SECOND PROGRESS REPORT

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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

Results of X-ray diffraction line sharpening (internal stress relaxation) studies on the carbon bearing "commercially pure" Titanium showed that temperatures in excess of 500° F at times in excess of 100 hours were necessary to initiate recovery in this particular stock. Two creep tests of 1000 hours duration were completed on the same material in the annealed condition; at a stress of 55,200 psi the rate was 6.0×10^{-5} percent per hour, at a stress of 50,200 psi the rate was 1.45×10^{-5} percent per hour.

With the arrival on March 1, 1951 of the test stocks of Ti 75A (commercially pure) and Ti 150A (iron and chromium alloy) the tenor of the investigation changed from that of being general preliminary work to one of active preparation for the systematic study of the creep of these materials as related to processing variables.

The first work undertaken with the Ti 150A stock was the carrying out of a heat treating program to form the microstructures to be dealt with in subsequent work. In the case of the Ti 75A material it was necessary to carry out various anneals to provide a base for further cold work and recovery treatments. Following these treatments recourse was made to metallographic examination, hardness determinations and X-ray traverses to provide further information. In the case of Ti 150A this involved surveying the range of microstructures obtained upon heat treating. For Ti 75A, this involved principally the establishment of suitable "strain free" conditions for each material on which to base subsequent X-ray line broadening measurements to measure of internal strain.

Detailed discussion of this work follows in subsequent sections, so the tentative program arrived at is summarized here without explanation of the basic

processing treatments decided upon. The Ti 75A, annealed at 1600° F, will be given reductions of 7, 13 and 40% prior to recovery treatments. The Ti 150A will be water quenched from 1500°, 1650° and 1750° F prior to tempering or aging treatments. The recovery and tempering treatments will be similar in both cases and consist of low temperature anneals for various times in a systematic manner.

As far as the actual creep testing work goes, the following has been done on the new stock. The Ti 75A has been given its preliminary annealing and reduction treatments. Creep tests are now underway on the as-annealed material. The Ti 150A is now being machined into test specimens prior to any heat treating.

INTRODUCTION

Two of the three sheet materials, commercially pure titanium (Ti 75A) and an iron-chromium alloy (Ti 150A) to be used for test purposes, were received on March 1, 1951. The third material, the manganese alloy (Ti 130A), has not yet been received. Up until this time the investigation was confined to extension of the preparation of equipment and development of techniques as outlined in the first progress report on January 1, 1951. Since March 1st the work necessary for the proper preparation of specimens and interpretation of data for the objectives of the investigation on the two available test materials, has been in progress.

The objective of the investigation is to obtain some explanation of the factors controlling the creep of commercially pure titanium and two commercially available alloys in the atmospheric temperature range (-100° to 600° F). Major effort is to be directed to the creep at room temperature. As outlined in the first progress report the investigation will determine the effects of such metallurgical variables as cold rolling and heat treatments on the creep characteristics and explain the results in terms of internal strain, initial microstructures and structural changes during testing. The fundamental explanations are the major objective rather than the accumulation of engineering design data.

TEST MATERIALS

The majority of experimental results obtained to date have been on a "commercially pure" lot of Titanium donated for preliminary studies which were carried out prior to the receipt of the desired test materials. This stock has been analyzed and found to contain 0.34 percent carbon.

Shipments of the desired materials were received from the Allegheny Ludlum Steel Company about March 1, 1951. These were the Ti 75A (commercially pure) and the Ti 150A (iron-chromium alloy). The Ti 130A Manganese alloy on order from the Rem-Cru Corporation has not been received.

Details reported by the manufacturers for the composition, both actual and nominal, physical properties and manufacturing procedure for the materials received are:

Chemical Composition (percent)

Material		C	Si	W	N_2	Fe	Cr	O_2	Ti
(Ti 75A)	Nominal	-	-	-	.02	.10	-	trace	99.88
(Heat X-934)	Actual	.037	.023	.50	.063	.08	nil	not reported	99.2
(Ti 150A)	Nominal	.02	-	-	.02	1.3	2.7	.25	95.7
(Heat X-672)	Actual	.051	.092	.27	.049	1.42	2.72	not reported	95.3

Physical Properties

Material	T.S. psi	Y.S.(2% Offset)psi	Elong. 2"	Hardness	Bend Test
Ti 75A	96,200	76,000	18%	98.5 R-B	180° dia 3XT.
Ti 150A	140,000	124,500	15%	34 R-C	110° dia 3XT.(GW)

Manufacturing Details

The billet of Ti 75A was heated to 1300° F, hot rolled to strip thickness of 3/16-inch, and then annealed at 1300° F. This was followed by a caustic Virgo treatment and a hot HNO_3-HF pickle. Then the strip was cold rolled to the finished gauge (.060") in 3 stages, each stage being followed with an anneal and cleaning as above. No special atmosphere was used in gas fired open annealing furnaces.

The sheet bar of Ti 150A was heated to 1300° F before rolling to a 1/16" thick sheet. The stock was then solution treated at 1275° F, water quenched, and then held at 1200° F for 2 hours. The usual cleaning procedure described above was used, followed by scrubbing and drying. Again no special atmosphere was used in low temperature muffle annealing furnaces.

The actual condition of the materials as received was fair. The Ti 75A had a good surface; however, cracks, tears and inclusions will render about 10 to 15 percent of the Ti 150A stock useless for the preparation of creep specimens.

EXPERIMENTAL PROCEDURES

The experimental procedures used, as well as the underlying philosophy of the test program were covered in the First Progress Report dated January 1, 1951.

RESULTS AND DISCUSSION

Hardness Measurements

Figure 1 shows the hardness of the Ti 75A material as a function of annealing. As a rough guide it can be assumed that hardness and degree of internal strain are proportional. Since it is desired to start all creep testing or further processing, with the Ti 75A material as strain-free as possible (See First Progress Report dated 1 January 1951) it was decided that a 1600° F anneal was a necessary preliminary treatment for the Ti 75A stock. This finding was substantiated by X-ray line width observations, which are in this case a direct measure of internal strain.

X-ray Studies

The majority of the X-ray investigations carried on during the period covered by this report were on the old "commercially pure" stock. Before an intelligent study could be made, an X-ray line had to be chosen which fulfilled the following three conditions: a. It must be of sufficient intensity to insure minimum error in energy "pick-up" by the Gieger tube and in recording sensitivity. The highly preferred structure must therefore be considered. b. It must be as isolated as possible from surrounding X-ray lines. As a material is cold worked, X-ray lines broaden. If two lines are close enough together their "tails" will blend in together, falsely raising the background and hence obviously introducing error. c. It must be at the highest possible value of θ , the Bragg angle, for the greatest possible accuracy. Differentiation of the Bragg Equation shows that for a given value of Δ d, the change in interplanar spacing, the largest values of $\Delta\theta$ occur at values of θ approaching 90°. On the basis of the above conditions, the diffraction line (114) which occurs at $2\theta \cong 114.6°$ was chosen.

Since one purpose of this project is to relate the internal state of the material to its properties, namely creep resistance, it was necessary to determine the representative internal states to be studied. For this end, a study of the effect of various percents of cold working on the X-ray line widths was made on the old stock. The result obtained, (See Figure 2), was that typical of most metals. Armed with these data, a rolling schedule for the Ti 75A was drawn up; it consisted of cold working the stock to states of equally spaced internal strain as measured by X-ray line widths. Values of 5%, 11% and 40% were set, the upper value representing the approximate state of maximum cold working, (further cold working might result in "recovery" of some internal strain) the lower limit representing the approximate minimum of uniform working throughout the section. Since the accuracy obtainable in rolling thin sheet is not good, the obtained reduction values of 7%, 13% and 41% which still give equally spaced strain states, were considered quite satisfactory.

An X-ray study was completed on the recoverability from an internally strained state of the "commercially pure" stock cold worked 40 percent. This consisted of

X-ray line width studies after recovery had been allowed to take place for times of 1, 10 and 100 hours at temperatures of 500° F, 700° F, 800° F and 900° F. (See Figure 3). This study showed that recovery did not take place, on this particular stock, until a temperature somewhat in excess of 500° F was reached, for the times at recovery temperature investigated. A preliminary study of a similar nature has been started on the new Ti 75A stock. This study tends to indicate a somewhat lower temperature for the start of recovery for a given time interval. It seems reasonable to attribute this to the carbon content of the commercially pure stock. Thus, guided by these sketchy results from the Ti 75A and the information obtained from the old stock, the tentative creep testing temperatures for these as rolled material were chosen. These are: 20° F, 75° F, 200° F, 400° F, 600° F and possibly 800° F. The first two temperature choices are based on the temperature range of most interest. The temperature of 200° F was chosen as an elevated testing temperature at which recovery should not start; 400° F as the temperature at which recovery starts on the 40% cold worked samples but not on the 7% cold worked samples; 600° F as a temperature well into the recovery region; 800° F as a temperature at which recovery is so fast it should have no effect on the latter stage of creep. It is expected that increased creep resistance from cold work will be maintained at temperatures up to those where recovery occurs. As an aid to interpretation of creep test results, aging of the specimens before testing may be introduced as another variable.

Again it should be stated that these temperatures are only tentative. Further studies on the new Ti 75A may necessitate some revision. However, the final selection of temperatures will be such as to give the representative testing conditions described above.

X-ray diffraction studies have been made on Ti 150A treated as follows: Water quenched from 1400°, 1500°, 1550°, 1600°, 1700°, 1700°, 1800° F; and furnace cooled from 1400°, 1500°, 1550°, 1600°, 1700°, 1750° and 1800° F. The work has been performed with two objects in mind. First the identification of phases as represented by the lines present and, secondly, the selection of a suitable line for line broadness studies in connection with strain recovery treatments. Such broadness studies will be found discussed on pages 13 and 14 of the First Progress Report.

Comparison of the actual line positions with calculated positions reveal the existence of mixed alpha-beta structures as a temperature dependent phenomena. Metallographic examination of the sheet just below the surface led to the postulation of a complete beta retention following quenching from high temperatures. However, X-ray traverses show the persistence of alpha lines even under these conditions. This indicates incompleteness of any suppression of alpha. This may be due to the inhomogeneous response of the sheet to quenching. (See Metallographic Studies). A further complicating factor is the presence of lines unaccountable either to alpha or beta. This suggests the presence of yet another phase. Unfortunately microstructural examination has not as yet revealed any visual evidence of this third phase.

Again in the study of line broadnesses of Ti 150A it is necessary to fulfill the conditions stated above. Slow traverses have been made in the region from 108° to 132° = 2Θ and two alpha lines have been selected for further study. One is the 114 line, at 114.6° = 2Θ , and the other 300 line, at 129° = 2Θ . Although the 300 line appears to be quite promising it was not found present with sufficient intensity with lower quenching temperatures. Consequently it appears that the 114 line will be used for strain recovery studies.

A further point in connection with this particular phase of the work has recently come to light and is to be the subject of investigation in the immediate future. That is the establishment of a suitable strain-free condition to which all broadening studies of Ti 150A can be referred. Slow traverses of the 114 and 300 lines even after furnace cooling from a high annealing temperature have revealed the presence of a certain amount of residual strain. It is felt that this strain may be a result of the alpha-beta transformation (possibly a shear mechanism) and that even the slow rate of 5° per minute attained by furnace cooling is too fast to allow recovery from any transformation strains. Consequently long time (on the order of 24 hours) anneals at temperatures below the transformation temperature are being studied as a means of attaining the strain-free condition. Qualitatively it may be stated that quenching of the alloy introduces strains of the same order of magnitude as is found in the pure material cold-worked up to 40%.

Metallographic Studies

Micrographic examination (see Figures 4 to 13) perpendicular to the surface of the heat treated Ti 150A stock revealed the existence of three "characteristic" structures in range from 1400° to 1800° F, especially after quenching. At the lower temperatures the material consisted of alpha plus beta titanium, and as the temperature was increased, greater and greater amounts of beta were obtained. The transformation from alpha plus beta to the beta region appeared to take place between 1550° and 1600° F. This was evidenced by the fact that examination of the 1500° F annealed material showed the beginning of the Widdmanstatten or "basketweave" type of structure and also by the very marked difference between the quenched samples from 1550° and 1600° F. The 1600° F quench showing formation of characteristic acicular particles while the 1550° F samples merely show the globular, enlarged alpha particles. Further increase in annealing temperature above 1600° F shows little change in the structure save that of the increasing fineness of the lamellae with temperature.

Quenching, however, from higher temperatures had a much more striking result. Beginning at 1700° F, and becoming more noticeable as the temperature was raised, what appeared to be a suppression of the beta transformation occurred.

At 1800° F the microstructure was almost completely beta. The grains were quite large and were outlined very distinctly on etching. Examination of samples in an edgewise direction, across the thickness of the specimen showed a cruous "banding" which consisted of a layer of somewhat different appearance sandwiched between the surface layers. The main thing observable about this layer was the suggestion of an acicular structure.

Concern manifested over the nature of the "banding" led to the performance of an experiment in which two "half thickness" samples were quenched from 1785°F. The specimens, however, were reduced in thickness in different ways. One one the reduction was performed one face, while on the other the specimen was reduced in thickness by removing a quarter of the thickness from each side. Thus the original center was in one case still in the center, while in the other it was displaced toward one edge. Microstructural examination revealed that the banded structure was displaced toward one side in the sample where the original center had been similarly displaced. The banded structure remained in the center in the sample where the original center was not displaced. Thus the "banding" seems to be associated with the original center (i. e. composition variation) of the material rather than a quenching effect. What the effect of this banding will be on mechanical (creep) properties is not known at the present time.

Choice of temperatures for processing prior to creep testing or further processing now becomes more readily apparent. The lowest temperature approximates the effects of quenching from the mixed alpha-beta region, the intermediate temperature the effects of so-called "complete" quenching to obtain acicular particles, while the highest temperatures should show the effects of suppressing the transformation.

Second heat treatments planned are designed to account for two possible effects. One is stress relief and grain growth while the other is the precipitation from the retained beta areas. By varying systematically the time and temperature of reheat treatments, these effects should be accounted for, if present.

Because of the implications involved in such phenomenon as the beta-suppression, it has been decided to begin the investigation of creep properties on material treated from this range. The material is expected to have a greater amount of strain trapped within the lattice and consequently is also expected to respond most readily to re-heat treatments according to the time-temperature schedule mentioned previously.

Creep Testing

During this period creep testing has been confined mainly to testing at 75°F the "commercially pure" material in the annealed state. Several tests on the Ti 75A in the annealed state have just been started at 75° F but these data will not be complete before the next progress report.

Difficulty was encountered in testing the "commercially pure" material due to the propagation of fine cracks within the specimens resulting in fracture. These crack formations were found in the material as received and were due, in some manner, to the processing of that particular sheet stock. The new 75A sheet shows no such cracks; no difficulty with fracturing has been met and none is expected.

Two acceptable creep tests on the "commercially pure" material were allowed to run for 1000 hours with the results given in Figure 14.

On the creep test at 55,200 psi, an optical system was attached to the specimen in order to check the reliability of the electric strain gages under the test conditions. The results proved the validity of the strain gage measuring method, at least up to strain rates of 10^{-6} per hour (see Figure 15). Its validity will be further investigated at the higher strain rates.

CONCLUSIONS

The work completed to date permits the outlining of the future testing program.

Program for Testing Ti 75A

On the basis of the minimum hardness of the various annealed conditions of Ti 75A, an initial annealing temperature of 1600° F will be used.

On the basis of X-ray diffraction line width studies at various percentages of cold work of the first lot of "commercially pure" Titanium (see Figure 2). Three states of internal strain were chosen at approximately equal intervals for testing. These three states of strain correspond to 7, 13 and 40 percent cold working; material given the above mentioned anneal has been cold rolled at these percentages and is ready for testing.

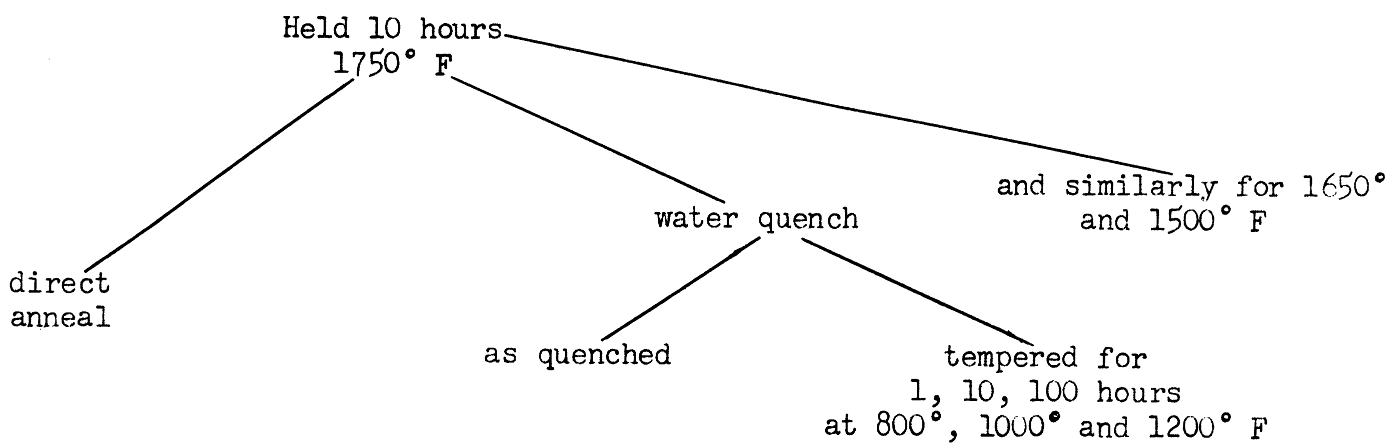
Guided by the recovery data collected on this first lot of "commercially pure" Titanium and the preliminary results on the new Ti 75A stock, the tentative creep testing temperatures have been chosen as -20° F, 75° F, 200° F, 400° F, 600° F and possibly 800° F. This choice of temperatures will allow evaluation of creep characteristics from sub-zero temperatures to temperatures at which the creep resistance of Ti 75A will be very low. It will allow investigation at temperatures where recovery does not take place, at a temperature where it is just starting, at a temperature at which a large amount of recovery takes place during the test and finally a temperature at which all recovery should have been completed by the time first stage creep has stopped. The results should define the effect of cold work on the creep of Ti 75A material and indicate if other metallurgical variables are affecting its creep.

It must be realized that the above temperatures of creep testing are only tentative. Complete comparative studies of X-ray diffraction line widths for recoveries of various percent reduction and various times at recovery temperatures on the new Ti 75A stock may alter the testing temperature slightly. The final selection of these temperatures though, will be such as to give the representative testing conditions described above.

Program for Testing Ti 150A

On the basis of the hardness data and metallographic examination of Ti 150A, after a program of preliminary heat treating, a tentative schedule of systematic heat treating of Ti 150A has been adopted. The program will be based upon the following heat treating or solution treating temperatures: 1500° F, 1650° F and 1750° F. Using these temperatures as a starting point, tests will be made on material directly annealed, water quenched and reheated from water quenching for periods of 1, 10 and 100 hours at 800°, 1000° and 1200° F.

Each specimen will be so designed as to permit three stress levels for room temperature creep testing. Conditions showing little or no creep will then be used as a basis for elevated temperature testing. Most creep tests will be made on specimens so designed as to be in the longitudinal direction of rolling. However, several transverse specimens will be tested under the same conditions to given an indication of what effects the known anisotropy of the material has on the creep resistance. The exact testing program may be better understood through reference to the following schematic diagram. Because of microstructural effects discussed previously, it has been decided to begin the testing program with the highest solution treating temperature of 1750° F.



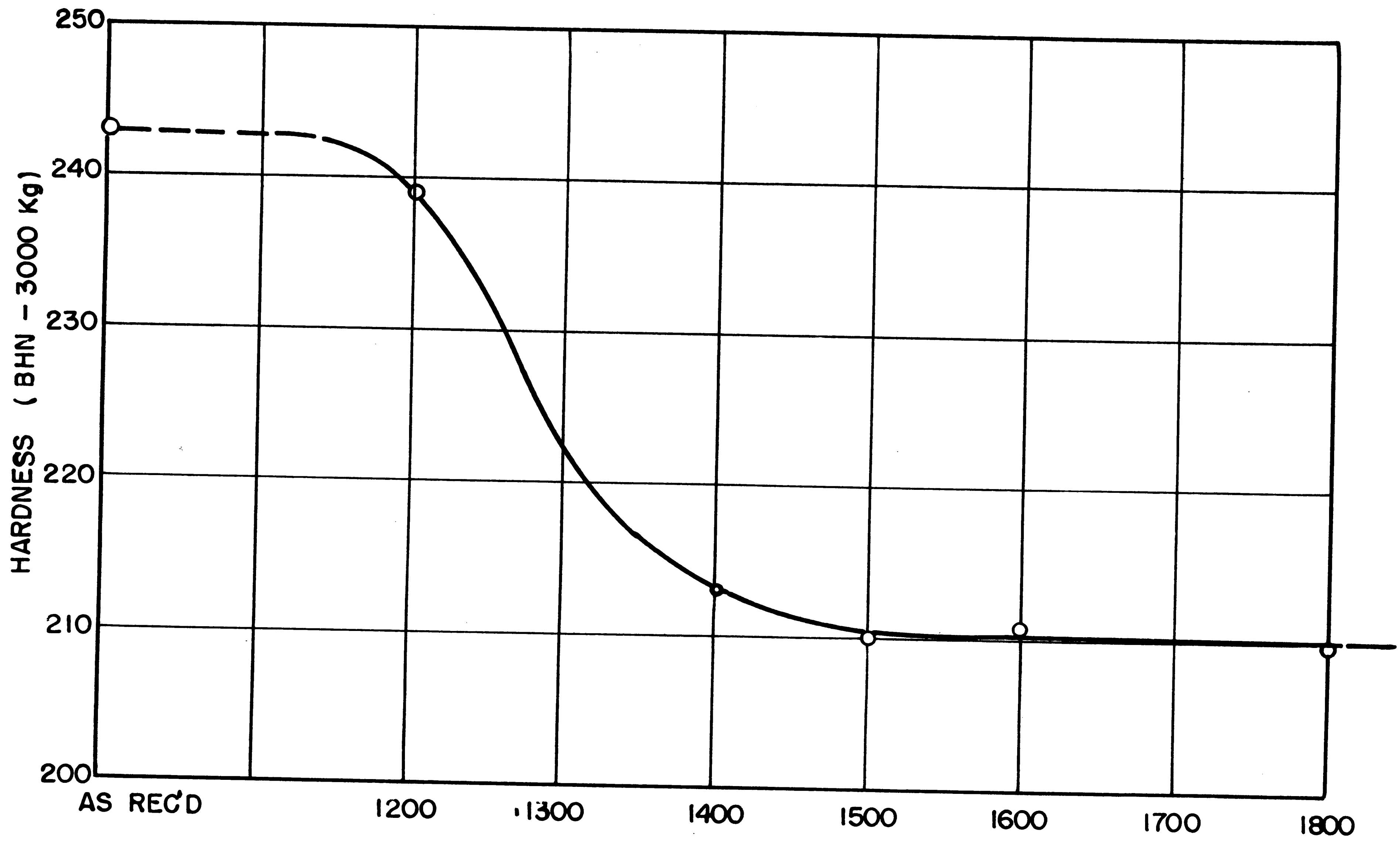


FIG. I HARDNESS OF TI-75A ANNEALED I HOUR AT VARIOUS TEMPERATURES AND SLOWLY COOLED (HARDNESS TAKEN AS 10 Kg VICKERS)

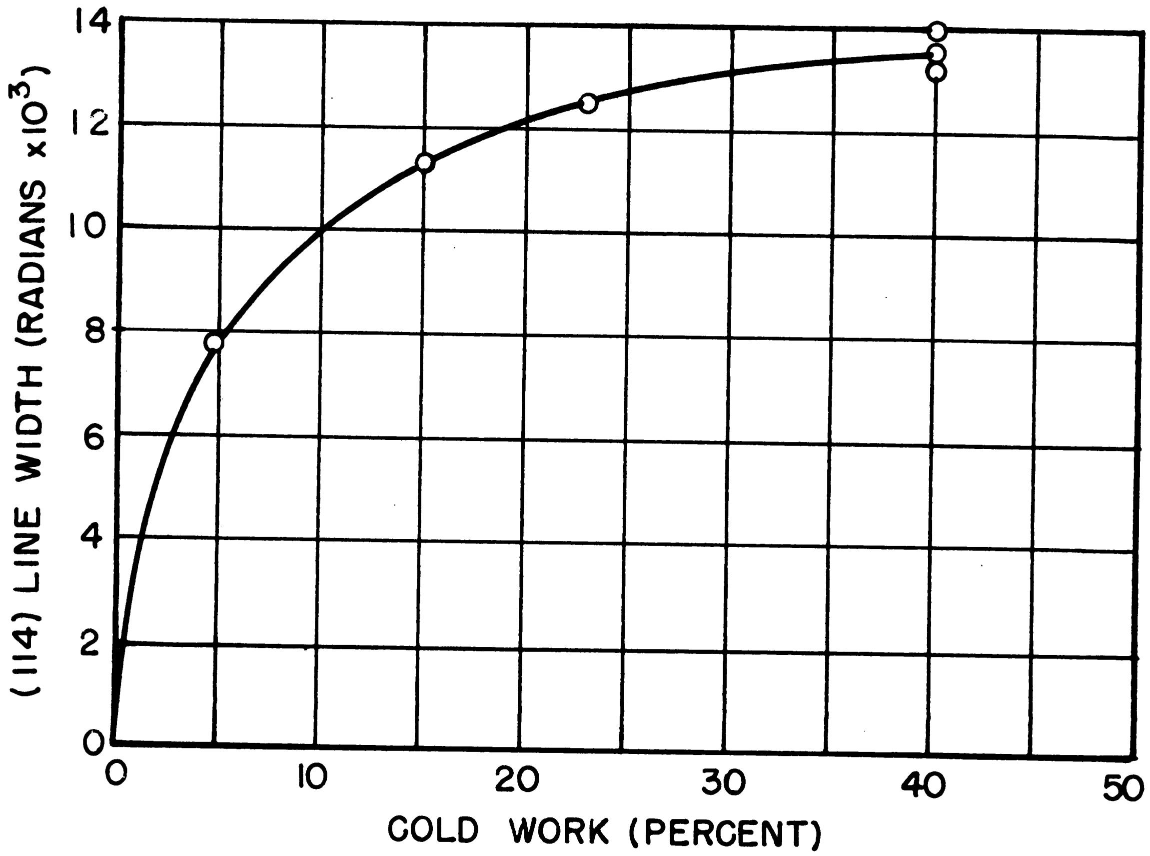


FIG. 2 EFFECT OF COLD WORKING ON THE (114) DIFFRACTED LINEWIDTHS OF THE "COMMERCIALLY PURE" TITANIUM (0.34C), ANNEALED AT 1600°

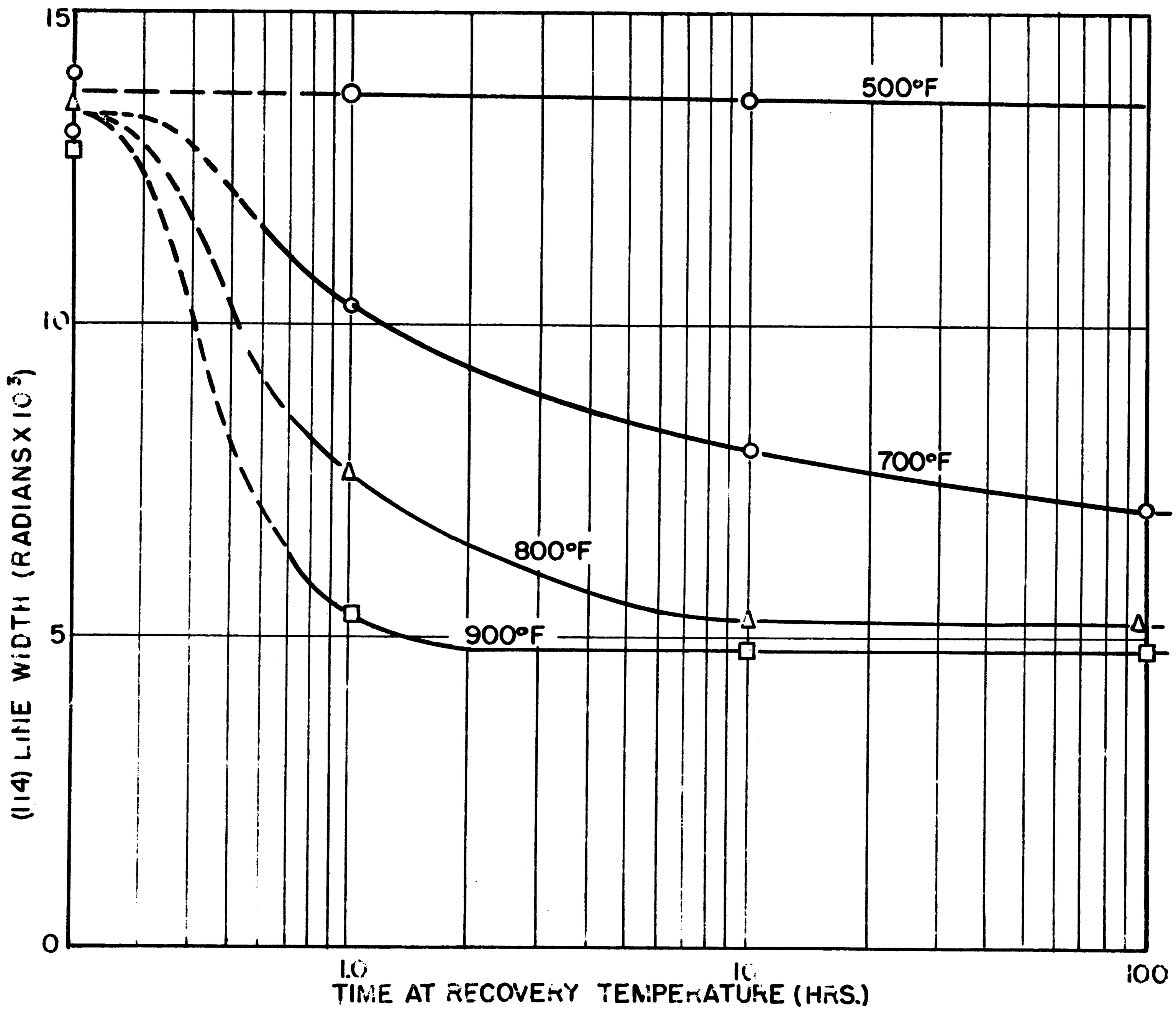
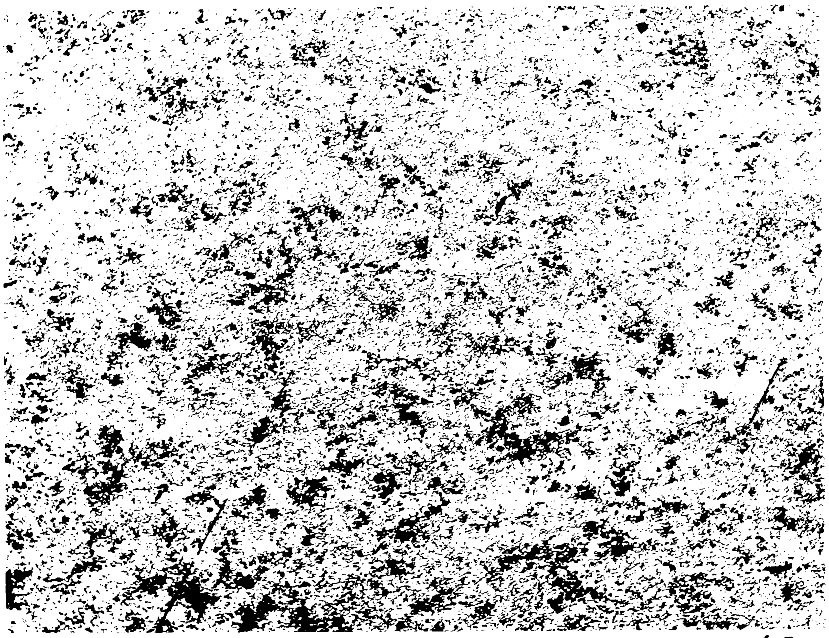


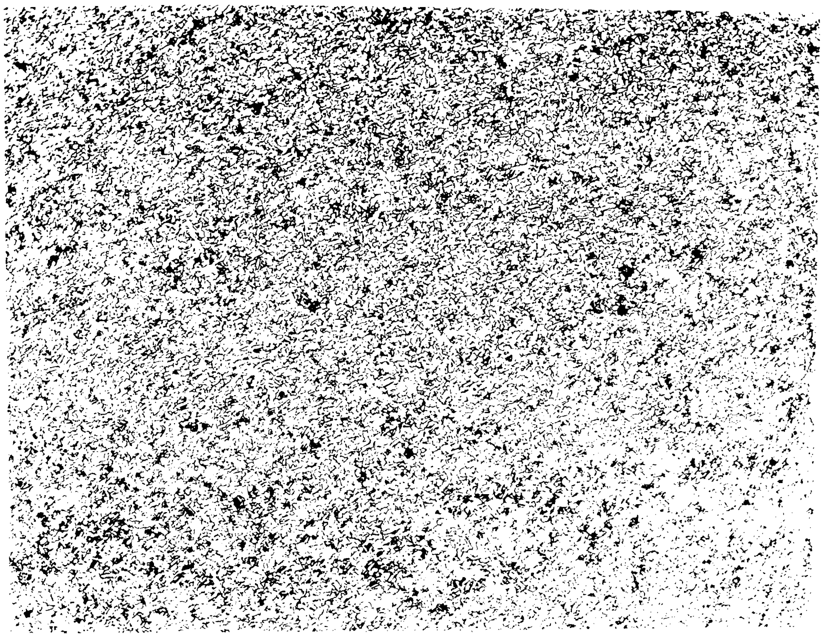
FIG. 3. EFFECT OF TIME AT VARIOUS RECOVERY TEMPERATURES ON THE WIDTH OF THE (114) LINE OF THE "COMMERCIALLY PURE" STOCK (0.34C) ROLLED, ANNEALED AT 1600°F AND REDUCED 40 PER CENT.



51-769

X100

Figure 4. - Ti 150 A furnace cooled from 1400° F.



51-770

X100

Figure 5. - Ti 150A furnace cooled from 1500° F

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

51-771

X100

Figure 6. - Ti 150A furnace cooled from 1600° F

51-772

X100

Figure 7. - Ti 150A furnace cooled from 1700° F

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

51-773

X100

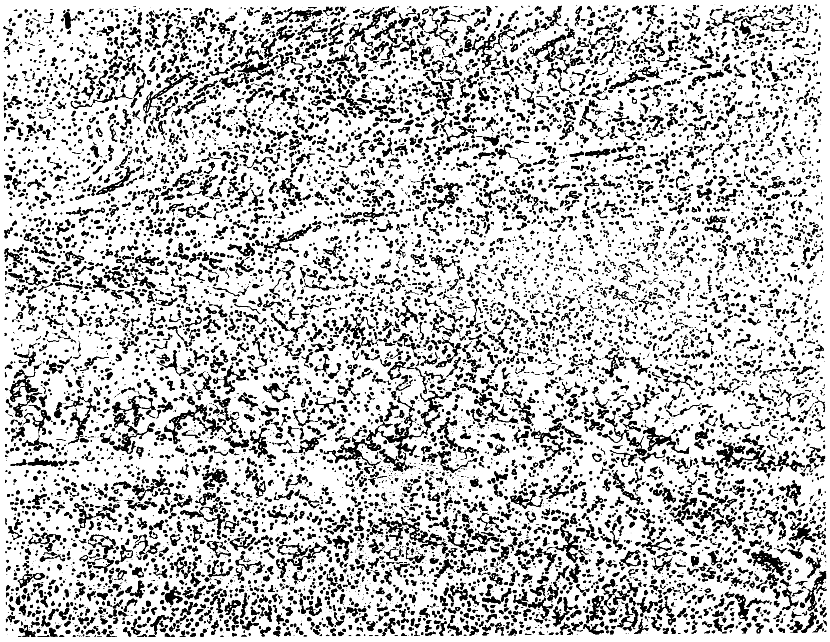
Figure 8. - Ti 150A furnace cooled from 1800° F

51-774

X100

Figure 9. - Ti 150A water quenched from 1400° F

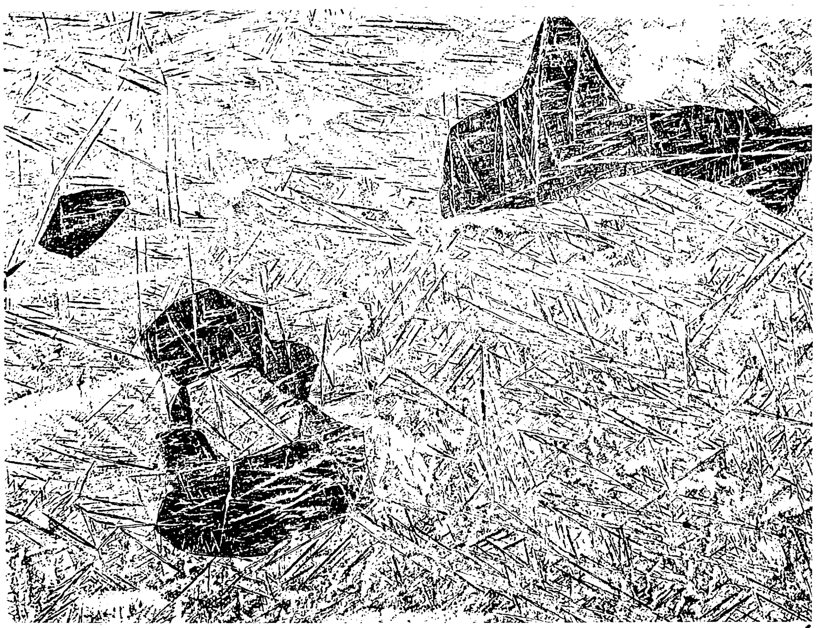
Etchant: 1 part HF, 1 part HNO_3, 20 parts H_2O.



51-775

X100

Figure 10. - Ti 150A water quenched from 1500° F

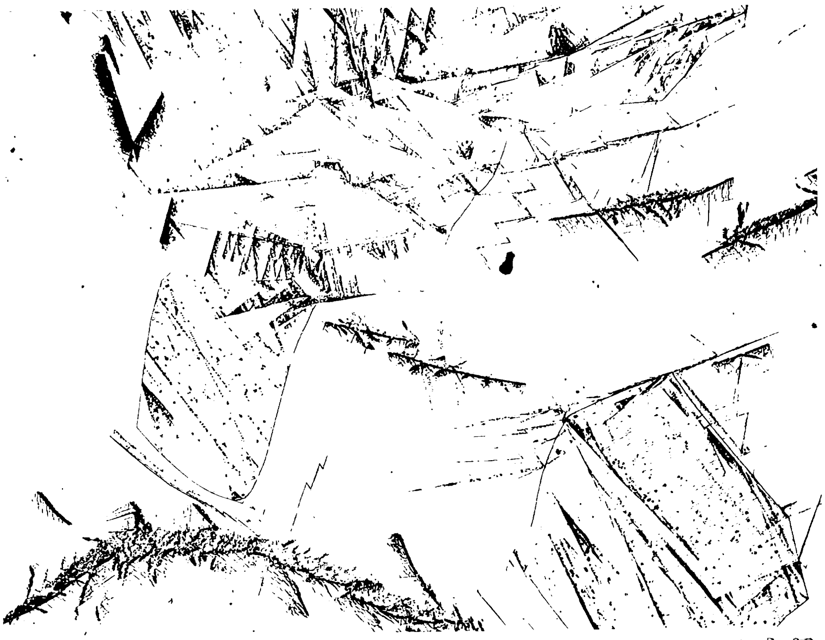


X100

51-776

Figure 11. - Ti 150A water quenched from 1600° F

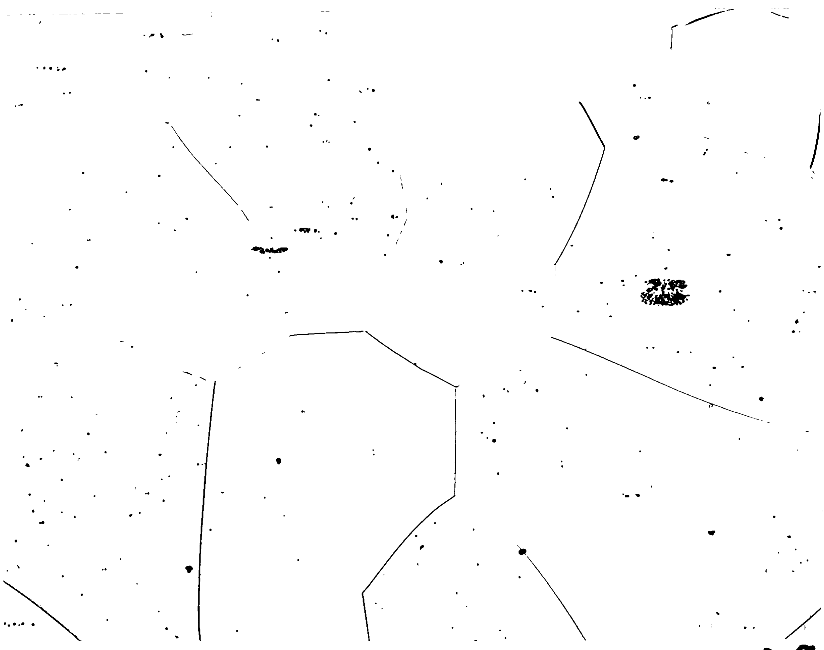
Etchant: 1 part HF, 1 part HNO_3, 20 parts H_2O.



51-777

X100

Figure 12. - Ti 150A water quenched from 1700° F



51-778

X100

Figure 13. - Ti 150A water quenched from 1800° F

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

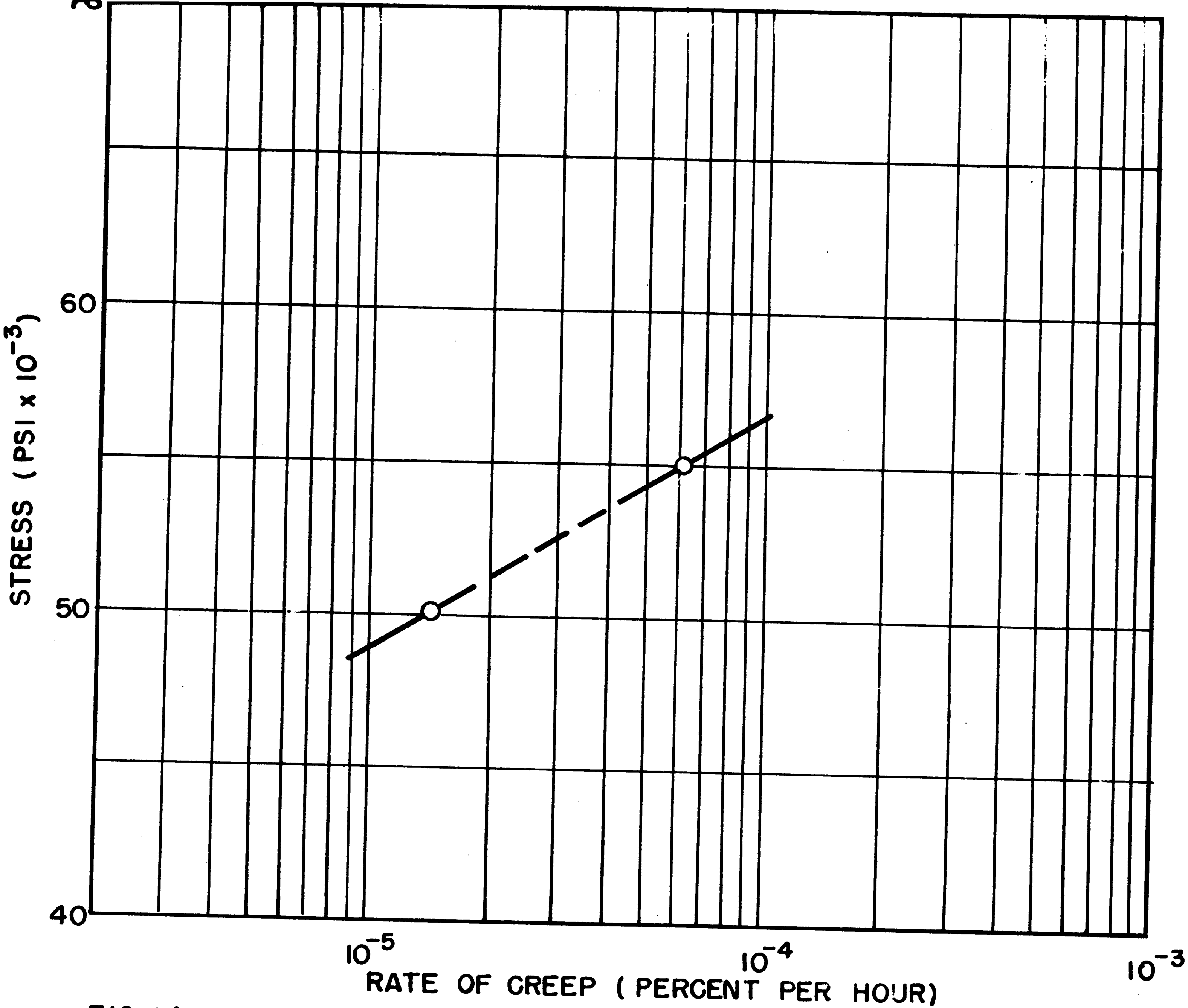


FIG. 14 CREEP RATES AT 75°F FOR THE "COMMERCIALLY PURE" TITANIUM (0.34C) ANNEALED AT 1600° F

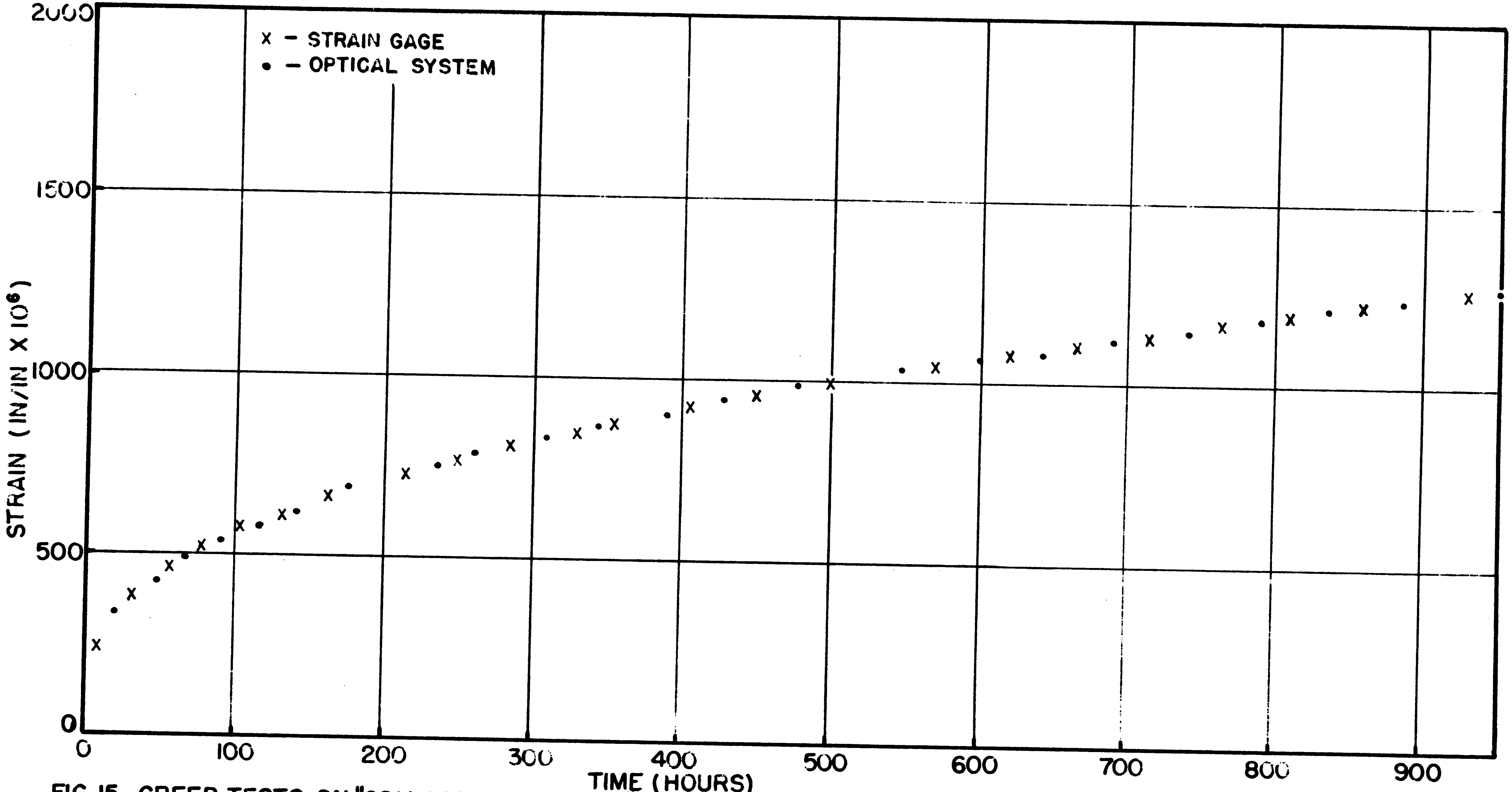


FIG. 15. CREEP TESTS ON "COMMERCIALLY PURE" TITANIUM (0.34C) AT 75°F AND 55,200 PSI. OPTICAL SYSTEM RUN IN CONJUNCTION WITH STRAIN GAGE TO PROVE VALIDITY OF LATTER. (FOR SAKE OF CLARITY ONLY ALTERNATE POINTS ARE GIVEN)