FIFTH PROGRESS REPORT
TO
THE OFFICE OF AIR RESEARCH
ON
A STUDY OF CREEP OF TITANIUM AND TWO OF ITS ALLOYS

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A STUDY OF CREEP OF TITANIUM AND TWO OF ITS ALLOYS

SUMMARY

During the period covered by this report, 1 October 1951 to 31 December 1951, creep testing of the Ti 75 has been extended to elevated temperatures. Work has also included the starting of a more complete program for the investigation of the effect of recovery treatments after cold working on the internal structure and the creep properties of the Ti 75.

Creep testing of the annealed, the 40 percent cold worked, and the 40 percent cold worked with a 100-hour recovery treatment at 550°F of Ti 75, have been completed and are presented. It has been found that the recovery treatment reduces the amount of first stage creep, both at 76°F and 210°F in comparison to the cold worked material at the same secondary creep rates. Furthermore, it was found that after the above recovery treatment the creep resistance was superior to that of the 40 percent cold worked material at 210°F, although the opposite was true at 76°F.

The work accomplished on the ferro-chrome alloy of titanium (Ti 150) has been confined principally to creep testing the as-received material at 210°F and room temperature creep testing of material heat treated in various ways from 1500°F. Some bend testing and considerable metallographic work has been done on the heat-treated material.

INTRODUCTION

This report, the fifth under Air Force Contract Number: AF33(038)-14111 (Expenditure Order Number: 460-36 BR-1) on the study of creep in titanium and two titanium base alloys in the form of commercially produced 0.060-inch thick sheet, covers the period from 1 October 1951 to 31 December 1951.
Work is still being concentrated on the commercially pure titanium (Ti 75) and the ferro-chrome alloy (Ti 150) since the third material, probably a manganese alloy (Ti 130), has not yet been shipped. Fundamental structural factors affecting the creep properties of the above materials continue to be the object of this work. This viewpoint has led to the determination of the effect of cold working and recovery treatments on the internal structure and on the creep resistance of Ti 75. In the study of Ti 150, heat treatments continue to be the main variables of investigation.

EXPERIMENTAL PROCEDURES

During this period the use of an electropolisher was obtained and the following procedure was found to be satisfactory in polishing the commercially pure materials and the ferro-chrome alloy for micro-examination:

An etching solution consisting of 1000 ml of glacial acetic acid and 60 ml of perchloric acid (sp. grav. 1.54) was carefully prepared. After preparing the samples by taking them through the 400 silicon carbide paper the Ti 75 was electropolished for 30 seconds at a current density of approximately 0.05 amperes per square centimeter (approximately 4 volts). The same current density was used with the Ti 150; however, approximately 1 minute of polishing time proved more satisfactory. In order to prevent the formation of a film and to limit the temperature rise, the electrolyte was recirculated at the maximum rate possible.

It was found that a close adherence to above conditions was not necessary in order to obtain a good polish.

After polishing, a light etch of 1 part hydrofluoric acid, 1 part nitric acid, and 2 parts glycerine was employed. It was found, especially in the case of the Ti 75, that examination must be made immediately after polishing and etching as there is a tendency for heavy staining to occur at the grain boundaries.

Heat Treatment Studies. Concern over the possible constant introduction of impurities in Ti 150 during heat treatment from the flowing argon atmosphere has led to the introduction of the following modification of procedure.
A chromel wire gauze cylinder packed with titanium and titanium alloy chips is placed in the heat treatment chamber inlet. Residual impurities are removed from the incoming gas as it passes through the chips. The first group of creep samples treated in this manner showed a definite improvement in surface appearance.

RESULTS AND DISCUSSION

Commercially Pure Titanium (Ti 75)

The work during the period covered by this report mainly involved the accumulation of creep data at 210°F on stock annealed at 1600°F, annealed and subsequently cold worked, and on stock reheated for recovery after cold working. Some further tests were conducted at room temperature on the latter material. Testing is in progress at 400°F and to a limited extent at 600°F.

The investigation is based on studying the relationship between internal strain and creep resistance. Previous reports presented results of measurement of internal strain from line widths of X-ray diffraction lines. A reasonably good correlation was obtained between creep resistance at room temperature and this measurement of internal strain. The work is now being extended to include testing temperature as a parameter and to introduce the effects of recovery from cold work. The recovery work is to cover both re-heating after cold working prior to creep testing and recovery effects during creep testing. One general objective is to determine if the correlation between internal strain and cold work is independent of the path by which the state of internal strain is reached.

Sufficient X-ray diffraction work had previously been completed for the purposes of the investigation to date. During the period covered by this report such work has therefore been limited to checks to insure that the creep specimens did have the internal strain predicted by the previous data.
Recent work has focused attention on primary creep effects. Heating for recovery apparently reduces the amount of primary creep to a very significant degree.

Creep Testing of Annealed Material. Creep tests at 210°F on annealed material gave the results shown in Figure 1. The results previously obtained at 76°F are included for comparative purposes. The decrease in creep resistance between 76°F and 210°F was substantial, the stress for a secondary creep rate of $10^{-6}$ per hour being about 23,000 psi at 210°F as compared to 37,000 psi at room temperature.

The stock being tested was annealed at 1600°F, the treatment found best to minimize internal strain.

Creep Testing of Cold Worked Material. Material annealed at 1600°F and then cold worked 40 percent at room temperature was creep tested at 210°F. The results obtained are presented in Figure 1 and compared with the data previously obtained at 76°F. Increasing the testing temperature reduced the creep resistance substantially and yielded a stress versus log secondary creep rate curve approximately parallel to the curve at 76°F.

Creep Testing of Cold Worked and Reheated Material. Creep tests were initially undertaken to determine if the correlation between internal strain of cold worked material (as measured by the width of X-ray diffraction lines) and secondary creep resistance at room temperature would hold for samples reheated after cold working. The last progress report indicated that tests at 50,000 psi on a sample cold worked 7 percent and held 10 hours at 600°F and a sample cold worked 40 percent and held 10 hours at 210°F did agree with the correlation.

Two additional tests at 50,000 psi did not agree with the correlation. The specimens were cold worked 40 percent and one was held for 1 hour at
400° and the other 100 hours at 550°F before testing. The creep rates obtained were lower than predicted by the correlation.

In view of this finding, a more extensive program was started to investigate the effects of recovery treatments of cold worked material. The work to date has been confined to material cold worked 40 percent. Samples were held for 100 hours at 210°, 400° and 550°F for recovery prior to creep testing. To date tests have been completed at 76° and 210°F on the samples recovered at 550°F with the results shown in Figure 1.

At 76°F the recovery treatment increased creep rate by approximately 50 percent over that of the cold worked condition. At 210°F, however, the effect of recovery was reversed, the cold worked condition having somewhat higher creep rates (lower creep resistance) than the recovered material.

During the next experimental period it is expected that it will be possible to obtain sufficient data to establish a more definite conclusion about the correlation between internal strain and creep resistance. More extensive data should enable the establishment of principles.

Primary Creep. The apparent lack of correlation between internal strain and creep resistance for recovered samples led to consideration of the shapes of the time versus elongation curves. This is still in progress and definite conclusions have not been reached. It was immediately evident, however, that the amount of primary creep for a given secondary creep rate was much less for the recovered condition than for the cold worked condition. This trend is shown by Figure 2. It should be recognized that annealed material also has relatively low primary creep on this basis of comparison.

The consideration of the amount of primary creep on the basis of the secondary creep rate is mainly of interest from a theoretical viewpoint. When the comparison is made at constant stress, the situation changes as is shown by Figure 3. The relative creep strengths become of more importance
and there is little difference between the cold worked and the recovered conditions. The annealed material has very large primary creep due to the low creep resistance.

The availability of more data and more complete analysis should prove useful in explaining results. Furthermore, the reduction of primary creep by reheating after cold work may become of practical importance for service at temperatures above room temperature where the recovery treatment also improves the creep resistance.

Theoretical Considerations. In the recovery treatment, thermal energy allows the low energy "flow units" to be dissipated. If it is assumed that these are the main contributors to primary creep when a stress is applied, then the primary creep should be reduced, as was observed.

This concept, however, must be extended if it is to cover the other experimental facts. Recovered samples had lower creep resistance at 76°F than in the cold worked condition. Thus the recovery treatment must not only eliminate the low energy flow units but must also reduce the general level of internal strain controlling secondary creep.

At 210°F the creep resistance was somewhat increased by recovery treatment. This, however, is not in contradiction to the behavior at room temperature. The previous progress report showed that material cold worked 40 percent continued to lose internal strain over a 1000-hour period at 210°F. Thus recovery occurred continuously during the creep tests. The amount of recovery in 1000 hours at 210°F for unstressed specimens was, however, less than would be expected from 100 hours at 550°F. Thus it would seem that a higher level of internal strain should have existed in the cold worked samples and, thus, a higher resistance to creep. Because the creep resistance at 210°F was less in the cold worked condition, it appears as if the instability induced by
recovery causes a larger loss in creep resistance than can be attributed to the recovery alone during testing.

The results of further testing and analysis of data should give considerable information about the factors controlling creep of titanium.

Ferro-Chromium Alloyed Titanium (Ti 150)

Creep tests and metallographic studies on Ti 150 have been continued on both the as-received material and material heat treated at a temperature of 1500°F. A previously unencountered brittleness was noted in the latter material; however, the modification (see page 3) of the heat treating procedure should correct the condition. Data from loading studies has enabled calculation of the elastic modulus of the material. Bend tests and metallographic examinations are also reported.

Creep Testing. Creep testing of Ti 150 has been carried out on the as-received material (solution treated 1275°F, water quench, 2 hours at 1200°F) at a temperature of 210°F. The data are presented in Figure 4 in conjunction with the room temperature results previously obtained on the same material. In testing at 210°F the approach to steady state creep appears to be more rapid than in testing at room temperature. As in the case of the room temperature tests, the creep rates were taken as the slope of the strain versus time curves at 500-hour increments. It will be noted, however, that the decrease in creep rates over the period from 500 to 1000 hours in the 210°F tests was greater than in the corresponding period at
room temperature. Examination of the strain versus time curves for the 210°F tests shows an essentially constant rate over the last 250 hours of the test and thus indicates the close approach to steady rate mentioned previously. As yet no 210°F test has been conducted at a high enough stress to check the existence of a "break" in the curve as obtained in the lower temperature tests. Further testing at both higher stresses and temperatures is planned to complete this study.

Tentative creep rate versus stress relationships are presented in Figure 5 for room temperature tests of Ti 150 heat treated from 1500°F. The heat treatments used were water quenching, air cooling, and annealing. The data are presented in the form of bands for water quenched and annealed material because there is a scatter in the points obtained. Only one test has been conducted on the air cooled material. The scatter may be due to both the inherent inhomogeneity of the material and also to the possible introduction of impurities during heat treatment. This latter possibility is expected to be checked in further tests by the modification of heat treatment procedures.

Apparently, at higher stresses and creep rates the samples having the larger amounts of β phase have the higher creep strength. An interesting point is the fact that the strength after annealing from 1500°F virtually coincides with the corresponding strength for the as-received material.

The last two sets of specimens heat treated at 1500°F seemed to be more prone to fracture prematurely in the grips and in the gage section than previously heat treated samples. Fractures were generally associated with slight imperfections on the surface of the samples, although these imperfections were not any more severe than for previously heat treated samples.
Samples ruptured in the gage section during tests at 76°F under the following conditions:

<table>
<thead>
<tr>
<th>Heat Treatment</th>
<th>Stress</th>
<th>Time for Rupture</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Received</td>
<td>86,000</td>
<td>1.5</td>
</tr>
<tr>
<td>Annealed from 1500°F</td>
<td>90,000</td>
<td>2</td>
</tr>
<tr>
<td>&quot;</td>
<td>85,000</td>
<td>1.5 (fillet fracture)</td>
</tr>
<tr>
<td>&quot;</td>
<td>80,000</td>
<td>7 (fillet fracture)</td>
</tr>
<tr>
<td>&quot;</td>
<td>80,000</td>
<td>(In progress 180 hours)</td>
</tr>
<tr>
<td>&quot;</td>
<td>70,000</td>
<td>(In progress 1200 hours)</td>
</tr>
<tr>
<td>Air Cooled from 1500°F</td>
<td>125,000</td>
<td>3.5</td>
</tr>
<tr>
<td>&quot;</td>
<td>118,000</td>
<td>5 minutes</td>
</tr>
<tr>
<td>&quot;</td>
<td>117,000</td>
<td>1.0</td>
</tr>
<tr>
<td>&quot;</td>
<td>113,600</td>
<td>775</td>
</tr>
<tr>
<td>Water Quenched from 1500°F</td>
<td>120,000</td>
<td>163</td>
</tr>
<tr>
<td>&quot;</td>
<td>115,000</td>
<td>1.0</td>
</tr>
<tr>
<td>&quot;</td>
<td>110,000</td>
<td>(In progress 1200 hours)</td>
</tr>
</tbody>
</table>

Elongation was virtually nil in every heat treated case and there has been no evidence of third stage creep.

**Creep Recovery Data.** Three samples of as-received material were held at room temperature after creep testing at room temperature in order to measure what recovery, if any, took place after the stress was removed. A slight amount of initial recovery was noted. The rate, however, falls off rapidly for longer recovery periods. Specific data were as follows:

<table>
<thead>
<tr>
<th>Original Creep Test</th>
<th>Creep Recovery Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stress (psi)</td>
<td>Time (hours)</td>
</tr>
<tr>
<td>80,000</td>
<td>1168</td>
</tr>
<tr>
<td>86,000</td>
<td>2580</td>
</tr>
<tr>
<td>92,500</td>
<td>33\frac{1}{6}</td>
</tr>
</tbody>
</table>
Bend Testing. Bend tests on material in either the as-received condition (1275°F solution treat, water quench, 1200°F for 2 hours) or when annealed from 1500°F showed cracking at a bend radius of 5/16 inch.

Modulus Data. Stress-strain data taken during loading of creep tests has enabled computation of the elastic modulus for Ti 150. The results obtained were:

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Test Temp. (°F)</th>
<th>$E \times 10^{-6}$ (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Received (W. Q. 1275°F + 2 hrs. at 1200°F)</td>
<td>76</td>
<td>15 ± 0.5</td>
</tr>
<tr>
<td>As Received (W. Q. 1275°F + 2 hrs. at 1200°F)</td>
<td>210</td>
<td>11.8 ± 0.5</td>
</tr>
<tr>
<td>Water Quenched from 1500°F</td>
<td>76</td>
<td>16.4</td>
</tr>
<tr>
<td>Air Cooled from 1500°F</td>
<td>76</td>
<td>15.1</td>
</tr>
<tr>
<td>Annealed from 1500°F</td>
<td>76</td>
<td>14.2</td>
</tr>
</tbody>
</table>

Attention is particularly called to the large decrease in modulus with increased temperature for the as-received material. Drastic cooling treatments from an elevated temperature apparently increase the modulus, while annealing results in a slight decrease. The apparent effect of heat treatment is unusual. The values reported were obtained from available data from creep tests and not from tests intended to establish modulus values with high precision; therefore, the tendency for modulus variation with heat treatment should be checked more precisely before acceptance.

Metallographic Work. A considerable number of specimens were examined microscopically after heat treatment at 1500°F. As far as could be ascertained from present knowledge, there was no variation in microstructure which could account for the erratic creep rates obtained.

A major reason for examining the specimens was to see if there was anything in the structures which might account for the seeming variable brittle-
ness of creep specimens. Isolated inhomogeneities were observed in the specimens, generally in the form of variations in structure parallel to the direction of rolling. These cannot be definitely related to the apparent variable brittleness and did not appear to be present in an abnormal amount. There was no evidence that variable contamination during heating would account for the observed brittleness.
Figure 1: Creep Rates (Minimum of 1754 A) as a Function of Stress at the Incipient Temperatures and 100 Hours at 550°F. 

- x: Reduced 40% and Recovered
- ▲: Reduced 40%
- ○: Annealed State
Creep Rate (hour⁻¹)

Solution heat 1275°F, water quench, 1200°F, 2 hours.

As a function of testing time for 1150°F.

Figure 1 - Stress vs. creep rate curves at 1600°F and 2100°F.
As shown.

Heat treated from 1000°F.

Figure 5 - Nominal stress vs creep rate relationships at 76°F.