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Effects of cyclic stress on the creep behaviour and dislocation microstructure of pure copper in the temperature range 0.4 to 0.5 T_m

JAE KON LEE, SOO WOO NAM
Department of Materials Science and Engineering, Korea Advanced Institute of Science and Technology, PO Box 131, Cheongryng, Seoul, Korea

The creep deformation behaviour of polycrystalline pure copper under static and cyclic stress was studied in the temperature range 0.4 to 0.5 T_m. Both cyclic creep acceleration and retardation occurred depending on the condition of peak stress and temperature combination. The comparison of dislocation microstructures, developed during steady state static and cyclic creep deformation, has also been performed to determine the effect of cyclic stress on the dislocation microstructure and evidence for the enhanced recovery of the cell wall under cyclic stress was found. These effects of cyclic stress on the creep rate and dislocation microstructure were interpreted on the basis of diffusion-controlled recovery creep theory and the cyclic creep acceleration mechanism is suggested as the enhanced recovery of the cell wall with the help of athermally generated excess vacancies.

1. Introduction

The effects of cyclic tensile stress on creep deformation have been investigated by many researchers, with increasing interest in the theoretical and practical aspects. The experimental results of their work show that the creep rate can be accelerated (cyclic creep acceleration, CCA) [1–12] or retarded (cyclic creep retardation, CCR) [4, 5, 11–16] by cyclic stress. More generally, it has been accepted that CCA or CCR is not a phenomenon for a special material but one which can occur in any material depending on the applied creep conditions [4, 11, 12]. With the experimental results, some theories [1–12] have been suggested to interpret the cyclic creep behaviour but there still exists some controversy between them.

In this study, on the basis of classical creep theory, the behaviour of cyclic and static creep of pure copper were compared in the temperature range 0.4 to 0.5 T_m and peak stress range 30 to 80 MPa. The dislocation structures, developed during static and cyclic creep, were also investigated to achieve a better understanding of the CCA mechanism.

2. Experimental procedure

Cold-rolled pure copper (> 99.9%) sheets were machined to tensile creep specimens of 25 mm gauge length. These specimens were annealed to recrystallize at 973 K in vacuum. The average grain size was about 0.04 mm.

Creep tests were carried out with an Andrade–Chalmers type constant stress creep machine. For cyclic creep test, a specially designed load elevator [17] was attached to that machine. Using this equipment, peak stress, stress amplitude, frequency and the loading–unloading time ratio could be kept constant throughout the creep test. In this work, the square wave form of stress cycle with a frequency of 3 c.p.m. (on-load time = off-load time = 10 sec) was used for cyclic creep testing. During the off-load period of the cycle, 10% of the peak stress was maintained for the stability of the test machine. The creep temperature was kept constant within ± 1 K by using an infrared radiant furnace. An argon gas atmosphere was used to prevent oxidation during the creep test. Creep strain was measured with LVDT and a strip-chart recorder within an accuracy of 1×10^{-6} m. Both the static and cyclic creep tests were performed under the peak stress range of 30 to 80 MPa and in the temperature range 550 to 670 K (0.4 to 0.5 T_m).

For dislocation structure investigation, some specimens were quenched by rapid air blowing under the stress during steady state. The use of radiant furnace and rapid air blowing enables specimens to be quenched as fast as 50 K sec^{-1}, a quenching rate which was thought to be fast enough to prevent the recovery effect during sampling. These crept specimens were carefully thinned mechanically and chemically to 0.1 mm. These were electropolished to make a thin foil by the Bollmann method with 30% nitric acid solution in methanol. Using these samples, dislocation structures were investigated by transmission electron microscope (TEM) at 160 kV.

3. Experimental results

3.1. Creep test results

The shapes of the static and cyclic creep curves show normal primary and steady state as observed in most pure metals. The cyclic creep rate is defined as the net increment of peak strain with time.

The steady state creep rates, measured at various

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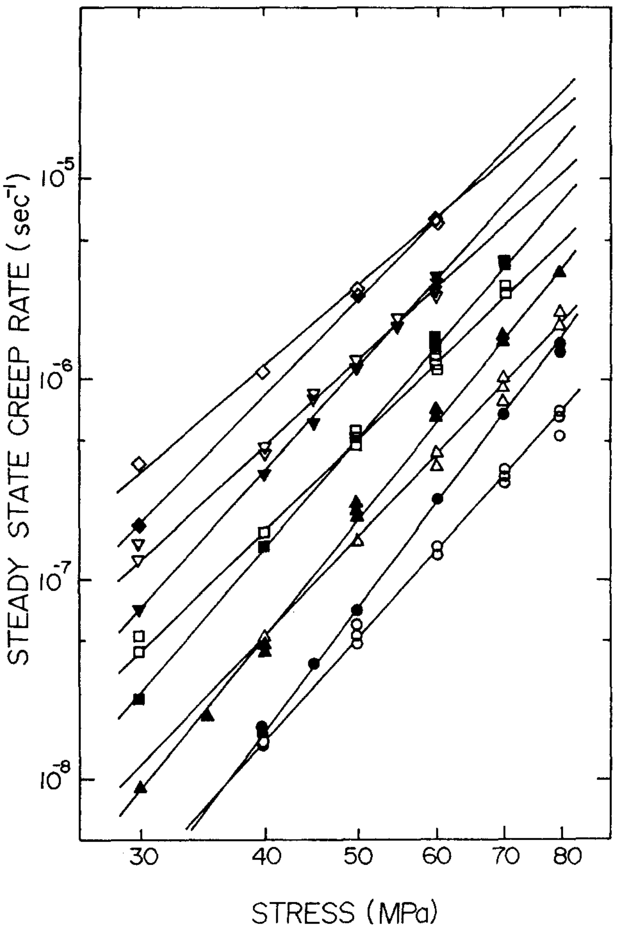

Figure 1 Steady state static and cyclic creep rates plotted against peak stress at various temperatures, for pure copper. The slopes of the lines imply the stress exponent *n*.

Static	Cyclic (3 c.p.m.)	T (K)	T/T_m
○	●	550	0.4
△	▲	580	0.425
□	■	610	0.45
▽	▼	640	0.475
◇	◆	670	0.5

TABLE I Dependence of stress exponent *n* on the stressing mode and temperature determined from Fig. 1

Temperature (K)	n_{st}	n_{cy}	Δn $(= n_{cy} - n_{st})$
550	5.43	6.57	1.14
580	5.16	6.11	0.95
610	4.78	5.78	1.0
640	4.55	5.46	0.91
670	4.29	5.07	0.78

peak stresses and temperatures, are plotted as a function of peak stress in Fig. 1 with the concept of thermally activated power law creep relation

$$\dot{\varepsilon} = A\sigma^n \exp\left(-Q_c/RT\right) \qquad (1)$$

where $\dot{\varepsilon}$ is steady state creep rate, σ is applied stress, n is stress exponent, Q_c is creep activation energy, R is gas constant and T is temperature. From this plot, although the stress exponent, n, is observed to change with temperature, it can be found that both the static and cyclic creep satisfy the power law relation. Another interesting result is that the stress exponents of cyclic creep are larger by about one than those of static creep over the entire temperature range of this work. The dependence of stress exponent on temperature and stressing mode is summarized in Table I. From Fig. 1 it is also found that the phenomena of CCA and CCR occur in certain stress and temperature ranges. In Fig. 2 the ratio of cyclic to static creep rate is shown for the various temperatures as a function of peak stress. This creep rate ratio is increased with increasing applied peak stress at a given temperature and with decreasing temperature at a given peak stress. The stress at which the creep rate ratio is one is the threshold stress for CCA, and it was found that the threshold stress also increased with increasing temperature. This temperature dependence of threshold stress is different from the recent suggestion of Lorenzo and Laird [11]. We will discuss this behaviour of threshold stress in elsewhere [18].

The static and cyclic creep rates are replotted in

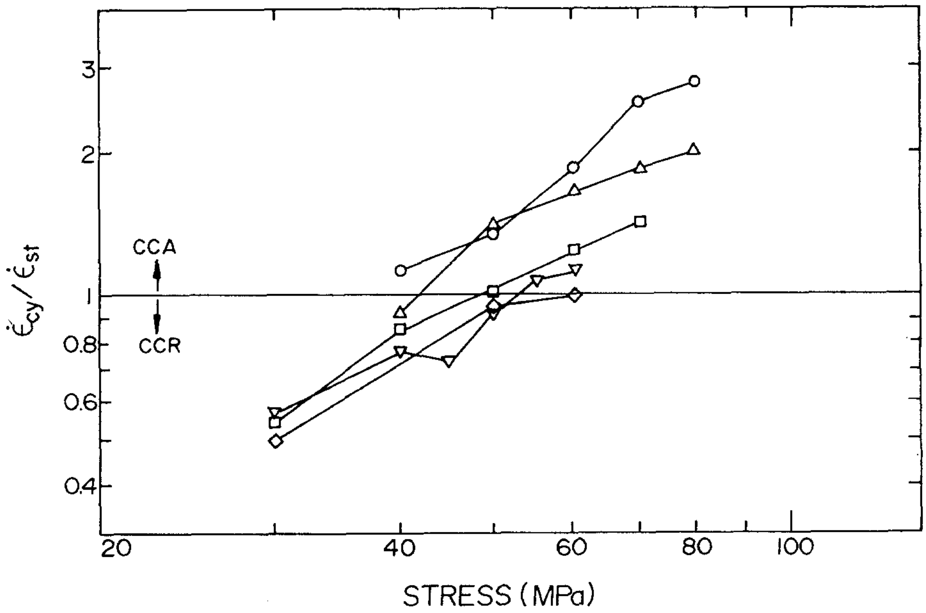

Figure 2 The ratio of cyclic to static creep rate plotted against peak stress at various temperatures for pure copper. (○) 550 K, (△) 580 K, (□) 610 K, (▽) 640 K, (◇) 670 K.

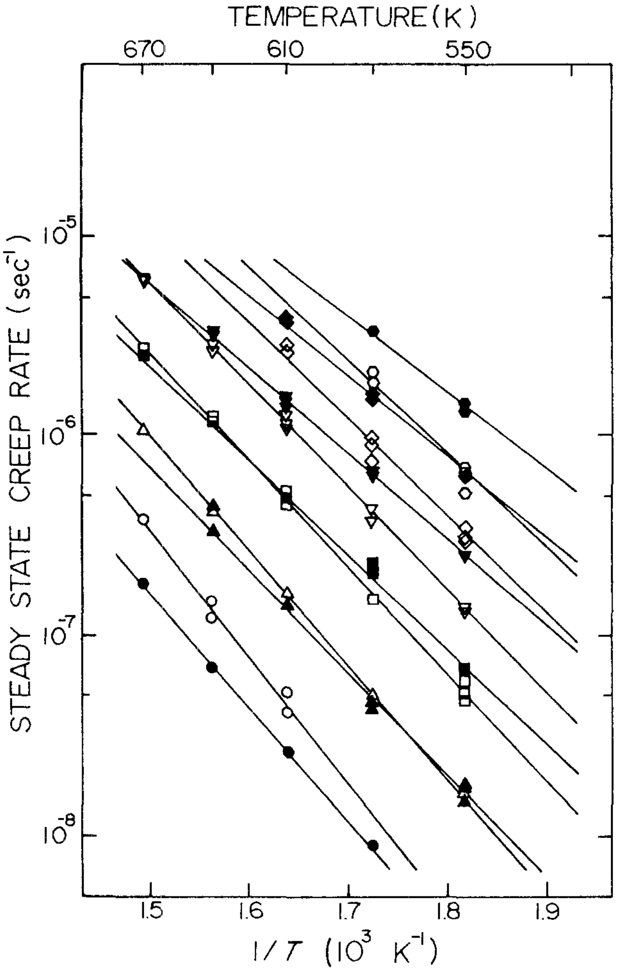

Figure 3 Steady state static and cyclic creep rates under various peak stresses plotted against inverse temperature for pure copper. From the slope of the line, creep activation energy can be determined.

Static	Cyclic (3 c.p.m.)	σ (MPa)
○	●	30
△	▲	40
□	■	50
▽	▼	60
◇	◆	70
⬡	⬢	80

Fig. 3 with respect to the inverse temperature. The creep activation energy at a given peak stress and stressing mode can be determined from this plot and the results are illustrated in Fig. 4 as a function of stress. The static creep activation energy measured at 30 MPa is about 120 kJ mol^{-1}. This value is close to the expected pipe diffusion activation energy of pure copper [19]. However, as the stress increases, the value of static creep activation energy is found to decrease to about 90 kJ mol^{-1} at 80 MPa. The cyclic creep activation energy also shows a similar trend but the value at a given stress is smaller than that of static creep and the difference between them is increased with increasing stress. We will discuss this dependence of the activation energy on the peak stress and stressing mode in Section 4. In Fig. 3 the threshold temperature below which the CCA phenomenon can occur at a given peak stress can be found. This threshold temperature is found to increase with increasing peak stress.

With the above experimental results, some significant characteristics of cyclic stress effects on the creep deformation of pure copper can be summarized: (1) cyclic stress increases the stress exponent of creep rate; (2) cyclic stress decreases the creep activation energy; (3) the threshold stress for the CCA phenomenon is increased with decreasing temperature and the threshold temperature for the CCA phenomenon is increased with increasing peak stress.

From these characteristic cyclic stress effects, it can be said that the CCA is a prevailing phenomenon under conditions of rather lower temperature and higher stress.

3.2. Observation of dislocation structure

Detailed study of the dislocation structure was performed using transmission electron microscopy. The purpose of this TEM work was to find the differences between the dislocation configurations developed under the different peak stress and stressing modes. The general feature of dislocation structure shows typical equiaxed cell structure. Figs 5a and b

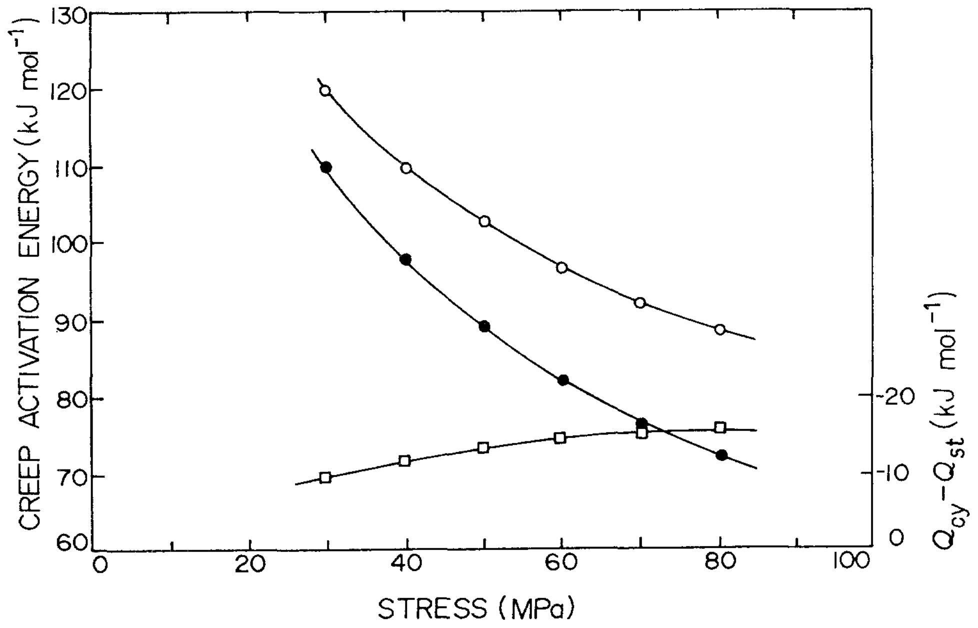

Figure 4 Dependence of the static and cyclic creep activation energy and their difference $(Q_{cy} - Q_{st})$ on the peak stress, for pure copper. (○) Q_{static}, (●) Q_{cyclic}, (□) $Q_{cy} - Q_{st}$.

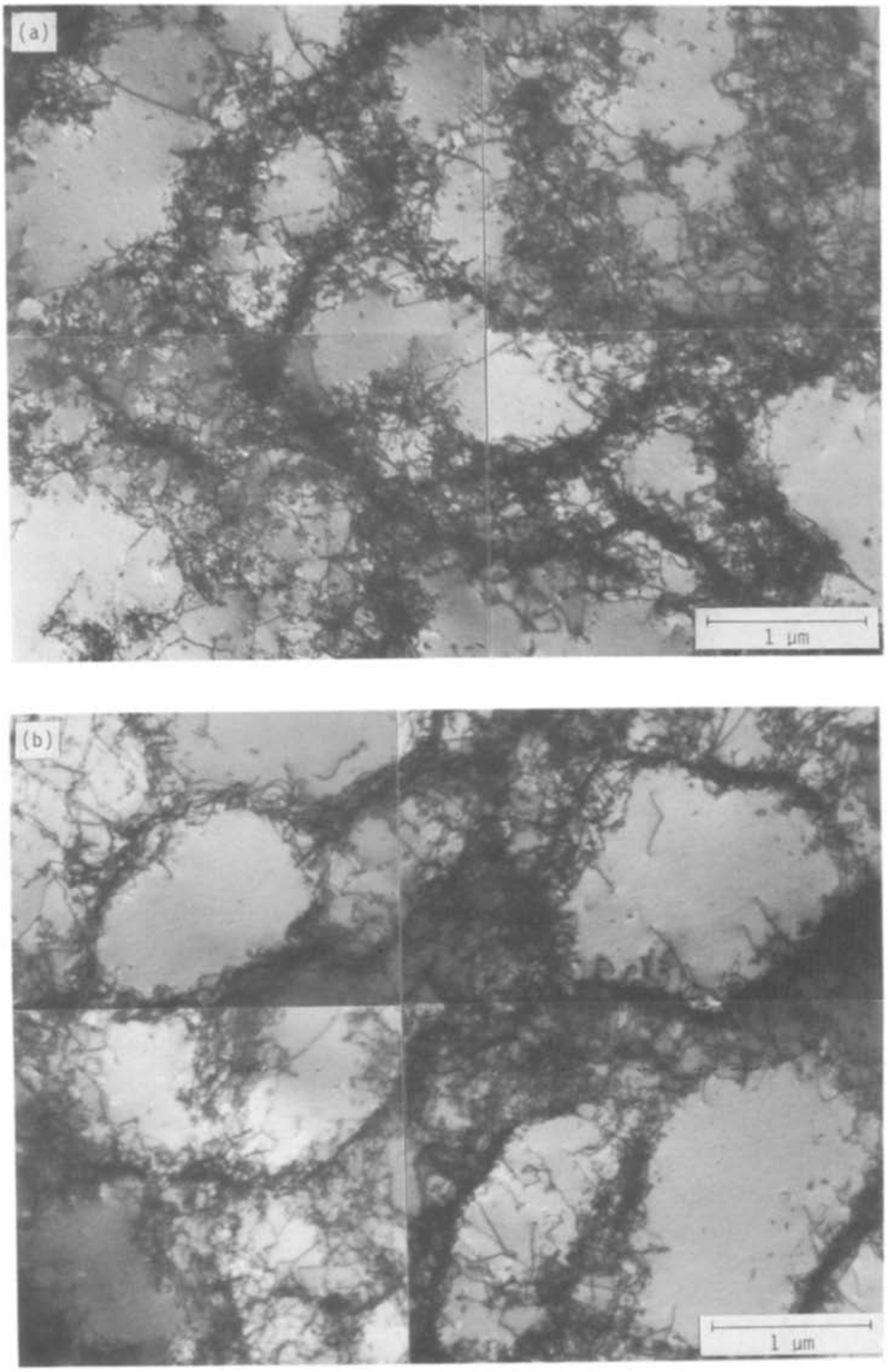

Figure 5 Typical transmission electron micrographs of dislocation structures developed during (a) static creep and (b) cyclic creep under the peak stress of 80 MPa at 580 K.

are the micrographs of the dislocation structure developed under static and cyclic peak stress of 70 MPa at 580 K, respectively. The cell interior is relatively free of dislocation and is bounded by thick cell wall. The cell wall is formed by highly tangled dislocations. Small dislocation loops and dipoles are also found around the wall. Dislocation structures, developed under the smaller stress condition (peak stress of 40 MPa at 580 K), are shown in Figs 6a and b for static and cyclic creep, respectively. It is seen that similar dislocation structures, but rather broad and incomplete cell walls, are developed under this lower stress condition. For a quantitative comparison of dislocation structures developed under the different peak stresses and stressing modes, representative micrographs for a given condition were taken. With these micrographs, the average cell size at a given condition was determined by a line intercept method. Over 100 cells for each condition were used to determine the average cell size. The results are plotted in Fig. 7 as a function of peak stress and stressing mode. From these results, it can be found that the average cell size is decreased with increasing peak stress but that of cyclic creep has larger size and smaller stress dependence than that of static creep at all stress ranges of this work. Generally, the cell size is represented by the following relation

$$d = Kb(G/\sigma)^p \qquad (2)$$

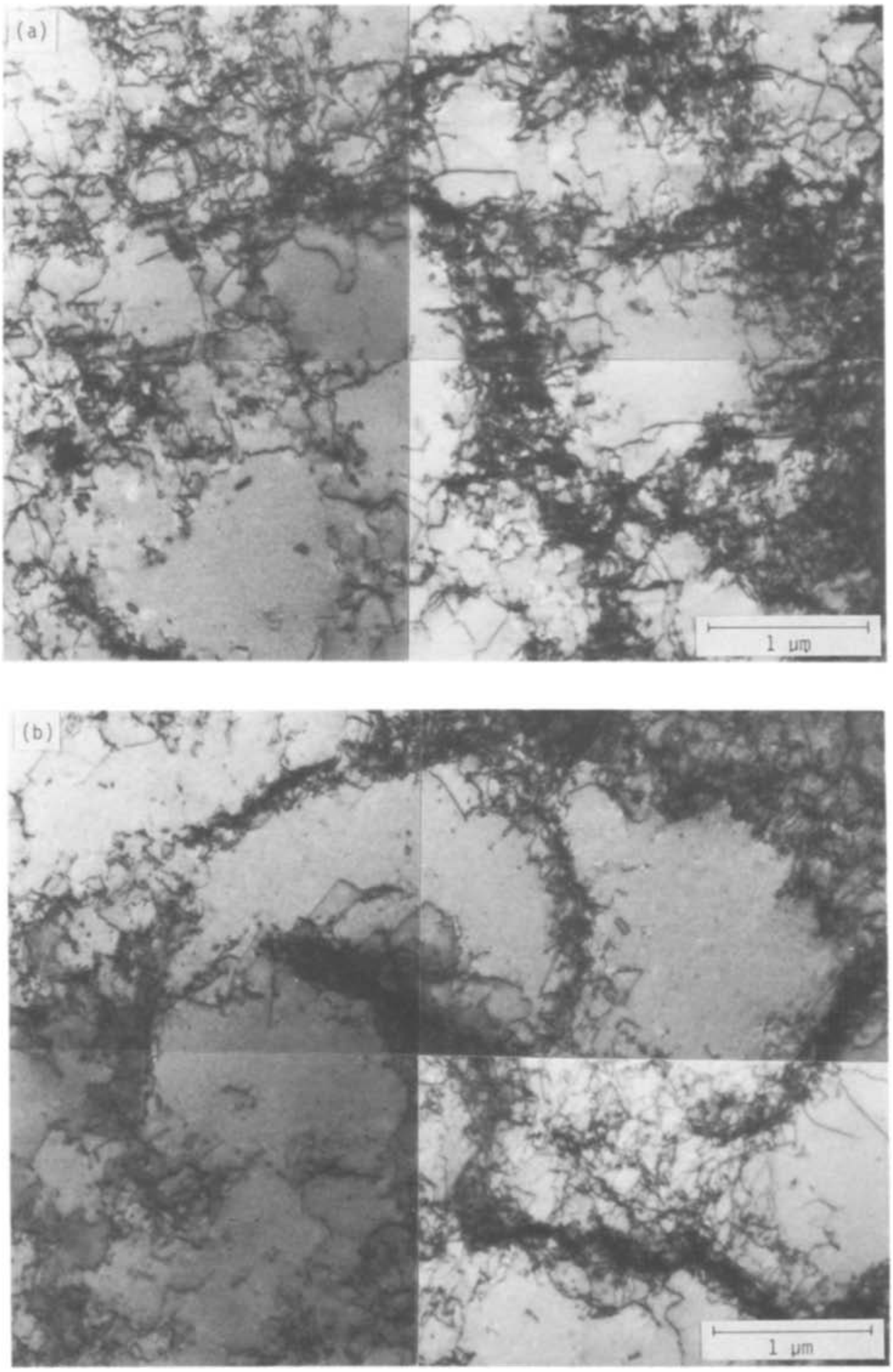

Figure 6 Typical transmission electron micrographs of dislocation structures developed during (a) static creep and (b) cyclic creep under the peak stress of 40 MPa at 580 K.

where d is the cell size, $\boldsymbol{b}$ is Burgers vector, G is the shear modulus, and K and exponent p are material constants. Most of the experimental results [20–22] and theoretical models [20, 23] show that the exponent p is close to unity and the constant K is about 20. From Fig. 7, the exponent p and constant K for static creep are calculated by the least square method to be about 0.9 and 15, respectively. These values are in good agreement with the results discussed above [20–23]. Similarly, the values of p and K for cyclic creep were found to be about 0.57 and 150, respectively. These results are quite different from those of static creep. Considering the morphology of dislocation structure, we think that these different values may arise from the effect of enhanced recovery process of the cell wall under cyclic stressing and are strongly correlated with the CCA phenomenon. These effects of stressing mode on the dislocation structure will be discussed in Section 4.

4. Discussion

As discussed previously, cyclic creep acceleration occurred at rather lower temperatures and in the higher stress range. Before beginning the discussion of the CCA mechanism, it may be helpful to consider the regular static creep deformation mechanism at that condition. It is well accepted that the high-temperature dislocation creep of pure metal is controlled by

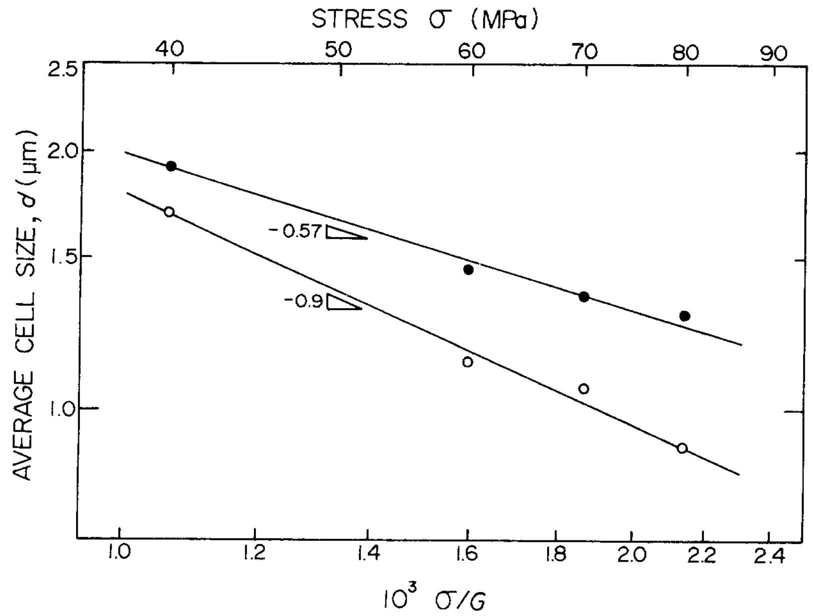

Figure 7 Dependence of the cell size on the peak stress and stressing mode, for pure copper. (○) Static, (●) cyclic, $T = 580\,\mathrm{K}$, $b = 2.56\,\mathrm{A}$, $G_{580} = 3.74 \times 10^4\,\mathrm{MPa}$. $d = Kb(G/\sigma)^p$.

dislocation climb through lattice diffusion, and the good agreement between the creep activation energy and the lattice diffusion activation energy supports this model. However, as the temperature is lowered below about half of the absolute melting temperature, it is generally observed that the creep activation energy is decreased and the stress exponent is increased. This characteristic behaviour of low-temperature creep means that another deformation process, whose activation energy is lower than that of lattice diffusion, operates in parallel to the high-temperature creep mechanism. For this creep deformation mechanism of the lower temperature region, the pipe diffusion assisted dislocation climb model is widely accepted as the possible rate controlling process [24–27].

The theories which explain the cyclic creep behaviour can be classified into two groups. Shetty and Meshii [4], Evans and Parkins [8], and Bennett and Evans [10] have suggested a cross-slip based model. In this suggestion, the piled up dislocations of screw character at the obstacle can be overcome by the cross-slip process during the off-load period of the stress cycle and easily glide away from the obstacle due to the next on-loading part of the stress cycle. Recently, Lorenzo and Laird [11] gave support to this model with their results of microstructural investigation.

Another suggestion is the point defect based model. Kennedy [1] found CCA in lead, and interpreted that phenomenon as the accelerated recovery of dislocation obstacles with mechanically produced excess vacancies. Feltner [3] interpreted CCA and CCR as the role of interaction of excess vacancies and moving dislocations. Bradley *et al.* [6], Jin and Nam [12], Konig and Blum [28], and Lee *et al.* [29] have found softening of dislocation substructure and an increase of effective stress under cyclic stressing, and accepted the point defect model as the possible mechanism of substructure softening. At this point, assuming that the rate controlling process of cyclic creep deformation is the same as that of static creep, i.e. pipe diffusion assisted climb of dislocation, the point defect model can be regarded as the more reasonable mechanism for CCA.

As shown in Fig. 4, cyclic stressing reduces the activation energy of creep deformation more effectively than the static stressing does, and both the static and cyclic creep activation energies in the higher stress region are smaller than the expected pipe diffusion activation energy of pure copper ($120\,\mathrm{kJ\,mol^{-1}}$) [19]. A similar result was observed in pure aluminium by Choi *et al.* [30]. This decrease in creep activation energy below the activation energy of pipe diffusion cannot be explained by the simple pipe diffusion concept. It has been suggested previously that the apparent creep activation energy, Q_c, can be reduced by athermal work according to the following relation [6, 12, 31]

$$Q_c = Q_d - W \qquad (3)$$

where Q_d is activation energy for diffusional process and W is the athermal work done by external stress. We think that some portion of athermal work appears in the form of excess vacancies and these reduce the activation energy barrier of the pipe diffusion process. The larger decrease of cyclic creep activation energy implies that more athermal work is done or a larger number of vacancies is generated athermally during cyclic creep deformation. There is much experimental evidence for the generation of excess vacancies with plastic deformation [32–35]. Recently, Shin *et al.* [36] found the existence of a higher vacancy concentration in a cyclically crept pure nickel than in a statically crept one.

Assuming that the deformation mechanism of cyclic creep is basically the same as that of static creep, i.e. pipe diffusion controlled recovery, the CCA mechanism is suggested on the basis of enhanced recovery of dislocation substructure with the help of excess vacancies. The athermal excess vacancies are known to be generated by the nonconservative motion of jog [37–40]. As the deformation proceeds, these vacancies are accumulated in the cell interior and should diffuse to the cell wall at which the vacancies are required to make dislocation climb. This athermal vacancy flux may reduce the activation energy for vacancy formation and, as a result, the climb rate will be enhanced. This effect of excess vacancy on the creep rate was previously suggested by Stüwe [39] and discussed by many researchers [32–36]. As it is known that the number of mechanically generated excess vacancies depends on the amount of plastic deformation [37–40], a higher concentration of excess vacancies may be obtained [31, 35, 36] with larger accumulated strain under cyclic stressing. As a result, the recovery of the cell wall can be enhanced more effectively than under static stress. The decrease in apparent creep activation energy with increasing peak stress and applying cyclic stress strongly supports the suggestion of the role of athermal vacancy for the reduction of activation energy barrier of diffusion. If the creep temperature is high or the stress is low enough, the concentration of mechanically produced vacancies may be decreased below the thermal equilibrium vacancy concentration. Therefore, under this condition, the CCA phenomenon will not occur. From the microstructural investigation of this work, it was found that a coarser cell structure was developed during cyclic creep than that of static creep and the size difference between them become larger with increasing peak stress. These results are thought to be evidence for the enhanced recovery of the cell wall with excess vacancies generated under cyclic stressing, because the cell wall cannot be recovered without a diffusional process. Previously, similar microstructural investigations have been performed for pure aluminium by Konig and Blum [28] and Lee *et al.* [29]. They found that the dislocation substructure of a cyclically crept specimen has more recovered boundary configurations and concluded that the effect of applying cyclic stress is similar to that of increasing temperature. As it is widely accepted that the cell wall is one of the most important barriers to dislocation motion in pure metal, and the internal back stress is induced by that cell wall [41], it may be said that the higher effective stress observed in cyclic creep [6, 12, 24, 31] may be due to the coarser cell structure. The effect of cell size on creep rate has been suggested by Robinson and Sherby [42] and Sherby *et al.* [43] as the constant structure creep model. They expressed this model as follows

$$\dot{\varepsilon} \propto d^q \sigma^m \tag{4}$$

Sherby *et al.* [43] have suggested the exponents q and m to be 3 and 8, respectively. For normal static creep, the cell size has the inverse stress dependence and their Equation 9 can be reduced to fifth power of stress as experimentally observed in most pure metals in the lower temperature region. Inserting the experimentally observed stress dependences of cell size of static and cyclic creep into Equation 4, one can easily find that the resultant stress exponents of static and cyclic creep become about 5.3 and 6.3, respectively, and these values are close to the experimentally observed stress exponents in this work (see Table I).

5. Conclusions

1. The effect of cyclic stress on enhancing the creep rate is found to become more significant with increasing peak stress and decreasing test temperature. Therefore, CCA is thought to be the prevailing phenomenon under conditions of higher stress and lower temperature.

2. A larger stress exponent and smaller creep activation energy are observed in cyclic creep in the stress and temperature range of this work. This implies that a larger amount of athermal work is done effectively by the cyclic stressing than the static stressing can do.

3. The dislocation structures show typical cell structures but the cell size developed under cyclic stress is larger and has a lower stress dependence than that of static creep. These results are considered to be evidence for the enhanced recovery of the cell wall with the help of athermally generated excess vacancies.

4. From these macroscopic and microscopic experimental results, it is concluded that the CCA phenomenon occurs when the athermal vacancies, generated by cyclic stress, can play a dominant role in the dislocation climb process required for the recovery of the cell wall.

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