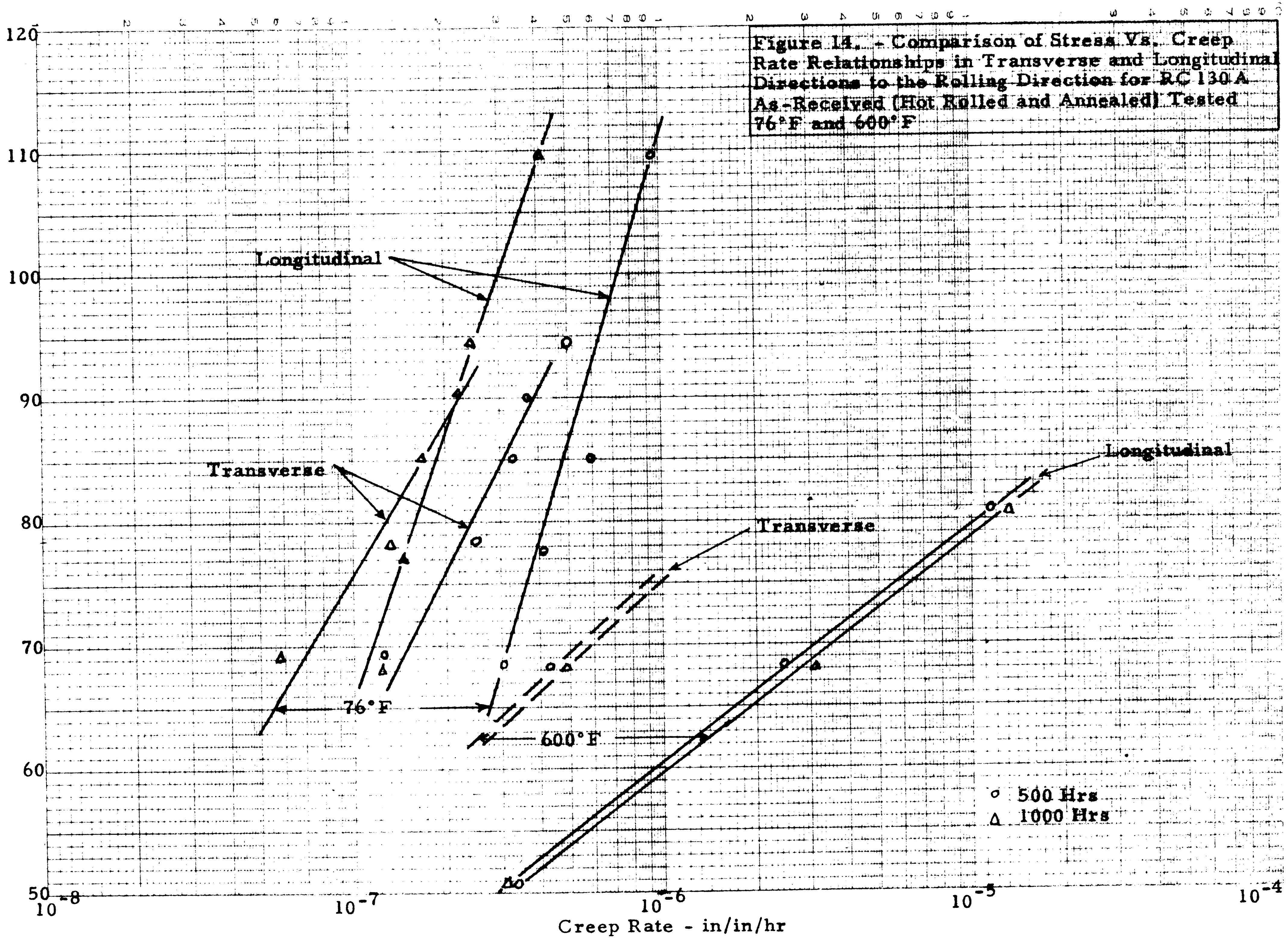

Figure 14. - Comparison of Stress Vs. Creep Rate Relationships in Transverse and Longitudinal Directions to the Rolling Direction for RC 130 A As-Received (Hot Rolled and Annealed) Tested 76°F and 600°F
Longitudinal
Transverse
Longitudinal
Transverse
76°F
600°F
o 500 Hrs
Δ 1000 Hrs
120
110
100
90
80
70
60
50
10^{-8}
10^{-7}
10^{-6}
10^{-5}
10^{-4}
Creep Rate - in/in/hr

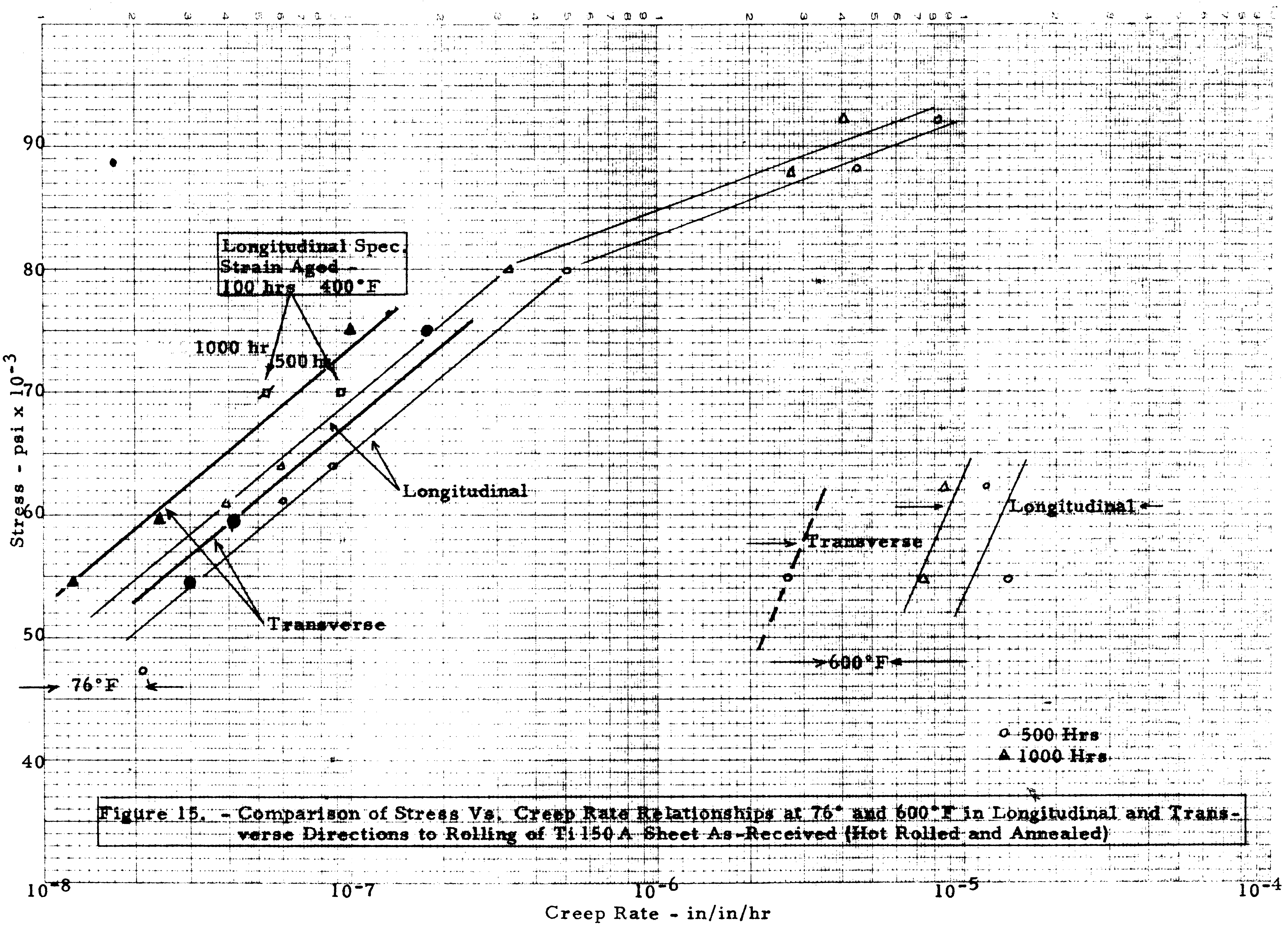

Figure 15. - Comparison of Stress Vs. Creep Rate Relationships at 76° and 600°F in Longitudinal and Transverse Directions to Rolling of Ti 150 A Sheet As-Received (Hot Rolled and Annealed)