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# EIGHTH QUARTERLY PROGRESS REPORT

TO

MATERIALS LABORATORY, WRIGHT AIR DEVELOPMENT CENTER

DEPARTMENT OF THE AIR FORCE

ON

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

By

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covering the period from

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## SUMMARY

This report, the eighth progress report to be issued under Contract No. AF 33(616)-244, covers the period from March 1, 1955 to May 31, 1955.

During this period work continued on the Second Summary Report on the contract which covers the period from November 1, 1953 to February 28, 1955.

The present progress report discusses the work to be carried on under a recent extension of the contract. The new work is to be principally concerned with a study of the effects of contaminents on the elevated temperature properties of titanium alloys. For these studies, a vacuum annealing and hydrogenation furnace was designed and constructed in order that control of hydrogen could be effected.

The initial operation and calibration of this furnace is described. In addition, limited data are presented covering a study of the effects of cold working on the 1000°F properties of an alpha alloy, Ti-6% Al-1/2% Si.

## INTRODUCTION

This report is the eighth progress report to be issued under Contract AF 33(616)-244 and covers the period from March 1, 1955 to May 31, 1955.

The work described in this report is the third phase of the research undertaken on titanium and its alloys in the temperature range to 1000°F. Under the first phase of the contract (1) four titanium alloys, representing typical and different transformation systems, were studied. Survey procedures were developed to study the tensile, rupture and creep properties.

The second phase of the work (2) extended this work in the following ways:

1. Creep and total deformation studies were extended to 1000 hours for materials previously studied.

2. The general validity of the result for each type of alloy was studied by surveying another material of the same alloy type; however, containing a different alloying element or elements.

3. A study of the effect of hot working variables was carried out on two alloys.

In addition, special studies were undertaken on the effects of exposure to test conditions and the effect of hydrogen content.

As a result of the preceding studies, a third phase of the work has been initiated under the present extension of the contract.

The additional research has been broken down to three major components. The first is a continuation as warranted of the study of effects of structure and/or processing. The second considers the effects of various contaminents on properties. The third covers some effects of structural instability.

In outline form, the anticipated work is as follows:

General Effects of Structure

a. Additional studies of cold worked alpha alloys.

b. Effects of commercial stabilization treatments on properties.

Effects of Contaminents

a. Effect of sponge hardness

b. Effect of hydrogen on long-time creep properties

c. Mechanism of room temperature embrittlement after exposure to elevated temperature testing.

Effect of Structural Instability

a. Correlation of negative creep phenomena and tensile embrittlement with transitional structures.

The test program has been set up so that the different phases of the above program complement one another. The alloys to be studied under each phase are indicated by (x) in the tabulation on the next page.

| Test Program | Material | | | | |
|---|---|---|---|---|---|
| | Ti-6% Al | Ti-30% Mo | Ti-8% Mn | Ti-10% Mo | Ti 150A |
| Sponge Hardness | (from previous data) | x | x | - | - |
| Hydrogen on Creep | x | x | x | x | - |
| Embrittlement | - | - | x | - | x |
| Structural Stability | - | - | x | - | - |

A more detailed discussion of the work follows.

## General Effects of Structure

As the work accomplished under the second phase of the contract is still being correlated, a detailed test program has not been set up. It is intended that supplementary work will be carried on as warranted by the results of the previous studies. Two topics presently appear to justify additional work. First, it is felt that additional work be done on cold-worked conditions of an alpha alloy, Ti-6Al-1/2 Si. Previous work on a Ti-6%Al Alloy indicated that cold working greatly lowered the stress required to reach a given total deformation in a given time period. Additional work on the Si modification of this alloy would indicate whether this effect was carried over.

A second field for additional work is the testing of Ti 155 AX in the stabilized conditions currently recommended for commercial practice. Such stabilization involves long-time heating at 1200°F. A survey test of such a condition showed no change in the 100-hour rupture properties over the as-produced conditions. However, it is possible that longer time testing might reveal some differences.

It is also felt that additional metallographic and X-ray studies may prove desirable in a continuation of a study of the effects of test conditions on structure of a number of alloys.

Effects of Contaminents

### Effect of Sponge Hardness

A convenient measure of the quality of the sponge material from which titanium alloys are prepared is its hardness. The hardness of titanium sponge is a function of its purity; the principal non-metallic contaminents being oxygen, hydrogen, nitrogen and carbon. Of these, only hydrogen can be conveniently removed by subsequent treatments.

In the present investigation, a survey is planned to delineate some of the effects of initial sponge hardness on the high temperature properties of titanium alloys. Previous results will be utilized in the case of an alpha alloy, Ti-6% Al, while new heats of low sponge hardness will be obtained of an alpha-beta alloy, Ti-8% Mn, and a beta alloy, Ti-30% Mo. Material for high sponge hardness alloys will be made of approximately 140 BHN sponge, while 107 BHN sponge will be used for preparation of low hardness alloys.

### Effect of Hardness on Creep

Under the second phase of the contract very limited studies were made of the effects of hydrogen on the creep-rupture properties of Ti-6% Al together with some studies of embrittling effects felt due to hydrogen in Ti-150A. This work indicated the need for an extensive study of the effects of hydrogen on the long-time creep properties of titanium alloys.

An alpha alloy, Ti-6% Al, an alpha-beta alloy, Ti-8% Mn, and a beta alloy, Ti-30% Mo, made from low hardness sponge will be used for these studies. Each material is to be tested with two hydrogen contents, approximately 50 ppm and 250 ppm. Initially, an annealed condition is to be tested for each material. Control of hydrogen content is to be effected by suitable vacuum annealing or hydrogenation treatments.

## Mechanism of Hydrogen Embrittlement

Studies under the second phase of the contract revealed that Ti 150A containing an appreciable amount of hydrogen was embrittled on room temperature tensile tests following exposure to creep test conditions. Conversely, a Ti-Mn alloy containing a low amount of hydrogen did not show this effect.

It is planned to study the mechanism by which such embrittlement occurs utilizing Ti 150A and a Ti-8% Mn alloy as test materials. Hydrogen levels of 50 ppm, 250 ppm, and 500 ppm are to be considered and initial testing will be confined to the hot rolled and annealed condition. The general procedure will be confined to the hot rolled and annealed condition. The general procedure will be to expose samples to stress-time-temperature conditions followed by a room temperature tensile test. A series of times are to be used in order to establish the time required for embrittlement. After establishment of conditions for embrittlement, metallurgical studies are to be made to delineate the mechanism by which it occurs.

## Effects of Deformation on Structural Stability (3)

In the course of another investigation it was observed that negative creep phenomena and embrittling tendencies were encountered at moderate test temperatures for a Ti-Mn alloy. These effects are believed due to unstable beta breaking down to omega phase and subsequently to alpha. Detailed work (4) had been done elsewhere on the nature of the reactions involved. In the present study it is intended to study the effects of time and deformation (i.e. test conditions) on such a meta-stable structure. The material to be studied is a Ti-8% Mn of low sponge hardness and low hydrogen content. Conditions known to be meta-stable are to be exposed to time and temperature either stressed or unstressed. Subsequent tensile and hardness studies will be used to delineate the effects of deformation on the structure.

## TEST MATERIALS

Special heats of the alloys to be studied under this contract are to be prepared by the Armour Research Foundation of Chicago, Illinois using a double melting technique (1).

The following alloys have been received or have been shipped:

From 107 BHN Sponge

| Nominal Composition | Actual Composition | Amount |
|---|---|---|
| Ti-10% Mo | Mo 10.47% C 0.051% $N_2$ 0.0034% | 5 lb. |
| Ti-6% Al | Al 6.13% (avg.) C 0.044% $N_2$ 0.045% | 7 lb. |

The following alloys are still on order:

| Nominal Composition | Amount |
|---|---|
| Ti-30% Mo | 10 lb. |
| Ti-8% Mn | 40 lb. |

From 140-150 BHN Sponge

The following alloy is still on order:

| Nominal Composition | Amount |
|---|---|
| Ti-8% Mn | 20 lb. |

The material is to be furnished as-forged to 1/2-inch round bars with the exception of the Ti-8% Mn alloy. This alloy is to be procured as 3/4-inch square bars and will be further worked at the University of Michigan in the alpha-beta phase region.

## EQUIPMENT

In order that control of hydrogen content of titanium alloys could be accomplished, a furnace was constructed during the period covered by this report for either vacuum annealing or hydrogenation treatments.

A schematic diagram of the furnace arrangement is shown in Figure 1.

Essentially the equipment consists of three sections: the heating section; vacuum chamber; and gas supply section.

Heating is accomplished by a resistance furnace. The heating elements are wound from Chromel-A wire and baked into an alundum core. Vermiculite insulation is used between the core and the furnace shell. Control is accomplished by a Pyr-O-Vane controller and "on-off" control is used.

The treatment chamber consists of a sillimanite tube placed within the furnace core. Sealing of the chamber is accomplished with O-rings with flanged covers. As the chamber is evacuated, atmospheric pressure forces the O-rings against the flanges, thus insuring a tight seal. The lower end of the vacuum chamber has been constructed so that a cover plate may be removed (after breaking the vacuum with an inert gas) and samples can be dropped out the bottom. Since specimens under treatment are hung in a stainless steel basket this is accomplished by melting the wire from which the basket is hung. Suitable power leads have been provided in the upper flange so that high amperage current may be introduced for this purpose. A MegaVac mechanical vacuum pump (ultimate vacuum 0.1 micron) is used for evacuation. Fine pressure measurement is accomplished with a thermocouple vacuum gage and coarse measurement is possible with a U-tube manometer. It is generally possible to pump below the lower limit of sensitivity of the thermocouple gage (approximately 1 micron) in less than five minutes.

The gas supply section of the system has been constructed so that a quantity of gas can be introduced into a measuring chamber and then transferred to the vacuum chamber. The volume of the measuring chamber has been determined and thus the gas quantity can be determined from a simple gas law calculation knowing the pressure and temperature. Vents have been provided so that excess gas may be withdrawn using an auxiliary pump. Arrangements have also been provided for drying and purifying gas prior to its introduction to the system.

# RESULTS AND DISCUSSION

## Second Summary Report

During the time period covered by this report, work continued on preparation of a Summary Report covering the second phase of the contract, November 1, 1953 to February 28, 1955.

## Effect of Cold Working on Ti-6% Al-1/2% Si

As indicated in the introduction, additional work has been undertaken on the 1000°F creep-rupture properties of cold worked Ti-6% Al-1/2% Si, an alpha alloy. The aim of this study is to compare the rupture and total deformation properties of as-forged or approximately 20% cold worked material for time periods up to 1000 hours. Survey work on this alloy (2) had indicated that this amount of cold working improved the tensile properties, but that the cold worked material was subject to a rapid loss of strength and accompanied by rapid creep.

The original tests were carried out on material cold worked 19%. A second batch of material was cold rolled using the same roll settings, however, the cold reduction was 21.6%. The slight amount of additional reduction did not appear to affect the strength to an excessive extent.

Test results obtained to date are summarized in Table I. An additional test was run on the as-forged condition in order to confirm the 100-hour rupture strength. A plot of stress versus rupture time is presented in Figure 2. Figure 3 shows approximate curves of time to reach given total deformations. Figure 4 indicates the stress-creep rate relationships.

Figure 2 shows that the cold worked condition was stronger up to about 100 hours, however, an instability exists that causes a weakening over longer time periods. Creep data show greater deformation occurs in a given time for the cold worked condition. Survey work (2) indicated that a recrystallization process takes place under this condition at 1000°F

Additional creep and rupture tests of these conditions will be run in order to better establish the effects of stress and time on the 1000°F properties. In addition, metallographic studies will be made of the completed test specimens.

Tests of Vacuum Annealing Furnace

Several test runs were made with the vacuum annealing furnace using Ti 75A stock (commercially pure titanium) of known hydrogen content. The purpose of these runs was three-fold:

1. Establish operational procedures
2. Check the leak rate of the system
3. Test the efficiency of hydrogen removal

An initial run was made at temperatures somewhat below those intended for actual hydrogen removal and for a somwhat longer time. A sample of Ti 75A was held at 750°F for 13 hours and then raised to 950°F for 25 hours. Following this it was furnace cooled, taking approximately 10 hours to reach room temperature. The system was evacuated throughout the run to a pressure below the limit of sensitivity of the thermocouple gage.

A second run was made using the operating conditions intended for treatment of the alloys under study. Five samples of Ti 75A were annealed for 24 hours at 1200°F and then furnace cooled in vacuum.

Hydrogen analyses(given as parts per million by weight) of the samples are as follows:

| | Detn. 1 | Detn. 2 |
|---|---|---|
| As-Produced (Not annealed) | 87 ppm (analysis furnished by WADC) | |
| | 83 ppm | 84 ppm (as analyzed at U. of M.) |
| 13 hours 750 plus 25 hours 950 | 81 ppm | 80 ppm |
| 24 hours 1200°F | 56 ppm | 52 ppm |

The analyses performed at the University of Michigan were run in a vacuum fusion analysis apparatus. A vacuum extraction technique (heating to 1400°C for

20 minutes at .01 micron) was employed.

All samples were weighed before and after the annealing treatment. It was assumed that almost quantitative absorption of oxygen and nitrogen would occur and thus the weight change would be a measure of effective leak rate of the system.

The weight gains measured were as follows:

| | |
|---|---|
| 13 hours 750°F plus 25 hours 950°F | 0.0136% (wt) |
| 24 hours 1200°F | 0.0069% (wt) |

After taking into consideration the length of time at temperature, the effective dynamic leak rate of the system was calculated to be of the order of 1 to 3 x $10^{-8}$ liters of air (STP) per second. This dynamic value was checked by timing the pressure rise occurring when the vacuum pump cut-off valve was shut. Under the static conditions the leak rate was equivalent to 4 x $10^{-6}$ liters per second. The agreement between the static and dynamic values is considered good.

The contamination from the 24 hour run at 1200°F was also considered from the basis of surface presented. The weight gain of 0.0069% was found to be equivalent to 0.091 mg/cm$^2$. This value was not considered to be excessive in relation to the typical initial oxygen contents of titanium alloys, i.e. approximately 0.1% (wt).

A micro-hardness traverse of several of the samples revealed that no significant change of hardness occurred. All samples are to be treated as 1/2-inch rounds. Since the final specimen size as-machined is 1/4-inch it is seen that a factor of safety exists to minimize any possible contamination problems.

On the basis of these results, it was concluded that operating conditions of 1200° - 1300°F for 24 hours were satisfactory for producing low hydrogen material of minimum contamination.

# 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. Op. Cit. Ref (1) - Part II (to be published in 1955).

3. Gluck, J. V. and Freeman, J. W. "A Study of Creep of Titanium and Two of Its Alloys" (forthcoming WADC Technical Report).

4. Robinson et al "Precipitation Hardening and Embrittlement of High-Strength Titanium Alloys" WADC Technical Report 54-355 (December 1954).

# TABLE I

## Creep-Rupture Test Data for Ti-6% Al-1/2% Si Alloy

| Condition | Test Temp. | Stress (psi) | Rupture Time (hours) | Elongation ( % in 1 in) | Reduction of Area (%) | Loading Def. (%) | Time to Reach Indicated Def. 0.5% | 1% | 5% | Minimum Creep Rate (in/in/hr) |
|---|---|---|---|---|---|---|---|---|---|---|
| As-forged | 1000 | 74,800 | Tensile Test | 11.7 | 22.4 | -- | -- | -- | -- | -- |
| | | 50,000 | 41.1 | 14.3 | 31.4 | .47 | 0.5 | 4 | -- | $1.4 \times 10^{-3}$ |
| | | 45,000 | 97.8 | 22.0 | 22.2 | -- | -- | -- | -- | -- |
| | | 36,000 | 473.9 | 15.0 | 26.9 | .34 | 4 | 15 | 330 | $1.2 \times 10^{-4}$ |
| | | 30,000 | >986.1 | (5.0)* | (7.9)* | .27 | 15 | 105 | 980 | $2.8 \times 10^{-5}$ |
| | | 20,000 | In progress | -- | -- | .19 | ~900 | | | $1.3 \times 10^{-6}$ |
| 19% | 1000 | 92,400 | Tensile Test | 11.7 | 34.9 | -- | -- | -- | -- | -- |
| | | 52,000 | 48.4 | 29.1 | 49.5 | .55 | -- | 14 | -- | $3.3 \times 10^{-4}$ |
| | | 44,000 | 126.0 | 35.6 | 53.5 | .48 | 0.5 | 5 | 46 | $7.7 \times 10^{-4}$ |
| 21.6% | 1000 | 44,000 | 145.6 | 33.0 | 32.2 | .47 | -- | -- | -- | -- |
| | | 32,000 | 393.4 | 30.0 | 46.4 | .33 | 4 | 16 | 180 | $2.1 \times 10^{-4}$ |
| | | 20,000 | In progress | -- | -- | .20 | 30 | 450 | -- | $7.6 \times 10^{-6}$ |

> = greater than; test stopped without rupture.
()* = permanent deformation on cooling.

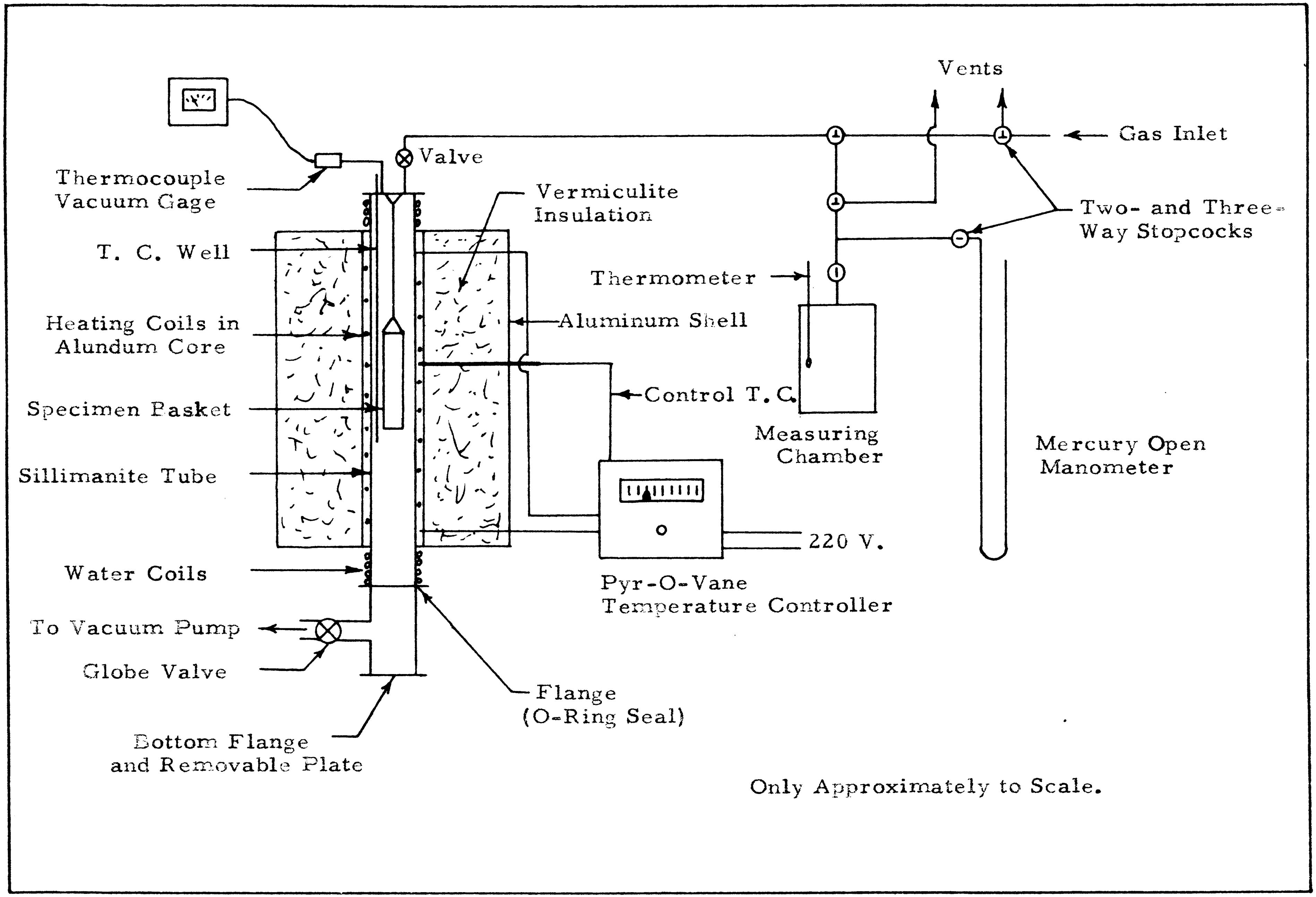


Figure 1. - Schematic Diagram of Vacuum Treatment Furnace.

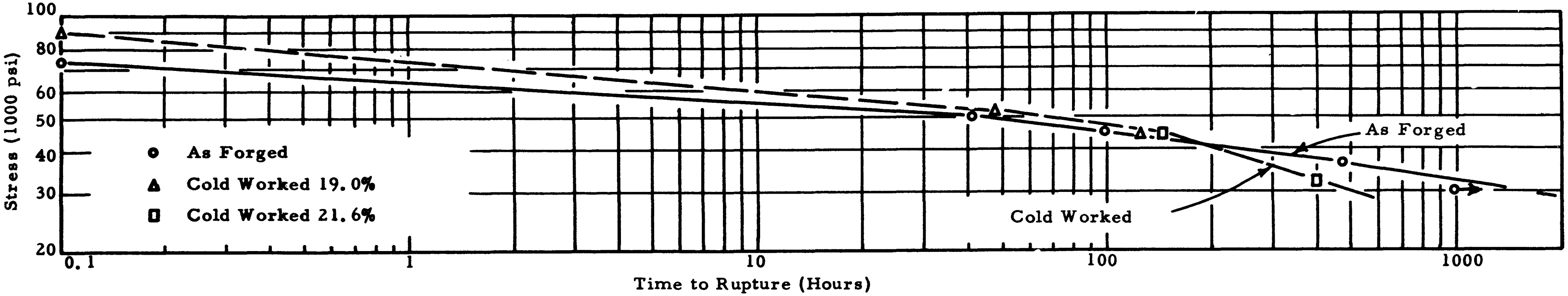


Figure 2. - Stress vs. Time to Rupture for Ti-6 Al-1/2 Si Alloy at 1000°F.

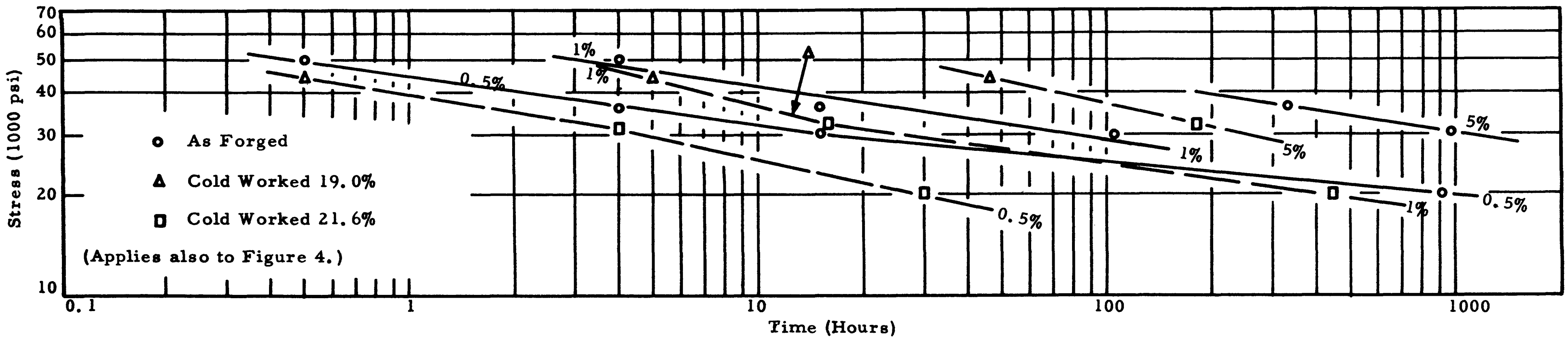


Figure 3. - Stress vs. Time to Reach Indicated Total Deformation for Ti-6 Al-1/2 Si Alloy at 1000°F.

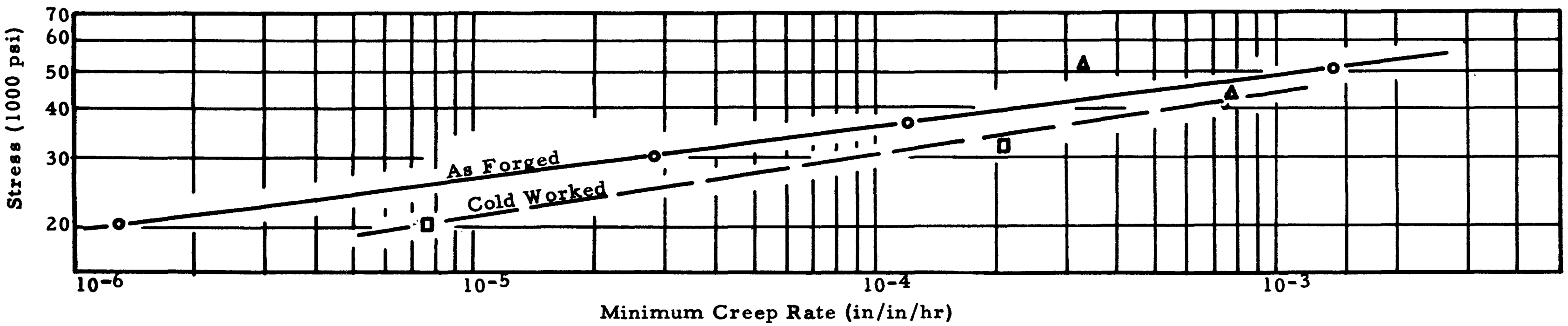


Figure 4. - Stress vs. Minimum Creep Rate for Ti-6 Al-1/2 Si Alloy at 1000°F.