ENGINEERING RESEARCH INSTITUTE
UNIVERSITY OF MICHIGAN
ANN ARBOR, MICH.

NINTH 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

J. V. Gluck

J. W. Freeman

Contract No. AF 33(616)-244
Supplemental Agreement No. S9 (55-1113)
Task No. 73510

Project 2076

covering the period from

June 1, 1955 to August 31, 1955

SUMMARY

This report, the ninth to be issued under Contract AF 33(616)-244, covers research carried out on the elevated temperature properties of titanium alloys during the period from June 1, 1955 to August 31, 1955.

During this period almost all of the test stock for study under the present program was received. This program is principally concerned with effects of contaminents on properties and the relative stability of various structures.

Vacuum annealing was carried out on a number of materials in order to produce material of low hydrogen content. In addition, hydrogenation tests were carried out on Ti 75A and Ti-10%Mo alloys to produce desired higher hydrogen contents. The study of the effects of cold working on the 1000°F properties of a Ti-6%Al-1/2%Si alloy was brought almost to completion. The study of the effect on room temperature tensile properties of exposure of Ti 150A to creep test conditions was started. The initial tests covered material of low hydrogen content and the effects of stressed exposure at 600°F and 1000°F.

INTRODUCTION

This report is the ninth progress report to be issued under Contract No. AF 33(616)-244 and covers research carried on during the period from June 1, 1955 to August 31, 1955.

The work herein described is a continuation of research into the properties of titanium and its alloys at temperatures up to 1000°F. Under one phase of this contract a number of titanium alloys, representing typical and different transformation behavior, were surveyed in order to develop information regarding the effects of structure and treatment on elevated temperature properties. Another phase of the investigation covered a study of the deformation properties for selected treatments of several alloys for time periods up to 1000 hours. Other research included a study of the effect of hot working conditions, and brief studies of the effects of hydrogen content and the effects of exposure to creep test conditions.

The present extension of the contract has been broken into three main divisions as outlined below.

<u>General Effects of Structure</u> (Continuation of previous work as warranted)

a. Effect of cold working on alpha alloys

b. Effects of commercial stabilization treatments

<u>Effect of Contaminents</u>

a. Relative effects of sponge hardness

b. Effect of hydrogen on long-time creep properties

c. Mechanism of room temperature embrittlement after exposure to creep test conditions

Effect of Structural Instability

a. Effect of instability on properties of transitional structures

The alloys to be studied under each phase of the program are indicated by (x) in the following tabulation:

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

A detailed discussion of each phase of the work was included in the Eighth Progress Report (1).

TEST MATERIALS

The analysis of all materials to be investigated under this contract is given in Table 1. All test materials were received by the end of the period covered by this report. However, forging cracks in the as-received material rendered about half the Ti-6%Al stock (Heat 4c) useless for machining to creep test specimens. Consequently, an additional heat of this material is being made by the Armour Research Foundation. The analyses given in Table I were made by the producer with the exception of the hydrogen analyses. Where available, the analysis of the sponge used to produce each heat is given.

RESULTS AND DISCUSSION

Operation of Vacuum Annealing Furnace

A number of runs were made with the vacuum furnace during the period covered by this report. Details of the design and construction of the apparatus were discussed in the Eighth Progress Report (1). A redetermination was made of the temperature distribution within the furnace and it was discovered that the nominal annealing temperature reported as 1200°F was actually 1350°F. Since this temperature did not result in contamination of the test stock (measured as weight gain) it was decided that standard vacuum annealing conditions for removal of hydrogen would continue to be 24 hours at 1350°F. Throughout this period pumping was carried on to a pressure below the limit of sensitivity of the thermocouple gage. A standard run size of 8 or 9 pieces was adopted. The pieces were one-half inch diameter rounds, three inches long. A blank slug of the same diameter and approximately one inch long was included with each run to furnish the samples for hydrogen analysis. At the conclusion of the run the blank slug was machined to a one-fourth inch square bar and then cut into cubes. Two random cubes were analyzed for each annealing run.

The results of annealing runs on four alloys are tabulated in Table 2. The final hydrogen content showed generally good agreement both between duplicate runs on the same materials and between the materials themselves. In addition, the agreement between the two random samples from each run was excellent. The initial hydrogen content of the various alloys ranged as high as 310 parts per million (by weight) and the vacuum annealing treatment produced final hydrogen contents averaging 48 parts per million with a deviation of plus or minus 15 parts per million.

During this period the furnace was also operated as a hydrogenation apparatus. A test run was made on a batch of Ti 75A.

With the information furnished from the test hydrogenation of Ti 75A, a run was planned to achieve a final hydrogen content of 250 parts per million in a batch of Ti-10% Mo alloy. The batch of 9 pieces was first vacuum annealed for 24 hours at 1350°F. Following this, the furnace temperature was raised to 1500°F and a calculated amount of hydrogen was added to the reaction chamber (which was isolated from the pumping system, of course). A hydrogen content of 50 parts per million was assumed as a starting point for hydrogenation and the additional hydrogen requirement was calculated from this value.

After absorption for 1 hour at 1500°F, the furnace was allowed to cool to 1250°F and held for one hour in order that structural equilibrium might be approached. Finally, the run was furnace cooled to room temperature. Throughout the period readings were made of the pressure within the reaction chamber. From these data it was determined that further absorption had ceased at the conclusion of the holding period at 1500°F.

The expected final hydrogen content of the metal was calculated from the difference between the weight of hydrogen added and that measured as the final pressure at the end of the run. For the run in question, it was expected that the final hydrogen level would be 265 parts per million. Analysis gave a value of 260 parts per million as the average of two determinations (see Table II).

Since the performance of this run several others have been made. Hydrogen analyses are not yet available, however, it appears that the rate of absorption is dependent to some extent on the material being treated. Materials of predominantly beta phase have shown much more rapid hydrogen absorption than did Ti 150A, a mixed alpha-beta structure. In order to reach what was considered a satisfactory final pressure during a run on a batch of Ti 150A, it was necessary to increase the absorption time to as much as 4 hours. On the other hand, a batch of Ti-30% Mo (stabilized beta structure) achieved almost complete absorption in less than one-half hour. These observations should be recognized as being purely qualitative since no effort has been made to determine accurately the kinetics of the absorption process.

Mechanism of Embrittlement After Elevated Temperature Exposure

As one phase of the general program on the effects of contaminents on the properties of titanium alloys a study is being made of the effect of elevated temperature test conditions on the room temperature tensile properties of both Ti-150A and a Ti-8% Mn alloy. A brief, previous study had shown that Ti 150A containing an appreciable amount of hydrogen showed room temperature embrittlement after creep test exposure while a Ti-8% Mn alloy having a low amount of hydrogen did not show this effect.

In the present program both materials in the annealed condition are to be exposed to stress, time, and temperature. Three levels of hydrogen, approximately 50 ppm, 250 ppm, and 500 ppm, are to be considered. Exposure is to be made at stresses of approximately 40 and 80% of the 1000-hour rupture strength at the temperature under consideration. The initial tests are to be for 100 hours and the subsequent exposure times are adjusted in order that the time for embrittlement might be established. Following this, metallographic and X-ray studies are planned in order that the mechanism by which such embrittlement occurs can be defined.

The data obtained to date cover studies of Ti 150A having a hydrogen content of 64 parts per million. The temperatures considered are 600° and 1000°F. The results are summarized in Table III and a plot of the exposure time versus tensile strength and elongation is presented in Figure 1. The data are not complete, however, it appears that embrittlement does not occur at 600°F, at least, at this hydrogen level. Exposure at 64,000 psi does result in some increase in tensile strength, however, a comparison with the 32,000 psi data suggests that this is due to strain hardening, particularly since the total deformation for the

64,000 psi tests was many times greater than for the tests at the lower stress.

However, the tests at 1000°F indicate that there was a significant drop in ductility accompanied by a slight decrease in tensile strength after exposure. This appeared to be the case for both stresses investigated, 2,800 psi and 5,600 psi. In addition, the mode of fracture changed from that encountered with samples exposed at 600°F. The 1000°F samples failed with no necking at the point of fracture. The elongation and reduction of area appeared to be uniform throughout the gage section of the specimen. The as-treated and 600°F samples failed with localized necking at the point of fracture and with a cup and cone type of fracture.

No explanation can be offered for this behavior at the present time. Additional tests are contemplated for longer exposure times at both these temperatures. In addition, one or more additional temperatures will be studied. Tests of specimens of higher hydrogen content are to be initiated shortly.

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

During the course of survey studies on both Ti 75A and a stabilized alpha alloy, Ti-6%Al, it was determined that an optimum amount of cold working existed above which instability was introduced to the material. This effect was further investigated during the studies of total deformation properties carried on under the second phase of the over-all program (2). In these studies it was shown that at 1000°F the effect of 12% cold work on Ti-6%Al was to increase the creep rate and lower the total deformation strengths from those of the as-forged material.

The general validity of the results obtained on the alpha alloys was checked by extending the survey studies to a modification of the Ti-6%Al alloy. The modified alloy contained 1/2% Si in addition to 6% Al. In these studies it was indicated that approximately 20% cold working would produce a similar instability at 1000°F (2). It was decided to extend the survey studies to provide data out to 1000 hours on this effect. In this way, it was hoped that the validity of the results on the Ti-6% Al could be confirmed.

The data obtained in this study are summarized in Table IV. It should be noted that the original survey studies were carried out on material cold worked 19 percent. A second batch of stock that was cold rolled using the same roll settings achieved 21.6% reduction. The slight amount of additional reduction did not appear to affect the properties.

A plot of the rupture data and the time to reach indicated total deformations for both the as-forged and the cold worked material is presented in Figure 2. As this plot shows, the immediate effect of cold working was to increase the tensile and rupture strength of the material. However, the strengthening was dissipated in about 100 hours and the as-forged material had a higher rupture strength throughout the remainder of the testing period.

The total deformation data show that the cold worked condition had consistently poorer properties than the as-forged condition. In Figure 2, it is shown that the time to reach a given deformation was less at all stresses for the cold worked material than it was for the as-forged material. Figure 3, a plot of stress versus minimum creep rate, shows that the creep strength of the as-forged condition was consistently higher at 1000°F than for the cold worked material.

Survey work indicated that the instability of the cold-worked material was manifested by recrystallization occurring during the course of the test. Additional metallographic studies are to be made on the completed test specimens from the present investigation.

The present work has confirmed the observation that the instability introduced to an alpha alloy by cold working can result in a loss of properties. Although the loss of rupture strength was not excessive, the time to reach a given total deformation at 1000°F was greatly reduced.

Other Work in Progress

In addition to the work described in detail in other sections of this report, work is in progress on several phases of the program covering the effects of contaminents. At the present time, the data are not sufficiently complete to warrant detailed discussion.

These studies include a survey of the Ti-30% Mo alloy to determine the relative effects of sponge hardness and a study of the effects of hydrogen contents on the creep properties of the Ti-10% Mo alloy. To date, tensile tests have been completed on these materials and the survey rupture and creep tests have been initiated.

REFERENCES

(1) Gluck, J. V. and Freeman, J. V. "Intermediate Temperature Creep and Rupture Behavior of Titanium and Titanium Alloys", Eighth Progress Report, Contract No. AF 33(616)-244, May 31, 1955, pages 1-5.

(2) Gluck, J. V. and Freeman, J. V. , Second Summary Report, Contract No. AF 33(616)-244 (to be published in 1955).

TABLE I

Producer's Chemical Analysis of Test Materials

				Alloying Constituents - Wt (%)				
	U. of M.	Suppliers	Sponge	Major		Minor		
Alloy	Alloy No.	Heat No.	Hardness	Fe	Cr	C	N	H_2
Ti 150A	2c	M739	-	1.47	2.76	.08	.026	.0310*
				Mo				
Ti-30% Mo	6		140	29.58		-	-	.0300*
		9042	107	30.50		.59	.020	na
				Al	Si			
Ti-6%Al-1/2%Si	8	7011	124	6.51	.48	-	.017	.017
				Mn				
Ti-8%Mn	3a	9056,58	148	8.62		.109	.051	na
	3b	9035-39	107	8.85		.063	.015	na
				Al				
Ti-6% Al	4c	9033	107	6.13		.044	.045	.0084
				Mo				
Ti-10% Mo	5a	9038	107	10.47		.051	.034	.0033

* hydrogen analysis by WADC, remainder at University of Michigan
na hydrogen analysis not yet available

Analysis of Sponge - Wt. (%)

Brinell Hardness	Si	C	N	Fe	Ti
107	<0.02	.045	.010	.012	bal.
148	.02	.054	.045	.240	bal.

TABLE II

Hydrogen Analysis of Vacuum Annealed Alloys

Material	U. M. Alloy No.	Run No.	No. Spec.	Hydrogen Content (parts per million by wt) Detn. 1	Detn. 2	Avg.
Ti-30%Mo	6	3	9	45	43	44
Ti-150A	2c	4	9	63	64	64
		5	9	57	57	57
Ti-10%Mo	5a	6	8	43	44	44
		7	9	44	42	43
Ti-6%Al	4c	10	9	34	34	34
		11	9	53	49	51

Hydrogen Analysis of Hydrogenated Alloy

Aim: 250 ppm

Ti-10%Mo	5a	12	9	286	234	260

TABLE III

Effect of Elevated Temperature Stressed Exposure on Room Temperature Tensile Properties of Vacuum Annealed Ti-150A

Stress-Exposure Conditions					Room Temperature Tensile Properties				
Temp. (°F)	Stress (psi)	Time (hrs)	Loading Def. (%)	Def. at End of Test (%)	Tensile Strength (psi)	0.2% Offset Yield Strength (psi)	Elongation (% in 1 in)	Reduction of Area (%)	Modulus[(a)] (psi x 10^6)
As Vacuum Annealed*					136,600	(136,600)	27.0	55.4	18.1
600	32,000	100	.32	-	133,700	--	30.0	52.9	-
	32,000	500	.33	.34	135,500	--	26.0	51.5	-
	32,000	In progress to 1000 hours							
	64,000	99.4	-	-	140,000	--	25.2	50.8	-
	64,000	500	3.03	3.71	150,800	--	21.0	43.5	-
	64,000	In progress to 1000 hours							
1000	2,800	100	.04	.26	136,500	(127,000)	9.0	7.9	-
	5,600	50	.10	.41	133,200	114,000	11.0	11.0	13.0
	5,600	99.5	.06	.38	132,800	120,500	10.0	10.0	13.4

* Hydrogen content: 64 parts per million (wt.)

(a) Modulus determined from slope of stress-strain curve.

TABLE IV

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

Condition	Test Temp. (°F)	Stress (psi)	Rupture Time (hours)	Elongation (% in 1 in)	Reduction of Area (%)	Loading Def. (%)	Time to Reach Indicated Deformation (hours) 0.5%	1.0%	5%	Minimum Creep Rate (in/in/hr)
As Forged	1000	74,800	Tensile Test	11.7	-	-	-		-	-
		50,000	41.1	14.3	31.4	.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	(5.0)*	(7.9)*	.27	15	105	(1000)est	2.8×10^{-5}
		24,000	>1581.4	(2.0)*	(0.8)*	.22	180	-	-	-
		20,000	>1083.8	(0)*	(0)*	.19	1030	-	-	1.3×10^{-6}
Cold Worked 19%	1000	92,400	Tensile Test	11.7	34.9	-	-	-	-	-
		52,000	48.4	29.1	49.5	.55	-	14	-	3.3×10^{-4}
		44,000	126.0	35.6	53.5	.48	0.5	5	46	7.7×10^{-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	2.1×10^{-4}
		25,000	1472.0	22.0	42.7	.27	8	35	800	4.0×10^{-5}
		20,000	>1034.7	(1.0)*	(0.8)*	.20	30	450	-	7.6×10^{-6}
		13,000	>1104.3	(0)*	(0)*	.12	690	-	-	1.8×10^{-6}

\> Test stopped without rupture at this time

( )* Permanent deformation after cooling, if measurable

Tensile Test Data for Ti-6%Al-1/2%Si Alloy

Condition	Test Temp. (°F)	Ult. Tensile Strength (psi)	0.2% Offset Yield Strength (psi)	Elongation (% in 1 in)	Reduction of Area (%)	Modulus (psi x 10^6)
As Forged	75	140,200	133,500	19.4	32.1	13.5
	600	97,000	86,200	15.7	21.8	13.5
	1000	74,800	60,700	11.7	22.4	9.8
Cold Worked 19%	75	178,800	162,700	8.2	24.2	12.7
	600	115,500	104,000	7.6	27.2	12.1
	1000	92,400	82,300	11.7	34.9	10.4

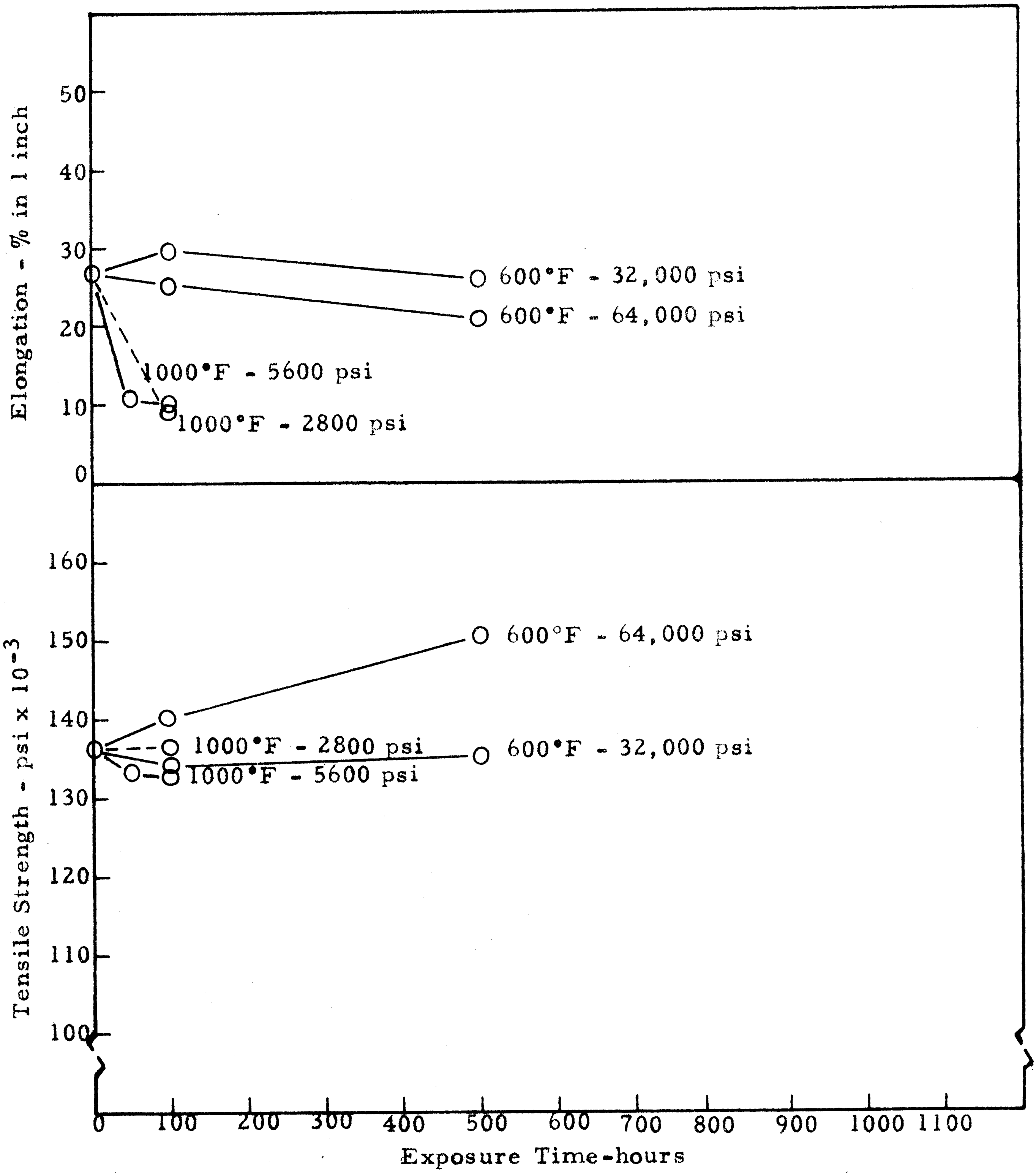

Figure 1. - Effect of Creep Test Exposure on Room Temperature Tensile Properties of Vacuum Annealed Ti 150A. (Hydrogen Content: 64 parts per million)

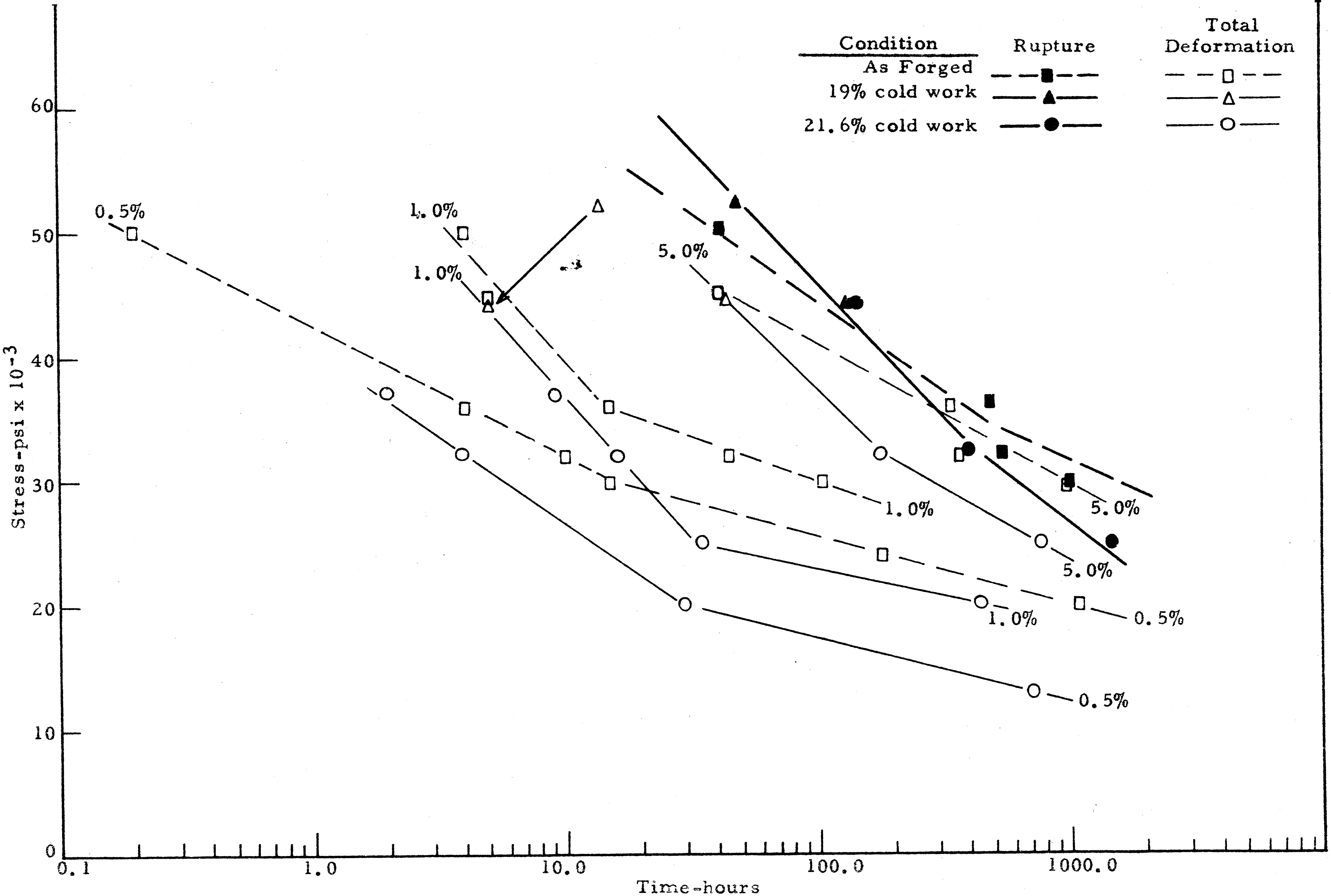

Figure 2. - Stress versus Rupture Time and Time to Reach Indicated Total Deformation at 1000°F for Ti-6%Al-1/2%Si Alloy.

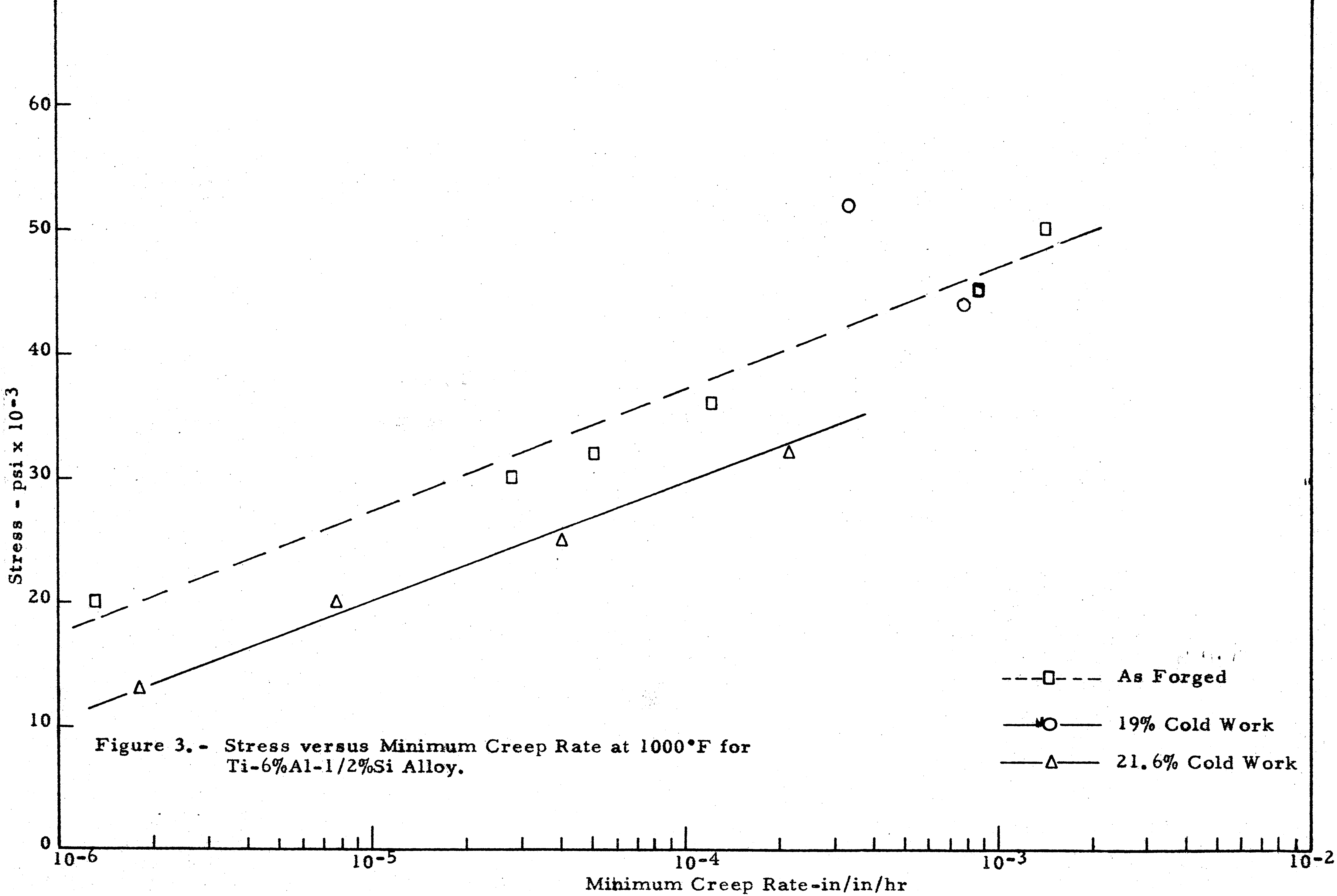

Figure 3. - Stress versus Minimum Creep Rate at 1000°F for Ti-6%Al-1/2%Si Alloy.