Compressive creep behavior of Ti-6Al-4V-xSi-ySc alloys under high-pressure at room temperature

Tongsheng Deng1,2,3,∗, Wei Cao3, Kemai Yang3 and Jianxin Shen1

1 College of Electrical Engineering, Zhejiang University, Hangzhou 310027, People’s Republic of China
2 Faculty of Materials Metallurgy and Chemistry, Jiangxi University of Science and Technology, Ganzhou 341000, People’s Republic of China
3 Department of Technology, Dongliang Aluminium Industry Co., Ltd, Huzhou 313000, People’s Republic of China
∗ Author to whom any correspondence should be addressed.
E-mail: dts115@jxust.edu.cn

Keywords: compressive creep, room temperature, scandium, silicon, titanium alloy

Abstract
One of the characteristics of Titanium alloys is that they are prone to creep due to the service conditions under high-pressure, such as deep-sea equipment. The compressive properties and deformation mechanism of Ti-6Al-4V-xSi-ySc alloys at room temperature are investigated in this study. The alloy samples are subjected to uniaxial compressive and creep tests. The obtained compression creep properties at room temperature follow the power-law function, and creep is mainly found in the first and second stage. Si and Sc are observed to improve the creep properties where the latter shows significant effect. This can be attributed to the strengthening of the solid solution of Si and Sc. Sc and Si strengthen the α and β phases respectively. The stability of these phases contributes to increasing the creep resistance of Titanium alloys. The creep resistance mechanisms of the alloys involve dislocation movement and twins.

1. Introduction
The good resistance of titanium (Ti) alloys to corrosion and their high strength to weight ratio [1, 2] mean that they are suitable for use in pressure-resistant structures in the deep-sea. However, one of the challenges of alloys used in such service conditions is that they are prone to creep at room temperature and under high stress (often 0.8 ∼ 0.9σ0.2), and most of the deformation occurs in the preliminary stage [3]. Moreover, service situations with similar concerns include those of pressure vessels, weight-bearing biomedical implants (e.g. hip joints), and aircraft components. Creep at room temperature may lead to component damage due to dimensional changes that exceed the allowable tolerance when accurate part tolerance is required. Therefore, in order to predict the properties of current alloys or design new alloys that exhibit resistance to creep at room temperature, it is important to have a good understanding of the creep deformation mechanisms of Ti alloys at room temperature and under high-pressure compression.

In the past few decades, studies have reported that Ti alloys creep at room temperature. Previous studies focused on tensile creep behavior of commercial titanium alloy at room temperature. The stability of the β phase increases with the addition of β-stabilizers to the alloys [4]. In two-phase α+β Ti alloys, many factors can potentially lead to the deformation during low-temperature creep [5]; including the grain size [6], β-phase stability [7], elastic interaction stresses [8], and crystallographic phase orientation relationships. Aiyangar et al [9] found that dislocation slip and twinning are the main mechanisms of creep deformation in alpha Ti-1.6V alloy. Oberson and Ankem [3] examined the creep of alpha-Ti alloy at room temperature by using scanning and transmission electron microscopies. They found that the main mechanism of deformation is dislocation slip and twinning. Twins were predominantly found in the larger-grain alloys but absent in those with a smaller grain size.
Moreover, the relative stability of the \( \beta \) phase of Ti alloys may lead to significant changes in the deformation mechanism at room temperatures \[10\]. Jaworski and Ankem \[7\] compared the deformation behavior of Ti-6.0Mn versus Ti-8.1V alloys and found that the creep strain in the latter is higher, which is attributed to the formation of stress-induced martensite in the \( \beta \) phase and more twins in the \( \alpha \) phase. The formation of stress-induced martensite is related to the reduced stability of the \( \beta \) phase. Therefore, the high stability of the \( \beta \) phase in Ti-6.0Mn prevents the formation of stress-induced martensite, and twinning is the main deformation mechanism. The findings above show that the creep behavior of alloys is not only affected by the phase composition but also the microstructure. More recently, the compressive creep mechanisms at room temperature are found to be dislocation slip and twinning. Chen et al \[11\] performed compression creep experiments on Ti-6Al-3Nb-2Zr-1Mo and Ti-6Al-4V alloys under high-pressure at room temperature and found that an increase in applied stress significantly increases both the creep rate and amount of creep deformation. The creep deformation is mainly caused by the dislocation slip in the \( \alpha \) phase.

Nevertheless, there are few studies on the effects of different alloying elements on the compression creep properties of Ti alloys at room temperature, especially the effects of silicon (Si) and scandium (Sc) \[12\]. Therefore, we study in-depth the creep behavior of Ti-6Al-4V-xSi-ySc alloys under a pressure of 0.9\( \sigma _{0.2} \) through long-time compression tests at room temperature. The creep mechanism of the Ti alloy samples is then discussed. The effects of Si and Sc on the amount of compression creep at room temperature are subsequently elaborated.

### 2. Materials and experiments

Ti-6Al-4V-xSi-ySc (\( x = 0.25 \) and \( y = 0.3 \) wt\%) alloys were prepared from pure Ti, aluminum (Al), Al-55 vanadium (V), Al-12Si and pure Sc by vacuum arc remelting (three times). The ingots were heated to 1050 °C for 2h, forged into 100 × 100 mm square rods, and air-cooled to room temperature. Then, they were kept at 980 °C for 2h, forged into 50 × 50 mm square rods, and air-cooled to room temperature. Finally, after being heated at 950, for 1h, they were rolled into 18 mm diameter rods. Their chemical composition was determined by using the inductively coupled plasma (ICP) method and the results are listed in table 1. The oxygen content of the four alloys is 0.083 wt\%, 0.098 wt\%, 0.073 wt\% and 0.092 wt\% respectively. The oxygen content of the alloys is controlled at a low level, and there is little difference in the four alloys.

The rods were subjected to heat treatment, which included solution treatment at 950 °C for 0.5 h, quenched in air, subsequently aged at 510 °C for 5 h and quenched in air again.

First, the compression creep load was determined through compression tests that were carried out on a YAW4306 servo-hydraulic press to obtain the yield strength of the alloy samples in accordance with Chinese standard GB/T 7314–2017 Metallic materials–compression testing at room temperature. Cylindrical samples with a diameter of 6 mm and length of 10 mm were prepared from the aged rod. The compression strain rate was \( 8.3 \times 10^{-3} \text{mm s}^{-1} \). The compression tests were performed twice for all of the alloy samples to ensure the repeatability of the results.

Creep tests were carried out on a GWT2015 creep testing machine with an applied stress of 0.9\( \sigma _{0.2} \) at room temperature (25 °C) for 100 h, as shown in figure 1. Since the testing machine is a tensile creep testing machine, a special device is made to apply compression load onto the samples. The tests were performed three times for all of the samples to ensure the repeatability of the results. The compression creep displacement of the samples was recorded by using an extensometer. The samples are 6 mm in diameter and 10 mm in gauge length.

The metallographic microstructures of the alloy samples were analyzed to determine their morphology during heat treatment and creep deformation. The metallographic samples were ground, polished, and etched in a solution of 1%HF + 3%HNO\textsubscript{3} + 96%H\textsubscript{2}O. The metallographic microstructures were then examined by using a ZEISS Axioskop optical microscope. In addition, the details of the microstructures were analyzed by using a Tecnai G2 F20 transmission electron microscope (TEM) formerly produced by FEI. The samples were mechanically polished until they were sheets with a thickness of 100 um. Disks with a diameter of 3 mm were

| Sample | Nominal composition | Nominal composition | Amount of element |
|--------|---------------------|---------------------|------------------|
| 1      | Ti-6Al-4V           | 5.90                | 4.26             | —     | —     |
| 2      | Ti-6Al-4V-0.25Si    | 6.27                | 4.22             | 0.26  | —     |
| 3      | Ti-6Al-4V-0.3Sc     | 5.75                | 4.22             | —     | 0.32  |
| 4      | Ti-6Al-4V-0.25Si-0.3Sc | 5.90            | 4.30             | 0.23  | 0.34  |

Table 1. Chemical composition of alloys (wt%).
polished on both sides by using a twin-jet electropolisher with a solution of 60%CH₃OH-34%CH₃(CH₂)₃OH-6%HClO₄ at −30°C and 20 V.

3. Results

3.1. Microstructure after heat treatment
The microstructure of the Ti-6Al-4V-xSi-ySc alloy samples after heat treatment is shown in figure 2. The alloy samples show one of two different microstructures after being subjected to the same heat treatment. The No.1 alloy forms a typical bimodal microstructure, which consists of equiaxed primary $\alpha$ phase dispersed in the transformed $\beta$ matrix. The average size of the primary $\alpha$ phase is determined to be 3.89 $\mu$m by using the linear intercept method with Image-Pro software. Moreover, the volume fraction of the primary $\alpha$ phase is 34.75%. The microstructure of the other three alloys is a basketweave structure with a transformed $\beta$ structure. No.2 alloy has a large number of colonies. Overall, the structure of alloy No.2, 3 and 4 becomes increasingly more fine-grained, which shows that the addition of Si or Sc greatly refines the prior $\beta$ grains in the alloy. The grain refinement can be attributed to the increased constitutional supercooling during $\beta$ solidification and the formation of high melting point dispersoids reduces the growth of the grains at high temperatures.

3.2. Compression creep behavior
The compressive yield strength of the four samples is shown in figure 3. There is an increase of 13.22% in the compression yield strength of No.2 compared to No. 1, which is a basic alloy sample. The main reason is that Si is solid-dissolved in the Ti alloy and strengthens the solid-solution. There is an increase of 5.88% in the compression yield strength of No. 3 compared to No. 1. Sc is a rare earth element and some of the Sc reacted with oxygen in the matrix to form a rare earth oxide called scandium oxide ($\text{Sc}_2\text{O}_3$). The formation of $\text{Sc}_2\text{O}_3$ reduces the oxygen content in the matrix and purifies the alloy. No. 2 and 4 sample have almost the same yield strength. Si is mainly dissolved in the $\beta$ phase and also has a strengthening effect on the $\beta$ phase. In Ti alloys, the $\alpha$ phase has a hexagonal close-packed (HCP) structure, and the $\beta$ phase has a body-centered cubic (BCC) structure. Therefore, the $\beta$ phase is preferentially deformed in the compression test.

There is large difference in the compressive yield strength of the four samples. The compressive creep stress is set to $0.9\sigma_{0.2}$. The creep test conditions are listed in table 2.

The plotted creep curves without considering the elastic stage are shown in figure 4. There are Stage I (Primary creep) and Stage II (Steady-state creep), without Stage III (Accelerated creep). The creep rate refers to how fast the creep strain changes with time, that’s the slope of the curves. It can be seen that the creep rate decreases gradually in the first stage. In the second stage it remains stable. The four alloys enter the second stage at different times. The creep performance is evaluated by the steady creep rate of the second stage. The lower the
creep rate is, the better the creep resistance of the material is. The creep variables are listed in Table 3, which include the initial, total and creep strains and steady-state creep rate. The total strain of Nos. 1, 2, 3 and 4 is 0.0975%, 0.0750%, 0.0455% and 0.0520% after creep testing, respectively. The creep strain of Nos. 2, 3 and 4 is reduced an additional 23.1%, 53.1% and 46.7% in comparison to that of No. 1. In addition, the steady-state creep rate of Nos. 1, 2, 3 and 4 is 1.56E-4, 1.23E-4, 8.13E-5 and 7.29E-5 respectively. The steady-state creep rate of Nos. 2, 3 and 4 is an additional 21.2%, 47.9% and 53.3% lower than that of No. 1.

Figure 2. Optical micrographs of alloys after heat treatment: (a) No. 1, (b) No. 2, (c) No. 3, and (d) No. 4.

Figure 3. Compressive yield strength at room temperature.

The deformation can be described by using the power-law function of creep strain with time: \( \varepsilon = at^b \) [13], where \( a \) and \( b \) are constants. The time exponent \( b \) indicates the exhaustion rate during creep. A smaller \( b \) value indicates more rapid exhaustion during creep. The parameters in the power law formula is determined based on the experimental data. The values of \( a \) and \( b \) of the different samples are shown in table 4. The fitted curve in Figure 4 shows the compression creep curve at room temperature.
Figure 5 shows a good agreement with the test results. During the creep of the Ti-6Al-4V-xSi-ySc alloy samples at room temperature, the primary creep is the dominant deformation mode in which there is a larger accumulation of primary creep strain.

4. Discussion

4.1. Creep properties

After the creep tests, the volume fraction of the equiaxed α phase of No. 1 is reduced to 13.4%. This shows that No. 1 has a stress-induced α-β phase transition. For Nos. 2, 3 and 4, the secondary α phase is coarsened to a certain extent. The overall microstructure retains the characteristics of a basketweave. When examined microscopically, the morphology does not show any significant changes; see figure 6.

Many dislocations are found in the microstructure of the samples, as shown in figure 7, thus indicating that dislocation movement is the main deformation mechanism. It is believed that creep deformation at room temperature is mainly controlled by the slip of the dislocations [14]. There are usually two types of dislocations, namely movable and fixed dislocations. Increased external stress on a metal material produces movable dislocations. When the material is subjected to a constant stress, the movable dislocation enables the atoms in the metal material to slide due to the external force. Macroscopically, the material creeps at room temperature. Phase boundaries, precipitates, or other obstacles during the sliding process inhibit movable dislocations. When there are many dislocations, the result is the formation of an area with densely packed dislocations.

In the compression creep experiment done at room temperature, a large number of movable dislocations are activated when the samples are subjected to a high pressure for a long period of time. The dislocations are entangled and kinked during the movement, thus forming a zone with many dislocations. At the same time, a large number of dislocations were found which formed dislocation walls at the phase boundaries of Nos. 1, 3 and 4, as shown in figure 8. The formation of dislocation walls prevents the movement of dislocations. The formation of the dislocation walls might be related to the some slip of of the β-phase during the compressive creep test. Si is a β-stabilizer, which can strengthen the alloy. The α phase of the Ti alloy has an HCP structure, and the β phase has a BCC structure. Therefore, the latter is preferentially deformed during the creep test.
According to the first-principles calculations, Sc has a good effect on Si. Sc is a stable element that is mainly distributed in the $\alpha$ phase. Therefore, the solid solubility of Si in the $\alpha$ phase can be increased. When the same amount of Si is added to the samples, there is less Si content of the $\beta$ phase in No. 4 as opposed to No. 2. Therefore, the strengthening effect of the $\beta$-phase in No. 4 is lower than that in No. 2. As a result, no dislocation walls can be found in No. 2.

A small amount of twins in Nos. 1 and 3 were found in figure 9, which indicates that twins are also a mechanism of the compression creep deformation of Ti alloys. Twins easily occur in coarse grains. When there is dislocation jamming, a larger grain size results in longer slip distance of the dislocations, and more jamming of the dislocations near the grain boundaries. This causes stress concentration at the grain boundaries, thereby inducing twins. Twins can enhance the creep properties of alloys and increase the strength of Ti alloys, which can also explain why the creep strain of No. 3 is smaller than that of No 4.

Sc$_2$O$_3$ is found in both Nos. 3 and 4, as shown in figure 10, which are mainly distributed at the grain boundaries, with irregular shapes that range from 1–2 um in size. There is an area with many dislocations around the Sc$_2$O$_3$, thus indicating that Sc$_2$O$_3$ has an obstructive effect on the movement of the dislocations.

4.2. Creep mechanism

The presence of an equiaxial $\alpha$-phase in the bimodal structure reduces the size of the phase interface, and the movement of the dislocations is less inhibited [15]. Therefore, the steady-state creep rate and creep strain values of No. 1 are both higher. The microstructure of No. 2 has a $\beta$ transformed structure, and the phase boundaries are obviously increased, which can well inhibit the movement of the dislocations. At the same time, Si is mainly found in the $\beta$ phase in a solid solution state, which strengthens the $\beta$ phase [16, 17]. Therefore, No. 2 has a lower steady-state creep rate and creep strain than No. 1. Sc is $\alpha$ stabilizing element, mainly distributed in the $\alpha$ phase, and has a solid solution strengthening effect on Ti alloys [18, 19]. At the same time, Sc reacts with oxygen in the matrix to form Sc$_2$O$_3$, which is distributed between the lamellar structures and inhibits the movement of the dislocations [20, 21]. Therefore, the compression creep properties of Nos. 3 and 4 are greatly enhanced as...
opposed to those of No. 1. Compared with the other rare earth elements, Sc has a higher solubility in the $\alpha$ phase. In the examined alloy system, Sc is expected to enhance $\alpha$ phase via solid solution strengthening. On the other hand, the addition of Sc leads to the formation of high melting point oxides, thus inhibiting the movement of dislocations. Furthermore, the addition of Sc refines the grain size of the alloys. Therefore, yield strength at room temperature is enhanced by the addition of Sc.

It is difficult to activate twins in Ti-6Al-4V alloys due to the inhibition of Al and V on twins. Only under extreme conditions, such as low temperatures $^{[22]}$, or high strain rates $^{[23]}$, can twins be found. Twins were present in both Nos. 1 and 3, thus indicating that Ti-6Al-4V alloys can be activated to produce twins in compression creep tests. However, no twins were found in Nos. 2 and 4, thus indicating that Si can inhibit the formation of twins in Ti-6Al-4V alloys. There have been studies that conclude that interstitial elements can cause lattice distortion in Ti alloys $^{[24]}$. Reducing the amount of interstitial elements is therefore beneficial to the generation of dislocation slips and deformation twins at low temperatures. Si is an interstitial $\beta$-stabilizer, which increases the amount of interstitial atoms in Ti alloys and can prevent the production of dislocations and deformation twins $^{[25, 26]}$.

As discussed above, the addition of Si and Sc enhances the compression creep properties of Ti alloys at room temperature. The compression creep mechanism of Ti-6Al-4V alloys at room temperature is mainly dislocation slip and a small amount of twinning. The addition of Si inhibits the formation of twins and strengthens the $\beta$-phase. Some of the Sc reacts with oxygen in the matrix, and the solid solution Sc has a good effect on Si because it increases the solid solubility of Si in $\alpha$ phase and strengthens the $\alpha$ phase.

Figure 7. Dislocations in samples after compression creep (a) No. 1, (b) No. 2, (c) No. 3, and (d) No. 4.
Figure 8. Dislocation wall after creep test: (a) No. 1, (b) No. 3, and (c) No. 4.

Figure 9. Twins after creep test: (a) No. 1 and (b) No. 3.
5. Conclusions

The compressive properties and deformation mechanism of Ti-6Al-4V-xSi-ySc alloys at room temperature are investigated. The following conclusions are made based on the findings in this study:

1. The experimental data of the creep compression test at room temperature are in good agreement with the plotted power-law function curves, and the creep is mainly found in the first and second stages of creep.
2. Both Si and Sc enhance the creep resistance of Ti alloys. Sc significantly reduces the amount of compression creep.
3. The mechanisms of compression creep of Ti-6Al-4V alloys are mainly dislocation slip and a small amount of twinning at room temperature. The addition of Si inhibits the formation of twins during the creep process.

Acknowledgments

The authors acknowledge financial support from the Natural Science Foundation of Jiangxi province (NO.20192BAB216005), Science and Technology Research Program of Jiangxi Educational Committee (NO. GJJ170550), and Natural Science Foundation of China (51837010).

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

ORCID iDs

Tongsheng Deng © https://orcid.org/0000-0001-8417-5059

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