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V. EFFECT OF HEAT TREATMENT ON CREEP PROPERTIES OF THE TANTALUM BASE

ALLOY ASTAR-811C*

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INTRODUCTION

ASTAR-811C has the nominal composition Ta-8W-1Re-0.7Hf-0.025C and thus contains both solid solution and dispersed phase strengthening features. The present study was initiated to investigate the response of ASTAR-811C to heat treatment and the associated changes in mechanical properties. The first phase of this study has just been completed, and this paper presents the results. Specimens were heat treated for times of 5 to 60 minutes at temperatures between 3000° and 3800° F, and their creep properties and microstructures were evaluated.

EXPERIMENTAL PROCEDURES

Material Preparation

All test specimens came from a single heat of ASTAR-811C, designated Heat NASV-20. Sandwich-type electrodes were double consumable electrode vacuum arc melted to yield an 80-pound, 4-inch-diameter ingot. A 1-inch-high right-circular section of the ingot was upset forged at 2550° F by a single blow on a Dynapak to give a thickness reduction of 59 percent. After annealing 1 hour at 3000° F the forged disk was rolled first at 900° F and then at room temperature to give a combined 89 percent thickness reduction. Following another anneal for 1 hour at 3100° F, the material was cold rolled 33 percent at room temperature to give a finished sheet thickness of 0.035 inch from which test specimens were prepared.

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Heat Treatment

Heat treatments were done in a cold wall vacuum furnace at $<1 \times 10^{-5}$ torr. Unless otherwise specified, cooling was accomplished by injecting helium gas into the vacuum chamber as the power was turned off. This cooled the specimens to below 2000° F in less than 1 minute. Specimens heat treated at 3600° F and above were encapsulated in an evacuated tantalum envelope prior to heat treatment to prevent decarburization.

Creep Tests

Creep specimens were of the pin-loaded type having a 1-inch gage length and 1/4-inch width in the gage section. The specimens were electropolished after heat treatment. Hydrogen bakeout following electropolishing was accomplished during heating for creep testing. Scribe lines were cut at the ends of the gage section to serve as reference marks for the optical strain measurements. The creep tests were run in ultrahigh vacuum ion-pumped systems with the pressure at test temperature being $<1 \times 10^{-8}$ torr. Heating was by radiation from a split tantalum resistance heated element. Uniaxial stress was applied by dead weight loading.

RESULTS

Creep Behavior

Incremental type creep tests were employed in which the stress and temperature were progressively changed, beginning generally at 2200° F and 25 ksi and ending after about 300 hours at 2600° F and stresses as low as 5 ksi. An example of the type of creep curves obtained is shown in figure V-1.

The specimens all showed between 0.1 and 0.2 percent strain on loading to a stress level of 25 ksi at 2200° F. Most of the segments of the creep curves were linear. Where nonlinear segments were observed, the minimum creep rate is reported. The applied stresses were based upon the initial cross-sectional area. Thus, the true stress on the specimen during the later stages of test are higher than the values reported by up to about 5 percent. The reported creep rates have not been corrected for this effect, except for the test mentioned below.

To provide some measure of the effect of the prior creep strain on the creep rate at a given instant in the latter part of the test, a test like that shown in figure

V-1 was run and then the sequence was reversed. The creep rates for the up-cycle and down-cycle were compared after correcting for the true stress effect, and as shown in figure V-2 they are in good agreement.

The effect of annealing temperature on the creep behavior of ASTAR-811C is summarized in the Larson-Miller plot shown in figure V-3. The times to 1 percent strain values were calculated from the creep rate data obtained during the incremental temperature-stress tests. It is apparent that recrystallized ASTAR-811C has significantly better creep properties than the cold worked material. Increasing the final annealing temperature results in still further improvement in creep properties. The change is gradual though, with specimens annealed at 3400° F and below showing considerable data scatter, while there is little if any difference between the specimens annealed at 3600° and 3800° F. The spread in the band for the annealed specimens amounts to a vertical separation of about 5 ksi, or a horizontal separation of about 150° F in test temperature. (A change by one unit in the Larson-Miller parameter in fig. V-3 is equivalent to a change in test temperature of about 50° F.)

In figure V-4 the creep rate is plotted against stress for each test temperature. The circular symbols in this figure are for specimens annealed between 3000° and 3400° F, while the triangles represent specimens annealed at 3600° and 3800° F, the annealing time being 5, 30, and 60 minutes at each temperature. The trend of decreasing creep rate with increasing annealing temperature is again evident.

The solid-line and dashed-line curves in figure V-4 are plots of equations (1) and (2), respectively:

$$\dot{\epsilon} = A e^{B\sigma} e^{-Q/RT} \quad (1)$$

$$\dot{\epsilon} = A \sigma^n e^{-Q/RT} \quad (2)$$

These curves were drawn using values for the constants in equations (1) and (2) determined by treating all of the creep data as a group, that is, irrespective of heat treat condition. The values for these constants determined by a least-squares-computer program are as follows:

Eq. (1)($e^{B\sigma}$)			Eq. (2)(σ^n)		
A, %/hr	B, (ksi) ⁻¹	Q, kcal/mole	A, (%/hr)/(ksi) ⁿ	n, dimensionless	Q, kcal/mole
1.6×10 ¹⁴	3.73	135.5	1.5×10 ⁸	4.61	111.8

No particular physical significance is attached to the values obtained for these constants at the moment. However, equation (1), in particular, is useful for estimation purposes.

The effect of heat treatment on creep is shown in figure V-5 in terms of conventional (constant T, σ) creep curves. The specimen annealed at 3000°F shows a monotonically increasing creep rate, while the sample annealed at 3600°F shows a linear creep rate out to at least 1000 hours. The difference in their creep behavior therefore increases with increasing time or strain. These two creep curves also depict the magnitude of the effect on creep due to the range of heat treatments being investigated. It is noted that the two curves start out about the same and even after 50 hours it is difficult to distinguish between them. This suggests the possibility that the incremental creep tests may minimize the differences in creep behavior due to heat treatment effects because of the short time increments employed at each combination of temperature and stress.

Microstructure

After moderate to heavy cold working, ASTAR-811C recrystallizes in 1 hour at about 2400° to 2600°F . With increasing annealing temperature and time the grain size increases (fig. V-6) and the precipitates present in the cold worked material are gradually taken into solution (figs. V-7 to V-9). The precipitates in ASTAR-811C have been identified as the hexagonal close packed tantalum rich dimetal-carbide, $(\text{Ta}, \text{W})_2\text{C}$, regardless of the mechanical-thermal conditions.

The optical microstructure of samples annealed at 3600°F and above appears to be single phase (fig. V-9). There is no doubt that the carbides have been taken completely into solution. However, reprecipitation on a scale unresolvable by optical microscopy occurs during cooling, as shown by the electron transmission micrographs in figure V-10.

The precipitates shown in figure V-11 were extracted from creep specimens after being tested as shown in figure V-5. The initial precipitates (fig. V-10) in the specimen annealed at 3630°F grew into long, thin platelets (fig. V-11(b)) during creep testing. These precipitates are still much smaller than those in the specimen annealed at 3000°F (fig. V-11(a)). Figure V-12 shows a transmission micrograph of the creep specimen which had been annealed at 3630°F prior to testing. The dislocations appear to be pinned by the small precipitates and tangled around the larger ones.

DISCUSSION AND FUTURE WORK

The two principal microstructural features which change for the range of heat treatments investigated are grain size and precipitate dispersion. Since both of these features can influence creep deformation, it is of interest to consider their relative contribution to the creep resistance of ASTAR-811C. Creep rate (from incremental tests) is plotted as a function of grain size in figure V-13 for several combinations of test temperature and stress. The circular points represent specimens annealed at 3400⁰ F (below carbon solvus) and below while the triangular points are for specimens annealed at 3600⁰ F (above carbon solvus) and above. There is little difference in the creep rates of specimens annealed at the higher temperatures (above the carbon solvus), even though the grain size varied by a factor of 4 or 5. In view of the observed changes in precipitate structure, it is believed that the trends shown in figure V-13 reflect for the most part the contribution of the precipitate dispersion.

Experimental work now in progress concerns the influence of cooling rate and various thermal-mechanical processing schedules, including welding, on the creep resistance of ASTAR-811C.

Figure V-1. - Creep behavior of ASTAR-811C during incremental stress and temperature testing. Specimen 1-34-30-H-C; prior treatment, 1/2 hour at 3400° F; grain size, 0.056 millimeter.

Figure V-2. - Effect of test sequence on creep behavior of ASTAR-811C during incremental stress and temperature testing. Prior treatment, 1/2 hour at 3600° F, cooled 50° F per minute.

Figure V-3. - Larson-Miller plot of incremental creep test data on ASTAR-811C.

Figure V-4. - Temperature and stress dependence of creep of ASTAR-811C.

Figure V-5. - Effect of heat treatment on creep of ASTAR-811C in conventional test at constant stress (15 ksi) and constant temperature (2400° F).

Figure V-6. - Grain size of ASTAR-811C as function of annealing temperature and time. Specimen thickness, 0.035 inch, reduced 33 percent by cold working.

Figure V -7. - Microstructure of ASTAR-811C as-cold-worked 33 percent. Optical micro-graphic montage, X1500.

Figure V-8. - Microstructure of ASTAR-811C after annealing cold-worked material (fig. V-7) for 5 minutes at 3000^o F. Optical micrographic montage, X1500.

Figure V-9. - Microstructure of ASTAR-811C after annealing cold-worked material (fig. V-7) for 30 minutes at 3600° F. Optical micrographic montage, X1500.

(a) Interior of grain.

(b) Grain boundary.

Figure V-10. - Electron transmission micrographs of ASTAR-811C after annealing for 1 hour at 3630° F.

(a) Annealed 1 hour at 3000° F and creep strained 2 percent in 500 hours.

(b) Annealed 1 hour at 3630° F and creep strained 2 percent in 1000 hours.

(c) Annealed 1 hour at 3630° F and creep strained 2 percent in 1000 hours. X40 000.

Figure V-11. - Electron micrographs of tantalum dimetal carbide (Ta_2C) precipitate extracted from creep specimens (fig. V-5) tested at 2400° F and 15 ksi.

Figure V-12. - Electron transmission micrograph of ASTAR-811C creep specimen after testing at 2400° F and 15 ksi to 2 percent strain in 1000 hours. Pretest heat treatment, 1 hour at 3630° F. (See fig. V-5 for creep curve and fig. V-11 for pretest microstructure.)

Figure V-13. - Creep rate (incremental tests) of ASTAR-811C as function of grain size for several combinations of test temperature and stress.