Creep Behavior of UFG CP Ti at Room Temperature

Lei Luo, Xicheng Zhao, Xiao Yan Liu and Xirong Yang

College of Metallurgical Engineering, Xi’an University of Architecture and Technology, Xi’an 710055, China.
Email: luolei0301@126.com

Abstract. Ultra-fine grained commercial purity titanium (UFG CP Ti) is processed by Composite refining process (Equal channel angular pressing (ECAP), cold rolling and rotary swaging) at room temperature. The grain size is refined from 19 μm to 180 nm, and the ultimate tensile strength increase to 870 MPa. Creep tests were carried out on Ultra-fine grained commercial purity titanium with the stresses of 640, 660, 680, 700, 720, 740, 760 MPa at room temperature. Steady state creep rate and stress exponent n at various stresses were calculated for Ultra-fine grained commercial purity titanium, and creep deformation mechanism was also investigated. (With the rise of stress, the steady creep rate increases while the creep time decrease). The steady state creep rate reached maximum 1.416×10⁻⁶ s⁻¹ (under) stress of 760 MPa. The stress exponent is 17.3 when the stress was 640 ~ 700MPa, while the stress exponent is 55.7 when the stress was 700 ~ 760MPa, UFG CP Ti shows good creep property at room temperature. The creep deformation mechanism of UFG CP Ti is the dislocation creep.

1. Introduction
Titanium and titanium alloys are widely used in aerospace, shipbuilding, automobile manufacturing, chemical industry, biomedicine, etc. because of the good general mechanical performance, such as low density, high specific strength, good corrosion resistance and excellent biocompatibility[1]. The application in biomedicine as implant is restricted due to its low strength, poor abrasive resistance and other disadvantages of commercial purity titanium. However, it is an effective approach to improve mechanical property of commercial purity titanium and expand its application by acquiring ultra-fine microstructure [2]. Equal Channel Angular Pressing, abbreviated for, ECAP, is one of the main techniques for preparing bulk UFG CP Ti to improve comprehensive mechanical properties of materials via the method of refining grain [3]. Nevertheless, the microstructure of CP Ti after ECAP deformation is inhomogeneous, and the strength is less than the strength of TC4 titanium alloy. The study on further cold working to improve the strength of commercial purity titanium after ECAP has been conducted in order to improve mechanical property of commercial purity titanium after ECAP[4]. In this study, composite refining process of ECAP + cold rolling + rotary swaging is applied to prepare UFG CP Ti at room temperature. UFG CP Ti has overall mechanical properties (room-temperature tensile property, fatigue property, thermal stability and the like) at room temperature, UFG CP Ti as biomedical materials does not only demand good room-temperature tensile property, fatigue property and fracture property, but also good creep property. Creep usually refers to the rheological phenomenon that strain increases with prolongation time under constant stress less than yield strength under constant temperature. Creep property is affected by stress, time, structure property and other factors [5]. Creep performance of UFG CP Ti is one of the main bases to evaluate comprehensive mechanical properties. As biomedical material, the creep property of UFG CP Ti has a decisive effect for the service life and safety of material, UFG CP Ti is required to possess very small creep plastic strain at room temperature [6]. This experiment is focused on the creep property of UFG CP.
Ti under different levels of stress at room temperature, and the creep mechanism of UFG CP Ti at room temperature is analyzed.

2. Experimental Material and Method

Experimental material is the UFG CP Ti prepared via complex refinement (ECAP+ cold rolling + rotary swaging) at room temperature, and its chemical component (mass fraction, %) is shown in table 1, tensile strength is 870 MPa.

Table 1 Chemical composition of the UFG CP Ti (wt%)

| Fe  | C   | N   | H   | O   | Ti  |
|-----|-----|-----|-----|-----|-----|
| <0.20 | <0.014 | <0.03 | <0.0015 | <0.18 | Bal. |

TEM texture image and SADP pattern of UFG CP Ti are shown in Fig.1. It can be observed that, after severe plastic deformation, amount of dislocation aggregates in microstructure and a mass of high density dislocation tangles to cellular structure whose boundary is indistinct. Subgrain forms in internal microstructure with the misoritation between adjacent dislocation cells increasing[7]. In selected-area diffraction pattern, the diffraction spot distributes approximately like a ring, indicated that the grain of CP Ti gets refined remarkable after complex refinement process. The average grain size is about 180 nm.

Creep tests are carried out on UFG CP Ti on a RD-50 creep testing machine at room temperature under different creep stresses of 640, 660, 680, 700, 720, 740 and 760 MPa respectively. The gauge length is 25 mm and gauge diameter is Ф5 mm. The creep curve constructed according to test results is analysed, and the steady creep rate is calculated with the slope of the curve and the gauge length.

JEM-200CX transmission electron microscopy is adopted to observe microscopic structure of UFG CP Ti after creep test. 0.5 mm sample is cut up from the creep sample, coarsely grinded to 0.1 mm, and then, mechanically grinded to 40μm. MTP-1A automatic twin-jet electropolisher is adopted for electrolytic polishing to obtain transmission sample, the electrolyte is methyl alcohol: n-butyl alcohol: perchloric acid=12:7:1, voltage is 30V, power supply is 50mA and temperature is about -30°C.

3. Results and Discussion

3.1 CreepCurves of UFG CP Ti

The creep curves of UFG CP Ti under different stress constructed according to experimental data is shown in Fig. 2.
Figure 2. Creep curves of UFG CP Ti
(a) 640MPa; (b) 660MPa; (c) 680MPa; (d) 700MPa; (e) 720MPa; (f) 740MPa; (g) 760MPa
It can be seen from Fig.2 that when creep stress from 640MPa to 700MPa, creep curve of UFG CP Ti only presents stages I and II of typical creep curve, while the creep stress is 720MPa, 740MPa and 760MPa, creep curve of UFG CP Ti would present 3 stages I, II and III of the typical creep curve. Stage I is primary creep, the creep rate of primary creep stage is large; with the prolongation of time, it decreases gradually, and then, to a constant value. Stage II is steady state creep, the creep rate in such stage nearly keeps same, and the graph of creep stress and time is a straight line whose slope is steady state creep rate. Stage III is accelerated creep, with the prolongation of time, creep rate increases gradually till samples rupture [8]. Under low stress level, the creep is slow; the steady-state creep stage is big and the accelerated creep even does not happen, as shown in Fig.2 (a), (b), (c), (d), which contributes to the slowly intergranular dislocations slipping and climbing. With the creep stress level increasing, the steady-state creep stage is shortening and the creep enters the accelerated stage quickly, as shown in Fig.2 (e), (f), (g). Under the high stress level, the intergranular dislocations motion occurs due to the experiment load [9]. The dislocation movement is the dominated creep mechanism of UFG CP Ti at room temperature.

The room temperature creep presents \( \alpha \)-type creep under the condition of the test creep stress far blow the tensile strength of UFG CP Ti. The transient creep rate, namely, is high at the beginning, and it decreases gradually with the time, even to a certain constant range, as shown in Fig.2 (a). Thus, the room temperature creep of UFG CP Ti behaves saturation phenomena of creep under high stress level [10]. The creep value of UFG CP Ti at room temperature increases with the creep stress increasing, as shown in Fig. 2 (b), (c), (d), (e), (f) and (g). Therefore, room temperature creep is sensitive to creep stress, namely, room temperature creep value increases much more under high stress level when creep stress increment is same.

### 3.2 Steady Creep Rate of UFG CP Ti

According to creep mechanics theory, steady state creep rate is the minimum creep rate, which is one of the important indicators to characterize creep property of materials. Depending on Fig.2, the steady state creep rate can be calculated out according to the slope of steady-state creep and the gauge length of sample.

**Table 2 Steady state creep rate of UFG CP Ti at room temperature**

| Creep stress /MPa | Total testing time /h | Experimental result | Steady state creep rate/s\(^{-1}\) |
|-------------------|-----------------------|---------------------|----------------------------------|
| 640               | 174.5                 | not rupture         | 3.194\times10^{-9}              |
| 660               | 258.4                 | not rupture         | 4.806\times10^{-9}              |
| 680               | 190.6                 | not rupture         | 8.722\times10^{-9}              |
| 700               | 185.4                 | not rupture         | 1.472\times10^{-8}              |
| 720               | 110.4                 | creep rupture       | 1.023\times10^{-7}              |
| 740               | 24.9                  | creep rupture       | 4.763\times10^{-7}              |
| 760               | 6.4                   | creep rupture       | 1.416\times10^{-6}              |

As shown in table 2, the steady state creep rate increases with the creep stress increasing at room temperature and the steady-state creep stage is shorten. When Creep stress increases from 640 MPa to 700 MPa, UFG CP Ti creep sample fail to rupture, and creep curve only represents stages I and II (as shown in Fig.2 (a), (b), (c), (d)). While creep stress increases to 720 MPa, 740 MPa and 760 MPa, UFG CP Ti creep sample suffers creep rupture failure, and creep curve shows 3 complete stages (as shown in Fig.2 (e), (f), (g)). And with the increase of creep stress, the time of suffering creep rupture in UFG CP Ti sample shortens, creep rapidly transits to stage III from stage I. From Fig.2, under the creep stress of 760 MPa, the maximum steady state creep rate is 1.416\times10^{-6} s\(^{-1}\), while the steady state creep rate of commercial purity titanium on the codition of 150 \(^\circ\)C/240 MPa, is 4.339\times10^{-6} s\(^{-1}\) [11]. Hence, room temperature creep resistance of UFG CP Ti is higher and creep property is good.
3.3 Creep Stress Exponent of UFG CP Ti

The steady state creep rate of most metals or alloys can be represented as follows [12]:

\[ \dot{\varepsilon} = A\sigma^n \exp\left(-\frac{Q_{App}}{RT}\right) \]  

(1)

In the formula, \( \dot{\varepsilon} \) is steady state creep rate, \( A \) is material constant, \( \sigma \) is Apparent Creep Activation Energy, \( Q_{App} \) is nominal creep activation energy, \( R \) is universal gas constant, \( (R=8.314 \text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}) \), \( T \) is absolute temperature, unit is K, \( n \) is creep stress exponent.

When the temperature of creep process is constant, the expression of stress exponent \( n \) is as follows by equation (1) differential:

\[ n = \left(\frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sigma}\right)_T \]  

(2)

According to the data of table 2, the graph of \( \ln \dot{\varepsilon} - \ln \sigma \) is shown in Fig.3. The slope of \( \ln \dot{\varepsilon} - \ln \sigma \) relation curve is namely the creep stress exponent \( n \). Through calculation, when creep occurs in UFG CP Ti at room temperature, creep stress is constant., which is 640 ~ 700 MPa, the creep stress exponent \( n \) of UFG CP Ti is 17.3; when creep stress is 700 ~ 760MPa, the creep stress exponent is 55.7.

Figure 3. The plot of \( \ln \dot{\varepsilon} \) against \( \ln \sigma \) for UFG CP Ti

In this experiment, UFG CP Ti has higher creep stress exponent \( n \) (17.3 ~ 55.7), which shows that UFG CP Ti has better creep resistance at room temperature. Combined with literature[13], under 500 ~ 600 °C, when stress is 97 ~ 472 MPa, the creep stress exponent \( n \) of Ti-6Al-4V alloy is approximate 5.2 ~ 11.3, consistenting with the rules of the experimental result. In this study, the creep stress exponent \( n \) of UFG CP Ti is bigger than the high-temperature creep stress exponent \( n \) of Ti-6Al-4V alloy, which means that room temperature creep property of UFG CP Ti is good.

3.4 Microscopic Structure of UFG CP Ti

Comparing the TEM images of UFG CP Ti after creep test with the before, as shown in Fig.4, it can be investigated that, after composite refining deformation, amount of cellular substructures forms in the microstructure of CP Ti and the twin crystal develops into subgrain by self-intersection. At the same time, severe plastic deformation causes greatly dislocation accumulation in the microstructure; high-density dislocation accumulates into cell wall; and dislocations tangle into clusters, the boundary of cellular structure is distinct. With the increase of cell dislocation density, misorientation between adjacent dislocation cells gradually increases to develop into subgrain.
Micromechanism of creep is closely related to the change of internal material microstructure and the behavior of dislocation configuration. Creep deformation mainly includes two mechanisms, one is dislocation deformation and the other is diffusion creep deformation. Under big stress and low temperature (< 0.5Tm), the creep mechanism is mainly the dislocation movement, while under higher temperature (0.6 ~ 0.7Tm) and relatively less stress, it is mainly diffusion creep deformation. At room temperature, external load causing stress applied on materials, intergranular dislocation is enable to move, resulting in plastic deformation, and new dislocation forms during the process of plastic deformation, causing multiplication. Dislocation moves on the glide plane, avoiding from accumulating on grain boundary, so as to make deformation on-going[14].

It can be seen from Fig.4 that, under applied stress, at the beginning of creep, intergranular dislocation could move freely and multiply constantly. Therefore, the deformation rate of creep in stage I is very high. With creep proceeding, dislocation pileup happens on the grain boundary, the original dislocation with free distribution is transformed into dislocation cell, as shown in Fig.4 (b), (c), (d). Screw dislocation leaves blocking area via dislocation gliding and climbing, at this time, dislocation cell wall is the small angle grain boundary resulted by dislocation gliding and climbing. The dislocation distribution of intracellular sliding and cell wall climbing forms a long-range inner gravitational field. Under the action of external force, dislocation moves on glide plane along with the gliding direction, till to surface and then disappearing. Therefore, the equilibrium state of the original inner gravitational field is broken, and dislocation source starts moving and generates new mobile dislocation. The dislocation desity is very high in the microstructure of UFG CP Ti and dislocations movement results in the creep. Therefore, the creep mechanism of UFG CP Ti room temperature creep is dislocation creep, its dominate deformation is dislocation glide, and at the same time, the creep property of material is also affected by the interaction between UFG CP Ti microstructure, such as, grain size, the number of grain boundary, and dislocation.

Figure 4. TEM micrographs of UFG CP Ti
(a) before creep; (b) 660 MPa; (c) 700 MPa; (d) 740 MPa
4. Conclusions
1) Under the low stress level, the creep behave saturation phenomenon. With the increase of creep stress, creeps value of UFG CP Ti room temperature creep increases gradually.
2) At room temperature, when creep stress of UFG CP Ti is 760 MPa, steady state creep rate is 1.416×10^{-6} s^{-1}, and creep property is good.
3) When creep stress is 640~700 MPa, creep stress exponent n of UFG CP Ti is 17.3, and when creep stress is 700~760 MPa, the creep stress exponent n is 55.7.
4) The creep mechanism of UFG CP Ti during room temperature creep is dislocation creep, and it’s dominate creep deformation is dislocation glide.

5. References
[1] N. S. Weston, F. Derguti, and A. Tudball, “Spark plasma sintering of commercial and development titanium alloy powders,” Journal of Materials Science, vol. 50, pp. 4860-4878, 2015.
[2] S. Hariprasad, M. Ashfaq, and T. Arumailaippan, “Role of electrolyte additives on in-vitro corrosion behavior of DC plasma electrolytic oxidation coatings formed on Cp-Ti,” Surface and Coatings Technology, vol. 3, pp.16-44, 2016.
[3] Christopher S. Meredith and Akhtar S. Khan, “The microstructural evolution and thermo-mechanical behavior of UFG Ti processed via equal channel angular pressing,” Journal of Materials Processing Technology, vol. 219, pp. 257-270, 2015.
[4] P. Rodriguez-Calvillo and J.M. Cabrera, “Microstructure and mechanical properties of a commercially pure Ti processed by warm equal channel angular pressing,” Materials Science and Engineering: A, vol.625, pp. 311-320, 2015.
[5] D.V. Gunderov, A.V. Polyakov, and I.P. Semenova, “Evolution of microstructure, macrotexture and mechanical properties of commercially pure Ti during ECAP-conform processing and drawing,” Materials Science and Engineering: A, vol. 562, pp. 128-136, 2013.
[6] P. SudharshanPhani and W.C. Oliver, “A direct comparison of high temperature nanoindentation creep and uniaxial creep measurements for commercial purity aluminum,” Acta Materialia, vol. 111, pp.31-38, 2016.
[7] D. Andrés, R. Lacalle, and J.A. Álvarez, “Creep property evaluation of light alloys by means of the Small Punch test: Creep master curves,” Materials & Design, vol. 96, pp. 122-130, 2016.
[8] W. Pachla, M. Kulczyk, and S. Przybysz, “Effect of severe plastic deformation realized by hydrostatic extrusion and rotary swaging on the properties of CP Ti grade 2,” Journal of Materials Processing Technology, vol.221, pp. 255-268, 2015.
[9] Qiao Dai, Chang-Yu Zhou, and Jian Peng, “Room-temperature creep behavior on crack tip of commercially pure titanium,” Materials & Design, vol. 85, pp. 618-625, 2015.
[10] B. Barkia, V. Doquet, and J.P. Couzié, “Room-temperature creep and stress relaxation in commercial purity titanium—Influence of the oxygen and hydrogen contents on incubation phenomena and aging-induced rejuvenation of the creep potential,” Materials Science and Engineering: A, vol. 624, pp. 78-89, 2015.
[11] Ş. Țălu, Micro and nanoscale characterization of three dimensional surfaces. Basics and applications. Napoca Star Publishing House, Cluj-Napoca, Romania, 2015.
[12] Peng Jian, Zhou Changyu, and Dai Qiao, “The temperature and stress dependent primary creep of CP-Ti at low and intermediate temperature,” Materials Science and Engineering: A, vol. 611, pp. 123-135, 2014.
[13] D.A.P. Reis, C. Moura Neto, and C.R.M. Silva, “Effect of coating on the creep behavior of the Ti–6Al–4V alloy,” Materials Science and Engineering: A, vol. 486, pp. 421-426, 2008.
[14] Yi-jie Zhao, Yong Zheng, and Wei Zhou, “Effect of carbon addition on the densification behavior, microstructure evolution and mechanical properties of Ti(C, N)-based cermets,” Materials Characterization, vol. 106, pp. 266-272, 2015.