WADC TECHNICAL REPORT

INTERMEDIATE TEMPERATURE CREEP AND RUPTURE BEHAVIOR OF TITANIUM AND TITANIUM BASE ALLOYS

PART III:

EFFECTS OF HOT ROLLING, EMBRITTLEMENT, AND INTERSTITIAL ELEMENTS

Jeremy V. Gluck
James W. Freeman

September 1957

Materials Laboratory
Contract No. AF 33(616)-244
Supplement No. S9(55-1113)
RDO No. 615-11
RDO No. 615-13

Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

FOREWARD

This report was prepared by the Engineering Research Institute of the University of Michigan, Ann Arbor, under USAF Contract No. AF 33(616)-244, Supplement No. S9(55-1113). The contract was initiated under Research and Development Order No. 615-11,"Titanium Metals and Alloys", and Research and Development Order No. 615-13, "High Temperature Alloys" and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Lt. H. M. Burte acting as project engineer. The research was identified by the Engineering Research Institute as Project No. 2076. The period covered by this report is from March 1, 1955, to December 31, 1955. Work initiated under a previous supplement, Supplement No. S4(54-288), and brought to completion during this period is also reported.

ABSTRACT

Three major factors involved in the properties of titanium alloys at high temperatures were investigated:

1. A limited study was made of the influence of hot-working conditions on creep-rupture properties at 600°F to 1000°F.

2. The changes in room temperature tensile test ductility as a measure of embrittlement during creep testing at 75°F to 1000°F were evaluated for a considerable number of specimens of several alloys.

3. A limited study was carried out on the influence of hydrogen content and the hardness of the sponge (interstitial alloying elements) used in making alloys. Creep-rupture properties and embrittlement during stressed exposure were evaluated for temperatures of 600°F to 1000°F.

PUBLICATION REVIEW

This report has been reviewed and is approved

FOR THE COMMANDER:

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

TABLE OF CONTENTS (Continued)

LIST OF TABLES

LIST OF TABLES (Continued)

LIST OF ILLUSTRATIONS

Figure

Figure

Figure

INTRODUCTION

Information is presented covering several aspects of the inter-relationships of type of microstructure and chemical composition of titanium alloys to creep-rupture properties at 600°F to 1000°F and to their embrittlement under creep testing conditions. The investigations carried out extend and supplement the results of two previous studies concerned with the relationship of microstructures to creep-rupture properties (Refs. 1 and 2).

The influence of hot-working conditions on the creep-rupture properties of a stable alpha alloy 6 Al and an alpha-beta alloy Ti 150A was surveyed. Instability effects on creep-rupture properties were evaluated for a stable alpha alloy 6 Al - 0.5 Si . These two investigations extend the information of References 1 and 2 on relationships between type of microstructure to creep-rupture properties.

Creep specimens available from the tests carried out for References 1 and 2 were subjected to tensile tests at room temperature to determine embrittlement characteristics of the various types of alloys when exposed to creep at 600°F to 1000°F. This was supplemented by tests on specimens in the files of the University from the investigation of creep of titanium and titanium alloys at 75°F to 600°F (Ref. 3). The results of preliminary surveys of the influence of vacuum annealing on embrittlement of the stable alpha alloy 6 Al and the alpha-beta alloy Ti 150A are also included in this section. Data were also obtained on the embrittlement time for the Ti 150A alloy.

The last section of the report deals with a planned comprehensive investigation of the role of hydrogen and interstitial elements on the creep-rupture and embrittlement characteristics at 600°F to 1000°F for typical alloys. Hydrogen was expected to be a major factor in embrittlement. Its effect on creep-rupture properties was to be determined. The part played by interstitial elements in relation to hydrogen content was to be evaluated. The relationship to type of microstructure was to be evaluated by studying the effects for stable alpha 6 Al , alpha-beta Ti 150A and 8 Mn , meta-stable beta 10 Mo and stable beta 30 Mo alloys. The major effort was to be placed on alpha-beta alloy 8 Mn.

EXPERIMENTAL TECHNIQUES

Tensile and creep-rupture testing procedures, metallographic examination and x-ray diffraction techniques are described in this section. Certain specialized techniques are discussed in the sections where they are applicable.

Tensile and Creep-Rupture Testing

All tensile and creep-rupture tests were carried out in air in accordance with ASTM Recommended Practices. Standard 0.250-inch diameter specimens were used for all tests. The specimens were 2-3/4 inches long with a reduced gage section of 1 inch. The ends of the specimen were threaded with 3/8 - 16 threads.

Creep-rupture tests were carried out in individual lever-type University of Michigan creep testing machines. The stress was applied to the specimen through a third-class lever system ratio of 10 to 1.

Tensile tests were performed in a Baldwin-Southwark hydraulic tensile machine. A head speed of 0.050 inches per inch per minute was used for all tests.

Elevated temperature tests were carried out in individual wire-wound resistance furnaces fitting over the entire specimen assembly. Three chromel-alumel thermocouples were attached to the specimen gage section and a temperature distribution of ± 3°F was required before initiation of the test. All specimens were placed in hot furnaces at approximately 50°F below the test temperature and could usually be brought up to proper temperature and distribution in about one hour.

Strain measurement was accomplished with a modified Martens optical extensometer system having a sensitivity of about 5×10^{-6} inches per inch. The extensometer bars were attached to pinned collars threaded to the specimen shoulder. Consequently, a correction factor was added to the reduced section in order to account for the fillets included in the effective gage length.

Readings were taken at least once a day on all long-time tests and only after the temperature had been checked to ± 3°F of the nominal test temperature. During tensile tests, readings were taken at applied load intervals of approximately 4,000 psi.

Metallographic and X-ray Techniques

Sections for metallographic or X-ray examination were cut using Krystolon or Allison glass-cutting wheels with continuously recirculating coolant. Following a rough grind on a wet-belt sander, the specimens were taken through 240-, 400-, and 600-mesh wet silicon carbide paper mounted on rotating caps. Intermediate polishing was accomplished with 4 - 8 micron diamond compound on glass-backed photographic print paper. The specimens were finished with Linde-B compound on a rotating wheel. Occasionally, it was necessary to lightly etch and then return to the final polishing wheel several times before a satisfactory surface could be obtained. Electro-polishing was not employed in this investigation in order to minimize the possibility of hydrogen contamination.

The etchants employed were as follows: for alpha alloys a mixture of 2 parts HF, 2 parts HNO_3, and 100 parts H_2O; for alpha-beta and beta alloys, a mixture of 1 part HF and 1 part glycerine.

Standard techniques were employed for photomicrography.

The samples used for X-ray examination were usually metallographic specimens given further etching in the standard etchants mentioned above. The etchants were enriched with HF in order to speed their action.

X-ray diffraction photographs were taken in a forward reflection Debye camera. A glancing angle of 15° or 20° was used and the specimens were rotated throughout the exposure. Copper radiation at 45 kilovolts and 12 milliamperes was used with nickel filters mounted at both the tube part and film plane in order to screen out extraneous radiation. The filter at the film plane served to reduce the background radiations produced by fluorescence of the sample. Depending on the area of the sample, exposures of one to three hours were used.

INFLUENCE OF HOT AND COLD WORK ON PROPERTIES

The properties of alloys at high temperatures can be influenced to varying extents depending on the type of alloy and the efficiency of subsequent heat treatments, when applied, in minimizing the influence of prior working. In theory, controlled hot working conditions can supplement heat treatment as a means of controlling microstructures to obtain desirable properties; or to avoid conditions of working leading to unfavorable properties. In practice, working conditions are usually governed by the least costly procedure to produce the desired geometrical form free from cracks and with at least minimum physical properties at room temperature. The possible influence of hot-working conditions on creep-rupture properties is generally little known and usually receives little attention. Accordingly, a limited investigation was undertaken to explore the possible influence of hot-working conditions on the properties of a stabilized alpha titanium alloy containing 6-percent aluminum and on the alpha-beta alloy Ti 150A.

In previous investigations (Refs. 1 and 2) it appeared that the stable alpha alloy 6 Al was subject to recrystallization during creep testing. The survey tests of Reference 2 on 6 Al - 0.5 Si alloy indicated a similar effect on properties with less indication of recrystallization. A more comprehensive study was carried out to better define the effects for the 6 Al - 0.5 Si alloy.

Hot-Working Conditions and Properties of Stable Alpha Alloy 6 Al

Experiments were carried out in which rolling reductions were made with the alloy heated to temperatures where (1) only beta phase existed and (2) when the structure was all alpha. This procedure was used so that the effects could be evaluated with and without transformation subsequent to working. Evaluations of properties were also carried out for stock heated and cooled in the same manner as the rolled stock. In addition, the properties of the as-rolled material as furnished by the supplier were established for comparative purposes.

The evaluation of properties was based on tensile tests at 75°F and on creep-rupture properties at 1000°F. A temperature of 1000°F was selected so that the properties would be mainly dependent on creep characteristics since References 1 and 2 indicated that creep was a minor factor at 600°F and 800°F, the other temperatures generally used to evaluate structural effects in this research.

Experimental Material

The stock used for the investigation was in the form of as-forged 3/4-inch square bars produced by the Armour Research Foundation from an approximately 10-pound heat (Heat 4b). They reported the chemical composition to be as follows:

Al (%)	C (%)	N (%)
6.28	0.044	0.02

Small ingots were first melted in a non-consumable tungsten electrode furnace using sponge titanium having a Brinell hardness of 124. These small ingots were forged to bars and remelted as consumable electrodes. Forging was carried out from 2000°F.

Hot-Working Conditions

The stock was worked by hot-rolling at two temperatures. One temperature, 2150°F, was selected to study the effects of working when the alloy was in the beta form. The other temperature, 1400°F, was selected to study the effect of working when the structure of the alloy was alpha. The bars were approximately 12 inches long and were heated for 1/2-hour in a gas-fired furnace with a reducing atmosphere.

All reductions were confined to one pass through the mill to keep working conditions nearly iso-thermal. Closed passes were used. The maximum reduction in area obtainable with the mill in one pass was 24 percent at 1400°F and 37 percent at 2150°F. Intermediate reductions of 12 percent at both temperatures were also evaluated.

In addition, stock was heated and air cooled without reduction in the same way as the stock subsequently rolled.

Tensile Properties

The hot rolling had rather little effect on tensile properties at 75°F or 1000°F (Table 1 and Fig. 1a). Simply heating to 1400°F or 2150°F slightly reduced strength at 75°F in comparison to the as-forged condition. Subsequent reduction increased strength slightly with a larger increase at 1400°F than at 2150°F. Heating to 1400°F increased strength at 1000°F and subsequent reduction reduced strength. Heating to 2150°F reduced strength and subsequent reduction raised it. The variation in tensile strength was at most 10,000 psi.

Elongations were not greatly changed by conditions of hot working and were not greatly different between 75°F and 1000°F.

Rupture Properties

The rupture strength for 100 hours at 1000°F ranged between 32,500 and 39,000 psi. (See Table 2 and Figs. 1b and 2.) Rolling at 1400°F resulted in the lowest strength. There was relatively little effect of reduction at either 1400°F or 2150°F. Ductilities were also affected very little.

The 100-hour rupture strengths were established by two tests. The stress-rupture time curves are therefore not well established. The available data do, however, suggest (Fig. 2) that the conditions of rolling would have had a greater effect on long-time strength than on 100-hour strength. Those specimens rolled at 1400°F might have rather low strength. The original as-forged stock and the material reduced 37 percent at 2150°F may have stress-rupture time curves with considerably less slope than the material simply heated to 1400°F or 2150°F or rolled to 13-percent reduction at 2150°F.

Creep Properties

The conditions of hot working appeared to have more effect on creep properties at 1000°F than on tensile or rupture properties. The material exposed to or rolled at 2150°F underwent practically no creep under 15,000 psi (Table 2). The as-forged condition and the material reduced 12 percent at 1400°F underwent considerable creep; and when reduced 24 percent at 1400°F extensive creep occurred. This relationship between treatment and creep resistance is reflected in the stress-creep rate curves (Fig. 3) and persists at high stresses. At the higher stresses the as-forged and material exposed to 1400°F were intermediate in creep resistance.

These results would tend to strengthen the indications of considerable differences in the slopes of the stress-rupture time curves beyond 100 hours as discussed in the preceding section. The higher creep resistance of the material exposed to 2150°F would apparently considerably increase rupture strength at the longer time periods.

The very pronounced difference in creep resistance at 1000°F as affected by temperature of treatment and percent reduction is demonstrated by the time-elongation curves of Figure 4. Apparently, treatment at 2150°F was the major factor in the high creep resistance and not reduction at this temperature. On the other hand, percent reduction was fairly influential for 1400°F.

The greater creep resistance of the materials exposed to or worked at 2150°F is reflected in much longer times to attain a given total deformation. This can be seen by inspection of the total deformation data in Table 2.

Microstructures

Microstructures of the as-forged and as-treated test materials together with microstructures of similar samples after creep-rupture testing are presented in Figure 5.

The as-forged material (Fig. 5a) consisted of a mixture of some variable-size equi-axed alpha grains and some elongated grains. Apparently, the as-cast structure had not been completely broken up during forging. Hot reduction at 1400°F (Figs. 5b, d) did not change the basic structure appreciably although the as-cast structure is more evident in this particular photomicrograph. Hot reduction at 2150°F (Figs. 5f, h) resulted in a structure of plate-like alpha within the configuration of the beta grains in existance at the hot rolling temperature.

The effect of creep-rupture testing at 1000°F on these structures is shown in Figures 5c, e, g, and i. Samples of material hot reduced at 1400°F (Figs. 5c, e) show some evidence of homogenization as a result of test exposure.

The material hot reduced at 2150°F and tested at 1000°F (Figs. 5g, i) shows well-defined structures of lamellar alpha in the vestigal beta grain. Figure 5g indicates almost a "basket-weave" structure, while in Figure 5i, all alpha lamellae within vestigal beta grains appear to be aligned parallel. The structures are considerably better defined than in the corresponding as-treated conditions.

Discussion

Hot working at 2150°F resulted in a substantial increase in creep resistance of the stable alpha alloy 6 Al at 1000°F. The temperature of hot working rather than the degree of reduction, however, appeared to be the more important factor. Increasing amounts of reduction at 1400°F apparently lowered creep strength in relation to the as-forged condition. Cold working at room temperature was found to have a similar effect previously (Refs. 1 and 2).

When subjected to tensile tests at 75°F or 1000°F, there was relatively little effect from hot-rolling conditions. The effect of temperature of hot rolling on creep resistance was not very evident in 100-hour rupture strengths, although there were indications it would be reflected in long-time rupture strengths.

The reason for the lower creep strength at 1000°F after rolling at 1400°F is not certain from the available data. The lack of increase in tensile strength indicates that there was very little cold work imparted by the rolling and, therefore, recovery should not have been a factor. There was little microstructural evidence to indicate a cause.

Hot-Working Conditions and Properties of Alpha-Beta Alloy Ti 150A

Alpha-beta alloys are generally hot-worked in the alpha-beta temperature range to avoid undesirable structures associated with the coarse grain size of beta and the resultant acicular structure formed during cooling. Hot-rolling was carried out on samples at several temperatures from 1200°F to 1650°F with the latter temperature lying near the beta transus. Single pass reductions to provide isothermal working were used.

Properties were evaluated by tensile tests at 75°F and by creep-rupture tests at 800°F. The temperature of 800°F was selected in order to have creep a prominent factor in properties, and to keep the evaluation temperatures consistent with those used for References 1 and 2. The other two evaluation temperatures used in the prior research were 600°F and 1000°F. Creep was not a factor in properties at 600°F; and creep resistance was nearly independent of prior history at 1000°F due to structural instability.

No heat treatments were applied after rolling so as to keep the properties characteristic of the temperature and amount of reduction with subsequent air cooling.

Experimental Material

The stock used was in the form of 0.75-inch square bars purchased from the Titanium Metals Corporation as typical of commercial material. The chemical composition of the heat (Heat L897) was reported by the producer to be:

Fe (%)	Cr (%)	C (%)	N (%)
1.35	2.63	0.070	0.12

Hot-Working Conditions

Approximately 12-inch lengths were heated to 1200°F, 1350°F, 1500°F and 1650°F for 1/2-hour in a gas-fired furnace. The bars were then given one pass through a closed pass rolling mill to obtain reductions which varied from 14.4 to 37.8 percent reduction of area. The larger reductions were the maximum which could be obtained in the mill with one pass. The restriction to single passes resulted in nearly isothermal reduction. The bars were air cooled from the mill.

Microstructures

The microstructures of the as-rolled samples are illustrated by Figure 6. The structures shown are for the largest reduction at each temperature. The structures were not appreciably different from those shown for the smaller reductions.

The reduction at 1200°F deformed both alpha and beta and apparently induced a small amount of fine transformation product to form. Rolling at 1350°F agglomerated the alpha. When rolled at 1500°F the amount of alpha was reduced and a good deal appeared in the form of elongated plates, apparently forming during cooling. When rolled at 1650°F, possibly a small amount of globular alpha remained. The coarse beta transformed to form an acicular product during cooling.

Tensile Properties

The relatively small effect of the conditions of hot rolling on tensile properties at 800°F are shown by Table 3 and Figures 7 and 8. The values for no reduction were estimated using the data from Reference 1 for Heat L-1006.

Tensile strength increased slightly with percent reduction at all temperatures. Those samples rolled at 1350°F and 1500°F had the highest strength. This, however, appeared to be mainly a function of the heating temperature for rolling rather than the rolling itself as can be seen from the comparative data from Reference 1 in Figure 8 for heating Heat L-1006 without reduction.

There was relatively little effect of temperature or amount of reduction on ductility.

Rupture Properties

The hot-rolling conditions had very little effect on the stress for rupture in 100 hours at 800°F. (See Table 4 and Figure 9.) Reductions of 15 to 20 percent may have reduced strength slightly from simple heating alone. Larger reductions slightly increased strength except at 1650°F. The rupture strengths varied from 45,000 to 56,000 psi. Strengths for material heated as for rolling but not reduced were estimated from single tests. The indicated range in rupture strengths for heating without reduction was from 47,000 to 54,000 psi.

Elongations and reduction of area were high in the rupture tests. There was some variation depending on the rolling temperature and degree of reduction.

Creep Characteristics

Both degree and temperature of reduction had a small effect on creep resistance (Table 4 and Figs. 10 and 11). Slight maximums in strength existed for heating to 1500°F and for the larger reductions at each temperature.

Examples of time-elongation curves are shown in Figure 12 for tests conducted under 30,000 psi. All reductions were not tested at 30,000 psi, notably the largest reduction at 1350°F and 1500°F, with the highest creep resistance. Consequently, Figure 12 does not illustrate creep for these conditions of rolling. The figure shows that, in general, primary creep was influenced in about the same way as minimum creep rates by temperature and amount of reduction.

Discussion

The investigation of the influence of conditions of hot working on creep resistance was extremely limited. No broad conclusions should be derived from the results. There are important factors qualifying the results:

1. Working was restricted to the alpha-beta temperature range, a requirement generally considered necessary to obtain good properties in the alloy.

2. Properties were not evaluated at lower temperatures where tensile strength and, in particular, ductility may have been influenced far more than the creep characteristics at 800°F.

3. There is some reason to expect that 800°F may be a temperature where the structural changes during testing tend to reduce differences in properties due to initial variations in microstructure. Additional transformation of beta takes place quite rapidly. It was concluded in Reference 2 that the degree of transformation was the most important factor in measured strength values for alpha-beta alloys.

4. The initial stock used for the experiments had been commercially produced to have optimum properties. This included a considerable amount of hot reduction under conditions shown by experience to be best for the alloy. Consequently, the restriction of conditions of rolling to the alpha-beta range avoided the rather pronounced effects which could come from improper working conditions or the improvements from applying the working conditions to material with initially poor properties.

5. The hot-rolling was limited to isothermal conditions as well as restricted in the degree of reduction. The inclusion of working

over a temperature range, larger reductions, and reheat effects could have been expected to show more influence on properties.

6. Heat treatments were not applied after hot rolling. It intended to try to show the maximum effects by evaluating the material in the hot-worked condition.

7. The reasons for the slight decrease in creep-rupture strength for reductions of the order of 15 percent and slight increases for larger reductions were not ascertained. The increase in strength with increasing rolling temperature up to 1500°F checks the results of References 1 and 2 where increasing amounts of beta were found to increase strength. The decrease at 1650°F was apparently the decrease normally found in the alloys when the structure becomes largely acicular alpha in beta instead of equi-axed alpha grains or relatively coarse alpha plates.

Effect of Cold Work on the Stability and Properties of Stable Alpha Alloy 6 Al - 0.5 Si

The main objective of the study was to better define the relation of stability to properties of stable alpha alloys when cold worked. In References 1 and 2, it was found that alpha titanium Ti 75A and the stable alpha alloy 6 Al when subjected to prior cold work underwent a loss in properties above some temperature due to structural instability. The 6 Al alloy appeared to undergo recrystallization during testing at 1000°F. Survey tests on 6 Al - 0.5 Si also indicated an instability effect but the survey tests did not define its influence too well. Accordingly, a further investigation of the creep-rupture properties of the 6 Al - 0.5 Si alloy was carried out at 1000°F on material cold reduced 20 percent. Comparative data were obtained for as-forged stock.

Experimental Material

The same stock used for the studies reported in Reference 2 was tested. The approximately 10-pound ingot had been produced by Armour Research Foundation using a double melting technique. The 0.5-inch square bars were forged from about 1850°F.

The chemical composition reported by the producer for this heat was as follows:

Heat No.	Sponge Hardness (BHN)	Al (%)	Si (%)	N (%)	H_2 (%)
7011	124	6.51	0.48	0.017	0.0170

Hydrogen was analyzed by the Materials Laboratory, WADC

Cold Reduction

The as-forged bars were reduced in several steps to approximately 20-percent reduction in area between open passes. The bars were rotated between passes so as to obtain equal reduction in both directions. The reduction obtained was 21.6 percent as compared to 19 percent for the material used for the many tests of Reference 2.

Results

The data obtained at 1000°F in the tensile and creep-rupture tests are summarized in Table 5. The immediate effect of the cold work was to increase the tensile and rupture strengths for short time periods. By 100 hours, however, the strengthening imparted by cold work disappeared and the as-forged material had higher rupture strengths. (Compare Figures 13 and 14.) Total deformation and creep strengths (Fig. 15) were also consistently higher for the as-forged condition with increasing time beyond 100 hours. Comparative strength values at 1000°F for the two conditions are summarized as follows:

	As Forged	Cold Reduced 20%
Tensile strength, psi	74,800	92,400
Rupture strengths, psi		
100 hours	45,000	46,000
1000 hours	31,500	27,000
0.5-percent total deformation strengths, psi		
100 hours	25,000	17,000
1000 hours	20,000	12,000
1.0-percent total deformation strengths, psi		
100 hours	30,000	23,000
1000 hours	~23,000	18,000
5-percent total deformation strengths, psi		
100 hours	41,000	37,000
1000 hours	30,000	24,000
Stress for a creep rate of 10^{-6} in/in/hr, psi	20,000	9,000

Discussion

These results confirm the damage effect from cold work on creep-rupture properties of stable alpha alloys at 1000°F. The survey tests at 1000°F

for Reference 2 were uncertain in this respect due to limitation of tests to 100 hours where not too much difference existed for the two conditions and the single creep tests did not give too definite an answer. The reduction in properties was greater for creep than for rupture. Instability introduced by cold work and the resultant recovery and recrystallization effects were responsible.

It is not known to what degree the conditions of forging of the 6 Al - 0.5 Si alloy influenced the comparative properties for the two conditions. It can be noted in Figures 13 and 14 that the curves tend to flatten out at the lower stresses and longer time periods. Apparently the structural changes reach an end point after which the strength improves in relation to higher stress tests when the instability manifested itself throughout most of the test.

SURVEY OF EMBRITTLEMENT OF TITANIUM ALLOYS DURING CREEP TESTING

Embrittlement of titanium alloys was recognized as a problem during the course of the investigation. To obtain information on this subject a survey of embrittlement during creep testing was carried out using all the available specimens from the creep tests at 600°F to 1000°F performed in the studies reported in References 1 and 2. Specimens available from the investigation of creep at 75°F to 600°F for Reference 3, mainly specimens of sheet, were also surveyed. Embrittlement, as a result of creep testing, was evaluated by the change in room temperature tensile test ductility. As the results will show, severe embrittlement was observed in alpha-beta, meta-stable beta and stable beta alloys after creep testing.

Hydrogen content had been recognized as a major source of embrittlement. However, the research covered by References 1, 2 and 3 had identified certain other structural changes which could cause reduced ductility. Alpha-beta and meta-stable beta alloys underwent transformation of beta to form finely divided transition structures. There was some evidence that stable beta alloys might be subject to the same reactions. The formation of the finely divided transformation products had been shown to be accompanied by increased tensile strength and reduced ductility in tensile tests at 75°F and to at least 800°F. Reactions of the strain aging type which might contribute to embrittlement were also identified in the research of Reference 3.

In view of these possibilities, certain preliminary studies were carried out to obtain more information on the factors involved. Experiments were carried out to obtain information on the time required for embrittlement of the alpha-beta alloy Ti 150A under stress at 600°F and 800°F. Cooperative studies were undertaken with the Materials Laboratory, WADC, to obtain information on the influence of hydrogen. A few samples of Ti 150A were vacuum annealed to remove hydrogen and then exposed to 600°F and checked for embrittlement. Samples of alpha alloy 6 Al were vacuum annealed and used to check the influence of hydrogen on creep resistance. The Materials Laboratory cooperated by conducting the vacuum anneals and analyzing for hydrogen.

Tensile Properties at Room Temperature after Creep Tests

Tensile tests were conducted at room temperature on the specimens from the creep tests carried out for prior investigations (Refs. 1, 2 and 3). All creep specimens which had not been used for some other purpose, such as metallographic examination, were tensile tested.

The alloys for which data are presented were:

1. Alpha titanium Ti 75A

2. Stable alpha alloys 6 Al and 6 Al - 0.5 Si

3. Alpha-beta alloys Ti 150A, Ti 155AX, and RC 130A

4. Meta-stable beta alloy 10 Mo

5. Stable-beta alloy 30 Mo

Part of the Ti 150A and RC 130A specimens had been creep tested as sheet at 75°F to 600°F for Reference 3. The other specimens were from tests at 600°F to 1000°F for References 1 and 2.

Experimental Materials

The chemical compositions reported for the alloys are given in Table 6. Hydrogen analyses were made by the Materials Laboratory, WADC. Oxygen analyses were procured from the National Research Corporation. The remainder of the analyses were those reported by the producers.

The Ti 75A, Ti 150A, RC 130A and Ti 155AX were procured from commercial producers. The other alloys were made as small heats by Armour Research Foundation.

Alpha Titanium Ti 75A

Specimens from two heats (Table 6) were subjected to tensile tests at 75°F. The specimens from Heat L-984 were from the creep tests of Reference 1 and those from Heat M-626 from creep tests reported in Reference 2. The results obtained (Table 7) showed the following:

1. Specimens annealed prior to creep testing showed some loss in ductility and an increase in tensile strength. The elongation was reduced more than reduction of area.

2. Specimens cold worked prior to creep testing increased in ductility and decreased in tensile strength.

3. No evidence of embrittlement was found.

4. The data were not sufficiently complete to define effects of temperature of prior treatment.

5. The hydrogen levels were 87 and 150 ppm. It is possible that the somewhat greater loss in ductility of the Heat M-626 specimen was due to the 150 ppm of hydrogen in this heat.

6. The changes in ductility were no more than would be anticipated from creep testing alone, except possibly the test on the Heat M-626 specimen. Relief from cold work during testing is the cause of the changes in the properties in that condition.

Stable Alpha Alloys 6 Al and 6 Al - 0.5 Si

The specimens of the stable alpha alloys tensile tested had been creep tested for References 1 and 2. The chemical compositions are given in Table 6. Hydrogen levels were 150 to 170 ppm.

Ductility changed very little as a result of creep testing (Tables 8 and 9) for the available specimen of either alloy. Even the recrystallization during creep testing observed in References 1 and 2 for cold-worked material did not change ductility. Tensile strengths increased for annealed or as-forged stock and decreased for cold-worked material.

The 6 Al - 0.5 Si specimen had been cold worked prior to creep testing. Because prior cold work seemed to inhibit loss of ductility for Ti 75A, it is possible that material not cold worked would show embrittlement. A precipitate at the grain boundaries of the 6 Al - 0.5 Si alloy was previously observed (Ref. 2).

The stable alpha alloys did not show the decrease in ductility observed for alpha titanium Ti 75A even though hydrogen contents were similar.

Alpha-Beta Alloys Ti 150A, Ti 155AX and RC 130A

Several sets of specimens of Ti 150A alloy were tensile tested at 75°F after creep testing. Specimens made from bar stock and creep tested at 600°F to 1000°F for References 1 and 2 are covered by Table 10. Bar stock material creep tested at 75°F to 600°F for Reference 3 is covered by Table 11. Sheet samples creep tested at 75°F to 400°F for Reference 3 are covered by Table 12. Data for one specimen of Ti 155AX alloy is included in Table 9. Data for RC 130A alloy in sheet form after creep testing at 76°F to 400°F are given in Table 13.

Available chemical composition for the alloys are given in Table 6.

The tensile properties after creep testing indicate the following effects:

1. Heat L1006 of Ti 150A alloy underwent severe embrittlement in creep tests at 600°F to 1000°F. All specimens were so brittle that premature fracture occurred in the threads at a low stress.

2. Heat M739 was also severely embrittled. It may possibly have been embrittled slightly less than Heat L1006. Specimens tested at 1000°F fractured in the gage length with some ductility. The material annealed at 1500°F was somewhat less subject to embrittlement than that air cooled from 1500°F.

3. The material from Heat L774 became very brittle during creep tests (Table 11) at 400°F and 600°F. There may have been a slight decrease in ductility in tests at 76°F and 210°F. The severity of embrittlement appeared to increase as prior treatment was varied from as produced and the 1500°F air cool to the 1500°F water quench.

4. There was little evidence of embrittlement of Ti 150A sheet in a number of conditions after creep testing at 76°F (Table 12). A very few tests on as-produced material after creep testing at 210°F and 400°F showed some decrease in ductility. Data were not available for ductility prior to testing for most of the treatments. Such variations as were observed could be due to the heat treatments.

5. The RC 130A sheet quenched from 1625°F was severely embrittled (Table 13) during creep testing at 400°F. This material initially had very low ductility. One sample air cooled from 1250°F also showed low ductility after testing at 400°F. Otherwise, the specimens did not show an appreciable change in ductility.

6. One specimen of Ti 155AX alloy (Table 9) subjected to creep at 600°F showed loss in ductility but not severe embrittlement.

The hydrogen analyses available for these materials are incomplete. The sheet Ti 150A (Heat X672) had 510 ppm hydrogen and bar stock (Heat M739) had 310 ppm. It can probably be assumed that the other two heats had high hydrogen. There is, however, no definite proof that the slightly lower susceptibility to embrittlement of Heat M739 was due to lower hydrogen. The hydrogen of the Ti 155AX was only 180 ppm which may account for the one specimen tested not being severely embrittled. The hydrogen in the RC 130A sheet was 83 ppm with this very much lower value than the 500 ppm in the Ti 150A sheet again being a possible cause for its greater resistance to embrittlement.

The data show that loss in ductility increased with testing temperature up to 800°F. The embrittlement was also more severe the higher the initial tensile strength, the higher the beta content and possibly was most severe for the initially brittle structures quenched from the beta range. It is uncertain at this point which is the more important factor: transformation of beta during testing or hydrogen effects.

Meta-Stable Beta Alloy 10 Mo

Two specimens of 10 Mo alloy were available for tensile tests at room temperature after creep tests at 600°F. Both were severely embrittled (Table 9) and fractured in the threads prematurely at a higher stress value than the as-treated tensile strength.

The alloy contained 130 ppm of hydrogen. The treatments used prior to creep testing were known to produce structures which were essentially all beta and were known (Ref. 2) to be subject to transformation at 600°F to a finely dispersed transition product between beta and alpha. Formation of this product was accompanied by an increase in tensile strength and a severe loss in ductility at 75°F. It is presumed that the structural changes during testing were mainly responsible for the observed embrittlement.

Stable Beta Alloy 30 Mo

Both specimens of 30 Mo alloy available for tensile tests at room temperature after creep testing were severely embrittled (Table 9). The as-forged material tested at 1000°F was embrittled more than a specimen quenched from 1325°F and creep tested at 600°F.

The heat contained 300 ppm of hydrogen (Table 6). This amount of hydrogen would be expected to contribute to loss in ductility. In Reference 2 it was noted, however, that there was some metallographic evidence of precipitation or transformation during creep-rupture testing. It is, therefore, possible that structural changes as well as hydrogen were involved in the observed embrittlement.

Discussion

The alpha-beta, meta-stable beta and stable beta alloys were all severely embrittled during creep testing. The degree of embrittlement increased with creep-test temperature from 75°F to 1000°F with a probable maximum effect at about 800°F.

Stable alpha alloys were not embrittled by creep testing at 600°F to 1000°F for the conditions examined. Alpha titanium Ti 75A suffered some loss in ductility in creep tests at 600°F and 800°F except when cold worked prior to creep testing.

The degree of embrittlement of the alpha-beta alloys apparently increased with hydrogen content although this was not well established. The presence of fine transformation products was associated with increased embrittlement during creep testing in both alpha-beta and meta-stable beta alloys. This was particularly evident in the increased severity of embrittlement as the amount of beta which could transform during testing was increased.

The degree to which hydrogen and the increases in strength and losses in ductility due to beta transformation were involved in the embrittlement during creep testing is uncertain. Stable beta alloy 30 Mo was also very subject to embrittlement although there is not very good evidence of hydrogen being the major factor. The work of Reference 2 suggested that the stable beta alloy might have been unstable during creep testing. Certainly alpha alloys without a beta transformation were nearly immune to embrittlement at hydrogen levels where the other alloys were embrittled.

There is a further possibility that elements such as carbon, nitrogen and oxygen were involved in the embrittlement. Furthermore, Reference 3 had indicated strain aging type reactions during creep testing which would be expected to reduce subsequent ductility at 75°F.

Embrittlement Time for Alpha-Beta Alloy Ti 150A

The severe embrittlement of alpha-beta alloy Ti 150A during creep testing raised the question of the time required for embrittlement to occur. A limited investigation of the subject was undertaken. Specimens of material heat treated at 1500°F for one hour and air cooled were exposed at 800°F under a stress of 35,000 psi. Another group of specimens which had been heat treated one hour at 1350°F and furnace cooled were exposed at 600°F under 83,000 psi. Exposure times were varied in order to establish the time for embrittlement in subsequent tensile tests at room temperature.

The choice of temperatures for exposure was based on two considerations. Transformation of beta had been found to occur at a maximum rate at 800°F (Ref. 2). Also creep was a major factor at 800°F. The temperature of 600°F was selected because creep was very slight and it was desired to test the more stable structure where little creep deformation took place.

Experimental Material

The specimens were made from stock from Heat L1006 having the composition given in Table 6. The hydrogen content of Heat L1006 was not determined although it was probably quite high. The survey of properties after creep testing indicated that Heat L1006 was embrittled more than Heat M739 which contained 310 ppm of hydrogen.

Procedure

The stock was heat treated in argon and specimens machined. These specimens were started in creep-rupture units in the usual way. The first exposure time was for 200 hours when the specimens were cooled and removed

from the test units. Tensile tests were then conducted at room temperature. Based on these results, shorter and longer exposure times were selected to define embrittlement characteristics.

Embrittlement at 600°F

Significant decrease in ductility resulted from 50 hours at 600°F under a stress of 83,000 psi for the stock initially annealed at 1350°F (Table 14). There was still measurable ductility after 300 hours. A specimen exposed for 1173 hours had no measurable ductility.

Embrittlement at 800°F

Severe embrittlement occurred in 20 hours at 800°F (Table 14) for the material air cooled from 1500°F exposed at 800°F under 35,000 psi. All specimens exposed for a longer time fractured prematurely in the threaded ends of the specimens.

Discussion

The data indicate that embrittlement at 800°F was a matter of a few hours and a somewhat longer time at 600°F. It is not known to what degree the differences in heat treatment were involved. The results of the preceding section suggest that the material air cooled from 1500°F and stressed at 800°F should have been more susceptible to embrittlement than the 1350°F furnace-cooled material used for the stock exposed at 600°F. The data for 600°F exposure do show that a fairly stable structure can be embrittled under condition where little creep occurred. Both the creep and the increased rate of beta transformation would be involved in the more rapid embrittlement at 800°F.

Preliminary Surveys on the Influence of Hydrogen on Properties of Titanium Alloys

Hydrogen was known to be an important factor in embrittlement of titanium alloys. There was a question, however, as to whether it influenced creep resistance and the degree to which it was a factor in embrittlement during creep testing at temperatures of 600°F to 1000°F. Two small survey-type investigations were carried out to obtain information regarding the factors involved:

1. The influence of vacuum annealing on the embrittlement of alpha-beta alloy Ti 150A at 600°F.

2. The influence of vacuum annealing on the creep-rupture properties of stable alpha alloy 6 Al at 1000°F.

Vacuum annealing was expected to reduce hydrogen content. A temperature of 1000°F was used for the study of the effect on creep characteristics of the 6 Al alloy because creep was the predominate factor governing strength at this temperature. The Ti 150A was checked for embrittlement at 600°F after vacuum annealing because it was desired to study the phenomena at a temperature where creep was a very minor factor.

The investigation was carried out cooperatively with the Materials Laboratory, WADC. The vacuum annealing and hydrogen analyses were carried out by the Materials Laboratory.

Influence of Vacuum Annealing on Embrittlement of Alpha-Beta Alloy Ti 150A at 600°F

An annealing time of 24 hours at 1200°F in vacuum was used. Comparative specimens annealed in argon were also prepared on the assumption that hydrogen would not be removed but the influence of heat treatment would otherwise be similar. The specimens were prepared from Heat M739 which had been annealed at 1350°F. Heat M739 initially contained 310 ppm of hydrogen (Table 1). The vacuum-annealed samples were not analyzed for hydrogen. The Materials Laboratory estimated the hydrogen to be 20 ppm.

Exposure conditions for embrittlement were as follows:

1. Sealed in Vycor tube under vacuum and heated for 100 hours at 600°F.

2. Heated for 100 hours at 600°F in air without stress.

3. Heated for 100 hours at 600°F in air under a stress of 83,000 psi.

The specimens were then tensile tested at room temperature to compare with the properties after the respective treatments at 1200°F.

The 24 hour treatments at 1200°F increased ductility over that of the condition of annealing from 1350°F (Table 15). There was little difference from the vacuum or argon atmospheres.

The argon annealed specimen underwent a significant decrease in ductility (Table 15) during exposure at 600°F for 100 hours in the evacuated Vycor tube. The vacuum-annealed material was not changed. The results were practically the same when exposed for 100 hours in air without stress.

The vacuum-annealed specimen fractured in 1.8 hours at 600°F under 83,000 psi. The argon-annealed sample showed expected strength and when subsequently tensile tested at room temperature the expected embrittlement occurred.

Specimens were examined under the microscope and no definite evidence of titanium hydride formation was observed.

The experiments were too limited to be certain of the significance of the data. The data suggest that the removal of hydrogen during vacuum annealing at 1200°F practically eliminated susceptibility to embrittlement at 600°F. There are, however, a number of uncertainties before this can be accepted as an established fact.

The probability is, however, that hydrogen was a major factor in the embrittlement of the argon-annealed material. The degree of embrittlement was nearly that to be expected after exposure to stress, assuming that the treatments at 1350°F and 1200°F would reduce the tendency for embrittlement previously discussed.

The premature rupture of the vacuum-annealed specimen in the stressed exposure test was unexpected and is not yet explained. All test conditions were carefully checked and found to be correct.

Stress during exposure to 600°F increased the degree of embrittlement to a marked extent over unstressed exposure in the argon anneal material. It is known that transformation of beta can occur during testing at 600°F. It is possible that this was a contributing factor to the increased embrittlement. The influence of such transformation will not be established, however, until stressed tests on low hydrogen material are available. Stress may simply intensify the embrittling effect of hydrogen.

Creep-Rupture Properties of Vacuum Annealed Stable Alpha Alloy 6 Al

Creep-rupture tests were conducted at 1000°F on samples of stable alpha alloy 6 Al which had been vacuum annealed at 1200°F for 24 hours. Comparative data were obtained for samples similarly annealed in argon. The assumption was made that the vacuum anneal would reduce hydrogen content while it would remain unchanged during argon annealing. The results were expected to show whether hydrogen had a substantial effect on an alpha-type alloy under conditions where creep was the predominate factor in the determination of strength properties.

As-forged stock from Heat 4a having the composition given in Table 6 was used.

The hydrogen content of Heat 4a was 150 ppm. It was assumed that this was unchanged during the argon anneal. Hydrogen was presumed to be reduced by the vacuum anneal. A definite analysis was not obtained.

The data obtained from the creep-rupture tests are given in Table 16 and Figure 16. No appreciable differences in either tensile or creep-rupture properties were found. The vacuum-annealed samples had slightly lower elongation in the rupture tests.

Two tests on the vacuum-annealed material under 30,000 psi showed less creep and lower creep rates (Fig. 17) than the material annealed in argon. Single tests at 27,000 psi (Fig. 18) gave practically identical creep curves for both conditions of annealing.

The only differences observed between the vacuum and argon-annealed samples were the somewhat lower ductility of the vacuum-annealed samples and a somewhat higher creep resistance in some tests. The tensile and rupture strengths were nearly the same. Moreover, they were nearly the same as those found for the as-forged condition in Reference 2. Any of the differences which did exist could easily be due to unknown slight differences in conditions of heating at 1200°F before testing.

It should be recognized that the difference in hydrogen content was not very large. The material which was vacuum annealed probably contained about 20 - 50 ppm in comparison to 150 ppm in the argon-annealed samples. The results should not therefore be considered to eliminate the possibility of there being a hydrogen effect on creep at 1000°F for the stable alpha alloy 6 Al.

INFLUENCE OF SPONGE HARDNESS AND HYDROGEN CONTENT ON PROPERTIES

The previous results from the investigation together with general developments in the metallurgy of titanium alloys had raised questions as to the role of interstitial element levels and hydrogen on creep-rupture resistance and embrittlement during exposure under creep conditions. Accordingly, a systematic program was planned to investigate the role of these two factors in affecting properties in the range from 600°F to 1000°F as a function of the type of microstructure of the alloys.

The alloys to be investigated included:

Stable alpha	6-percent aluminum (6 Al)
Alpha-beta	2.75-percent chromium - 1.5-percent iron (Ti 150A)
Alpha-beta	8-percent manganese (8 Mn)
Meta-stable beta	10-percent molybdenum (10 Mo)
Stable beta	30-percent molybdenum (30 Mo)

With the exception of the alpha-beta alloy, Ti 150A, each alloy was to be studied when made with low (100 - 110 BHN) and with medium hardness (140 - 150 BHN) titanium sponge. In addition, each material was to be investigated at two or three levels of hydrogen. The major research was to be conducted on the 8 Mn alloy. Because the data for each alloy apply to more than one objective, results are presented for each alloy separately.

The investigation was only partly completed. One reason was the delay in obtaining delivery of the alloy stock. The problems associated with the development of equipment to remove and add controlled amounts of hydrogen and in the analytical determination of the hydrogen content of experimental materials proved difficult. A further major delay developed when it was found that the 8 Mn alloy, on which delivery had been delayed, would not respond properly to heat treatment.

General Procedure

The supply of alloy stocks remaining from previous investigations was reviewed and the additional materials required for the objectives of the investigation determined. After some delay Armour Research Foundation proved

to be the only source of alloy stock made with titanium sponge having the desired hardness levels.

A unit was designed and built to produce controlled amounts of hydrogen in the experimental materials. The unit consisted of a chamber which could be heated under controlled conditions under a vacuum or with hydrogen atmospheres. When the unit was completed, experiments were conducted to establish suitable operating conditions to provide the necessary hydrogen levels. Samples were analyzed for hydrogen in a vacuum fusion apparatus.

When suitable samples could be produced, tests were conducted to establish the influence of the variables on creep-rupture and embrittlement characteristics.

Sponge Hardness

Stock made from titanium sponge of 100 - 110 and 140 - 150 BHN was to be evaluated for the 8 Mn and 30 Mo alloys. Material made with 100 - 110 BHN sponge was to be used for the stable alpha alloy 6 Al. The data in References 1 and 2 were to be used to provide comparative properties for 6 Al alloy made with the higher hardness sponge.

The materials were to be evaluated by survey type creep-rupture tests at 600°F and 1000°F after being vacuum annealed to about 50 ppm of hydrogen. The object was to study interstitial element effects without interference from hydrogen. Very little creep would occur at 600°F and creep would be the predominant factor at 1000°F.

Hydrogen Content

The influence of hydrogen on creep resistance was to be carried out using all four alloys in the program as made with 100 - 110 BHN sponge. Initial tests were to heat 600°F and 1000°F. All materials were to be tested in the annealed condition with approximately 50 and 250 ppm of hydrogen.

The only exception to this was that the 8 Mn alloy was to be included in both programs when heat treated to a high hardness and low ductility (high in alpha-beta range and rapidly cooled).

Mechanism of Embrittlement

The planned research was limited to alpha-beta alloys Ti 150A and 8 Mn. Structures were to be made close to equilibrium by annealing except for some work to be done on the 8 Mn alloy when quenched from 1625°F and from 1400°F. Variables were to be exposure temperature, time and stress, as well

as sponge hardness and hydrogen content. Stress levels were to be approximately 40 and 80 percent of the estimated 1000 hour rupture strength and the times were not to exceed 1000 hours. The hydrogen levels were to be approximately 50, 250 and 500 ppm. Evaluation of embrittlement was done by tensile tests at 75°F.

After delineation of the conditions of embrittlement, microstructural and x-ray diffraction studies were to be used to define the mechanism. Particular attention was to be directed to the relative effects of beta decomposition and hydrogen.

The whole scheme of testing was set up so that each test could supply appropriate data for the three phases of the research.

Experimental Materials

The materials for the investigation were as follows:

Alloy	Heat No.		Amount Ordered (lb.)	Bar Size	Sponge Hardness	Date Received
	Supplier	U of M				
6 Al	9033	4c	7	0.5-in. dia.	107	June 1955[a]
6 Al	Replacement Heat			0.5-in. dia.	107	April 1956
Ti 150A	M739	--	--	0.5-in. dia.	(na)	On hand
8 Mn	9035-9	3a	40	0.75-in. sq.	107	Sept. 1955
8 Mn	9056	3b	20	0.75-in. sq.	148	Sept. 1955
10 Mo	9038	5a	5	0.5-in. dia.	107	July 1955
30 Mo	--	6	--	0.5-in. dia.	~140	On hand
30 Mo	9042	6a	10	0.5-in. dia.	107	Sept. 1955

(a) -- After preliminary work, this heat was rejected due to an excessive number of cracks found in the bars during machining of specimens.
(na) - Not available.

The available chemical compositions for the experimental materials are given in Table 17. The table also gives the composition of the sponge titanium used to melt the new heats ordered for the investigation. The compositions given are those reported by the producers with the exception of those given for hydrogen. The hydrogen values are for the materials as supplied. Those materials which

were on hand had been analyzed by the Materials Laboratory, WADC, from samples furnished for the work reported in the preceding section. The new heats were analyzed for hydrogen by the University of Michigan.

The compositions are generally normal except for the low hardness sponge 30 Mo alloy, which had a high carbon content. The producer reported that the Mn was higher in the 8 Mn alloy than expected. It will be noted that carbon was somewhat high also.

On the basis of the reported compositions of the titanium sponge, the iron and nitrogen were higher in the 148 BHN sponge heats. Oxygen values, another important element in sponge hardness, were not available.

All materials except the Ti 150A were made as small laboratory heats by the producer as described under the results for the individual alloys. The Ti 150A was obtained from a commercial heat.

Hydrogen Control Apparatus

A furnace with a chamber permitting annealing in vacuum or hydrogen atmosphere was constructed to provide experimental materials with controlled hydrogen contents.

A schematic diagram of the apparatus is shown by Figure 19. The unit consisted essentially of a sealed tube in an electrical resistance furnace. The tube could be attached to a vacuum pump or to a gas supply system. Suitable controls and gages were provided.

The chamber was Sillimanite tube 3-inches in diameter by 42-inches long. The tube rested on a flanged plate attached to the under side of the furnace shell. The plate opened into a reducing tee. The tube was sealed by O-rings between flanged covers such that the rings were tightly pressed against the tube. The projecting ends of the tube were water cooled to protect the O-rings.

Heat was supplied by electrical current passing through Chromel-A resistance wire in baked alumina cores. Three separate cores were stacked and hooked up so that current could be proportioned between the three sections by shunts. Closer windings of the wire were used at the ends to compensate for heat loss. Temperature was controlled by a Pyro-O-Vane automatic temperature controller actuated by a thermocouple imbedded in the resistance wire heater. Temperature of the chamber was measured by a thermocouple inserted in a sealed tube extending through the top cover plate.

Specimens were placed in a stainless steel mesh basket hung from the underside of the top cover plate. If the specimens were to be slow cooled

the basket was attached to a hook. If rapid cooling was to be used, a thin wire was introduced and attached and leads through the cover plate so that the wire could be melted with electric current. A removable cover plate was attached to the bottom end of the chamber. An inert gas could be introduced into the system; the cover plate removed; and the specimens dropped out of the furnace for air cooling or liquid quenching.

The outlet to the chamber was made through a refrigeration valve mounted on the top of the chamber. A Mega Vac mechanical pump provided a final vacuum pressure of about 0.1 micron. Arrangements were made so that a diffusion pump could have been added if it had been necessary. Pressures were measured with a thermocouple vacuum gage. It was generally possible to pump below the lower limit of sensitivity of the vacuum gage (1 micron) in less than 5 minutes. A vacuum valve was placed in the line to the pump. A manometer was also attached to the chamber for measuring pressure.

A series of two- and three-way stopcocks provided a manifold for introducing measured amounts of hydrogen. Hydrogen could be introduced into the measuring chamber and the desired quantity introduced into the treatment chamber. Excess gas could be withdrawn through a vent connected to an auxiliary pump. All hydrogen used for hydrogenation treatments was extra-dry, high purity hydrogen. Provision was made for drying and purifying tank argon to supply an inert atmosphere when needed.

Calibration of Apparatus

Temperature measurements defined a zone 4-inches long in the chamber where temperature varied less than 5°F and a 6-inch zone with less than 10°F variation. Temperature cycling was less than 5°F. The controller had to be set 50°F to 75°F low while the temperature in the chamber was being raised and then gradually brought to the desired temperature to avoid overheating. Because specimens were never longer than 3 inches, temperature variations were no problem.

The system was continuously pumped for two test runs. In the first run a sample of Ti 75A, 0.5-inch in diameter by 3-inches long, was carefully weighed and placed in the chamber. In the second run, five such pieces were used. The operating conditions and weight gains were:

1. 13 hours at 900°F + 25 hours at 1100°F, furnace cool - 0.0136% weight gain.

2. 24 hours at 1350°F, furnace cool - 0.0069% weight gain.

Assuming oxygen and nitrogen absorption in the proportions in air, the weight gains were calculated to indicate an effective dynamic leak rate of about 1 to 3×10^{-8} liters of air (STP) per second. This derived value was checked by pressure rise measurements in which the static leak rate was found to be about

4×10^{-6} liters of air (STP) per second. The contamination of a 0.5-inch diameter bar specimen resulting from 24 hours at 1350°F was equivalent to 0.091 mg. per sq. cm. On an absolute basis the weight gain of 0.0069 percent was not excessive in relation to the 0.1 to 0.2 percent oxygen and nitrogen initially present in alloys. Microhardness measurements showed no hardness variation from the surface inward. It should be noted that specimens were machined to 0.25-inch diameter in the gage length from 0.500-inch rounds which would remove surface contamination.

The Ti 75A samples were analyzed for hydrogen by duplicate determinations with the following results:

Condition	Hydrogen (ppm)
As-produced	83, 84
13 hours at 900°F + 25 hours at 1100°F, furnace cool	81, 80
24 hours at 1350°F, furnace cool	56, 52

Table 18 gives the analyzed hydrogen contents after vacuum annealing for the successful runs using 24 hours at 1350°F and furnace cooling. The only exception was 8 Mn alloy where hydrogen was not removed at 1350°F.

Introduction of Hydrogen

The general procedure was:

1. Anneal in vacuum for 24 hours at 1350°F to reduce hydrogen to a known value. (Approximately 50 ppm)

2. Increase temperature to 1500°F, shut off vacuum pump and add hydrogen. Reference 4 indicated that this was about the lowest temperature at which a reasonable rate of hydrogen adsorption could be expected.

3. Furnace cool to approximately 1250°F and hold for one to two hours for attainment of structural equilibrium and finally furnace cool.

The measuring chamber was filled with hydrogen to a fixed pressure and the temperature measured. The weight of hydrogen was then calculated. From the weight of hydrogen to be transferred to the specimens, a final pressure could be calculated for the measuring chamber. The hydrogen was then bled into the evacuated furnace chamber until the required amount was added. Pressure in the chamber was measured as a function of time. When the results

of hydrogen analyses became available it was possible to compare the amount of hydrogen absorbed with values calculated from pressure drop with time. It was found that the initial pressure could be relied upon as a start in computing the amount of hydrogen absorbed. There was no rapid initial absorption to upset calculations.

Accordingly, an effective gas temperature for the whole system was established as 477°F. This then permitted calculation of hydrogen absorbed as a function of pressure drop.

Normal procedure was to place from 7 to 9 specimen blanks in the basket along with a small slug for analysis for hydrogen for each run. An experimental run established that there was no significant variation in hydrogen from point to point on specimens or with specimen location in the basket. (See Table 19, "Calibration Run on Ti 75A".) Specimen blanks were 3-inches long by 0.5-inch in diameter.

The operating experience with the unit is shown by Table 19. Proper operation and consideration of all factors allowed production of hydrogen contents within about 20 percent of aim amounts. Difficulty was encountered in placing the unit in operation and learning how to produce desired hydrogen levels. Other runs were made where operating difficulties did not permit calculation of the amount of hydrogen to be absorbed.

Considerable variation in the time necessary to obtain the necessary hydrogen absorption was encountered between alloys. This is reflected in the time necessary at 1500°F for the pressure to drop to the necessary value. Comprehensive time-pressure data were not taken because this behavior had not been anticipated. The general behavior of the different alloys is, however, shown schematically in Figure 20. The rate was initially high for the Mo alloys and then fell off to low values. The Ti 150A and 8 Mn alloys absorbed hydrogen at a slower and more uniform rate. Ti 75A was intermediate. Apparently, mixed alpha-beta structures absorb hydrogen at 1500°F at a slower rate than alpha or beta. Alpha in turn is slower than beta.

Hydrogen Analyses

The hydrogen analyses performed at the University of Michigan were run in a National Research vacuum fusion gas analysis apparatus. The technique used was that of hot-extraction. The samples for analysis were machined to 3/16 to 1/4-inch cubes. After machining they were polished on 600-mesh silicon carbide paper.

In the analysis procedure, the sample was dropped into a graphite crucible and heated to 1400°C (2552°F) for 20 minutes. The hydrogen analysis was calculated from the sample weights and the total pressure of the gas collected.

Duplicate samples were run for each analysis and an average hydrogen content was calculated. Blanks were run on the unit between sample analyses.

Stable Alpha Alloy 6 Al

Only tensile tests at 75°F, 600°F, and 1000°F with a few creep-rupture tests at 1000°F were conducted on the low hardness sponge Heat 4c. During machining of specimens the stock was found to be so badly cracked that only a few sound specimens could be obtained. The heat was then rejected, but the replacement heat was not received in time for inclusion in the investigation. The chemical compositions are given in Table 17.

The as-forged stock from Heat 4c was annealed in vacuum at 1350°F for 25 hours, which reduced the hydrogen from 84 to 34 - 51 ppm. All test results are limited to this material (Table 20). There were not sufficient specimens to produce material with 250 ppm of hydrogen. The limited number of specimens resulted in incomplete tests on the low hydrogen condition. Comparative properties for high hardness sponge alloy were limited to results from previous studies.

The results of the few tests (Table 20) did not show any definite effect from the low hardness sponge or the low hydrogen. Strictly comparative data for similar heat treatments were not available. Previous results (Refs. 1 and 2) had not shown much variation in properties with heat treatment. Accordingly, the tensile properties of the vacuum-annealed samples from Heat 4c are compared with previous data in Table 21 to show that any trends of the data point to a heat-to-heat difference attributable to factors other than the sponge hardness and hydrogen content.

The rupture and total deformation values were nearly the same (or slightly stronger) as those for Heat 4a in Reference 2. They were, however, below those for Heat 4 in Reference 1. The minimum creep rates indicated higher creep resistance than for Heat 4a. The Heat 4c properties had similar relations to the Heat 4a specimen vacuum annealed at 1200°F (Fig. 16) as previously presented in this report.

The data are too sparse for any conclusions. The trends, however, seem to indicate that there was no proven effect of sponge hardness or hydrogen content; and that if any existed they were relatively minor.

Alpha-Beta Alloy Ti 150A

The variable investigated for Ti 150A alloy was the influence of hydrogen content on creep-rupture properties and on embrittlement under stress. Sponge hardness effects were not studied.

Material from Heat M739 (Table 17) with a hydrogen level of 310 ppm was vacuum annealed to 57 - 64 ppm. Hydrogen was then added to samples to raise the content to 162 and 274 ppm. These samples were exposed to stress for various lengths of time under stresses approximately 40 and 80 percent of the 1000-hour rupture strength in the temperature range of 600°F to 1000°F. Creep data were taken during the tests. The samples were then subjected to tensile tests at room temperature to define the effects of hydrogen on the embrittlement characteristics. There was insufficient time to complete all tests for the higher hydrogen levels.

In evaluating the effect of hydrogen content, consideration must be given to the differences in thermal history introduced to obtain the variations in hydrogen (Tables 18 and 19), since thermal treatments also influence the properties.

Results

The creep data (Table 22) are not sufficiently complete to define any definite effect of hydrogen. There was a tendency for the high hydrogen material (274 ppm) to show more creep. The range of results obtained was rather small and, in view of the variation between specimens and the difference in heat treatment, neither proves nor disproves an effect. Apparently, however, there is no large effect.

The tensile properties after exposure to stress and temperature (Table 23) show that embrittlement is influenced by hydrogen content. The material with 274 ppm of hydrogen was subject to more loss in ductility at 600°F than were the two lower levels of hydrogen (Fig. 21) during 100 hours of exposure. The materials containing 162 and 274 ppm hydrogen suffered more loss at 1000°F than the material with 57 - 64 ppm. Tensile strengths also increased at 600°F with increasing hydrogen.

The influence of time of exposure (Fig. 22) was defined for the material with 57 - 64 ppm of hydrogen. There was a slight decrease in ductility and an increase in tensile strength at 600°F in 500 hours with little change to 1000 hours. Apparently, changes were complete in 30 hours at 1000°F. The data are incomplete at 800°F and 900°F.

For 274 ppm of hydrogen the difference in subsequent properties after different exposure time at 1000°F was small (Fig. 21). Table 23 includes a value previously determined for exposure for 1000 hours where the material containing 310 ppm of hydrogen was argon annealed at 1500°F. This result suggests some recovery of ductility at longer times.

Stress level during exposure had a considerable effect (Fig. 22a) at 600°F on both tensile strength and ductility of the 57 - 64 ppm hydrogen material. Changes were greater for a stress level of 64,000 than for 32,000 psi. There was relatively little difference at 1000°F for 2,800 and 5,600 psi

(Fig. 22). A sample of 274 ppm material had higher ductility after 100 hours under 5,600 psi than under 2,800 psi. It is possible that the stressed exposure for material argon annealed at 1500°F as discussed in the previous paragraph had higher ductility due to the stress of 5,000 psi rather than the longer time period.

The influence of temperature on embrittlement is not well defined. The material with 57 - 64 ppm of hydrogen showed a drop in room temperature ductility (Fig. 22) between 800°F and 900°F in 100 hours. The high hydrogen material showed a substantial drop at 600°F whereas there was little effect from 162 ppm of hydrogen at 600°F. Apparently the temperature and time for embrittlement changes with hydrogen content.

Discussion

The results show two main features of the influence of hydrogen on embrittlement of alpha-beta alloy during exposure to creep conditions:

1. Some embrittlement can be introduced at temperatures of 900°F or higher even when the hydrogen is of the order of 50 ppm. It is not known to what degree this is due to hydrogen or to transformation of beta to alpha as discussed in the previous section. The possibility of other reactions in addition to these two also exist as a cause for embrittlement.

2. The temperature at which embrittlement occurs decreases with increasing hydrogen content. A fair degree of ductility was lost at 600°F when the hydrogen was 274 ppm. Very low ductility existed after exposure at 1000°F with 162 ppm of hydrogen. Again it is uncertain as to what degree transformation of beta was involved in these changes. Increasing stress (faster creep) slightly accelerates embrittlement.

There was a difference in heat treatment between the hydrogen levels which may have influenced the results to some extent. This should, however, be minor in this case.

A previous section presented a small amount of data for the same heat of Ti 150A vacuum annealed for 24 hours at 1200°F after an anneal from 1350°F. The tensile strengths (Table 15) were substantially higher than those obtained for this section. The degree of hydrogen removal is not known. The data in Table 15 did indicate reduction in ductility after exposure for 100 hours at 600°F without stress when the hydrogen level was high (annealed in argon - 310 ppm) and quite brittle material when exposed under stress for 100 hours. The vacuum anneal prevented lower ductility for heating without stress. It seems that an initially stronger structure intensifies the subsequent embrittlement for a given hydrogen level. This seems to be evident also in the data for specimens tensile

tested after creep (Table 10). Thus, embrittlement is at least a function of initial structure as well as hydrogen content.

Alpha-Beta Alloy 8 Mn

The alpha-beta alloy had been selected as the major experimental material for the study of interstitial elements as reflected in the hardness of the titanium sponge and hydrogen content as it influenced creep-rupture properties and embrittlement. The main effort on mechanism of embrittlement was also to be carried out on this alloy. The stock was, however, not received until September 1955. Further delay developed due to the resistance of the alloy to hydrogen removal and difficulties in obtaining the type of microstructures desired for the test program. No test data were obtained. The information presented describes the experiments carried out to develop proper structures and hydrogen contents.

Experimental Materials

The material needed for the bulk of the research was 0.5-inch square stock containing about 50 ppm of hydrogen and having a microstructure of equiaxed alpha grains in a beta matrix. The stock was supplied as 0.75-inch squares in the as-forged condition so that it could be treated to remove hydrogen and properly hot rolled and heat treated to produce the desired structure.

The chemical compositions (Table 17) show that the heats had somewhat high manganese contents. The producer reported that the manganese loss during melting was only 5 to 7 percent whereas prior experience had indicated a loss of 15 percent.

Heat 3a was made with 107 BHN sponge and 3b with 148 BHN sponge. The latter heat contained higher carbon and nitrogen than the former. The hydrogen in Heat 3a was 114 ppm and it was not determined for Heat 3b.

Procedure

The initial step was to hot roll bars in the alpha-beta temperature range and then vacuum anneal to remove hydrogen. When this was unsuccessful in producing the experimental material with the proper characteristic, other combinations of treatments were tried. The results of the experiments were evaluated by metallographic examination and hydrogen analyses.

In addition, a small amount of X-ray diffraction work was carried along in preparation for the mechanism studies.

Results

The as-forged stock from the 107 BHN sponge heat (Heat 3a) had a structure of equi-axed beta grains. This was unexpected and indicated that the heat was sluggish in transforming. The results of the attempts to heat treat to obtain the prescribed experimental material are given in Table 24.

When hot rolled at 1380°F in the alpha-beta range and subsequently annealed at 1360°F, there was only slight transformation. This is a common treatment to produce the spheroidal alpha in a beta matrix for such alloys.

Increasing the heat treatment time to 24 hours at 1320°F in vacuum did develop some spheroidal alpha. A similar structure was developed by first vacuum annealing and then rolling at 1300°F and subsequently annealing at 1200°F. While both of these treatments developed structures which might have been suitable for testing, the vacuum anneals at 1320°F did not reduce the hydrogen. A check analysis on treatment 2 did not indicate hydrogen pick-up during hot rolling.

At this point attention was directed towards removal of hydrogen with the development of structure and hot rolling to be final steps. A vacuum anneal at 1365°F did not reduce the hydrogen. It was decided to vacuum anneal for 24 hours in the beta range at 1500°F. (The alpha-beta to beta transus was about 1400°F for the alloy.) The hydrogen was reduced to 83 ppm. Some feathery alpha resulted from the furnace cool rather than the coarse basket-weave alpha characteristics of the alloy with normal transformation rates. A subsequent treatment of 24 hours at 1270°F did develop some spheroidal alpha along with some plate and feathery alpha.

At this stage the time for the investigation expired.

Some X-ray diffraction patterns were taken in preparation for the embrittlement mechanism studies intended to be performed on the 8 Mn alloy. The main objective of these studies was to develop familarity with the alloy and they were undertaken before the full importance of the sluggish nature of transformation and hydrogen removal was appreciated.

The analysis of the diffraction patterns gave the results indicated in Table 24. Only the number of identifiable lines are listed with no attempt to establish relative intensities. It will be noted that the diffraction lines indicated more transformation than was evident in the description of the microstructures. For instance, the as-forged material which appeared to be all beta gave lines for alpha. Moreover, all the samples indicated the presence of the transitional omega phase.

Discussion

The very sluggish transformation rate resulted in material which was very difficult to process to microstructures required for the experiments to define properties. In addition, the difficulty of removing hydrogen complicated the problem.

It would be necessary to use temperatures of 1500°F to 1600°F for vacuum annealing to reduce hydrogen to 50 ppm in 24 hours. This would require sufficient subsequent hot rolling to break up the coarse beta grain size and to precipitate alpha in a form which could be made equi-axed by subsequent treatment. It appears that the alloy furnished is of very doubtful suitability for the purposes of the investigation as planned. Some compromise in structures would have to be accepted and the results would be characteristic of a far more sluggish alpha-beta alloy than was the intent of the investigation.

Meta-Stable Beta Alloy 10 Mo

The influence of hydrogen on the creep-rupture properties of a meta-stable beta structure was evaluated using the 10 Mo alloy. The levels of hydrogen used were 44 and 242 - 260 ppm. Properties were evaluated by tensile tests and creep-rupture tests at 600°F and 1000°F.

Experimental Material

Stock from the heat made with 107 BHN sponge titanium (Table 17) was used. The as-forged bars were annealed in vacuum at 1350°F for 23.3 hours and furnace cooled. This treatment resulted in a final hydrogen content of 44 ppm. The stock with 242 - 260 ppm of hydrogen was similarly heated in vacuum, the temperature raised to 1500°F for one hour, when the hydrogen was added and then furnace cooled with a 2-hour hold at 1250°F.

Tensile and Creep-Rupture Properties

The results of the tests (Table 25) show the following:

1. There was no significant change with hydrogen content in tensile properties at 75°F, 600°F, or 1000°F.

2. The 44 ppm material showed little evidence of creep at 600°F for stresses just under the tensile strength. However, where similar tests were run on the 242 - 260 ppm hydrogen stock fracture occurred immediately. It is possible that the tensile test gave an abnormally high value; or high hydrogen develops a sensitivity for rapid fracture at 600°F when stresses are just under the tensile strength.

3. There was no difference in total deformation strengths at 1000°F indicated by the limited data (Table 25).

4. The one rupture test at 1000°F for each hydrogen level indicated slightly longer time for rupture for the low hydrogen stock. Consideration of total deformation and creep data suggests that the specimen of the 44 ppm hydrogen material tested at 22,000 psi was abnormally strong.

Data for as nearly comparable a heat treatment as possible is included in Figure 23 for the 142 BHN sponge heat from Reference 2. This was a water quench from 1300°F which resulted in slightly higher tensile strengths and similar ductilities. Because the results of Reference 2 work showed that tensile properties were very sensative to a degree of transformation, it would be expected that the annealed stock used for the study of hydrogen would have lower tensile strengths. Thus, the main reason for the difference between the two heats was probably heat treatment. This is borne out by the rupture times being within the range of rupture times indicated in Reference 2 for the heat made from higher hardness sponge. The deformation data were also similar.

Discussion

The results fail to show any effect of hydrogen on tensile properties at 75°F, 600°F or 1000°F for the meta-stable beta alloy 10 Mo; or for rupture and creep data from a limited number of survey tests. Rather poor and meager comparative data also indicate little or no effect from the use of sponge with a hardness of 107 in comparison to the heat of Reference 2 which was made with 146 BHN sponge.

Stable Beta Alloy 30 Mo

The influence of both hydrogen content and sponge hardness was studied for the stable beta alloy 30 Mo. Hydrogen levels of about 50 ppm were surveyed for creep-rupture properties at 1000°F on material from heats made with 107 and 140 BHN sponge titanium. Tests were conducted on the 107 BHN sponge material with 216 ppm of hydrogen. Tensile tests were also conducted at 75°F and 600°F. Data for the 140 BHN sponge heat with 300 ppm of hydrogen were available from References 1 and 2. Unfortunately, the 107 BHN sponge heat was found to have an undesirably high carbon content and only limited testing was carried out.

Experimental Materials

The compositions of the heats are given in Table 17. It should be noted that the 107 BHN sponge heat had high carbon. Samples of both heats were vacuum

annealed at 1350°F for 25 hours and furnace cooled to remove hydrogen. The 25-hour treatment in vacuum was supplemented by raising the temperature to 1500°F for 1 hour in hydrogen, furnace cooling to 1250°F for 2 hours and then furnace cooling to produce the high hydrogen condition of the 107 BHN sponge material. The hydrogen content of the specimens used for tensile tests on the low hydrogen, low hardness sponge material was somewhat higher than desired.

Results

The tensile and creep-rupture data are given in Table 26. The tensile properties (Fig. 24) were not appreciably altered by hydrogen. The low sponge hardness heat was brittle in tensile tests at room temperature at both hydrogen levels. This may have been due to its relatively high carbon content (Table 17). The high sponge hardness heat was not brittle. The heat made with high hardness sponge had appreciably higher tensile strength than that made with the low. Apparently, the interstitial elements caused a significant increase in strength.

The hydrogen level did not have much effect on rupture strength at 1000°F (Fig. 25). The high hydrogen-high sponge hardness heat had slightly higher strength. This could have been due to this material being in the as-forged condition while the others had been annealed. Ductility in rupture tests tended to be higher for the low hydrogen version of the low sponge hardness heat. The low hydrogen-high sponge hardness material tended to be less ductile than the as-forged condition with high hydrogen tested for Reference 2. Values were so erratic, however, that no definite conclusions are possible.

The creep and total deformation data at 1000°F are very difficult to interpret (Table 26 and Figs. 26 and 27). Apparently, inconsistent results were obtained. In Reference 2 where more tests were conducted on the high hydrogen-high sponge hardness heat, it was found that those were apparent instability effects which complicated creep results between 100 and 1000 hours. It is believed that similar and possibly intensified effects existed for the tests in this investigation. In general, the higher hydrogen levels seemed to have somewhat higher creep resistance. The differences between the heats with the two sponge hardness levels were not large enough to be definite. The general results seem to indicate no large effect on creep properties for either hydrogen level or the interstitial elements from varying sponge hardness.

Discussion

The only appreciable difference found was the brittleness at room temperature for the heat made with low hardness sponge. This presumably was due to high carbon. Otherwise the results are contrary to the expected effect of increased interstitial elements. The high hardness sponge heat seemed to have slightly higher tensile strength. Any differences due to hydrogen level

were very small. Apparently creep in the beta alloy is complicated at 1000°F due to an unidentified structural instability. Consequently, the survey tests to which the investigation was limited did not define creep with sufficient detail to show any small effects which might exist. Furthermore, such differences as did exist could just as well be due to differences in heat treatment as to the primary variables. The undesirably high carbon of the 107 BHN sponge heat also complicated results.

SUMMARY AND CONCLUSIONS

The experiments conducted indicate the following generalized results and conclusions:

1. Influence of Hot-Working Conditions on Creep-Rupture Properties of Titanium Alloys

Hot rolling the stable alpha alloy 6 Al in the beta range (2150°F) resulted in considerably higher creep resistance at 1000°F than did rolling in the alpha range (1400°F). Tensile and rupture properties up to 100 hours at 1000°F were not appreciably influenced. It is presumed that transformation during cooling from the beta range minimized effects arising from heating and working at 2150°F, whereas this was not true for working at 1400°F in the alpha range where cooling did not include subsequent transformation.

Hot rolling the alpha-beta alloy Ti 150A at several temperatures in the alpha-beta range (1200°F to 1650°F) did not have much effect on tensile, rupture or creep properties at 800°F. The heating temperature had more effect than the amount of reduction. Apparently, structural instability during testing at 800°F greatly reduced any effects from the conditions of hot working.

The hot-rolling experiments were limited to single pass isothermal reductions with little variation in temperature and amount of reduction. Testing conditions were also very limited. Any broad conclusions as to relationships between conditions of working and properties appear to be unwarranted.

2. Influence of Coldwork on Creep-Rupture Properties at 1000°F for Stable Alpha Alloys

A cold reduction of 20-percent increased short time strength and reduced long time strength and creep resistance at 1000°F for the stable alpha alloy 6 Al + 0.5 Si. This confirms the results of Reference 2 for damage to creep resistance at 1000°F for stable alpha alloys. The damage appeared to be due to a recovery-recrystallization mechanism and apparently 6 Al - 0.5 Si was more resistant to this than 6 Al alloy.

3. Embrittlement of Titanium Alloys During Creep Testing

A considerable number of specimens from creep tests were subjected to subsequent tensile tests at room temperature to evaluate embrittlement during creep testing by the changes in ductility. The heat treatments applied to the specimens prior to creep testing varied over wide ranges for experimental purposes. In those cases where embrittlement was observed, the use of non-standard

heat treatments may have been a contributing factor to the embrittlement and the observation of embrittlement does not necessarily preclude the possibility of producing the alloys in conditions resistant to embrittlement.

Alpha titanium Ti 75A exhibited some loss in ductility from creep tests at 600°F and 800°F when tested in the annealed condition. Cold work prevented loss in ductility.

Stable alpha alloys 6 Al and 6 Al - 0.5 Si were not embrittled.

Varying degrees of embrittlement were observed in specimens of alpha-beta alloys Ti 150A, Ti 155AX, and RC 130A depending on the alloy, the prior heat treatments and the test conditions. Heat treatments which resulted in initially high strength and low ductility from finely dispersed transformation products increased susceptibility to embrittlement. Embrittlement, presumably due to transformation during creep testing, was also increased by treatments which increased the amount of beta phase. High hydrogen levels appeared to increase embrittlement. Embrittlement increased with prior test temperature up to 800°F or 1000°F. Cases of severe embrittlement were developed during creep testing at temperatures as low as 400°F. The inter-relationships of transformation effects, hydrogen and alloy composition were not clarified by the data. It was recognized that interstitial element impurities and other metallurgical reactions such as strain aging could be contributing factors to embrittlement.

A small amount of data disclosed embrittlement of high hydrogen alpha-beta alloy Ti 150A in less than 10 hours at 800°F and in less than 50 hours at 600°F under stress. Again, heat treatment could be a factor in the results.

4. Influence of Hydrogen and Sponge Hardness (Interstitial Elements) on Creep Embrittlement and Creep-Rupture Properties of Titanium Alloys

The comprehensive investigation planned on this subject was not completed due to delays in procuring alloys and experimental difficulties. Delivery of the main experimental material (alpha-beta alloy 8 Mn) was not obtained until September and then the material was found to have such a slow transformation rate and to be so resistant to hydrogen removal that it is doubtful that the types of microstructures required for the objectives of the investigation could have been produced.

No large effect of hydrogen or sponge hardness on tensile or creep-rupture properties up to 1000°F was observed for hydrogen levels in the range of approximately 50 to 250 ppm for alloy made with titanium sponge of 107 or approximately 145 BHN. This observation was based on very limited data for stable alpha alloy 6 Al, meta-stable beta alloy 10 Mo and stable beta alloy 30 Mo. There may have been a slight increase in creep resistance due to reduction of hydrogen. The effect, if any, was smaller than both normal heat-to-heat differences and to normal response to a given heat treatment.

Embrittlement of alpha-beta alloy Ti 150A under stress at 600°F to 1000°F was found to increase with hydrogen content in the range of 60 to 274 ppm. Severe embrittlement occurred in 50 hours at 600°F at the high hydrogen level. Increasing stress was found to slightly increase rate of embrittlement. The 50 ppm stock was not severely embrittled although the ductility was considerably reduced in 100 hours at 800°F to 900°F. It was not established whether this decrease in ductility with the 50 ppm hydrogen was due to residual hydrogen or to beta transformation effects. Environmental effects such as the activation of stress-corrosion type embrittlement during creep were not considered in the experiments. The stressed specimens for this investigation were exposed in an air atmosphere free from such effects insofar as is known.

REFERENCES

1. Gluck, J. V. and Freeman, J. W., "Intermediate Temperature Creep and Rupture Behavior of Titanium and Titanium-Base Alloys", WADC Technical Report 54-112, September 1954.

2. Gluck, J. V. and Freeman, J. W., "Intermediate Temperature Creep and Rupture Behavior of Titanium Base Alloys, Part II: Influence of Microstructure in Creep-Rupture Properties". Report submitted to WADC, September 1957.

3. Gluck, J. V. and Freeman, J. W., "A Study of Creep of Titanium and Two of Its Alloys", WADC Technical Report 54-54, March 1956.

4. Lenning, G. A., Craighead, C. M., and Jaffee, R. I., "First Summary Report on the Effect of Hydrogen in Mechanical Properties of Titanium", Battelle Memorial Institute Report to Watertown Arsenal, Ordnance Dept., Dept. of the Army, Contract No. DA33-019-ORD-220, Project No. TB4-15, July 31, 1952.

TABLE 1

TENSILE DATA FOR HOT-ROLLED STABLE ALPHA ALLOY 6 Al

Hot Rolling Temp. (°F)	Hot Reduction (%)	Test Temp. (°F)	Tensile Strength (psi)	0.2% Offset Yield Strength (psi)	Elongation (% in 1 in)	Reduction of Area (%)
As Forged	0	75	131,000	-	17.6	40.2
		1000	74,000	-	15.0	50.7
1400°F	0	75	125,800	119,000	10.3	28.6
		1000	80,600	67,400	14.3	47.6
	12	75	133,600	130,000	15.3	47.3
		1000	77,400	64,600	24.8	67.0
	24	75	136,500	132,000	13.1	44.6
		1000	73,500	62,100	19.2	51.6
2150°F	0	75	126,300	115,000	15.0	29.3
		1000	67,300	54,000	12.6	57.8
	13	75	128,200	115,000	16.3	33.1
		1000	74,000	57,300	18.2	51.4
	37	75	128,200	117,000	18.8	34.0
		1000	69,600	55,200	16.3	47.6

TABLE 2

CREEP-RUPTURE PROPERTIES AT 1000°F FOR HOT-ROLLED STABLE ALPHA ALLOY 6 Al

Hot Rolling Temp. (°F)	Hot Reduction (%)	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in)	Reduction of Area (%)	Loading Deformation (%)	Time to Reach Specified Deformation (hours)					Minimum Creep Rate (in/in/hr)	100-hr Rupture Strength (psi)
							0.5%	1.0%	3.0%	5%	Special (c)		
As forged	0	74,100T	-	15.0	50.7	-	-	-	-	-	-	-	-
		37,000	57.5	21.9	30.5	.42	-	-	-	-	-	-	-
		35,000	156.8	36.2	57.8	.18	4	10		62	-	5.8×10^{-4}	-
		15,000	>1171.0	-	-	.15	30	140	1000	-	-	2.2×10^{-5}	36,000
1400°F	0	80,600T	67,400Y	14.3	47.6	-	-	-	-	-	-	-	-
		36,000	121.7	22.1	51.6	.42	-	7	-	49	-	9.8×10^{-4}	-
		28,000	1025.2	39.0	39.1	.27	-	50	200	380	-	1.1×10^{-4}	37,000
	12	77,400T	64,600Y	24.8	67.0	-	-	-	-	-	-	-	-
		35,000	63.9	38.1	62.1	.52	-	3	15	-	-	2.1×10^{-3}	-
		32,500	86±10	62.1	76.8	.51	-	2	9	-	-	2.2×10^{-3}	-
		15,000	>1121	-	-	.16	30	140	775	-	-	2.8×10^{-5}	32,000
	24	73,500T	62,100Y	19.2	51.6	-	-	-	-	-	-	-	-
		35,000	75.6	67.5	64.0	.31	-	-	-	-	-	-	-
		32,000	106±10	65.0	59.0	.32	1	4	-	28	(8%) 42	1.6×10^{-3}	-
		15,000	>1001	-	-	.12	15	65	-	555	(8%)1000	6.9×10^{-5}	32,000
2150°F	0	67,300T	54,000Y	12.6	57.8	-	-	-	-	-	-	-	-
		38,000	141.6	18.5	24.7	.59	-	7	-	69	-	5.1×10^{-4}	-
		35,000	281.5	32.0	30.1	.34	-	5	-	87	-	2.4×10^{-4}	-
		15,000	>1121	-	-	.13	-	-	-	-	(.3%)800	8.3×10^{-7}	39,000
	13	74,000T	57,300Y	18.2	51.4	-	-	-	-	-	-	-	-
		39,000	114.0	15.8	30.1	.39	-	8	-	52	-	7.7×10^{-4}	-
		35,000	255.0	20.4	31.3	.33	-	8	-	142	-	2.5×10^{-4}	-
		15,000	>1025.0	-	-	.18	-	-	-	-	(.3%)125 (.4%)550 (.46%)1000	1.4×10^{-6}	39,000
	37	69,600T	55,200Y	16.3	47.6	-	-	-	-	-	-	-	-
		38,500	60.4	12.0	16.9	.43	-	-	-	-	-	-	-
		35,000	310.2	22.5	33.4	.50	-	18	-	60	-	1.4×10^{-4}	-
		15,000	>1025.0	-	-	.15	-	-	-	-	(.3%)175 (.4%)650 (.43%)1000	1.3×10^{-6}	37,500

> Test stopped without rupture
c Specified deformation in parentheses
T Tensile Strength
Y Yield Strength

TABLE 3

TENSILE DATA AT 800°F FOR HOT-ROLLED ALPHA-BETA ALLOY Ti 150A

Hot Rolling Temp. (°F)	Hot Reduction (%)	Test Temp. (°F)	Tensile Strength (psi)	Yield Strength 0.2% Offset (psi)	Elongation (% in 1 in)	Reduction of Area (%)
1200	14.9	800	79,700	54,500	33.3	74.7
	18.1	800	82,800	61,200	29.8	70.2
	30.7	800	86,300	60,200	30.9	74.5
1350	14.4	800	84,400	55,000	35.4	76.2
	17.7	800	87,600	59,300	28.2	64.0
	29.3	800	93,800	62,900	34.0	75.2
1500	15.8	800	83,200	51,600	35.3	81.1
	20.4	800	87,700	55,000	43.6	82.1
	34.8	800	93,700	60,000	30.8	79.2
1650	19.1	800	75,100	48,600	34.0	76.2
	25.6	800	77,400	53,400	34.0	76.9
	37.8	800	81,500	52,700	33.0	75.3

TABLE 4

CREEP-RUPTURE DATA FOR HOT-ROLLED ALPHA-BETA ALLOY Ti 150A

Hot Rolling Temp. (°F)	Hot Reduction (%)	Test Temp. (°F)	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in.)	Reduction of Area (%)	Loading Deformation (%)	Time to Reach Specified Deformation (hours)			Minimum Creep Rate (in/in/hr)	100-hr Rupture Strength (psi)	Stress for Minimum Creep Rate of 10^{-5} (in/in/hr)
								1%	5%	10%			
1200	0	800	42,000	300.1	61.0	76.9	.45	7	75	170	5.5×10^{-4}	47,000	-
	14.9	800	79,700	Tensile Test	33.3	74.7	-	-	-	-	-	-	-
			43,000	155.6	46.0	78.0	.45	15	95	125	3.3×10^{-4}	-	-
			40,000	296.8	42.0	79.5	.40	5	50	140	4.7×10^{-4}	-	-
			35,000	772+6	48.6	82.5	.35	7	130	480	1.3×10^{-4}	-	-
			30,000	>1131.0	-	-	.28	17	730	-	3.2×10^{-5}	45,000	26,000
	18.1	800	82,800	Tensile Test	29.8	70.2	-	-	-	-	-	-	-
			47,000	141.6	66.5	77.0	.60	3	30	70	1.1×10^{-3}	-	-
			44,000	264.5	55.9	75.4	.40	7	55	130	5.0×10^{-4}	-	-
			30,000	>1013.0	-	-	.35	20	~800	-	2.2×10^{-5}	48,000	28,000
1350	0	800	50,000	138.1	42.0	79.0	.55	3	15	60	8.6×10^{-4}	51,000	-
	14.4	800	84,400	Tensile Test	35.4	76.2	-	-	-	-	-	-	-
			45,500	74.7	45.6	82.3	-	-	-	-	-	-	-
			44,500	175.5	48.0	80.8	.45	2	30	85	8.6×10^{-4}	-	-
			35,000	>1057.4	-	-	.37	10	230	-	4.9×10^{-5}	-	-
			30,000	>1147.0	-	-	.28	40	-	-	9.5×10^{-6}	45,000	30,000
	17.7	800	87,600	Tensile Test	28.2	64.0	-	-	-	-	-	-	-
			48,000	98.5	59.5	79.5	.60	5	20	-	2.9×10^{-3}	-	-
			35,000	> 810.0	-	-	.39	12	280	-	6.2×10^{-5}	-	-
			30,000	>1011.8	-	-	.36	20	~900	-	1.8×10^{-5}	48,000	28,000
	29.3	800	93,800	Tensile Test	34.0	75.2	-	-	-	-	-	-	-
			53,500	73.6	35.4	76.2	.55	2	18	40	2.6×10^{-3}	-	-
			51,000	177.8	31.0	76.4	.50	3	37	105	5.9×10^{-4}	52,000	35,000
1500	0	800	52,000	193.1	41.0	79.0	-	-	-	-	-	54,000	-
	15.8	800	83,200	Tensile Test	35.3	81.1	-	-	-	-	-	-	-
			48,000	188.8	35.3	80.9	.60	4	50	120	4.9×10^{-4}	-	-
			45,000	239.0	34.6	80.8	.50	5	90	175	4.8×10^{-4}	-	-
			40,000	990.2	50.5	84.4	.55	7	220	-	8.1×10^{-5}	-	-
			30,000	>1146.0	-	-	.29	65	-	-	6.7×10^{-6}	49,000	32,000
	20.4	800	87,700	Tensile Test	43.6	82.1	-	-	-	-	-	-	-
			49,500	143.6	48.0	79.4	.70	2	30	90	7.2×10^{-4}	-	-
			47,500	175.9	40.0	82.1	.60	4	50	-	6.1×10^{-4}	-	-
			35,000	>1197.9	-	-	.39	20	1100	-	1.5×10^{-5}	-	-
			30,000	>1012.0	-	-	.33	25	-	-	9.5×10^{-6}	50,000	32,000
	34.8	800	93,700	Tensile Test	30.8	79.2	-	-	-	-	-	-	-
			55,500	147.7	39.6	76.7	.80	2	45	-	6.1×10^{-4}	-	-
			51,000	383.5	45.2	78.9	.65	4	85	255	2.9×10^{-4}	-	-
			35,000	>1057.6	-	-	.42	10	-	-	1.1×10^{-5}	56,000	35,000
1650	0	800	45,000	296.3	36.0	78.0	.35	7	115	235	-	50,000	-
	19.1	800	75,100	Tensile Test	34.0	76.2	-	-	-	-	-	-	-
			46,000	211.3	66.4	79.7	.70	2	70	125	7.4×10^{-4}	-	-
			41,000	535.5	37.4	78.2	.50	35	410	515	2.4×10^{-4}	-	-
			35,000	>1057.3	-	-	.44	15	~800	-	3.2×10^{-5}	-	-
			30,000	>1940.6	-	-	.34	40	-	-	5.3×10^{-6}	50,000	32,000
	25.6	800	77,400	Tensile Test	34.0	76.9	-	-	-	-	-	-	-
			47,000	199.7	38.8	76.9	.75	1	20	125	2.5×10^{-3}	-	-
			42,000	519.0	33.0	77.5	.55	5	150	405	2.0×10^{-4}	-	-
			40,000	743.9	48.6	81.5	.53	10	250	555	1.3×10^{-4}	-	-
			25,000	>1941.0	-	-	.32	130	-	-	2.5×10^{-6}	50,000	30,000
	37.8	800	81,500	Tensile Test	33.0	75.3	-	-	-	-	-	-	-
			51,000	153.6	39.6	80.2	.70	5	50	90	5.3×10^{-4}	-	-
			46,000	460.2	35.9	78.6	.60	8	135	355	2.4×10^{-4}	-	-
			30,000	>1438.1	-	-	.34	35	-	-	5.6×10^{-6}	53,000	32,000

> Test stopped without rupture

TABLE 5

CREEP-RUPTURE DATA AT 1000°F FOR STABLE ALPHA ALLOY 6 Al - 0.5 Si

Stress (psi)	Test Time (hr.)	Elongation (% in 1 in.)	Red. of Area (%)	Loading Deformation (%)	Time to Reach Indicated Total Deformation (hours) 0.5%	1.0%	5%	Other	Minimum Creep Rate (in/in/hr)
				As-Forged					
74,800 T	(60700Y)	11.7	22.4	--	--	--	--	--	--
50,000	41.1	14.3	31.4	0.47	0.2	4	--	--	1.4×10^{-3}
45,000	97.8	22.0	22.2	.39	2	5	40	--	8.3×10^{-4}
36,000	473.9	15.0	26.9	.34	4	15	330	--	1.2×10^{-4}
32,000	526.4	15.0	22.0	.37	10	45	365	--	1.0×10^{-4}
30,000	>986.1	--	--	.27	15	105	1000	--	2.8×10^{-5}
24,000	>1581.4	--	--	.22	180	--	--	(0.7%) 1400	1.2×10^{-6}
20,000	>1084.	--	--	.19	1030	--	--	--	1.3×10^{-6}
				Cold Reduced 20 Percent					
92,400 T	(82300Y)	11.7	34.9	--	--	--	--	--	--
52,000	48.4	29.1	49.5	0.55	--	14	--	--	3.3×10^{-4}
44,000	126.0	35.6	53.6	.48	--	5	46	--	7.7×10^{-4}
44,000	145.6	33.0	32.2	.47	--	--	--	--	--
32,000	393.4	30.0	46.4	.33	4	16	180	--	2.1×10^{-4}
25,000	1472.0	22.0	42.7	.27	8	35	800	--	4.0×10^{-5}
20,000	>1034.7	--	--	.20	30	450	--	(1.36%) 1000	7.6×10^{-6}
13,000	>1104.3	--	--	.12	690	--	--	(0.55%) 1104	1.8×10^{-6}

T = Tensile Strength
Y = Yield Strength
> = Time Test Was Stopped.

TABLE 6

CHEMICAL COMPOSITION OF ALLOYS SUBJECTED TO TENSILE TESTS AT 75°F AFTER CREEP TESTING

Heat Number	Supplier	Sponge Hardness BHN	Fe (%)	Cr (%)	Mo (%)	Al (%)	C (%)	N_2 (%)	H_2 (%)	O_2 (%)
			Alpha Titanium Ti 75A							
L984	TMC	--	0.19	--	--	--	0.025	0.061	0.0087	--
M626	TMC	--	0.14	--	--	--	0.042	0.026	0.0150	--
			Stable Alpha Alloy 6 Al							
*4	ARF	146	--	--	--	6.21	--	--	0.017	0.21
*4a	ARF	124	--	--	--	5.89	0.075	0.055	0.015	0.097
			Stable Alpha Alloy 6 Al - 0.5 Si							
7011	ARF	124	--	--	--	6.51	--	0.017	0.017	-- (Si - 0.48)
			Alpha-Beta Alloy Ti 150A							
M739	TMC	--	1.47	2.76	--	--	0.08	0.026	0.031	--
L1006	TMC	--	1.52	2.68	--	--	0.046	0.124	--	--
L774	TMC	--	1.55	2.72	--	--	0.055	0.086	--	--
X672	TMC	--	1.42	2.72	--	--	0.051	0.049	0.052	0.45 (Si - 0.092, W-0.27)
			Alpha-Beta Alloy Ti 155AX							
M400R	TMC	--	1.45	1.28	1.16	5.11	0.037	0.017	0.018	--
			Alpha-Beta Alloy RC130A							
A5036	RC	--	--	--	--	--	0.10	0.018	0.0083	-- (Mn - 6.9)
			Meta-Stable Beta Alloy 10 Mo							
*5	ARF	146	--	--	10.50	--	0.05	0.022	0.013	--
			Stable Beta Alloy 30 Mo							
*6	ARF	--	--	--	29.56	--	--	--	0.030	0.017

TMC Titanium Metallurgical Corporation
ARF Armour Research Foundation
RC Rem-Cru Titanium Corporation
* U Identification

TABLE 7

INFLUENCE OF CREEP TESTING ON THE TENSILE PROPERTIES AT 75°F OF ALPHA TITANIUM ALLOY Ti 75A

Heat No.	Heat Treatment	Creep-Test Conditions			Tensile Properties at 75°F			
		Temp. (°F)	Stress (psi)	Time (hr)	Tensile Strength (psi)	Yield Strength 0.2% offset (psi)	Elongation (% in 1 in.)	Red. of Area (%)
L-984	As Received		As treated		90,700	67,500	30.2	50.4
		600	35,000	911	96,800	89,200	21.7	47.0
L-984	1700°F Anneal		As treated		87,600	65,600	34.0	44.6
		800	6,000	1178	89,300	71,000	27.5	45.8
L-984	1500°F Anneal		As treated		87,900	68,400	30.4	48.6
		1000	2,500	1126	86,000	65,000	16.3	46.6
M-626	1500°F Anneal		As treated		88,600		26.5	44.3
		600	33,000	1322	104,800		12.5	30.9
L-984	1500°F Anneal + 10% Cold Reduction		As treated		115,000	102,000	12.7	43.5
		800	12,000	1052	94,900	83,000	21.8	44.6
L-984	1700°F Anneal + 10% Cold Reduction		As treated		109,000	99,400	10.7	32.5
		600	40,000	1026	100,900	89,300	22.2	42.9
M-626	1500°F Anneal + 31% Cold Reduction		As treated		130,500		15.3	40.8
		600	44,000	1252	116,700		21.0	36.7
L-984	1700°F Anneal + 31.3% Cold Reduction		As treated		127,000	114,000	11.5	34.1
		800	15,000	1188	96,700	79,300	24.0	47.8

TABLE 8

INFLUENCE OF CREEP TESTING ON THE TENSILE PROPERTIES AT 75°F OF STABLE ALPHA ALLOY 6 Al

Heat No.	Heat Treatment	Creep-Test Conditions			Tensile Properties at 75°F			
		Temp. (°F)	Stress (psi)	Time (hr.)	Tensile Strength (psi)	Yield Strength 0.2% offset (psi)	Elongation (% in 1 in.)	Red. of Area (%)
4	As forged	As-treated			146,000	135,000	18.8	34.1
		600	87,000	3001	162,000	159,000	17.1	36.7
4	17% Cold Work	As-treated			177,200	163,800	10.7	28.0
		600	102,000	955	170,000	168,000	8.8	27.4
4	17% Cold Work	As-treated			177,200	163,800	10.7	28.0
		1000	15,000	1337	159,000	152,000	10.0	39.8
4	17% Cold Reduction + 1500°F Anneal	As-treated			137,000	129,800	15.5	20.7
		600	84,000	3167	153,800	151,300	14.9	31.4
4	17% Cold Reduction + 1500°F Anneal	As-treated			137,000	129,800	15.5	20.7
		1000	17,000	3000	140,400	135,700	9.6	11.2
4	17% Cold Reduction + 1700°F Anneal	As-treated			135,000	129,000	13.0	34.6
		600	79,500	1556	147,600	144,000	17.0	37.3
4a	12% Cold Reduction	As-treated			154,000		11.0	30.1
		1000	17,000	913	143,300		11.9	42.0
		1000	12,500	1000	139,600		16.0	34.3
		1000	10,000	1102	146,800		18.0	36.1

TABLE 9

INFLUENCE OF CREEP TESTING ON THE TENSILE PROPERTIES AT 75°F FOR FOUR ALLOYS

Treatment	Creep-Test Conditions			Tensile Properties at 75°F		
	Temp. (°F)	Stress (psi)	Time (hr.)	Tensile Strength (psi)	Elongation (% in 1 in.)	Red. of Area (%)
Stable Alpha Alloy 6 Al-0.5 Si						
19% Cold Reduction		as-treated		178,800	8.2	24.2
	600	106,000	962	179,000	6.8	22.6
Alpha-Beta Alloy Ti 155AX						
1700°F Furnace Cool		as-treated		149,000	22.4	43.4
	600	100,000	983	175,000	10.2	21.5
Meta-Stable Beta Alloy 10 Mo						
1800°F Water Quench		as-treated		107,200	48.0	80.6
	600	98,000	962	(Fractured in threads at 134,000 psi)		
1800°F + isothermal transformation at 1200°F for 1 hour, water quenched		as-treated		109,600	34.0	44.4
	600	88,500	938	(Fractured in threads at 149,000 psi)		
Stable Beta Alloy 30 Mo						
As-Forged		as-treated		144,700	14.0	27.4
	1000	20,000	912	(Fractured in threads at 101,800 psi)		
1325°F Water Quench		as-treated		145,800	12.6	28.0
	600	102,500	983	162,500	5.6	0

TABLE 10

INFLUENCE OF CREEP TESTING ON TENSILE PROPERTIES
AT 75°F OF ALPHA-BETA ALLOY Ti 150 A

Creep-Test Conditions			Tensile Properties at 75°F			
Temp. (°F)	Stress (psi)	Time (hrs.)	Tensile Strength (psi)	Yield Strength 0.2% offset (psi)	Elongation (% in 1 in.)	Red. of Area (%)
Heat M739 -- 1500° for 1 Hour, Furnace Cool						
	as-treated		138,500	130,600	25.5	42.8
800	23,000	1146	193,000	---	3.7	3.9
1000	5,000	1028	131,500	112,000	7.1	7.1
Heat M739 -- 1500°F for 1 Hour, Air Cool						
	as-treated		153,000	135,600	11.0	13.2
600	95,000	1133	(Fractured in threads at 71,500 psi)			
800	35,000	1099	(Fractured in threads at 48,000 psi)			
1000	5,000	1028	141,000	119,500	7.2	8.8
Heat M739 -- 1800°F for 1 Hour, Isothermally Transformed at 1300°F for 1 Hour, Water Quenched						
	as-treated		156,500	153,500	16.0	21.2
800	35,000	1099	(Fractured in threads at 49,000 psi)			
1000	5,000	1099	90,600	---	0	0
Heat L-1006 -- 1350°F for 1 Hour, Furnace Cool						
	as-treated		156,200	154,100	25.7	43.5
600	83,000	1173	206,500	206,000	0	0
Heat L-1006 -- 1500°F for 1 Hour, Furnace Cool						
	as-treated		153,800	147,600	26.7	35.2
800	29,000	1535	(Broke in threads at 100,500 psi)			
1000	5,000	860	(Broke in threads at 104,400 psi)			

TABLE 10 (Continued)

INFLUENCE OF CREEP TESTING ON TENSILE PROPERTIES AT 75°F OF ALPHA-BETA ALLOY Ti 150 A

Creep-Test Conditions			Tensile Properties at 75°F			
Temp. (°F)	Stress (psi)	Time (hrs.)	Tensile Strength (psi)	Yield Strength 0.2% offset (psi)	Elongation (% in 1 in.)	Red. of Area (%)
		Heat L-1006 -- 1350°F for 1 Hour, Water Quench				
	as-treated		170,100	159,900	18.2	39.2
600	116,000	1052	(Broke in threads at 126,200 psi)			
		Heat L-1006 -- 1500°F for 1 Hour, Air Cool				
	as-treated		177,200	162,500	16.7	32.0
800	35,000	1190	(Broke in threads at 73,500 psi)			
1000	5,000	978	(Broke in threads at 140,800 psi)			
		Heat L-1006 -- 1500°F for 1 Hour, Water Quench				
	as-treated		222,000	220,000	3.9	3.5
800	41,000	811	(Broke in threads at 58,600 psi)			
1000	5,000	1035	(Broke in threads at 71,900 psi)			

TABLE 11

INFLUENCE OF CREEP TESTING AT LOW TEMPERATURES ON THE TENSILE PROPERTIES AT 75°F OF ALPHA-BETA ALLOY Ti 150A

(Specimens from Creep Tests of Reference 3 -- Heat L774 bar stock)

Treatment	Creep-Test Conditions			Tensile Properties at 75°F		
	Temp. (°F)	Stress (psi)	Time (hr.)	Tensile Strength (psi)	Elongation (% in 1 in.)	Red. of Area (%)
As-Produced	As Received (manufacturer)			156,000	29.0	54.8
	76	65,000	2016	155,300	21.5	37.0
		80,000	1632	156,100	17.9	15.4
	210	60,000	1462	156,000	18.2	44.8
		80,000	1250	156,000	13.7	11.8
	400	55,000	1030	168,000	1.9	4.3
		72,000	1120	158,300	----	----
	600	55,000	1120	177,000	1.6	0
		62,000	1120	Broke in threads at 181,000		
1500°F Water Quench	As Treated			222,000	3.9	3.5
	76	110,000	1465	230,000	<1.0	<1.0
	400	70,000	1000	232,000	--very brittle--	
		90,000	1000	216,000	--very brittle--	
1500°F Air Cool	As Treated			177,200	16.7	32.0
	76	90,000	1945	168,000	10.5	31.5
		100,000	1490	168,000	10.6	15.5

TABLE 11 (Continued)

INFLUENCE OF CREEP TESTING AT LOW TEMPERATURES ON THE TENSILE PROPERTIES AT 75°F OF ALPHA-BETA ALLOY Ti 150 A

(Specimens from Creep Tests of Reference 3 -- Heat L774 bar stock)

Treatment	Creep-Test Conditions			Tensile Properties at 75°F		
	Temp. (°F)	Stress (psi)	Time (hr.)	Tensile Strength (psi)	Elongation (% in 1 in.)	Red. of Area (%)
1500°F Air Cool	210	80,000	1140	167,000	9.2	13.9
		98,000	1120	169,800	13.1	46.0
	400	80,000	1120	169,700	3.5	11.6
		97,000	1120	173,000	7.1	14.7
	600	80,000	1120	187,000	3.6	0
		93,000	1120	179,100	1.1	1.0

TABLE 12

INFLUENCE OF CREEP TESTING AT LOW TEMPERATURES ON THE TENSILE PROPERTIES AT 75°F OF ALPHA-BETA ALLOY Ti 150A IN SHEET FORM

(Specimens from Creep Tests of Reference 3 -- Heat X672, 0.064 inch thick sheet)

Treatment	Creep-Test Conditions			Tensile Properties at 75°F	
	Temp. (°F)	Stress (psi)	Time (hr.)	Tensile Strength (psi)	Elongation (% in 1 in.)
As Produced	As-received (U. of M.)			147,000	13.0
	As-received (Manufacturer)			140,000	15.0
	210	40,000	1175	145,000	9.7
		54,000	1175	143,200	9.3
	400	55,000	not available	152,200	7.3
1300°F, Anneal	76	70,000	1000	140,600	13.7
		75,000	1000	143,800	9.7
1300°F, Air Cool	76	80,000	1370	156,100	9.7
		92,500	1350	159,000	8.6
1300°F, Water Quench	76	80,000	1340	156,000	10.3
		96,000	1195	162,800	10.9
1500°F Water Quench + 1300°F, 1 Hour, Furnace Cool	76	65,000	1000	142,100	5.1
1400°F Water Quench	76	70,000	1500	151,000	4.5
1500°F, Air Cool	76	110,000	1340	171,000	3.0
		100,000	1000	169,600	1.3

TABLE 13

INFLUENCE OF CREEP TESTING AT LOW TEMPERATURES ON THE TENSILE PROPERTIES AT 75°F OF ALPHA-BETA ALLOY RC 130A IN SHEET FORM

(Specimens from Creep Tests of Reference 3 -- Heat A5036, 0.064 inch thick sheet)

Treatment	Creep-Test Conditions			Tensile Properties at 75°F	
	Temp. (°F)	Stress (psi)	Time (hr.)	Tensile Strength (psi)	Elongation (% in 1 in.)
As Produced	As Produced			129,300	21.5
	210	72,000	1300	128,000	20.0
		90,000	1250	133,000	19.0
	400	65,000	1010	132,000	20.5
		65,000	1170	131,800	21.5
		80,000	1170	135,000	21.6
800°F, Air Cool	As Treated			129,000	25.0
	76	80,000	1270	133,000	19.0
		105,000	1360	134,000	18.0
	210	90,000	1000	132,800	19.5
	400	75,000	1010	135,000	17.5
1100°F, Air Cool	As Treated			130,000	20.0
	76	95,000	1470	132,200	21.0
	210	80,000	1195	129,000	21.0
1250°F, Air Cool	As Treated			135,000	12.0
	76	75,000	1445	138,000	17.5
	210	75,000	1030	140,100	18.0
	400	75,000	1010	144,700	12.5
		85,000	1000	139,000	4.0

TABLE 13 (Continued)

INFLUENCE OF CREEP TESTING AT LOW TEMPERATURES ON THE TENSILE PROPERTIES AT 75°F OF ALPHA-BETA ALLOY RC 130A IN SHEET FORM

(Specimens from Creep Tests of Reference 3 -- Heat A5036, 0.064 inch thick sheet)

Treatment	Creep-Test Conditions			Tensile Properties at 75°F	
	Temp. (°F)	Stress (psi)	Time (hr.)	Tensile Strength (psi)	Elongation (% in 1 in.)
1400°F, Water Quench	As Treated			168,200	5.5
	400	110,000	810	188,900	3.5
1625°F, Anneal	As Treated			118,400	4.0
	210	70,000	1025	126,600	9.0
1625°F, Water Quench	As Treated			167,050	1.5
	76	105,000	1250	(shoulder failure at 144,000 psi)	
	210	80,000	955	(shoulder failure at 118,100 psi)	

TABLE 14

EMBRITTLEMENT TIME FOR ALPHA-BETA ALLOY Ti 150A DURING EXPOSURE TO STRESS AT 600° AND 800°F

Heat Treatment	Temp. (°F)	Stress (psi)	Time (hrs)	Tensile Strength (psi)	Yield Strength 0.2% Offset (psi)	Elongation (% in 1 in)	Reduction of Area (%)
1350°F (1 hr) + Furnace Cool	As Treated			156,200	154,100	25.7	43.5
	600	83,000	50	180,300	178,500	4.5	21.8
	600	83,000	100	181,200	--	1.0	1.7
	600	83,000	300	189,000	187,800	1.9	1.8
	600	83,000	1173	206,500	206,000	0	0
1500°F (1 hr) + Air Cool	As Treated			177,200	162,500	16.7	32.0
	800	35,000	20	Broke in threads at 112,900 psi			
	800	35,000	50	Broke in threads at 54,400 psi			
	800	35,000	100	Broke in threads at 74,400 psi			
	800	35,000	1190	Broke in threads at 73,500 psi			

TABLE 15

EFFECT OF VACUUM ANNEAL ON EMBRITTLEMENT OF ALPHA-BETA ALLOY Ti 150A

Exposure Conditions	Tensile Properties at 75°F after Exposure			
	Tensile Strength (psi)	Yield Strength 0.2% offset (psi)	Elongation (% in 1 in.)	Reduction of Area (%)
1350°F for 1 hour and furnace cooled				
As-treated	157,000	151,000	14.0	19.6
1350°F for 1 hour, furnace cooled + 24 hours in vacuum at 1200°F				
As-treated	155,000	150,000	22.0	37.8
600°F - 100 hours in vacuum	154,300	147,900	25.0	33.2
600°F - 100 hours in air	156,100	150,500	23.0	31.4
600°F - 83,000 psi	(Fractured at 600°F in 1.8 hours with 27.6% elongation and 58.6% reduction of area)			
1350°F for 1 hour, furnace cooled + 24 hours in argon at 1200°F				
As-treated	154,000	146,000	20.9	32.3
600°F - 100 hours in vacuum	156,000	145,500	13.0	14.6
600°F - 100 hours in air	157,000	152,000	10.2	13.2
600°F - 100 hours - 83,000 psi	179,000	175,000	4.5	5.9

TABLE 16

EFFECT OF VACUUM ANNEALING ON CREEP-RUPTURE AND TENSILE PROPERTIES OF STABLE ALPHA ALLOY 6 Al

Tensile Data

Treatment	Test Temp. (°F)	Tensile Strength (psi)	Yield Strength (psi)	Elongation (% in 1 in)	Reduction of Area (%)
Argon Annealed (24 hr 1200°F + furnace cool)	1000	73,800	58,700	15.0	57.4
Vacuum Annealed (24 hr 1200°F + furnace cool)	1000	74,200	59,000	17.4	57.1
As Forged	1000	73,800	59,200	13.0	54.0

Creep-Rupture Data

Treatment	Test Temp. (°F)	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in)	Red. of Area (%)	Loading Deformation (%)	Time to Reach Specified Deformation (hours) 0.5%	1.0%	5.0%	Minimum Creep Rate (in/in/hr)
Argon Annealed (24 hr 1200°F + furnace cool)	1000	39,000	32.3	64.4	73.3	.53	-	-	-	-
		35,000	123.9	34.9	34.8	.34	2	8	0	-
		30,000	279.0	38.0	75.0	.17	6	16	118	4.1×10^{-4}
		30,000	301.6	54.8	68.9	.29	3	10	117	2.5×10^{-4}
		27,000	597.0	55.4	68.6	.27	10	30	214	2.0×10^{-4}
Vacuum Annealed (24 hr 1200°F + furnace cool)	1000	39,000	26.1	10.8	26.7	.32	-	-	-	-
		35,000	69.6	14.3	42.0	.28	-	-	-	-
		30,000	351.0	23.8	45.7	.44	4	20	183	2.1×10^{-4}
		30,000	450.5	50.5	73.8	.36	4	17	170	2.1×10^{-4}
		27,000	494.5	29.1	79.5	.29	13	22	190	2.1×10^{-4}

TABLE 17

CHEMICAL COMPOSITION OF EXPERIMENTAL MATERIALS

(Producer's Reported Composition Except for Hydrogen)

Alloy	Heat	Sponge Hardness (BHN)	Major Alloying Elements	C (%)	N_2 (%)	H_2 (%)
6 Al	4c	107	Al - 6.13%	0.044	0.045	0.0084
6 Al	4	146	Al - 6.21%	---	---	0.017
6 Al	4a	124	Al - 5.89%	0.075	0.055	0.015
Ti 150A	M739	(na)	Cr - 2.76%; Fe - 1.47%	.08	.026	.0310*
8 Mn	3a	107	Mn - 8.85%	.063	.015	.0114
8 Mn	3b	148	Mn - 8.62%	.109	.051	(na)
10 Mo	9038	107	Mo - 10.47%	.051	.034	.0033
30 Mo	6	~140	Mo - 29.58%	(na)	(na)	.0300*
30 Mo	6a	107	Mo - 30.50%	.59	.020	.0097
Sponge Composition		107	Si - <0.02%; Fe - 0.012%	.045	.010	--
Sponge Composition		148	Si - 0.02%; Fe - 0.24%	.054	.045	--

* Hydrogen analysis by Materials Laboratory, WADC; other hydrogen analysis by University of Michigan

(na) Not available

TABLE 18

HYDROGEN CONTENTS AFTER ANNEALING IN VACUUM

(Continuously Pumped Vacuum of Less than 1 Micron)

Alloy	Heat	Run	Vacuum Conditions		Original H_2 (ppm)	Analyzed H_2 (ppm) After Vacuum Anneal		
			Temp. (°F)	Time (hr.)		1st	2nd	Average
6 Al	4c	10	1350	26	84	34	34	34
6 Al	4c	11	1350	25	84	53	49	51
Ti 150A	M729	4	1350	26	310	63	64	64
Ti 150A	M729	5	1350	26	310	57	57	57
8 Mn	3a	19	1320	25	114	110	106	108
8 Mn	3a	19a	1365	24	114	112	113	112
8 Mn	3a	24	1500	26	114	84	83	84
10 Mo	5a	6	1350	23.3	33	43	44	44
10 Mo	5a	7	1350	23.3	33	44	42	43
30 Mo	6	3	1350	26	300	45	43	44
30 Mo	6a	16	1350	24	97	112	113	112
30 Mo	6a	17	1350	24	97	49	50	50
30 Mo	6a	22	1360	25	97	74	71	73

Note: Results of Run 16 on 30 Mo are not understood. H_2 content of Heat 5a of 10 Mo was probably not properly established and 33 ppm represents a low H_2 sample from the heat.

TABLE 19

HYDROGEN ADDITION RESULTS

Alloy	Heat	Run	Exposure Time to H_2 at 1500°F (hrs.)	Aim H_2 (ppm)	Analyzed H_2 (ppm) Determination 1st	2nd	Average	Location of Analysis Specimen for Ti 75 A Calibration Run
Ti 75A	(Calibration)		1.25	250	214	222	218	Middle of basket -- Middle of bar
					216	222	219	Middle of basket -- End of bar
					203	207	205	Edge of basket -- Middle of bar
					222	203	213	Edge of basket -- Top of bar
							214	Average from calibration run
Ti 150A	M729	14	2	175-200	161	162	162[a]	
Ti 150A	M729	15	4.3	250	274	---	274	
8 Mn	3a	23	7	500	573	602	587	
10 Mo	5a	12	1.16	250	286	234	260	
10 Mo	5a	13	1.0	250	188, 194	218, 266	216-242[b]	
30 Mo	6a	18	1.0	250	213	218	216[a]	
30 Mo	6a	20	1.0	250	302	303	302	

(a) H_2 was lost, therefore, aim value was not reached, but could be accounted for in calculations to agree with analyzed values.

(b) Results of 1st determination on slug; 2nd determination on pure furnace specimen. Results of 1st determination probably represent analytical error.

General Operating Procedure:

1) Heat for 24 hrs. at 1350°F in vacuum to reduce hydrogen to 50 ppm.
2) Raise temperature to 1500°F in 1 hr. and add hydrogen.
3) Furnace cool to 1250°F in about 1 hr. and hold 1 to 2 hrs., furnace cool. (Furnace cooling about 2°F per minute to 850°F and about 0.7°F per minute to room temperature.)

TABLE 20

TENSILE AND CREEP-RUPTURE DATA FOR STABLE ALPHA ALLOY 6 Al

(Sponge Hardness 107 BHN -
Vacuum Annealed 25 Hours at 1350°F to 34-51 ppm of H_2)

Test Temp. (°F)	Stress (psi)	Rupture Time (hrs.)	Elongation (% in 1 in)	Red. of Area (%)	Loading Deformation (%)	Time (Hrs) for Indicated Total Deformation			Minimum Creep Rate (in/in/hr)
						0.5%	1.0%	Other	
75	140,000 T	(127,600Y)	17.0	31.6	--	--	--	--	--
600	90,750 T	(73,800Y)	14.3	42.7	--	--	--	--	--
600	85,000	>1340.8	--	--	1.62	--	--	(1.7%) 800	4.4×10^{-7}
1000	79,100 T	(66,000Y)	16.0	56.1	--	--	--	--	--
1000	35,000	138.6	34.0	47.0	0.34	7	16	(5%) 72	6.2×10^{-4}
1000	20,000	>1341.0	--	--	.20	105	385	(2%) 930	1.7×10^{-5}
1000	15,000	>1341.5	--	--	.14	--	--	(0.4%) 1340	7.4×10^{-7}

T - Tensile Strength
Y - Yield Strength
> - Test Stopped at Indicated Time

TABLE 21

INFLUENCE OF SPONGE HARDNESS AND HYDROGEN CONTENT ON THE TENSILE PROPERTIES OF STABLE ALPHA ALLOY 6 Al

Heat	Treatment	Sponge Hardness (BHN)	Hydrogen (ppm)	Temp. (°F)	Tensile Strength (psi)	Yield Strength, 0.2% offset (psi)	Elongation (%)	Red. of Area (%)
4c	25 hr. vacuum anneal at 1350°F	107	34/51	75	140,000	127,600	17.0	31.6
4	2025°F Water Quench	146	170	75	142,000	122,000	18.2	38.6
4a	2025°F Water Quench	124	150	75	127,800	115,000	17.0	34.5
4c	25 hr. vacuum anneal at 1350°F	107	34/51	600	90,750	73,800	14.3	42.7
4	2025°F Water Quench	146	170	600	97,900	78,500	17.6	34.1
4a	2025°F Water Quench	124	150	600	77,700	60,700	17.0	43.5
4c	25 hr. vacuum anneal at 1350°F	107	34/51	1000	79,100	66,000	16.0	56.1
4	2025°F Water Quench	146	170	1000	80,300	64,800	14.9	33.8
4a	2025°F Water Quench	124	150	1000	72,500	58,000	16.0	37.8
4a	24 hr. vacuum anneal at 1200°F	124	?	1000	73,800	58,700	15.0	57.4

(Data for Heats 4 and 4a taken from References 1 and 2)

TABLE 22

CREEP DATA FOR ALPHA-BETA ALLOY Ti 150A WITH VARIABLE HYDROGEN

H_2 (ppm)	Creep Conditions: Temp. (°F)	Stress (psi)	Time (hr)	Loading Deformation (%)	Time (hr) for Indicated Total Deformation (%)	Minimum Creep Rates (in/in/hr)
57-64	600	64,000	100	--	- - - - - - - - -	6.2×10^{-6}
57-64	600	64,000	500	3.03	3.25%-25; 3.5%-250; 3.71%-500	9.1×10^{-6}
57-64	600	64,000	1000	2.98	3.25%-65; 3.5%-350; 3.9%-1000	5.4×10^{-6}
274	600	64,000	100	2.91	3.28%-100	1.3×10^{-5}
57-64	600	32,000	100	.32	- - - - - - - - -	- - - - -
57-64	600	32,000	500	.33	0.34% - 500	3×10^{-7}
57-64	600	32,000	1032.7	.29	0.27% - 1000	nil
57-64	800	28,000	101.6	.35	0.41% - 100	3.6×10^{-6}
57-64	900	14,000	100	.16	1.5% - 100	1.4×10^{-6}
57-64	1000	2,800	30	.06	0.18% - 30	1.1×10^{-5}
57-64	1000	2,800	100	.04	0.26% - 100	6.2×10^{-6}
162	1000	2,800	30	.04	0.145% - 30	6.2×10^{-6}
274	1000	2,800	30	.03	0.15% - 30	7.1×10^{-5}
57-64	1000	5,600	100	.06	0.1%-6; 0.2%-18; 0.38%-100	1.2×10^{-5}
274	1000	5,600	100	.08	0.55% - 100	1.9×10^{-5}
57-64	1000	5,600	500	.04	0.55% - 500	3.1×10^{-6}

Heat Treatments:

57-64 ppm H_2 -- 26 hours in vacuum at 1350°F + furnace cooled

162 ppm H_2 -- 26 hours in vacuum at 1350°F + 2 hours at 1500°F in H_2 + furnace cool with a 2-hour hold at 1250°F

274 ppm H_2 -- 26 hours in vacuum at 1350°F + 4.3 hours at 1500°F in H_2 + furnace cool with a 2-hour hold at 1250°F

TABLE 23

EFFECT OF HYDROGEN CONTENT
ON THE EMBRITTLEMENT OF ALPHA-BETA ALLOY Ti 150A
DURING EXPOSURE TO TEMPERATURE AND STRESS

Hydrogen	Stressed Exposure Conditions			Tensile Properties at 75°F			
	Temp.	Stress	Time	Tensile Strength	Yield Strength 0.2% offset	Elongation (% in 1 in.)	Red. of Area
(ppm)	(°F)	(psi)	(hr)	(psi)	(psi)		(%)
Heated for 26 Hours in Vacuum at 1350°F + Furnace Cool							
57-64	As-treated			136,600		27.0	55.4
	600	64,000	100	140,000		25.2	50.8
	600	64,000	500	150,800		21.0	43.5
	600	64,000	1000	152,600		20.0	37.0
	600	32,000	100	133,700		30.0	52.9
	600	32,000	500	135,500		26.0	51.5
	600	32,000	1000	137,000	127,000	27.0	53.4
	800	28,000	100	137,300	133,000	24.5	52.7
	900	14,000	100	138,000	123,500	9.9	14.0
	1000	2,800	30	135,500	124,000	11.0	11.7
	1000	2,800	100	136,500	127,000	9.0	7.9
	1000	5,600	50	132,200	114,000	11.0	11.0
	1000	5,600	100	132,800	120,500	10.0	10.0
	1000	5,600	500	134,000	118,500	12.0	13.1
Heated for 26 Hours in Vacuum at 1350°F + 2 Hours at 1500°F in Hydrogen + Furnace Cool with a 2-hour hold at 1250°F							
162	As-treated			135,500	131,000	22.0	33.4
	600	64,000	98.2	143,000	138,000	20.0	23.4
	1000	2,800	30	132,500	124,500	3.0	2.4

TABLE 23 (Continued)

EFFECT OF HYDROGEN CONTENT
ON THE EMBRITTLEMENT OF ALPHA-BETA ALLOY Ti 150A
DURING EXPOSURE TO TEMPERATURE AND STRESS

Hydrogen	Stressed Exposure Conditions			Tensile Properties at 75°F			
	Temp.	Stress	Time	Tensile Strength	Yield Strength 0.2% offset	Elongation (% in 1 in.)	Red. of Area
(ppm)	(°F)	(psi)	(hr)	(psi)	(psi)		(%)
	Heated for 26 Hours in Vacuum at 1350°F + 4.3 Hours at 1500°F in Hydrogen + Furnace Cool with a 2-hour hold at 1250°F						
274	As-treated			135,000	131,000	18.1	46.6
	600	64,000	100	146,300	146,300	10.0	12.5
	1000	2,800	29.8	136,500	126,500	2.9	10.5
	1000	5,600	100	134,700	--	6.0	9.4
	(a) Annealed 1 Hour at 1500°F in Argon						
310	As-treated			138,500	130,600	25.5	42.8
	800	23,000	1146	193,000	--	3.7	3.9
	1000	5,000	1028	131,500	112,000	7.1	7.1

(a) Data from Reference 2.

TABLE 24

EFFECT OF HEAT TREATMENTS AND HOT-ROLLING
ON MICROSTRUCTURE AND HYDROGEN CONTENT OF 8 Mn ALLOY

Treatment Conditions	Microstructure	H_2 (ppm)	Number of Diffraction Lines Identified		
			α	β	ω
1. As-Forged	equi-axed β	114	3(2?)	7	1(2?)
2. 0.5 hr. at 1380°F and reduced 26% by rolling, AC, heated 0.5 hour at 1360°F, FC to 1020°F and then AC	equi-axed β with slight transformation at grain boundaries	118			
3. 0.5 hr. at 1250°F and reduced 25% by rolling, AC, heated for 24 hours in vacuum at 1320°F, FC	small evenly distributed α in a β matrix-α conc. at grain boundaries	108			
4. Annealed in vacuum for 24 hours at 1320°F, FC - heated to 1300°F for 0.5 hr. and reduced 25% by rolling - heated to 1200°F for 0.5 hr, FC	small evenly distributed α in a β matrix-α conc. at grain boundaries	106			
5. Annealed in vacuum for 24 hours at 1365°F, FC	small evenly distributed α - some Widmanstatten	112	8(1?)	7	5(1?)
6. 24 hours at 1350°F in vacuum + 8 hours at 1500°F in hydrogen, FC	β with some feathery α at grain boundaries	213	6(1?)	7	3(2?)

TABLE 24 (Continued)

EFFECT OF HEAT TREATMENTS AND HOT-ROLLING
ON MICROSTRUCTURE AND HYDROGEN CONTENT OF 8 Mn ALLOY

Treatment Conditions	Microstructure	H_2 (ppm)	Number of Diffraction Lines Identified		
			α	β	ω
7. Annealed in vacuum for 24 hours at 1500°F, FC	some feathery α at β grain boundaries	83	5 (1?)	5 (1?)	3
8. Annealed in vacuum for 24 hours at 1500°F, FC + 24 hours at 1270°F in vacuum	small evenly distributed α; some feathery and plate α near β grain boundaries	(83)	10	4	4 (1?)

AC - Air cooled
FC - Furnace cooled

TABLE 25

TENSILE AND CREEP-RUPTURE PROPERTIES FOR META-STABLE BETA ALLOY 10 Mo AT TWO HYDROGEN LEVELS

(Heat 9038 - Sponge Hardness 107)

Temp. (°F)	Yield Strength 0.2% offset (psi)	Stress (psi)	Rupture Time (hrs.)	Elongation (% in 1 in)	Red. of Area (%)	Loading Deformation (%)	Time for Indicated Total Deformation (hours)				Minimum Creep Rate (in/in/hr)
							0.5%	1%	2%	Other	
Annealed in Vacuum for 23 hours at 1350°F to 44 ppm Hydrogen											
75	106,500	115,600	(TT)	17.0	31.6	--	--	--	--	--	--
600	56,250	74,750	(TT)	16.0	51.3	--	--	--	--	--	--
600	--	73,000	>1340.9	--	--	(72.0)	--	--	--	--	2.5×10^{-6}
600	--	71,000	>1081.5	--	--	1.46	--	--	30	(2.21%) 1000	nil
1000	40,500	51,500	(TT)	12.0	21.8	--	--	--	--	--	--
1000	--	22,000	142.0	23.0	36.8	.26	10	35	80	--	2.0×10^{-4}
1000	--	10,000	>1319.7	--	--	.125	50	260	655	(3%) 990	1.8×10^{-5}
1000	--	5,000	>1940.2	--	--	.061	500	1870	--	--	3.3×10^{-6}
Vacuum annealed 23 hours at 1350°F + 1 hour at 1500°F in H_2 + furnace cooled to 1250°F for 2 hours + furnace cool - 242/260 ppm hydrogen											
75	98,000	111,000	(TT)	15.0	1.6	--	--	--	--	--	--
600	62,500	78,500	(TT)	18.0	62.0	--	--	--	--	--	--
600	--	74,000	on load	18.5	67.0	--	--	--	--	--	--
600	--	70,000	on load	13.0	66.0	--	--	--	--	--	--
1000	42,100	52,000	(TT)	16.2	35.3	--	--	--	--	--	--
1000	--	22,000	60.3	34.3	54.0	.32	--	--	--	--	--
1000	--	10,000	>1323.5	--	--	.11	35	240	575	(4%) 1320	2.0×10^{-5}
1000	--	5,000	>1200	--	--	.04	510	1270 (est)			

(TT) - Tensile Test

TABLE 26

TENSILE AND CREEP-RUPTURE PROPERTIES
FOR STABLE BETA ALLOY 30 Mo AT VARIOUS HYDROGEN LEVELS

(Heat 9042 - Sponge Hardness 107 BHN and Heat 6 - Sponge Hardness 140 BHN)

Hydrogen (ppm)	Temp. (°F)	Yield Strength 0.2% offset (psi)	Stress (psi)	Rupture Time (hr.)	Elongation (% in 1 in)	Red. of Area (%)	Loading Deformation (%)	Time for Indicated Total Deformation (hours)				Minimum Creep Rate (in/in/hr)
								0.5%	1%	2%	Other	
Sponge Hardness 107 Vacuum annealed at 1350°F for 25 hours, furnace cooled												
112	75	--	>116,000	(TT)	(Fractured in threads)		--	--	--	--	--	--
112	600	79,800	92,600	(TT)	14.0	37.8	--	--	--	--	--	--
112	1000	66,250	75,000	(TT)	17.6	42.7	--	--	--	--	--	--
50	1000	--	36,000	86.6	40.0	--	0.41	--	--	--	--	--
50	1000	--	32,000	140.4	54.0	--	.31	3	7	17	--	1.0×10^{-3}
50	1000	--	27,000	277.6	27.2	--	.26	22	45	75	(6.5%)185	1.0×10^{-4}
50	1000	--	20,000	>1154.9	--	--	.19	22	60	128	(5%)318	1.2×10^{-4}
Sponge Hardness 107 Vacuum annealed at 1350°F for 25 hours + raised to 1500°F for 1 hour, furnace cooled with a 2 hour hold at 1250°F												
216	75	122,000	122,000	(TT)	3.0	4.0	--	--	--	--	--	--
216	600	90,600	96,200	(TT)	14.7	49.3	--	--	--	--	--	--
216	1000	63,200	72,700	(TT)	18.0	42.2	--	--	--	--	--	--
216	1000	--	36,000	122.0	37.3	--	.32	3	13	26	(8%) 69	5.2×10^{-4}
216	1000	--	27,000	322.4	(20)	--	.31	45	72	112	(7%) 236	4.1×10^{-5}
216	1000	--	20,000	>1154.9	--	--	.22	36	86	190	(12%) 980	5.7×10^{-5}

(TT) = Tensile Test

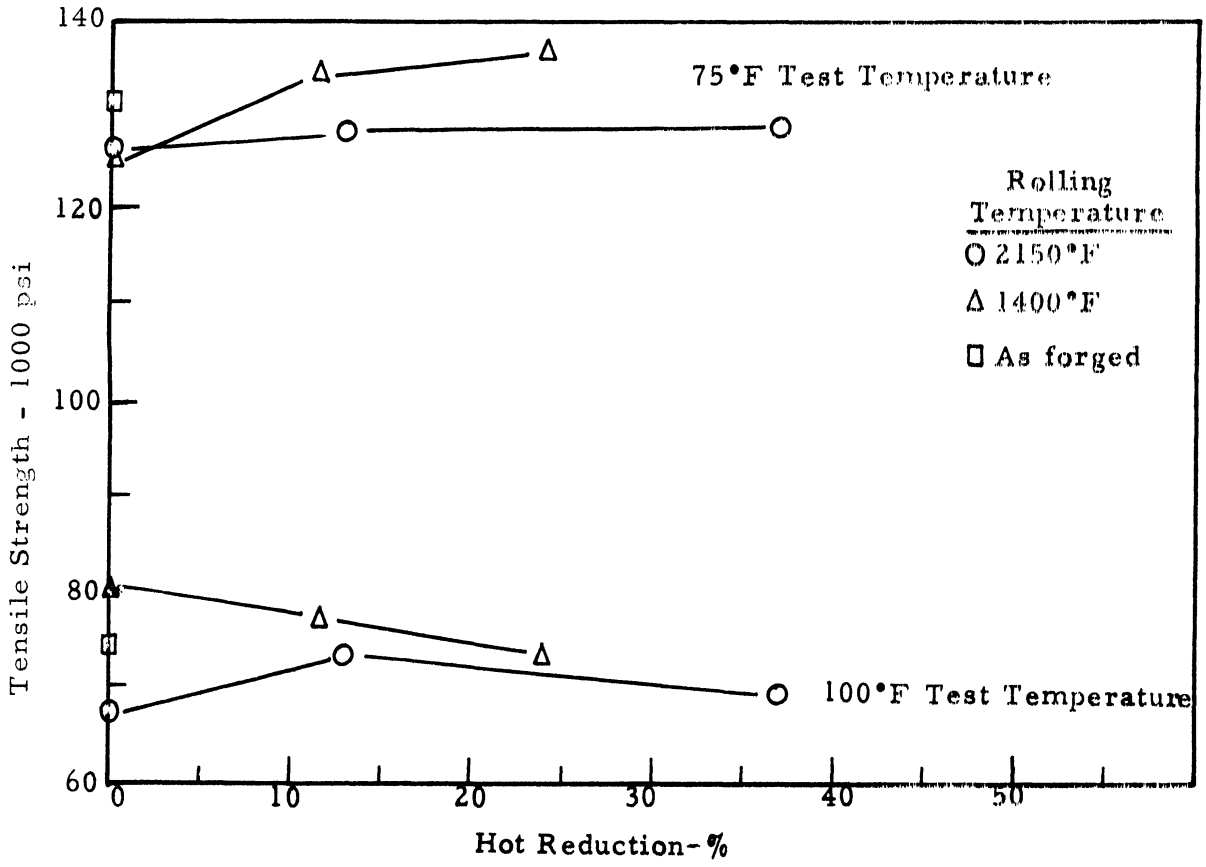
TABLE 26 (Continued)

TENSILE AND CREEP-RUPTURE PROPERTIES
FOR STABLE BETA ALLOY 30 Mo AT VARIOUS HYDROGEN LEVELS

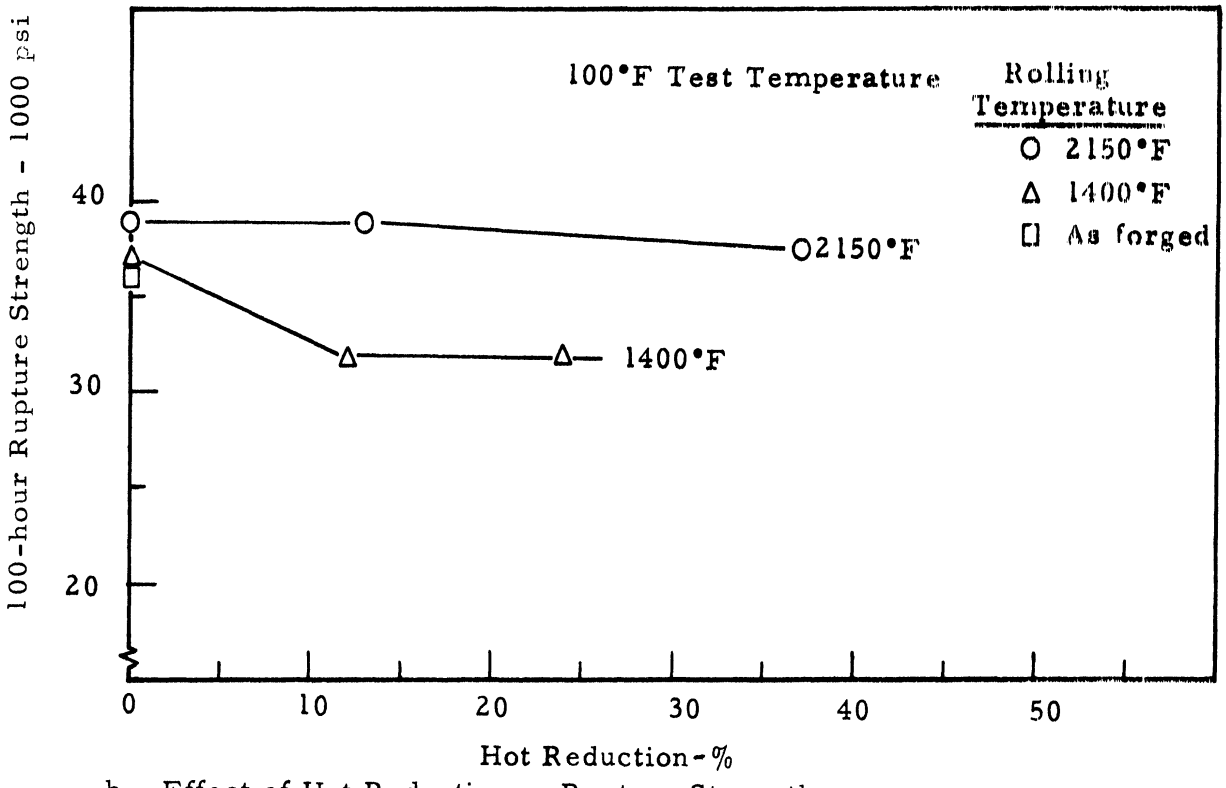
(Heat 9042 - Sponge Hardness 107 BHN and Heat 6 - Sponge Hardness 140 BHN)

Hydrogen (ppm)	Temp. (°F)	Yield Strength 0.2% offset (psi)	Stress (psi)	Rupture Time (hr.)	Elongation (% in 1 in)	Red. of Area (%)	Loading Deformation (%)	Time for Indicated Total Deformation (hours) 0.5%	1%	2%	Other	Minimum Creep Rate (in/in/hr)
Sponge Hardness 140 BHN Vacuum annealed at 1350°F for 25 hours, furnace cooled												
44	75	133,700	133,700	(TT)	11.0	20.7	--	--	--	--	--	--
44	600	90,000	101,600	(TT)	18.0	51.1	--	--	--	--	--	--
44	600	--	96,000	>1153	--	--	1.80	--	--	150	(2.47%) 1100	nil
44	600	--	90,000	>1270.9	--	--	1.14	--	--	--	(1.17%) 750	nil
44	1000	72,250	79,700	(TT)	18.0	56.9	--	--	--	--	--	--
44	1000	--	37,500	75.5	67.0	75.4	.33	1	4	--	(5%) 26	1.6×10^{-3}
44	1000	--	26,000	282.5	32.0	64.0	.19	50	90	--	--	2.6×10^{-5}
44	1000	--	16,000	>1132.9	--	--	.16	230	410	600	(5%) 960	1.3×10^{-5}

(TT) - Tensile Test



a. Effect of Hot Reduction on Tensile Strength



b. Effect of Hot Reduction on Rupture Strength

Figure 1. Influence of Hot Rolling on the Tensile and Rupture Strengths of Stabilized Alpha Alloy 6 Al.

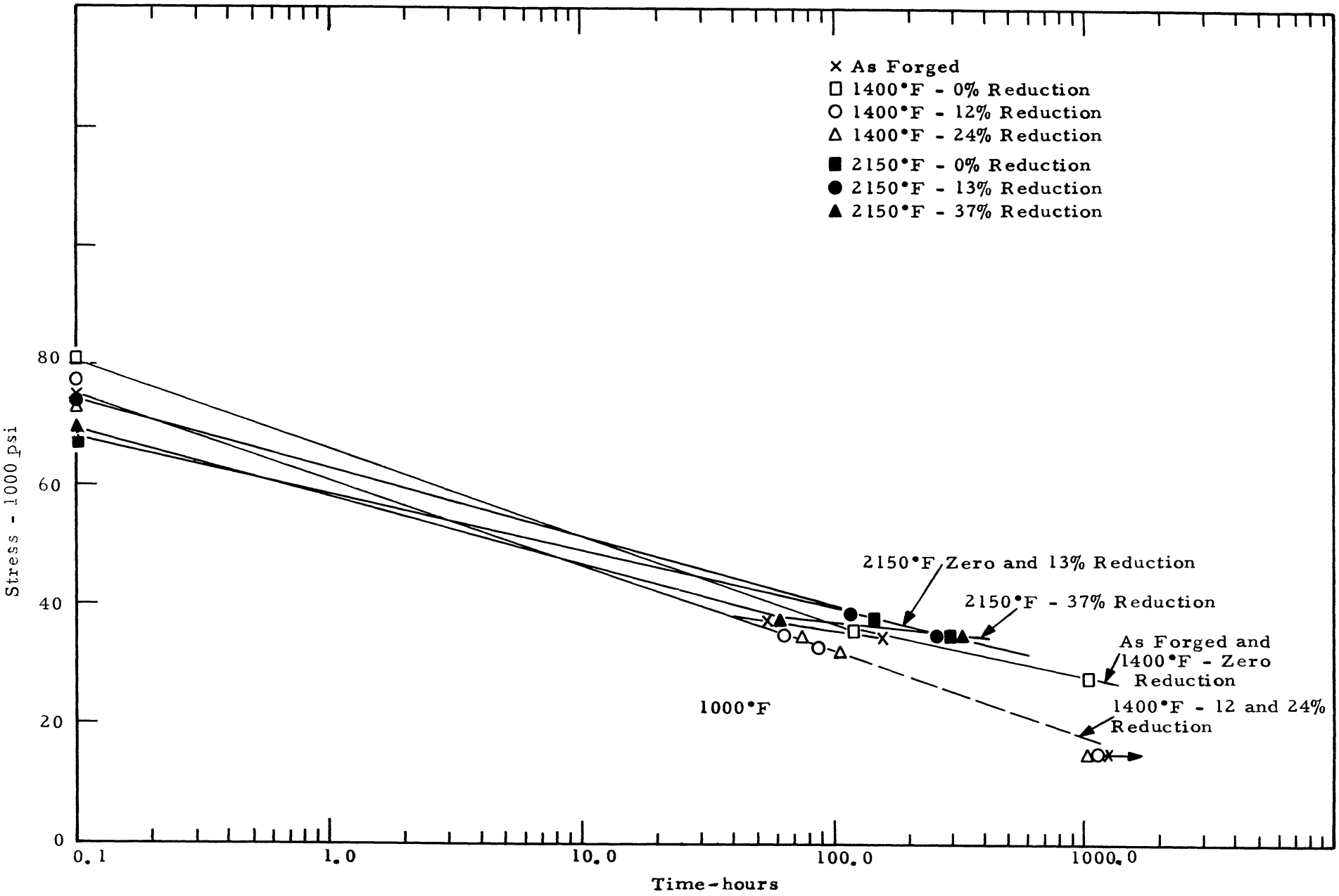


Figure 2. Stress Rupture Time Curves at 1000°F for Hot Rolled Stabilized Alpha Alloy 6 Al.

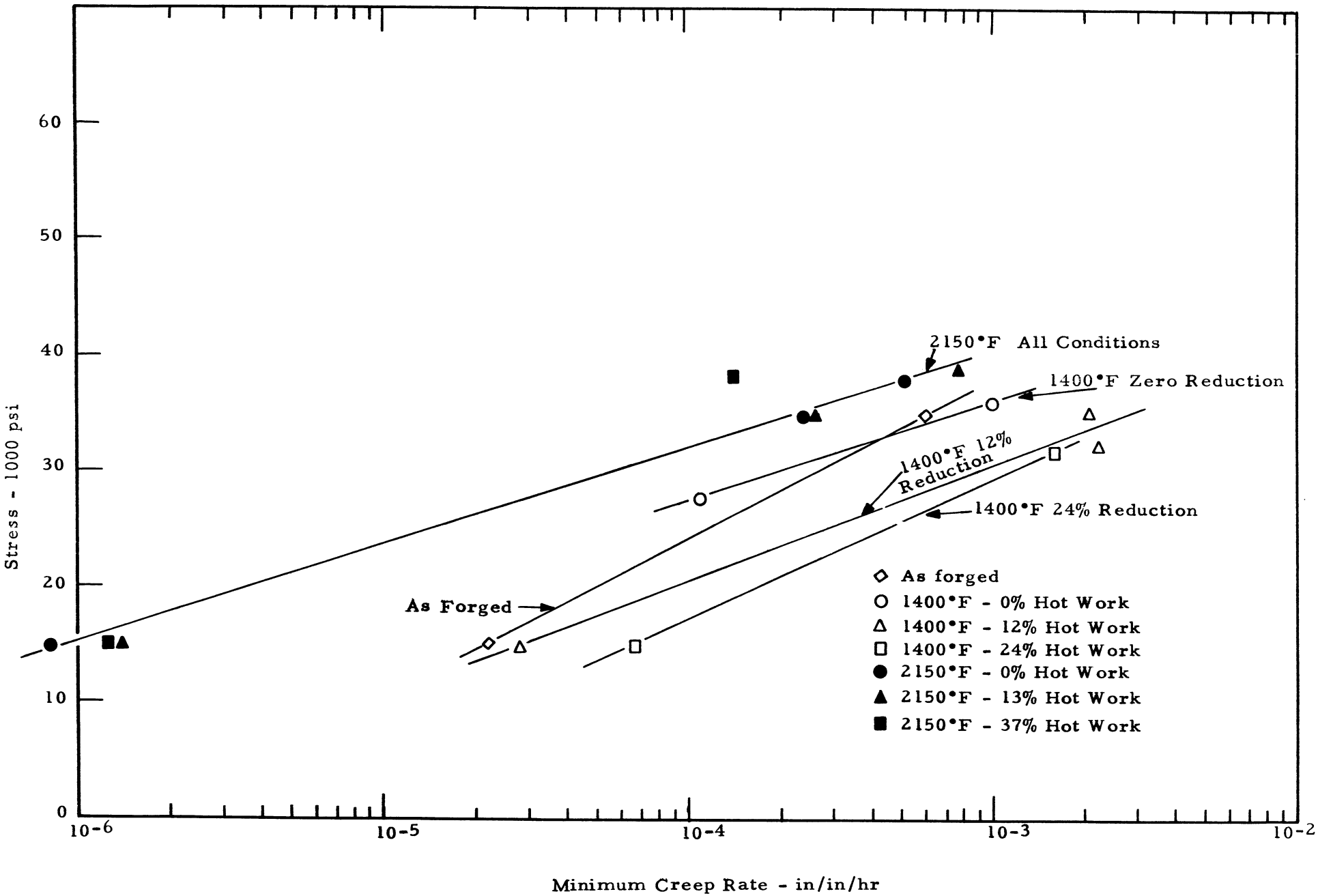


Figure 3. Stress-Minimum Creep Rate Curves at 1000°F for Hot Rolled Stable Alpha Alloy 6 Al

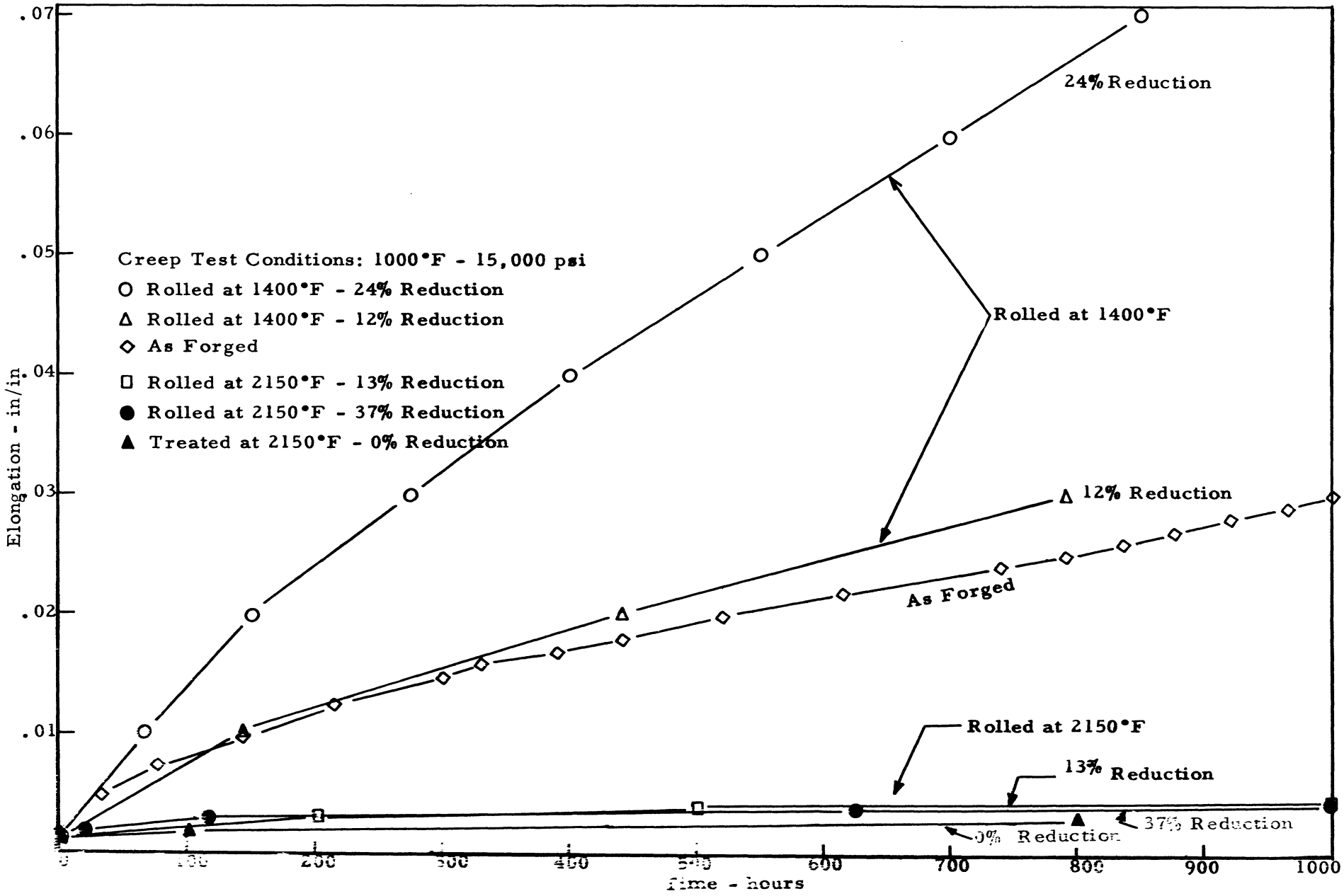
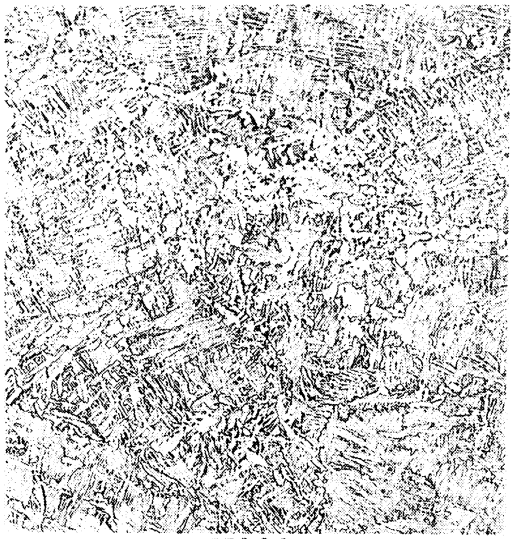


Figure 4. Time-Elongation Curves at 1000°F and 15,000 psi for Hot Rolled Stabilized Alpha Alloy 6 Al.

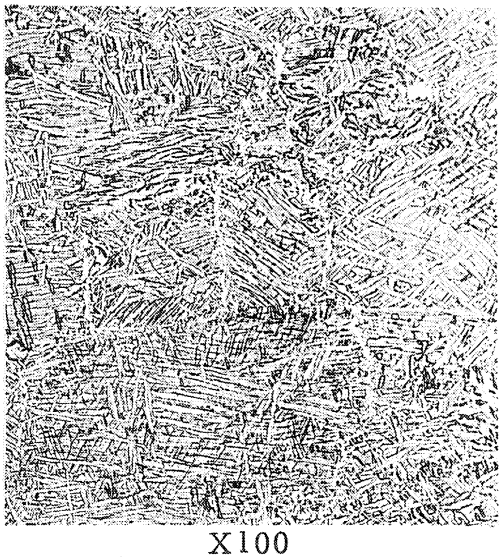


X100

a. As Forged

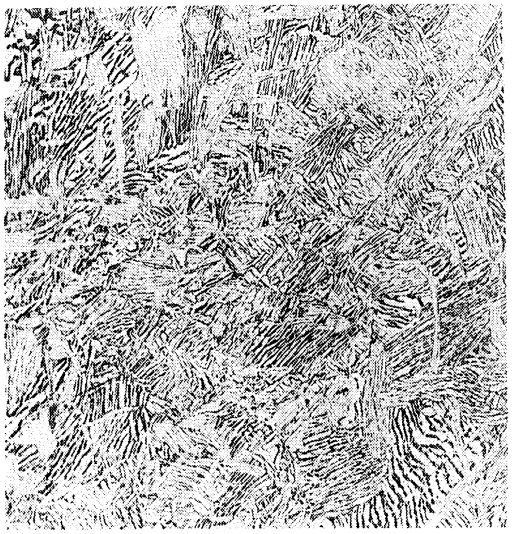
X100

b. 12% Hot Reduction at 1400°F



X100

c. 12% Hot Reduction at 1400°F plus 86 hours at 1000°F and 32,500 psi



X100

d. 24% Hot Reduction at 1400°F

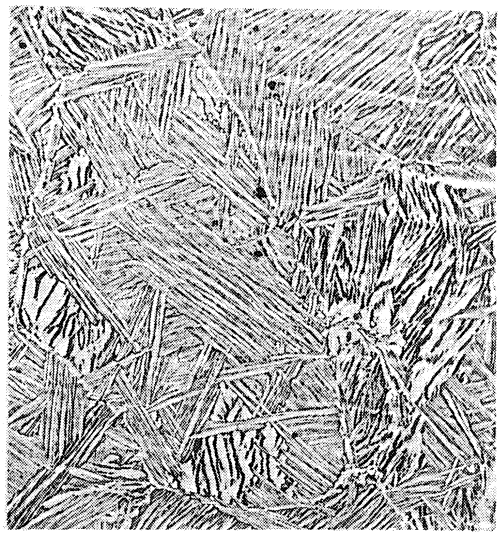
X100

e. 24% Hot Reduction at 1400°F plus 106 hours at 1000°F and 32,000 psi

Figure 5. - Microstructures of Hot Rolled Stabilized Alpha Alloy 6 Al before and after Rupture Testing at 1000°F.

X100

f. 13% Hot Reduction at 2150°F

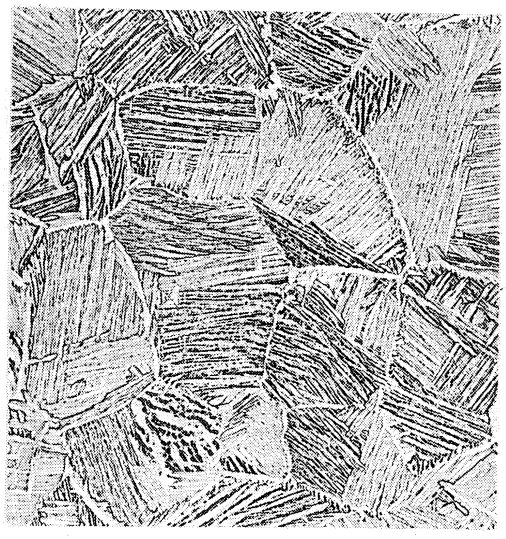


X100

g. 13% Hot Reduction at 2150°F plus 255 hours at 1000°F and 35,000 psi

X100

h. 37% Hot Reduction at 2150°F



X100

i. 37% Hot Reduction at 2150°F plus 60 hours at 1000°F and 38,500 psi

Figure 5. (concluded) - Microstructures of Hot Rolled Stabilized Alpha Alloy 6 Al before and after Rupture Testing at 1000°F.

X1000

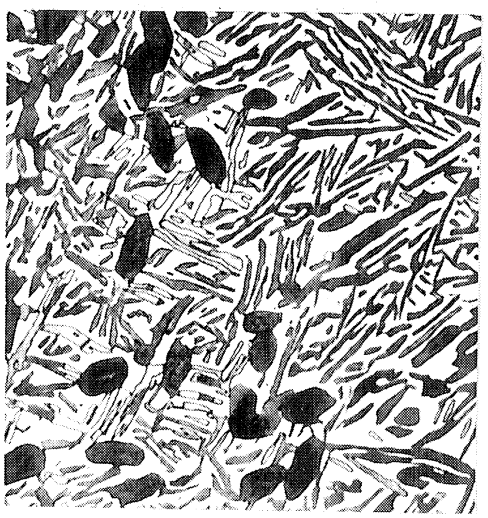
a. As Received

X1000

b. Reduced 30.7% at 1200°F

X1000

c. Reduced 29.3% at 1350°F



X1000

d. Reduced 38.2% at 1500°F

X1000

e. Reduced 37.8% at 1650°F

Figure 6. - Microstructures of Alpha-Beta Alloy Ti 150A in the As-Rolled Conditions.

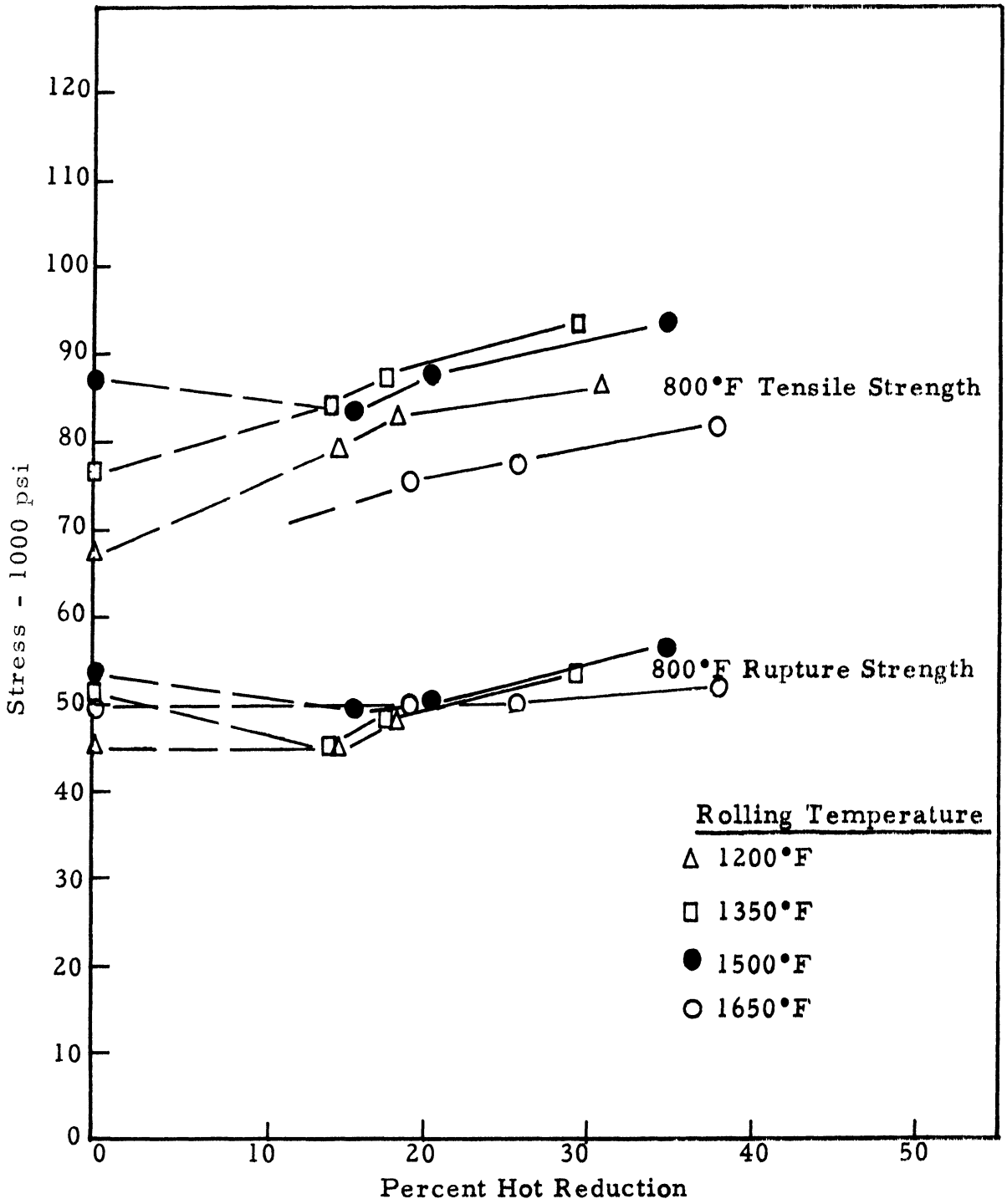


Figure 7. Effect of Hot-Rolling Conditions on Tensile Strength and 100-Hour Rupture Strength at 800°F for Alpha-Beta Alloy Ti 150A.

Data for zero reduction from Reference 1.

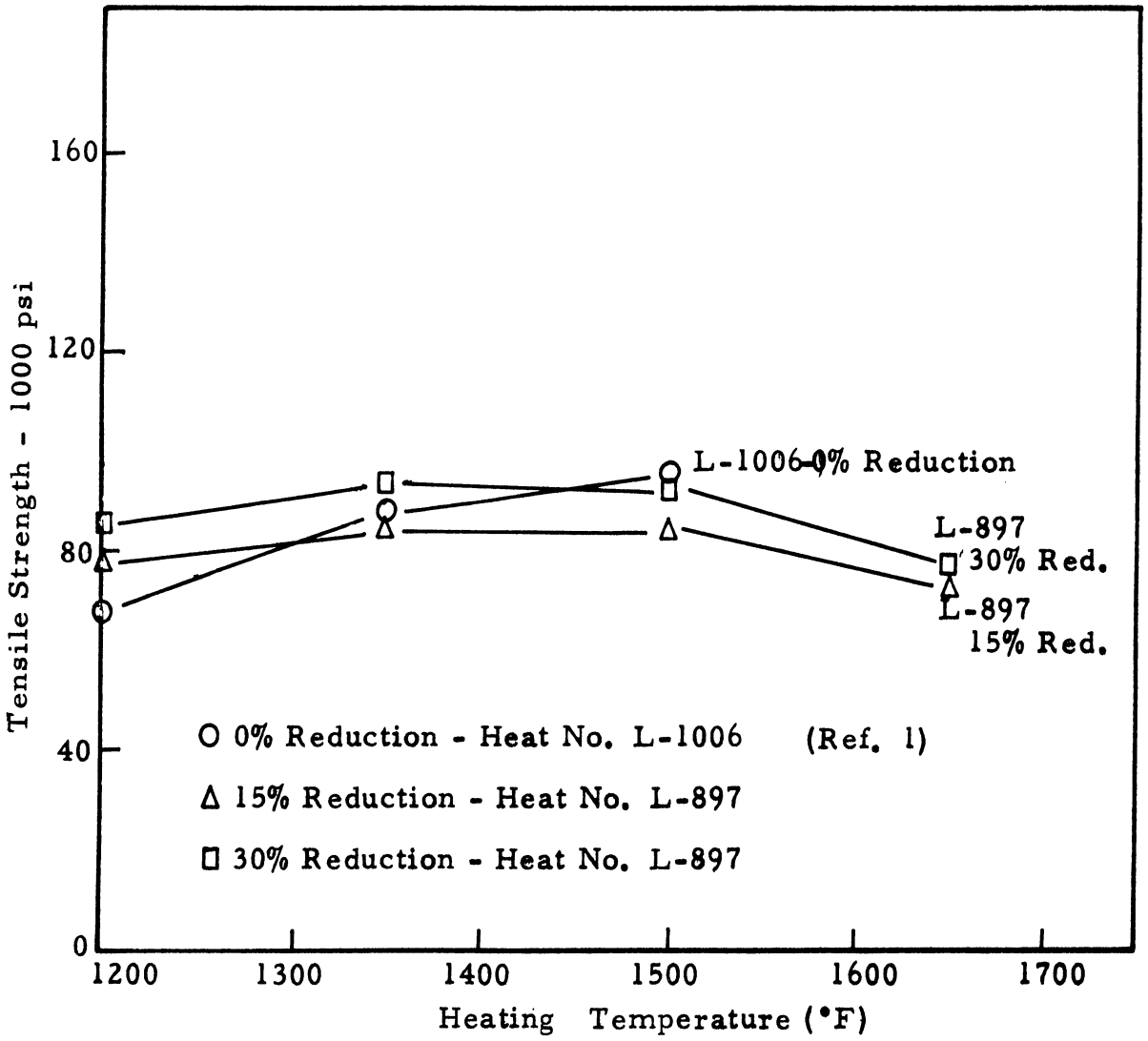


Figure 8. Effect of Heating Temperature on Tensile Strength of Alpha-Beta Alloy Ti 150A.

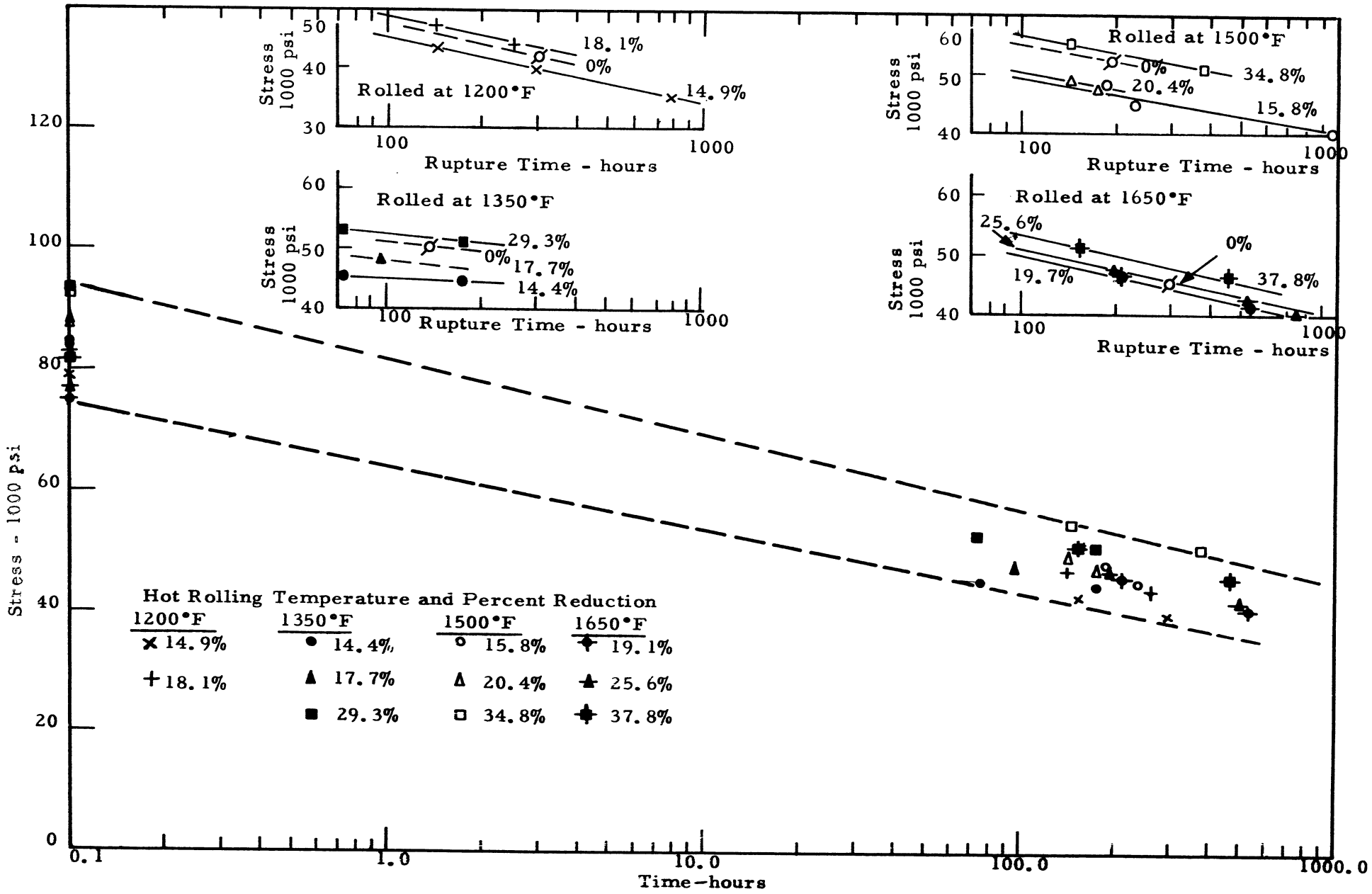


Figure 9. Stress-Rupture Time Curves at 800°F for Alpha-Beta Alloy Ti 150A after Hot Rolling.

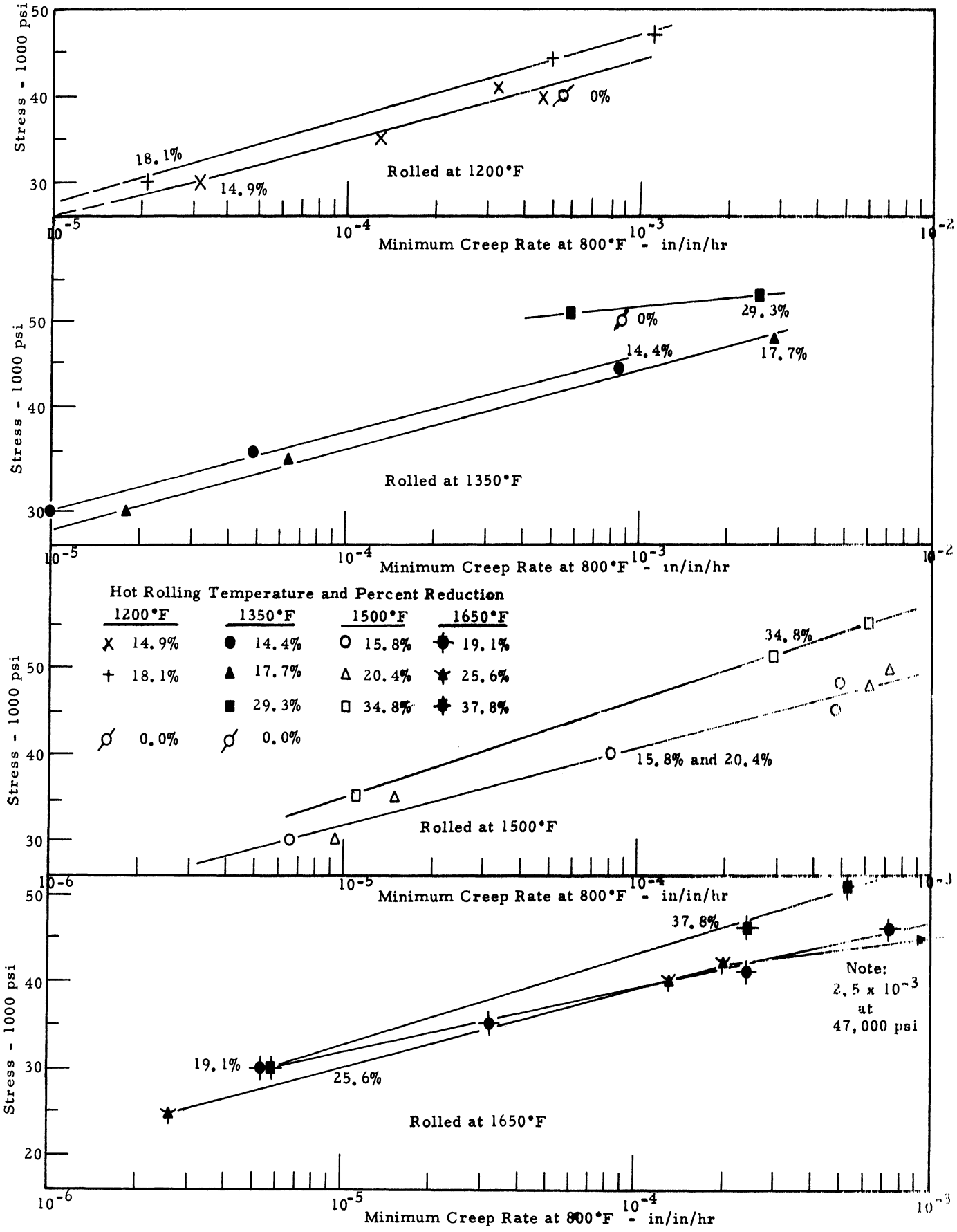


Figure 10. Stress-Minimum Creep Rate Curves at 800°F for Alpha-Beta Alloy Ti 150A After Hot Rolling

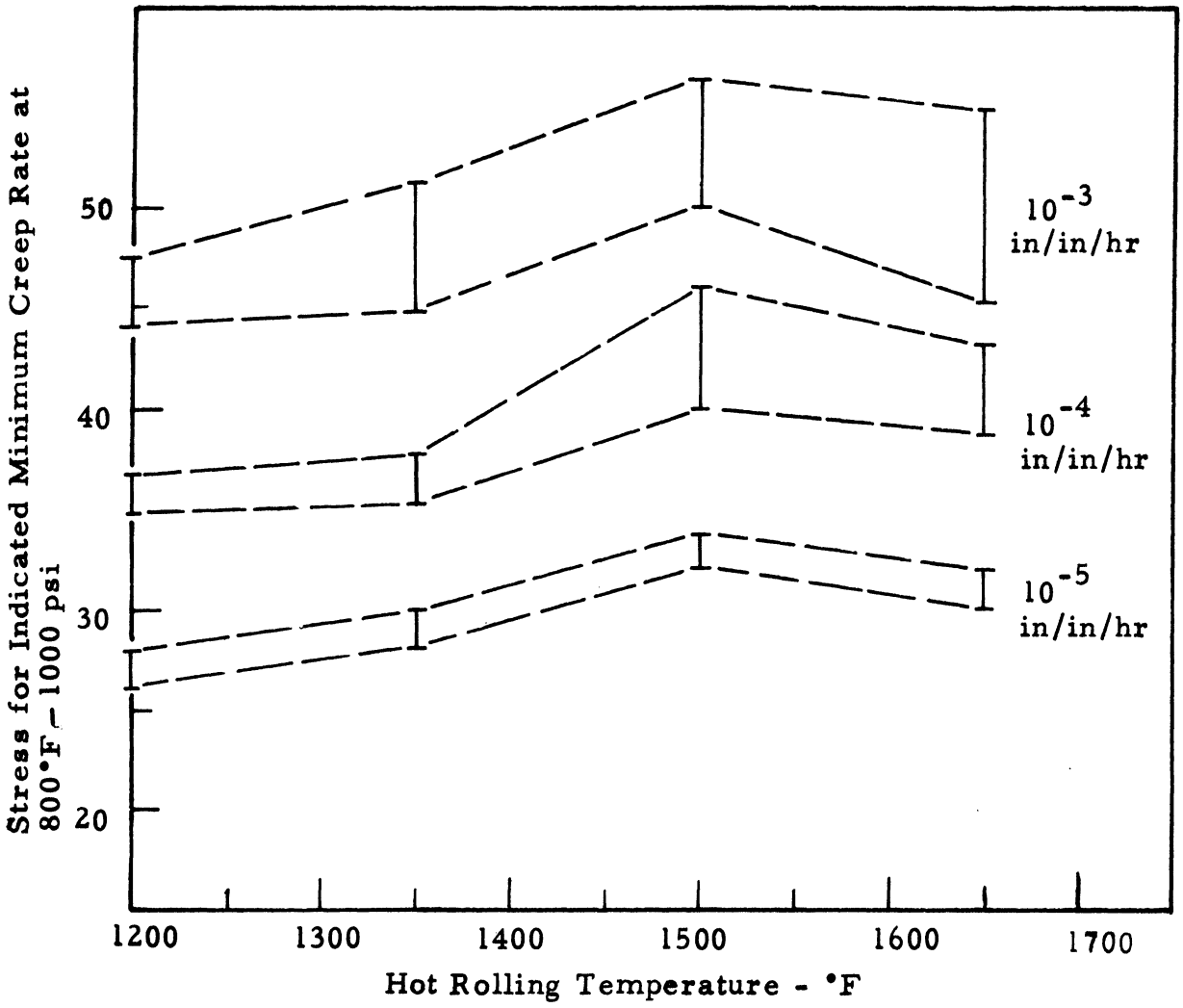


Figure 11. Effect of Hot Rolling Temperature on Stress for Indicated Minimum Creep Rates at 800°F for Alpha-Beta Alloy Ti 150A.

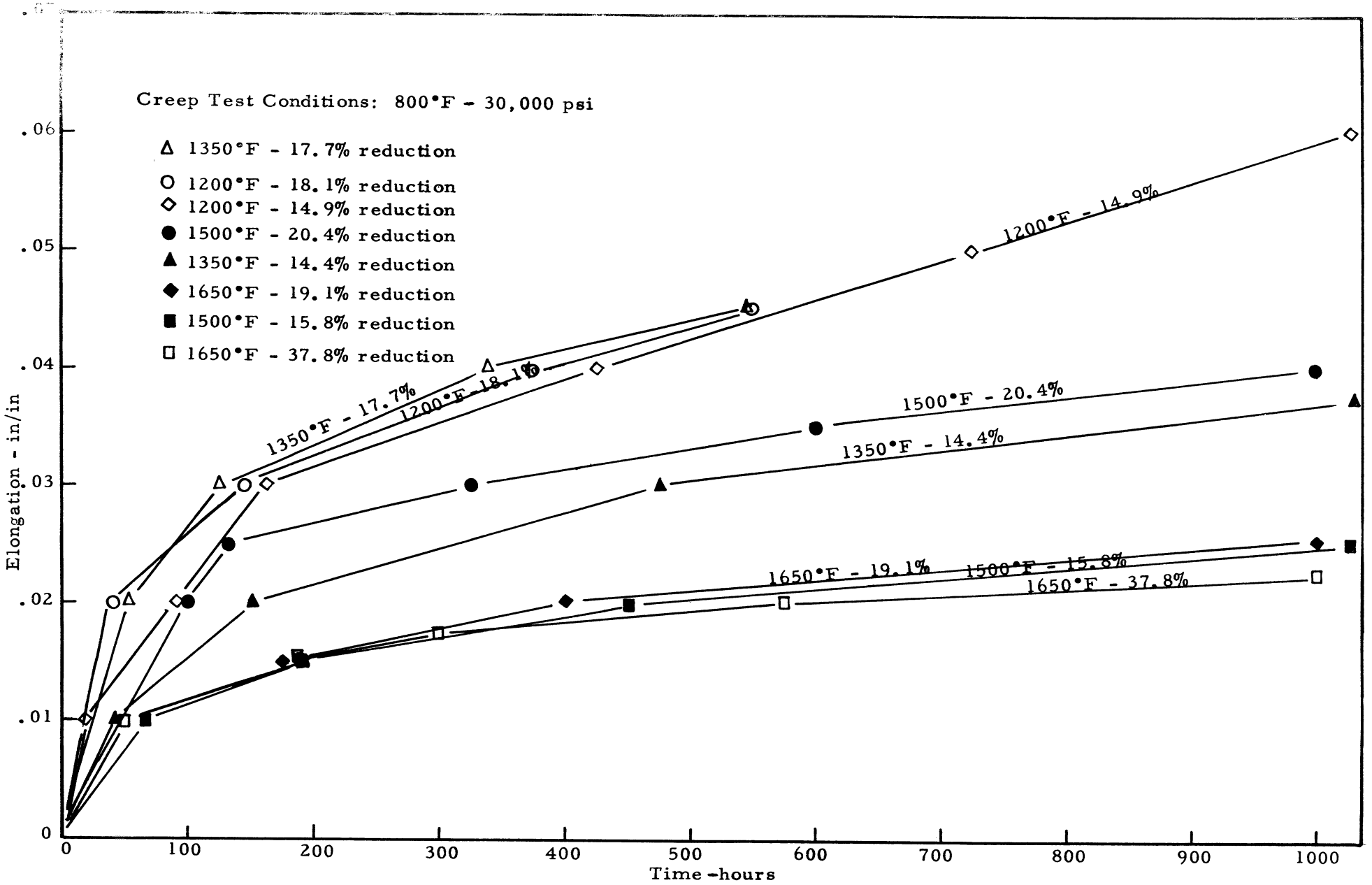


Figure 12. - Time-Elongation Curves at 800°F and 30,000 psi for Hot-Rolled Alpha-Beta Alloy Ti 150A.

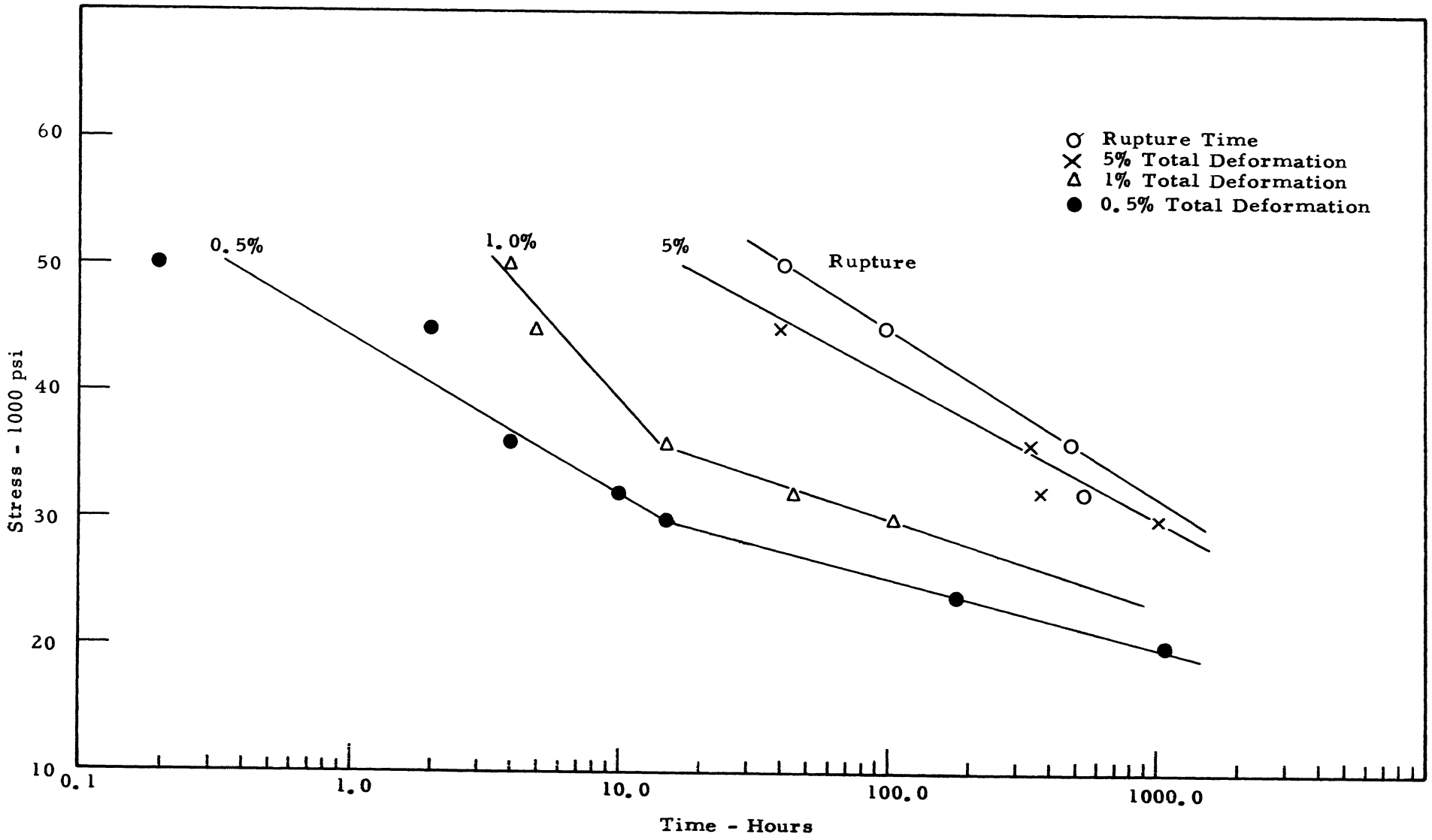


Figure 13. Stress-Rupture Time Curves at 1000°F for Stabilized Alpha Alloy 6 Al - 0.5 Si in the As-Forged Condition.

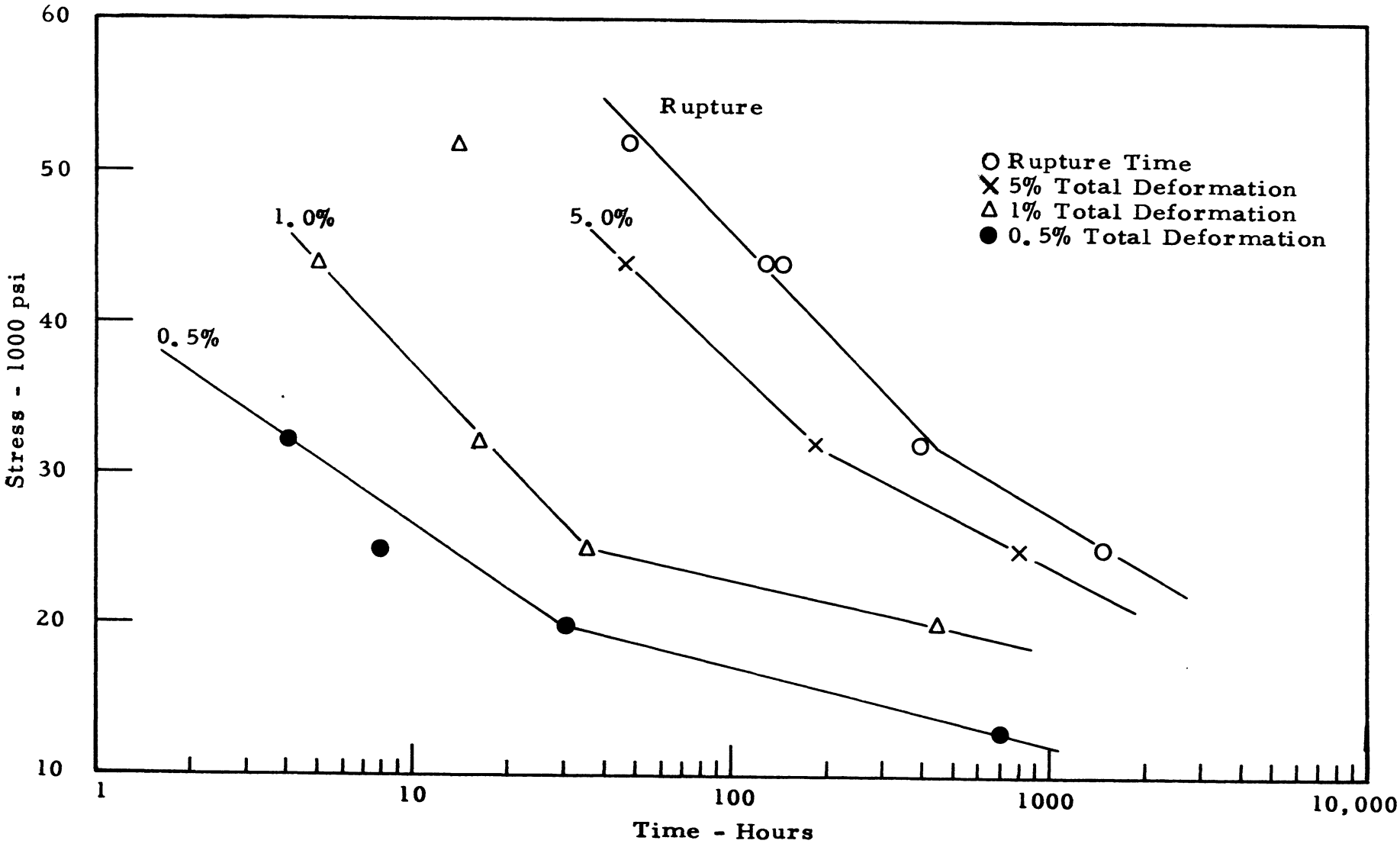


Figure 14. Stress-Rupture Time Curves at 1000°F for Stabilized Alpha Alloy 6 Al - 0.5 Si Cold Reduced 20 Percent.

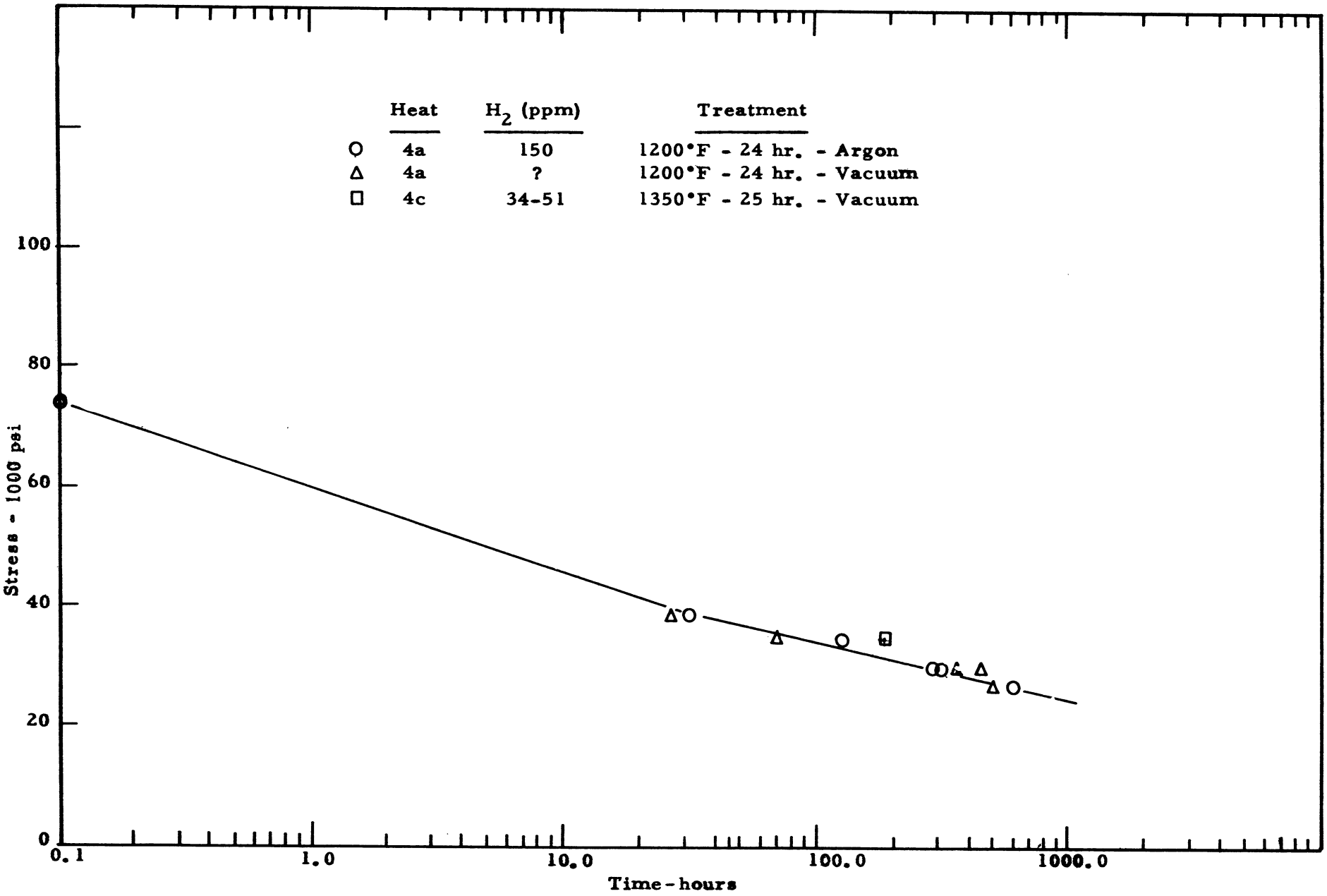


Figure 16. - Stress-Rupture Time Curves at 1000°F for Stabilized Alpha Alloy 6Al After Annealing in Argon or Vacuum for 24 hours at 1200°F

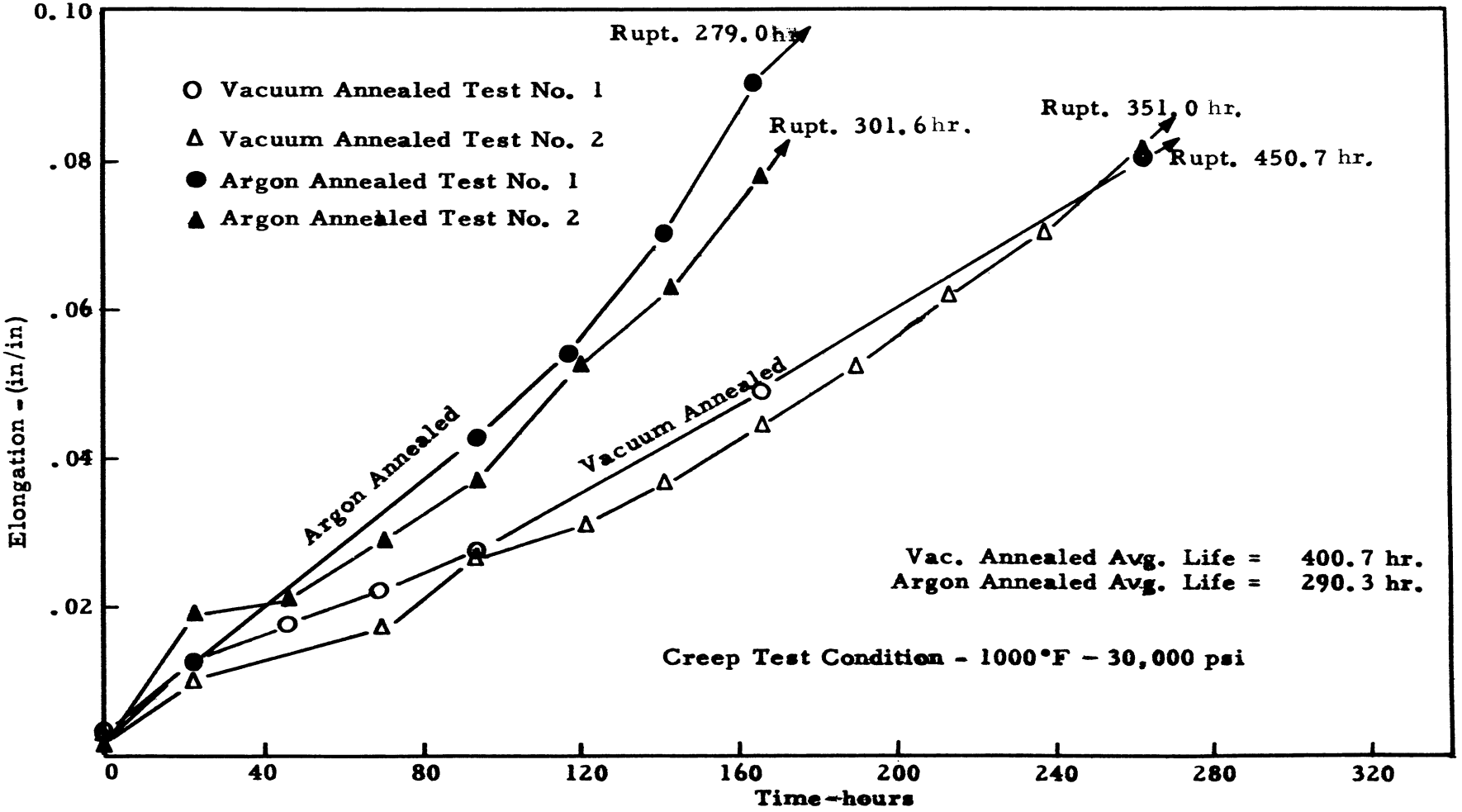


Figure 17. Time Elongation Curves at 1000°F and 30,000 psi for Stabilized Alpha Alloy 6 Al after Annealing in Argon or Vacuum for 24 Hours at 1200°F.

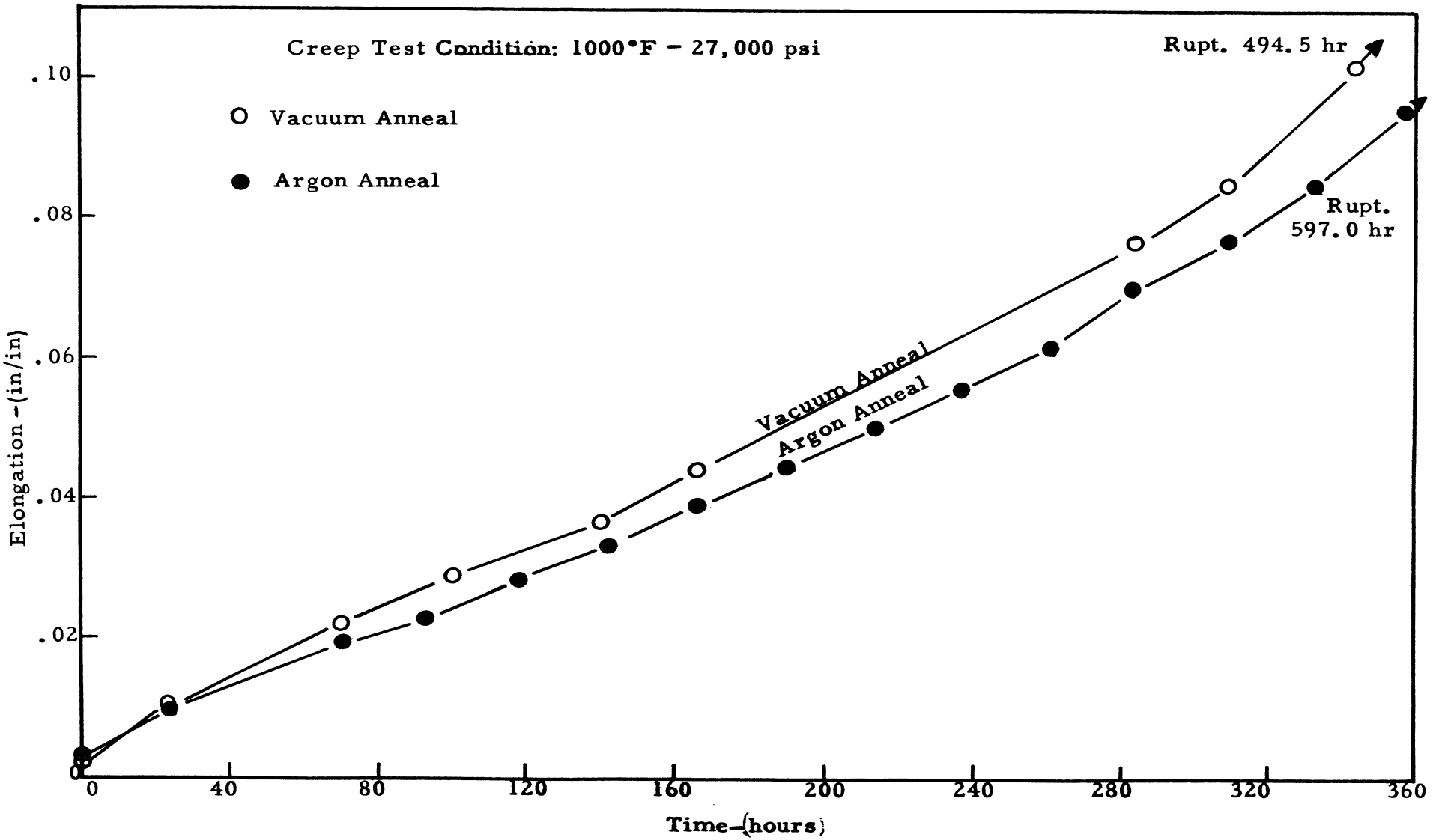


Figure 18. Time Elongation Curves at 1000°F and 27,000 psi for Stabilized Alpha Alloy 6 Al after Annealing in Argon or Vacuum for 24 Hours at 1200°F.

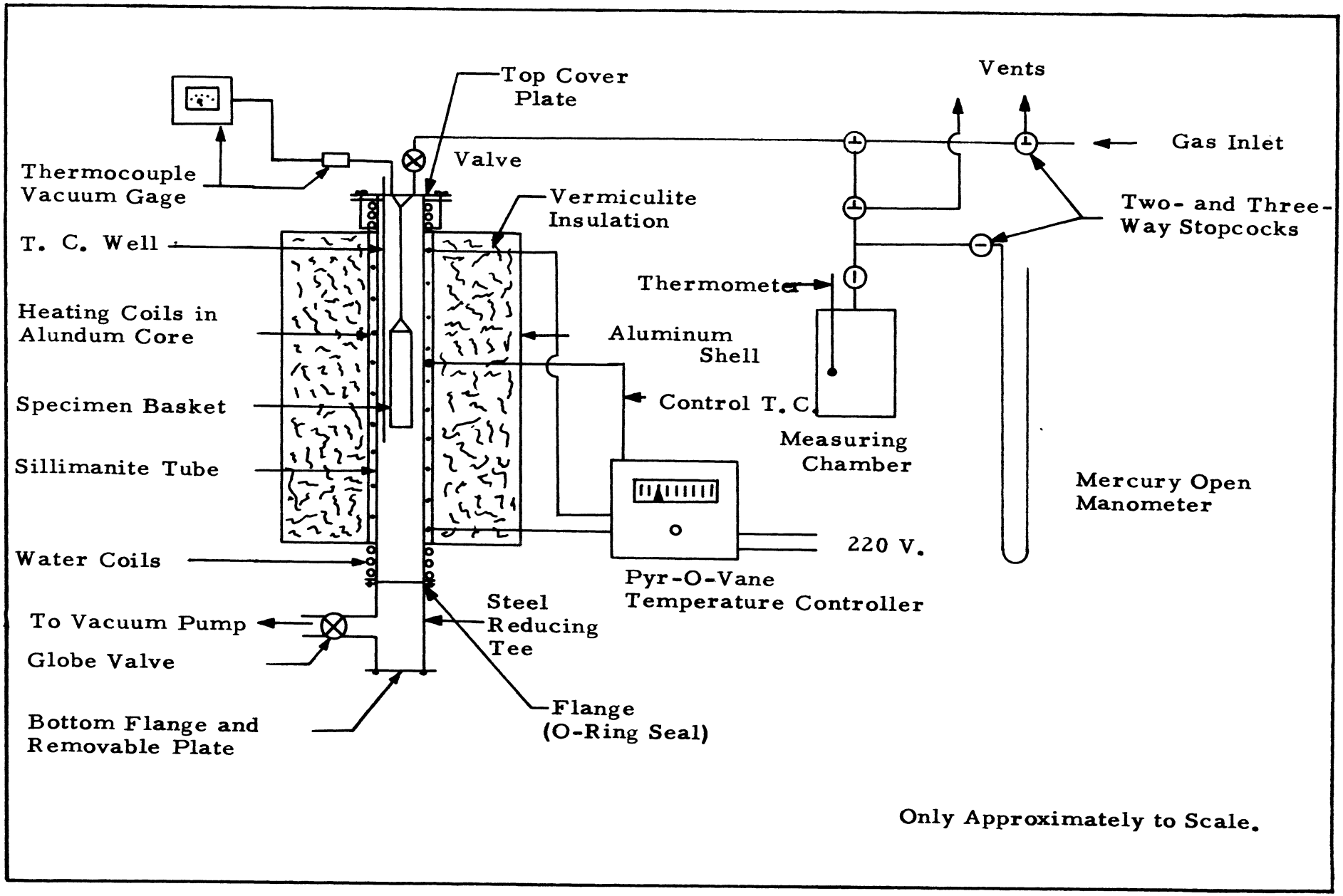


Figure 19. Schematic Diagram of Vacuum Treatment Furnace.

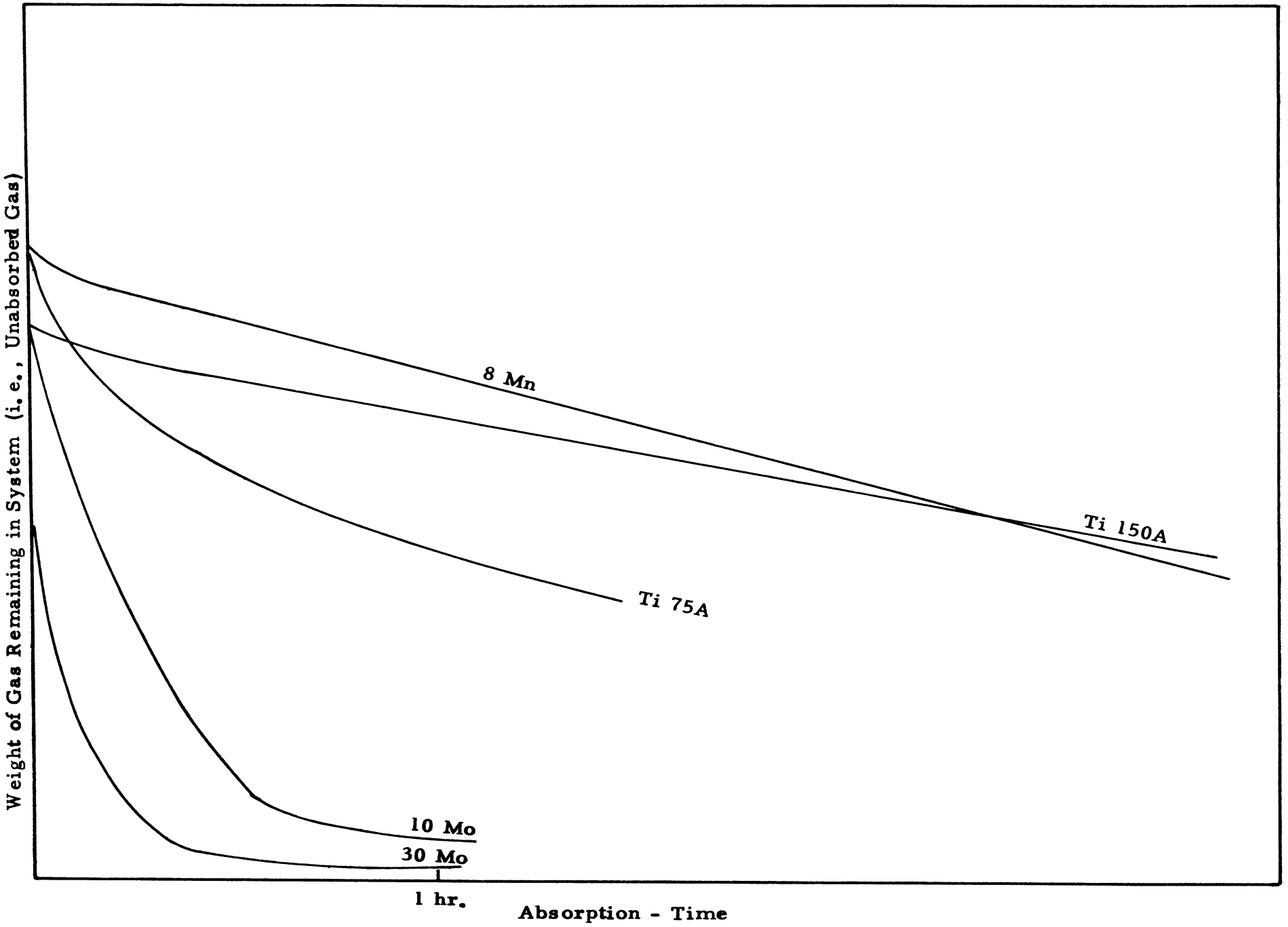


Figure 20. Schematic Diagram of Effect of Alloy Type on Rate of Hydrogen Absorption at 1500°F

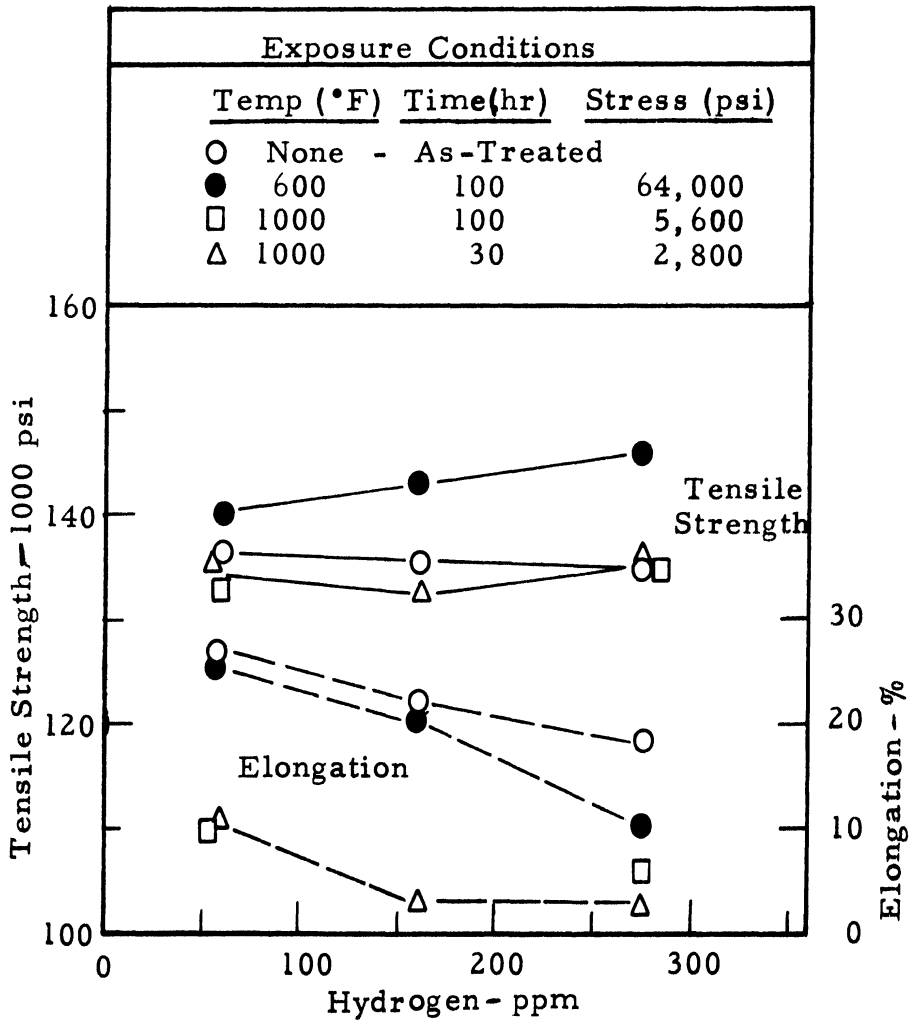


Figure 21. Influence of Hydrogen Content on Tensile Properties at Room Temperature for Alpha-Beta Alloy Ti 150A after Exposure to 600°F and 1000°F under Stress.

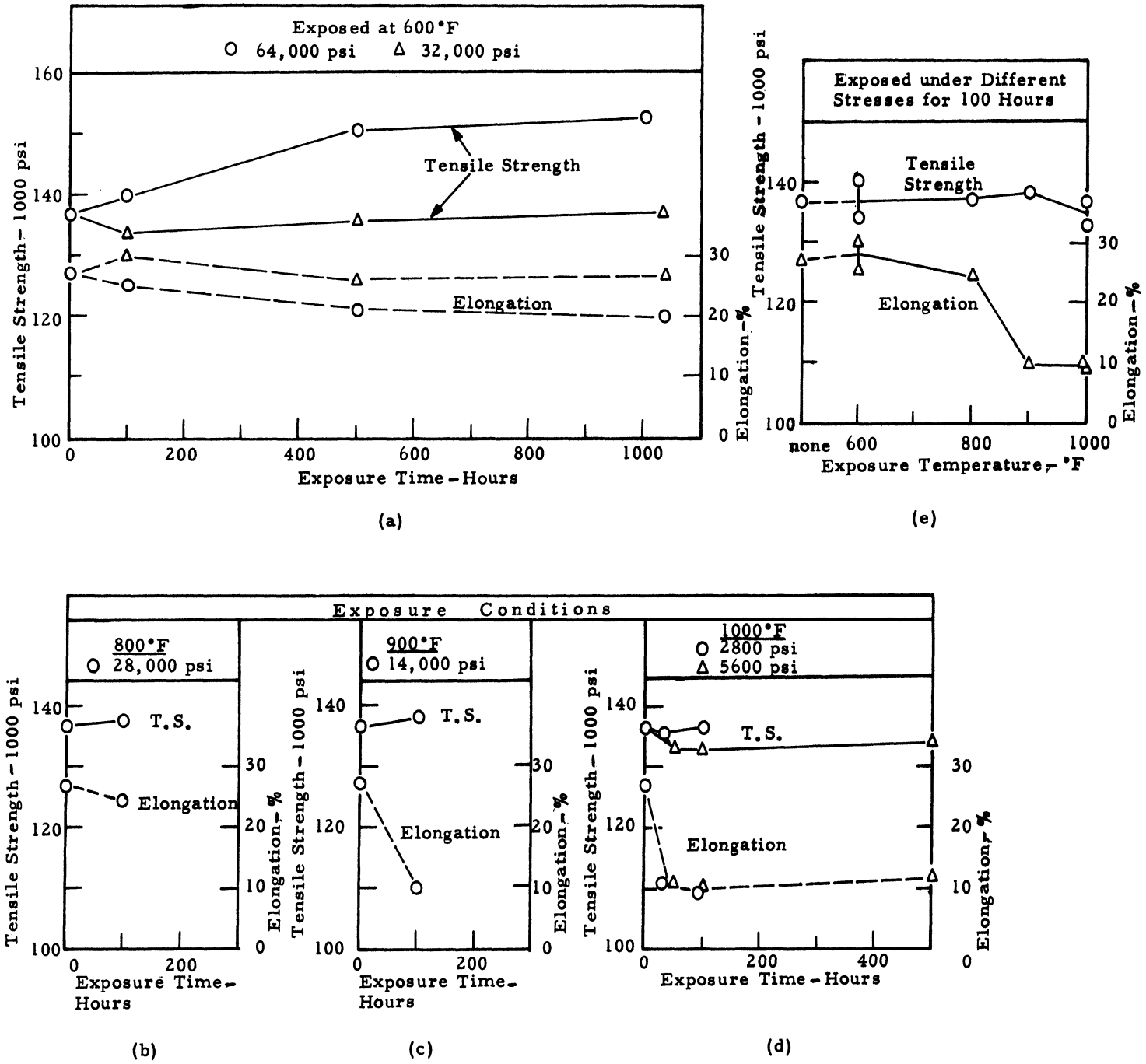


Figure 22. Influence of Exposure Temperature, Time and Stress on the Tensile Properties at Room Temperature of Alpha-Beta Alloy Ti 150A with 60 ppm of Hydrogen.

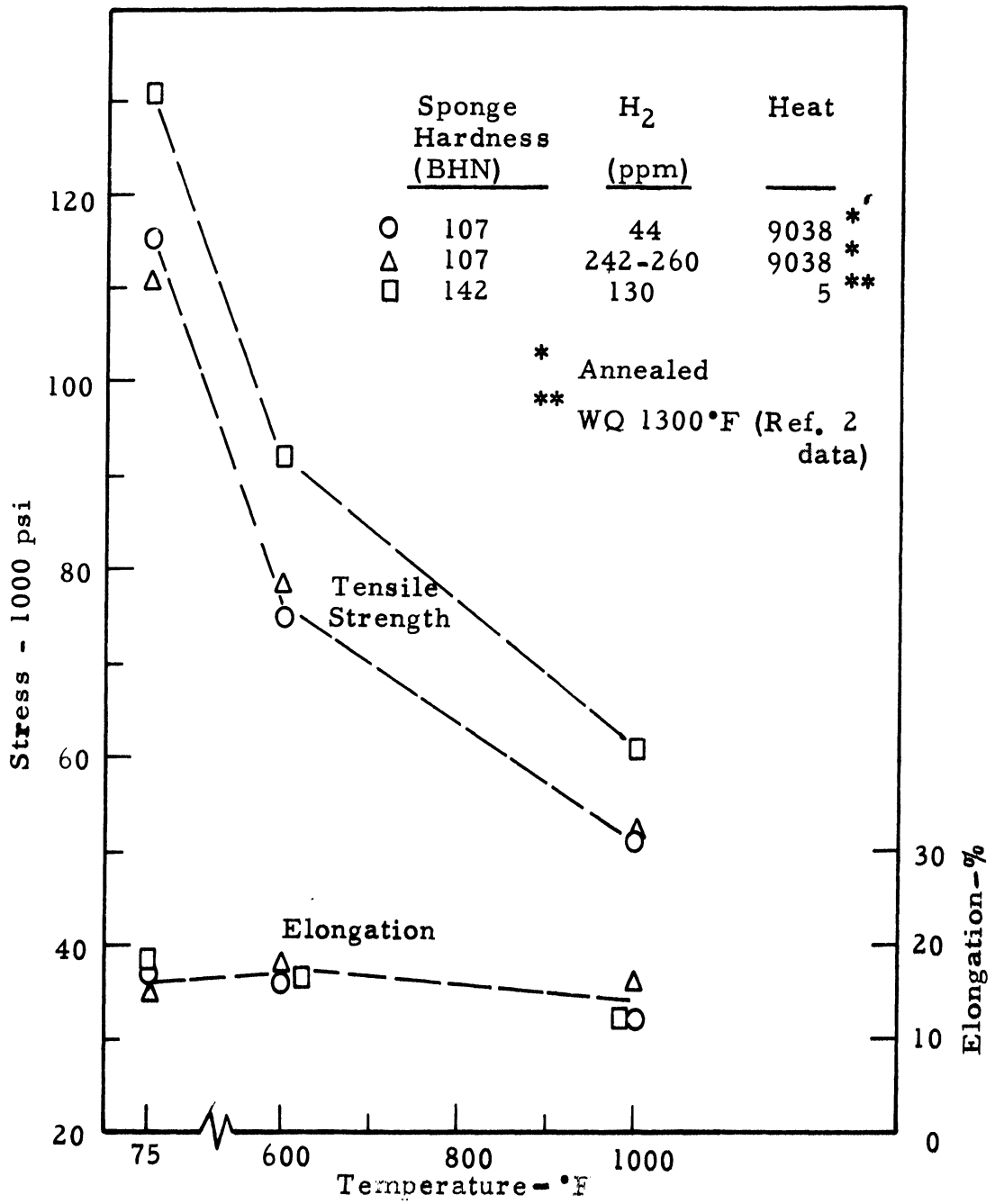


Figure 23. Influence of Hydrogen Content on Tensile Properties of 10 Mo Alloy Made with 107 BHN Titanium Sponge.

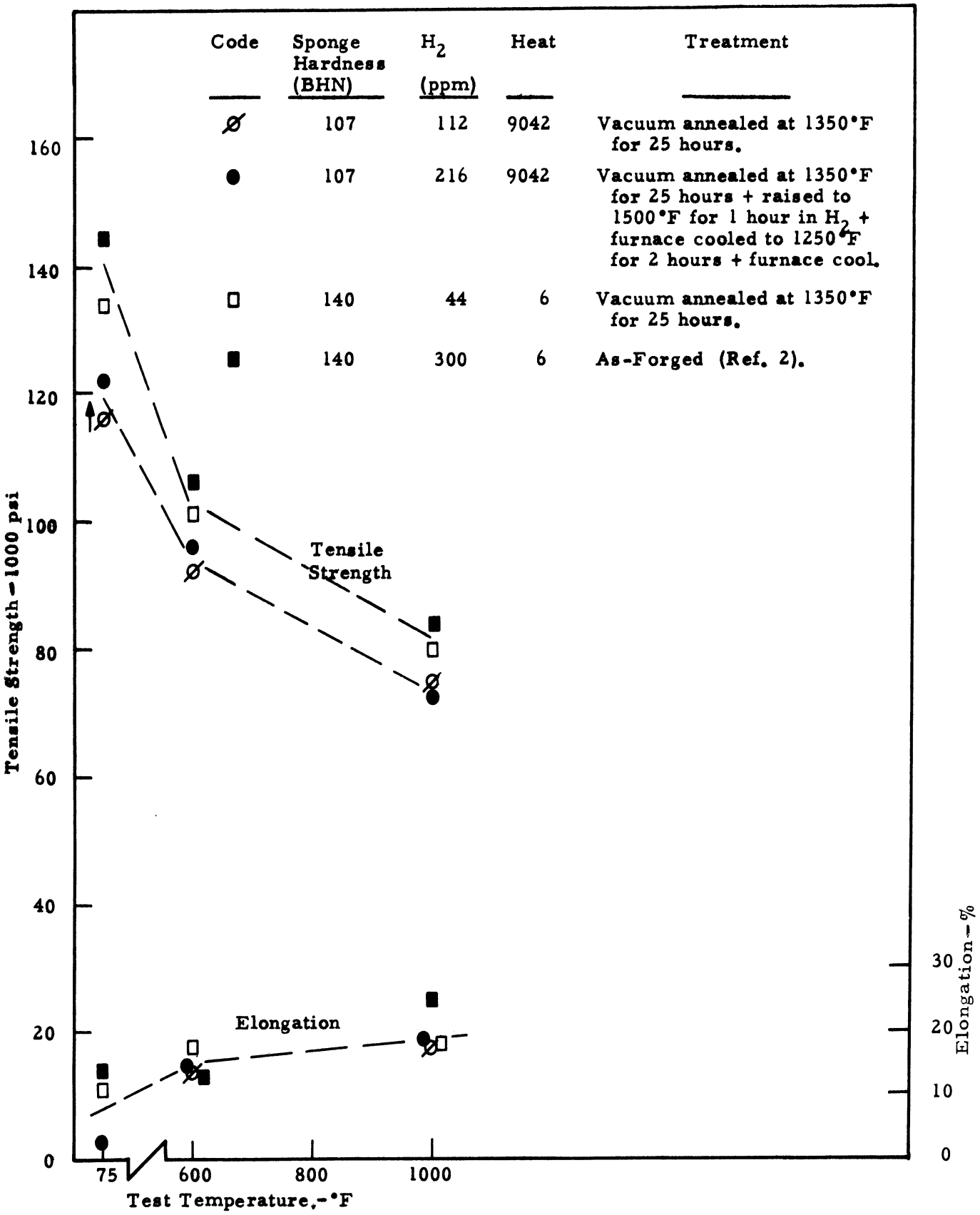


Figure 24. Influence of Hydrogen Content and Sponge Hardness on Tensile Properties of Stable Beta Alloy 30 Mo.

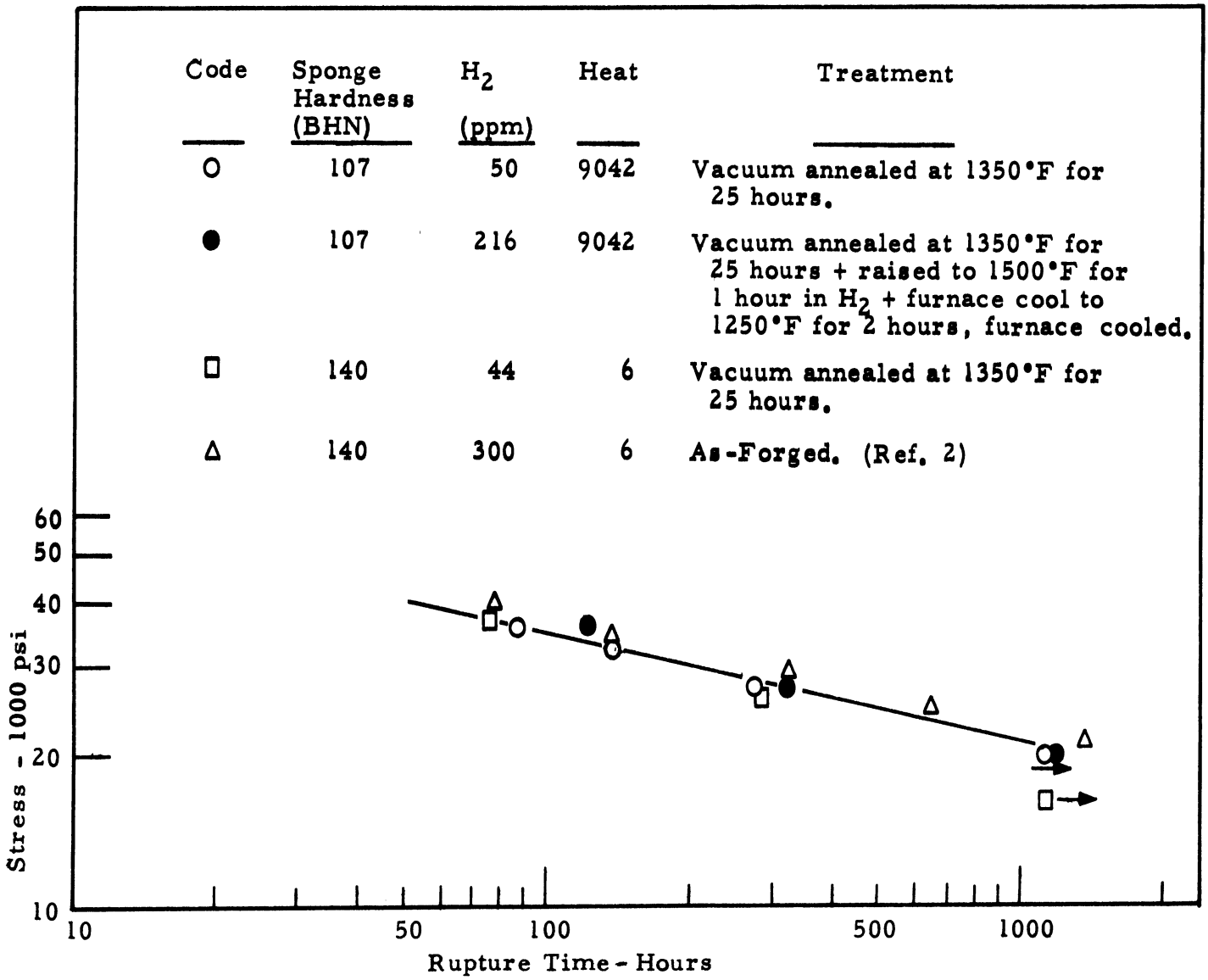


Figure 25. Influence of Hydrogen Content and Sponge Hardness on the Comparative Stress-Rupture Time Curves at 1000°F for Stable Beta Alloy 30 Mo.

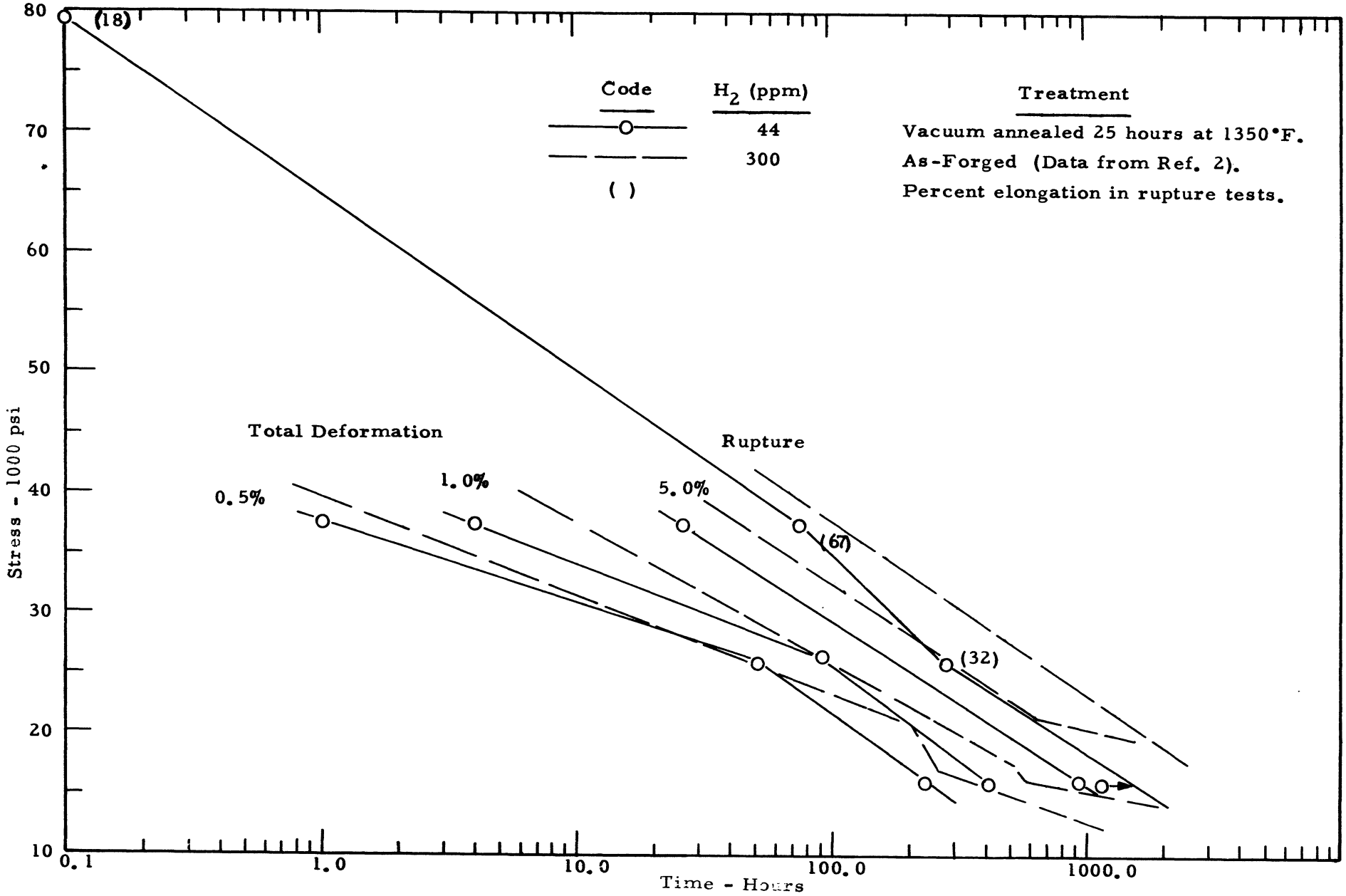


Figure 26. Influence of Hydrogen Content on Stress-Time for Total Deformation Characteristics at 1000°F for Stable Beta Alloy 30 Mo Made with 140 BHN Titanium Sponge.

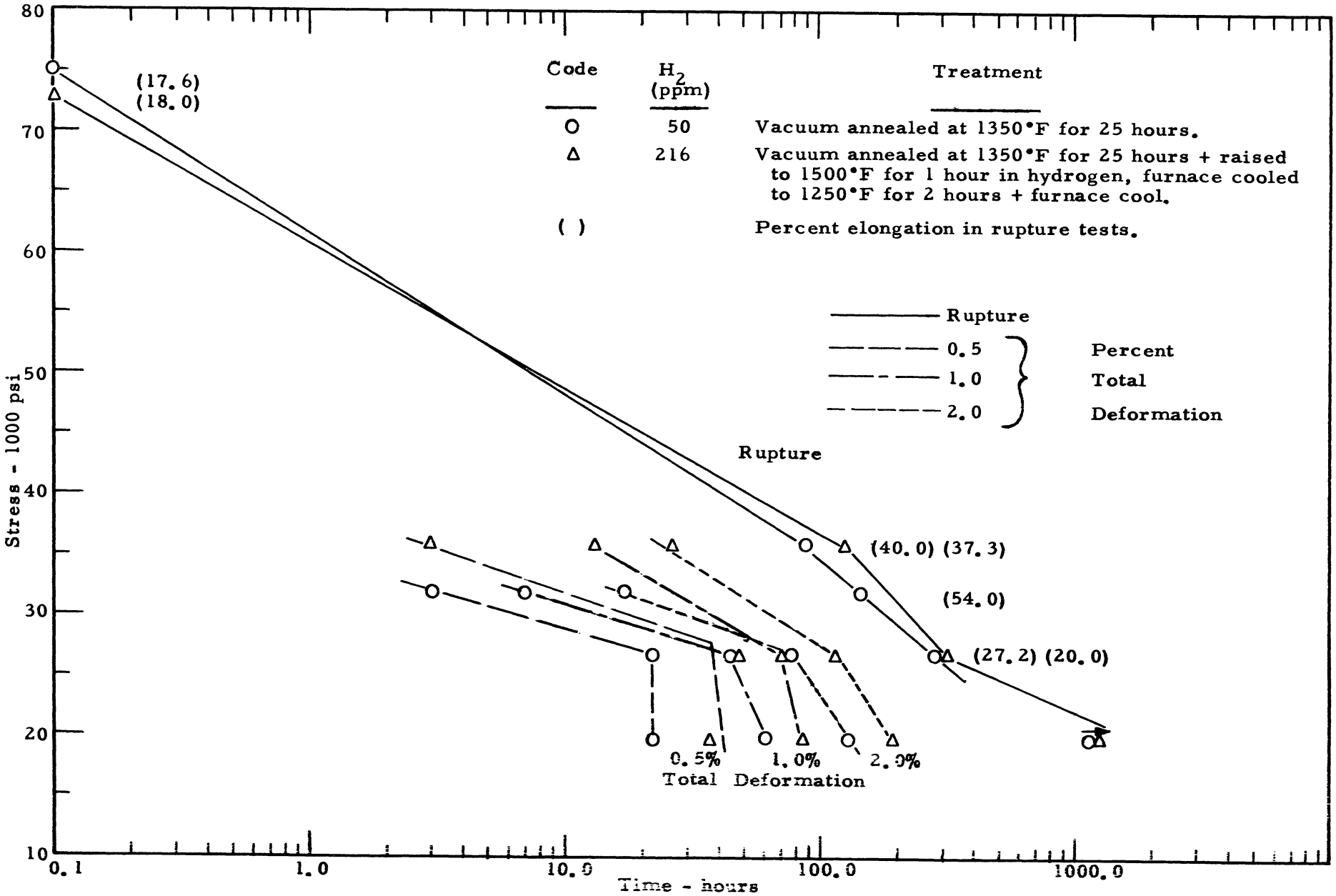


Figure 27. Influence of Hydrogen Content on Stress-Time for Total Deformation Characteristics at 1000°F for Stable Beta Alloy 30 Mo Made With 107 BHN Sponge Titanium.