Threshold stress for cyclic creep acceleration in copper

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The phenomenon of cyclic creep acceleration (CCA) is well known. Also, it has been observed experimentally that there exists a peak stress level above which CCA begins to occur. For copper the value of this peak stress is known to be independent of cyclic stress conditions such as stress amplitude and frequency. This stress level or the threshold stress was observed to increase with temperature in the range 0.4 to $0.5 T_m$. A model is suggested to predict the threshold stress level and to explain the temperature dependence of the stress. This model is based on the dislocation core diffusion process by the athermally generated excess vacancies. The concept of critical length of dislocation core is used for core diffusion fast enough to enhance dislocation climb. This critical length is expressed as a function of stress and temperature. From these concepts, the threshold stress is expressed as $\tau/G = A \exp(-\Delta Q/RT)$. The experimentally observed values of the threshold stress are in good agreement with this model, and the temperature dependence is also experimentally proved.

1. Introduction

It is well known that under certain conditions of test temperature and stress ranges, the creep rate under cyclic stress becomes faster than that of static creep for the same peak stress. This phenomenon is called cyclic creep acceleration (CCA). The phenomenon of CCA is found to occur up to a temperature below about $0.5 T_m$ [1, 2]. The degree of acceleration decreases with decreasing peak stress at a given temperature and finally at a certain stress level, the cyclic acceleration diminishes and cyclic creep retardation (CCR) begins to occur. This stress is called the threshold stress for CCA [3].

In this investigation, the threshold stress for pure copper is experimentally obtained and a model for the physical meaning of this stress is suggested.

2. Experimental procedure

Electrolytic copper (> 99.99% pure) was melted in a graphite crucible in a vacuum using an induction furnace to give the chemical composition of 99.99% Cu. The ingot was homogenized at 1170 K for 12 h, forged and rehomogenized. The forged ingots were rolled into sheets 1 mm thick. These sheets were machined to make tensile creep test specimens, whose tensile direction was the same as the rolling direction. The specimen had a gauge length of 25 mm and a width of 4 mm. Annealing was carried out in vacuum to obtain the average grain size of 0.04 mm.

Static creep tests were performed under a constant peak stress using a creep machine equipped with an Andrade-Chalmers constant stress arm. For cyclic creep tests, a specially designed load elevating apparatus [4] was attached to the Andrade-Chalmers arm to maintain not only constant peak stress, but also constant stress amplitude, frequency and loading– unloading time throughout a test. A trapezoidal wave shape of repeated tensile loading is used and for calculation of the cyclic creep rate, the total time for one cycle is used rather than the time of on-load only.

The test temperature was kept constant within $\pm 1 \text{ K}$ using an infrared reflection furnace. Strain was measured by a Schaevitz model HR 1000 LVDT.

3. Results and discussion

In the temperature range of $0.4 T_{\rm m}$, static and cyclic creep tests are conducted at a frequency of 3 cycles per minute (c.p.m.) and a stress amplitude of 90% peak stress. It is shown in Fig. 1 that the cyclic creep rate is observed to be faster than that of static creep rate in the stress range higher than $\tau = 2.3 \times 10^{-4}$ G. At high stress, CCA is very significant, however, because as the peak stress decreases, the degree of CCA tends to decline and finally acceleration disappears and retardation begins to occur at a certain stress level. This stress is called the threshold stress for CCA.

Further experiments have been performed to see the effect of the experimental parameters of frequency (Fig. 2) and amplitude (Fig. 3) on the threshold stress. As shown in Figs 2 and 3, regardless of the various conditions, the threshold stress remains unchanged. These unique experimental observations strongly suggest that the threshold stress should have a specific physical meaning which can be explained in terms of micromechanistic motion of dislocations. On the basis of these experimental observations, we have tried to explain the physical meaning of the threshold stress. As shown in Figs 2 and 3, at a stress higher than the



Figure 1 Stress dependence of the steady state creep rate for static (\odot) and cyclic (\triangle) creep in copper at 0.4 $T_{\rm m}$. (3 c.p.m., $\Delta\sigma/\sigma = 0.9$).

threshold stress the cyclic creep rate becomes faster as the cyclic stress effects become more severe (frequency and amplitude of cyclic stress increase). However, below the threshold stress level, the cyclic creep rate becomes more retarded as the effects of cyclic stress become more severe. Thus it can be said that above and below the threshold stress the cyclic stress assisted and retarded the creep deformation, respectively. The threshold stress may be the transitional stress where the cyclic stress effects on the creep deformation are reversed. To investigate the physical meaning of threshold stress more precisely, it is necessary to know the mechanism of creep deformation. For this purpose, the activation energies both for static and cyclic creep have been measured and the results are shown in Fig. 4. At 0.4 $T_{\rm m}$, the activation energy for static creep deformation is observed to be lower than 137 kJ mol⁻¹ which is 70% of the activation energy $(196 \text{ kJ mol}^{-1})$ of self diffusion or creep deformation of copper above



Figure 2 Stress dependence of the steady state creep rate for cyclic creep ($\Delta\sigma/\sigma = 0.9$) in copper at 543 K, and at various frequency conditions. (Δ) frequency 3 c.p.m., (∇) frequency 1 c.p.m., (\bigcirc) frequency 0.5 c.p.m., (\diamondsuit) frequency 0.2 c.p.m., (\Box) static creep.



Figure 3 Stress dependence of the steady state creep for cyclic creep (3 c.p.m.) at 543 K (0.4 $T_{\rm m}$), and at various stress amplitude conditions. (Δ) $\Delta\sigma/\sigma = 0.9$, (∇) $\Delta\sigma/\sigma = 0.75$, (\odot) $\Delta\sigma/\sigma = 0.5$, (\diamondsuit) $\Delta\sigma/\sigma = 0.3$, (\Box) static creep.

the 0.65 $T_{\rm m}$ range. This experimental evidence supports that static creep deformation is controlled by the pipe diffusion process [5–7].

Further evidence for the pipe diffusion controlled creep in this work is that the stress exponent is measured to be 4.4 and this is shown in Fig. 1. Recently, Nam *et al.* [8] have shown that the stress exponent is 3 for high temperature $(0.7 T_m)$ static creep if the creep deformation is controlled by lattice diffusion. On the other hand, they showed that the exponent should be 4.5 if pipe diffusion becomes the predominant deformation mechanism in the temperature range around $0.4 T_m$. Therefore, the controlling micromechanism of this static creep deformation of copper at $0.4 T_m$ is believed to be the total dislocation climb supported by the pipe diffusion process.

With this understanding of the deformation mechanism of static creep of copper, the controlling process of cyclic creep deformation at $0.4 T_{\rm m}$ is now discussed. It is well known [1, 9] and also shown in Fig. 4, that in the CCA region, the activation energy for cyclic creep is much smaller than that of static creep. Nam and Bradley [9] have measured the effective stress for static and cyclic creep. They found that the cyclic creep provides higher effective stress to reduce the apparent thermal activation energy in terms of $Q_{\rm L} - \sigma_{\rm e} V^* = Q_{\rm app}$, where $Q_{\rm L}$ is the activation energy for lattice diffusion, σ_e is the measured effective stress, V^* is the activation volume and Q_{app} is the apparent activation energy of the cyclic creep. On the basis of this result they have suggested the reason why cyclically crept specimens have higher effective stress. Their suggestion is that cyclic stressing generates more athermal vacancies and these excess vacancies can enhance the fast recovery during the unloading period to decrease the internal stress.

Having carried out static and cyclic creep tests with



fully annealed and quenched aluminium and copper specimens, Shin and Nam [10] have suggested that the jog can generate more vacancies under cyclic loading and they have also shown [11] by resistivity measurements that the cyclic stressing generates more athermal vacancies and these can promote dislocation recovery. Feltner [12] has also observed that excess vacancies are formed more easily during cyclic than static creep. The above experimental observations suggest that the apparent thermal activation energy for cyclic creep has to be reduced by an amount equivalent to the fraction of athermal vacancy generation by the mechanically applied external stress. This idea is quantitatively shown to be physically valid by Shin and Nam [13].

On the basis of creep mechanism at the intermediate temperature and the excess vacancies generated during cyclic creep, the cyclic creep acceleration mechanism can be suggested as follows. From Figs 1 to 4 it can be shown that in the region of CCR the stress dependence of cyclic creep rate and the apparent activation energy of cyclic creep have the same trend as those in the CCA region. Thus, in the region of CCR, some fraction of isothermal vacancy generated by external work seems to exist. So, for CCA, it can be said that the excess vacancies may not only be generated by external work but also consumed by recovery of the dislocation structure. If excess vacancies formed by the cyclic stress are consumed to make climb dislocations from the obstacles, deformation can be easier as the excess vacancy generation is accelerated. However, if the excess vacancies cannot be consumed to make climb dislocations, dislocation movement is retarded by the vacancy causing core dragging. In this case, deformation can be retarded more as the excess vacancy generation is accelerated. Therefore, the threshold stress can be explained as the critical point where the excess vacancy begins to assist dislocation climb.

When the deformation mechanism at the intermediate temperature is controlled because of the local climb by the pipe diffusion, the excess vacancies can assist in dislocation climb by moving along the dislocation core only. However, a critical length exists through which the excess vacancies can move along the dislocation. Therefore, if the distance between the sites of source and sink of excess vacancies is larger than that, the excess vacancies cannot be consumed by

Figure 4 Creep activation energy of the static (O) and cyclic creep at various stress frequency conditions. (a) $0.5 T_m$, $\sigma/E = 4 \times 10^{-4}$, $\Delta\sigma/\sigma = 0.9$, (\Box) 0.2 c.p.m., (Δ) 3 c.p.m.(b) $0.4 T_m$. For $\sigma/E = 10 \times 10^{-4}$ (-O-) static ($-\Delta$ -) 0.2 c.p.m., ($-\Box$ -) 0.5 c.p.m., ($-\overline{\Box}$ -) 1 c.p.m., ($-\overline{\nabla}$ -) 3 c.p.m. For $\sigma/E = 9 \times 10^{-4}$ (-O--) static, ($-\Delta--$) 0.2 c.p.m., ($-\Box$ -) 0.5 c.p.m., ($--\overline{\Box}$ -) 1 c.p.m., ($--\overline{\Box}$ --) 3 c.p.m. Q (kJ mol⁻¹).

local climb. From the above idea, it may be assumed that CCA can only occur when the critical length is larger than the distance between the sink and source of excess vacancies. Then the threshold stress can be interpreted as the stress where the critical length is equal to that distance. The critical length is calculated by Hirth and Lothe [14] as

$$L_{\rm c} = (2a)^{1/2} \exp(\Delta Q/2kT)$$
 (1)

where L_c is the mean free path of vacancy diffusion along the dislocation core prior to evaporation and ΔQ is the difference between the diffusion activation energy in lattice and dislocation core. The mean spacing between dislocations is reported by Weertman [15] to be

$$\bar{X} = G \boldsymbol{b} / 4\pi\tau \qquad (2)$$

where τ is the shear stress, G is the shear modulus and **b** (2.58 × 10⁻⁸ cm) is Burgers vector. Then, the distance between the nodal points can be obtained, by assuming the octahedral network structure, as

$$L = \bar{X}/3^{1/2} = 0.46Gb/\tau$$
 (3)

If L is larger than L_c , the excess vacancies cannot contribute to dislocation climb through core diffusion. Therefore, it is assumed that L must be at least the same as L_c to make CCA possible. Using Equations 1 to 3, the threshold stress is obtained as

$$\tau/G = (A/2^{1/2}) \exp\left(-\frac{\Delta Q}{2RT}\right)$$
(4)

Equation 4 shows that the threshold stress increases as the temperature becomes higher, which is a different tendency from the result of Lorenzo and Laird [2]. The threshold stresses at various temperatures are shown in Fig. 5. For copper the activation energy for pipe diffusion was reported to be 148 kJ mol⁻¹ [16], and for lattice diffusion to be 196 kJ mol^{-1} [5] in the absence of stress. Using these values, the temperature dependence of threshold stress is observed to be slightly larger than the experimental result as shown in Fig. 5 and the values of threshold stress are larger than the experimental results by 1.8 times. This discrepancy seems to be due to problems in deciding the activation energy for diffusion. Another problem can originate from determining the mean spacing between nodal points. L can also be experimentally obtained by



Figure 5 Temperature dependence of threshold stress for cyclic stress acceleration. $\bullet \sigma_{\rm th}$ from (ε versus 1/T), $\oint \sigma_{\rm th}$ from ($\dot{\varepsilon}$ versus σ). $\Delta Q = -21 \,\rm kJ \,mol^{-1}$.

measuring the stress dependence of activation energy and dislocation density. From the experimental results of Lee and Nam [17] using the above methods, the relationship of L and stress can be expressed by Equation 3, but the experimental value of the constant, 0.3, appears to be close to the calculated value of 0.46. If this experimental relation may be used instead of Equation 3, the measured threshold stresses are observed to be the same as the calculated values. With the correspondence of the measured and calculated values in the activation energy and the threshold stress, it can be said that the threshold stress may be explained as the critical stress of CCA for the local climb of dislocations assisted by excess vacancies moving through the dislocation core.

Lorenzo and Laird [2] recently suggested the idea of temperature and modulus normalized threshold stress for copper at 0.21 T_m and reported that this value is very close to those reported by Shetty and Meshii [3]. The threshold stress values reported by Lorenzo and Laird and by Shetty and Meshii are obtained in a very low temperature (0.08 to 0.3 T_m) range and they are much higher than those of our values. The modulus normalized threshold stress is inversely proportional to the homologous temperature. For this discrepancy between our results and those of Lorenzo and Laird, there are two possible interpretations. Below 0.3 T_m , the creep deformation mechanism seems to be different from the climb controlled recovery process to which the model of dislocation pipe diffusion cannot be applied. For another interpretation, it can be suggested that the tests by Lorenzo and Laird and by Shetty and Meshii were conducted under constant load condition. Thus the reported initial stress would be different from the stress in the secondary state (there will be no real steady state under constant load from which the creep rates are supposed to be obtained). Because under this constant load condition a minimum creep rate rather than a steady state creep rate can be obtained, the comparison of static and cyclic creep rate may not be so meaningful.

4. Conclusions

1. The threshold stress for cyclic creep acceleration increases with temperature in the range 0.35 to $0.55 T_{\rm m}$.

2. From the temperature dependence and the value of threshold stress, the threshold stress for CCA may be explained as the critical stress, where the excess vacancies moving through the dislocation line can be used for local climb for dislocations.

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