

# Separating the Effects of $Ti_3Al$ and Silicide Precipitates on the Tensile and Crack Growth Behavior at Room Temperature and 593 °C in a Near-Alpha Titanium Alloy

A. Madsen and H. Ghonem

Aging of silicon-bearing, near-alpha titanium alloys produces two types of precipitate,  $Ti_3Al$  and  $(TiZr)_6Si_3$  (silicide). A postaging heat treatment that relies on the phenomenon of the critical ordering temperature is used to remove the  $Ti_3Al$  precipitate while leaving the silicide intact. Three materials—unaged (precipitate free), overaged ( $Ti_3Al$  plus silicide), and post aging heat treated (silicide only)—are compared. The objective is to identify the separate effects of the  $Ti_3Al$  and silicide precipitates on tensile and fatigue crack growth behavior at room temperature and 593 °C. The  $Ti_3Al$  precipitate is largely responsible for the increase in yield stress and for the decrease in ductility at both test temperatures. In contrast, the increase in room temperature fatigue crack growth rate associated with aging is attributed to the silicide, with  $Ti_3Al$  playing only a minor role. Aging produces a slight improvement in fatigue crack resistance at 593 °C, which also appears to be due to the presence of the silicide precipitate.

## Keywords

titanium alloys, fatigue crack, aging, heat treatment

## 1. Introduction

AGING of silicon-bearing, near-alpha titanium alloys results in the formation of two types of precipitate,  $Ti_3Al$  and  $(TiZr)_6Si_3$  (silicide) (Ref 1-5). These two precipitates differ markedly in their size and formation characteristics. At aluminum concentrations below 8 wt%, the  $Ti_3Al$  precipitate nucleates homogeneously and remains coherent with the matrix with a size ranging from 10 to 100  $\mu m$  depending on the aluminum concentration, aging time, and temperature. In contrast, the silicide nucleates heterogeneously, is incoherent with the matrix, and is larger and ellipsoidal in shape. In near-alpha alloys that have a Widmanstätten microstructure, the silicides tend to form preferentially along the alpha platelet boundaries.

A new high-temperature titanium alloy, designated Ti-1100, has been employed to study precipitation and its effects on mechanical behavior in modern silicon-bearing, near-alpha titanium alloys. A previous study showed that aging of Ti-1100 at 593 °C requires times greater than 1000 h to complete the formation of both the aluminide and the silicide precipitates (i.e., the overaged condition) (Ref 1). Peak hardness occurs at approximately 166 h aging time at 593 °C. In another study, the changes in uniaxial tensile and fatigue crack growth behavior that accompany aging were also studied. The focus was placed on two test temperatures, room temperature and 593 °C, which is the intended service temperature of Ti-1100 (Ref 6). At both

temperatures, aging causes an increase in the yield stress and a decrease in ductility. Also aging increases the fatigue crack growth rate at room temperature, while at high temperature, aging produces a slight decrease in growth rate. However, since this alloy system contains two types of precipitates, it has been difficult to assign the origin of these changes in tensile and fatigue behavior to a particular precipitate.

The primary purpose of the present work is to distinguish between the influence of the silicide and  $Ti_3Al$  precipitates on the monotonic uniaxial tensile behavior and the cyclic fatigue crack growth behavior at room temperature and 593 °C. To study the separate roles of the two precipitates, a postaging heat treatment (PAHT) was devised that produces a material in which only one of the two precipitates, the silicide, is present. The removal of  $Ti_3Al$  by the PAHT relies on the phenomenon of the critical ordering temperature. Figure 1 shows schematically the comparison that is made possible among the unaged, overaged ( $Ti_3Al$  plus silicide), and PAHT (silicide only) materials. The next section describes aspects of the experimental procedure, followed by a discussion of the results and a summary of major conclusions.

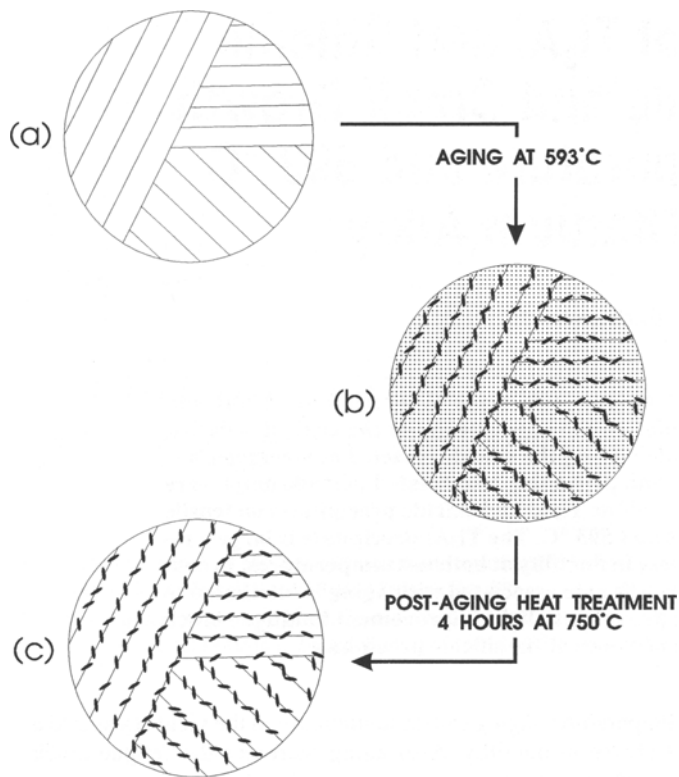
## 2. Experimental Procedure

The alloy used in this study was provided by TIMET Corporation as  $1/2$  in. pancake plate that was forged above the beta transus, solutionized above the beta transus for  $1/2$  h, and air-cooled followed by an aging of 8 h at 593 °C. The chemical composition of the alloy is Ti-6Al-2.8Sn-4Zr-0.4Mo-0.45Si-0.070- $<0.03Fe$ . This heat treatment resulted in a Widmanstätten microstructure, which is described in detail in Ref 1, 7, and 8.

Tensile specimens were end-threaded, smooth, round specimens with a diameter of 0.30 in. and a uniform gage length of 1.28 in. Heating of tensile specimens during tests performed at

A. Madsen\* and H. Ghonem, Mechanics of Solids Laboratory, Department of Mechanical Engineering and Applied Mechanics, University of Rhode Island, Kingston, RI 02881, USA.

\*Current address: Dept. of Materials Science and Engineering, University of Washington, Seattle, WA 98195, USA.

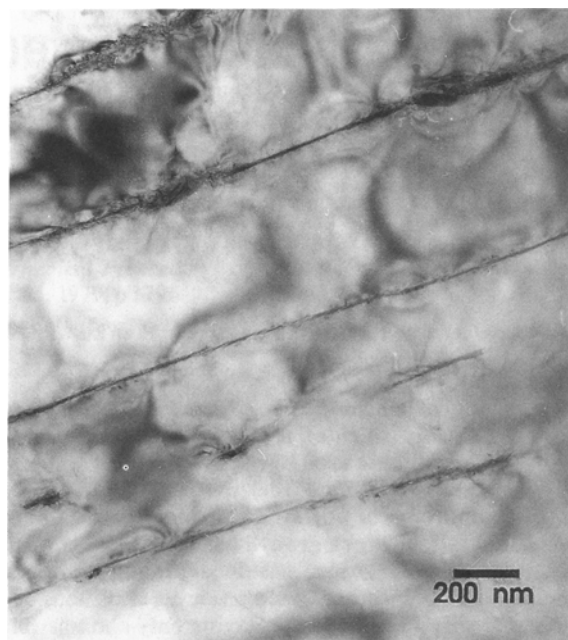


**Fig. 1** Schematic of the PAHT concept. (a) Unaged material representing alpha platelet colonies. (b) Overaged material with ellipsoidal silicides along the alpha platelet boundaries and Ti<sub>3</sub>Al uniformly distributed in the matrix. (c) PAHT material where the silicide remains unchanged, but the Ti<sub>3</sub>Al precipitate was removed by exposure above the critical ordering temperature

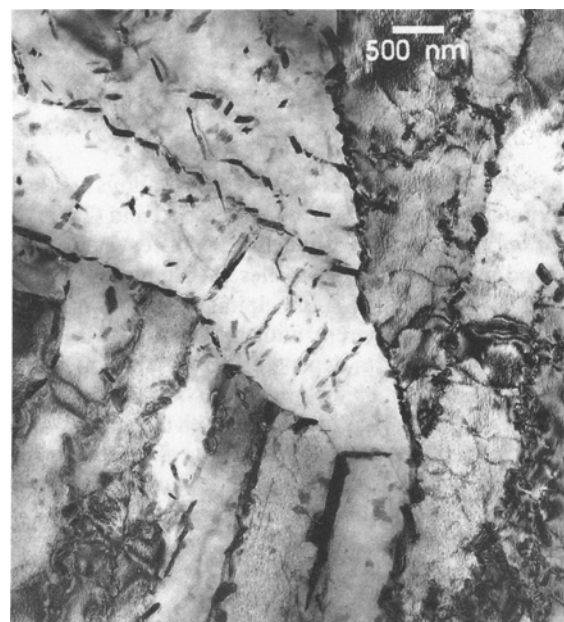
593 °C was accomplished with a low-frequency induction system.

Compact tension (CT) specimens dimensioned according to ASTM standard E 647-88 were used to investigate fatigue crack growth behavior. Two sizes of CT specimens were employed: W (width) = 40 mm (1.575 in.), B (thickness) = 10 mm (0.394 in.), which is designated as CT-40; and W = 25 mm (0.984 in.), B = 6 mm (0.236 in.), which is designated as CT-12.5. Both tensile and fatigue specimens were machined from the plates with the loading axis of the tensile specimen corresponding to the loading direction of the CT specimens. The primary method of crack length measurement was optical using a traveling microscope. Direct current potential drop was also used to supplement optical measurements when occasionally required. Compact tension specimens were heated using a resistive heating furnace. Additional details regarding the execution of the tensile and fatigue tests are found in Ref 6.

Aging prior to testing was accomplished in air in a resistive heating furnace at 593 °C. This aging temperature was chosen to coincide with the intended service temperature of Ti-1100. All aged specimens were exposed at the aging temperature as oversized blanks and were subsequently machined down to the final specimen size to minimize the influence of surface oxide effects. Additional specimens were initially overaged (aging



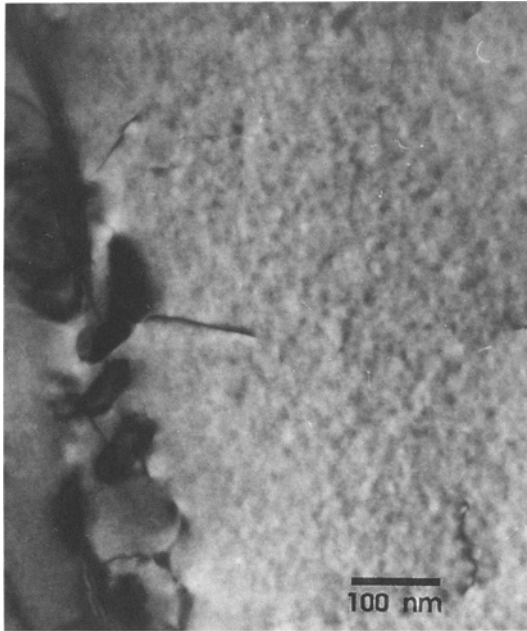
(a)



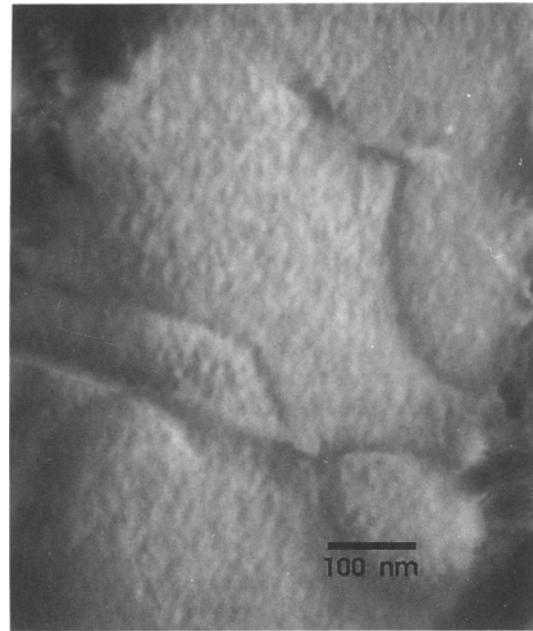
(b)

**Fig. 2** TEM micrographs of Ti-1100. (a) Unaged condition. (b) 6000 h aged condition (overaged)

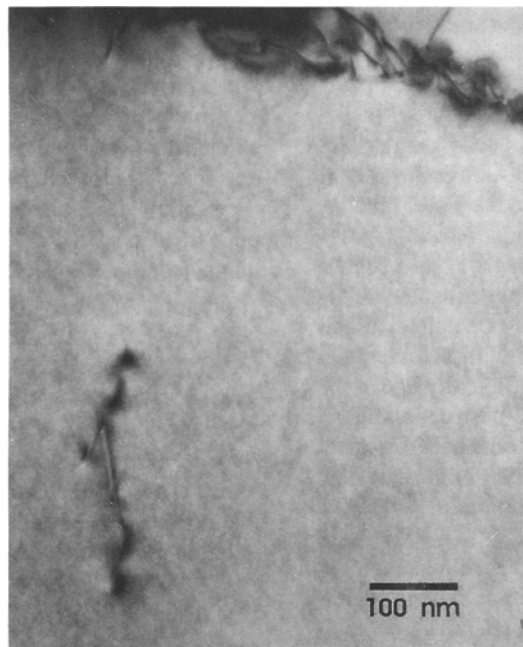
time >1000 h) and then were subjected to a PAHT of 4 h at 750 °C followed by fan cooling. Tensile specimens that are referred to as “overaged” were aged for 3000 h while compact tension specimens that are referred to as “overaged” were aged for 6000 h. The aging time of specimens that received the PAHT were initially aged for 1000 h. Previous studies indicate that these aging times (1000, 3000, and 6000 h) are identical in terms of hardness and mechanical properties (Ref 1, 6).



(a)



(b)



(c)

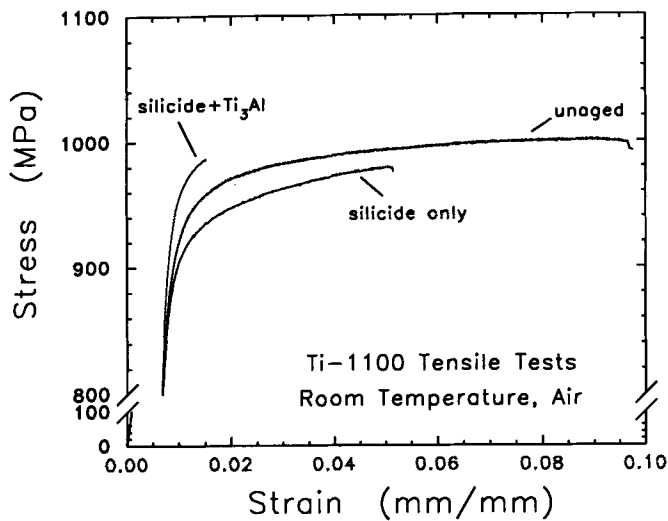
**Fig. 3** TEM micrographs of Ti-1100. (a) 1000 h aged at 593 °C with no additional heat treatment. (b) 1000 h aged at 593 °C plus 2 h at 700 °C with fan cooling. (c) 1000 h aged at 593 °C plus 2 h at 750 °C with fan cooling

### 3. Experimental Results and Discussion

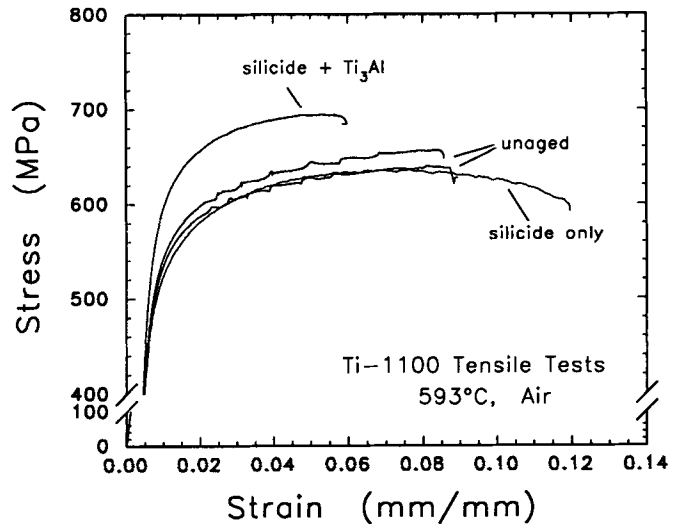
#### 3.1 Aging and Post-aging Heat Treatments

Transmission electron microscopy (TEM) micrographs of the unaged and overaged materials are shown in Fig. 2(a) and

(b). In the overaged material, silicides exist primarily along the alpha platelet boundaries, and the aluminide precipitates are seen throughout the matrix. A more thorough discussion of the size, distribution, and evolution of these precipitates is found elsewhere (Ref 1).



(a)



(b)

**Fig. 4** Stress-strain curves for Ti-1100. (a) For tests performed at room temperature for three aging conditions: unaged, overaged ( $\text{Ti}_3\text{Al}$  plus silicide), and PAHT (silicide-only). (b) For tests performed at 593 °C for three aging conditions: unaged (repeated), overaged ( $\text{Ti}_3\text{Al}$  plus silicide), and PAHT (silicide-only)

The purpose of the PAHT is to apply a time and temperature combination to the overaged material that results in the dissolution of the  $\text{Ti}_3\text{Al}$  precipitate while leaving other elements of the microstructure unchanged, especially the silicide precipitate. A feature of many ordered systems is the existence of a temperature called the critical ordering temperature above which entropy considerations lead to a destruction of long-range order in the system. Exposure of overaged Ti-1100 above the critical ordering temperature was used to eliminate the  $\text{Ti}_3\text{Al}$  precipitate while leaving the silicide precipitate unaffected. Figures 3(a) to (c) show bright-field TEM micrographs from material that was aged for 1000 h at 593 °C (i.e., overaged) and two different postaging heat treatments: 2 h at 700 °C, and 2 h at 750 °C. Comparison of these two PAHT cases with the overaged material indicates that only 750 °C was sufficiently above the critical ordering temperature to ensure the destruction of the  $\text{Ti}_3\text{Al}$  precipitate in a time of 2 h. An additional heat treatment at 800 °C (not shown) did not show any change in bright field appearance from the 750 °C case. Optical examination of the etched microstructure indicated that no change in the Widmanstätten microstructure occurred because of exposure at 750 °C. Although the  $\alpha$  and  $\alpha+\beta$  transus temperature was not determined for Ti-1100, in a study of IMI 829, which has a similar alloy chemistry, beta phase did not form again until an aging temperature of approximately 900 °C (Ref 9). The actual equilibrium critical ordering temperature may be much lower than 750 °C, perhaps as low as 650 °C (Ref 10). However, the value of 750 °C was chosen in the present study in the context of the relatively short 2h heat treatment time. Finally, a PAHT time of 4 h at 750 °C was selected for test specimens to further guarantee the destruction of ordered  $\text{Ti}_3\text{Al}$  precipitate. The strength of the PAHT concept lies in the fact that when comparing unaged, overaged, and PAHT materials, all other aspects of the alloy chemistry, silicide precipitation, microstructure, aging time and temperature, etc. remain constant while only the  $\text{Ti}_3\text{Al}$  precipitate is removed by the PAHT.

### 3.2 Tensile Test Results

Table 1 summarizes the results of the tensile tests discussed in this work. True uniform plastic elongation values are determined by the measurement of the reduced cross-sectional area of the specimen gage length away from the site of failure. These values are calculated as the logarithmic ratio of the original to the reduced cross-sectional area. Similarly, zero-gage length elongation values are derived from measurement of the percent reduction in area ( $RA\%$ ) at the plane of fracture and according to the formula  $RA\%/(100 - RA\%)$ .

Figures 4(a) and (b) show tensile stress-strain curves for tests run at room temperature and 593 °C for three conditions: unaged, overaged, and PAHT. At room temperature, the increase in the yield stress that occurs in the overaged material appears to be due entirely to the present of the  $\text{Ti}_3\text{Al}$  precipitate. Indeed, in the silicide-only material, a lowering of the yield stress is observed compared to the unaged case. This can be understood as a balance between increasing yield stress due to the silicide precipitate and decreasing yield stress when silicon is taken out of solution.

At high temperature, the same relative trends among the unaged, overaged, and silicide-only cases are observed; however, the reduction in yield stress of the silicide-only (PAHT) material compared to the unaged material is less pronounced than at room temperature, which may be due to an increase in silicide precipitate strengthening effect with temperature. The importance of the temperature dependence of the silicide precipitate strengthening effect may even be underestimated by the present results because the silicon solid solution strengthening effect is also known to increase with temperature (Ref 11). This latter effect should result in a trend where the difference in yield stress between unaged and silicide-only materials is greater at high temperature than at room temperature (contrary to the experimental trend).

Turning attention to the effect of precipitation on ductility, it is observed that at room temperature, both the silicide and the  $Ti_3Al$  precipitates are responsible for the decline in ductility seen in the overaged material. However, the  $Ti_3Al$  precipitate must be present to cause the most severe decline in ductility that provides for nearly brittle behavior at room temperature in the overaged alloy. It was suggested that a synergistic relationship may exist between the silicide and  $Ti_3Al$  in reducing the ductility of aged silicon-containing titanium alloys (Ref 4). In this mechanism, the presence of  $Ti_3Al$  causes the formation of heterogeneous, planar slip in the form of widely spaced intense slip bands. These slip bands encounter the larger silicide precipitates that fracture, having the effect of further concentrating slip. The consequence of this cooperative mechanism is highly concentrated slip and brittle macroscopic behavior. The current results do not contradict this mechanism; however, neither do

they provide supporting evidence. Clearly, however, if  $Ti_3Al$  formation can be suppressed in alloys that contain silicides, then the room temperature ductility will be improved.

At high temperature, the decline in ductility with aging is less severe than at room temperature. This is an important point because alloys that are subjected to aging during high-temperature service will presumably also encounter the highest stresses and strains at high temperature where the reduction in ductility with aging is rather modest. Nevertheless, the results from the silicide-only material are interesting because they show an increase in ductility compared to the unaged case. The cause of this increase in ductility may be due to a tendency for the silicide to foster a more homogeneous slip distribution and less planar slip character during high-temperature deformation by promoting cross-slip and dislocation loop formation around the silicide precipitate, although the present study does not provide direct evidence to support this mechanism (e.g., TEM analysis).

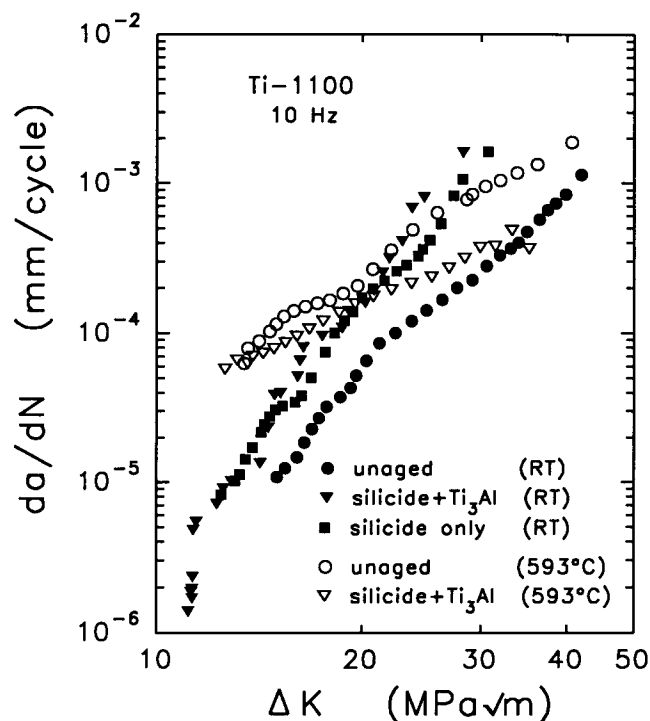


Fig. 5 Fatigue crack growth rate curves for Ti-1100 at room temperature and 593°C

### 3.3 Fatigue Crack Growth Results

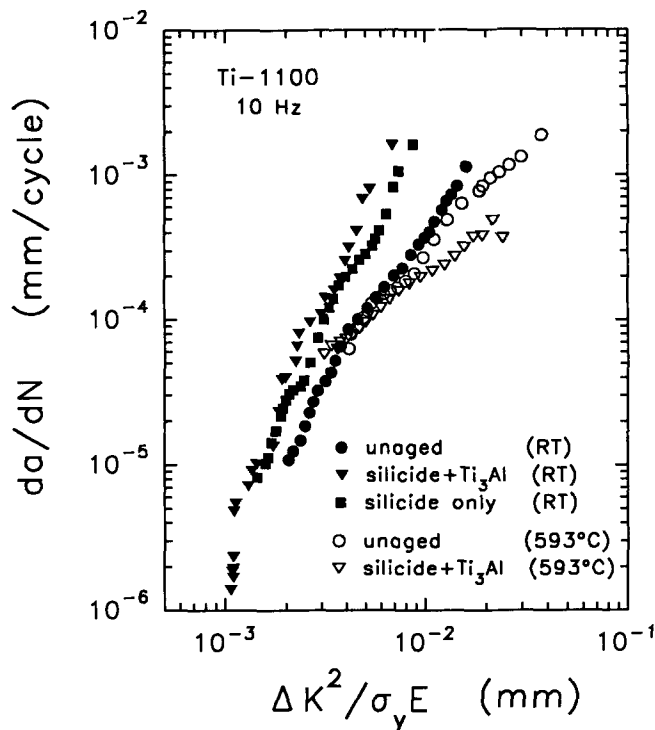
Fatigue crack growth test results are shown in Fig. 5 in the form of crack growth per loading cycle versus  $\Delta K$ . All tests were performed in air environment. A cyclic load frequency of 10 Hz was chosen so that the total test time was generally less than 8 h, thereby avoiding significant additional aging during the high temperature tests. At room temperature, the effect of aging is to increase the fatigue crack growth rate, especially at high  $\Delta K$ . To explore the separate roles of the silicide and  $Ti_3Al$  precipitates, a comparative test was performed using material that had undergone the PAHT to remove the  $Ti_3Al$  precipitate. This fatigue crack growth rate is identical with the result from the overaged material.

At high temperature, the overaged material shows a slight improvement in fatigue crack growth when compared to the unaged material, again especially at higher  $\Delta K$ . (These results were first introduced in Ref 6.) Figure 6 illustrates the consolidation of the fatigue crack growth curves that is possible when changes in mechanical properties are considered. Apparently, the yield strength and Young's modulus are important parameters in controlling fatigue crack growth rates in these materials because consolidation is good among the cases where monotonic ductility is similar. See Fig. 4(a) and (b). Ductility also appears to play a role in fatigue crack growth because the room temperature silicide-only case is not well consolidated and at

Table 1 Summary of tensile test data for Ti-1100

Test No.	Aging condition	Test temperature, °C	Strain rate, s <sup>-1</sup>	Young's modulus, GPa	0.002 Yield, MPa	n(a)	K(a), MPa	True uniform plastic strain(b)	Zero gage length elongation(c)
T1	Unaged	593	1e-4	86	500	0.103	900	0.0584	0.182
T4	Unaged	593	1e-4	89	505	0.110	940	0.0662	0.211
T3001	Overaged	593	1e-4	91	565	0.089	955	0.0477	0.135
T10	PAHT	593	1e-4	89	485	0.113	953	0.0911	0.235
T5	Unaged	RT	1e-4	120	915	0.060	1250	0.0546	0.175
T3002	Overaged	RT	1e-4	122	955	0.066 (d)	1323(d)	0.0018	0.048
T9	PAHT	RT	1e-4	120	895	0.0689	1264	0.0415	0.087

(a) Defined as  $\sigma = K\epsilon^n$  where  $\sigma$  and  $\epsilon$  are true stress and true strain. (b) From measurement of cross-sectional area away from the necked region. (c) Defined as  $RA/(1 - RA)$  where  $RA$  is percent reduction in area at the site of fracture. (d) Values must be treated with caution because plastic portion of curve is too short to allow accurate determination of a linear portion.



**Fig. 6** Same as Figure 5 but modified to account for differences in elastic modulus and yield strength with test temperature and aging condition

the same time displays a reduced monotonic ductility compared with the unaged material. In this respect, the room temperature overaged case represents an anomaly because its monotonic ductility is much less than the silicide-only case, and yet their fatigue crack growth rates are identical. A possible explanation for this comparative behavior may involve the destruction of the  $Ti_3Al$  precipitates within the crack tip plastic zone by repeated dislocation shearing during the cyclic fatigue process. A similar mechanism has been seen in other materials (Ref 12, 13). In this fashion, the microstructure within the crack tip plastic zone of the overaged material becomes essentially identical to the microstructure of the PAHT material, albeit by different mechanisms. This notion is also consistent with the results of fatigue crack growth at high temperature. If the shearing action of dislocations in the plastic zone acts to destroy the  $Ti_3Al$ , then the comparison of fatigue crack growth rates between the unaged and overaged materials should follow the trend set by the comparison of ductility between the unaged and PAHT conditions. The PAHT material is more ductile than the unaged material (Fig. 4b), and in fact the fatigue crack growth behavior of the overaged material is slightly improved over the unaged case. Due to the small difference in fatigue crack between the unaged and overaged cases and due to uncertainty regarding the reformation characteristics of the  $Ti_3Al$  precipitate, a fatigue growth rate test at 593 °C was not performed using PAHT material.

As in the results of the tensile ductility, the effect of aging on fatigue crack growth is more pronounced at room temperature

than at high temperature. Therefore, the practical importance of aging effects in the use of Ti-1100 and similar alloys can be considered slight if affected parts are subjected to significant monotonic or cyclic stresses only at high temperatures. This leads to the suggestion that structural component life may benefit from preheating before being subjected to significant stresses. Although aging causes a reduced performance at room temperature in terms of both monotonic ductility and fatigue crack growth, the underlying reason behind the degradation in performance is different for the two cases.  $Ti_3Al$  is largely responsible during monotonic deformation. The silicide is largely responsible during cyclic (fatigue) deformation. Thus it seems that suppressing the formation of both precipitates would be desirable regarding both room temperature ductility and room temperature fatigue crack growth. On the other hand, at high temperature, the existence of the silicide enhances both ductility and resistance to fatigue crack growth, while suppression of  $Ti_3Al$  formation is apparently desirable with respect to monotonic ductility (and probably to fatigue crack growth as well). However, any discussion of the merits or drawbacks of these precipitates must include their effects on creep behavior, an arena where titanium alloys are typically at a disadvantage compared to other high temperature alloys, such as nickel-based superalloys. It was suggested that short-range order in Ti-Al alloys does enhance creep properties (Ref 14). On the other hand, silicide formation was deleterious to creep behavior (Ref 15). Work is currently in progress to utilize the PAHT concept to explore the effects of these precipitates on creep behavior.

#### 4. Conclusion

Aging in silicon-bearing near-alpha titanium alloys results in the formation of two types of precipitate:  $Ti_3Al$  and the silicide. A PAHT was used to remove the  $Ti_3Al$  precipitate in overaged material. A comparison of mechanical behavior among unaged, overaged, and a PAHT alloy permits a greater understanding of the separate roles of the two types of precipitate in affecting tensile and fatigue crack growth behavior at room temperature and 593 °C.

- The increase of the monotonic yield stress that accompanies aging of Ti-1100 at both room temperature and 593 °C is caused by the formation of  $Ti_3Al$ . When only the silicide is present, a decrease in yield stress compared to unaged material occurs. In addition, the effect of the silicide precipitate is temperature dependent, with greater silicide strengthening (i.e., less weakening effect) occurring at 593 °C than at room temperature.
- The decrease in room temperature monotonic tensile ductility that occurs with aging in Ti-1100 is caused by both  $Ti_3Al$  and the silicide precipitate. However, when only the silicide is present, the decline in room temperature ductility is much less severe than when both precipitates are present. A decrease in ductility also occurs at high temperature, but the magnitude of the decline in ductility is much less than that occurring at room temperature. Furthermore, the decline in ductility at high temperature is due only to  $Ti_3Al$

because when only the silicide is present, the high temperature ductility is increased compared to the unaged case.

- Aging causes an increased fatigue crack growth rate compared to the unaged case for 10 Hz tests run in air and at room temperature. The precipitate responsible for this increased fatigue crack growth rate appears to be the silicide, with the aluminide playing little if any role. In contrast, at high temperature, aging produces a slight decrease in fatigue crack growth rate when compared to the unaged material. For both test temperatures, the aging effect becomes more pronounced at higher  $\Delta K$ , and the trends of aging effect in fatigue crack growth appear to follow the trends of monotonic ductility in the material in which only the silicide is present.
- The deleterious effects of aging are less severe during deformation at high temperature than at room temperature. This suggests that the service life of aged components may benefit from a period of preheating before being subjected to significant stresses in service. This conclusion should be applicable to near-alpha titanium alloys as a general class.

### Acknowledgments

This work was supported by TIMET Corporation, Henderson, Nevada. The authors thank Professor R. Brown of Chemical Engineering Department, University of Rhode Island, and Dr. E. Andrieu and Ms. Regine Moline of Ecole des Mines de Paris, France, for their useful discussions and assistance in completion of TEM work in this study. The encouragement and assistance of Dr. P. Bania of TIMET Corporation are also greatly appreciated.

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