EVIDENCE OF HETEROGENEOUS SUBSTRUCTURE DEVELOPMENT DURING PRIMARY CREEP OF Ti-6Al-2Sn-4Zr-2Mo

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Introduction

In conventional titanium alloys as well as in more advanced titanium alloys based upon the ordered Ti_3Al (DO_{19} structure), primary creep is a very important aspect of the high temperature creep behavior. This is due in part to the fact that depending on the initial microstructure, primary creep strains as large as 1.0% can be obtained in titanium alloys. Primary creep strains of this magnitude far exceed most design limitations for high temperature components which will be employed under conditions where creep is an important material property. Typical design requirements for Ti-6Al-2Sn-4Zr-2Mo impose a limitation of 0.1% creep strain in 35 hours at 510°C and 241 MPa.

Although microstructures which lead to low primary creep strains and lower minimum strain rates are fairly well established, a fundamental understanding of why is still very much lacking. Following Gibeling and Nix [1] we have monitored the recoverable anelastic strain and have analyzed the magnitude and the kinetics of the time dependent backflow. By comparing these measurements with those obtained on aluminum, copper and lead [1] we are able to make a connection between the heterogeneous sub structure which develops during primary creep of Ti-6Al-2Sn-4Zr-2Mo and the anelastic backflow. We are also able to show that the initial microstructure of this material plays a significant role in the development and evolution of substructure during primary creep.

Our preliminary correlation between the development of dislocation substructure during primary creep including the role of initial microstructure and the anelastic strain following forward creep in Ti-6Al-2Sn-4Zr-2Mo are presented and discussed in this paper

Experimental Procedure

Material:

A commercial grade of Ti-6Al-2Sn-4Zr-2Mo (hereafter Ti-6-2-4-2) was used for this work. The material was in the form of a large forged ring. Preliminary metallographic analysis indicated that the microstructure was uniform throughout the part and consisted of a mixture of equiaxed primary alpha phase (≈ 40 vol %) in a transformed beta matrix [2]. This is a typical microstructure for material requiring a reasonable balance of tensile, fatigue and creep properties. This microstructure will hereafter be referred to as the as-received microstructure.

Creep Specimens and Testing:

Several creep specimen blanks were cut from the Ti-6-2-4-2 forging. In order to produce a creep resistant microstructure by which to compare the as-received microstructure, some of the specimen blanks were given a 0.5 hour heat treatment at 1000°C followed by an air cool. Following heat treatment, all specimen blanks were fabricated into threaded round creep specimens having a gauge diameter of 12.7 mm and a length of 25.4 mm. Grooves were machined onto the shoulders of the specimens for placement of the creep extensometer.

Creep testing was performed on constant load Satec Systems creep machines having a 16:1 lever arm ratio. Creep strain measurements were obtained using dual dial gauge extensometers having a strain resolution of 10^{-5} . Creep temperatures were measured by placement of two high temperature thermocouples directly to the specimen gage section. All temperatures were controlled to within $\pm 2^\circ\text{C}$ of the desired temperature. Uniaxial tension creep tests were conducted on specimens of each microstructure at 510°C and 538°C at an initial applied stress level of 344.7 MPa. Following creep deformation into the minimum strain rate region at 510°C, specimens of each microstructure were given nearly full unloadings by employing an 87% stress reduction. Under these conditions, the load remaining on the specimen is high enough to maintain axial specimen alignment however, no continued forward creep is expected. Creep strain measurements were taken during the load removal which was conducted manually.

Following the load removal, the time dependent anelastic strain was measured on each specimen with measurements being continued out to roughly 27 hours. Strain measurements were taken at 6 min intervals over the first five hours following unloading.

Results

Microstructures:

The microstructures of the as-received material as well as the material heat treated at 1000°C are presented in Figure 1. For the as-received material, a typical microstructure consisting of a relatively large volume of primary alpha in a matrix of transformed beta is exhibited. The volume of the primary alpha phase has decreased significantly following heat treatment at 1000°C relative to that of the as received microstructure.

Creep Deformation and Anelastic Backflow:

The creep deformation response of the two microstructures is presented in Figure 2. The strain rate is plotted against strain for creep tests at 538°C and 344.7 MPa. From Figure 2 several points are noteworthy. First, the primary creep transient of the as-received material exceeds that of the heat treated material. Second, the strain rates are significantly lower in the minimum strain rate region for the heat treated structure compared to the as-received structure. Each of these trends are in excellent agreement with those reported in the literature [3]. Figure 3 shows the anelastic strain as a function of time following an 87% stress reduction for the as-received microstructure crept to 0.6% strain at 510°C. Figure 4 shows the anelastic strain as a function of time following an 87% stress reduction for the 1000°C heat treated material crept to 0.23% strain at 510°C. Each of the above strain levels are within the minimum strain rate region of creep. The anelastic strain rate is initially rapid and appears to decrease with increasing time. The trends shown in Figures 3 and 4 in general, are in very good agreement with those found for FCC aluminum, copper and lead by Gibeling and Nix [1].

Gibeling and Nix [1] have pointed out that in order to examine the physical origins of backflow, it is necessary to characterize both the magnitude and the kinetics of the backflow process. An analysis of the kinetics of the backflow process requires consideration of the dependence of rate on the driving force for backflow. Thus, following Gibeling and Nix [1] we have plotted the log of the anelastic strain rate versus the log of the remaining strain to be recovered which is a direct measure of the driving force for backflow. These plots are shown in Figure 5 for the as-received material and in Figure 6 for the material heat treated at 1000°C. Gibeling and Nix [1] have shown that for aluminum, copper and lead, two distinct regions corresponding to a nonlinear region at large remaining strains which transitions into a linear region with decreasing remaining strain are found. The slope of the log anelastic strain rate versus log remaining strain in the linear region has been found to range from about 0.8 to 1 for the high purity FCC Metals [1]. In Figures 5 & 6 of the

present work we have obtained only the linear portion of the data. Calculation of the slopes of Figures 5 & 6 gives a value of 0.62 for the as-received condition and a value of 0.76 for the material heat treated at 1000°C. The present values are slightly below the range obtained for the high purity FCC materials but still in reasonable agreement.

Given the limited data thus far, we can estimate the apparent activation energy for creep based on creep tests at constant initial applied stress at two temperatures. Table 1 lists the data with respect to the temperature dependence of the minimum strain rate.

TABLE 1 Summary of Creep Conditions and Results for Ti-6Al-2Sn-4Zr-2Mo

<u>Condition</u>	<u>Temperature</u> (°C)	<u>Stress</u> (MPa)	<u>Minimum Strain Rate</u> (Hr ⁻¹)
1000°C/0.5 hour	510	344.7	4.2 X 10 ⁻⁶
	538	344.7	1.48 X 10 ⁻⁵
As Rec'd	510	344.7	2.83 X 10 ⁻⁵
	538	344.7	9.84 X 10 ⁻⁵

Taking the data in Table 1 and employing the following formula

$$Q_c = -R \ln (\dot{\epsilon}_1 / \dot{\epsilon}_2) / (T_2 - T_1) / (T_1 T_2) \quad 1.$$

where Q_c is the apparent activation energy for creep and R is the gas constant we have for the as-received material $Q_c \approx 235$ kJ/mole and for the 1000°C /0.5 hour heat treatment, $Q_c \approx 238$ kJ/mole. Although qualitative, these values are in good agreement with published values for self diffusion in alpha titanium ($Q_{S.D.}$ 240 kJ/mole). This implies that creep under the present experimental conditions is governed by diffusion although there is not enough data to confirm any specific mechanism.

Discussion

Creep Deformation:

As discussed previously, higher primary creep strains and minimum strain rates are found in microstructures having higher volumes of primary alpha. Heat treatments approaching the beta transus, thus reducing the volume of primary alpha reduce both the primary creep strain as well as the minimum strain rate. This trend is well documented for titanium alloys [3]. Thiehsen et al. [3] have pointed out that correlation of the creep response with the volume of primary alpha phase only is likely an over simplification. The magnitude of the primary creep strain may be related to the dislocation source density upon initial loading. It is possible that the interfaces between the primary alpha phase and the transformed beta phase may be a source of high dislocation density. At this point, this is only postulated however, this suggestion does correlate with the trends regarding primary creep strains in titanium alloys. Another strong possibility is the residual dislocation density left from the forging or hot working operation. Although most alloys are given an annealing treatment following the hot working operation, the annealing temperature range can be quite large. Therefore, depending on the specific hot working parameters, such as temperatures and reductions, it is not unreasonable to propose that a substantial dislocation density available to contribute to creep deformation may be present following the annealing treatment. If this is the source of dislocations which contribute to the high primary creep strains in titanium alloys, this may in part explain the scattered results which have been reported in various studies in the literature [3]. At present we offer the above as possibilities realizing that more extensive and definitive work is needed in order to draw any sound conclusions.

Based on creep experiments at two temperatures at constant initial applied stress, the apparent activation energy for creep has been estimated and found to agree fairly well with the value for self diffusion in alpha titanium. At this time the data is not sufficient for elaboration on any specific creep

mechanism. The value of the creep activation energy does however suggest the possibility of diffusion controlled deformation processes under the present experimental conditions. These results are interesting given that the temperatures of the present creep experiments are on the order of $0.40 T_m$ (510°C) and $0.42 T_m$ (538°C) where T_m is the melting temperature of titanium (1943K). In addition, the primary creep transients exhibited under the present creep conditions would at least suggest dislocation motion as being involved in the creep deformation process.

Anelastic Backflow:

The plots presented in Figures 3 and 4 of the present study provide direct evidence of the development of a heterogeneous substructure and thus a nonuniform stress distribution within the Ti-6-2-4-2 creep specimens [4-6]. The basic shapes of the curves displayed in Figures 3 and 4 are in good agreement with those obtained on high purity FCC aluminum, copper and lead [1]. In comparing Figures 3 and 4 it is found that the anelastic strain recovered is somewhat less following the 1000°C heat treatment. It is important to mention that a relationship between anelastic strain recovered and grain size was found with larger grain sizes giving rise to less anelastic strain [1]. The microstructural unit size is larger following the 1000°C heat treatment (compare Figures 1a and b). We feel that in the present case, the low anelastic strain recovered following the 1000°C heat treatment reflects the fact that the driving force for backflow in this microstructure is reduced relative to that of the as-received microstructure.

Gibeling and Nix [1] have shown that the anelastic backflow process consists of two regions. This is found on plots of log anelastic strain rate versus log of the remaining strain. At large remaining strains the anelastic strain rate is rapid and decreases in a nonlinear fashion. This region has been attributed to the thermally activated glide of dislocations within the subgrain interior. As the remaining strain decreases, the anelastic strain rate decreases in a time dependent linear fashion. This has been attributed to thermally activated recovery processes within the subgrain walls [1,6]. In the present experiments, only the linear region of the back flow process was obtained (Figures 5 & 6). Although careful inspection of the data of Figure 5 (as-received microstructure) indicates a small portion of the nonlinear region at the largest remaining strain. The absence of the non linear region of backflow from the present data can be attributed to the limited strain and time resolution available from the experimental method. However these results, do indicate that the kinetics of anelastic backflow, of the present Ti-6-2-4-2 material exhibits very similar behavior to that of aluminum, copper and lead [1]. With respect to the present material, it is significant that the kinetics of anelastic backflow appears to be unchanged by heat treatment at 1000°C .

Gibeling and Nix [1] made a comparison between the amount of time dependent anelastic strain recovered and the elastic strain. It was found that for aluminum, the time dependent anelastic strain exceeded the elastic strain by 7 to 8 times, for copper the anelastic strain and the elastic strain were about equal and for lead, the anelastic strain was equal to about one quarter of the elastic strain. These results were attributed to the differences in stacking fault energy (SFE) of these materials. For the aluminum which has a high SFE, thermally activated glide within the subgrain interior as well as recovery within the subgrain walls which may be climb controlled, takes place rather easily due to the non extended dislocations. Thus, large time dependent anelastic strains are found relative to the elastic strains. For copper which has an intermediate SFE, thermally activated dislocation motion is more restricted due to the larger splitting width between extended partials, making glide and climb more difficult. In lead which has a very low SFE, thermally activated glide within the subgrain interior as well as climb within the subgrain wall is very difficult owing to the widely extended partial dislocations [1]. A comparison of the time dependent anelastic strain to the measured elastic contraction of the present Ti-6-2-4-2 reveals that the anelastic strain is roughly an order of magnitude less than the elastic strain regardless of the microstructural condition. It is not known whether the SFE of Ti-6-2-4-2 is known with precision however, the present results would suggest that compared to the pure FCC metals [1], thermally activated glide and recovery occurs with much greater difficulty within the present material. Given the complex two phase structure of Ti-6-2-4-2 these findings are not surprising. The preliminary results presented in this study suggest that the anelastic backflow process of Ti-6-2-4-2 may be related to the heterogeneous substructure and accompanying non uniform distribution of stresses which develop during primary creep just as has been shown for the FCC metals previously [1, 4-6].

Conclusions

Preliminary creep tests conducted on the titanium alloy Ti-6Al-2Sn-4Zr-2Mo show that both primary creep strain and the minimum strain rate decreases with decreasing volumes of primary alpha phase in the microstructure. This is in agreement with well established trends. We have suggested a relationship between the initial dislocation source density and the primary creep behavior and have pointed toward some microstructural variables which may give rise to a high initial dislocation source density. Measurements of anelastic backflow and the characterization of the kinetics of the backflow process indicates that the Ti-6Al-2Sn-4Zr-2Mo exhibits very similar behavior to that of pure FCC aluminum, copper and lead. It is suggested that the anelastic backflow provides direct evidence of a heterogeneous substructure and a nonuniform distribution of stresses within the material which drives the backflow process. The backflow process is discussed along the lines of what has been discussed regarding time dependent anelastic backflow in high purity FCC Metals. Finally, the apparent creep activation energy estimated from creep tests at two temperatures at constant initial applied stress indicates that creep under the present experimental conditions is diffusion controlled. At present no specific mechanism can be defined. More definitive and extensive work is planned in order to better address the issues regarding primary creep in titanium alloys.

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(a)

(b)

Fig 1. Microstructures of (a) as received material and (b) following 1000°C heat treatment.

Fig 2. Creep deformation response at 538°C and 344.7 MPa.

Fig 3. Backflow transient obtained for 87% stress reduction during minimum strain rate of as received Ti-6-2-4-2.

Fig 4. Backflow transient obtained for 87% stress reduction during minimum strain of Ti-6-2-4-2 following 1000°C heat treat.

Fig 5. Anelastic strain rate vs. remaining strain for as received material data of Fig. 3.

Fig 6. Anelastic strain rate vs. remaining strain for heat treated material data of Fig. 4.