ELSEVIER

Materials Science and Engineering A345 (2003) 215–222

MATERIALS SCIENCE & ENGINEERING A

www.elsevier.com/locate/msea

The observation of dislocation reversal in front of crack tips of polycrystalline copper after reducing maximum load

H.L. Huang [a,*], N.J. Ho [b]

[a] *Department of Mechanical Engineering, Chinese Military Academy, Fungshan 830, Taiwan, ROC*
[b] *Institute of Materials Science and Engineering, National Sun Yat-sen University, Kaohsiung 812, Taiwan, ROC*

Received 28 January 2002; received in revised form 18 June 2002; accepted 25 June 2002

Abstract

The loading at a crack tip varies during fatigue crack propagation. As a result, overloading causes retardation of crack propagation, and underloading causes the acceleration of crack propagation. In addition, reducing the load range by changing either the minimum load or maximum load can cause a reduction or retardation of crack propagation to occur. Examining a fatigue cracked specimen made of polycrystalline copper with back scattered electron images (BEI) in a scanning electron microscope (SEM) revealed that (1) the dislocation structures close to the crack tips gradually evolved from a cell structure into a new loop patch structure during the crack retardation period which follows after reducing the maximum load; (2) restoring the crack propagation rate is a result of re-establishing the cell structure from new loop patches or PSBs; and (3) the evolution of the dislocation structure at the crack tip due to the maximum loading reduction is affected by residual active slip systems.

Keywords: Crack propagation; Dislocation evolution; Fatigue

1. Introduction

It is well known that both crack initiation and crack propagation during fatigue are the results of dislocation interactions in the material [1]. For crack initiation, the persistent slip bands (PSBs) accompanying a ladder-like wall dislocation structure embedded in the PSBs [2–4] develop from trapping of edge dislocations. Further interactions between the PSBs and the grain boundaries (PSB-GB) accompanying the dislocation cell structure [5,6] develop more dislocations generated from different activated slip systems. For crack propagation in front of the crack tip, usually a wide region of cell structure [7–11] develops at a higher rate of crack propagation. The dislocation morphology ahead of the crack tip is cellular in a small region at a low crack propagation rate, but then followed by ladder-like walls of the PSBs, veins and loop patches [7]. The cell size in front of the tip of a propagating a crack cannot be larger than 0.7 μm.

It is also well known that a sudden increase of the peak load followed by cycling at normal loading conditions, or dropping the peak load to the so called crack closure or opening level for the subsequent cycling, will delay subsequent crack propagation. Accelerating the crack propagation rate can be achieved by underloading for one time followed by cycling at the normal loading conditions. The retardation phenomenon is commonly explained by a compressive residual stress in the region ahead of the crack tip, resulting in crack tip closure, due to the large plastic strain induced from a single overloading [12–19]. In the case of an underload, the crack propagation will be accelerated [12,18] by a tensile residual stress in the region ahead of the crack tips to partially annihilate the compressive residual stress built up by the positive load. Nevertheless, the results of Huang et al. [12] indicate that the crack passes through the cell structure into vein or loop patch structures when the crack is subjected to a single peak overload. The period of retardation time can be considered as the cycles needed re-development of dislocation cell structure to the size smaller than 0.7 μm in front of the crack tip. And the region of

* Corresponding author. Tel.: +886-7-747-9510; fax: +886-7-747-9515
E-mail address: hlhuang@cc.cma..edu.tw (H.L. Huang).

0921-5093/02/$ - see front matter
PII: S0921-5093(02)00472-0

dislocation cell structure in front of the crack tip is enlarged and more fully evolved than the dislocation structure before underloading when the crack propagation rate is subjected to a peak underloading. At the same time, it is also reported [7] that the propagation rate seemed to be proportional to the degree of extension of the area that dislocation cells occupied at the crack tip.

In addition to the above loading conditions, it is important to investigate the dislocation morphology at the crack tip while the peak load changes from high to low for crack propagation (CT specimen) in fatigue, because the propagation rate can be decreased to a level quite similar to an overloading condition. According to Laird [20], the dislocations evolve from cells into loop patches when the amplitude of loading changes from high to low during low cycle fatigue. It is difficult to determine which grain in the sample is with the fastest or the slowest dislocation development. At the same time, Huang et al. [7] indicates that the dislocation morphology varies in front of the crack tip when the rate of the crack propagation varies. Therefore, in order to reveal the dislocation evolution under reducing the maximum load condition during fatigue, this study used the single compact tension specimen of polycrystalline copper to investigate the crack propagation until the rate of crack propagation reached a specific steady state. Finally, the back scattered electron image (BEI) of scanning electron microscopy (SEM, JEOL 6400) was employed to observe the microstructure in front of the crack tips.

2. Experimental

A plate of high purity polycrystalline copper (99.95%) was used in this study. The specimens were annealed at 850 °C for 2 h in a vacuum of 10^{-5} torr and then cooled in the furnace. The grain sizes of the specimens were about 100–120 μm. The preparation of specimens followed the instruction of ASTM E647 for a single edge compact tension specimen. The crack propagation was conducted by a computerized Instron 1332 hydraulic testing machine at $R = 0.1$ (stress ratio, $R = P_{min}/P_{max}$) at a frequency of about 20 Hz. During crack propagation at each step, the cracks increased by a length of 0.254 mm with the effective load reduced by 5–8% of the previous step. The crack length was measured by a traveling microscope to an accuracy of ±0.01 mm. For each specimen, when the crack reached a specific steady propagation rate, the peak load was then reduced to a small value. These specimens were cycled subsequently to a different number of cycles until re-propagation of the crack occurred. A schematic diagram of Fig. 1 showed the time intervals required for the specimens to be sliced for microstructure observation. The stress intensity (ΔK) was determined from ASTM E647 as:

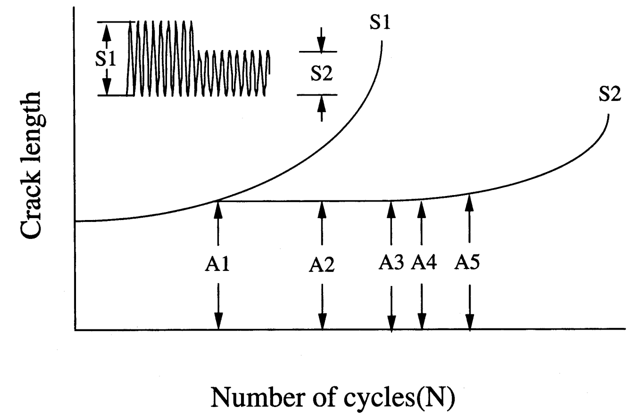


Fig. 1. Schematic diagram of the loading condition to produce retardation.

$$\Delta K = \Delta P/(B \times w^{0.5})(a/w)$$

where $(a/w) = [(2 + a/w)(0.866 + 4.72(a/w) - 5.56(a/w)^2 + 13.32(a/w)^3 - 14.72(a/w)^4)/(1 - a/w)^{1.5}$, B is thickness, a is crack length, w is effective width, and ΔP is range of loading, $\Delta P = P_{max} - P_{min}$.

To observe the dislocation evolution at the crack tip, the fatigue-cracked specimens were cut into tiny squares of 10 mm that contained a crack longer than 3 mm (as shown in Fig. 2(a)). The squares were then cut into slices of 0.6 mm thickness. The slices were then further ground to a thickness of 0.15–0.2 mm by grid paper and then punched into disks of 3 mm in diameter (as shown in Fig. 2(b)). These 3 mm disks were twin-jet polished using Struers D2 polishing solution at 8–10 V and −20 °C. A JEOL 6400 SEM was used to investigate the microstructures at the crack tip and the crack path by means of BEI [21]. The accelerating voltage was set at 25 kV, and the working distance was 8 mm.

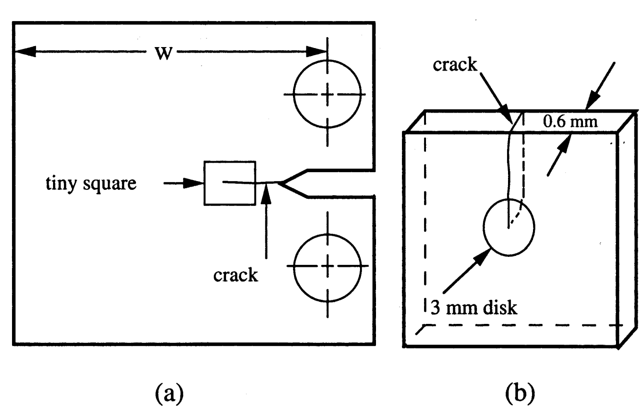


Fig. 2. Schematic diagrams of the foil preparation. (a) Position of tiny square. (b) Position of discs.

3. Results

The loading conditions versus their effects on the retardation of cracks which propagated at two steady propagation rates, 2.0×10^{-5} and 5.0×10^{-6} mm cycles^{-1}, respectively, were summarized in Tables 1 and 2. In sample A1, propagating at a rate of about 2.0×10^{-5} mm cycle^{-1} without peak load reduction, a very wide range of misorientation cells was developed in front of the crack tip, as shown in Fig. 3. The misorientation cells and the following ordinary cells almost occupied the whole grain containing the crack tip. Micrograph of the A2 specimen which was cycled to 3.5×10^5 cycles after peak load reduced from 320 to 112 Kgf, revealed that larger dislocation cells with the same contrast occupied most of the portion of the region in front of the crack tip (Fig. 4a). Further cycling to 5.5×10^5 cycles for A3, newly developed loop patches, shown in Fig. 4b, replaced the cells and misorientation cells in front of the crack tip and misorientation cells and cells could only be observed beyond the crack path. It was noted that no walls or PSBs could be seen in this stage. After cycling to 7.0×10^5 cycles for A4, the dislocation cells developing in large with a portion of walls, PSBs appeared in front of the crack tip again (Fig. 4c). When the crack propagation re-started for A5, the dislocation structures in front of the crack tip are low energy cells (Fig. 4d). A clear picture can be drawn to show the evolution stages of the dislocation structure. In the first stage, misorientation cells changing to larger cells, loop patches; in the second stage, loop patches gradually develop into walls, PSBs and then into cells.

In order to investigate the dislocation morphology changes at near threshold condition, another fatigued sample, B1 was cycled at 230 Kgf peak load with a propagation rate of 5×10^{-6} mm cycles^{-1}. The dislocation morphologies at the crack tip were a sequence of misorientation cells and cells occupied a very limited area where walls, multiple PSBs, veins and loop patches were followed and occupied a larger area, as shown in Fig. 5 [7]. Cycling to 3.0×10^5 cycles after reducing the peak load from 230 to 100 Kgf for sample B2, newly developed loop patches replaced most of the cells and multiple PSBs ahead of crack tip where the region of cells and PSBs reduced to a very small portion (shown in Fig. 6a). In Fig. 6b, the development of veins from loop patches can be seen in the sample B3 that was cycled to 6.0×10^5 cycles. It was also noted that no walls or PSBs could be found in this stage. Re-appearance of cells ahead of the crack tip could be seen in Fig. 6c for the sample B4 that was cycled to 9.0×10^5 cycles after load reduction.

Table 1
The loading condition to produce retardation of a crack propagating from the propagation rate of 2.0×10^{-5} mm cycle^{-1}

Sample	Crack length (mm)	da/dN (mm cycles^{-1})	Cycles[a]	Peak load (Kgf)
A1	15	2×10^{-5}	0	320
A2	15	–	3.5×10^5	112
A3	15	–	5.5×10^5	112
A4	15	–	7.0×10^5	112
A5	15.05	Re-propagation	7.6×10^5	112

[a] Cycles accumulated after load reduction.

Table 2
The loading condition to produce retardation of a crack propagating from the propagation rate of 5.0×10^{-6} mm cycle^{-1}

Sample	Crack length (mm)	da/dN (mm cycles^{-1})	Cycles[a]	Peak load (Kgf)
B1	15	5×10^6	0	230
B2	15	–	3.5×10^5	100
B3	15	–	6.0×10^5	100
B4	15	–	9.4×10^5	100

[a] Cycles accumulated after load reduction.

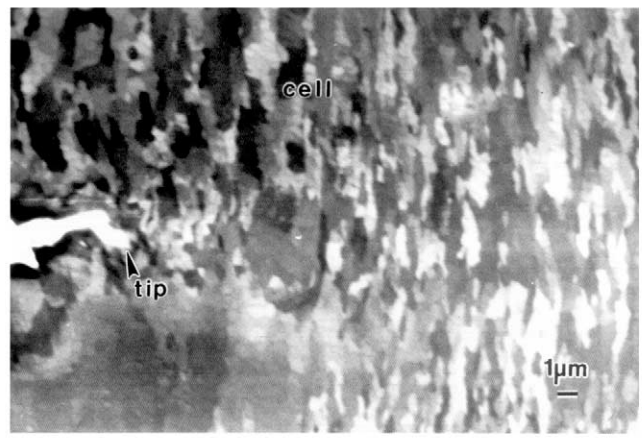


Fig. 3. A wide range of misorientation cells developed in front of the crack tip at a crack propagation rate of 2.0×10^{-5} mm cycle^{-1} and peak load of 320 Kgf.

4. Discussion

It was reported that the retardation of the crack propagation from overload is a result of the crack passing through cells into the region of loop patch structure which would need substantial cycles in order to accumulate dislocations to develop cells according to the result of low cycle fatigue of copper [12]. The retardation was explained by many researchers [13–19] that the reversed residual stresses (compressive stresses) were built up after overload to suppress the tensile stress in the region (commonly called the reversed plastic zone) ahead of the crack tip. After many cycles, the crack tip slowly advanced away from the region of high compressive residual stresses to the region of low compres-

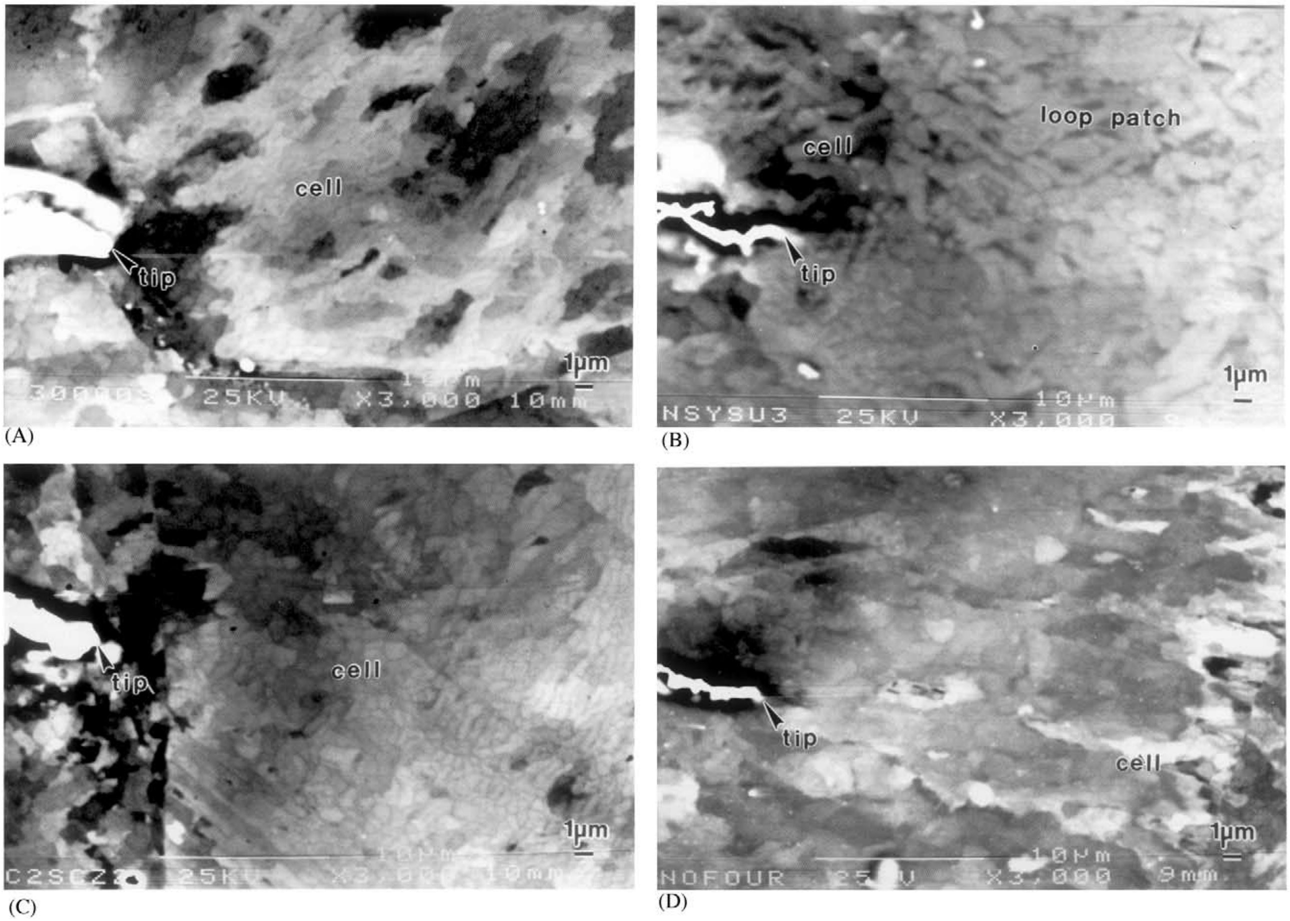


Fig. 4. (a) Misorientation cells collapsed into larger cells after 3.0×10^5 cycles after peak load dropping from 320 to 112 Kgf. (b) Cells vanished and new loop patches appeared without a sign of 2-D walls, in front of the crack tip at 4.0×10^5 cycles after peak load dropping from 320 to 112 Kgf. (c) New cells developed from new walls and PSBs in front of the crack tip at 7.0×10^5 cycles after peak load dropping from 320 to 112 Kgf. (d) Cells and misorientation cells restored at 9×10^5 cycles after peak load dropping from 320 to 112 Kgf, where the crack tends to re-propagate again.

sive residual stress to re-obtain the propagation. As for the condition of reducing the peak load, there should not have been any change of reversed compressive residual stress except the changes of the level of tensile stress in front of the crack tip. It was commonly considered that less driving force (effective ΔK) or less accumulated plastic strain for the crack to propagate was below the original critical value so that the crack stopped propagating. The argument that should reflect the dislocation structures developed in front of the crack tip. It is implied that they should remain unchanged as before, until the re-accumulation of strain energy or plastic strain reaches the original critical value. Any changes of dislocation in front of the crack tip under peak load reduction should be the reduction of mobile dislocations within cells or walls, but not the cells or walls themselves. Nevertheless, the facts shown in Fig. 4a and Fig. 6a were that cells could not hold their forms in the low energy state, while further cycling for a certain period of time after the peak load dropped to a low level. They were gradually transformed back into loop patches, veins or walls, depending on the amount of load reduction. This kind of reverse evolution of dislocation structures from saturated cells with low energy at high strain into loop patches at low strain at the crack tip demonstrates the metastability of dislocation structure in a variable deformation process and is worthwhile for detailed discussion.

It is clearly known that, in low cycle fatigue test, edge dislocation trapping, loop patches and veins develop, while only one primary slip system is activated; ladders in PSBs and walls develop, while a secondary slip is activated by higher back stress which is proportional to the accumulation of dislocations; 2-D cells, cells and misorientation cells develop, while more than three slip systems are activated gradually by accumulated plastic strains. Formation of dislocation cells is a result of multiple slip systems regardless of whether the amplitude of the plastic strain is low or high. The multiple slip systems operate one after another in the region of low plastic strain and operate simultaneously in the region of high plastic strain. Cells usually develop much faster at lower plastic strain in low cycle fatigue test than in a monotonic test. This result is caused by forward and

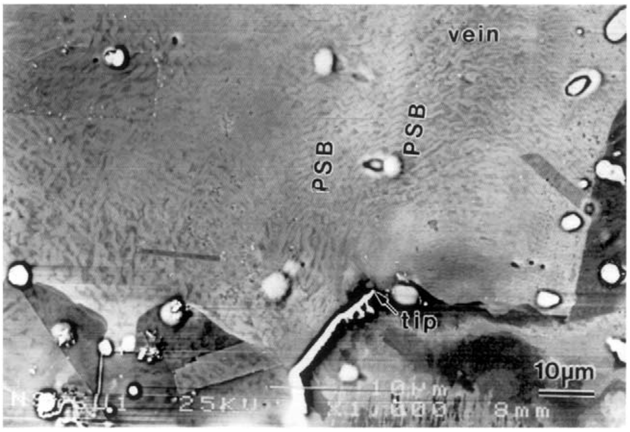
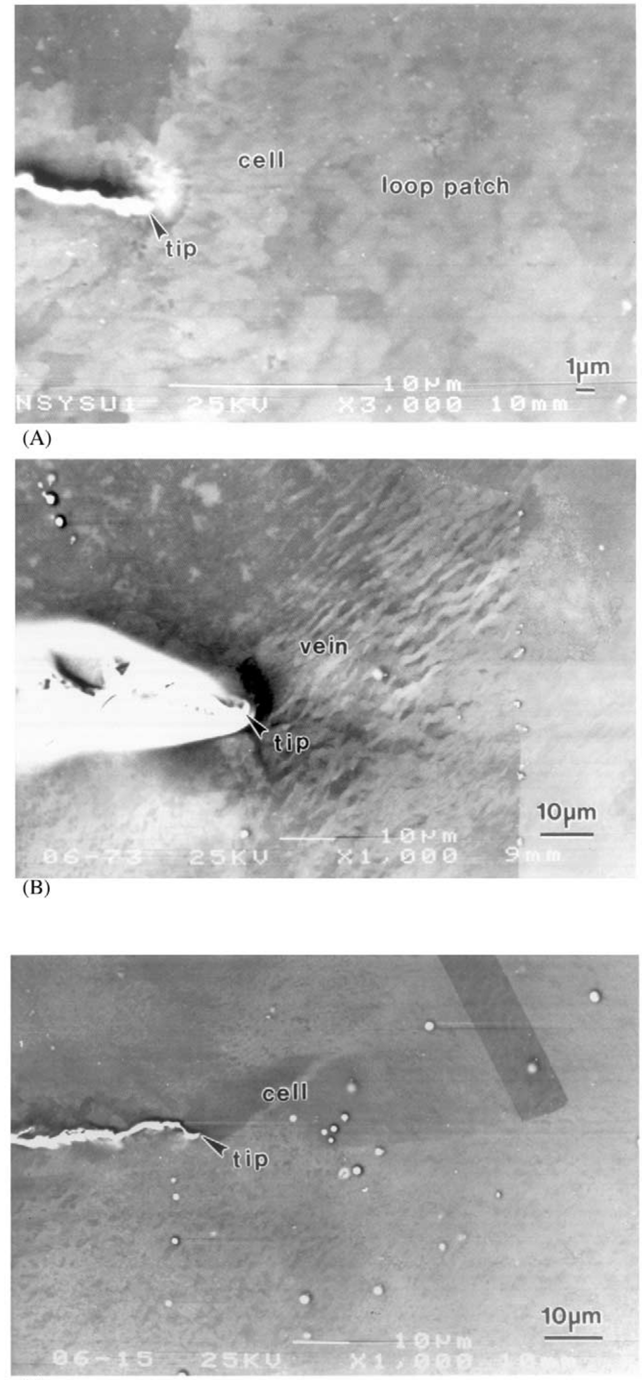


Fig. 5. Full evolution of cells with limited range, walls, veins and loop patches can be seen in front of the crack tip at a propagation rate of 5×10^{-6} mm cycles^{-1} and 230 Kgf peak load.

back motion of dislocations in slip systems, and also the forward and back operation of participating slip systems.

Morphologies of dislocation structures [7–11] in front of the fatigue crack tip were reported to be similar to those found in the low cycle fatigue test, as well as the mechanisms. They vary along the stress/strain distribution ahead of the crack tip. Owing to the uneven stress/strain distribution in tension and in compression around the crack tip, activated slip systems are more in tension than in compression. The operating stress (net stress) to the slip system is composed of applied stress from outside and back stress from entangled dislocations, as illustrated in Fig. 7. During fatigue crack propagation, upon loading to the peak value, the operating stress at crack tip is high enough in tension to activate many slip systems, and to propagate the crack by retaining dislocation structures into the saturation state of low energy cells. Upon unloading to a minimum value, the operating stress in reverse direction is, by no means, compatible with its tension counterpart, but enough to hold the morphology. Otherwise the stability of the dislocation morphology cannot be kept. When the peak load decreases, the operating stress decreases with a consequence of reducing the number of slip systems in tension and in compression. Once the operating slip systems in tension and/or in compression drop to cause less than two slip systems to operate, the back stress should be strong enough to maintain the cell formation; therefore, the observation of cells in an electron microscope can be possible. Otherwise, the residual active slip systems should play some kind of important role in altering the cell morphology.

In this study, the prior fatigued samples further fatigued to 3.5×10^5 cycles with the peak loads of two different values dropping to 112 Kgf, a value just above the threshold value. The loop patches (Fig. 4b) and veins (Fig. 6b) appeared in the prior cell regions to demonstrate that no more than two slip systems can operate at the moment. The following argument will illustrate how a few slip systems develop loop patches or veins from cells.



(A)

(B)

(C)

Fig. 6. (a) Cells collapsed, walls or PSBs vanished and new loop patches developed at 3.0×10^5 cycles after peak load dropping from 230 to 110 Kgf. (b) Only newly developed veins and hardly cells can be seen at 6.0×10^5 cycles after peak load dropping from 230 to 110 Kgf. (c) Re-appearance of cells developed from new slip systems can be seen in front of the crack tip at 9.0×10^5 cycles after peak load dropping from 230 to 110 Kgf.

It can be seen in Fig. 8, taken from another sample at 3.0×10^5 cycles after load reduction that the slip systems rotate the original structures toward the opposite direction and result in transforming the misorientation on cell structures (Fig. 4(a)), or broadening the cell walls in one direction then another direction followed. Retrapping of edge dislocations and/or dipoles occurred to develop the loop patches (Fig. 7(b)).

To demonstrate the broadening of the cell walls, a smooth specimen was tested in low cycle fatigue at 0.3% strain amplitude for 500 cycles to ensure the formation of cells, and then further fatigued at lower strain amplitude of 0.1% up to 3.0×10^5 or 8.05×10^5 cycles. It is clear that the cells walls were broadened (Fig. 9(a), 8.05×10^5 cycles) and the Labyrinth structure was broadened along one direction (Fig. 9(b), 3.0×10^5 cycles).

The schematic diagram shown in Fig. 10 is provided to illustrate that once the loading is reduced the slip systems in operation in repeated loading are also reduced to one. Pairs of edge dislocations of opposite sign are generated by each forward and back movement of screw dislocations moving inside the cell. These balance the back stress to hold the part of the wall that traps them. However, when these moving dislocations encounter the other parts of the walls, the balance of the cell walls gradually breaks to release trapped dipoles or to form new dipoles by cross-slip. Then, broadening of the cell walls occurs. Later on, most of the cell walls become similar to clustering of dipoles in Taylor lattice form. As the cycling continues, re-trapping of dipoles to form new loop patches and vein structures can be expected with the traditional mechanisms.

According to Huang et al. [7], whether the rate of the crack propagation is low or high, it is necessary that the dislocation morphology in front of the crack tip consist of low energy cells in order to propagate a crack for polycrystalline copper. Hence, furthering cycling is needed for the dislocation structure to continuously evolve into low energy cells. In that regard, an additional 7.0×10^5 cycles were needed for a few active slip systems to accumulate the plastic strain and dislocations to re-establish dislocation cells (Fig. 3(c)) and to propagate the crack again after peak load reduction. The requirement of these additional cycles indicates the source of crack retardation after a peak load reduction.

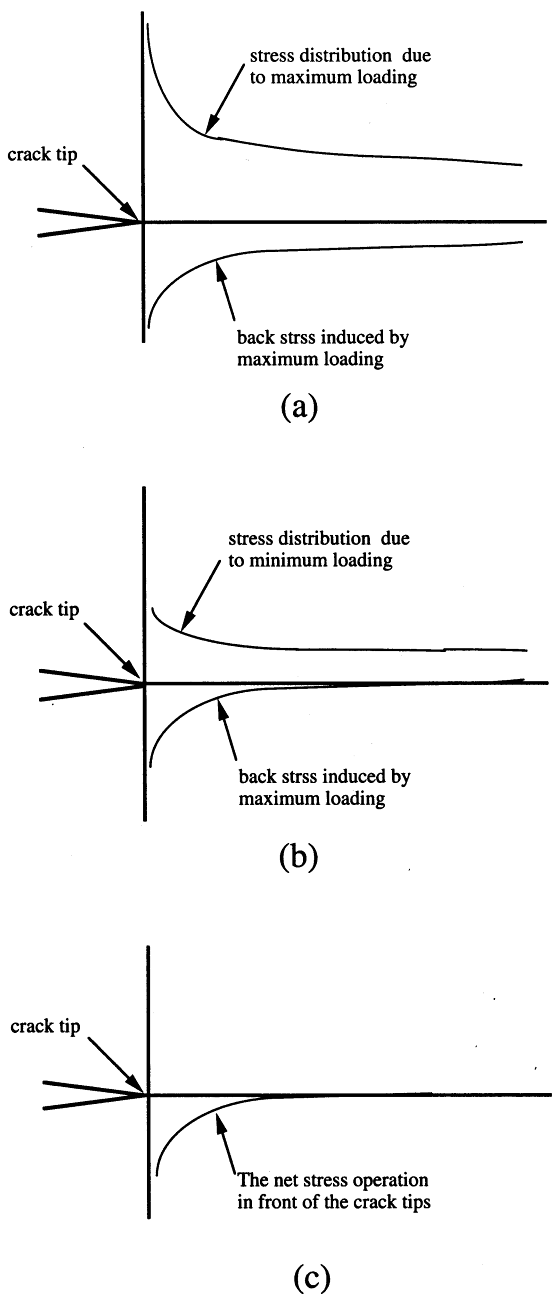


Fig. 7. Schematic diagrams show the stress distribution at the crack tip. (a) Steady state maximum loading. (b) Reduced the maximum loading. (c) The net stress after the reduced loading.

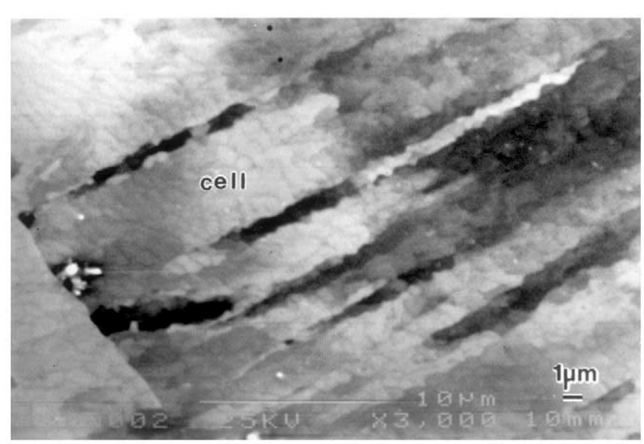


Fig. 8. The misorientation cells possessing one direction in the sample cycled about 3.0×10^5 cycles after maximum loading reduce from 320 to 112 Kgf.

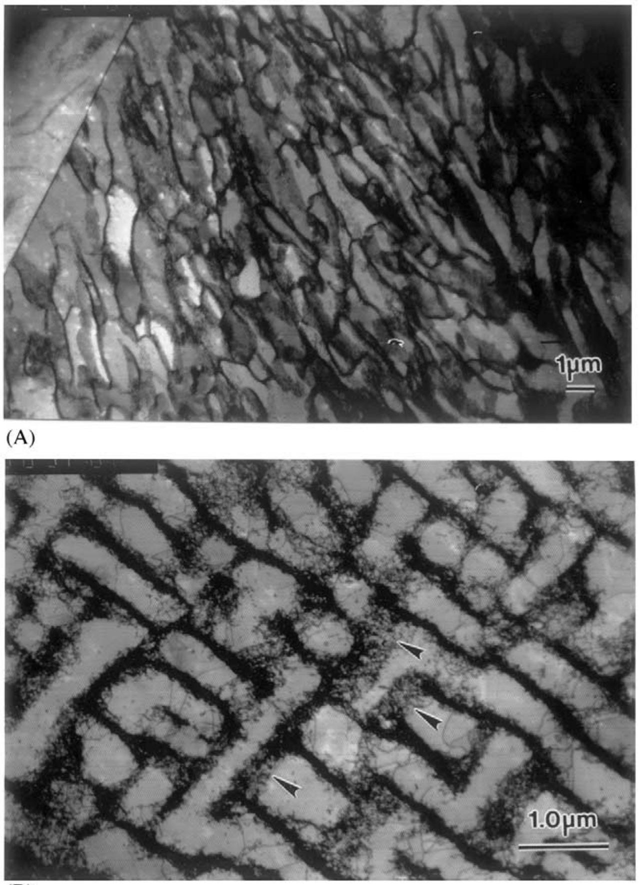


Fig. 9. (a) The dislocation cell structure with the broadened cell walls at 8.05×10^5 cycles after the strain amplitude reduced from 0.3 into 0.1%. (b) The dislocation Labyrinth structure with the broadened cell wall at 3.05×10^5 cycles after the strain amplitude reduces from 0.3 into 0.1%.

5. Conclusions

A peak load drop was employed to a propagating crack to change the crack propagation rate. Conclusions about the dislocation evolution in front of the crack tip from retardation to re-propagation can be summarized as follows.

1. Development of new dislocation loop patches, veins and PSBs occurred in the prior region that is occupied by misorientation cells (low energy cells) or ordinary dislocation cells while the crack is subject to further cycling under the condition of peak load dropping to a value near threshold condition.

2. Broadening of the cell walls to leave space for development of new loop patches is a result of a decrease in the number of slip systems to a low level after peak load reduction from multiple slip systems before peak load reduction.

3. Additional cycles are needed to resume the crack propagation after the peak load drop; the additional cycles are needed to transform the old low energy cells to new low energy cells to the propagating size of 0.7 μm through new loop patches or PSBs mechanism.

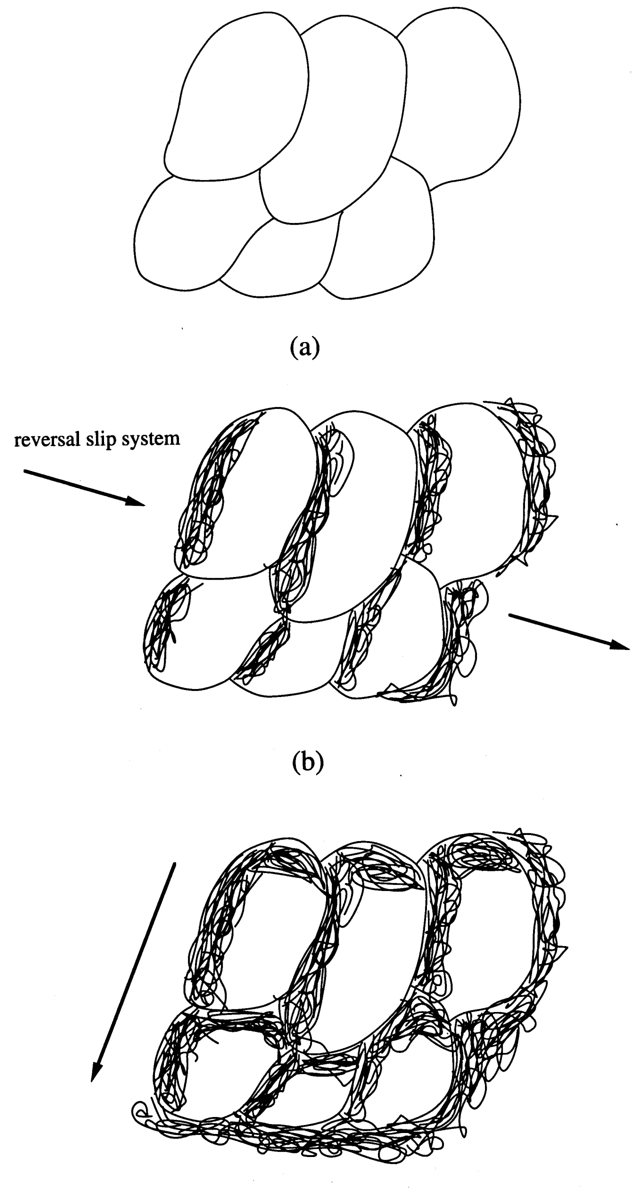


Fig. 10. Schematic diagrams show that dislocation structure develops from cells into loop patches resulting from residual slip system operations. (a) Original cells structure; (b) single residual slip system operation; (c) multiple residual slip systems operation.

Acknowledgements

The authors would like to acknowledge the financial support of National Science Council of ROC through contract NSC89-2216-E-110-013.

References

[1] C. Laird, Mat. Sci. Eng. 25 (1976) 1187.
[2] J. Awatani, A. Omura, K. Koyanagi, K. Katagiri, T. Shiraishi, H. Kaneshrio, Metall. Trans. 8A (1977) 176.
[3] Z.S. Basinski, R. Pascual, S.J. Basinski, Acta Metall. 31 (1983) 591.
[4] Z.S. Basinski, R. Pascual, S.J. Basinski, Acta Metall. 33 (1985) 1307.
[5] W. Liu, M. Bayerlein, H. Mughrabi, Acta Metall. 40 (1992) 1763.
[6] C. Li, T. Bretheau, Acta Metall. 37 (1989) 2645.
[7] H.L. Huang, N.J. Ho, W.B. Lin, Mater. Sci. Eng. A279 (2000) 261.
[8] J. Awatani, K. Katagiri, K. Koyanagi, Metall. Trans. 10A (1979) 503.
[9] H. Ishii, K. Yukawa, Metall. Trans. 10A (1979) 1881.
[10] K. Katagiri, J. Awatani, K. Koyanagi, Y. Onishi, M. Tsuji, Metall. Trans. 11A (1980) 2029.
[11] K. Katagiri, J. Awatani, K. Koyanagi, Phil. Mag. 38 (1978) 349.
[12] H.L. Huang, N.J. Ho, Mater. Sci. Eng. A298 (2000) 241.
[13] J. Mcevily, Z. Yang, Metall. Trans. 21A (1990) 2717.
[14] E.W. Lee, S.B. Chakrabortty, E.A. Starke, Jr., Metall. Trans. 15A (1984) 511.
[15] D. Gan, J. Weertman, Eng. Fracture Mech. 18 (1983) 155.
[16] T.K. Chaki, J.C.M. Li, Scripta Metall. 18 (1984) 703.
[17] Y. Higo, S. Nunomura, Acta Metall. 32 (1984) 1029.
[18] J. Schijve, D. Broke, Aircraft Eng. 34 (1962) 314.
[19] D.M. Corby, P.F. Packman, Eng. Fracture Mech. 5 (1973) 479.
[20] B.T. Ma, C. Laird, Sci. Eng. A102 (1988) 247.
[21] R. Zauter, H.J. Christ, H. Mughrabi, Phil. Mag. 66 (1992) 425.