Reproducibility of Small-Strain Creep Tests of Ti 6242Si

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Titanium 6242Si was creep tested at 510 °C and 241 MPa to plastic strains of about 0.002. These conditions are common in commercial specifications for acceptance of this alloy. Nineteen specimens were extracted from four identically thermally and mechanically processed coupons from the same ingot and creep tested. The results indicate relatively poor reproducibility despite essentially identical sample preparation and specimen testing procedures. The causes of the scatter are unclear. The average creep strain of the 19 specimens after 35 h was 0.067%, and the standard deviation was 0.0367%. Significant scatter is also evident among the average properties of the four coupons. The results suggest that impurity levels and processing procedures may require adjustments to ensure relatively low expected or "average" creep strain levels (half the allowable) in some common acceptance specifications to ensure a high probability of acceptance.

Keywords
creep tests, titanium alloys

1. Introduction

EARLIER work (Ref 1) established that variation in the volume fraction of primary alpha phase and Ni composition could significantly affect the small-strain creep properties of titanium alloy Ti 6242Si with nominal concentrations of Al, Sn, Zr, Mo, and Si. An equation was formulated to predict the creep strain in Ti 6242Si after 35 h at 241 MPa and 510 °C as a function of volume fraction primary alpha, weight fraction Ni, and weight fraction Fe (which also has been known to degrade the creep properties of 6242Si. (See Ref 2-4.):

$$\varepsilon_{p35h} = -1.44 \times 10^{-4} + 2.40 \times 10^{-2} W_{Ni} + 8.50 \times 10^{-3} W_{Fe} - 5.56 \times 10^{-4} V_{\alpha} + 2.61 \times 10^{-3} V_{\alpha}^2 \quad \text{(Eq 1)}$$

where W_{Fe} and W_{Ni} are Fe and Ni concentrations in weight percent, respectively (e.g., insert 0.005 for W_{Fe} if weight percent iron is 0.005%), and V_{α} is the volume fraction of primary α (e.g., insert 0.12 for V_{α} if the volume fraction of primary alpha is 12%).

This stress, temperature, and duration (510 °C, 241 MPa over 35 h) is relevant to common acceptance specifications for this alloy (e.g., Ref 5), which often limits plasticity to 0.001. This equation is valuable in predicting creep strain as a function of V_{α}, W_{Fe}, and W_{Ni}. However, statistical considerations are not incorporated. For example, the equation might predict that a specimen of a specific W_{Ni}, W_{Fe}, and V_{α} will deform to 0.0009 after 35 h. However, the probability of passing is not available. A high level of reproducibility suggests that a higher fraction of specimens would "pass" a 0.001 strain specification while poorer reproducibility predicts a lower fraction passing. The data of our earlier study suggest that significant scatter (or a significant lack of reproducibility) was observed despite very careful specimen preparation and testing procedures. That is, the lack of reproducibility may be due to variations within the alloy rather than the testing procedure.

The purpose of this study then, was to determine the reproducibility of the creep strain after 35 h in a significant number of specimens from an identical 6242 ingot (in this case, the same as utilized in the previous study, Ref 1) with identical thermal and mechanical processing procedures. A statistical analysis was performed. This would allow a calculation of the probability that the creep strain in any given specimen will be less than 0.001 based on predictions of the average or "expected" strain (less than 0.001) from Eq 1.

2. Experimental Procedure

The testing procedures and materials were identical to those of Ref 1. Briefly, the samples were forged from a production billet of Oregon Metallurgical Corporation (OREMET) Ti-6242Si (heat number T 90847), which had a beta transus, T_{β}, of 1004 °C (determined by differential thermal analysis). The composition is listed in Table 1.

The billet was processed by forging a vacuum arc-melted 914-mm-diam ingot to a 152-mm-diam billet using a two-stage process. The initial reduction was in the beta temperature range between 1065 and 1149 °C (T_{β} + 61 °C and + 145 °C) where the ingot was forged to a 254-mm-diam octagon. The final forging at T_{β}–36 °C (36 °C below the beta transus) reduced the diameter to 152 mm. The forged ingot was then cooled to ambient

Table 1 Composition of primary alpha test ingot for alloying and impurity elements

Element	Composition, wt%
Al	6.000
Sn	2.020
Zr	3.980
Mo	2.000
Si	0.080
N	0.011
Cr	0.008
V	0.010
Ni	0.005
Fe	0.088
O	0.119

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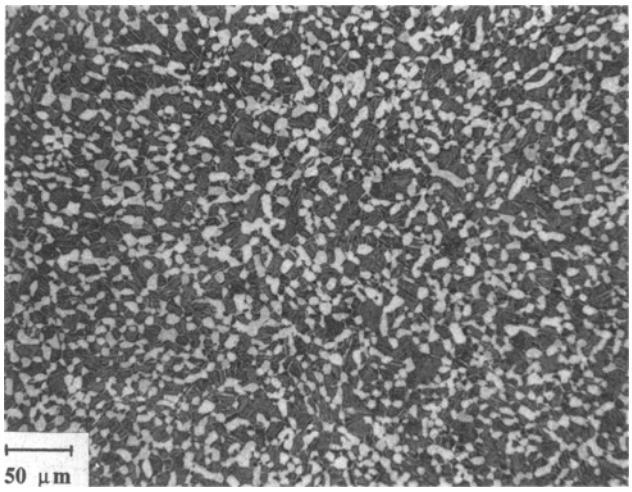

Fig. 1 Optical micrograph of Ti 6242Si microstructure that is representative of the specimens of this study (solution anneal T_β–15 °C). Equiaxed (light) phase is primary alpha. The dark phase is transformed beta phase.

temperature, which resulted in a microstructure of principally elongated primary alpha due to relatively slow cooling because of the large diameter of the ingot. Sample coupons were then extracted from the billet and forged. The samples had initial dimensions of 64 × 64 × 95 mm. These coupons were heated for 2 h at T_β–36 °C ± 14 °C before forging. Hammer forging of the samples was performed at OREMET at a strain rate of between 63/s and 252/s. Six forging passes were required to reduce the sample coupon to the desired cross-section dimensions of 41 × 16 mm. The resulting true and engineering strains were 5.1 to 1.8, respectively, for the coupon forging sequence. The samples were air cooled after forging and solution annealed at T_β–14 °C for 1 h and then air cooled. This resulted in lenticular alpha for the transformed beta phase rather than elongated primary alpha due to the faster air cooling rate of the smaller samples. The samples were then aged at 643 °C for 8 h and air cooled to ambient temperature as is consistent with standard practices for this alloy (Ref 5). Tensile samples with a 25.4-mm-gage length and a 6.35-mm-diam were extracted from the coupons. The axes of the tensile samples were parallel to the long axis of the forgings. Four coupons were forged from which a total of 19 specimens were extracted. An optical micrograph of the resulting microstructure is shown in Fig. 1.

The creep strain was measured using a single Applied Test Systems* model 4112 extensometer configured for 6.35-mm-diam samples with 25.4-mm-gage lengths and a Measuretron model L1-12 linear variable capacitance transducer. The extensometer was determined to have an error of less than 50 microstrain over a 72-h period. The creep tests were performed on an Arcweld** (SATEC) model UC creep testing machine. Because of the very low plastic strains measured in this investigation (<0.002) and the relatively high elastic strain on loading (~0.0035), less than very well aligned testing systems can have bending moments that can cause a significant stress gradient. Stress gradients can cause anomalously high or low creep rates, as measured by a single extensometer. Four universal joints were present in the load train to help mitigate bending stresses.

The standard recommended practices for determining the magnitude of bending were utilized prior to all creep tests (Ref 6, 7). It was determined that Ti 6242Si did not creep measurably at ambient temperature at 241 MPa (the standard applied creep stress). This allowed strain gages to be mounted on all creep test samples for ambient temperature alignment at 241 MPa. Four strain gages were mounted at 90 degree intervals around the sample; then the bending in the sample was measured for various specimen orientations within the threaded universal grips. A configuration that minimized the bending at ambient temperature was identified. Bending values were calculated according to the formula:

$$\text{pct bending} = (\text{average elastic strain}) - (\text{maximum or minimum elastic strain}) / (\text{average elastic strain}) \times 100 \quad \text{(Eq 2)}$$

Final bending at ambient temperature was always less than 3.5% (and as low as 0.7%). The strain gages were then removed while the sample was still loaded to prevent misalignment during stripping. The adhesive was removed; then the sample was given a final cleaning with methanol. Three thermocouples were attached at the top, middle, and bottom sections of the specimen gage length. The extensometer was attached to the sample, which was then unloaded to 25 MPa (a stress at which no measurable creep occurs at 510 °C) and heated in air to 510 °C. The creep tests were commenced once the temperature stabilized at 510 °C ± 2 °C for 3.5 to 4.5 h. A three-zone furnace with Eurotherm* power supplies and controllers was utilized. The samples were tested for 72 h or longer, and a mechanical steady-state was approached. It was found that a "true" steady state may not be reached until after 150 h, with steady state creep rates being a factor of approximately 2 lower than those measured at 72 h. The "steady state" values reported in this study were calculated after only about 72 h. The temperature typically did not vary by more than 2 °C from 510 °C at any point along the specimen throughout the test.

Each sample was sectioned, and the microstructure was examined by optical metallography after creep testing. Metallographic sections were taken parallel and perpendicular to the tensile axis. Two parallel sections that were perpendicular to each other were extracted. Area fractions of primary α (alpha) were determined using computerized image analysis and were verified by manual point counting. Chemical analysis was performed by OREMET using induction coupled plasma chemical spectrographic analysis.

3. Results and Discussion

The results of the reproducibility tests are illustrated in Fig. 2. Substantial scatter is evident. The primary alpha composi-

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*Eurotherm is a trademark of Eurotherm Corporation, Reston, VA.

Table 2 Creep testing results of Ti 6242Si at 241 MPa and 510 °C

Coupon No.	Specimen No.	Ni composition, wt%	Fe composition, wt%	V_α, %	ε_p(35 h), %	$\dot{\varepsilon}_{ss} \times 10^{-9}$/s
1	1	0.006	0.083	14.8	0.086	1.39
	2	0.006	0.083	15.2	0.068	0.96
	3	0.006	0.083	17.7	0.047	0.61
	4	0.006	0.083	13.9	0.086	1.55
					$SD_{coupon1} = 0.019$	
2	1	0.005	0.083	10.3	0.049	0.79
	2	0.005	0.083	8.9	0.085	1.73
	3	0.005	0.083	16.6	0.155	2.52
	4	0.005	0.083	24.1	0.097	1.71
					$SD_{coupon2} = 0.044$	
3	1	0.006	0.088	8.8	0.011	0.26
	2	0.006	0.088	13.0	0.022	0.28
	3	0.006	0.088	10.2	0.052	1.26
	4	0.006	0.088	9.3	0.026	0.27
	5	0.006	0.088	7.0	0.027	0.36
	6	0.006	0.088	11.3	0.038	0.83
					$SD_{coupon3} = 0.014$	
4	1	0.006	0.088	12.0	0.111	1.62
	2	0.006	0.088	14.5	0.080	1.15
	4	0.006	0.088	9.2	0.039	0.65
	5	0.006	0.088	15.0	0.092	1.21
	6	0.006	0.080	16.8	0.104	1.32
					$SD_{coupon4} = 0.025$	
Mean	...	...	...	13.1	0.067	1.08
	...	...	...	$SD_{19} = 4.1$	$SD_{19} = 0.0371$	0.61
Adjusted mean $V_\alpha = 30\%$	...	...	...	30	0.076 (adjusted)	...
	...	...	...	...	$SD_{19} = 0.0367$ (adjusted)	...

Note: SD is standard deviation.

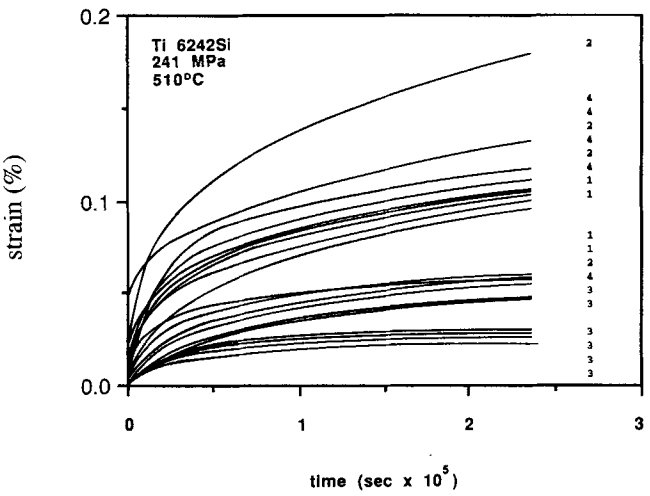

Fig. 2 Creep strain versus time for 19 Ti 6242Si specimens. Data are grouped according to the coupon from which the specimens were extracted.

tion was determined on each specimen to assess reproducibility more accurately. Additionally, the Fe and Ni composition was determined of each coupon that was individually thermally and mechanically processed (four to six specimens were extracted from each coupon). The data from the 19 creep tests to 35 h are reported in Table 2.

The Fe and Ni compositions did not vary, within error, among four coupons. There was, however, some variation in primary alpha phase among the 19 specimens. The range of primary alpha varied from 7 to 24%, with an average of about 13.1% and a standard deviation of 4.1%. This average was lower than the expected value of 30% (Ref 1) based on the solution treatment of T_β–14 °C. The average strain value after 35 h at 510 °C and 241 MPa was 0.067% for $\bar{V}_\alpha$ = 13.1%. The standard deviation (based on a normal distribution) was 0.0371%. Equation 1 was used to normalize the data to the expected strain at 35 h assuming 30% V_α in each test specimen. This generally resulted in a minor correction. Once corrected to 30% V_α, the expected average strain value increased to 0.076%. A very similar value (0.080%) was obtained in an earlier study (Ref 1). The 30% V_α standard deviation was 0.0367%. This is essentially identical, though slightly better than the "uncorrected" value. Figure 2 shows that the data are grouped with the average strain from different coupons varying. It is apparent from Table 2 that the standard deviation (ε_{p35}) within each coupon is generally better than the standard deviation for the entire ingot. The average standard deviation for specimens of a given coupon is 0.026%, less than that for all 19 specimens.

The explanation for the scatter is unclear because significant variations in the average V_α, Ni, and Fe concentrations among coupons were not detected. Also, testing variations will not explain the variations in creep strain, particularly as different coupons appear to have different behavior. As Fe and Ni

Table 3 Probability of an accumulated plastic strain of <0.1% after 35 h at 510 °C and 241 MPa in Ti 6242Si for various mean strain values

Predicted strain (from Eq 1)	Predicted passing, %	Ni = 0 composition Fe, wt%	Fe = 0 composition Ni, wt%
0.1	50	0.151	0.054
0.09	61	0.140	0.049
0.085	66.5	0.134	0.047
0.08	71	0.128	0.045
0.075	76	0.122	0.043
0.07	80	0.116	0.041
0.065	83.5	0.110	0.039
0.06	87	0.104	0.037
0.05	92	0.093	0.033
0.04	95.5	0.081	0.029
0.03	97.5	0.069	0.024
0.02	98.5	0.057	0.020
0.01	99.5	0.046	0.016

Note: Maximum Ni and Fe concentrations are listed for each strain.

may segregate into the minority, or β phase, this phase may strongly influence the low creep strain behavior (Ref 1, 8, 9). Chemical or morphological changes in this phase, which cannot be assessed by optical microscopy, may be an explanation.

The observed standard deviation for all 19 specimens is significant. For example, an expected (average) creep strain (of a given ingot chemistry followed by a specified thermal and mechanical processing series) of 0.05% (half the maximum allowable for many specifications) is required to ensure at least a 92% pass rate. Maximum allowable Fe and Ni concentrations for various expected creep strains and the associated probabilities of accumulated strains less than 0.1% are listed in Table 3. For example, Table 3 suggests a target 35 h creep strain of 0.05% to ensure a 92% pass rate. If the weight percent of Fe is zero, then maximum permissible Ni is 0.033 wt% for this maximum strain for this pass rate. Conversely (for 92% pass rate), if the weight percent of Ni is zero, the maximum Fe is 0.093 wt%.

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