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The Dislocation Evolution in Polycrystalline Copper Affected by Loading Ratio under Pure Tension*

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Abstract. The rate of crack propagation is determined by the rate of dislocation evolution. However, dislocation evolution is dominated by the loading condition in fatigue. To study the dislocation structure evolution affected by reversal slip systems, the rates of crack propagation variation were measured by changing the R ratio (fixed maximum loading and increased minimum loading) under pure tension condition in fatigue. In this study, compact tension specimens of polycrystalline copper were completed as the following ASTM. In order to measure the rates of changing in the experiment, a steady state rate of crack propagation was reached by Instron fatigue machine, and then it was subjected to an increasing of the minimum loading. The microstructure at crack tip was observed by means of the back electron image (BEI) of scanning electron microscope (SEM). In order to ensure that the dislocation development is affected by reversal slip system, the low cycle fatigue was performed for different R ratios at the same fatigue cycles (constant maximum loading) under pure tension loading. At the same time, after a complete low cycle, the specimens' micro-hardness was measured. The results reveal that: (1) the crack propagation rate is decreased and the range of dislocation evolution is widespread when the R ratio increases; (2) the micro-hardness of low cycle fatigue specimens is increased with an increased R ratio; (3) the dislocation structures in low cycle fatigue specimens show ladder-like wall structure embedded in loop patches at low R ratio and the cells' structure is created by multiple slip systems induced by hardness at a high ratio; (4) no matter what the crack propagation and low cycle fatigue, the results were determined by the reversal slip systems operation.

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1 Introduction

It is known that the development process of fatigue failure can be divided into steps of crack initiation and crack propagation. No matter the crack initiation or crack propagation, they result in dislocation interactions in the interior of the material [1]. For crack initiation, they are initiated in the persistent slip bands (PSBs) with a ladder-like wall dislocation structure embedded in the PSB [2-5], and also initiated by interactions between the persistent slip bands and the grain boundaries (PSBs-GB), in which the dislocation is a cell structure [3,6,7]. For crack propagation, the dislocation morphology ahead of the crack tip is usually cell structure in spite of the rate of crack propagation — high, or low [8-11]. The difference of dislocation structure between high and low crack propagation rate is the range of area that is occupied by dislocation morphology. In other words, the interface of cells, walls, PSBs, vein and loop patches structure is clear and the range of dislocation morphology is wide at high rate of crack propagation. Nevertheless, the interface is distinguished, the area of dislocation is small and dominated by veins and loop patches at low rate of crack propagation [11].

However, the condition of loading is a variable during the fatigue crack propagation. For example, the loading may have suddenly subjected to a peak of overload or underload or change the R ratio (change maximum loading or change minimum loading) during fatigue. Based on the theory of plasticity, the overload or underload will affect the rate of crack propagation. In the condition of overload, the crack behavior will retard the propagation rate [12-19]. The rate of retardation due to an overload can be explained that the overload will enlarge the plastic zone size, which will induces a residual stress in the region ahead of the crack tip, and it promotes crack tip closure. In case of underload, the crack propagation will be accelerated [17,19]. The explanation of this phenomenon is that the acceleration of the crack propagation rate is a result of a partially annihilation of the residual stress, which is built up by the previous positive load and it helps the crack tip to open. Nevertheless, the results of Huang et al. [19] reveal that the microstructure ahead of the crack tip passes from a cell structure to a vein structure when the crack propagation rate is subjected to a single peak overload. At the same time, it also exhibit the region, which is occupied by the dislocation cell structure in front of the crack tip is enlarged when the crack propagation tip is subjected to a single peak underload. According to the results of Huang et al. [3], no matter whether the rate of crack propagation is high or lows, the dislocation structure ahead of the crack tip is a cell with an average about 0.7 μm in diameter. Based on the dislocation evolution, the veins or loop patches structure developed into cell with an average about 0.7 μm in diameter need more much fatigue cycles at original loading amplitude. Therefor, the rate of crack propagation will be retarded when the loading is subjected to a single peak overload. Similarly, the dislocation structure (cell) in front of tips after underloading evolved into cell with an average about 0.7 μm need less fatigue cycles. Hence, the crack propagation rate will be accelerated when the loading is subjected to a peak underload.

In order to determine dislocation development affected by reversal slip system during fatigue in this experiment, it is important to study the minimum load change from low to high (increase R ratio) for crack propagation. Compact tension specimens of polycrystalline copper were used in this study to measure the rates of changing as increasing R ratio (increase the minimum loading and fixed the maximum loading) after the rate of crack propagation reached steady state during fatigue under pure tension condition. Since the dislocation evolution study is widest and deepest, the results can be compared with experimental results. At the same time, the grain boundaries and orientation are more much affected dislocation development. In other words, the dislocation evolution is difference at each grain in spite of the same accumulation of during fatigue. Although the grain boundaries can be decreased as the fatigue sample with large grain size, the grain orientation is more difficult to decrease in polycrystalline material. However, it is known the crack tip with concentration factor. Therefor, the dislocation evolution is fastest in specimen during fatigue crack propagation. Based above reason, the CT specimen of polycrystalline copper is used in this study.

The loading condition in this experiment, the plastic zone size ahead of the crack tip is not affected by changing the minimum loading because the maximum load is fixed. At the same time, the crack growth rate is affected by the minimum load changing from a low value to a high value under pure tension had seldom reported. According to the theory of material mechanics, the maximum loading is fixed during fatigue and the minimum load change is smaller than the crack opening force for pure tension, in which the rate of crack propagation is not affected by minimum loading change. Since the maximum loading is constant and the R ratio is positive, the maximum residual stress is constant ad against to apply loading direction always. In other words, change the minimum loading from low to high is variation the back stress which is created the reversal slip systems. Therefor, the results will ensure the affection of reversal slip systems in dislocation development. In addition, the low cycle fatigue with same constant maximum load and difference R ratios under pure tension loading condition was performed to make sure the dislocation evolution affected by reversal slip systems.

2 Experimental

A plate of high purity polycrystalline copper (99.95%) was used in this research. The specimens were annealed at 850°C for 2 hours in a vacuum of 10^{-5} torr and then cooled in a furnace. The grain sizes of the specimens were about 100–120 μm in average. The preparation of specimens followed the instruction of

ASTM E647 for a single edge compact tension specimen (CT). Crack propagation was performed on a computerized Instron 1332 hydraulic testing machine at different R (0.1, 0.3, 0.5, or 0.7) at a constant frequency of 20 Hz. During stable crack propagation, 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 travelling microscope to an accuracy of ± 0.01 mm. When the crack propagation reached a special rate, the specimen was then increased the minimum load (increase the R ratio). The stress intensity (ΔK) was determined from ASTM E647 as:

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

where

$$f(a/w) = \frac{(2+a/w)(0.866+4.72(a/w)-5.56(a/w)^2+13.32(a/w)^3-14.72(a/w)^4)}{(1-a/w)^{1.5}};$$

B — thickness; a — crack length; w — effective width; ΔP — range of loading, $\Delta P = P_{\max} - P_{\min}$.

After the rate of crack propagation is stabilized, the minimum loading is increased. In other words, the R ratio is increased from 0.1 to 0.3, 0.5, or 0.7. After increase the R ratio, the crack propagation will retard and then continue fatigue. Once upon the crack propagation again, record the fatigue cycles of retardation and hold fatigue up to measure the rate of crack propagation. In order to observe the dislocation evolution from crack retardation to propagation again, as crack propagation again from retardation stop fatigue to remove the specimen from fatigue test machine. In addition, the low cycle fatigue with same constant maximum load and difference R ratios under pure tension loading condition was performed. The specimens of micro-hardness and dislocation structure were detected after completed low cycle fatigue to make sure the dislocation evolution affected by reversal slip systems.

To observe dislocation morphology evolution at the crack tip from crack retardation to re-propagation, the test specimens (subjected to increase minimum loading) were cut into tiny squares of 10 mm that contained a crack longer than 3 mm (as shown in Figure 1(a)). The squares were then cut into slices having a thickness of 0.6 mm. The slices were ground to a thickness of 0.15–0.2 mm using abrasive paper. A 3 mm in diameter disks, which contain a crack length at least 1 mm, were punched (as shown in Figure 1(b)). For low cycle fatigue specimen, the gage portion was sliced into 0.6 mm in thickness along the cross section by a slow cutter. The slices were divided into two parts. In order to measure the micro-hardness of low cycle fatigue slice, one part of slice is used cold mount by epoxy and then polish the surface. The other parts were ground to a thickness of 0.15–0.2 mm using abrasive paper. The slices were disks 3 mm

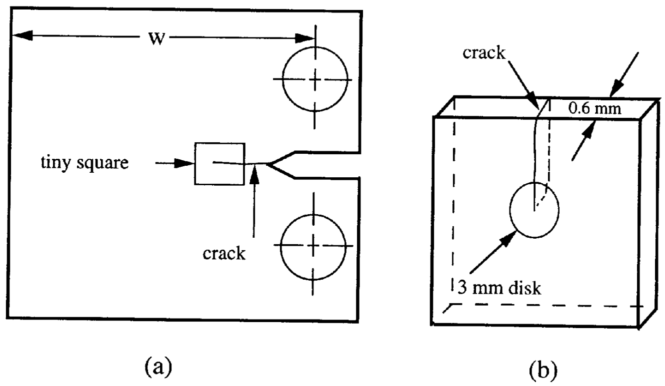

Figure 1. Schematic diagrams of the TEM foil preparation: (a) position of tiny square; (b) position of discs.

in diameter were punched. The 3 mm disks were twin-jet polished using Sturus D2 polishing solution at 7–9 V and -10°C to prepare the specimen for electron microscope. A JEOL 6400 SEM was used to investigate the microstructures at the crack tip and the crack path by means of back electron microscope (BEI) [20]. The accelerating voltage was set at 25 KV, and the working distance was 8 mm. For low cycle fatigue specimen, the microstructure was observed in a JEOL 200CX TEM at acceleration voltage 200 KV.

3 Results

The crack tip propagation is retarded with the increase of R ratio as shown in Table 1. No matter what the loading R ratio increase is, it is clear that the rate of crack propagation is decreased with increased minimum loading for pure polycrystalline copper under pure tension loading condition. At the same time, the rate of crack propagation retardation is more decreased when R ratio increased is larger. The dislocation structure in front of the crack tips is misorientation cell and followed the dislocation cell when R ratio is 0.1 (rate of crack propa-

Table 1. The rate of crack propagation at different R ratio under increase minimum load

R ratio	Rate of crack [mm/cycle]	Maximum load [Kgf]
0.1	1.29×10^{-5}	220
0.2	3.40×10^{-6}	220
0.3	9.40×10^{-7}	220
0.5	4.50×10^{-7}	220
0.7	4.70×10^{-8}	220

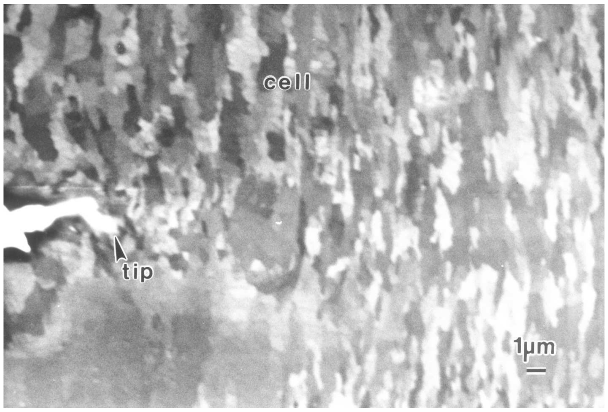

Figure 2. The dislocation structure in front of the crack tips at rate of about 2×10^{-5} mm per cycle under $R = 0.1$.

gation is about 2×10^{-5} mm per cycle) as shown in Figure 2. The dislocation morphology in front of the crack tips when the crack propagation restart after the R ratio increase from 0.1 to 0.3, 0.5, and 0.7 is shown in Figures 3(a), (b), and (c). Figure 3(a) points out that the dislocation structure ahead of the crack tips is misorientation cell and following the dislocation cell when the R ratio increase from 0.1 to 0.3 (the crack propagation rate decrease from 2×10^{-5} to 7×10^{-6} mm per cycle). As the R ratio change from 0.1 to 0.5, the dislocation structure ahead of the crack tips is dominated by uncondensed cell (Figure 3(b), the crack propagation rate decrease from 2×10^{-5} to 2×10^{-7} mm per cycle). Figure 3(c) shows the dislocation structure at crack tips is dominated by uncondensed cell when R ratio increase from 0.1 to 0.7 (the crack propagation rate decrease 2×10^{-5} to 8×10^{-8} mm per cycle). The micro-hardness of specimens after completed low cycle fatigue to 15000 cycles is listed in Table 2. It shows the micro-hardness with little difference between sample 1 and 2, but sample 3 is of distinguishable different. In order words, the micro-hardness is higher when the R ratio is larger. At the same time, the microstructure of low cycle fatigue test specimens is shown in Figure 4. Figure 4(a) reveals the dislocation structure is dominated by loop patches structure and the PSBs structure embedded in loop

Table 2. The micro-hardness was revealed after 15000 cycles low cycle fatigue under different minimum loading (R ratio)

R ratio	Micro-hardness [HV]	Number of cycles
0.3	61.88	1.5×10^4
0.5	60.25	1.5×10^4
0.7	70.9	1.5×10^4

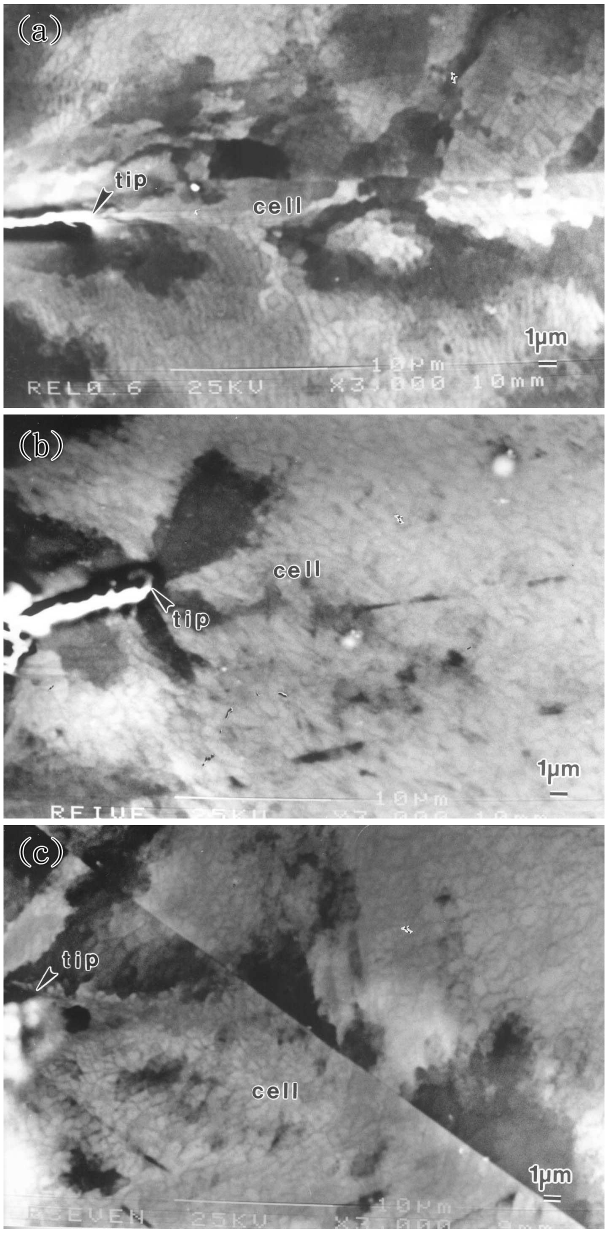

Figure 3. The dislocation structure ahead of the crack tips: (a) $R = 0.3$ (7×10^{-6} mm per cycle); (b) $R = 0.5$ (2×10^{-7} mm per cycle); (c) $R = 0.7$ (8×10^{-8} mm per cycle).

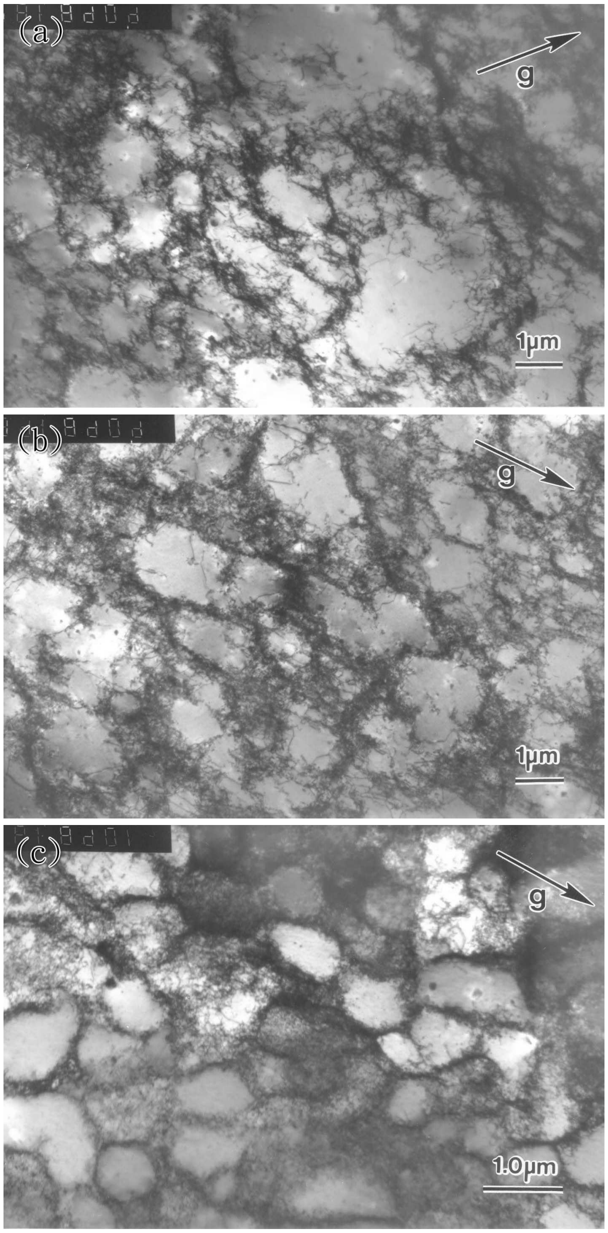

Figure 4. The dislocation structure at the same as number of 1.5×10^4 cycles under different R ratio in low cycles fatigue: (a) $R = 0.3$; (b) $R = 0.5$; (c) $R = 0.7$.

patches structure at $R = 0.3$. The dislocation structures (Figure 4(b)) is similar to those of Figure 4(a) when R is 0.5. The dislocation structure in Figure 4(c) is dominated by loop patches structure, but the uncondensed cell structure had formatted in some grains ($R = 0.7$).

4 Discussion

Based on the theory of material mechanics, it is well known that there is a minimum stress intensity factor (Δk_{op}) to open the crack. In other words, the crack is unable to propagate when the applied stress intensity factor (Δk) is smaller than Δk_{op} in fatigue. Similarly, the applied minimum stress intensity factor ($\Delta k_{\min}$) is unable to affect the rate of crack propagation when $\Delta k_{\min}$ is smaller than Δk_{op} in fatigue ($\Delta k_{\text{eff}} = \Delta k_{\max} - \Delta k_{\min}$, when $\Delta k_{\min} < \Delta k_{\text{op}}$, $\Delta k_{\min} = \Delta k_{\text{op}}$). Therefore, it is no affected the crack propagation when increase the minimum Δk under Δk_{op} during fatigue. As shown in Table 1, it is distinguish that the crack of propagation rate is changed in spite of the increases of minimum applied loading (R ratio, $\Delta k_{\min}$) in pure tension fatigue ($R = P_{\max}/P_{\min}$, $R =$ positive). This result implies that there is no real Δk_{op} in pure tension fatigue on polycrystalline copper.

The morphologies of dislocation for different R ratios (difference crack propagation rate are shown in Figures 2 and 3. It is revealed that the evolution of dislocation structure ahead of the crack tip is slow when R ratio is lower after increased the minimum loading. Based on this experiment design, they are with same maximum loading in spite of increase R ratio from 0.1 to 0.3, 0.5, and 0.7. These results exhibit that the rate of the crack re-propagation is decreased after retardation (Table 1). At the same time, the crack propagation rate decreases with a promoted the R ratio. According to Huang et al. [AA], it is point out that the dislocation structure in front of crack tips is cell structure in spite of the rate of propagation is high or low. It also indicate the dislocation cell with an about 0.7 μm in diameter ahead of crack tips, the crack will be able to propagate. Therefore, the period of crack retardation is modulation the dislocation structure to balance between applied and repulsive stress and toward cell development after increased the R ratio. However, the dislocation structure is misorientation cell (Figure 2) dominated at $R = 0.1$ (rate crack propagation is 2×10^{-5} mm per cycle). Hence, the period of retardation is regulated misorientation cell toward low energy cell with a about 0.7 μm in diameter. Since the crack propagation with same constant maximum loading at difference R ratios, they have same residual stress (repulsive stress), which is created by previous apply maximum loading. Once the crack propagation reaches to steady state at $R = 0.1$, the stress is balance between applied and repulsive stress.

As soon as the maximum loading is applied in fatigue, the maximum repulse stress was created by dislocation interaction and regulated the dislocation development. Once upon the minimum loading is applied in fatigue, the net force is created by difference between the applied minimum loading and the repulse stress, which was generated by the previous maximum loading during fatigue, as shown in Figure 5. Because the crack propagation with same constant maximum loading at difference R ratios, they have same residual stress (repulsive stress). As the crack propagation reaches to steady state at $R = 0.1$, the stress is balance between applied and repulsive stress. During fatigue, promote the R ratio (increase minimum loading) is violate the balance, which build in crack propagation at $R = 0.1$. In order words, the net stress of back is smaller between applied and repulsive stress when the minimum loading applied during fatigue (Figure 5). At the same time, the effect is increase with promoted R ratio.

It is known that the reversal slip systems are created by net stress of back during fatigue. Based on above discussion, the net stress of back is smaller with higher R ratio. However, the dislocation development is nonhomogenous in the interior of material due to strain localization [21] and concentration effect due to crack tip. The reverse slip system is operated in spite of the net back stress is small and it occupied range is narrow. Therefore, the reversal slip system can also be created at high R ratio loading condition. Compare with the same maximum loading condition in pure tension fatigue, the reversal slip system is easily operated for small R ratio loading condition.

Based on the Table 2, the hardening rate increase with increased R ratio at the same constant maximum loading under pure tension fatigue. Therefore, the dislocation development in low R ratio is more completely than high R ratio. It is because that the reversal slip system is easily to be operated in fatigue, and the dislocation pile up can be degraded. Meanwhile, the hardening effect due to dislocation pile up in fatigue can be slow down. Under this condition, multiple slip systems are difficult to be created by dislocation pile up. Hence, the dislocation evolution with small R ratio (Figure 4(a)) is slower than with high R ratio (Figure 4(c)). At the same time, the dislocation development for low R ratio is step by step. Therefore, the cell is low energy saturation dislocation cell (with small diameter) as soon as cell formation for low R ratio. However, the cell formation at high R ratio is due to hardening induce the multiple slip systems to create dislocation cell structure. Accordingly, the dislocation evolution is fast and reveal an uncondensed (unsaturation) dislocation cells at $R = 0.7$ (Figure 4(c)). Based on the theory of two phases reported by Winter [22], the PSB is soft phase and loop patches and the cells are hard phase. Compare with Figure 4(a) and (c), it is reasonable that the hardness is high when R ratio is high.

In addition to those mentioned above, the reversal slip system and set up a new balance between applied and repulsive stress at low R ratio is easier than high R ratio. Therefore, the period of crack retardation for modulation dislocation to build up a new balance is increase with a promoted R ratio. At the same time, with higher R ratio have higher hardening rate induce the dislocation evolution slow at same maximum loading condition. Hence, the rate of crack propagation

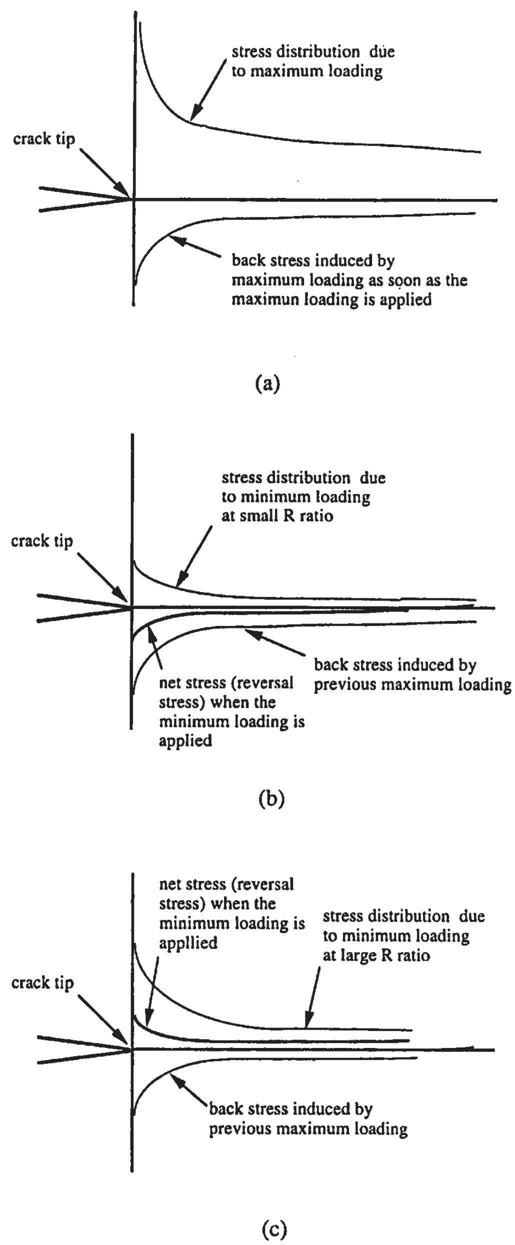

Figure 5. Schematic diagrams of: (a) the back stress under different minimum load; (b) the net stress in low R ratio. (c) the net stress in high R ratio.

decreased with a promoted R ratio and the effect proportional R ratio increased. Based on the same reason, the dislocation structure in front of crack tips is similar $R = 0.1$ (Figure 2) for lower R ratio (Figures 3(a) and (b)). Since higher R ratio with higher hardening rate, the accumulation of plastic strain is small. At the same time, the area in front of the crack tip is affected by concentration factor of crack tip. The area ahead of crack tips have higher hardening rate. As a result, the dislocation development is slowest among R ratio in this experiment. Conversely, the energy of plastic strain accumulation is store in dislocation structure. Thus, the dislocation development of near the crack tip is faster than crack tip. Because the plastic strain is small, it is unnecessary more slip system to regulate dislocation compared with low R ratio. Therefore, the misorentation cell structure (Figure 2) would be transferred into uncondensed cell structure (Figure 3(c)).

5 Conclusions

For pure tension, the retardation of crack propagation after the crack tip is subjected to the minimum load increased in the steady state of loading. At the same time, retardation of crack propagation increases with increasing R ratio.

The retardation of crack propagation after the R ratio increases, is due to rate of dislocation structures evolution into low energy cells was slow down. Because the R ratio increases, it is difficult to create the reversal slip systems.

The micro-hardness increases with the increase of the R ratio at 150,000 fatigue cycles. The rate of formation cells increases with an increase in R ratio. Since the hardening effect results from dislocation pile up is easier for high R ratio, the result induces multiple slip systems to create the cells.

Acknowledgments

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