SIXTH PROGRESS REPORT

TO

THE OFFICE OF AIR RESEARCH

ON

A STUDY OF CREEP OF TITANIUM AND TWO OF ITS ALLOYS

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A STUDY OF CREEP OF TITANIUM AND TWO OF ITS ALLOYS

SUMMARY

During the period covered by this report, 1 January 1952 to 31 March 1952, creep testing of the Ti 75 has been continued at elevated temperatures. At room temperature the study of the effect of recovery over a range of temperatures on the material cold worked 40 percent has been initiated. Tensile testing of the material in various conditions has been carried out both at room temperature and at elevated temperatures where necessary.

Creep testing of the annealed material has been completed at 400° and 600°F. A maximum in the creep resistance has been observed at 400°F. Results of creep testing at room temperature of the material cold worked 40 percent and recovered 100 hours at temperatures up to 750°F are also presented. Here again a maximum in the creep resistance was found for a recovery temperature of 400°F. Creep testing at 400°F of the material cold worked 40 percent, and the material cold worked 40 percent and recovered 100 hours at 550°F, showed a higher creep resistance for the latter. This was also found true for testing at 210°F. Generally, it can be stated that line width measurements made on cold worked and recovered stock do not correlate with creep resistance.

Tensile tests made on the annealed stock over a temperature range from 75° to 1000°F disclosed the criteria necessary to describe a strain-aging process. Additional tensile tests run at room temperature on the material recovered 100 hours at temperatures up to 550°F after cold working 40 percent showed a maximum in tensile properties at a recovery temperature of approximately 400°F.

Creep testing of the ferro-chrome alloy of titanium (Ti 150) has continued at room temperature on material heat treated in various ways from 1500°F. The testing of the as-received material has been extended to 400°F. Creep rates have been shown to be sensitive to both heat treatment and testing temperature. Metallographic examinations have been utilized for the study of the effect of testing on microstructure with little, if any, effect noted.

INTRODUCTION

This report, the sixth under Air Force Contract Number: AF33(038)-14111 (Expenditure Order Number: R-463 BR-1) on the study of creep of titanium and two of its alloys in the form of commercially produced 0.060-inch thick sheet, covers the period from 1 January 1952 to 31 March 1952. Work is still being concentrated on the commercially pure titanium (Ti 75) and the ferro-chrome alloy (Ti 150) since the third material, probably a manganese alloy (Ti 130), has not yet been received. The object of this work continues to be the study of fundamental structural factors affecting the creep properties of the above materials. In line with this viewpoint, determinations are being made on the effect of cold working and recovery treatments on the internal structure and creep resistance of Ti 75. In the case of Ti 150, the main variable of investigation is heat treatment.

EXPERIMENTAL PROCEDURES

Atmosphere Efficiency. In the Fifth Progress Report it was reported that a modification of the heat treatment atmosphere had been effected through the use of titanium chips for argon purification. Subsequent hardness traverse studies were employed to check the efficiency of the atmosphere. Figure 1 presents results of this investigation. For three different cooling treatments from 1500°F it was found that in each case the use of titanium chips resulted in an over-all lower hardness level across the section of the specimen, excluding local variations. The anamolous brittleness noted before (Fifth Progress Report, page 10) has not been encountered since the initiation of the modified heat treatment procedure.

RESULTS AND DISCUSSION

Commercially Pure Titanium (Ti 75)

The work during the period covered by this report involved the further accumulation of creep data on the stock annealed at 1600°F and the stock given additional treatments of cold working and recovering. Elevated testing temperatures of 400° and 600°F, together with room temperature tests, were utilized.

Previous reports have presented a reasonable correlation of internal strain, as measured by the broadening of the (114) X-ray diffraction line, with room temperature creep resistance of the cold worked material. Attempts to add cold worked and recovered samples to this correlation were not always successful. Thus, a more generalized program has been initiated so that these discrepancies might be understood.

The work has been extended to include the effect of the recovery treatment over a range of temperatures on the room temperature creep resistance of the cold worked stock. At present the effect of time at recovery temperatures, as it affects creep resistance, is also being evaluated.

To help in evaluating the results of the above creep tests, microstructural investigations have been carried out. Also, an evaluation of the effect of testing temperature on the tensile properties of the annealed stock has been started, together with the accumulation of data concerning the room temperature tensile properties of the cold worked and recovered stock.

Creep Testing of Annealed Material. Creep testing at 400° and 600°F on the annealed material has been completed. These results, together with those previously obtained on the same material at room temperature and at 210°F, are presented in figure 2. It will be noted that a considerable increase in the creep resistance has occurred at 400°F as compared to the results at 210°F. At 600°F the creep resistance again drops off.

The effect of temperature is graphically illustrated in figure 3 where the stress necessary to give a secondary creep rate of 10^{-6} per hour, as determined in the testing interval of 1000 hours, is plotted as a function of testing temperature. Since the curves for the effect of stress, at varying temperatures, on the creep rate are approximately parallel, the results shown in figure 3 hold for all creep rates.

Because of the nature of the creep results at 600°F, a minimum number of creep tests on the annealed stock are now in progress at 750°F.

Creep Testing of Cold Worked Material. Creep testing of the stock cold worked 40 percent after annealing 1 hour at 1600°F has been completed at 400°F. These data are presented in figure 4 together with the data obtained previously. Improved creep resistance is still evident when compared to the stock which was annealed only.

It will be noted that at 400°F, as was the case at 210°F, the stock cold worked and recovered 100 hours at 550°F prior to testing possesses better creep resistance than the material cold worked only. At 210°F the continued recovery processes during testing, as demonstrated by the recovery of the diffracted X-ray line breadths, probably accounted for the lessened creep resistance of the cold worked stock which had received no recovery treatment before testing. Although no recovery was evident after approximately 20 hours at 400°F in the X-ray line breadth studies, the dynamic conditions of creep testing must allow such recovery to take place during the testing time considered. This subject will be further discussed in ensuing sections.

Creep Testing of the Cold Worked and Recovered Material. After it was found that the state of internal strain, as measured by X-ray line breadth measurements in the materials which were recovered after cold working, did not always correlate with the room temperature creep resistance, a more inclusive testing program was started on these materials. Samples which were cold worked 40 percent were held for 100 hours at 210°, 400°, 550°, and 750°F for recovery prior to testing. To date, testing of these conditions has been completed at room temperature. These data, plotted as the log of the creep rate as a function of stress, are given in figure 5. It has been found that recovery treatments from 210° to 400°F resulted in an improved creep resistance as compared to the material cold worked only. A further increase in the temperature of recovery to 500° and 750°F resulted in a decrease in the creep resistance. It will be noted that as the temperature of recovery is increased the slope of the stress versus log of the creep rate curves tends to decrease. Previously, in evaluating the creep results for the stock recovered 100 hours at 550°F, the data were found to parallel approximately the data for the stock cold worked and given no recovery treatment. Since the results at the lower stress on the later material were poor, a re-evaluation of these data was made in view of the present results at the other temperatures and the present curve tentatively drawn. Tests now in progress will clarify the exact slope.

At present the effect of time at recovery temperatures of 210°, 400°, and 550°F is being investigated.

Creep testing of the stock recovered 100 hours at 550°F has been completed at 400°F. These data together with previously reported data on this stock creep tested at room temperature and 210°F are shown in figure 4.

The creep data on the material recovered and then tested at room temperature showed an improved creep resistance after recovering at 400°F as contrasted to recovery at 550°F. There is reason to believe the same relation should hold at elevated temperatures and, hence, the recovery taking place during the first period of the 400°F creep test on the material cold worked only should give an improved creep resistance. However, it will be noted that this was not the case.

The higher temperature of the 550°F pretesting recovery treatment must have prevented all but a minimum of instability during testing at 400°F while the recovery processes must have continued throughout the testing period on the material with no recovery treatment. To check this supposition, creep tests at 400°F are to be conducted on material cold worked 40 percent and recovered at 450°F.

Tensile Testing. In order to obtain a clearer understanding of the creep results previously reported, recourse has been made to tensile tests of varying conditions and states of the material. The choice of tensile testing was made in order to take advantage of the great amount of information available concerning the manner in which various phenomena manifest themselves in this type of test. Through tensile testing relatively large amounts of data are available within short periods of time.

The tests were run in air with a Baldwin-Southwark hydraulic tensile machine using a dial range of 2400 pounds. Normal strain rates were used and all data recorded automatically. The properties determined were the normal tensile ones of ultimate strength, 0.2% offset yield strength, percentage elongation in 2 inches, and modulus of elasticity.

The first series of tests were run on the annealed stock in order to determine the effect of temperature on the above properties. Figure 6 presents these results. It is to be noted that, in the range from 600° to 800°F, the tensile strength remains essentially constant. If anything, it increases slightly. The 0.2% offset yield strength follows in a like manner while the percent elongation does the inverse. Of interest is the fact that a yield phenomenon was detectable in the range from 210°F to approximately 600°F.

In figure 7 are presented the results of tests at room temperature on material cold worked 40 percent and recovered 100 hours at various temperatures up to 550°F. The curves obtained are not those expected of a true recovery process. Instead, at about 400°F, the tensile properties reach a maximum while the ductility suffers. Above this temperature the tensile properties decline. No definite appearance of a yield point was apparent in these latter tests.

Microstructural Investigations. Since the results obtained on tensile testing were indicative of an aging process, the materials previously cold worked 40 percent and recovered at temperatures to 600°F were re-examined. No evidences of a precipitate were visible. An additional series of samples cold worked

40 percent were allowed to recover at 750°F for time intervals to 100 hours and also examined. An overaged precipitate was still not visible. Photomicrographs of the material cold worked 40 percent and cold worked plus 100-hour recovery at 750°F are presented in figures 8 and 9.

Theoretical Considerations. The recently obtained results on creep testing (see figures 2 through 7) and tensile testing indicate the occurrence of some type of aging phenomenon. On microscopic examination of the structures no visible precipitate has been detected, even at times and temperatures which should result in extreme overaging. Furthermore, precipitation is usually accompanied by a change in internal strain great enough to cause changes in the line breadths of the diffracted X-ray lines, a condition not found in the previous X-ray investigation. Examination of the tensile curves, however, presents a fairly convincing argument for a strain aging mechanism. In general, strain aging is characterized by the yield phenomenon, serrated stress-strain curves, and strengthening, all of which have been evident to a greater or lesser degree over some temperature range studied.

The increased creep resistance of the annealed stock at the testing temperature of 400°F is analogous to the increased tensile properties at approximately 600°F. This is evident since the increased rate of straining of the tensile test has been shown by Nadai and Manjoine (1) to raise the temperature of the maximum tensile properties and to inhibit the extent of the maximum.

Testing at room temperature of the material which had been cold worked 40 percent and recovered 100 hours at various temperatures to 750°F, again resulted in the best creep resistance after treatment at approximately 400°F as shown in figure 5. It would appear here that the strain aging process resulting from the cold working during recovery again produced a maximum effect at approximately 400°F.

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(1) Nadai, A., and Manjoine, M. J., J. Applied Mechanics, pp. 63, 77-91 (1941).

With the introduction of the relatively complex phenomenon of strain aging during creep testing, and prior to testing in the recovered specimens, the application of the present creep theories become a difficult task. However, in the near future, with the completion of the creep testing program on the commercially pure titanium, an attempt will be made to analyze these data on an empirical basis.

Ferro-Chrome Alloyed Titanium (Ti 150)

Extension and corroboration of previously initiated testing results has been the principal objective in the work on Ti 150 in the past three months. Metallographic data have been studied in an attempt to discover the effects of creep testing on microstructure. A possible X-ray method for quantitative determination of the proportions of α and β has been considered and the factors necessary for such analyses have been computed.

Creep Testing. The Fifth Progress Report, dated December 31, 1951, presented tentative stress versus creep rate relationships for tests at room temperature of Ti 150 heat treated in various manners from 1500°F. Additional data have now been accumulated to permit revision of these tentative relationships. These data are reported in figures 10, 11, and 12. Three methods of heat treatment from 1500°F have been studied previously: water quenching, air cooling, and annealing; a fourth treatment, air cooling followed by an anneal from 1300°F, has been added to the list of heat treating variables.

The principal change to be noted in these data is that of the shape of the curves. Rather than using a simple straight line relation on semi-log paper, it has been found necessary to introduce a break into the curve in order to better follow the points obtained. In the case of air cooling and annealing from 1500°F (see figure 10), the break occurred at a creep rate of approximately

1.5×10^{-8} hr^{-1} and is characterized by a change of slope in the order of 3:1. The actual stress levels for the phenomenon are different, being 65,000 psi for the annealed conditions and 94,000 psi for the air cooled condition. For stock annealed from 1500°F the practical limiting stress appears to be 75,000 psi. Those tests attempted at 80,000 psi showed rupture in periods ranging from 10 to 195 hours. A similar practical limit for air cooled stock appears to be in the region from 105,000 to 110,000 psi. Rupture occurred in just over 1000 hours at 110,000 psi and in 775 hours at 113,600 psi. The effect of testing time on creep rate is not as marked with heat treated specimens as that previously encountered in the as-received specimens. This variation is also less noticeable at higher stress levels.

Quite a bit of scatter was found in the data for the stock water quenched from 1500°F (see figure 11). This is particularly evident in the points for the 500-hour creep rate. No simple usable relationship can be presented for these points, although at 1000 hours the data are capable of correlation. Little variation in creep rate with stress is evident up to 100,000 psi; however, at this point there is a sharp break in the relationship. The change in slope is approximately 16:1. The strengthening effect of water quenching from 1500°F is a very marked one. A practical upper stress limit is between 112,000 and 115,000 psi and here the creep rate is a tenth of that under a similar stress for the air cooled material which, incidentally, is above the 1000-hour rupture strength of the latter.

The study of the effect of subsequent reheating treatments on material first cooled at an accelerated rate has been started with samples originally air cooled from 1500°F. Reheating for 1 hour at 1300°F results in a significant drop in the creep strength. These data are presented in figure 12. The effect on the microstructure of such treatment is discussed elsewhere. Of particular interest is the fact that, for the case investigated, annealing at 1300°F following air cooling from 1500°F gives about the same stress versus creep rate relationship as that for direct annealing from 1500°F. It is too early to state any definite conclusions regarding these limited results. To check them it is planned to test material water quenched from 1500°F and reheated at 1300°F and also material annealed from 1300°F.

It is of importance to note here that all the foregoing heat treatments have been conducted entirely within the two-phase α and β region on the equilibrium diagram. The microstructural effects involved depend principally on the solution of α in β as the temperature is raised and the subsequent non-equilibrium structure produced on accelerated cooling.

Elevated temperature testing at 400°F has been started on the as-received material (solution treat 1275°F, water quench, + 1200°F for 2 hours) at stresses of 55,000 psi and 72,000 psi. The creep rates at 500 and 1000 hours are presented in conjunction with the previously reported data in figure 13. The slope of the stress versus creep rate curves appears to be approximately the same for all temperatures investigated to date.

However, an effect similar to that noted in the case of the annealed Ti 75 has been observed. The creep resistance at 400°F is superior to that at 210°F. That this effect is a long-time one is borne out by examination of loading data for the two temperatures. Attention is particularly called to the relative elastic moduli (see page 11). At 210°F the modulus is 11.8×10^6 psi while at 400°F it is 9.6×10^6 psi. Apparently the material also has poorer short-time tensile properties at the higher temperature. That this contention is valid is qualitatively corroborated by reference to the complete strain-time curves for the tests. The primary creep in the 400°F test was so great as to preclude any reliable measurement. At 210°F it was possible to obtain a reasonable idea of the primary component as it was much less. Furthermore, it can be inferred that the strengthening effect on creep rate at 400°F occurs principally in the first hundred hours of the test, since the strain versus time curve levels off sharply at approximately this point. Further decrease in creep rate was small as reflected by the difference between the 500- and 1000-hour rates. This change in rate was in the same order of magnitude as previously observed at lower temperatures.

The phenomenon is to be further investigated in greater detail.

Modulus Data. With the extension of creep testing to 400°F it has been possible to add to the modulus data previously computed from stress-strain data taken during loading. The "double" heat treatment is also reported.

Treatment	Test Temp. (°F)	E x 10^{-6} (psi)
As Received (W.Q. 1275°F + 2 hrs. at 1200°F)	76	15 ± 0.5
As Received (W.Q. 1275°F + 2 hrs. at 1200°F)	210	11.8 ± 0.5
As Received (W.Q. 1275°F + 2 hrs. at 1200°F)	400	9.6 ± 0.5
Air Cooled 1500°F + 1 hr. 1300°F + Anneal	76	15

Metallographic Work. Following the completion of creep testing, a number of specimens have been re-examined metallographically in an attempt to discover what, if any, microstructural changes may have occurred as a result of the time, stress, and/or temperature conditions.

Figures 14 through 19 show the effects of creep testing at elevated temperatures on the structure of Ti 150 as-received (solution treat 1275°F, water quench, + 2 hours at 1200°F). Figure 14 shows the material before testing while figures 15, 16, and 17 show the effect of creep testing for long time periods at 76°, 210°, and 400°F, respectively. The stresses used were in the order of 70,000 to 90,000 psi. Very little change in microstructure can be seen as a result of testing at 76° and 210°F (see figures 15 and 16), however, figure 17 shows that at 400°F the effect of time and temperature does change the microstructure. Some doubt exists at present regarding the exact nature of the change. X-ray determinations are to be used in order to check the microstructural evidence which seems to indicate enlargement of the alpha grains. Consequently, the effect on creep resistance as mentioned previously (figure 13 and page 10) cannot be assigned solely to this cause. Instead, the explanation is to be sought perhaps in terms of possible precipitation or strain-aging processes.

Heat treated structures were also examined metallographically with little, if any, significant change in microstructure evidenced as a result of testing conditions. All such tests have been performed to date only at 76°F. Such variation as exists is mainly accounted for by local variability from point to point in the sample. All microstructures are taken perpendicular to the rolling direction and, as such, may be considered typical of the cross-section of the sample.

Figure 20 shows the result of annealing for 1 hour at 1300°F after air cooling from 1500°F, while figures 18 and 19 show the results of air cooling from 1500°F and annealing from 1300°F, respectively. These are presented for comparison purposes. In creep properties the treatment appears to produce results similar to those of stock annealed from 1500°F.

The microstructure produced is similar to the annealed condition; however, the dark etching constituent is more finely distributed. While the alpha grains in the "single" heat treated specimens (figures 18 and 19) are clearly outlined, they do not appear to be so in the case of the "double" treatment (figure 20).

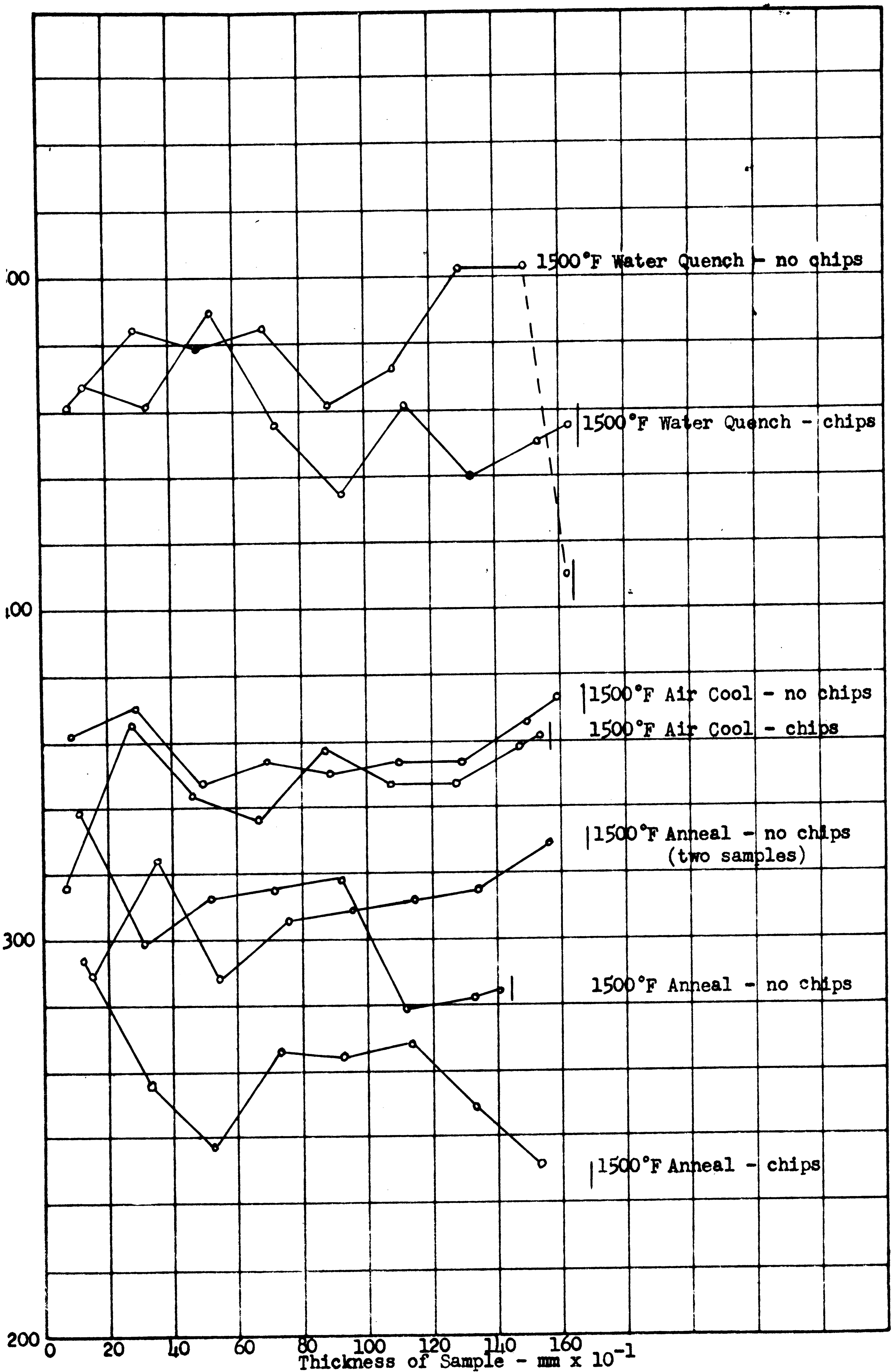


Figure 1. - Hardness Traverse of Ti 150 Heat Treated as Indicated to Show Effect of Ti Chips in Purifying Argon Atmosphere.

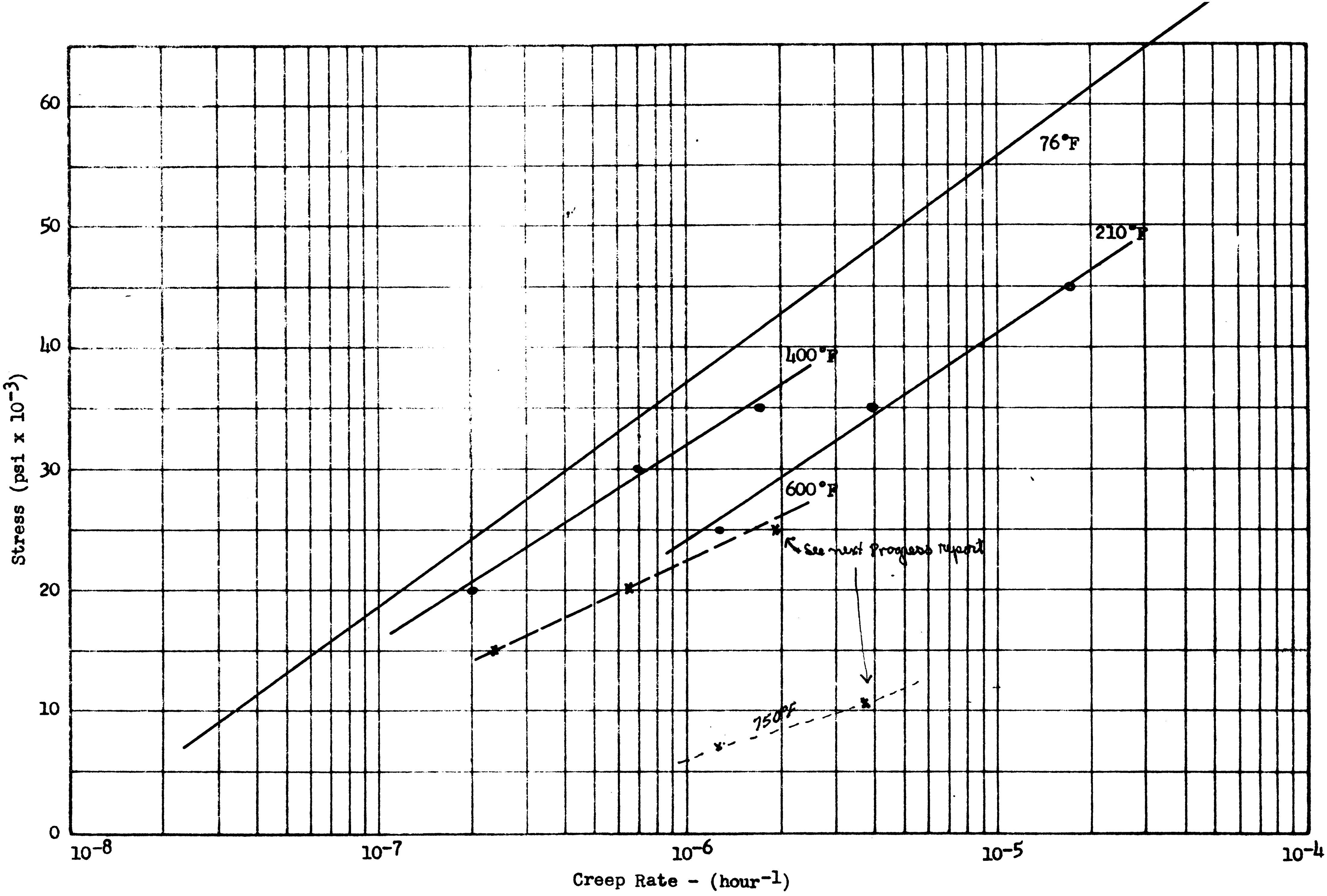


Figure 2. - Effect of Testing Temperature on the Creep Resistance of Annealed Ti 75.

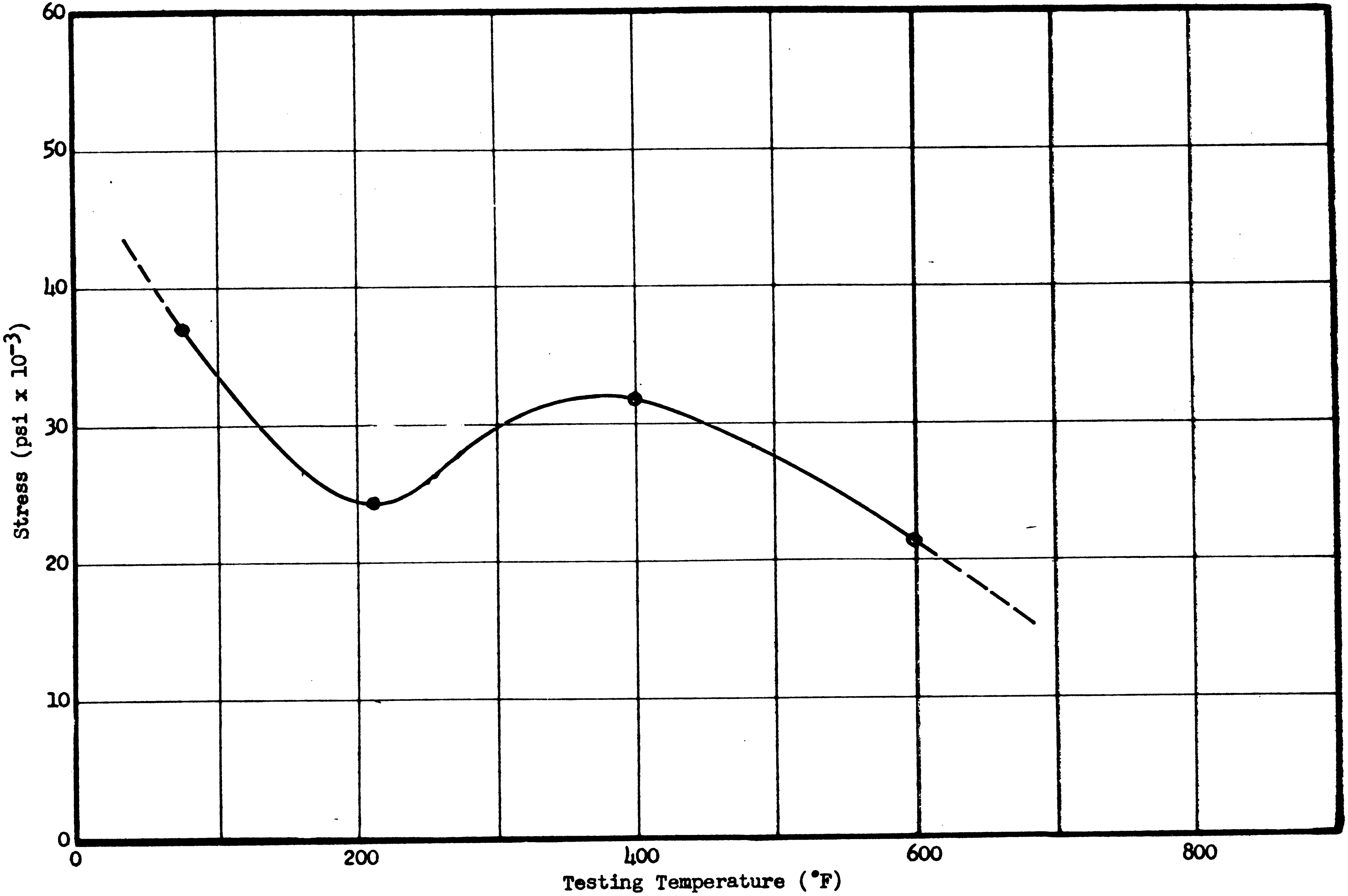


Figure 3. - Stress Required for a Creep Rate of 10^{-6} (hr^{-1}) at Various Testing Temperatures on Annealed Ti 75.

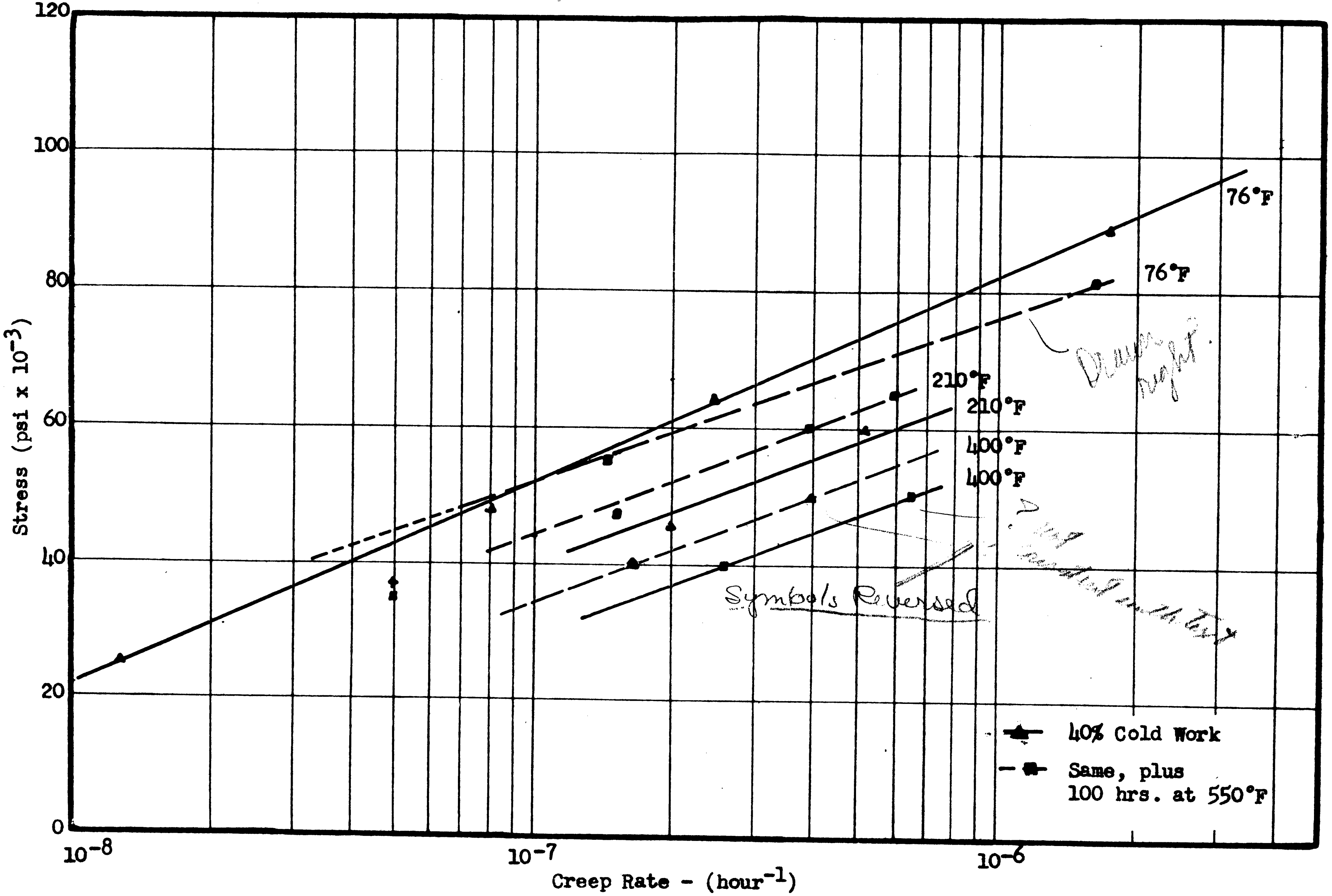


Figure 4. - Effect of Stress and Temperature on the Creep Resistance of Annealed Ti 75 in the Above Conditions Prior to Testing.

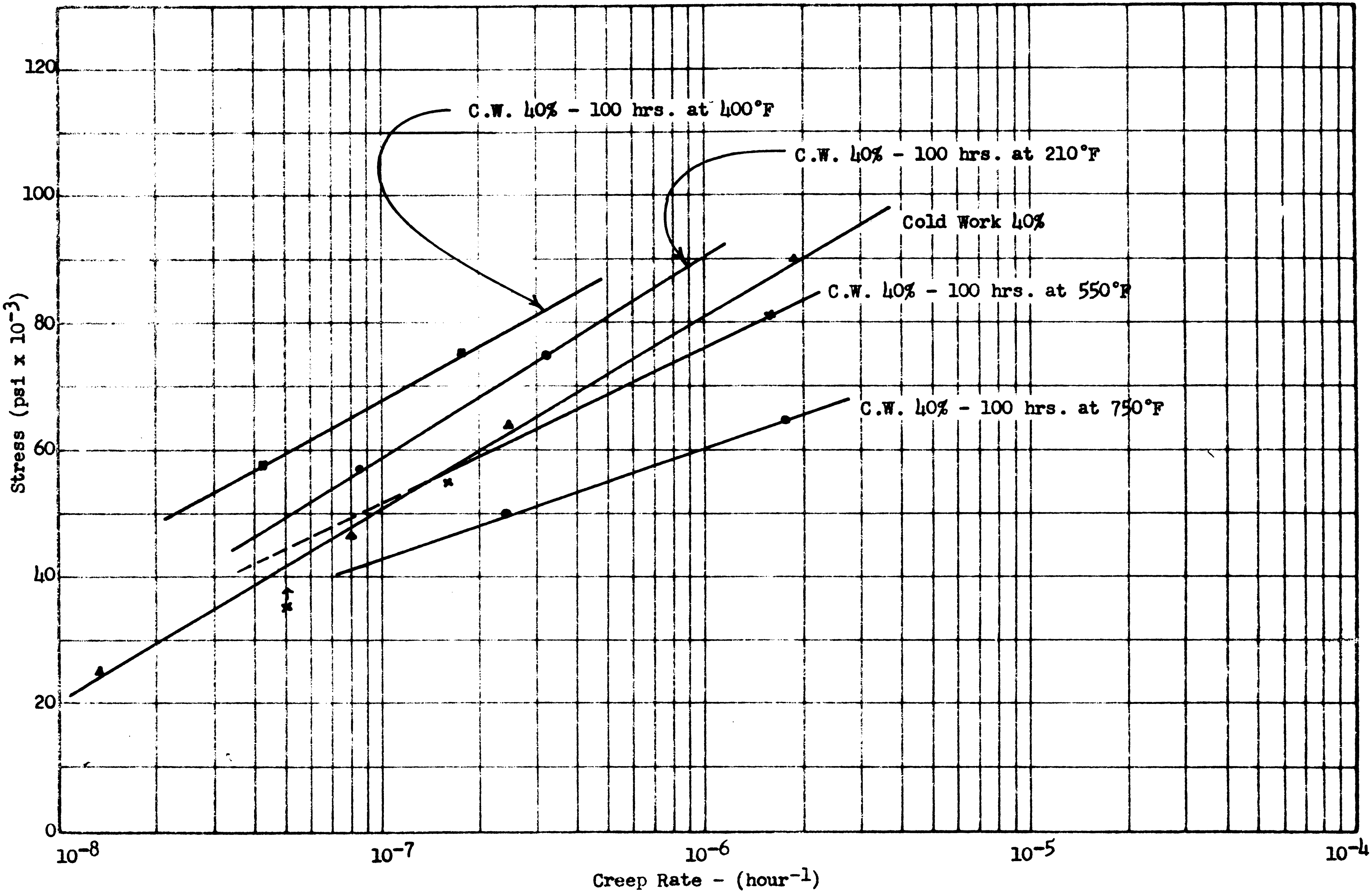


Figure 5. - Effect of 100 Hours at Various Recovery Temperatures After 40 Percent Cold Working on the Creep Resistance of Annealed Ti 75. at ?

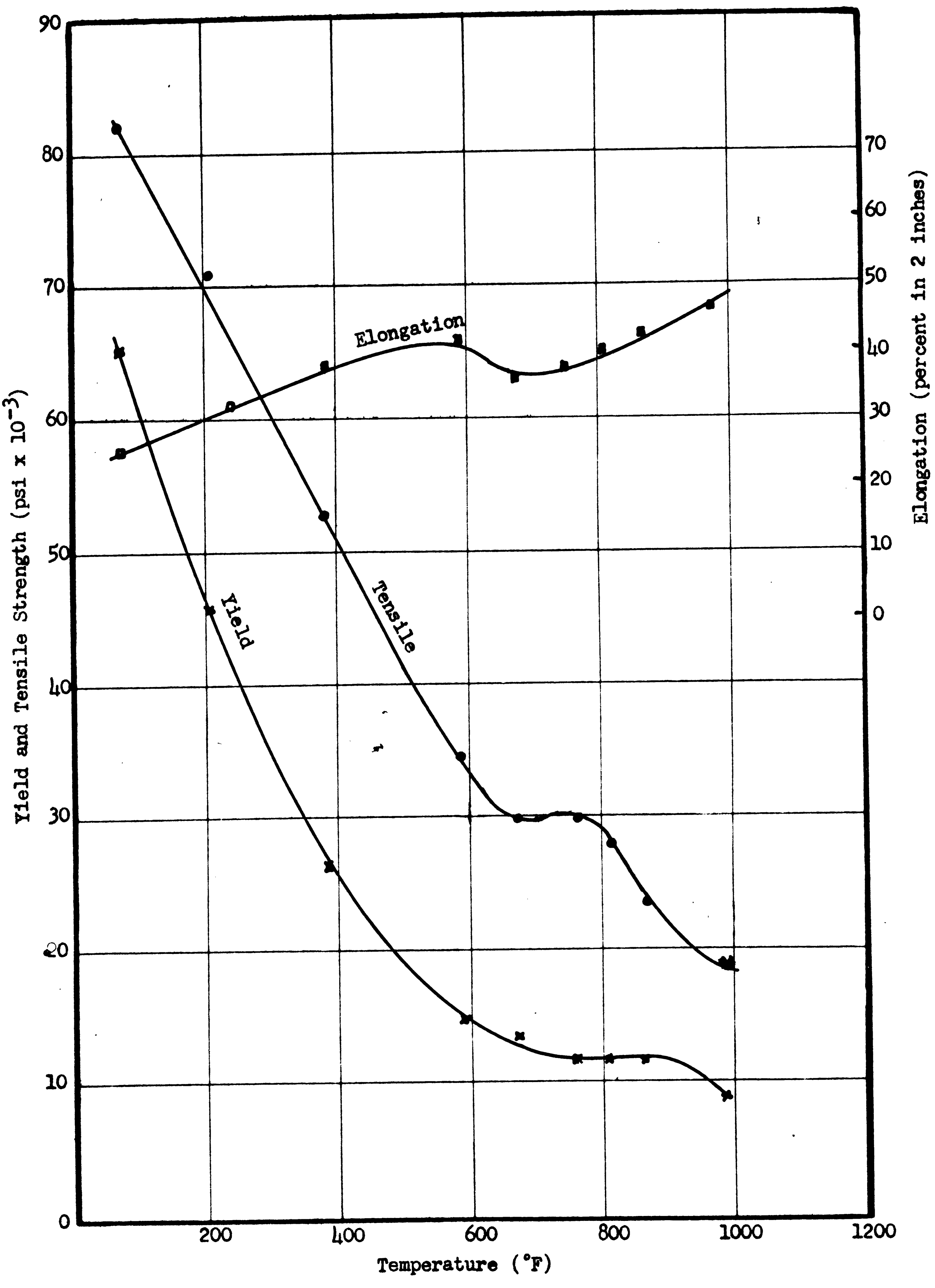


Figure 6. - Effect of Temperature on the Tensile Properties of Annealed Ti 75.

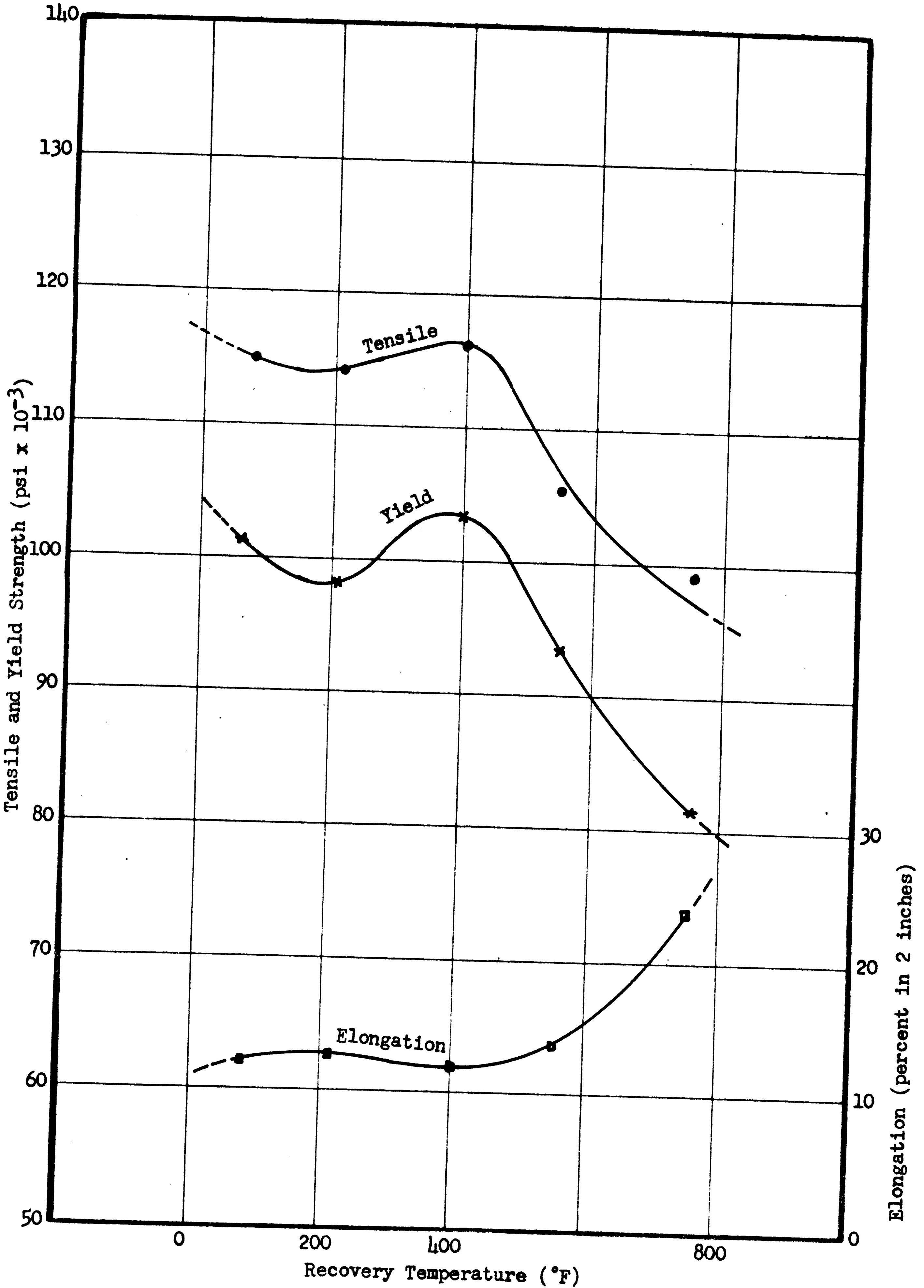


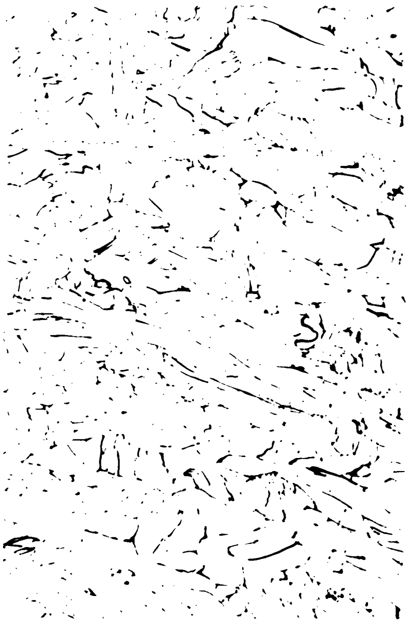
Figure 7. - Effect of 100 Hours Recovery Time at Various Temperatures on the Tensile Properties of Annealed Ti 75 Cold Worked 40 Percent.

52-34

X250

Figure 8. -

Ti 75 Cold Worked 40 Percent



52-35

X250

Figure 9. -

Ti 75 Cold Worked 40 Percent and Recovered 100 Hours at 750°F

Etchant: 2 HF, 2 HNO_3, 100 H_2O

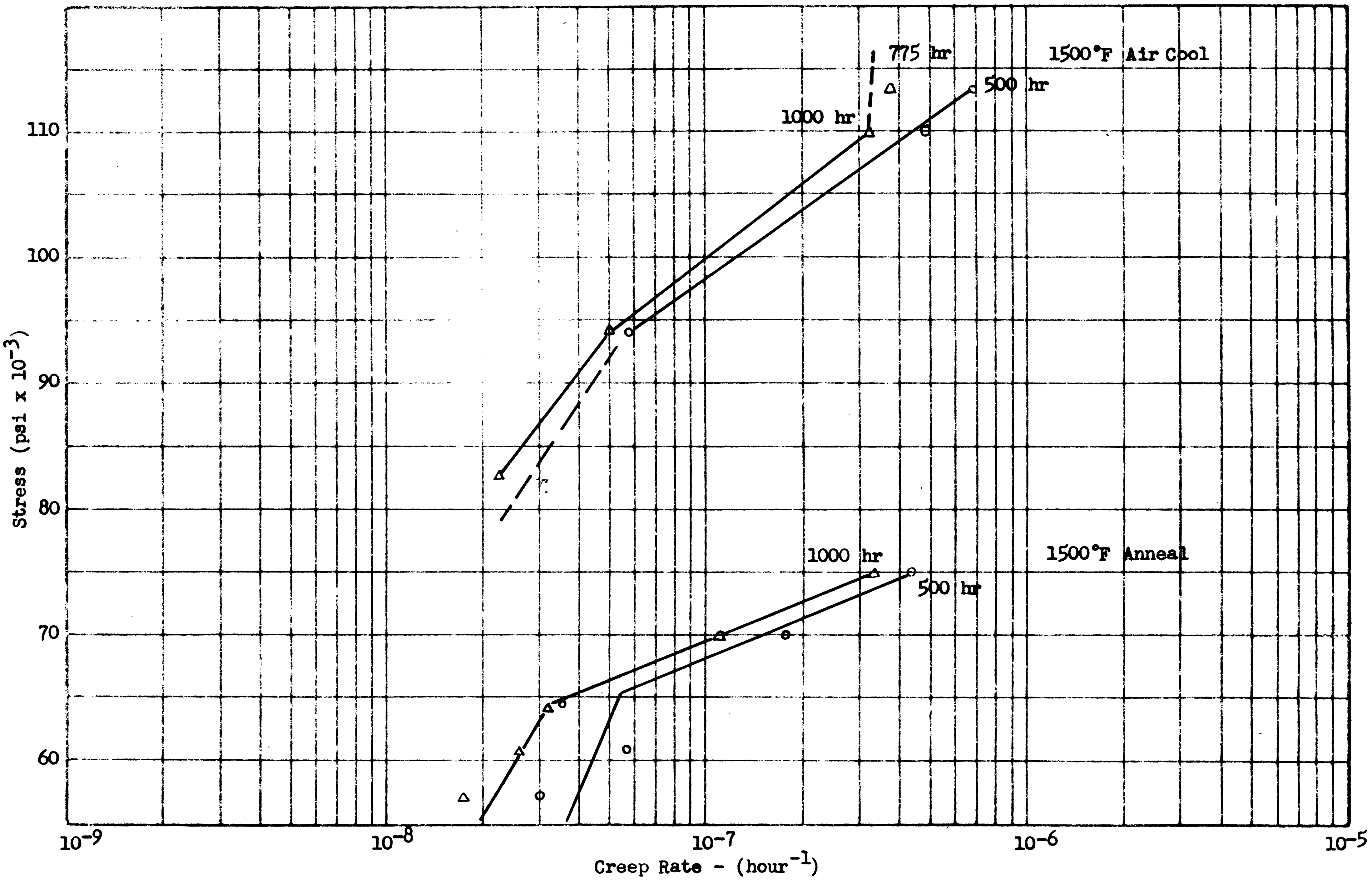


Figure 10. - Stress Versus Creep Rate at 76°F for Ti 150 Heat Treated from 1500°F as Shown.

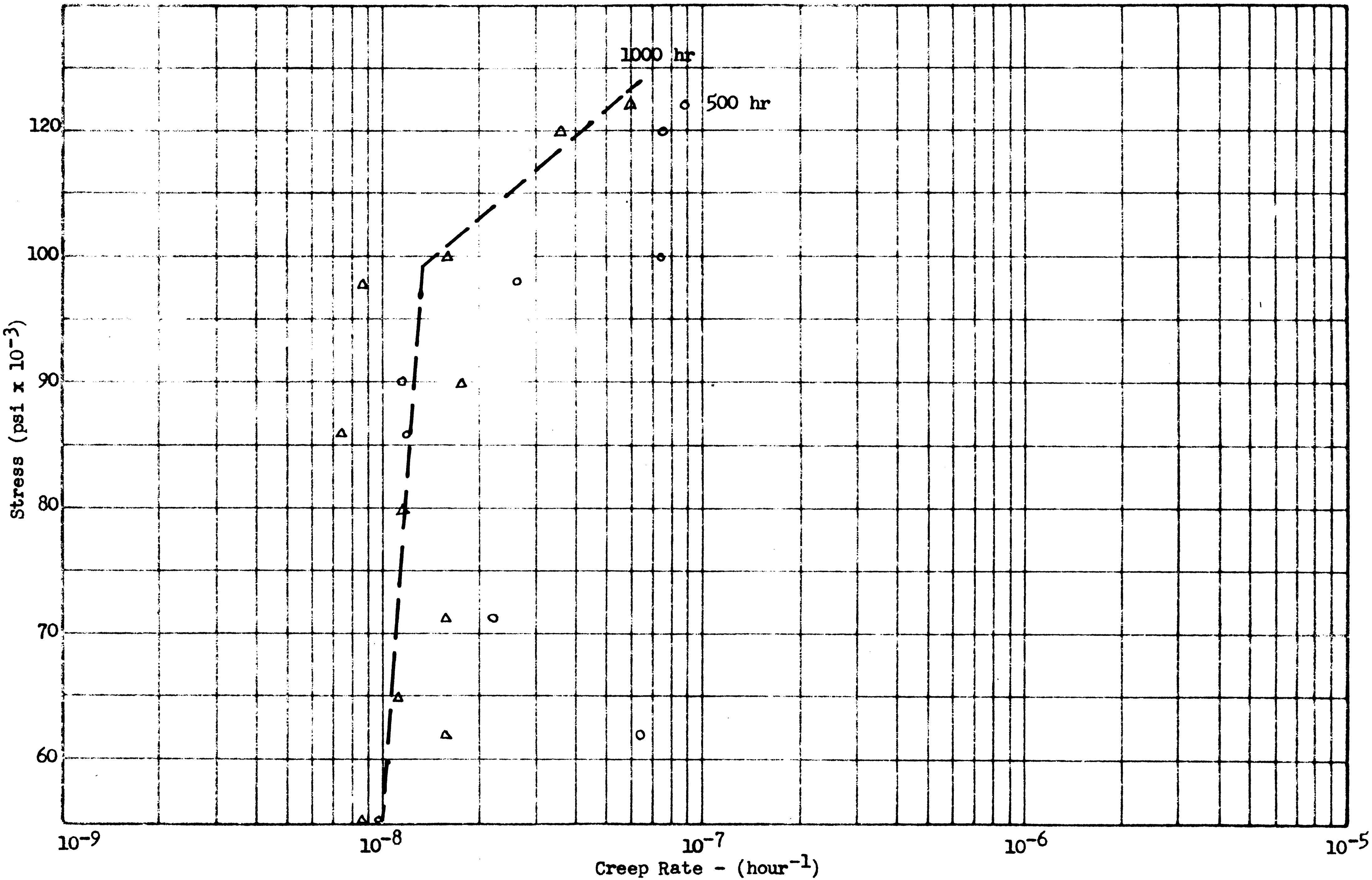


Figure 11. - Stress Versus Creep Rate at 76°F for Ti 150 Water Quenched from 1500°F.

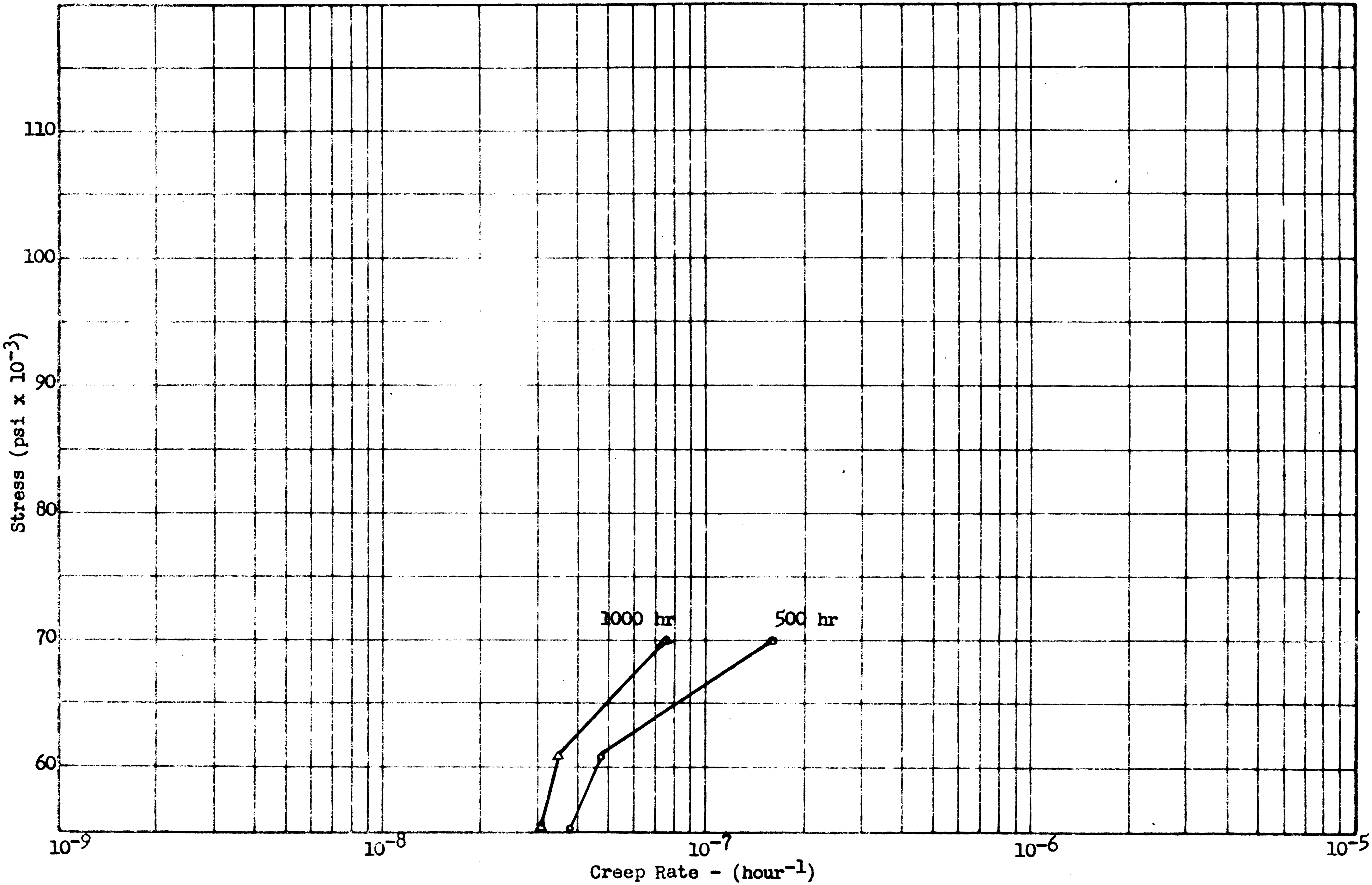


Figure 12. - Stress Versus Creep Rate at 76°F for Ti 150. Heat Treatment: 1 hour 1500°F + Air Cool + 1 hour 1300°F + Anneal.

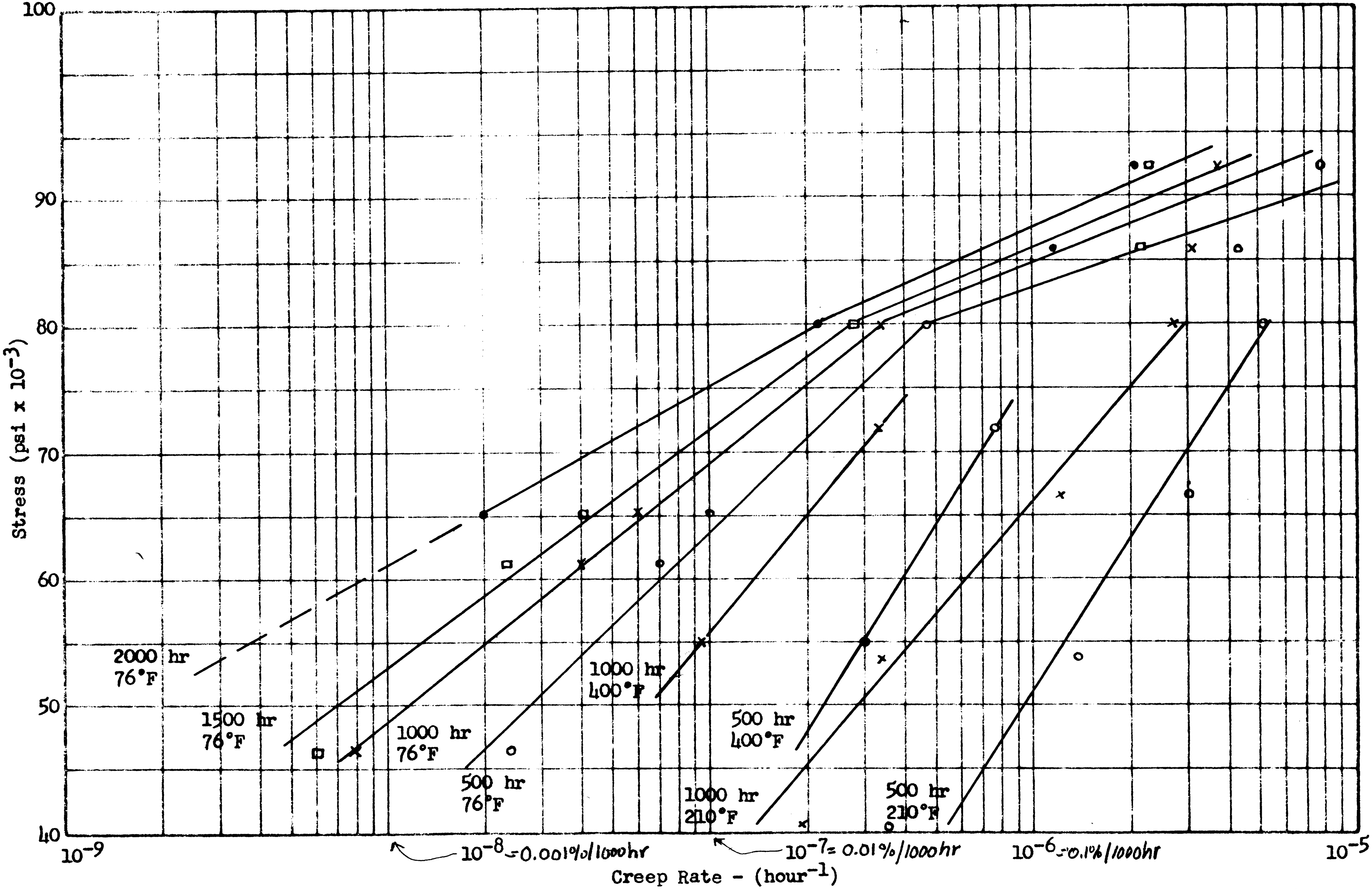


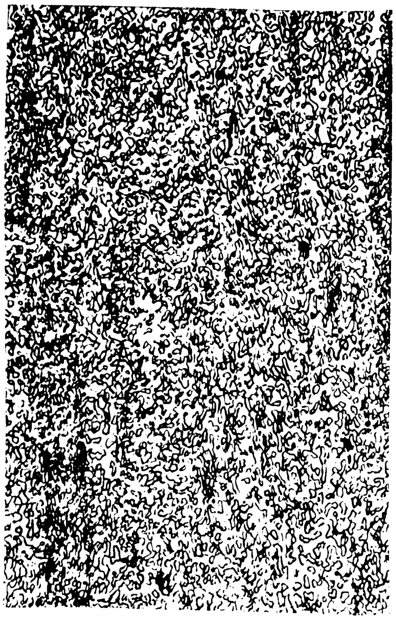
Figure 13. - Stress Versus Creep Rate Curves at 76°, 210°, and 400°F for Various Periods of Testing Time for Ti 150 As Received (1275°F Solution Treat, Water Quench, 1200°F 2 Hours).

Effect of Creep Testing on As-Received Structure of Ti 150
(Solution treat 1275°F, W.Q., 2 hrs. at 1200°F)

52-36

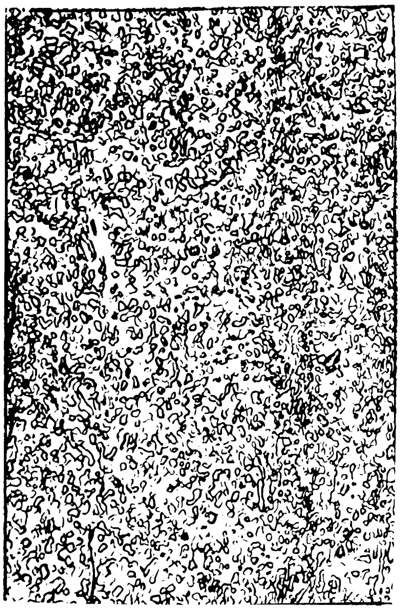
X500

Figure 14. - As Received
Before Testing



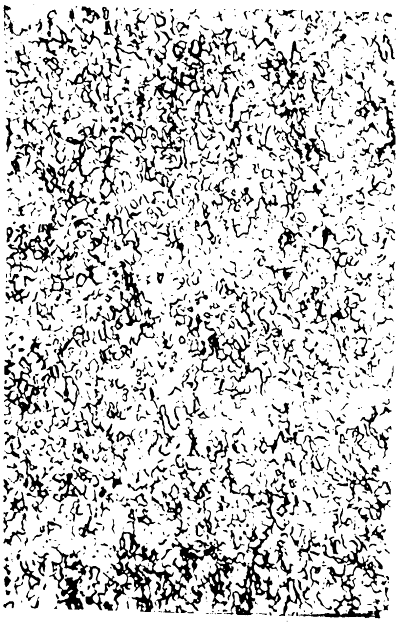
X500

Figure 15. - 2800 hrs. at 76°F
- 92,500 psi



X500

Figure 16. - 1000 hrs. at 210°F
- 80,000 psi



X500

Figure 17. - 1079 hrs. at 400°F
- 72,000 psi

Etchant: 1 part HF, 1 part Glycerine

Effect of Double Heat Treatment on Microstructure of Ti 150

X500

Figure 18. - Air Cooled
from 1500°F

X500

Figure 19. - Annealed
from 1300°F

X500

Figure 20. - Air Cooled from 1500°F
+
Annealing from 1300°F

Etchant: 1 part HF, 1 part Glycerine