# FOURTH PROGRESS REPORT

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THE OFFICE OF AIR RESEARCH

ON

# A STUDY OF CREEP OF TITANIUM AND TWO OF ITS ALLOYS

By

W. R. Kiessel

J. V. Gluck

J. W. Freeman

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# A STUDY OF CREEP OF TITANIUM AND TWO OF ITS ALLOYS

## SUMMARY

During the period covered by this report, 1 July 1951 to 30 September 1951, creep testing at 76°F of the Ti 75 material cold worked 0, 7, 12, and 40 percents has been completed. It has been found that a reduction of 7 percent greatly increases the creep resistance and that further cold working continued to increase the creep resistance but at a decreasing rate. At 40 percent reduction, approximately 230 percent increase was realized. Two creep tests at 76°F, on material which had recovered after cold working, have been completed. In these tests a large decrease in the total amount of extension was noted.

Measurement of the hardness and the (114) line breadths have been completed on Ti 75 material which had been cold worked 7, 12, and 40 percent and allowed to recover at temperatures of 210°, 400°, and 600°F for time intervals to 1000 hours. It was found that the line breadth measurements show recovery before the hardness measurements, this being especially evident on the materials cold worked 7 and 12 percent and recovered at 210°F and the material cold worked 7 percent and recovered at 400°F. The line breadth measurements showed a decrease while the hardness values give no apparent signs of changing.

On the basis of the above determinations a preliminary correlation of second-stage creep rates was attempted. It appears that line width determinations may be a better indication of the second-stage creep properties than hardness measurements.

An extension of the (114) line breadth studies on Ti 75 to reductions of 85 percent showed a relatively flat maximum in the range of 50 to 75 percent reductions. Further reductions resulted in decrease of line breadths.

Creep tests on Ti 150, which had been cooled from 1500°F at various cooling rates, are being carried out. It is apparent that the faster the cooling rate

the more creep resistant the material. Examination of photomicrographs of these materials showed a change in the amount and size of the $\alpha$ grains in the $\beta$ matrix as the cooling rate was varied.

Creep tests at 76°F of the Ti 150 in the as-received condition are reported. Tests extended to times of 2000 to 3000 hours failed to show termination of first stage creep.

Anomalous results obtained on line breadth studies on Ti 150 have resulted in the abandonment of this method of measuring internal strain, at least for the immediate future.

## INTRODUCTION

This report covers the period from 1 July 1951 to 30 September 1951 on Air Force Contract Number: AF33(038)-14111 (Expenditure Order Number: 460-36 BR-1) for the study of creep in titanium and two titanium base alloys in the form of commercially produced 0.060-inch thick sheet.

Work has continued on the commercially pure titanium (Ti 75) and a ferro-chrome alloy (Ti 150). The third material, a manganese alloy (Ti 130), has not yet been received. The object of this work is to study the fundamental structural factors affecting the creep properties.

Ti 75 is being studied from the viewpoint of determining the effect of cold work on the internal structure and on creep resistance. Heat treatments are the main variable being investigated in Ti 150.

## RESULTS AND DISCUSSION

### Commercially Pure Titanium (Ti 75)

The first variable being investigated for Ti 75 is the effect on the creep characteristics of internal strain introduced by cold work. An anneal from 1600°F reduced the internal strain to the minimum value obtainable. Both the

line breadths of the (114) X-ray diffraction line and the hardness are being used as a measure of internal strain. Recovery from cold work by annealing is being studied for two reasons. First, if creep resistance is mainly a function of internal strain, then the same creep resistance should be characteristic of materials either cold worked to a given internal strain or initially cold worked to a higher degree of strain and reduced to the lower level of strain by recovery during annealing. Furthermore, the maintenance of increased creep resistance imparted by cold work should be limited to the temperature below which significant recovery cannot occur during the tests.

The creep characteristics are to be evaluated at those temperature from the sub-zero range to as high as 600°F as may be necessary to develop the fundamental principles of creep in titanium.

Line Broadening Studies. The (114) diffraction line width measurements have been extended from reductions of 40 percent to those of 80 percent at room temperature with the results shown in figure 1. Check determinations for reductions of 7 and 12 percent are included. The line width reaches a fairly flat maximum for reductions in the range of 30 to about 75 percent. A significant decrease resulted from further reductions.

It thus appears that a reduction of 40 percent will be typical of the maximum internal strain which can be imparted by cold work. Further consideration is being given to the significance of the wide range in reductions without change in line breadths and the decrease at an 85 percent reduction. However, one explanation of the reduction of line breadths at 85 percent is possible. Since lower amounts of reduction will start recovery at temperatures slightly above room temperature, it is probable that the extreme amount of reduction depressed the temperature for the start of recovery to a value somewhat less than room temperature.

Line Recovery Studies. Line recovery studies on Ti 75 reduced 7, 12, and 40 percent have been completed and are given in figures 2 through 4. Times to 1000 hours were used at temperatures of 210°, 400°, and 600°F.

Two important observations can be made. First, it will be noted that at 400° and 600°F recovery is extensive at all degrees of cold working studied. At 210°F appreciable recovery is to be observed in the 12 and 40 percent reductions, while that at 7 percent reduction has probably just started. This evidence, plus negative results on line breadth tests run on samples of cold worked material aged at room temperature, is proof of no recovery at room temperature for reductions up to 40 percent. Second, it will be noted that after a period of approximately 20 hours all samples have reached a condition of no further apparent recovery with increased time. This evidence suggests the possibility of a wide variation in the activation energies of the flow units, or dislocations, present in the material after cold working. Holding the material at an elevated temperature allows thermal energy to dissipate those dislocations of low energy requirements. The probability of the remaining dislocations acquiring sufficient thermal energy for motion out of the lattice is small, and recovery appears to reach an equilibrium level. More is to be said concerning this observation when the creep results are considered.

Hardness Measurements. In conjunction with the line breadth recovery measurements described above, hardness determinations were made. These results are given in figures 5 through 7.

At 600°F all degrees of cold working exhibited the expected decrease in hardness during recovery. However, little or no recovery was noted at 210°F for the 7 and 12 percent reductions and at 400°F for the 7 percent reduction, in the time interval considered.

The difference in the behavior of recovery between hardness and line breadth measurements is not without precedent; a similar observation having been made by Wilson and Thomassen[1]. However, an exact explanation for the variation remains to be found. Because of this difference in behavior it is to be expected that a difference in their respective correlations with creep properties will exist.

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(1) Wilson, J. E. and Thomassen, L., Trans. ASM., 22, 769, (1934)

Since hardness measurements are taken through the cross section and line breadth measurements are taken on the surface of the specimens (after removal of approximately 0.006-inch), the question might be raised as to whether representative material was tested in both cases. In order to check this, additional surface layers of the sample cold worked 7 percent were carefully etched off (without considerable care the bath temperature could become high enough to start recovery) and the line breadth measurements taken. No change was observed.

Creep-Test Data. The "steady-state" creep rates (average rate between 500 and 1000 hours) at room temperature are shown in figure 8 as a function of stress for reductions of 0, 7, 12, and 40 percent. The same data is replotted in figure 9 as a percent of the yield stress. The yield stress was only established for the material annealed from 1600°F (55,400 psi). The values used for the cold-worked conditions were estimated from hardness data inasmuch as there is not enough test material available for tensile tests on all degrees of reduction.

Cold working greatly increases the creep strength at room temperature, the degree of increase becoming somewhat greater with stress. The degree of improvement increases markedly with greater reductions up to the 40 percent investigated. The effect of cold work on creep strength when the latter is expressed as a percent of the yield strength is quite different than when expressed as a function of stress alone. Cold work does increase the creep resistance at a given fraction of the yield strength, however there is little effect from increasing amounts of reduction. Furthermore, the curves tend to be parallel rather than to converge as for the case where stress alone is considered.

Referring again to figure 9 it will be noted that the results of the present investigation are compared with findings of Adenstedt[2]. It is shown that the steady state creep rates for the material being used in this investigation are less dependent upon stress than those found by Adenstedt.

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[2] Adenstedt, H., Met. Prog., 56, p. 658, (1949).

This variation may be due to either composition differences (Adenstedt's material contained approximately 0.40 percent carbon and 0.05 percent tungsten while this investigation is concerned with material containing 0.04 percent carbon and 0.50 percent tungsten) or to differences in prior history.

Previous reports had noted that cold working did not greatly reduce primary creep. However, the time necessary for reaching a reasonable steady creep rate was reduced.

Tests are in progress to measure creep resistance after cold working followed by a recovery treatment. To date two such tests have been completed. Upon examination of the extension versus time curves for the cold-worked and recovered samples it was noted that a considerable reduction in the amount of first-stage creep had taken place. In continuing the discussion started in "Line Recovery Studies", it may be postulated that this reduced first-stage creep is due to the dissipation of the low energy dislocations during the recovery treatment. Since first stage creep is the dissipation of the dislocations existing in the material at the start of the test, such a mechanism as described for the recovery treatment should lead to the observed results.

Tests at 210°F are also in progress on the annealed stock. These data, as well as data on the annealed stock at a sub-zero temperature, will be presented in the next report.

Correlation of Internal Strain with Creep Resistance. Sufficient data have been obtained to begin fundamental correlation. The relationship between line width and creep rate under a stress of 50,000 psi at 76°F is shown in figure 10. A similar curve of hardness is included. To further explore the relationship existing between line breadth measurements and stead-state creep rates, two cold worked creep samples were given recovery treatments to various states of internal stress and the steady state creep rates were determined. It will be noted in figure 10 that these results also fall within the correlation.

There appears to be a direct relationship between line width and creep rate, except for the slightly faster creep rate for the annealed sample. In the latter case the test had not reached a constant creep rate in the 1000 hour test and the rate reported represents a faster one than true minimum creep. The latter, if available, would probably fall on the straight line.

There seems to be an unquestionable curvature in the plot of hardness versus creep rate. While the deviation is small it seems that the internal strain as measured by line width is a more direct measure of the creep resistance than hardness.

It cannot be emphasized too strongly that these latter observations are based on limited results. More work must be done, and is being done, to verify these findings.

## Ferro-Chromium Alloyed Titanium (Ti 150)

Creep studies of Ti 150 have been conducted using heat treatment as the variabl to be investigated. The objective has been the establishment of the relationship between micro-structural changes and creep resistance. Wide variations in microstructure are obtained by the effect on transformation characteristics of the alloy additions (2.7% chromium and 1.4% iron).

Considerable difficulty in testing has been encountered both from extreme brittleness of heat-treated structures and the non-homogeneity of the material. Relation of internal strain to creep characteristics through X-ray studies has produced anomalous results.

The necessity for conservation of the limited supply of test material has led to the introduction of a bend test as a sorting measure for possible heat treatments in the planning of creep tests.

Heat Treatment Studies. Heat treatment studies are to be extended using bend and hardness tests, as well as microstructures, to select representative samples

for creep testing. This is to be an extension of previous work which showed that samples heat treated from the $\beta$ phase (above approximately 1575°F) were extremely brittle regardless of whether they were quenched or annealed. Samples treated from 1500°F (the $\alpha + \beta$ region) had sufficient ductility for testing purposes. Microstructures representative of this last treatment are shown in figures 13, 14, and 15.

The bend test equipment is undergoing further development inasmuch as the as-received material was found to fail when bent on the 1/4-inch radius jig originally used. A new jig with a 3/4-inch maximum radius has been constructed.

Creep Testing. Creep testing on Ti 150 has been carried out at 76°F on the as-received stock(solution treated 1275°F, water quench, 1200°F for 2 hours) to the point where curves of stress versus creep rate can be presented (figure 11). Time-elongation curves for these tests indicated in no case that "steady state" (second-stage) creep had been reached at the end of the 1000-hour period originally proposed for creep testing. Prolongation of the tests to 2000 - 3000-hours failed to show any further approach to "steady state" creep in most cases. The data has been plotted to show this effect; the creep rates being taken as the tangent to the time-elongation curves at the indicated times. A fairly consistent trend seems to be indicated from examination of the curves (figure 11). Scatter in the data may be due to both the non-homogeneity of the test material (see Third Progress Report, July 1, 1951, page 6) and the fact the upper limit (about 1.5% total elongation) of strain-gage validity is being approached in the tests run at the highest stress levels. Tensile data taken on a sample of the Ti 150 as-received has shown the yield strength to be 131,200 psi. Creep tests have thus been conducted at stresses up to 70% of this value.

Other tests are in progress at 76°F on material water quenched, air cooled and annealed from 1500°F. Results to date, shown in figure 12, indicate the rapid decrease in creep resistance with a decreasing cooling rate. Annealing produces

the weakest material, while air cooling results in slightly lower creep resistance than water quenching. The annealed material had considerable elongation at fracture.

Micrographs (figures 13, 14, and 15) at 1000X magnification show the variation in microstructure resulting from these treatments. The structure is essentially one of $\alpha$ grains distributed in a $\beta$ matrix. The amount and size of the $\alpha$ grains is a function of the cooling rate.

The equipment for conducting creep tests at elevated temperatures was put into satisfactory operating condition during the period covered by this report. Tests are now in progress at 210°F on the as-received material but as yet not enough data has been accumulated to warrant presentation at this time.

Line Broadening Studies. Anomalous results obtained in line broadness studies on Ti 150 subjected to various cooling treatments from 1500°F have led to the abandonment, for the present at least, of this method of characterizing internal strain. It was expected that the slowest cooling rate, annealing, would show the sharpest X-ray diffraction line, while the fastest rate, water quenching, would be associated with the broadest line. This is in accordance with accepted theories for the mechanism of line broadening due to internal strains. However, the results obtained were exactly the reverse of expectations and indicate for this alloy that the broadest lines are associated with the slowest cooling rate. Creep data (figure 12) shows that the broader lines were obtained from the material with least creep resistance.

The explanation for the effect is believed to be in the fact that compositional variations associated with non-equilibrium phases are present in this material. Other work carried out at the University has shown the dependence of "d", the lattice parameter, on isothermal transformations to be a great one. Such lattice parameter dependence is traceable to variation in the composition of the micro-constituents and indicates the sluggish nature of the difficusion process in this material.

Variations in "d" values obtained as noted above, are thus believed responsible for anomalous line broadening effects. This explanation is being checked and it is hoped that quantitative data will be available soon. Consequently, further attempts at internal strain characterization of the alloy material in terms of X-ray line broadnesses are to be held in abeyance.

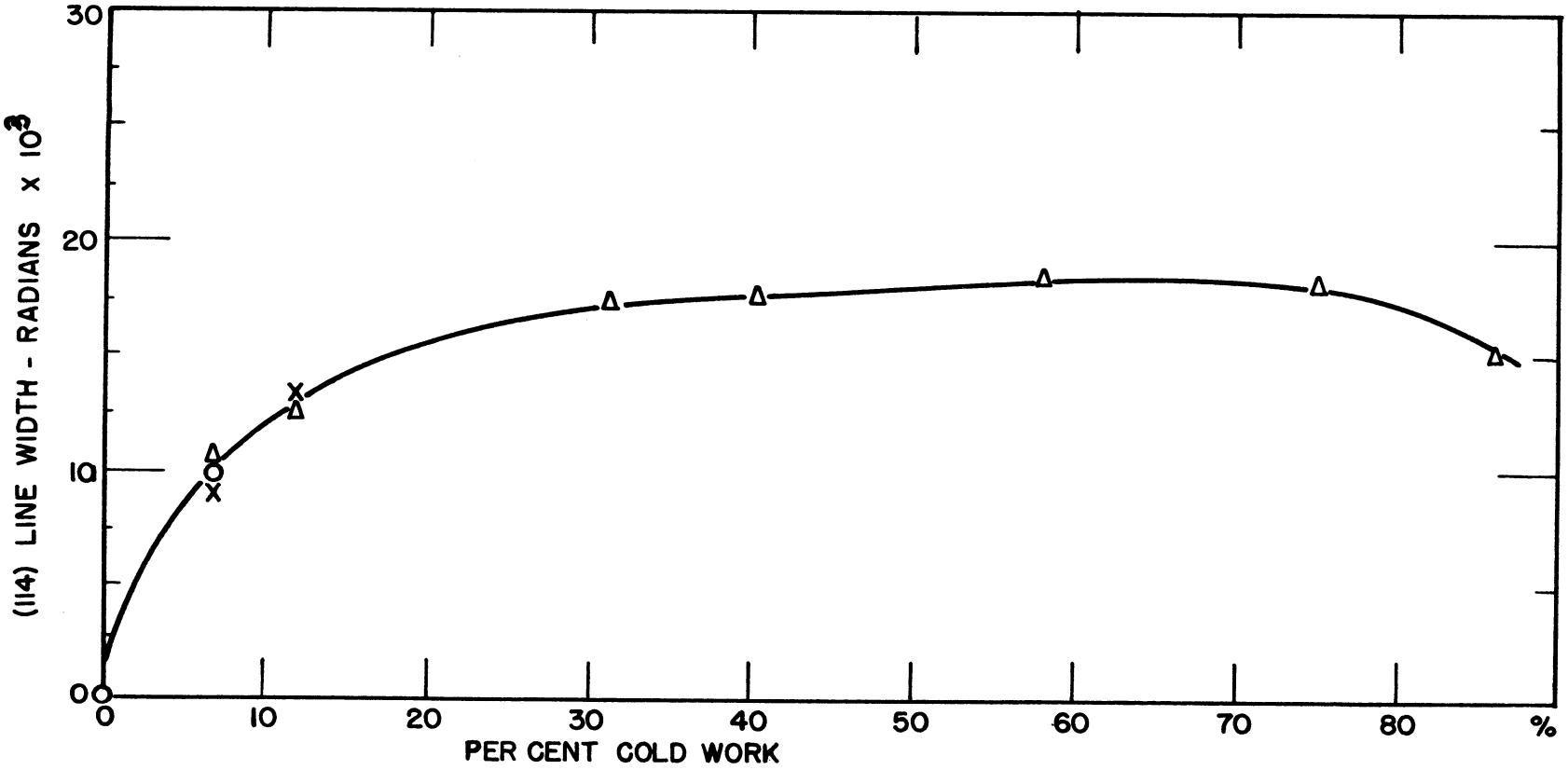



FIGURE 1 STATE OF INTERNAL STRAIN OF Ti 75 DESCRIBED BY THE BROADENING OF THE (114) DIFFRACTION LINE, AS A FUNCTION OF COLD WORKING, AT 80° F

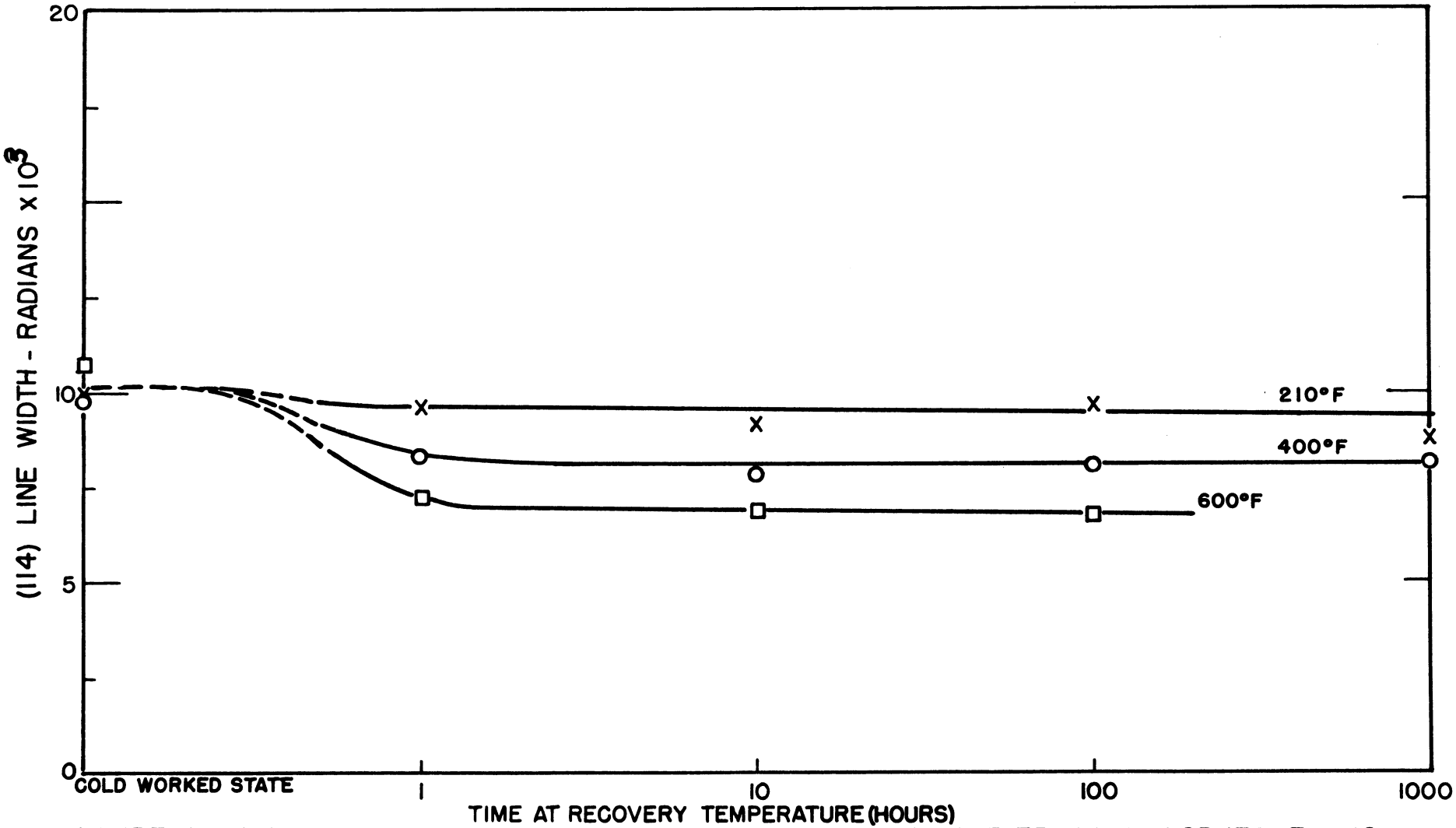



FIGURE 2 LINE WIDTHS OF THE (114) DIFFRACTION LINE AS Ti 75, COLD WORKED 7%, IS ALLOWED TO RECOVER AT THE INDICATED TEMPERATURES.

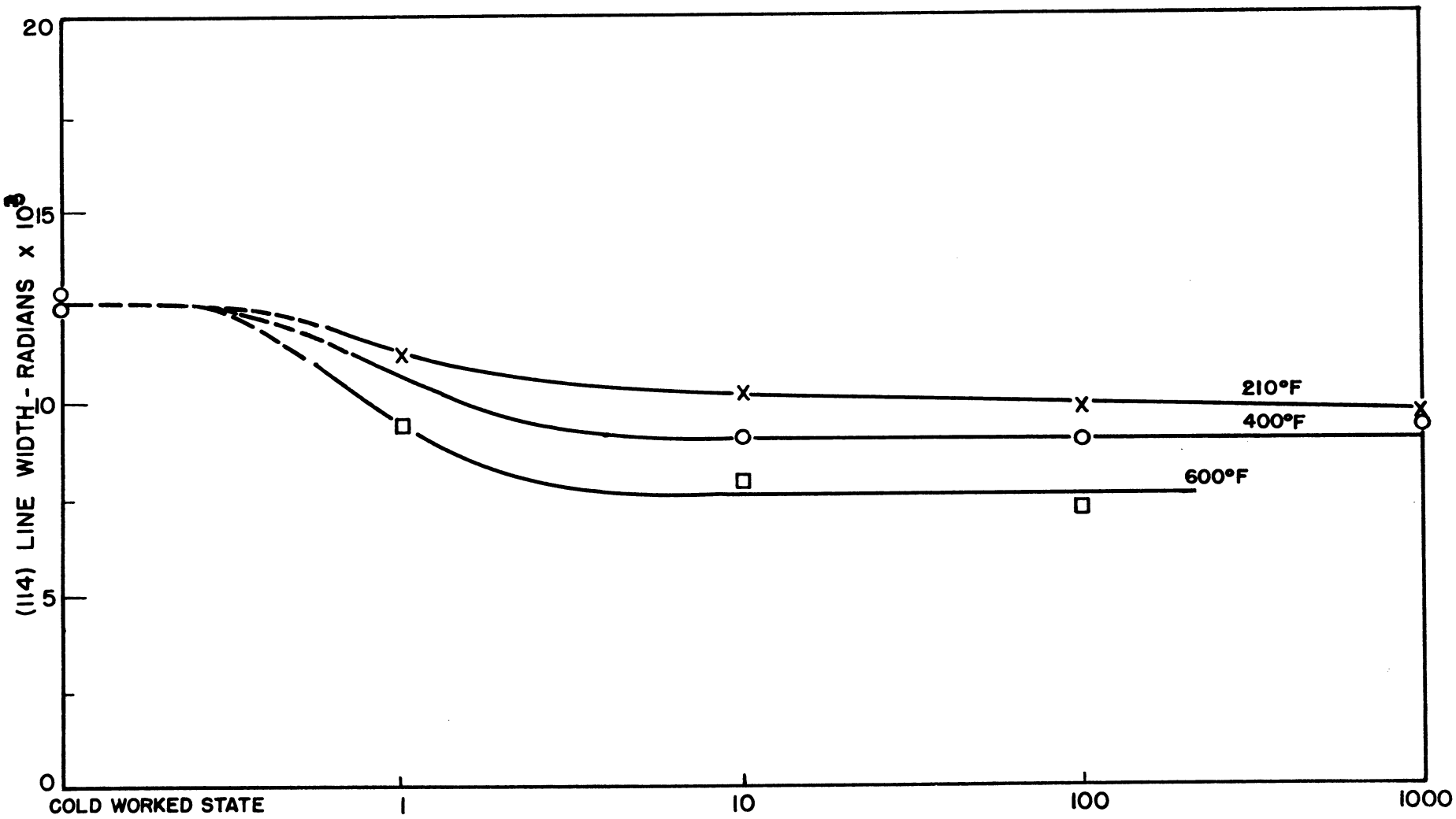



FIGURE 3 LINE WIDTHS OF THE (114) DIFFRACTION LINE AS Ti 75, COLDWORKED 12% IS ALLOWED TO RECOVER AT THE INDICATED TEMPERATURES.

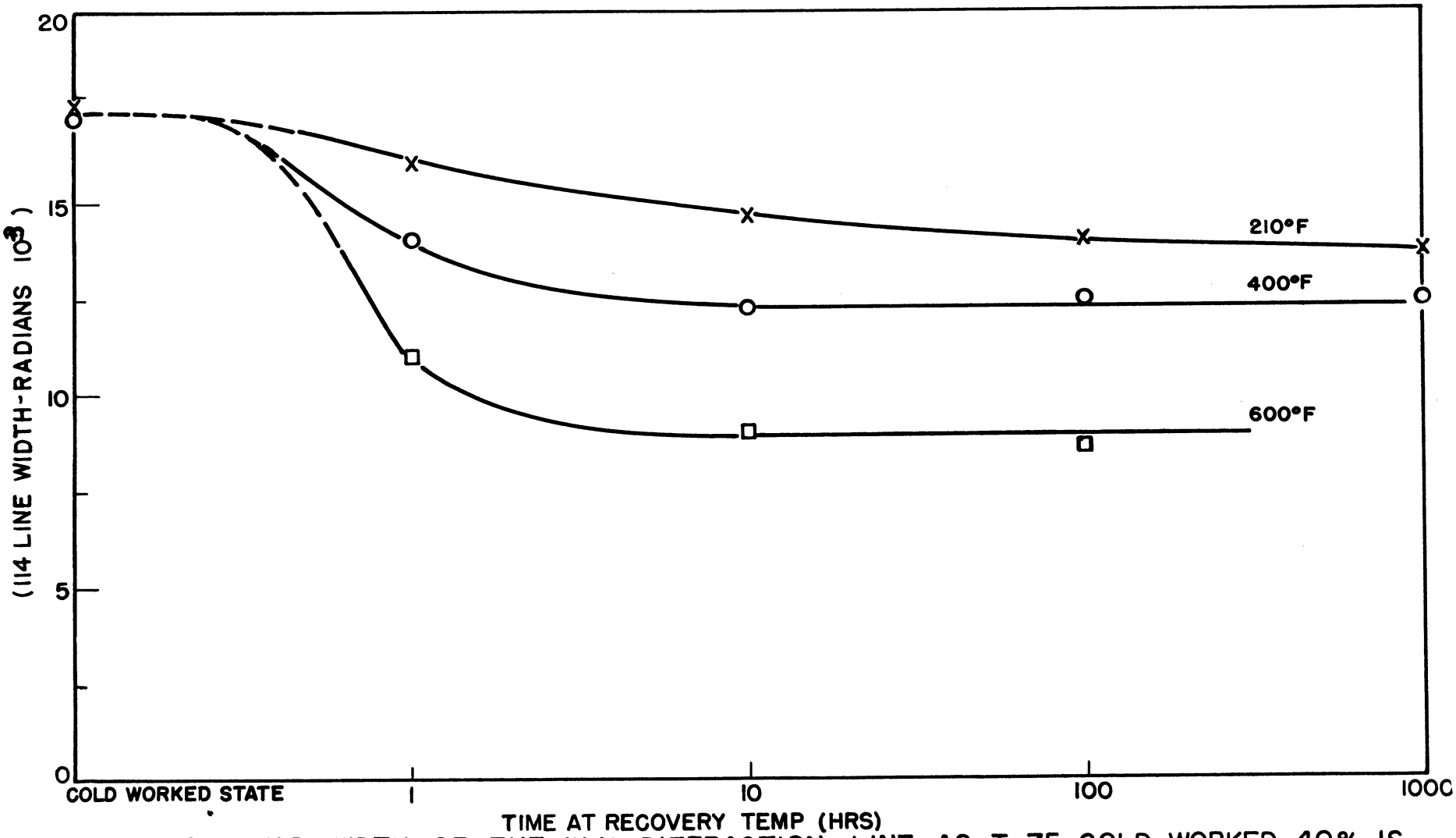



FIGURE 4 LINE WIDTH OF THE (114) DIFFRACTION LINE AS Ti 75, COLD WORKED 40%, IS ALLOWED TO RECOVER AT THE INDICATED TEMPERATURES.

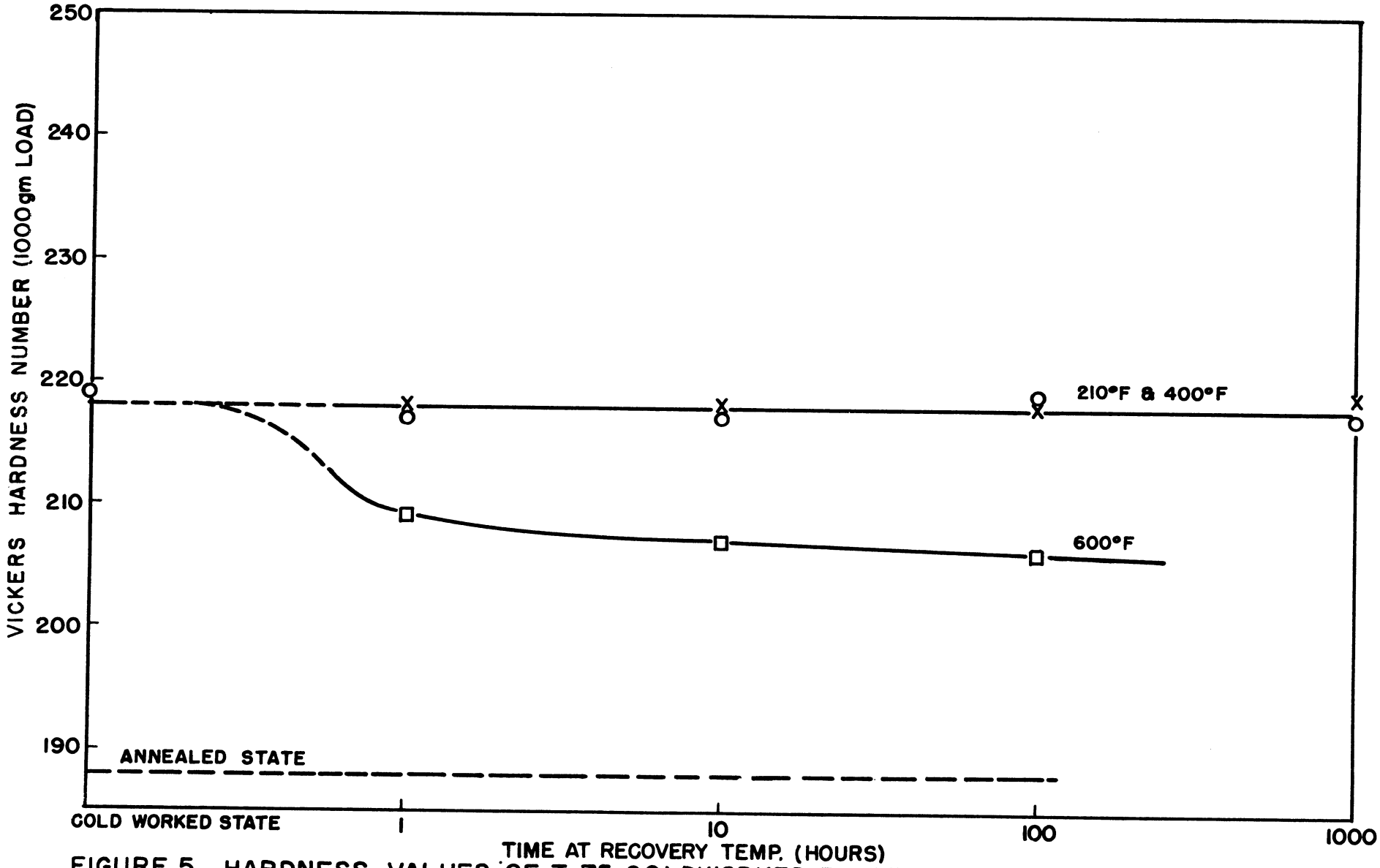



FIGURE 5 HARDNESS VALUES OF Ti 75 COLDWORKED 7% AFTER RECOVERY OF VARIOUS TEMPERATURES

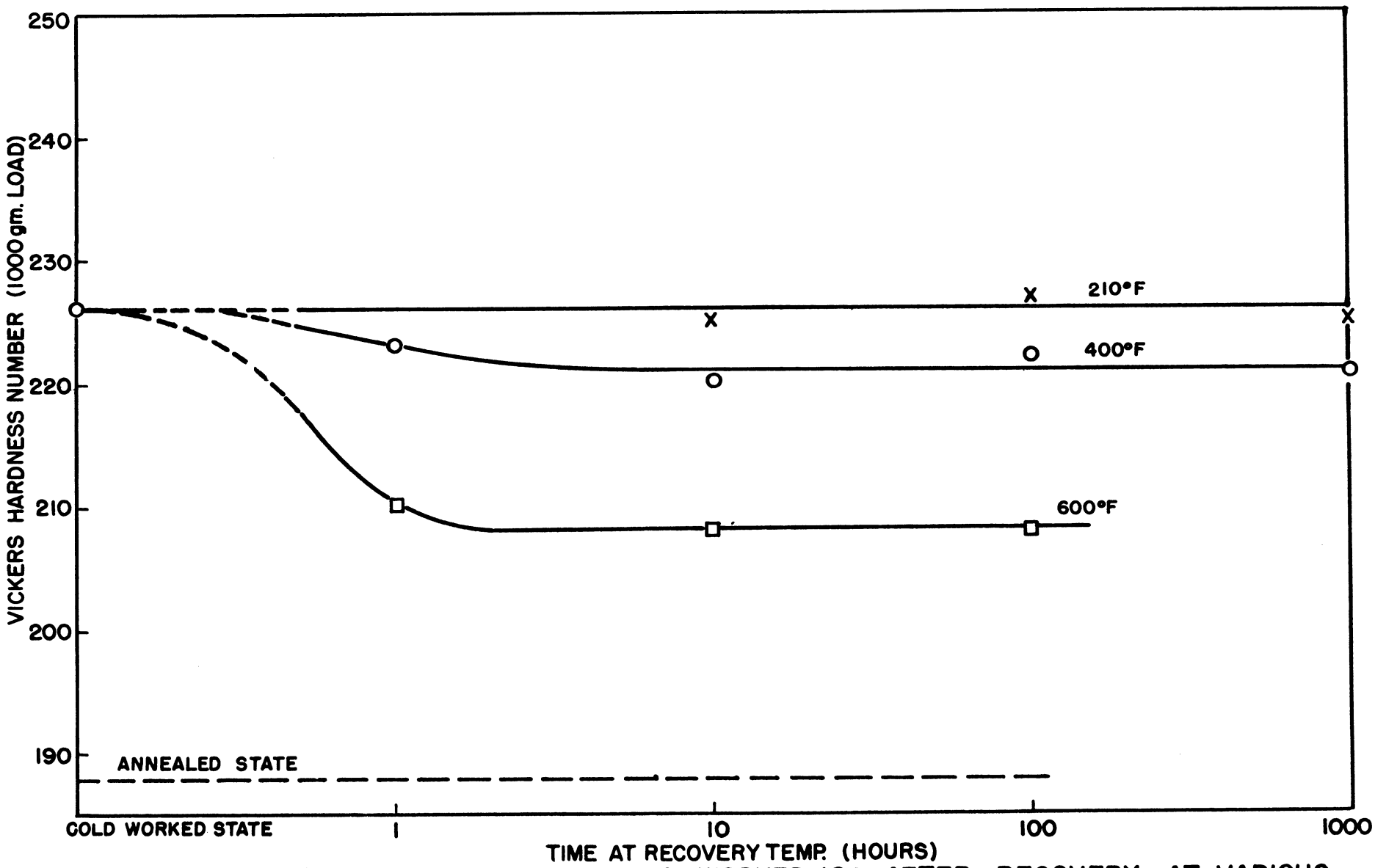



FIGURE 6 HARDNESS VALUES OF Ti 75A COLDWORKED 12% AFTER RECOVERY AT VARIOUS TEMPERATURES.

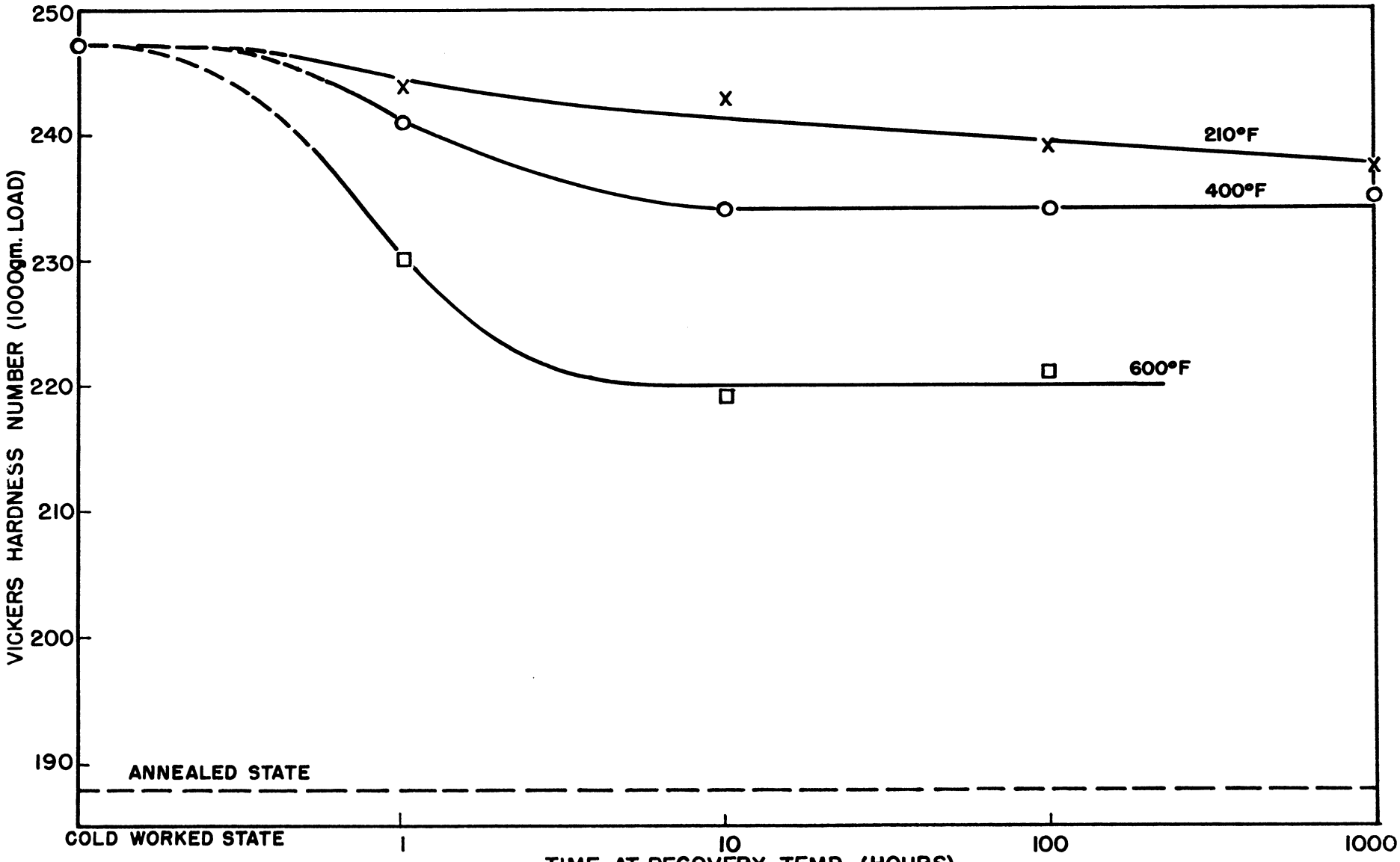



FIGURE 7 HARDNESS VALUES OF Ti 75, COLDWORKED 40% AFTER RECOVERY AT VARIOUS TEMPERATURES.

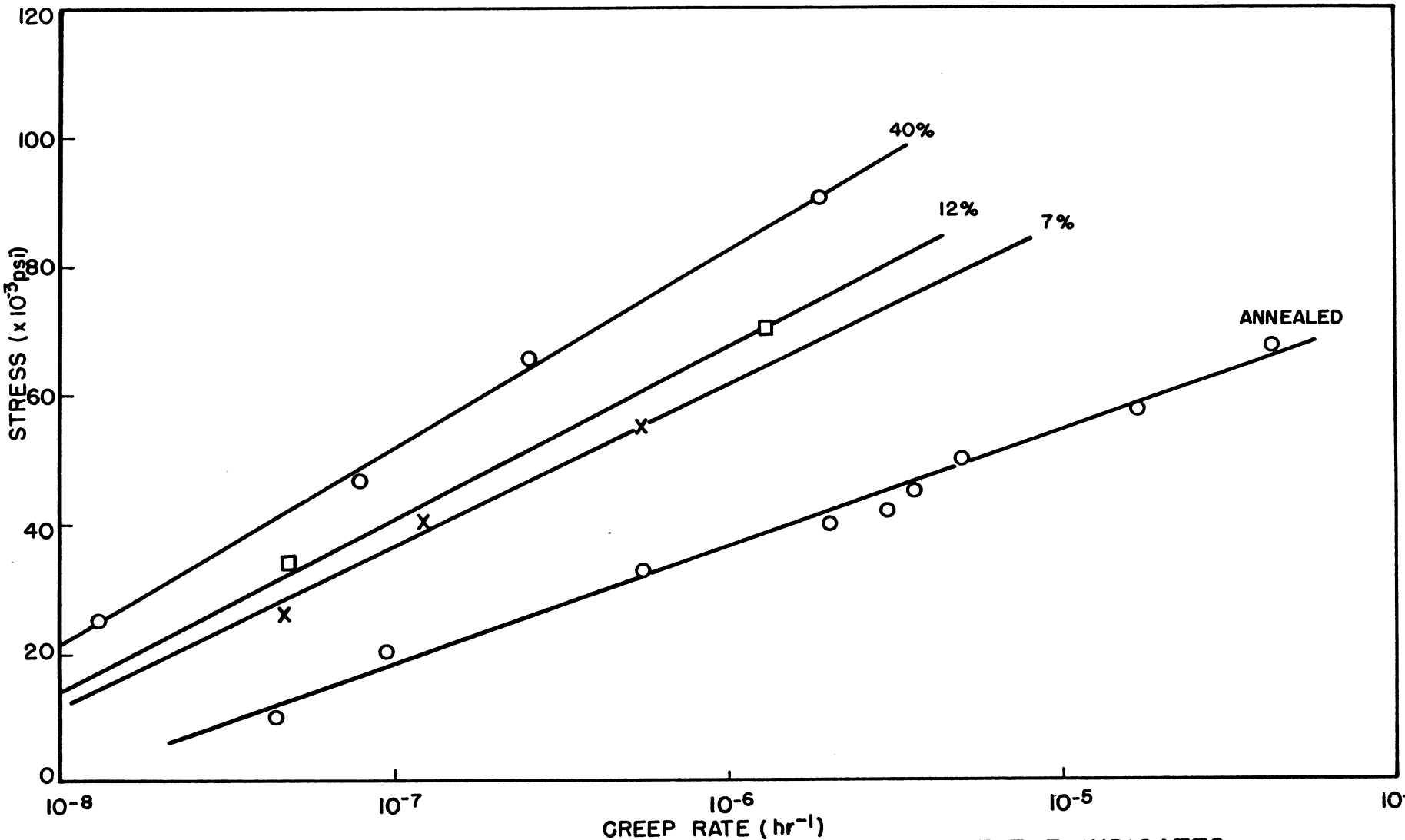



FIGURE 8 CREEP RATE VS STRESS OF Ti 75 AT 76°F AND AT THE INDICATED PERCENTAGE OF REDUCTION

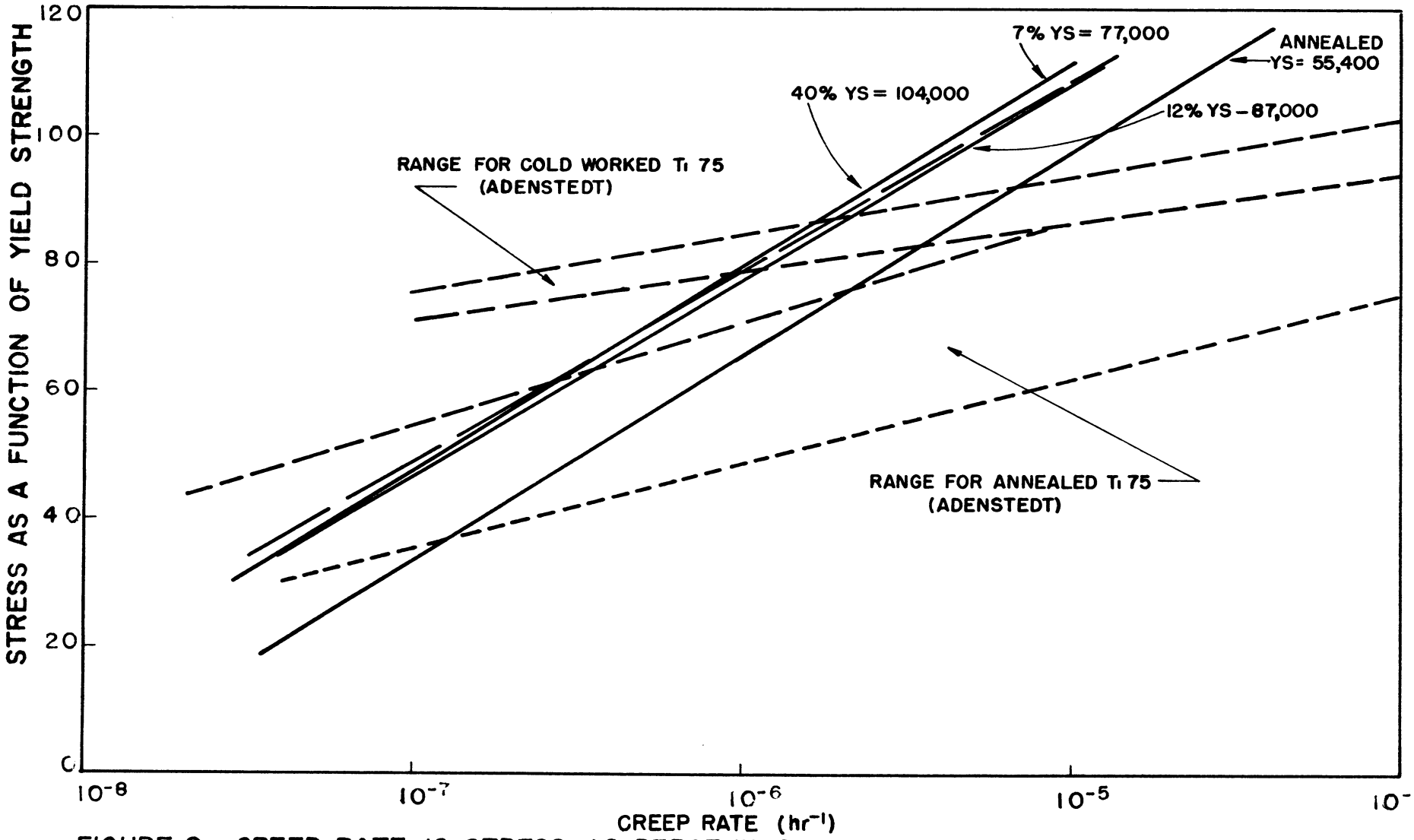



FIGURE 9 CREEP RATE IS STRESS, AS PERCENT OF THE YIELD STRENGTH, FOR Ti 75 AT 76° (EXEPT WHERE INDICATED YIELD STRENGHS ARE ESTIMATED FROM HARDNESS)

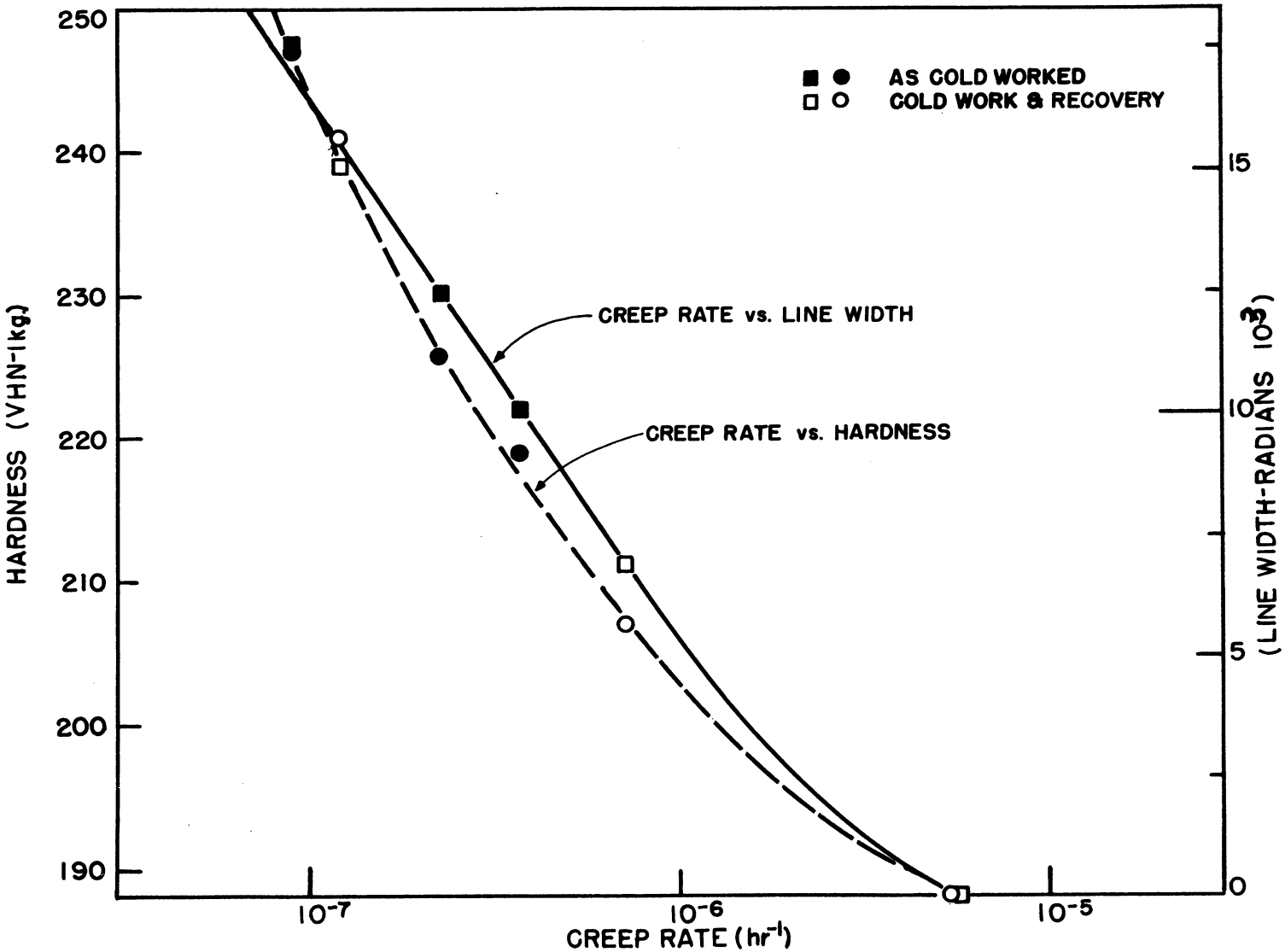
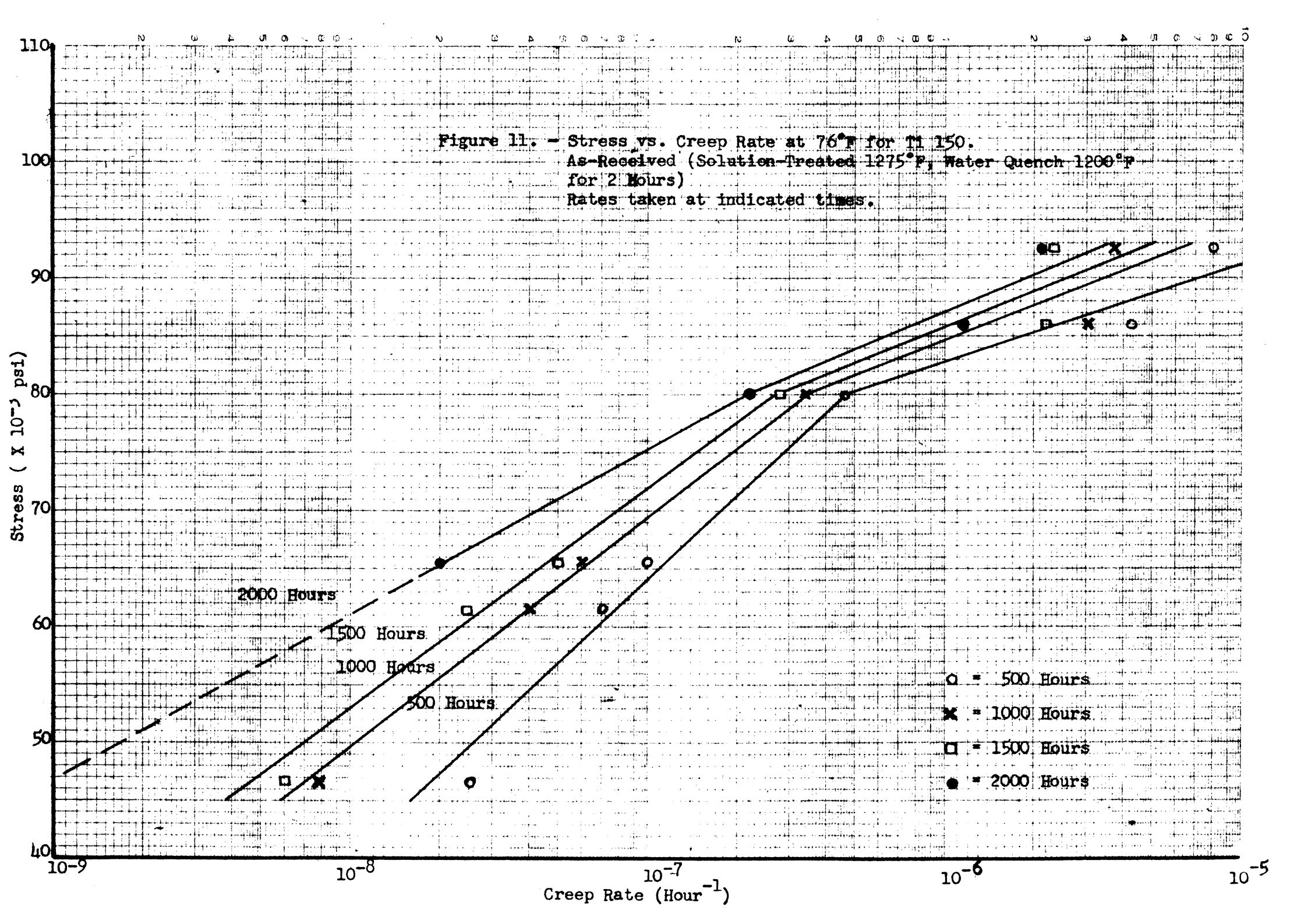



FIGURE 10 CREEP RATE OF Ti 75 AT 50,000 PSI & 76°F AS A FUNCTION OF THE (114) DIFFRACTION LINE WIDTH AND HARDNESS.


Figure 11. - Stress vs. Creep Rate at 76°F for T1 150.
As-Received (Solution-Treated 1275°F, Water Quench 1200°F for 2 Hours)
Rates taken at indicated times.
Stress ( X 10⁻³ psi)
Creep Rate (Hour⁻¹)
110
100
90
80
70
60
50
40
10-9
10-8
10-7
10-6
10-5
2000 Hours
1500 Hours
1000 Hours
500 Hours
o = 500 Hours
x = 1000 Hours
□ = 1500 Hours
● = 2000 Hours

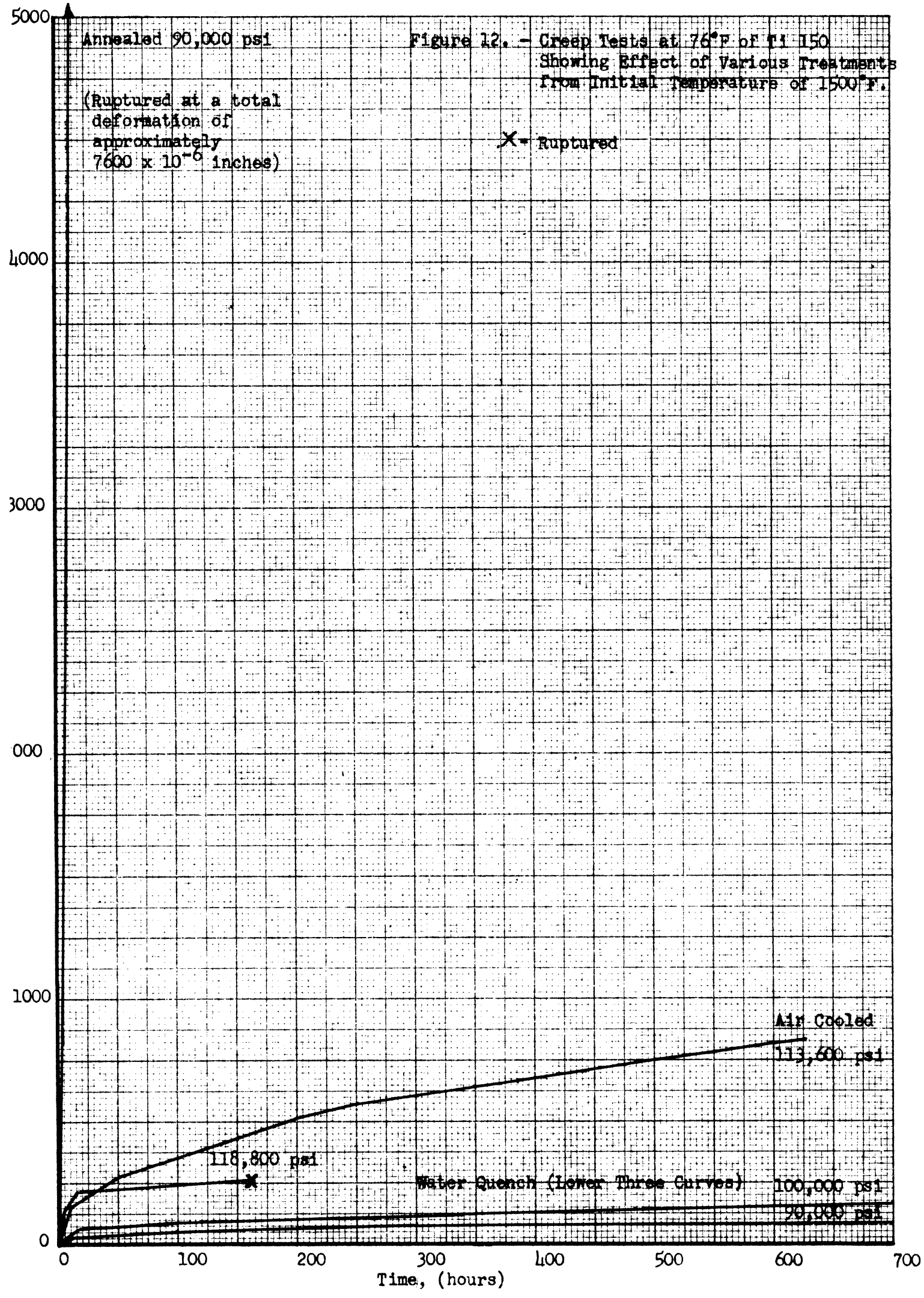



Figure 12. - Creep Tests at 76°F of Ti 150 Showing Effect of Various Treatments from Initial Temperature of 1500°F.

Effect of Heat Treatment from Holding at 1500°F for One Hour on Microstructure of Ti 150.




Figure 13. - Ti 150 Water Quenched from 1500°F




Figure 14. - Ti 150 Air Cooled from 1500°F




Figure 15. - Ti 150 Annealed from 1500°F

Etchant: 1 part HF, 1 part $HNO_3$ , 20 parts $H_2O$.