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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

A brief outline is presented of an experimental investigation to be carried out to develop the relationship between microstructures of five typical titanium alloys and their creep characteristics in the temperature range from 600° to 1000°F. The alloys are Commercially Pure titanium (Ti 75), a commercial martensite forming alloy (Ti 150A), a meta-stable beta alloy (10% Mo), a stable beta alloy (30% Mo), and an alpha alloy (6% Al).

Work during the period covered by this report, July 1, 1952 to October 15, 1952, has been confined to the procurement of test materials and preparation of equipment. The Ti 75 and the 6% Al alloy were received at the end of this period.

INTRODUCTION

A study of the principles of the physical metallurgy of titanium base alloys for use in the temperature range 600° to 1000°F was authorized under Contract AF 33(616)-244 and Supplemental Agreement No. S-1 (53-247). During the period covered by this report, July 1, 1952 to October 15, 1952, work was confined to procurement of material for experimental purposes and building of authorized experimental equipment.

The investigation involves development of general principles relating creep and rupture properties in the temperature range of 600° to 1000°F to the response to heat treatment of typical titanium base alloys. The alloys specified represent five general structural types:

(1) Commercially pure titanium (Ti 75)
(2) Martensite-forming alloy (Ti 150A)
(3) Meta-stable Beta alloy (10% Mo)
(4) Stable Beta alloy (30% Mo)
(5) Alpha alloy (6% Al)

In 1949 Adenstadt* reported that titanium underwent significant amounts of continued deformation by creep at room temperature under stresses well below the yield point. This tendency to creep increases rapidly with increasing temperature. In general, alloys of titanium of the type considered in this investigation are considerably more resistant to creep than titanium itself. At temperatures of 600°F or higher, however, the load carrying ability of the alloy is limited by the relationships between stress and the time to reach by creep the allowable deformation for the application considered.

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* Metal Progress 56, No. 5, p. 658 (1949)

The creep resistance is a function of both the composition and heat treatment of the alloys. Variations in the conditions of treatment can cause greater variations in creep resistance than wide changes in chemical composition. The situation is similar to that encountered in steel, for instance, where heat treatment has a well known and marked effect on creep strength.

The alloys being considered in this investigation each have characteristic responses to heat treatment. In general, these are reflected in changes in the microstructure in accordance with their phase diagrams. The Ti 75 and Ti 150A are well known but complex commercial alloys. The other three alloys were derived from phase diagram work sponsored by the Materials Laboratory, AMC, at the Armour Research Foundation. The main basis for selecting the three compositions was to obtain representative structural types based on simple two component phase diagrams. The alpha alloy (6% Al), however, was known to have very attractive creep properties in comparison to other titanium alloys.

GENERAL PROCEDURE

All test materials are being purchased from outside sources as bar stock. The Ti 75 and Ti 150A have been ordered from the Titanium Metals Corporation. The 6% Al, 30% Mo, and 10% Mo alloys are being prepared by Armour Research Foundation of Illinois Institute of Technology, who developed the alloys under sponsorship of the Materials Laboratory, WADC. In each case ten pound ingots will be melted and forged to bar stock.

The proposed experimental program involves preparing specimens with systematic variation of the conditions of heat treatment applicable to the several alloys so as to produce typical structural conditions in the metal. Experience obtained from a concurrent investigation of sheet

Ti 75 and Ti 150 which has been in progress for the Office of Air Research, WADC, for some time is available as a guide for those materials. The objectives of that investigation are similar to those of the present investigation except that elevated temperature testing is confined to the temperature range of 76° to 600°F. The work on the other three alloys will be guided mainly by the phase diagrams developed for the Materials Laboratory, WADC, by Armour Research Foundation and by the experience Armour has had in producing the alloys.

The main structural variations being considered for each alloy are:

1. Commercially pure titanium (Ti 75)

This material is not subject to the control of microstructure by regulating the cooling rate from the all beta phase region. Consequently, microstructures are governed by hot rolling or by cold working and annealing. Cold work in itself is an important consideration in controlling properties. Test stock is being supplied in the commercial hot-rolled condition. Samples will be prepared using at least two annealing temperatures and two degrees of cold work for each. In addition, the influence of grain size as obtained by cold work and recrystallization should be included. The work on sheet has shown that cold work improves creep resistance remarkably up to 600°F. One result of the investigation will be to define the maximum temperatures for improvement of strength by cold work.

The work on sheet has also shown that Ti 75 is subject to considerable alteration in creep resistance due to a strain aging type of reaction between 210° and 600°F. It is not expected that this will be important in the present investigation although care will be exercised to determine if other significant structural changes might occur.

2. Martensite-forming alloy (Ti 150)

This alloy is subject to considerable variation in properties due to variation in the relative proportions of α and β and their form by control of the influence of initial temperatures and cooling rates on the β to α transformation. It appears necessary to limit heating to below the all beta phase region in order to avoid excessive brittleness at room temperature. Consequently, the heat treatments will be almost entirely restricted to the $\alpha + \beta$ region. Indications are, however, that creep properties are very dependent on both temperatures and cooling rates. It is planned to include at least two temperatures of heating followed by water quenching, air cooling, and annealing. Suitable subsequent heat treatments may be used. In addition, some experimenting involving the properties of structures obtained by isothermal transformation from the beta region are contemplated to obtain some idea of the creep resistance of transition structures.

Variation in temperature of heating controls the relative amounts of α and β, the amount of β increasing with increasing temperature. Cooling rate controls the amount of β retained in the structure, the more rapid the cooling the more β retained at room temperature. The previous work on sheet indicates that increasing amounts of retained β are accompanied by greatly increased creep resistance. The retained β, however, tends to transform during testing with a decrease in volume which causes complex creep characteristics. It is expected that this latter behavior will require considerable attention at the higher temperatures of the present investigation.

3. Meta-stable Beta alloy (10% Mo)

Attempts are being made to obtain more information regarding the characteristics of this alloy. Pending more complete information only a tentative program involving direct quenching to retain β followed by reheating for transformation, and direct transforming by isothermal means are contemplated at present.

It is expected that considerable experimentation to determine response to heat treatment will be necessary before conditions for creep testing can be established.

4. Beta alloy (30% Mo)

In theory this alloy should respond to heat treatment mainly through grain size changes with temperature of heating. Likewise, it should be amenable to considerable variation in creep resistance by cold work. It is, therefore, planned to establish proper conditions of variation in these two treatments for creep testing.

5. 6% Al Alpha alloy

The program for this material is still being formulated. It is expected, however, that the main variable to be studied will be cold work. Initial cold work followed by recrystallization to two grain sizes is planned. Some testing will also be carried out using reheating without recrystallization subsequent to cold work.

Because the 6% Al alloy has a stable structure, hot working or cold working following by annealing are the ways the structure can be varied. Consequently, grain size and degree of cold work are the major variables which might be expected to control creep resistance.

EXPERIMENTAL PROCEDURE

The Ti 75 and Ti 150A were ordered from the Titanium Metals Corporation. The three experimental alloys were ordered from the Armour Research Foundation.

Fifty feet of 1/2-inch round bar stock is to be supplied for the work on Ti 75 and Ti 150A. Ten pound ingots of the three experimental alloys are to be melted and forged to bar stock by Armour.

The Ti 75 and the 6 percent aluminum alloy stock were received at the end of the period covered by this report.

Bar stock was ordered for several reasons. Contamination difficulties during heat treatment are minimized. In the case of the experimental alloys, it was almost mandatory to use bar stock in view of the problems associated with the production of sheet from experimental materials. From the viewpoint of establishing true properties of the structures, it may be that the use of bar stock will also reduce possible effects from reaction with the oxygen and nitrogen of the air during testing.

TESTING 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; however, if necessary for fundamental explanations, or to more completely establish the properties of a particular material, some intermediate temperature may also be employed. The effects of the variables will be 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.

(c) One creep test at a stress level aimed to 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 alloy, rupture tests will be extended to approximately 1000 hours and enough additional creep tests made to establish the total deformation characteristics. Mechanical properties will also be determined at room temperature.

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 changes.

Equipment

A total of eight creep units are to be assigned to the experimental work under this contract. Three of these units are for high precision creep tests and are now available. The remaining five units are under construction at present and are of the creep-rupture type, (i.e. suitable for both types of testing). In this manner complete time-extension curves can be established for all tests.

An existing heat treatment furnace and muffle have been modified to permit treatment of bar stock specimens. A protective atmosphere of argon is first purified by passing through titanium chips.

A rolling mill is available for cold working specimens.

In addition, complete equipment is available for tensile and hardness testing and standard metallographic examination. An electron microscope is available if the course of the investigation dictates its use. X-ray diffraction equipment includes a Norelco low angle x-ray spectrometer, a Norelco high angle spectrometer, and a focusing back-reflection camera. A Fourier analysis computer is available for line broadness computations.

Experimental Techniques

Bar stock in the form of 0.250-inch diameter specimens will be used for all creep and rupture tests. Tests will be conducted in individual units utilizing electric resistance furnaces for heating. A modified Marten's optical type extensometer system will be used for elongation measurements. All testing is to be carried out in accordance with A.S.T.M. Recommended Practices.

Metallographic procedures successfully used at the University on sheet material will be used for this investigation. Following a rough grind the specimens are taken through 240, 400, and 600-mesh silicon carbide paper. Finish polishing is accomplished with 4-8 micron and 0-1/2 micron diamond compound on suitable laps. Standard etchants applicable to the particular material under study will be used. An electro-polisher has also become available recently.

It is expected that previously developed techniques for the x-ray diffraction study of titanium alloys will be applicable to this investigation. The exact procedure used will be reported when such work is carried out.

Correlation of Data

The investigation will provide survey creep and rupture data for a number of treatments of each of the five types of alloys. These will be in turn correlated with the microstructures existing before and after testing. In general, tensile and hardness properties at room temperatures will be made part of the correlation. Insofar as is necessary, x-ray diffraction studies will be made where they can contribute to an understanding of the metallurgical principles involved in the creep and rupture behavior.