

# A study of dislocation evolution in polycrystalline copper during low cycle fatigue at low strain amplitudes

H.L. Huang\*

*Department of Mechanical Engineering, Chinese Military Academy, Fungshan, 830 Kaohsiung, Taiwan, ROC*

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## Abstract

The dislocation structures evolution on the polycrystalline copper at constant strain amplitude during low cycle fatigue is well understood. However, the dislocation structures developments at variable strains, which change from high to low strain amplitude, are seldom reported. In order to realize the dislocation morphology evolution at reduced strain amplitude, the polycrystalline copper is used in this study. The results show that. (1) The S–N curve at reduced strain amplitude from high to low reveals softening, and it is lower than S–N curve which is fatigue in constant strain amplitude at low strain amplitude. (2) The dislocation structures of walls, labyrinth walls, cells or misorientation cells transfer into scattering walls, loop patches or cells. (3) The dislocation morphology development at decreased strain amplitude is determined by the magnitude of reduction in strain amplitude.

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## 1. Introduction

It is known that the dislocation evolution in low-cycle fatigue for copper is a sequence of loop patches, vein structures, persistent slip bands (PSBs), walls, cells and misorientation cells [1–5]. According to the reported literature, the dislocation evolution is a step-by-step process from loop patches to cells at low plastic strain amplitudes [6,7] and the dislocation structure forms dislocation cells at high plastic strain amplitudes [6,8]. Moreover, the reported literature also points out that dislocation development is more at the grain boundaries and twin boundaries due to effect of strain localization [9–12]. At the same time, the result also reveals that the dislocation evolution in large size grain is more completed than in the small size grain [13,14].

The dislocation structure in front of the crack tip is seldom reported in the literature. It has been pointed out that the dislocation structure at high crack propagation rates in copper is cells [15,16]. Recent, results of Huang et al. [17] indicate that no matter what the rate of crack

propagation, the dislocation structure in front of the crack tips is essentially cells. However, it is well known that the crack propagation rate is retarded when the loading condition changes from high strain amplitude to low strain amplitude. Based on elementary theory of plasticity, the effect is due to plastic zone size, which induces compressive stresses. Nevertheless, it is known that the energy of plastic strain is stored in dislocation structures during fatigue [6]. Meanwhile, the dislocation morphology is variable when the loading sequence is changed during fatigue. To this point, Laird [18] has pointed out that the dislocation structure changes from cells to loop patches when the amplitude of loading changes from high to low. However, no experimental result were shown to substantiate this the dislocation structure evolution. In polycrystalline copper during low cycle fatigue at low strain amplitudes was studied and the deformation structures observed by means of transmission electron microscope (TEM).

## 2. Experimental

A polycrystalline copper plate of high purity (99.95%) was used in this study. The specimens were annealed at

\* Tel.: +886-7-74-9510; fax: +886-7-74-9515

E-mail address: [hluang@cc.cma.edu.tw](mailto:hluang@cc.cma.edu.tw) (H.L. Huang).

850 °C for 2 h in a vacuum of  $10^{-5}$  torr and then cooled in a furnace. The grain sizes of the specimens were about 100–120  $\mu\text{m}$ . The preparation of specimens followed specification out lined in ASTM E647. The low cycle fatigue tests were done on a computerized Instron 1332 hydraulic testing machine at  $R = -1$  (strain ratio,  $R = \epsilon_{\text{min}}/\epsilon_{\text{max}}$ ) and a frequency of 1 Hz.

At first, the low cycle fatigue is controlled at 0.3% strain amplitude during fatigue. Under this condition, Specimen is deformed to 500 cycles the strain amplitude is decreased from 0.3 to 0.1%. After drop tiny in the strain amplitude, the low cycle fatigue test is continued to a cycle which is design by this research. The specimen was removed from the fatigue test machine. A schematic diagram is shown in Fig. 1. In this study, the specimen was deformed for 0, 1, 2 and  $3 \times 10^4$  cycles after drop the strain amplitude (i.e. index A–D in Fig. 1). In order to compare the fatigue life of the specimen of a 0.1% constant strain amplitude with the fatigue life of specimens which were with 0.3% strain amplitude fatigue to 500 cycles and then decrease the strain amplitude to 0.1% strain amplitude, a strain amplitude of 0.1% low-cycle fatigue testing was completed in this study as well.

To observe the dislocation morphology, the specimens of the experiment deformed, were cut into a slice having 0.6 mm thickness along the cross section. The slices were ground to a thickness of 0.1–0.5 mm using abrasive paper and then punched into 3 mm diameter discs by using Gatan puncher. The 3 mm disks were twin-jet polished using Struer D<sub>2</sub> solution at 12 V and  $-10$  °C to prepare the foils of electron microscope. A JEOL 200CX transmission electron microscope (TEM) was used to examine the microstructures of the deformed specimen in low-cycle fatigue test.

### 3. Results

The fatigue tests data in this experiment is listed in Table 1. The results show that the fatigue life in variable strain amplitudes (amplitudes of strain change from 0.3

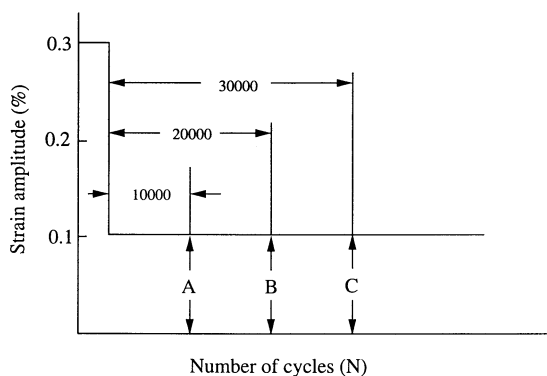


Fig. 1. Schematics illustration of the loading condition.

to 0.1%) is larger than 0.1% constant strain amplitude. The stress versus number of fatigue cycles (S–N) is shown in Fig. 2. It reveals the hardening rate to be high and reaches a stable value at about 70 cycles for 0.3% strain amplitude. This was followed by softening with a decrease in strain amplitude to 0.1%. Fig. 3(a) shows the dislocation structure at  $1 \times 10^4$  cycles following a strain amplitude decrease from 0.3 to 0.1% (B specimen). The dislocation is almost a type of low energy walls, but the wall structure near the grain boundary (labeled with arrow) similar diffusion. The local area (labeled with arrow in Fig. 3(a)) at high magnifications reveals the dislocation structure at the wall to be scattering (Fig. 3(b)). Fig. 4 indicates the dislocation structure at  $2 \times 10^4$  cycles after strain amplitude decrease from 0.3 to 0.1% (C specimen). Fig. 4(a) is a low magnification micrograph that shows the dislocation structures in several grains. The dislocation morphology in grain A (Fig. 4(a)) is essentially misorientation cells, and the dislocation structure is cell at the grain boundary (labeled with arrow). The dislocation structure in grains B and C (Fig. 4(a)) is a diffused wall (labeled with arrow). Fig. 4(b) reveals the dislocation morphology at the grain boundary to be loop patches and Labyrinth structure in the interior of grain. Fig. 5 indicates the dislocation structure at  $3 \times 10^4$  cycles following in strain amplitude decrease from 0.3 to 0.1% (D specimen). The dislocation structure in Fig. 5(a) and (c) is in the same grain. Fig. 5(a) reveal the dislocation structure near the grain boundary to be loop patches and well within the interior of grain to be scattering walls and Labyrinth structure. The local region (labeled 'a' in Fig. 5(a)) shows the dislocation structures to be loop patches which evolve from wall scattering. In Fig. 5(b) reveals that the dislocation structures near the grain boundary are essentially wall scattering along two directions (labeled 'a' and 'b' in Fig. 5(b)) and create the dislocation loop patches structure (labeled 'c' in Fig. 5(b)). In Fig. 5(c) is shown the dislocation to be Labyrinth structure. Fig. 5(d) a high magnification micrograph of Fig. 5(c). It shows the Labyrinth structure is scattering (labeled with arrow) along one direction.

### 4. Discussion

In Fig. 2, it is shown that the initial hardening rate is rapid until stabilized. It is reasonable that the high strain amplitudes will induce multiple slip systems to facilitate the formation of dislocation cell structure in low-cycle fatigue [6,8]. Based on the theory of dislocations, the hardening effect is determined by dislocation mobility, which means that higher the rate of hardening, more difficulties for dislocation motion. At the same time, dislocation pile up creates repulsive forces. During fatigue, the repulsive force and applied force would

Table 1  
The fatigue test data at different strain amplitude

Number of samples	Items				
	Strain amplitude (%)	Reduce strain amplitude	Reduce after cycle	Final cycle	Fracture
1	0.3	No	–	5124	Yes
2	0.3	No	–	4738	Yes
3	0.1	No	–	157 137	Yes
4	0.1	No	–	146 176	Yes
5	0.3	0.1	–	196 428	Yes
6	0.3	0.1	–	238 684	Yes
7	0.3	0.1	–	500	No
8	0.3	0.1	500	10 000	No
9	0.3	0.1	500	20 000	No
10	0.3	0.1	500	30 000	No

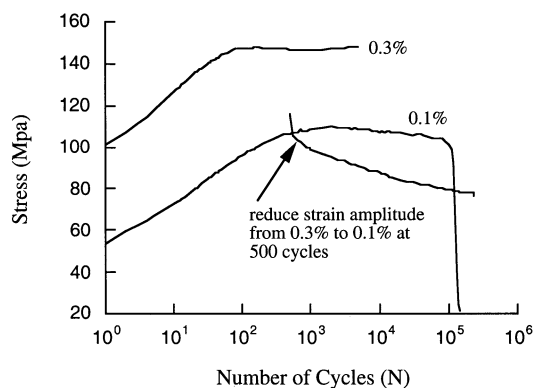


Fig. 2. The curve of stress vs. number of cycles in low cycle fatigue at various strain amplitudes.

regulate dislocation more much stability. Therefore, presence of a saturation fatigue stress implies a high repulsive force. Thus dislocation evolution drive force is determined by a balance of repulsive and applied forces. As the strain amplitude is decreased, the net force between the repulsive and applied forces renews in the direction of the applied force. These effects promote dislocation movement in a direction of the anti-applied force to accommodate the repulsive forces which are created by initial interaction of dislocations, up to the balance point between repulse forces and apply forces.

Examine the dislocation morphologies in specimens of B–D, the dislocation structures reveals wall scattering (Figs. 3 and 4(a) and Fig. 5), misorientation cells transfer to cells (Fig. 4(a)) and formation loop patches (Fig. 4(b) and Fig. 5(a) and (b)). These dislocation structures are observed at sites near the grain boundaries, and the dislocation structure is low energy walls or cells in interior of the grain. However, it is known that the dislocation structures in polycrystalline copper are loop patches, PSBs, walls, cells and misorientation cells. Which one of these dislocation morphologies is present in the microstructure is determined by the plastic strain accumulation and strain amplitude during fatigue.

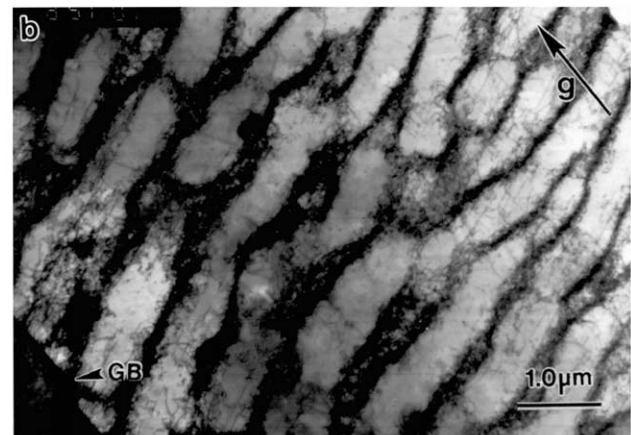
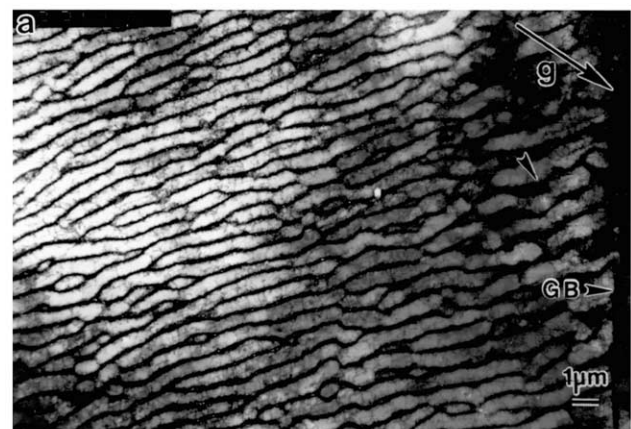


Fig. 3. The dislocation structures at  $1 \times 10^4$  cycles after a decrease of the strain amplitude from 0.3 to 0.1% (specimen B), (a)  $B = (011)$ ,  $g = [200]$ . (b)  $B = (011)$ ,  $g = [\bar{1}1\bar{1}]$ .

Meanwhile, the dislocation is developed step-by-step at low strain amplitudes [6,7] and easily to create cells structure due to the multiple slip systems at high strain amplitudes [6,8]. Also, in the dislocation structure at near the grain boundaries develops faster than that in interior of the grain [9–12] due to strain localization. However, in Figs. 3–5 shows that they are violate the normal evolution of dislocation at low strain amplitude

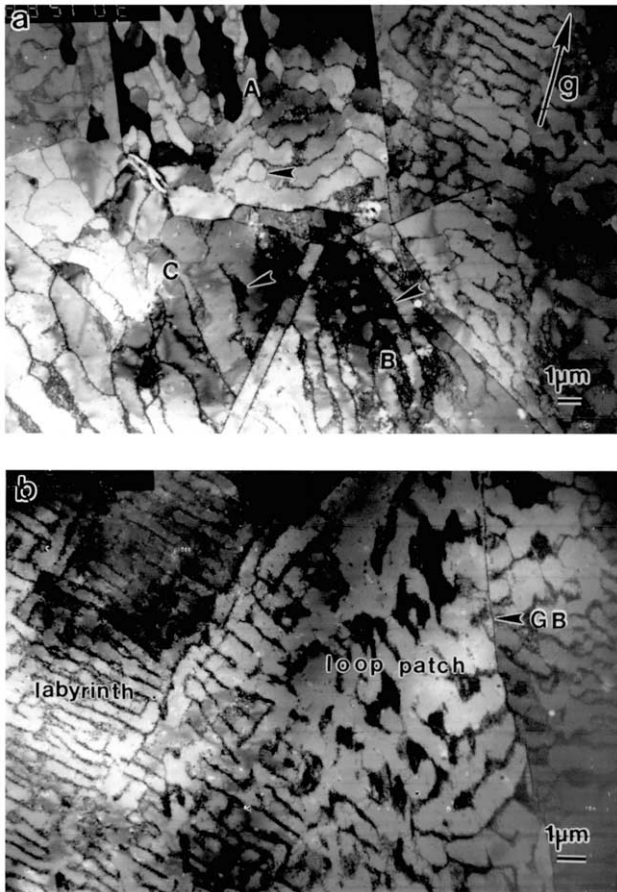


Fig. 4. The dislocation structures at  $2 \times 10^4$  cycles after a decrease of the strain amplitude from 0.3 to 0.1% (specimen C). (a)  $B = (011)$ ,  $g = [200]$ . (b)  $B = (112)$ ,  $g = [11\bar{1}]$ .

during fatigue. Because the net force (repulse force add apply force) is toward to opposite normal direction of dislocation evolution once the strain amplitude reduces from 0.3 to 0.1% (point A in Fig. 2), and the grain boundaries and twin boundaries are the preferential sites for strain localization. The back force is larger than the interior of the grain with a decrease in strain amplitude. Hence, the dislocation development is faster at the grain boundaries and twin boundaries. Based on above discussion, the dislocation evolve toward to opposite normal direction and take place near the grain boundary at first (Figs. 3 and 4(a)) and then extends into interior of the grain (Fig. 4(b) and Fig. 5). Since the dislocation development towards the opposite direction induces wall scattering, cells and formation of loop patches, the repulsive force due to mutual interaction of dislocation is reduced. Therefore, the apply force to balance the repulsive force is decrease during the dislocation reversal development. This result in the fatigue saturation stress declines (softening) with a decrease in strain amplitude and lower than that with a constant strain amplitude of same magnitude (Fig. 2). Based on the theory of dislocation evolution, the low

energy of wall structure results in only single slip system, and the cells, misorientation cells and Labyrinth structure are result from multiple slip systems. Hence, low energy of the wall structure result in the formation in wall of scattering along one direction (Fig. 3(a) and Fig. 5(c) and (d)). For cell structure, it is develop toward the opposite direction the theory of dislocation normal evolution. Meanwhile, the slip systems are determined by the magnitude of reduction in strain amplitude. For large reduction strain amplitude, it will induce the multiple slip systems to form loop patches structure. For small decrease in strain amplitude, the dislocation development is a one-by-one operation in multiple slip systems which were created by the high strain amplitude until reaches the balance the force between repulsive and applied force. It can be proved in Fig. 4(a) (grain A), which reveals misorientation cells transfer to cells. Therefore, the condition for dislocation development following a reduction in strain amplitude is determined by the magnitude of decrease in strain amplitude. In other words, the final dislocation structure is revealed in condition of after decrease in strain amplitude. At the same time, it is known that processing of the dislocation structure evolution is affected by grain orientation during fatigue. Hence, the reverse of the dislocation structure evolution is also affected by grain orientation. Thus, the dislocation structure is not definitely to evolve into loop patches structure in all grain of fatigue specimen when a reverse in dislocation development was revealed after a reduction in strain amplitude

However the S–N curve (stress vs. number of fatigue cycles) shows that in case of strain amplitude decrease from 0.3 to 0.1%, the stress is smaller than that of constant strain amplitude at 0.1% (Fig. 2). Based on the discussion above, a reasonable explanation can be obtained, i.e. when the strain amplitude decreased from 0.3 to 0.1%, the direction of the dislocation structure evolution towards the opposite direction of that of a strain amplitude 0.3%, and the dislocation evolution is inhomogeneous, which is due to the grain boundary effect. In other words, while the strain amplitude decrease from 0.3 to 0.1%; the evolution of the dislocation structures will be initiated in the region near the grain boundary because of the localization of the strain. However, when the dislocation structure near the grain boundary has evolve to a status that a constant strain amplitude of 0.1% should be, the dislocation structure will evolve along the direction which posses lower energy. But in the region out of this area, the dislocation evolve in a direction opposite to that of a 0.3% strain amplitude, and it is late and slower; therefore, before the dislocation structure evolve to the normal status of a 0.1% strain amplitude, the dislocation structures evolve towards the opposite direction of dislocation structure evolution at strain amplitude of 0.3%. Therefore, there are several different dislocation

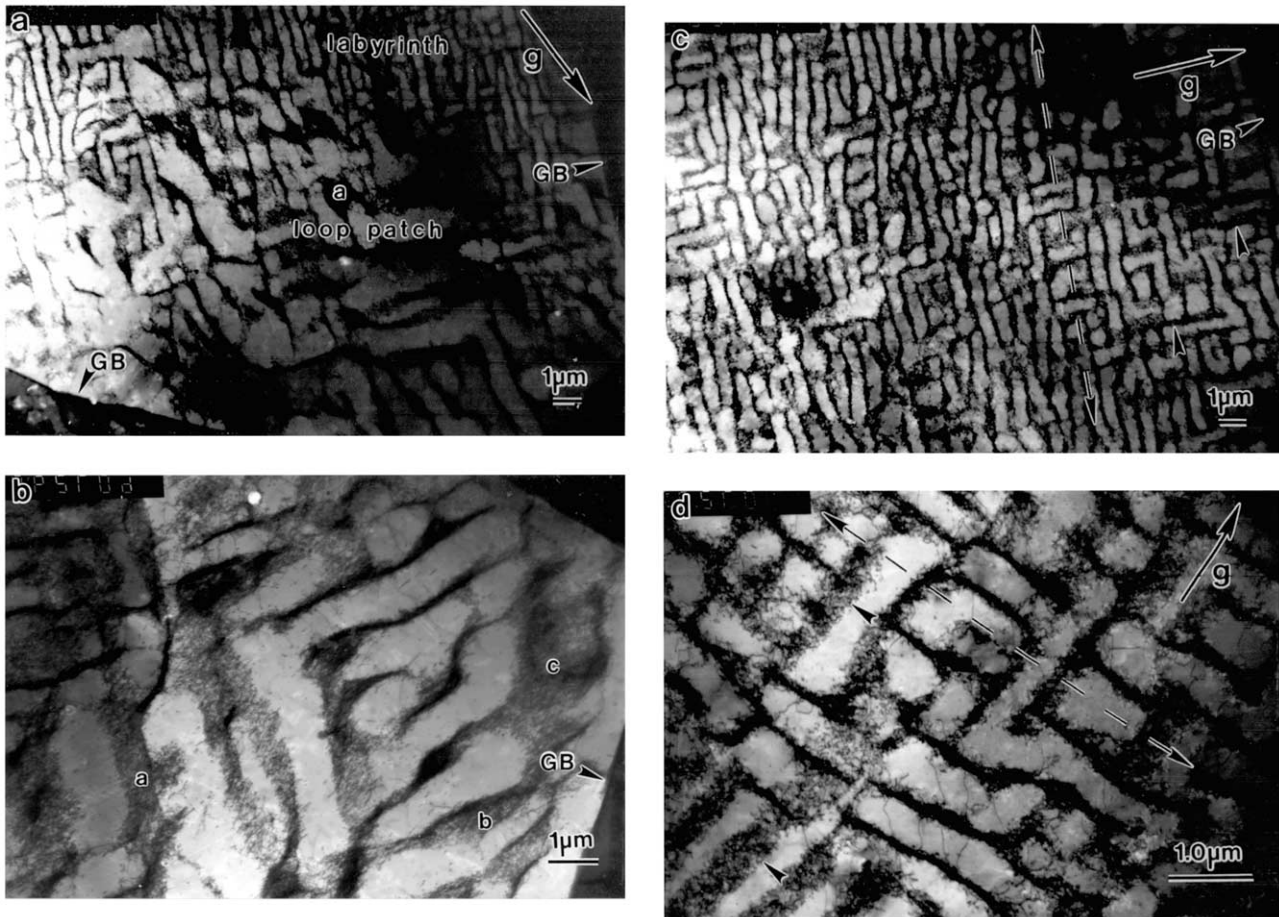


Fig. 5. The dislocation structures at  $3 \times 10^4$  cycles after a decrease of the strain amplitude from 0.3 to 0.1% (specimen D). (a)  $B = (011)$ ,  $g = [\bar{1}\bar{1}\bar{1}]$ . (b)  $B = (011)$ ,  $g = [11\bar{1}]$ . (c)  $B = (011)$ ,  $g = [\bar{1}\bar{1}\bar{1}]$ . (d)  $B = (011)$ ,  $g = [\bar{1}\bar{1}\bar{1}]$ .

evolution directions even in the same grain. Since the grain size of the copper specimens used in this study is 100–120  $\mu\text{m}$ , it needs a long fatigue cycles to make all the dislocation structure in the same grain evolve to the normal dislocation structure status of a 0.1% constant strain amplitude. Hence, while the strain amplitude decreased from 0.3 to 0.1%, the corresponding S–N curve is lower the curve of 0.1% constant strain amplitude. Moreover, for a polycrystalline copper with large grains, since there are several different dislocation evolution directions even in the same grain, the evolution rate of the dislocation structure near the grain boundaries starts to evolve towards to the low energy direction is smaller than the dislocation structure evolution rate of 0.1% constant strain amplitude. It is well known that the fatigue life can be distinguished into crack initiation and propagation, and no matter what the crack propagation rate is, the dislocation structure is low energy dislocation cell [17]. Therefore, the fatigue life of specimen with strain amplitude decreased 0.3–0.1% is longer that of constant 0.1% strain amplitude, as shown in Table 1.

## 5. Conclusion

For strain amplitude decrease from high to low during low cycle fatigue, the curve of stress versus number of cycles reveals softening and it is lower than the S–N curve at constant strain amplitude of 0.1% finally.

The dislocation structures evolve with a strain amplitude decrease from high to low, the low energy structure of walls, Labyrinth walls, cells and misorientation cells, which were formed at the higher strain amplitude, transfer to dislocation structure of scattering walls, loop patches, and cells structure.

The processing of dislocation structure development following a reduction in the amplitude of strain is determined by the magnitude of reduction strain amplitude. For large decrease  $\epsilon$ , the back stress (net stress, repulse stress add to apply stress) induces multiple reverse slip systems. For small reduction in strain amplitude, the back stress creates the reverse slip system step-by-step.

The reverse of dislocation structures evolution develop up to saturation stress (the net stress between

repulse and applies stress is toward the original direction) of the low strain amplitude, the dislocation evolve toward to low energy dislocation structures, which is in the condition of the low strain amplitude.

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