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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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SUMMARY

This report covers the period from October 16, 1952 to January 15, 1953.

The objective of the investigation is to relate types of microstructure obtained by variation in heat treatment and cold work to the creep resistance of typical titanium alloys in the range from 600° to 1000°F. The work will be guided to develop general fundamental metallurgical principles. Five typical alloys will be studied. These include commercially pure titanium (Ti 75), a commercial martensite forming alloy (Ti 150A), an alpha alloy (6% Al), a meta-stable beta alloy (10% Mo), and a stable beta alloy (30% Mo). The last three are being obtained in the form of small experimental heats. Variables of investigation include alloy type, heat treatment, and cold work. Microstructural examination and x-ray diffraction analysis will be the major tools utilized to attain the objective of the investigation.

Work to date has been confined principally to material procurement and preparation of equipment. Four of the five materials have been received, the exception being the meta-stable beta alloy. Tensile testing is almost complete on the commercially pure titanium and rupture testing is now underway. Heat treatment and tensile testing are being carried out on the other alloys.

INTRODUCTION

It is well known that for materials subjected to creep testing the effects of prior treatment have more influence on creep behavior than wide ranges of chemical composition. Prior treatment variables include hot and cold working, temperatures and cooling rates of solution treatments, temperatures and times of aging treatments, and, in those materials subject to phase changes, the type of microstructure produced. In general, melting practice has been shown to be of prime importance. Thus, observed creep properties are functions not only of final treatments, but even of the particular lot of stock being tested.

Relations between creep behavior and treatments are often very complicated. A treatment producing maximum creep resistance at a low testing temperature may produce just the opposite result at a higher temperature. Treatments best for short time periods of service may not be best for prolonged times. Relationships may change depending on the deformation criterion over the time interval under consideration.

Thus, in an investigation whose scope is as wide as the present one, it is difficult to select any one alloy and/or condition for singular emphasis. Instead, it is desirable to survey in as systematic a fashion as possible a wide range of properties.

Fortunately, a considerable amount of information is now available covering the general metallurgy of titanium and a number of its alloy systems. At low alloy contents and low temperatures an alpha phase (hexagonal close packed) exists which transforms at higher temperature to a beta phase (body centered cubic). At intermediate temperatures a mixture of these phases exists. Dependent upon cooling rate and alloy content, different transformation products are obtained from the beta phase. In addition, subsequent reheat or primary isothermal transformation are significant.

Purely on the basis of the metallographically observed response to such treatments, it is possible to characterize titanium alloys as "type" alloys. Selection of the systems for study under this contract has been made on this basis. Commercially pure titanium forms the basis for the work. Even this may be considered an alloy since the small amounts of impurities present have an effect on its properties. The other materials chosen for study have the following characteristics: an alloy in which the alpha is stablilized; an alloy undergoing the so-called martensitic transformation (i. e. formation of an acicular transformation product) on fast cooling from the beta region; a meta-stable beta alloy, one in which beta retention would be possible, yet which would permit structural variations from both isothermal and aging treatments; and a stable beta alloy, one permitting complete beta retention on slow cooling.

Complete details of composition and testing variables for each alloy are given in following sections of this report. The purpose of mentioning them here is to emphasize the philosophy of the testing program.

TEST MATERIALS

Five materials, four of which have been received, were ordered for the investigation after consultation between representatives of the Wright Air Development Center and the University of Michigan. The test materials were selected as mentioned previously in order to be represenative of the "type" transformation reactions possible with titanium alloy systems. Two are available from commercial sources. The remaining three are being obtained in the form of small (approximately ten pounds each) experimental ingots and will be melted for the University by special arrangements with the Armour Research Foundation of Chicago, Illinois.

Material in the form of bar stock was chosen for study in order to minimize both procurement difficulties and contamination problems during treating and testing. The available details of the test materials are given in Table I.

PROCEDURE

In general, the experimental program is planned to study the effects on creep behavior of the metallurgical variables appropriate to the alloy under consideration.

Testing will be guided by experience from other research work in progress at lower temperatures. In the present program it is anticipated that tests can be confined to 600° and 1000°F for the experimental alloys and 600°, 800°, and 1000°F for the commercial materials; however, if necessary for fundamental explanations or to develop more complete data some intermediate temperature may also be employed. The effects of the variables are being surveyed by the following approximate testing procedure at each temperature:

(a) A short time tensile test;

(b) Tests designed to cause fracture in 30-40 hours and 100 hours. It must be realized that it will be unusual to actually obtain these times. Every effort will be made to cover the time period with two tests.

(c) Creep tests at a stress level which will result in a creep rate of between 10^{-5} and 10^{-6} inches per inch per hour and for a length of time sufficient for the approximation of a second stage creep rate.

These tests should be adequate to indicate relative creep strength properties. If more complete engineering data appears desirable for any particular condition and/or alloy, rupture tests will be extended to approximately 1000 hours and enough additional creep tests made to establish the total deformation characteristics. In addition, mechanical properties will also be determined at room temperature.

The room temperature tensile tests will show the general mechanical properties and serve to indicate whether or not treatments used are giving normal results.

Tensile tests at elevated temperatures are mainly required to arrive at a reasonable stress for rupture testing. The limited number of testing units and the limited number of specimens of experimental materials require very little error be made in the selection of stresses.

In addition to creep, rupture, and tensile testing, the following general techniques will be employed to delineate the observed influences of metallurgical variables:

(a) Microstructural examination both before and after creep and rupture testing;

(b) Hardness changes due both to initial treatment and to the effect of testing;

(c) Changes in x-ray diffraction characteristics induced by treatments, particularly diffraction line broadness where applicable as a measure of internal strain and lattice parameter changes as a measure of precipitation effects.

The purpose of these measurements is particularly to establish the effects of grain size, cold work, and precipitation phenomena on creep characteristics. Where the $\beta \longrightarrow \alpha$ transformation changes not only the relative amounts of the phases but also their distribution, a correlation of the two effects will be attempted. Also included in such correlation will be the effects of initial structure and changes during testing.

Details of Planned Treatments

I. Commercially Pure Titanium (Ti 75) Tested at 600°, 800°, 1000°F

1. As produced (exact details not yet available.
2. Annealed at 1500°F.
3. Annealed at 1700°F.
4. Annealed at 1500°F + 10% cold work.
5. Annealed at 1500°F + 30% cold work.
6. Annealed at 1700°F + 10% cold work.
7. Annealed at 1700°F + 30% cold work.
8. Partially stress relieved specimens after cold working are to be tested for both reductions. Selection of the exact conditions will be delayed pending results of the above tests.

II. Martensite-forming Alloy (Ti 150) Tested at 600°, 800°, 1000°F

1. As produced (exact details not yet available).
2. Annealed at 1500°F.
3. Air Cooled from 1500°F.
4. Water Quenched from 1500°F.
5. Annealed from 1350°F.
6. Air Cooled from 1350°F.
7. Water Quenched from 1350°F.
8. * from 1500°F + 1350°F Anneal.
9. * from 1500°F + 900°F Anneal.
10. * from 1350°F + 900°F Anneal.
11. Solution treat at 1800°F + full isothermal transformation at 1300°F.
12. Solution treat at 1800°F + full isothermal transformation at 1000°F.

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* Cooling rate to be determined by work in items (3) through (7).

Items 1 - 7 represent variations in the properties of $\alpha + \beta$. Items 8 - 10 vary $\alpha + \beta$ properties through a different procedure. Items 11 and 12 are representative isothermal transformation structures. Sufficient stock is available for checking about three more conditions in addition to those listed on the preceding page.

III. Experimental Alpha Alloy (6% Al) Tested at 600° and 1000°F

1. Cold worked about 17% and annealed to fine grained structure.
2. Cold worked about 17% and annealed to coarse grained structure.
3. Water quenched from 2025°F (middle of β range).
4. Cold worked 10%.
5. Cold worked 17%.
6. Cold work as in (4) and (5) plus reheating to a temperature below which recrystallization occurs.

In addition, mechanical properties will be determined on the material as-forged.

IV. Experimental Meta Stable β Alloy (10% Mo) Tested at 600° and 1000°F

1. Water quench from β range to retain β.
2. Quenched from β range and aged to transform β at a lower temperature.
3. Quenched from β range to full isothermal transformation at 1100° and 1300°F.
4. Quenched from two temperatures in the $\alpha + \beta$ range in order to obtain the maximum difference in $\alpha + \beta$ properties.

The above planned procedure may be modified pending receipt of the material.

V. Experimental Stable β Alloy (30% Mo) Tested at 600° and 1000°F

1. Testing original retained β structure.
2. Quenching and aging β structure for transformation.
3. Quenched plus cold work.
4. Long time isothermal holding after cooling from all β region.

Little response to heat treatment is to be expected from this material.

As results are obtained it is anticipated that changes and modifications of the above schedules may become necessary; therefore, it should not be accepted as a hard and fast statement of work.

RESULTS

The following results are presented without comment at the present time:

Commercially Pure Titanium (Ti 75)

All specimens have been treated for conditions (1) through (7).

The following tensile and rupture data have been obtained to date:

(see following page)

Tensile Test Results - Ti 75

Treatment	Test Temp. (°F)	Tensile Strength (psi)	.2% Offset Yield Strength (psi)	Elongation (% in 1 in.)	Reduction of Area (%)
As Received	75	90,700	67,500	30.2	50.4
Ann. 1500°F	75	87,900	68,400	30.4	48.6
Ann. 1700°F	75	87,600	65,600	34.0	44.6
Ann. 1500°F + 10.9% Cold Worked	75	115,000	102,000	12.7	43.5
Ann. 1700°F + 10.9% Cold Worked	75	109,000	99,400	10.7	32.5
Ann. 1500°F + 31.3% Cold Worked	75	129,000	116,000	12.3	39.8
Ann. 1700°F + 31.3% Cold Worked	75	127,000	114,000	11.5	34.1
As Received	600	37,600	19,100	34.9	68.6
As Received	800	32,700	19,500	22.6	69.5
Ann. 1500°F	800	28,300	14,700	34.3	70.4
Ann. 1700°F	800	28,500	14,800	36.6	69.2
Ann. 1500°F + 10.9% Cold Worked	800	41,300	36,600	17.3	67.3
Ann. 1700°F + 10.9% Cold Worked	800	42,400	37,000	15.6	61.5
Ann. 1500°F + 31.3% Cold Worked	800	50,800	44,100	21.4	66.7
Ann. 1700°F + 31.3% Cold Worked	800	48,900	39,800	18.6	66.2
As Received	1000	22,000	12,700	72.0	83.4
Ann. 1500°F	1000	17,790	10,550	114.0	92.0
Ann. 1700°F	1000	19,750	11,200	93.4	87.0
Ann. 1500°F + 10.9% Cold Worked	1000	23,800	14,700	103.0	85.4
Ann. 1700°F + 10.9% Cold Worked	1000	20,900	14,000	144.5	94.6
Ann. 1500°F + 31.3% Cold Worked	1000	26,800	17,000	70.5	87.4
Ann. 1700°F + 31.3% Cold Worked	1000	23,700	15,000	91.4	91.8

Rupture Test Results - Ti 75

Treatment	Test Temp. (°F)	Stress (psi)	Time for Rupture (hours)	Elongation (% in 1 in.)
As Received	1000	9,500	4.9	109.2
As Received	1000	5,000	54.4	119.4

Martensite Forming Alloy (Ti 150)

Specimens of the as-received condition have been prepared for tensile testing. Specimens of the heat treated conditions are being prepared.

Stable Alpha Alloy (6% Al)

Experimental work has led to the following observations:

1. The maximum cold work the as-forged structure will take without cracking is about 17 percent.

2. It has not yet been determined if 17 percent cold work will give the desired recrystallization structures (i. e. fine and coarse grained).

3. The as-forged stock showed no appreciable microstructural change on reheating between 1300° and 1700°F except grain growth. Depending on the time period this was encountered between 1500° and 1700°F.

The following tensile data have been obtained:

Tensile Test Results - Alpha Alloy

Treatment	Test Temp. (°F)	Tensile Strength (psi)	.2% Offset Yield Strength (psi)	Elongation (% in 1 in.)	Reduction of Area (%)
2025°F 1 hr. + Water Quench	75	142,400	122,000	18.2	38.6
2025°F 1 hr. + Air Cool	75	141,200	121,000	7.1	14.7
2025°F 1 hr. + Water Quench	600	97,900	78,500	17.6	34.1
2025°F 1 hr. + Water Quench	1000	80,300	64,800	14.9	33.8

The other test materials have not yet been received or have been received too recently for experimental work in the period of time covered by this report.

Structural Studies

In addition, electro-polishing techniques have been developed for several of the materials; however, metallographic data is not yet extensive enough to warrant report at this time.

TABLE I

TEST MATERIALS

Designation and Supplier		Chemical Composition (percent)						Form and Date of Receipt
		O_2	N_2	C	Fe	Ti		
1. Ti. 75 (Commercial Pure Ti)	Nominal	trace	.02	-	.10	bal.		50 ft. 1/2" round bar stock
(Titanium Metals Corp.)	Actual	-	.061	.025	.19	bal.		October 15, 1952
2. Ti 150 (Martensite forming		O_2	N_2	C	Fe	Cr	Ti	
Alloy)	Nominal	.25	.02	.02	1.3	2.7	bal.	50 ft. 1/2" round bar stock
(Titanium Metals Corp.)	Actual	-	.124	.046	1.52	2.68	bal.	December 10, 1952
3. Experimental Alpha Alloy	Nominal	6% Al - bal. Ti.						20 ft. 1/2" rounds and squares
(Armour Res. Foundation)	Actual	6.21% Al - bal. Ti.						October 15, 1952
4. Experimental Stable Beta Alloy)	Nominal	30% Mo. - bal. Ti.						20 ft. 1/2" rounds
(Armour Res. Foundation)	Actual	Not yet available						January 15, 1953
5. Experimental Meta-Stable Beta Alloy (Armour Res. Foundation)	Nominal	10% Mo - bal. Ti.						Not yet received.