New creep region and mechanism in hexagonal close-packed metals

T Matsunaga¹,², T Kameyama²,³, S Ueda²,⁴, E Sato³

¹ Department of Space and Astronautical Science, School of Physical Sciences, Graduate University for Advanced Studies, Sagamihara, Kanagawa 229-8510, Japan
² Institute of Space and Astronautical Science, Japan Aerospace Exploration Agency, Sagamihara, Kanagawa 229-8510, Japan
³ Department of Materials Engineering, School of Engineering, University of Tokyo, Bunkyo, Tokyo 113-8656, Japan
⁴ Department of Aerospace Engineering, Faculty of System Design, Tokyo Metropolitan University, Hino, Tokyo 191-0065, Japan

Abstract. Only hexagonal close-packed (h.c.p.) materials show creep behaviour significantly at ambient temperature or less even below their 0.2% proof stresses with their stress exponents of 3.0 and their apparent activation energies of 20 kJ/mol. Transmission electron microscopy revealed dislocation arrays as a planar slip without any tangled dislocations inside each grain. Atomic force microscopy and electron backscatter diffraction pattern analyses brought about the occurrence of grain boundary sliding. The grain-size exponent was evaluated as 1.0, which means grain boundaries work as the barrier of the dislocation motion. Ambient-temperature creep of h.c.p. materials is schematically illustrated as that lattice dislocations move inside each grain without any obstacles and then pile up at grain boundaries. To continue the creep deformation, these dislocations are absorbed by grain boundaries to accommodate the internal stress and lead to grain boundary sliding.

1. Introduction

It has been revealed that several Ti alloys show significant creep behaviour at ambient temperature [1–6]. These works reported two types of deformation mechanisms: a planner slip in α-grains [1–3, 5]; time-dependent deformation twins [4]. Thompson et al. [1] evaluated apparent activation energy (Q) of 37 kJ/mol as dislocations overcame interstitial atoms. Neeraj et al. [5] and Hastija et al. [6] claimed that this creep was Andrade creep. However, the extra low Q value reported by ref. [1] contradicts that of Andrade creep which is 100 kJ/mol. Our previous works [7, 8] revealed that ambient-temperature creep appeared in not only Ti alloys but also hexagonal close-packed (h.c.p.) materials. Additionally, we introduced a new region, i.e. the ambient-temperature creep region [8], into the Ashby-type deformation mechanism map of annealed CP-Ti [9], where the Q value was 10 kJ/mol. According to these previous works [1–8], the deformation and the rate-controlling mechanism of ambient-
temperature creep have not been understood clearly. In the present paper, transmission electron microscope (TEM) and atomic force microscope (AFM) observations were conducted to reveal the both mechanisms. Samples were typical pure h.c.p. metals, i.e. commercially pure titanium (CP-Ti), pure magnesium (Mg) and pure zinc (Zn).

2. Experimental Procedure

Samples were rolled sheets of CP-Ti (JIS grade 1) with grain size, $d$, of 75 µm, Mg (99.95%) with $d$=120 µm and Zn (99.995%) with $d$=100 µm. Another Zn sample with $d$=210 µm was produced by annealing at 483 K for 24 h.

Tensile tests were performed to evaluate 0.2% proof stresses ($\sigma_{0.2}$) at ambient temperature with the initial strain rate of $1 \times 10^{-3}$ s$^{-1}$. Creep tests were conducted in tension at temperatures from 203 K to 873 K with loads of 0.5–0.9$\sigma_{0.2}$. The loading direction is parallel to the rolling direction in both tests. Strains were measured using strain gauges and an optical displacement camera. Creep curves were fitted by the logarithmic creep equation [10] to evaluate steady state creep rates:

\[
\dot{\varepsilon} = \dot{\varepsilon}_0 + \dot{\varepsilon}_p \ln(1 + \beta_p t) + s \dot{\varepsilon}_s t, \quad (1)
\]

where $\varepsilon$ is the total true strain, $\varepsilon_0$ is the instantaneous strain, $\varepsilon_p$ and $\beta_p$ are parameters characterizing the primary creep region, $\dot{\varepsilon}_s$ is the extrapolated steady state creep rate and $t$ is the elapsed time.

TEM observations were performed after creep tests lasting $4.3 \times 10^5$ s with $0.8\sigma_{0.2}$ to clear a dislocation structure. AFM observations were executed before and after creep tests to observe grain boundary sliding (GBS). Specimens were polished mechanically using colloidal silica before the tests.

3. Results

The $\sigma_{0.2}$ at ambient temperature were 176, 61, 40 and 24 MPa for CP-Ti, Mg and Zn with $d$=100 and 210 µm, respectively. The right part of Fig. 1 is a log-log plot of modulus compensated applied stress, $\sigma/E$, and $\dot{\varepsilon}_s$ at 298 K; all specimens show the stress exponents, $n$, of about 3.0. It is noted that $E$ values were obtained by tensile tests in previous reports, whereas they were the reference data [9, 11] in this study. A log-log plot of $b/d$ vs. $\dot{\varepsilon}_s$ (the left part of Fig. 1) at $\sigma/E$=0.001, where $b$ is the Burgers vector, shows that the $p$ value is evaluated 1.0 for Zn.

The Arrhenius plots, Fig. 2, are shown for CP-Ti, Mg and Zn with $d$=100 µm at $\sigma/E$=0.004. These

Figure 1. Right part is a double logarithmic plot of $\sigma/E$ and $\dot{\varepsilon}_s$ at 298 K for all specimens; left part is that of $b/d$ and $\dot{\varepsilon}_s$ for Zn.

Figure 2. Arrhenius plots for CP-Ti, Mg and Zn with $d$=100 µm. The apparent activation energy of ambient-temperature creep is about 20 kJ/mol.
plots are divided into two regions. The higher-temperature regions, at \( T > 330 \, \text{K} \) for Mg and \( T > 353 \, \text{K} \) for Zn, have larger \( Q \) values of 76 and 98 kJ/mol. This region corresponds to the low-temperature dislocation creep region with dislocation-core diffusion. The \( Q \) value of CP-Ti, 166 kJ/mol, is higher than that of dislocation-core diffusion, 97 kJ/mol, reported previously [9] and might indicate lattice diffusion. Because it was influenced by strain aging [11], steady state creep rates at temperatures from 423 K to 573 K were not obtained. Another region with extremely low \( Q \) value of about 20 kJ/mol is the ambient-temperature creep region.

TEM images of CP-Ti, Mg and Zn with \( d=100 \, \mu\text{m} \) are shown in Fig. 3. Their total true strains were 0.003, 0.021 and 0.048 for CP-Ti, Mg and Zn, respectively. All images show same dislocation structure: dislocation arrays as a planar slip without any tangled dislocations inside a grain.

Figure 4 shows a creep curve and strain induced by GBS, \( \varepsilon_{\text{GBS}} \), with 0.8\( \sigma_{0.2} \) for Zn with \( d=210 \, \mu\text{m} \). \( \varepsilon_{\text{GBS}} \) was estimated by equation (2) [12]:

\[
\varepsilon_{\text{GBS}} = k \left( \frac{\nu}{d} \right) \tag{2}
\]

where \( k \) is a geometrical averaging factor, \( \nu \) is the component of GBS perpendicular to the stress axis and to the specimen surface. In this study, the \( k \) value is 1.1 because the specimens were polished before the tests. The broken line in Fig. 4 is the fitting curve of \( \dot{\varepsilon}_{\text{GBS}} \) using eq. (1), which revealed the extrapolated steady state \( \dot{\varepsilon}_{\text{GBS}} \) as \( 2.1 \times 10^{-10} \, \text{s}^{-1} \). \( \dot{\varepsilon}_{\text{GBS}} \) is only 6% of \( \dot{\varepsilon}_{s} \).

Figure 3. TEM images of (a) CP-Ti, (b) Mg and (c) Zn after creep test. The same dislocation structure, i.e. a planar slip without any tangled dislocations, was observed.

Figure 4. Creep curve and \( \varepsilon_{\text{GBS}} \) with 0.8\( \sigma_{0.2} \) of Zn with \( d=210 \, \mu\text{m} \). The dotted line indicates the \( \dot{\varepsilon}_{\text{GBS}} \) of \( 2.1 \times 10^{-10} \, \text{s}^{-1} \). This value is smaller than \( \dot{\varepsilon}_{s} \) by one order.
4. Discussion

Although higher-temperature dislocation creeps do not show the $p$ value, ambient-temperature creep show the grain-size dependency. In ambient-temperature creep region, lattice dislocations lying on a slip plane piled up at GBs easily and then these piled-up dislocations have to be accommodated to continue the creep. Slip-induced GBS was proposed as an accommodation process [13]. This process is as follows: (1) lattice dislocations pile up at GBs; (2) these dislocations resolve at GBs and are absorbed by them; (3) the dislocations slip on a boundary plane and produce GBS. In addition, the $Q$ value of this mechanism is evaluated 15 kJ/mol for AZ31 [13]. Since the $\dot{\varepsilon}_{\text{GBS}}$ is smaller than $\dot{\varepsilon}_s$ by one order, main part of the strain is yielded by the dislocation motion inside grains. Summarizing the results, ambient-temperature creep is occurred by lattice dislocations as a planner slip without tangled dislocations inside each grain and slip-induced GBS to accommodate piled-up dislocations in series.

The constitutive relation for ambient-temperature creep is identified in the form of the modified Dorn equation including the grain-size dependency [14]:

$$\dot{\varepsilon}_s = A D_0 \exp(-Q/RT) \frac{G b}{k T} \left( \frac{\sigma}{E} \right)^n \left( \frac{b}{d} \right)^p$$

where $A$ is a constant, $D_0$ is a frequency factor, $R$ is the gas constant and $k$ is Boltzmann’s constant.

5. Conclusions

To reveal the features of ambient-temperature creep, TEM, AFM and EBSD analyses were performed before and after creep tests. Typical h.c.p. metals show the distinguishing features differing from conventional dislocation creeps. The conclusions obtained from this work are shown bellow:

(1) The creep parameters were identified as $Q \approx 20$ kJ/mol, $n = 3.0$ and $p = 1.0$.
(2) The dislocation structure, a planner slip without tangled dislocations, was observed.
(3) The mechanism of GBS is resulted from lattice-dislocation motion, i.e. slip-induced GBS.
(4) Constitutive relation was proposed as equation (3).

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