Aging Effects on the Creep Behavior of the Near-Alpha Titanium Alloy Ti-1100

A.H. Rosenberger, A. Madsen, and H. Ghonem

During aging of the silicon-bearing, near-alpha titanium alloy Ti-1100 at the service temperature of 593 °C, the precipitation of Ti3Al and silicide particles has been observed. The objective of this article is to determine the influence of these precipitates on creep behavior. Stress relaxation tests, with the advantage of needing only a short time to produce a complete creep curve, were used to determine the creep behavior of Ti-1100. These accelerated creep tests reduce the chance of metallurgical changes that could occur in the long time required to perform conventional creep tests. The creep results obtained from the stress relaxation tests were verified with those obtained from conventional monotonic creep tests. Aging of the material for 500 h at 593 °C, where full precipitation of Ti3Al is assumed to be incomplete, resulted in a small decrease in creep resistance compared to the unaged condition. Specimens aged for 1000 h, which corresponds to the averaged condition in which the precipitation of Ti3Al is expected to be completed, were also examined. It was observed that these specimens yielded creep resistance similar to that of the unaged condition. Material aging for 1000 h followed by a special heat treatment, which results in the dissolution of Ti3Al particles, although not affecting the silicide precipitates, resulted in the poorest creep resistance of the aging conditions investigated. The major conclusion of this study is that the presence of Ti3Al has a beneficial influence on the creep resistance of Ti-1100 alloy.

Keywords
aging, creep, dislocations, silicide, titanium—1100,

1. Introduction

THE present trend of increasing temperatures and component stresses in the development of today's high-performance gas turbine engines is placing higher demands on the creep performance of materials. The simultaneous desire to increase the thrust-to-weight ratio of the engines has led to an increased interest in the use of titanium alloys. One concern, however, with the increased use of titanium in critical components is the long-term stability of the alloy and the changes in mechanical properties that may occur during use. Some components in the present commercial gas turbine engines may see service for over 10^5 h.[1] This amount of time at high service temperatures may result in metallurgical changes in the components of the hot section of the engine.[2]

Modern near-alpha titanium alloys typically contain silicon in concentrations ranging from 0.3 to 0.5 wt%, which has been shown to significantly increase the creep resistance of this class of materials.[3-5] This is due to silicon atoms being attracted to mobile dislocations, thereby increasing the dislocation drag stress and reducing creep rate.[4,5] However, this strengthening approach requires that the silicon remain in solution. Aging of these alloys, however, causes the silicon to be removed from solution, which precipitates in the form of silicides. This generally has a detrimental effect on the creep resistance of these alloys.[5]

Commercial near-alpha titanium alloys also contain aluminum in concentrations of approximately 6 wt%, which is a strong alpha phase stabilizer and solid solution strengthener. Under service conditions, this will form Ti_3Al precipitates, which have been shown to affect the slip character and deformation behavior of titanium alloys.[6,7] There are, however, minimal data available regarding the effect of Ti_3Al precipitates on creep behavior.

The objective of this study is to determine the influence of aging on the creep performance of Ti-1100. The aging and creep testing temperature of 593 °C was chosen as relevant to the service conditions under which the Ti-1100 alloy will be used. Under these conditions, the combined influence of Ti_3Al and silicide precipitates on creep are examined as a function of aging time based on their natural formation kinetics, which has been examined by Madsen and Ghonem.[8] In an effort to separate the creep influence of silicide and Ti_3Al precipitates, a post-aging heat treatment (PAHT) devised by Madsen[9] will also be incorporated. This treatment reduces the size and/or number of Ti_3Al precipitates due to their dissolution while leaving the silicon distribution in the alloy relatively unchanged.

To develop a baseline data set for unaged material, test techniques were required that did not involve a long duration of time so that precipitation would not take place during the test. This was achieved using accelerated creep tests similar to those of Woodford et al.[10] Here, the creep behavior of the material is determined by a series of stress relaxation tests (SRT) where the stress relaxation versus time during a strain hold is converted to the strain rate versus stress behavior. These tests typically last less than 20 h and result in less than 0.2% inelastic strain so that several tests can be conducted on a single specimen. Woodford et al.[10] have shown good correlation between the creep behavior determined by stress relaxation tests and conventional creep tests for a nickel-base superalloy.

The next section of this article describes the material used in the study and the details of the stress relaxation tests. The creep results for different aging conditions are then presented, as well as a comparison of the naturally aged material with that given

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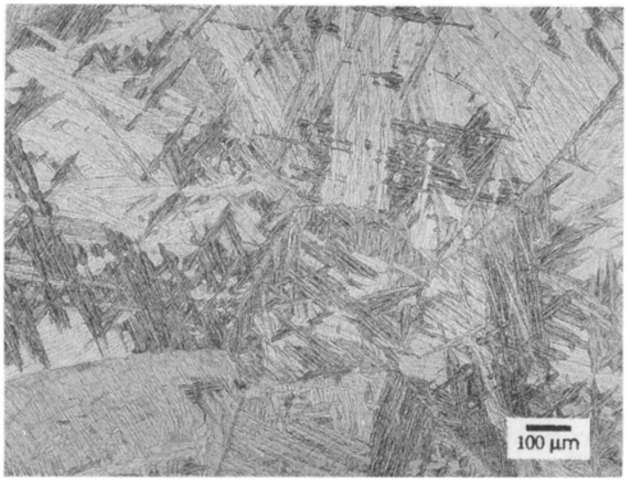

Fig. 1 Optical micrograph of the microstructure of Ti-1100 swaged bar.

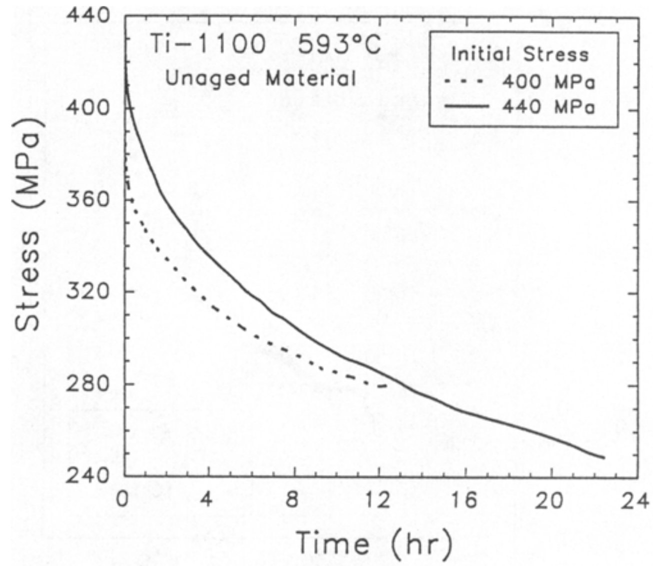

Fig. 2 Stress relaxation versus time for as-received material with initial stress levels of 400 and 440 MPa.

for the post aging heat treatment. Finally, these results are discussed in terms of the effect of precipitation on the creep resistance of the Ti-1100 alloy.

2. Materials and Experimental Procedure

The material used in this study was obtained from Timet Corporation in the form of 25-mm diameter bar. The bar was swaged and solutionized above the beta transus, air cooled, and given an 8-h stabilization treatment at 593 °C. The nominal chemical composition (wt%) of Ti-1100 is Ti-6Al-2.8Sn-4Zr-0.4Mo-0.45Si with 0.07 wt% O and 0.03 wt% Fe maximum. The above thermomechanical processing resulted in the Widmanstätten microstructure shown in Fig. 1. Based on optical microscopy, there was no observable grain size variation in the material due to swaging. The mechanical properties and microstructure of this material is similar to that of the plate material used in other studies.[8] Stress relaxation tests were conducted on cylindrical, threaded end specimens with a gage length of 40 mm and a diameter of 10 mm in accordance with ASTM Standard E 328-86.

Stress relaxation tests were conducted using a servohydraulic loading frame with computer-controlled waveform generation and data acquisition system. Specimens were heated to 593 °C using a low-frequency induction heating unit with temperature controlled to ±1 °C during the test. Prior to each test, the load train was aligned using a metal pot. Tests were conducted under total strain control using an axial quartz rod extensometer with a sensitivity of better than 10^{-5} strain. The stability of the strain control was maintained by the use of a constant-temperature recirculating water-cooling device attached to the extensometer. With the specimen under strain control and at elevated temperature, the accuracy of the control was better than $\pm 2.5 \times 10^{-5}$. The specimens were loaded with a strain rate of 10^{-4} s^{-1} to stresses of 400 or 440 MPa, which are slightly above the proportional limit of the material. On reaching the predetermined stress, the strain was held constant, and the time, strain, and load were simultaneously collected. The tests were typically terminated when the stress decreased to half of this initial value or the strain rate decreased to $\approx 10^{-9}$ s^{-1}.

Blanks of the material were aged in a resistance furnace at 593 °C in air and machined to the final specimen dimensions. The aging times of 0, 166, 500, and 1000 h were chosen based on the study by Madsen et al.,[8] where it was determined that 166 h corresponded to the peak in the hardness curve where only silicide precipitation was apparent in bright-field TEM. The aging during of 1000 h resulted in the overaged condition where complete precipitation of Ti_3Al and silicide particles was attained. The time of 500 h was chosen as an intermediate condition. All of the material initially received an 8-h stabilization treatment at 593 °C, as mentioned above, which in this study is referred to as the unaged material. No measurements of the oxygen content were performed after aging treatments. However, it was assumed that final machining of the specimen would remove any oxygen-affected layer. Also, Winstone et al.[4] noted a negligible influence of oxygen on the creep performance of a Ti-1Si alloy with oxygen concentrations of 1060 and 1800 ppm. The post-aging heat treatment (PAHT)[9] is based on the concept of the critical ordering temperature of an ordered precipitate. Heat treating a material above its critical ordering temperature leads to the destruction of the long-range order in a precipitate system, thereby destroying the particle. Hence, the temperature and time of the PAHT were chosen to result in the dissolution of the Ti_3Al while leaving the other elements of the microstructure unchanged, especially the silicide precipitates. The preparation of the PAHT material consists of 1000-h preaging at 593 °C to form both Ti_3Al and silicide precipitates. The material was then heated to 750 °C and held at this temperature for a period of 4 h followed by fan cooling. This temperature/time combination was sufficient to disorder the Ti_3Al particles without dissolving the silicide particles.

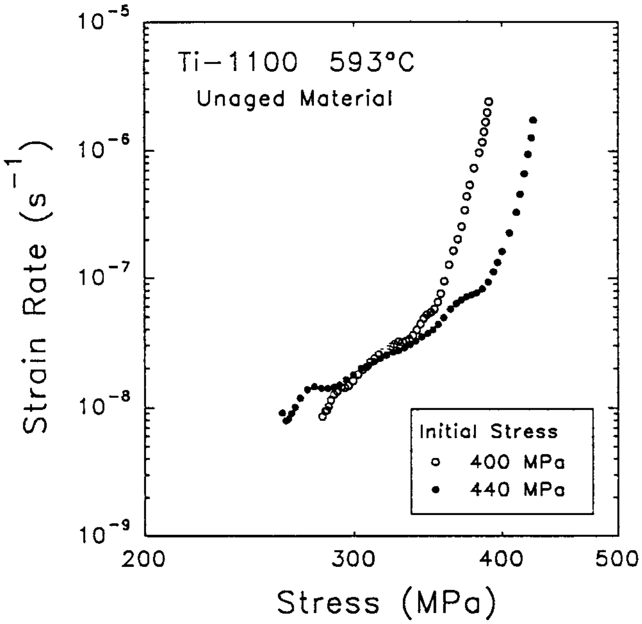

Fig. 3 Stress versus inelastic strain rate for as-received material with initial stress levels of 400 and 440 MPa.

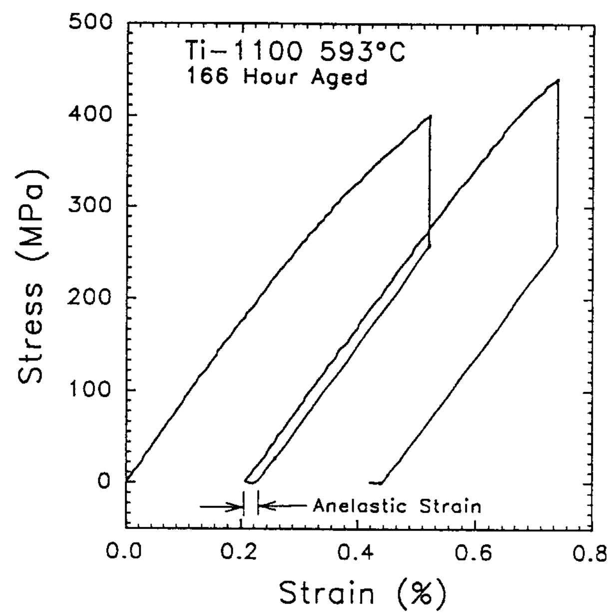

Fig. 4 Stress versus strain for two relaxation runs of the 166-h aged material showing anelastic strain recovery at zero stress.

3. Results and Analysis

Typical stress versus time results for the as-received material tested with initial stress values of 400 and 440 MPa are shown in Fig. 2. The initial stresses were chosen to be slightly above the proportional limit to minimize transient behavior.[11] In all tests, this stress versus time was converted to stress versus stress rate using the seven-point polynomial technique recommended by ASTM Standard E 647-88. The inelastic strain was calculated from

$$\varepsilon_e + \varepsilon_I = \varepsilon_t = \text{Const} \quad [1]$$

where ε_e is the elastic strain, ε_I is the inelastic strain, and ε_t is the total strain. The inelastic strain rate is calculated from the time derivative of Eq 1, and since ε_t is a constant, thus $\dot{\varepsilon}_I$ can be written as

$$\dot{\varepsilon}_I = -\dot{\varepsilon}_e = \frac{-1}{E}\frac{d\sigma}{dt} \quad [2]$$

where E is the elastic modulus measured during the loading and σ is the stress. In this fashion, the stress relaxation rate, $d\sigma/dt$, is converted to the creep strain rate, $\dot{\varepsilon}_I$. Figure 3 shows the strain rate versus stress calculated from Eq 2 for the two different initial stress levels, 400 and 440 MPa, for the relaxation data shown in Fig. 2. The non-unique behavior in the early (high strain rate) portion of each curve has been related to the existence of a recoverable anelastic strain. Similar results were obtained for high-purity aluminum,[11] where it was shown that the transient behavior is due to the accumulation of anelastic strain, which occurs due to the initial loading. Here, the inelastic strain, ε_I, is actually composed of the creep strain, ε_c, and the anelastic strain, ε_{an}, that results in the initially high inelastic strain rate, $\dot{\varepsilon}_I$. Only after saturation of the anelastic strain, i.e., $\dot{\varepsilon}_{an} = 0$, does the inelastic strain rate represent the pure creep behavior of the material. A characteristic of anelastic strain is that it is normally recovered on release of the applied stress. The stress versus strain curve for the 166-h aged material is shown in Fig. 4. After the completion of the first run, the specimen was maintained at test temperature and held at zero stress for approximately 30 min, which resulted in the recovery of approximately 0.02% strain. The existence of this recoverable (noncreep) strain indicates that the initial transient strain rate could be a result of the anelastic strain. The transients could not be eliminated due to the test technique, but are readily apparent in the results and were removed from the data sets prior to the analysis. An important result shown in Fig. 3, however, is that the results are self-consistent and repeatable as long as the transient effects are eliminated. After the initial transient response, the curves represent the same creep behavior, which is independent of the initial stress level or the previous number of tests performed on the same specimen. A similar repeatability of the results has been demonstrated by Woodford et al.[10]

The creep behavior of Ti-1100 after various aging times as well as the PAHT technique is shown in Fig. 5. In general, the materials follow the same creep trend regardless of aging time with the exceptions of the 500-h aged material and the PAHT material. Both of these show a decrease in the creep resistance, with the PAHT material exhibiting the poorest creep resistance of all tests. The influence of running multiple tests on a single specimen is demonstrated in Fig. 6, which shows five runs on the same specimen of the 500-h aged material. The first two runs show a higher strain rate as compared to the last three tests. The implications of this behavior will be discussed in the following section. The data in this investigation, as shown in Fig.

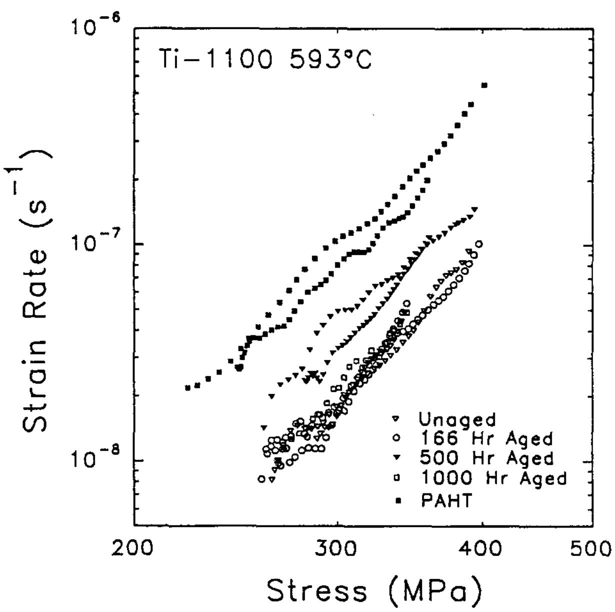

Fig. 5 Strain rate versus stress for aging times ranging from 0 to 1000 h. Results from the post-aging heat treatment (PAHT) material is also shown.

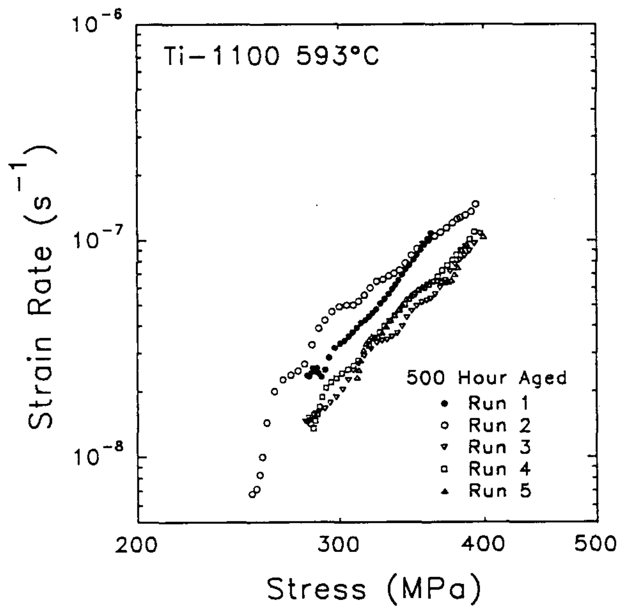

Fig. 6 Strain rate versus stress for 500-h aged material for five relaxation runs.

Table 1 Norton law coefficients for aged Ti-1100 and PAHT material

Aging time	Norton law coefficient, $MPa^{-n}\ s^{-1}$	Norton law exponent
Virgin	5.777×10^{-23}	5.855
166 h	7.549×10^{-22}	5.411
500 h (first two runs)	2.094×10^{-21}	5.344
500 h (first three runs)	1.589×10^{-22}	5.704
1000 h	1.114×10^{-22}	5.763
1000 h PAHT	7.610×10^{-21}	5.269

5 and 6, are accurately described by a Norton power law equation of the form

$$\dot{\varepsilon} = B\sigma^n \qquad [3]$$

where B is the Norton law coefficient and n is the Norton law exponent. Table 1 shows the Norton law coefficient and exponent for the different aging treatments in the study based on a least-squares analysis. Note that the exponent is relatively constant at a value of approximately 5.6, whereas the coefficient varies by over an order of magnitude for the different conditions.

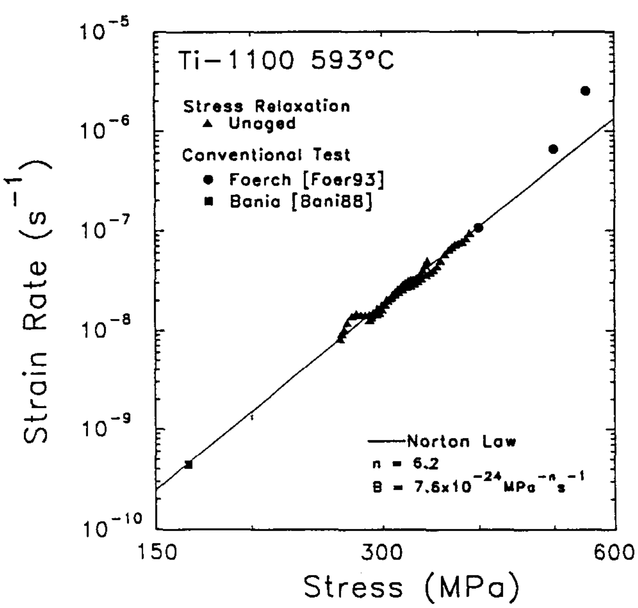

Fig. 7 Comparison of the creep behavior determined from stress relaxation tests and conventional monotonic creep tests.

4. Discussion

In the current literature, the creep behavior determined from stress relaxation tests has not been thoroughly compared to long-term creep tests to validate the stress relaxation test technique. In Ti-1100, a comparison of this type can be made for unaged material using existing data in the literature, which have been generated using traditional constant-load creep tests. Figure 7 compares the results from the present study with the high-stress creep behavior from Foerch[12] and low stress behavior from Bania.[13] The correlation of the data from the two test methods is good, indicating a Norton law coefficient of 7.6 $\times$ 10^{-24} MPa^{-n} s^{-1} and an exponent of 6.2. These coefficients, determined over a wider stress range, are similar to values de-

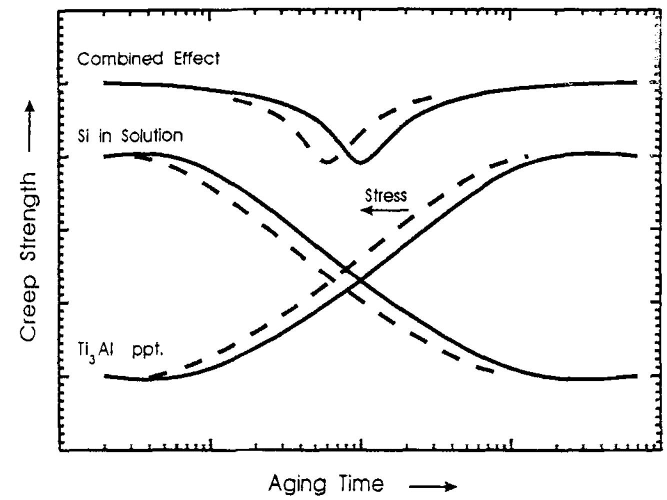

Fig. 8 Schematic of the influence of Ti_3Al and silicide precipitation on the creep behavior versus aging time. Also illustrated is the possible influence of stress on the precipitation (dashed line).

termined from the SRT data alone. The creep rate for the 550 MPa stress level from the results of Foerch is higher than the trend of the data, which appears to indicate an upper limit of applicability of the Norton power law in this material. The stress level of 550 MPa is also above the yield stress of Ti-1100, which may result in a different creep mechanism.

Previous studies have shown that removing silicon from solution through the formation of silicides is detrimental to the creep behavior of titanium alloys.[5,14] However, these studies typically are performed on simplified binary or ternary titanium alloys, with the heat treatment chosen to precipitate a particular morphology of silicide within a relatively short period of time.[5] In the current study, the aging temperature was chosen to be the maximum use temperature of the alloy (593 °C) to determine the change of the creep behavior during the service use conditions of the material. In this case, the creep behavior is affected by two naturally occurring precipitates, namely silicides and Ti_3Al particles. In Ti-1100, Madsen and Ghonem[8] have shown that silicides start to precipitate on alpha platelet boundaries at times less than 166 h and coarsen through ≈1000 h, whereas smaller Ti_3Al particles precipitate homogeneously throughout the material at a slower rate. These precipitates are apparent in bright-field TEM observation at 1000 h, although it is probable that some ordering occurs at lesser times. From these observations, it could be assumed that the specimens in this study represent a range of precipitation conditions from very few precipitates in the unaged condition to both silicide and Ti_3Al being fully existent in the 1000-h aged condition. The fact that the creep behavior for the unaged material and the 1000-h aged material is the same indicates that the decrease in creep resistance through the loss of silicon in solution is balanced by the creep-strengthening effect of Ti_3Al particles. Assadi et al.[14] have also proposed a creep-strengthening mechanism due to short-range ordering of aluminum during 550 °C aging of an experimental Ti-5Al-5Zr-1.02Mo-0.53Si (wt%) alloy. However, they did not present any direct proof of this mechanism. The beneficial influence of Ti_3Al on the creep resistance is further supported in this study by the PAHT results. Because PAHT reduces the size and number of Ti_3Al particles,[9] while producing no apparent change in either the silicides or general features of the microstructure, the change in the creep resistance between the 1000-h aged material and the PAHT material must be primarily due to a reduction in the ability of the Ti_3Al to control dislocation motion. It has been shown[5] that creep in a similar titanium alloy (IMI 685) is predominantly due to dislocation creep. If this is also the case in Ti-1100, then the presence of a homogeneously nucleated precipitate would decrease the creep rate as a function of its ability to inhibit dislocation motion.

The change in the creep behavior of the 500-h aged material due to multiple test runs appears to indicate a change in the material creep performance rather than an influence of the test procedure. The final three runs are repeatable and represent an identical creep behavior, which indicates that the influence of the accumulated strain due to the multiple runs is not significant enough to affect the measured creep behavior of the alloy. The aging time increase during the first two runs, however, is less than 26 h, which alone would produce a small change in particle precipitation. A possible mechanism that could account for the change in creep performance is stress-enhanced precipitation of Ti_3Al. This would speed the formation of creep-strengthening Ti_3Al particles such that the creep behavior would mimic the 1000-h aged material at much shorter times. A proposed map of the influence of the two precipitates on the creep performance is shown in Fig. 8. Here, with an increase in aging time, the silicon in solution is reduced, which decreases the creep resistance while the Ti_3Al particles are precipitated, thus causing an increase in the creep resistance of the alloy. The minimum in creep resistance, represented experimentally by the 500-h aged material, is due to an advanced stage of silicide precipitation, i.e, reduction of silicon in solution, and an immature stage of Ti_3Al precipitation. Overall, the creep behavior of Ti-1100 is not strongly affected by aging due to the overlap in the precipitation of silicides and Ti_3Al. The application of stress may affect both the silicide and Ti_3Al precipitation, which would shift the minimum in creep performance to the left as indicated. Direct proof of the map shown in Fig. 8, however, would require extensive TEM analysis of deformed specimens at different aging times, which was not attempted in this study.

5. Conclusions

A series of stress relaxation tests has been performed on Ti-1100 subjected to different aging times at a temperature of 593 °C. The influence of the natural formation of silicide and Ti_3Al precipitates has been demonstrated while a post-aging heat treatment technique has been incorporated to determine the separate influence of the precipitates. The main findings of the study can be summarized as follows:

- The creep behavior measured by a stress relaxation test is consistent with the behavior from typical monotonic creep tests. The benefit of the stress relaxation test in determining the creep behavior of a material lies in the speed in which a large amount of data can be generated at a constant metallurgical state.

- The creep performance of Ti-1100 in the as-received condition is identical to that after 1000 h aging at 593 °C. The material aged for 500 h indicated a small decrease in creep resistance. Generally, the overall creep behavior is not strongly affected by the aging time within the range of times investigated in this study.
- Creep performance appears to be aided by homogeneously precipitated Ti_3Al particles. This is supported by the creep behavior of post-aging heat treatment material, which has poor creep resistance.

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