Available online at www.sciencedirect.com
ScienceDirect
Trans. Nonferrous Met. Soc. China 23(2013) 1209−1214
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

"Abnormal transient creep" in fine-grained Al-5356 alloy observed at low strain rates by high-resolution strain measurement

Jun-jie SHEN[1], Kenichi IKEDA[2], Satoshi HATA[2], Hideharu NAKASHIMA[2]

1. School of Mechanical Engineering, Tianjin University of Technology, Tianjin 300191, China;
2. Department of Electrical and Materials Science, Faculty of Engineering Sciences, Kyushu University, Kasuga, Fukuoka 816-8580, Japan

Received 4 May 2012; accepted 11 March 2013

Abstract: Transient creep at very low strain rates (less than 10^{-10} s^{-1}) is still unclear. The traditional uniaxial creep testing is less useful due to unsatisfied resolution strain (~10^{-6}). To study transient creep behavior at such low strain rates, a high-resolution strain measurement using the helicoid spring specimen technique was employed in a fine-grained Al-5356 alloy at temperatures ranging from $0.47T_m$ to $0.74T_m$ (T_m: melting point). To clarify transient creep mechanism at such low strain rates, transmission electron microscopy (TEM) was used in microstructure observation of crept specimens. The abnormal transient creep, high temperature strengthening at $T>T_p$ (T_p: the phase transformation temperature, $0.58T_m$) or intermediate temperature softening at $0.4T_m<T\le T_p$ and double-normal type (creep curves including double work-hardening stages) at $T=T_p$, were firstly observed. The substructure observation in a crept specimen at $T=0.58T_m$ and $\varepsilon=1\times10^{-4}$ shows pile-up dislocations including many small jogs with equal interval, and dislocations emitted from grain boundaries. The β-Al_3Mg_2 phase dissolves under the condition of testing temperatures higher than 523 K, which causes solid-solution quantity of Mg atoms to increase. Therefore, the "abnormal transient creep" may be related to the difference of solid solution strengthening caused by phase change during the creep tests.

Key words: Al-5356 alloy; transient creep; phase change; low strain rate

1 Introduction

The demand for safe creep lives of industrial parts is frequently 20 or 30 years, even longer. In this case, the creep rate will be very low, in the order of 10^{-10} s^{-1} or even lower [1−3]. It is difficult or even impossible to carry out long-term creep experiment (>20 year) according to real service conditions. Therefore, study of creep at such low strain rates has to be based on short-term creep (accepted period of creep experiment). However, conventional uniaxial creep tests are less useful due to unsatisfactory strain resolution of strain measurements.

As an alternative method of short-term creep at very low strain rates, a helicoid spring specimen technique (based on the torsion deformation) has been attempted so far [4−10] because of its high strain resolution, even reaching 10^{-8} [8,10]. Analysis of all of these creep data is based on extrapolating steady-state creep rates by fitting primary transient creep curves. However, as for practical alloys, there is no steady-state creep rate, but minimum one [1−3]. Thus, this approach (analysis of creep data based on extrapolating steady-state creep rates) hides an error of taking a steady-state creep rate instead of the minimum one. The physical meaning of the steady-state creep rate and minimum creep rate is completely different. The former, steady-state creep rate, is associated with a balance of work hardening and creep recovery under the condition of stable microstructure, whereas the latter, the minimum one, may be related with a balance point of work-hardening and recovery caused by deterioration of microstructure.

As another creep stage, transient creep is less studied, but it is extremely important at very low strain rate because identification of the secondary creep stage requires very long testing time, even longer than 40 years [1]. Therefore, the objective of this study is to focus on transient creep in the helicoid spring specimen of a fine-grained Al-5356 alloy [10]. In this alloy containing

Foundation item: Project (12JCYBJC32100) supported by Tianjin Research Program of Application Foundation and Advanced Technology, and in part by Grants-in-Aid from the Japan Society for the Promotion of Science (JSPS)
Corresponding author: Jun-jie SHEN; Tel: +86-13516233995; E-mail: sjj1982428@sina.com
DOI: 10.1016/S1003-6326(13)62585-6

5% (mass fraction) Mg, dissolution of β-Al_3Mg_2 phase into Al solid-solution occurs at 523 K (In this study, we define a temperature higher than the phase transformation temperature as high temperature), and solute Mg atoms increase with temperature, as recognized in Fig. 1 [11]. Here, we firstly reported the “abnormal transient creep”: high temperature strengthening at $T>T_p$ (T_p: the phase transformation temperature, $0.58T_m$) or intermediate temperature softening at $0.4T_m<T\le T_p$ and double-normal type at $T=T_p$. The substructure observation in a crept specimen at $T=T_p$ showed pile-up dislocations including many small jogs with equal interval and dislocations emitted from grain boundaries. The phase transformation may be the most important reason of abnormal features of transient creep at low strain rates. The findings are important to understand creep behavior at very low strain rates.

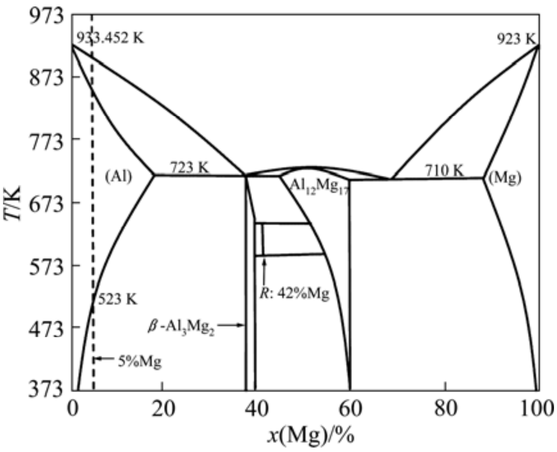

Fig. 1 Phase diagram of binary Al−Mg system [11] (Dashed line depicts Mg concentration in Al-5356 alloy)

2 Experimental

The experiments were performed on an Al-5356 alloy in the form of wires with diameter d=1.6 mm. Its chemical composition (mass fraction) was 4.81% Mg, 0.08% Si, 0.13% Fe, 0.01% Cu, 0.07% Mn and 0.08% Cr, with the balance Al. Helicoid spring specimens with a mean coil diameter, D=18.8 mm, and the coil-pitch spacing, δ=2.5 mm, were prepared by winding the wires on threaded stainless steel bolts [10]. The bolts with wound wires were heat-treated in air for 48 h at 723 K to fix the helicoid shape and obtain a recrystallized microstructure. The grain size after the heat treatment was d_g=(5±0.5) μm. Creep tests were performed using the helicoid spring technique at 423−673 K and applied stresses $\sigma\le$10 MPa. Figure 2 illustrates the apparatus of helicoid spring creep tests which has three adjacent heaters that are independently controlled to obtain uniform temperature distribution in the helicoid sample with an accuracy of ±1 K [10]. The pitches of the helicoids of the sample were measured using a light-emitting diode with an accuracy of 0.5 μm.

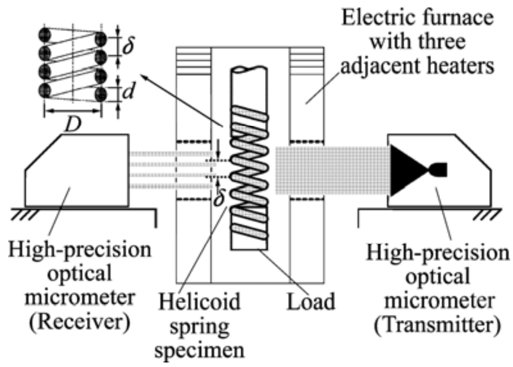

Fig. 2 Scheme of helicoid-spring creep machine

The following equations [12,13] were used to calculate the mean surface shear stress, τ, and the surface shear strain, γ, assuming pure torsion of the helicoid spring specimen:

$$\tau=\frac{8PD}{\pi d^3} \tag{1}$$

$$\gamma=\frac{\Delta\delta d}{\pi n D^2} \tag{2}$$

where P is the average load, D is the coil diameter (18.8 mm), d is the wire diameter (1.6 mm), n is the number of coils measured (5) and $\Delta\delta$ is the displacement of the mean coil-pitch spacing. In this study, the torsion deformation was the dominant component because D is much greater than d (D/d>12) [8], and the value of δ is between 2.5 mm and 4.0 mm [9]. Since the stress and strain in the helicoid spring have essentially shear components, they can be transformed to the equivalent tensile quantities using von Mises equations for tensile stress $\sigma=\sqrt{3}\tau$ and tensile strain $\varepsilon=\gamma/\sqrt{3}$. The minimum detectable value of strain for the helicoid spring creep test was evaluated from Eq. (2). For example, the minimum detectable strain for the present experiment is ε=8.4×10^{-8} under the following conditions: d=1.6 mm, D=18.8 mm and n=5, measured using a light-emitting diode with a resolution of 0.5 μm.

3 Results and discussion

3.1 Mechanical properties

The creep curves acquired at 423 K ($0.47T_m$), 523 K ($0.58T_m$) and 623 K ($0.70T_m$) [14] are shown in Figs. 3(a), (b) and (c), respectively. At 423 K and 623 K, the creep curves (Figs. 3(a) and (c)) exhibit typical normal type transient creep: a transient stage in which the creep rate decreases monotonically with time. The normal type transient creep is generally considered to be due to work-hardening. On the other hand, for the intermediate

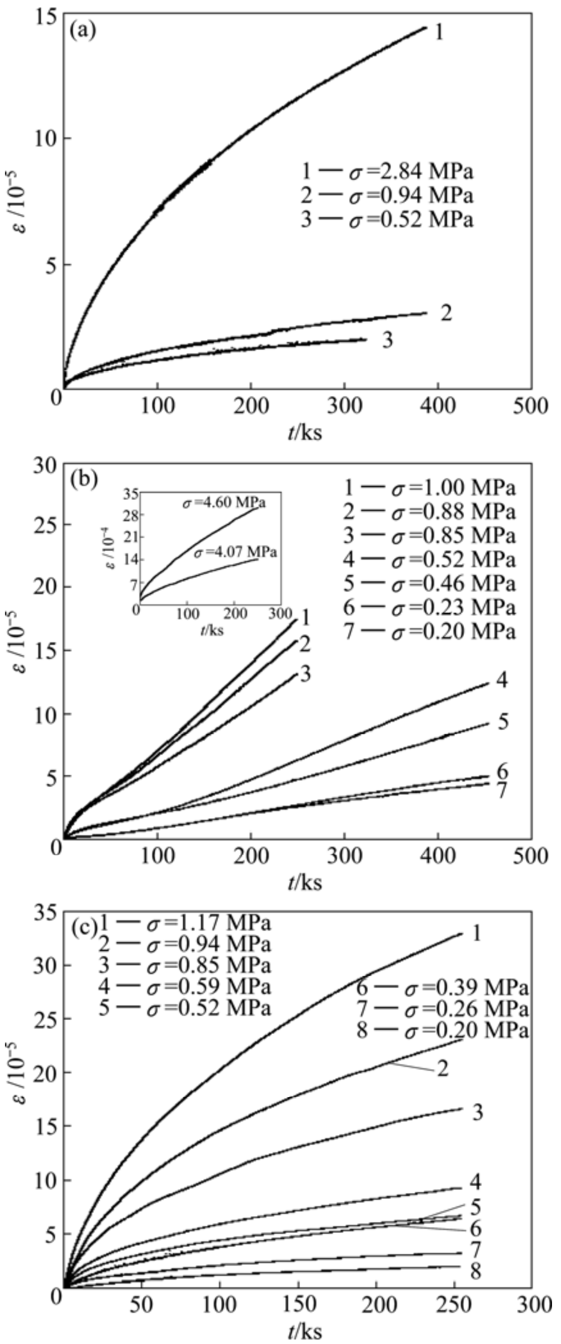

Fig. 3 Strain versus time curves at 423 K (a), 523 K (b) and 623 K (c) [10]

temperature, 523 K (The phase transformation temperature, $0.58T_m$), the creep curves (Fig. 3(b)) exhibit a different type of transient creep, which is called "double-normal type" transient creep because double stages of work-hardening are recognized on the creep curves. In other words, the creep rate firstly decreases with time until it reaches a minimum, and subsequently turns to increase with time until it reaches a maximum. Further, the creep rate decreases again after reaching the maximum. In Fig. 4, the strain rate vs time or strain curves at 523 K show again the "double-normal type"

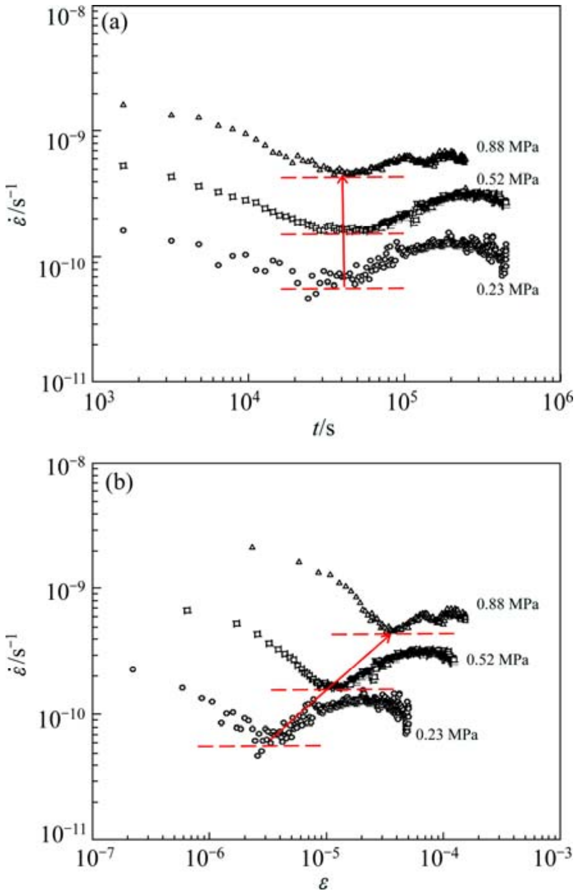

Fig. 4 Strain rate versus time (a) and strain (b) curves at 523 K

transient creep in detail. The time to reach minimum strain rates indicated by broken lines is independent on stress, as shown in Fig. 4(a), while the magnitude of strain to reach minimum strain rate is dependent on stress, as shown in Fig. 4(b). Such "double-normal type" transient creep is only observed at low stresses ($\sigma \le 1.0$ MPa) or low strain rates. The transient creep curves exhibit the normal type ones for high stresses or high strain rates, as shown in the inset of Fig. 3(b).

In the course of analyzing the transient creep at a certain stress and different temperatures, another unusual behavior of transient creep is observed, as shown in Fig. 5(a). When the temperature for the creep tests at 0.52 MPa is increased from 523 K to 573 K, the time evolution of creep strain becomes slower, which is called "high temperature strengthening" or "intermediate temperature softening" in the transient creep. The "high temperature strengthening" ($T>T_p$) or "intermediate temperature softening" ($0.4T_m<T\le T_p$) can be seen clearly on the creep rate vs time or strain curves, as shown in Figs. 5(b) and (c).

3.2 Substructure observation

The dislocation substructure in the crept specimen at T=523 K (the phase transformation temperature),

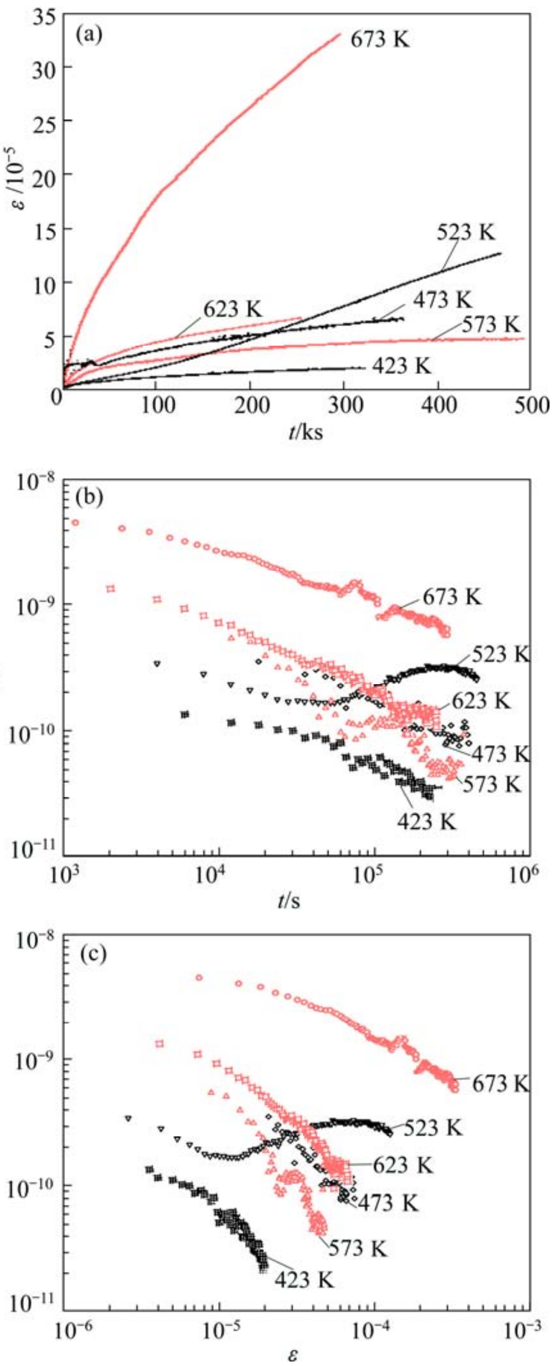

Fig. 5 Creep curves at stress of 0.52 MPa and different temperatures of 423 K, 473 K, 523 K, 573 K [10], 623 K [10] and 673 K [10]: (a) Strain versus time curves; (b) Strain rate versus time curves; (c) Strain rate versus strain curves

σ=0.46 MPa and ε=1×10^{-4} is shown in Fig. 6. The photography shows the following major features of dislocations substructure: non-uniform dislocation distribution; low dislocation density; dislocation pile-up including many small jogs with equal interval and the particle acting as a source of pile-up dislocations. The non-uniform dislocation distribution may be due to the non-uniform distribution of stress [10,12,13]. Low dislocation density is reasonable since the strain (1×10^{-4}) is low. The most notable feature different from that at high temperatures [10] is the formation of dislocation pile-up, which indicates strengthening of interior deformation at intermediate temperature. The pile-up dislocations including many small jogs with equal interval are also observed in Fig. 7 in the regions *B*, *C* and *D*. The figure also shows dislocations emitted from the junction of grain boundaries in the region *A*.

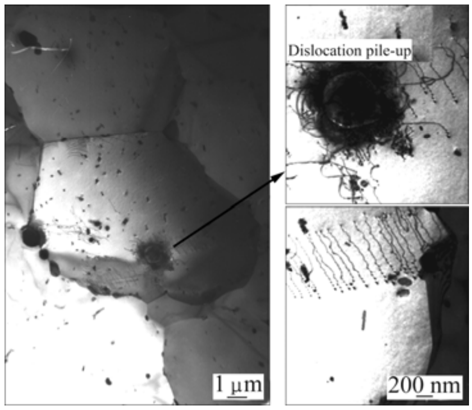

Fig. 6 Dislocation substructure in crept specimen at T=523 K, σ=0.46 MPa and ε=1×10^{-4} showing non-uniform dislocation distribution, low dislocation density, dislocation pile-up including many small jogs and particle acting as a source of pile-up dislocations

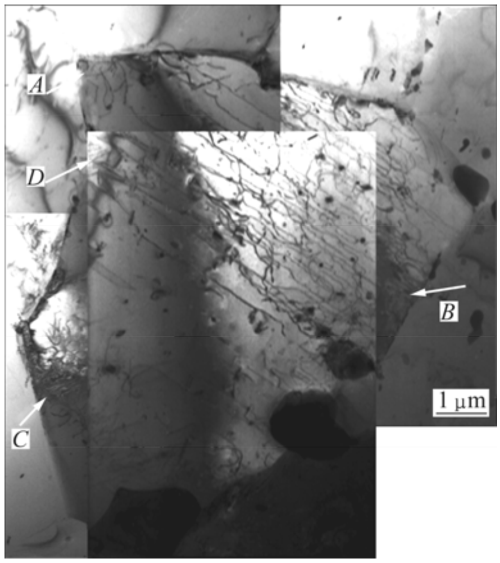

Fig. 7 Dislocation substructure in crept specimen at T=523 K, σ=0.46 MPa and ε=1×10^{-4} showing dislocation emission from junction of grain boundaries in region *A* and dislocation pile-up near grain boundaries in regions *B*, *C* and *D*

A more clear view and detailed analysis of dislocations in region *D* of Fig. 7 is shown in Fig. 8 by the Burgers vector analysis performed by putting the dislocations out of the contrast and using the standard $\boldsymbol{g}\times\boldsymbol{b}$=0 invisibility criterion. The Burgers vector for pile-up dislocations marked by arrow E is $\boldsymbol{b}=1/2[\bar{1}01]$. For the dislocations indicated by the arrow F, it is $\boldsymbol{b}=1/2[011]$. The results indicate intersection of dislocations in different slip planes that forms the jogs in pile-up dislocations.

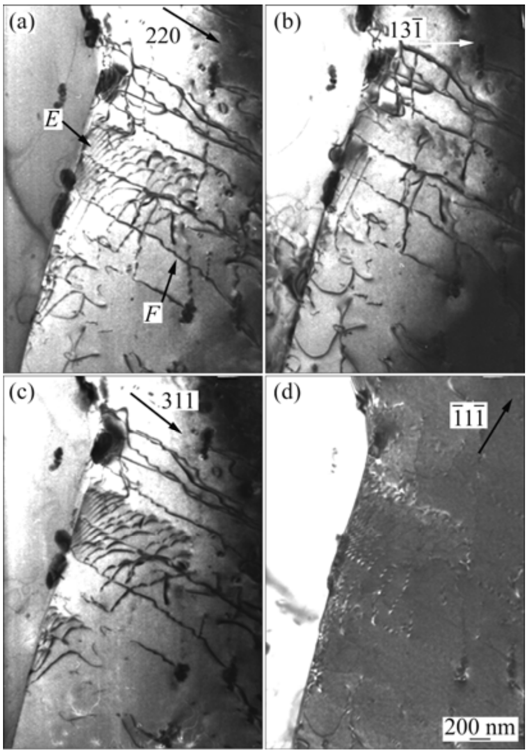

Fig. 8 Clearer view and detailed analysis of dislocations in region D of Fig. 7

Enlarged photographs of region A in Fig. 7 are shown in Fig. 9. A Burgers vector analysis indicates the emitted dislocation with $\boldsymbol{b}=1/2[0\bar{1}1]$ having the characters of lattice dislocations. The emission of lattice dislocations from junctions of grain boundaries is in a way of small arcs, which indicates that grain boundary sliding occurs and is hindered at the junctions of grain boundaries.

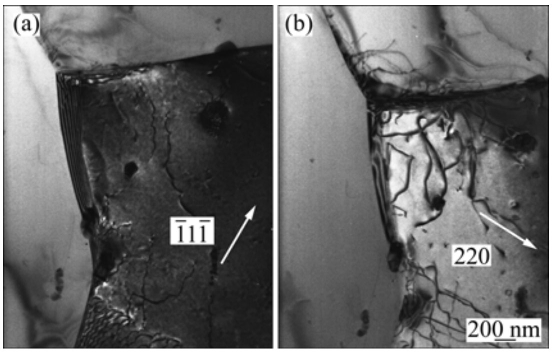

Fig. 9 Enlargement views of region A in Fig. 7

3.3 Likely mechanism

The high temperature strengthening at $T>T_p$ or intermediate temperature softening at $0.4T<T\leq T_p$ and double-normal type behavior at $T=T_p$ in the transient creep at very low strain rates are firstly observed. These creep features are totally different from those reported in class I alloy [14]. Those "abnormal transient creeps" may be related with phase changes in the Al-5356 alloy during the creep tests. The Al−Mg phase diagram in Fig. 1 shows that β-Al_3Mg_2 dissolves at $T>523$ K (250 °C) for 5% Mg, as indicated by a broken line. In this study, the β-Al_3Mg_2 exists in the initial specimen, since the specimen is treated after annealing at 723 K for 48 h. Therefore, the β-Al_3Mg_2 dissolves under the condition of testing temperatures higher than 523 K, which causes solid-solution quantity of Mg atoms to increase. The increase of the solid-solution quantity of Mg atoms (solid solution strengthening) may cause the high temperature strengthening ($T>523$ K). This suggested explanation is also supported by substructure observation. The notable difference of substructure in crept specimens at high and intermediate temperatures is pile-up dislocations that are not observed at high temperatures. The result indicates that solid solution strengthening has obvious effect at high temperatures than at intermediate temperatures.

The "double-normal" type transient behavior observed in crept specimens at $T=523$ K means at first work hardening, then work softening and work hardening again with the progress of creep. Generally, work hardening and softening are related with multiplication and annihilation of dislocations, respectively. The "double-normal type" may be correlated with double dislocation sources. In this study, the double dislocation sources should be grain boundaries and interior grain, since the specimen is a fine-grained ($d_g=5\pm0.5$ μm) alloy and the substructure observation in crept specimen at $T=523$ K shows pile-up dislocations and dislocations emitted from grain boundaries. The progress of creep may be described as follows: first grain boundary sliding occurs and is hindered somewhere, for example, junctions of grain boundaries that cause first work-hardening, then interior grains (for example β-Al_3Mg_2 phase) act as the other source of dislocations that cause work softening, and finally intersection of dislocations in different slip planes leads to work hardening again. On the other hand, when $T>523$ K, grain boundaries act as main dislocation source. When $T<523$ K, interior grain should act as main dislocation source. Therefore, the "double-normal type" transient behavior is only observed in crept specimens at $T=523$ K.

More detailed analysis of the abnormal transient creep by comparing quenched and annealed specimens with different grain sizes is being conducted.

4 Conclusions

The transient creep in a fine-grained Al-5356 alloy was investigated at low strain rates less than 10^{-8} s^{-1} and intermediate temperatures ranging from $0.47T_m$ to $0.74T_m$ by a high-resolution strain measurement using the helicoid spring specimen technique. The following “abnormal transient creep” phenomena were observed:

1) The high temperature strengthening at $T>T_p$ or intermediate temperature softening ($0.4T_m<T\le T_p$): Strain rate decreases with the increase of testing temperatures.

2) The double-normal type transient creep (double stages of work-hardening are recognized in the creep curves) at $T=T_p$: at first work hardening, then work softening and work hardening again with the progress of creep.

The substructure observation in crept specimen at $T=T_p$ shows pile-up dislocations including many small jogs with equal interval and dislocations emitted from grain boundaries. The β-Al_3Mg_2 phase dissolves under the condition of testing temperatures higher than 523 K, which causes solid-solution quantity of Mg atoms to increase. Therefore, the abnormal transient creep may be related to the difference of solid solution strengthening caused by phase change during the creep tests.

References

[1] KIMURA K, KUSHIMA H, SAWADA K. Long-term creep deformation property of modified 9Cr−1Mo steel [J]. Materials Science and Engineering A, 2009, 510: 58−63.

[2] KIMURA K, TODA Y, KUSHIMA H, SAWADA K. Creep strength of high chromium steel with ferrite matrix [J]. International Journal of Pressure Vessels and Piping, 2010, 87: 282−288.

[3] HANEY E M, DALLE F, SAUZAY M, VINCENT L, TOURNIÉI, ALLAIS L, FOURNIER B. Macroscopic results of long-term creep on a modified 9Cr–1Mo steel (T91) [J]. Materials Science and Engineering A, 2009, 510: 99−103.

[4] MALAKONDAIAH G, RAMARAO P. Creep of alpha-titanium at low stresses [J]. Acta Metallurgica, 1981, 29: 1263−1275.

[5] SKLENIČKA V, KUCHAŘOVÁK, SVOBODA M, KLOC L, BURŠÍK J, KROUPA A. Long-term creep behavior of 9–12%Cr power plant steels [J]. Materials Characterization, 2003, 51: 35−48.

[6] KLOC L, FIALA J. Harper–Dorn creep in metals at intermediate temperatures revisited: Constant structure test of pure Al [J]. Materials Science and Engineering A, 2005, 410: 38−41.

[7] KLOC L. Creep of ex-service 0.5CrMoV steel at low strain rates [J]. Materials Science and Engineering A, 2009, 510: 70−73.

[8] KLOC L, MAREČEK P. Measurement of very low creep strains: A review [J]. Journal of Testing and Evaluation, 2009, 37: 53−58.

[9] ISHIBASHI M, FUJIMOTO K, IKEDA K, HATA S, NAKASHIMA H. High temperature deformation behavior of Sn-based solder alloys under low stress conditions by a helical spring creep testing method [J]. The Japan Institute of Metals, 2009, 73: 373−380.

[10] SHEN J J, IKEDA K, HATA S, NAKASHIMA H. Creep mechanisms in a fine-grained Al-5356 alloy at low stress and high temperature [J]. Materials Transactions, 2011, 52: 1890−1898.

[11] PAHUTOVÁM, ČADEK J. On two types of creep behavior of F.C.C solid solution alloys [J]. Phys Status Solidi A, 1979, 56: 305−313.

[12] TIMOSHENKO S, YONG D H. Elements of strength of materials, [M]. 5th Ed. New York: Van Nostrand, 1968: 77−80.

[13] WAHI A M. Mechanical Springs [M]. 2nd Ed. New York: McGraw-Hill, 1963: 229−230.

[14] OIKAWA H, KURIYAMA, N, MIZUKOSHI D, KARASHIMA S. Effect of testing modes on deformation behavior at stages prior to the steady states at high temperatures in class I alloys [J]. Materials Science and Engineering, 1977, 29: 131−135.

细晶 Al-5356 合金在低应变速率下基于高分辨率应变测量的“异常瞬时蠕变”

申俊杰 [1], Kenichi IKEDA[2], Satoshi HATA[2], Hideharu NAKASHIMA[2]

1. 天津理工大学 机械工程学院 天津 300191；

2. Department of Electrical and Materials Science, Faculty of Engineering Sciences, Kyushu University, Kasuga, Fukuoka 816-8580, Japan

摘　要：极低应变速率下(<10^{-10} s^{-1})的瞬时蠕变仍然未知。由于应变分辨率低(~10^{-6})，传统的单轴拉伸/压缩等蠕变测试技术并不能满足此蠕变区的测试要求。采用具有高应变分辨率的螺旋弹簧蠕变试验法，研究 Al-5356 合金在温度范围为 $0.4T_m$~$0.8T_m$ (T_m 为熔点的绝对温度)、应变速率小于 10^{-10} s^{-1} 的瞬时蠕变行为。采用透射电子显微镜研究蠕变微观组织以揭示极低应变速率下的瞬时蠕变机制。实验结果首次显示了一些异常蠕变行为: “高温强化”($T>T_p$，T_p 为发生相变的温度，$T_p=0.58T_m$)或“中温弱化”($0.4T_m<T\le T_p$)和“双正常型蠕变”(蠕变曲线包含两次加工硬化阶段($T=T_p$))。试样经中温($0.58T_m$)蠕变后(1×10^{-4})，微观组织中发现有大量等间隔割阶的塞积位错和从晶界发射的弧形位错。当温度高于 523 K 时，β-Al_3Mg_2 相溶解引起固溶 Mg 量增加，因此，异常蠕变行为可能归因于蠕变过程中相转变引起的固溶强化的差异。

关键词：Al-5356 合金；瞬时蠕变；相变；低应变速率

(Edited by Sai-qian YUAN)