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Dislocation evolution in interstitial-free steel during fatigue near the endurance limit

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Abstract In order to clear the relationship between dislocation development and endurance limit in fatigued body-centered cubic (BCC) metals, the automotive grade interstitial-free steel (IF steel) was fatigued near the endurance limit in this study. When cycling just below the endurance limit, the dislocation structures are mainly composed of loop patches, moreover, a few large dislocation cells and dislocation walls can also be found, and thus these structures have no significant effect on fatigue failure. However, once cyclic strain slightly exceeds the endurance limit, the small dislocation cells tend to develop near grain boundaries and triple junction of the grains, and which provide a more appropriate structure for crack growth than do large dislocation cells.

Introduction

Recently, Majumdar et al. [1] determined that the fatigue endurance limit for interstitial-free (IF) steel is close to 0.98 YS (yield stress), which is much higher than that of most of low carbon steels (0.6–0.8 YS) [2]. Additionally, Majumdar et al. [1] and Narasaiah et al. [3] found that microcracks developed near grain boundaries when they were cycled below the endurance limit. Majumdar et al. [1] also noted that microcracks occur at inclusions. However, Narasaiah et al. [3] excluded small inclusions as potential sites for the nucleation of microcracks because of their low aspect ratio. Microcracks that do not propagate are called non-propagating cracks [1, 3]. However, once stress/strain amplitudes increase above the endurance limit, these microcracks close to grain boundaries will coalesce to form a major propagating crack that propagates near and along grain boundaries at the beginning of the crack propagation period while those within the grain interiors remain non-propagating. Once persistent slip bands (PSBs) develop in copper, for example, fatigue failure will definitely occur [4, 5] and, a non-propagating crack has never been observed during low-cycle fatigue. Dislocation structures such as dislocation cells, loop patches, and dipolar walls have been intensively studied [6–9]. The propagation of long crack depends on the development of dislocation cells in front of the fatigue crack tip of copper metals [10–12]. The dislocation morphologies within grains and grain boundaries in IF steel during fatigue close to the endurance limit has not been reported on, and this topic is the main focus of this work.

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Experimental

A hot rolled polycrystalline interstitial-free steel (IF steel) plate with a chemical composition of $C < 50$ ppm, $N < 50$ ppm, $S < 120$ ppm, $B \approx 2$ ppm, $Mn \approx 0.15$ wt%, $Ti \approx 0.04$ wt%, and balance Fe was used in this study. The material was annealed at 800 °C for 2 h and then cooled in a furnace to obtain an average grain size of about 80 μm in diameter. The preparation of specimens followed the ASTM E606 specification. A computerized Instron 8801 hydraulic testing machine was employed at a testing strain rate of 4×10^{-3} s^{-1} with $R = 0$ at room temperature. The tests on IF steels were performed with maximum strain (ε_{max}) ranging from 0.15 to 0.4% (i.e., total strain amplitude ($\Delta\varepsilon/2$) ranging from 0.075 to 0.2%). All fatigued samples were cut into slices of 1 mm thickness. The slices were ground to a thickness about of 0.3 mm for SEM and 0.1 mm for TEM observations using abrasive paper and punched into 3 mm-diameter discs. The 3 mm discs were twin-jet polished using a solution of 90% methanol diluted with 10% perchlorate at 15 V and −40 °C. For the microstructure observations, a scanning electron microscope (SEM) of Philips Quant 200 SEM under the BEI mode and a transmission electron microscope (TEM) of Philips CM200 by bright field imaging (BFI) in which a two-beam mode was employed.

Results and discussion

The plot in Fig. 1 of cyclic responding stress versus the number of cycles clearly reveals that fatigue failure does not occur when the strain amplitude is controlled below $\varepsilon_{max} = 0.2\%$. As the cyclic strain was increased above $\varepsilon_{max} = 0.25\%$, the maximum cyclic stress rapidly increased in the early stage of cycling and the secondary cyclic hardening occurred prior to fatigue failure. Majumdar et al. [1] indicated that the fatigue failure in cyclically deformed IF steel did not occur when stress was maintained below 0.98 YS, and they defined this as the endurance limit. In this study, the YS of IF steel is about 108 MPa. The corresponding cyclic stress for $\varepsilon_{max} = 0.2\%$ is around 105 MPa, this is about 0.97 YS. This result is very close to that suggestion by Majumdar et al. [1]. Therefore, $\varepsilon_{max} = 0.2\%$ in this study is a strain that is very close to the endurance limit. Additionally, fatigue failure

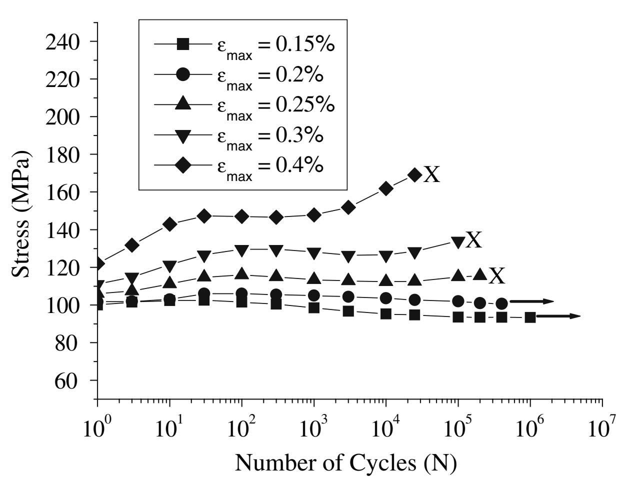

Fig. 1 Cyclic stress responses with number of cycles were plotted at various total strain amplitudes under $R = 0$ condition, where mark X is for fatigue failure and *arrow* for no failure

and secondary cyclic hardening did not occur when cyclic strains were controlled below $\varepsilon_{max} = 0.2\%$.

The dislocation structures after 1×10^6 cycles at $\varepsilon_{max} = 0.15\%$, as shown in Fig. 2a, mainly are composed of loop-patch structure. Interestingly, an inclusion of 10 μm in size does not transform surrounding loop patches into other higher stress forms (Fig. 2b). Apart from the argument of Narasaiah et al. [3] that small inclusions are not potential sites for nucleation of microcracks due to the low aspect ratio in fatigued interstitial-free steel, Murakami et al. [13, 14] noted that the fatigue limit of 0.13% C low carbon steel is independent of the presence of a hole (artificial defect, such as a drilled hole) with a diameter of <40 μm. Restated, fatigue failure is not affected by the defects that are smaller than 40 μm. Therefore, whether inclusions that are smaller than 40 μm are detrimental to fatigue has not been conclusively determined. Basically, the results of the experiment in this study are consistent with the finding of Narasaiah et al. [3] and Murakami et al. [13, 14], because the inclusions in this study are smaller than 40 μm and no divergence of dislocation structure occurs near the inclusions. Hence, the defects or inclusions are irrelevant to the development of dislocations and fatigue failure. Figure 2c–e displays detailed TEM images of loop patches, whose contrast is markedly reduced when $g = [10\bar{1}]$ at $B \approx [111]$ (where g is g vector and B is beam direction). Accordingly, the loop patches mainly are composed of dislocation with primary Burgers vector of $[1\bar{1}1]$, and the gliding behavior of dislocations for $\varepsilon_{max} = 0.15\%$ is a single slip principally.

The dislocation structures after 4×10^5 cycles at $\varepsilon_{max} = 0.2\%$ shown in Fig. 3a are mostly loop-patch structure. Figure 3b and c shows marks Y and Z in Fig. 3a at high magnification, in which the loop-patch structure predominates in the dislocation structures, and a few dislocation cells that are larger than 2 μm (large dislocation cells) and dislocation walls can also be found. However, dislocation cells that are smaller than 2 μm are very difficult to be found. Therefore, the loop-patch structure and large dislocation cells are not associated with fatigue failure. Majumdar et al. [1] and Narasaiah et al. [3] had indicated that the crack initiation (microcracks) occurs near grain boundaries when stress is below the endurance limit, and so even if crack initiation had already occurred, the microcracks are reasonably inferred to be unable to coalesce, resulting in the growth of cracks through loop-patch structure, large dislocation cells and dislocation wall. This process reasonably explains the lack of failure when cyclic strains were controlled below $\varepsilon_{max} = 0.2\%$. Therefore, these microcracks should be the non-propagating cracks.

Compared with $\varepsilon_{max} = 0.2\%$, $\varepsilon_{max} = 0.25\%$ shows an extreme difference in dislocation structure as shown in Fig. 4a–d, in which many small dislocation cells (smaller

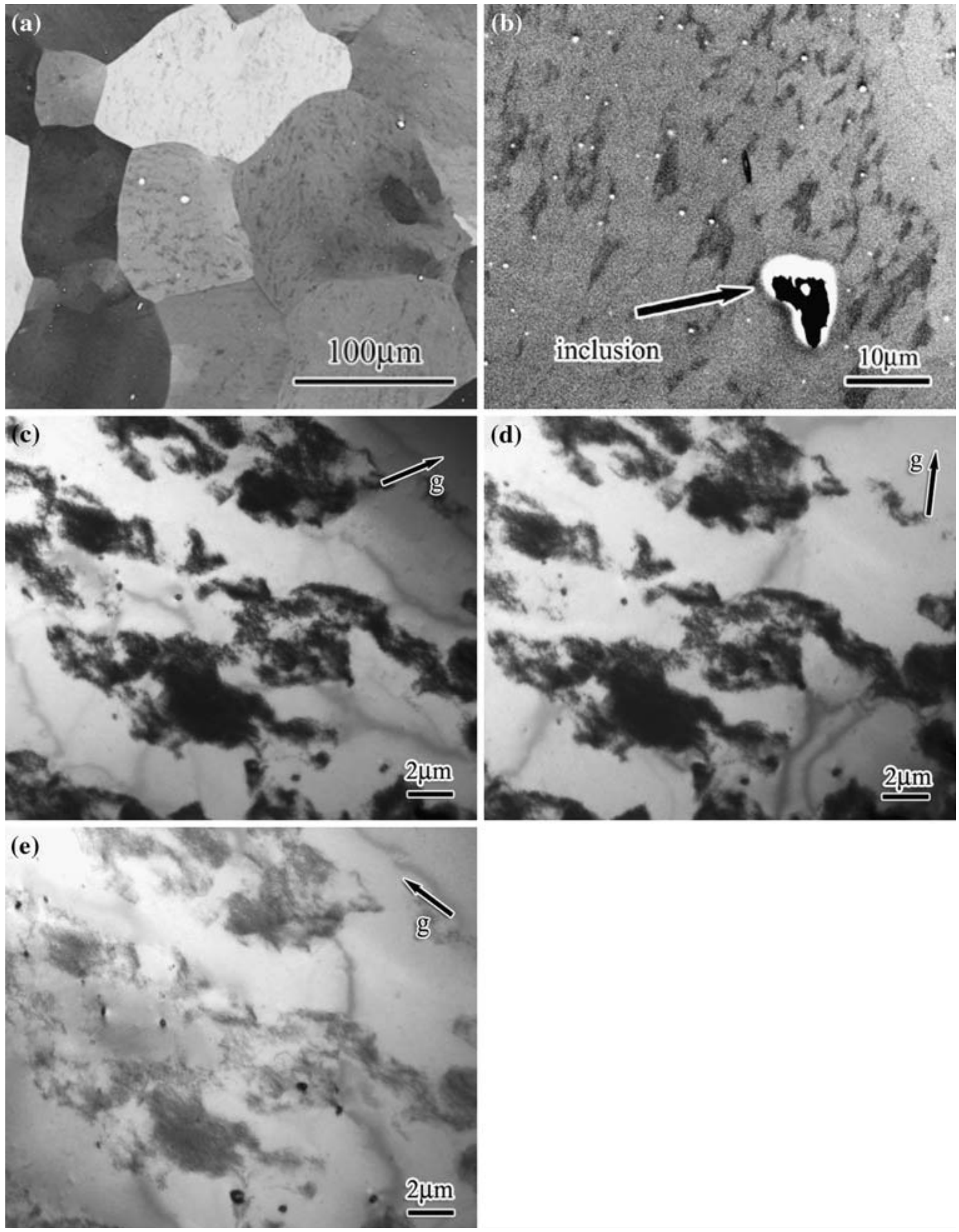

Fig. 2 SEM/BEI/ECCI micrographs taken from sample cycled to 1×10^6 cycles without failure at $\varepsilon_{max} = 0.15\%$ showing loop patches predominate: **a** in all grains; **b** around inclusion; **c** TEM/BFI showing the detail loop-patch structure, where $B \approx [111]$, $g = [0\bar{1}1]$; **d** same as (**c**), where $B \approx [111]$, $g = [1\bar{1}0]$; and **e** same as (**c**), where $B \approx [111]$, $g = [10\bar{1}]$

than 2 μm) can be seen, but the loop-patch structure is absent. The estimated volume fraction of the small dislocation cells and large dislocation cells are about 30 and 70%, respectively. Figure 4b presents in detail mark H in Fig. 4a, and Fig. 4c and d shows the TEM counterpart of Fig. 4b. The small dislocation cells had formed preferentially along the grain boundaries, and the size of the dislocation cell decreased as the distance from the grain boundary. Furthermore, the smaller dislocation cells also tend to form near the triple junction of the grains, as shown in Fig. 5a and b. Figueroa et al. [15] and Sommer et al. [16] indicated that the incompatibility between the neighboring grains would results in the localized deformation occurring near the grain boundary. Since the interior of each grain is far from its grain boundaries, the dislocation development in the grain interior should be affected only by the applied stress. Apart from the applied stress, incompatible stress will also affect the dislocation development in the areas near grain boundaries. These two kinds of stress simultaneously act on the regions near grain boundaries making the dislocation morphology dissimilar from that in the grain interior. This phenomenon is probably responsible for the different rates of development of the dislocation structures close to grain boundaries and in the grain

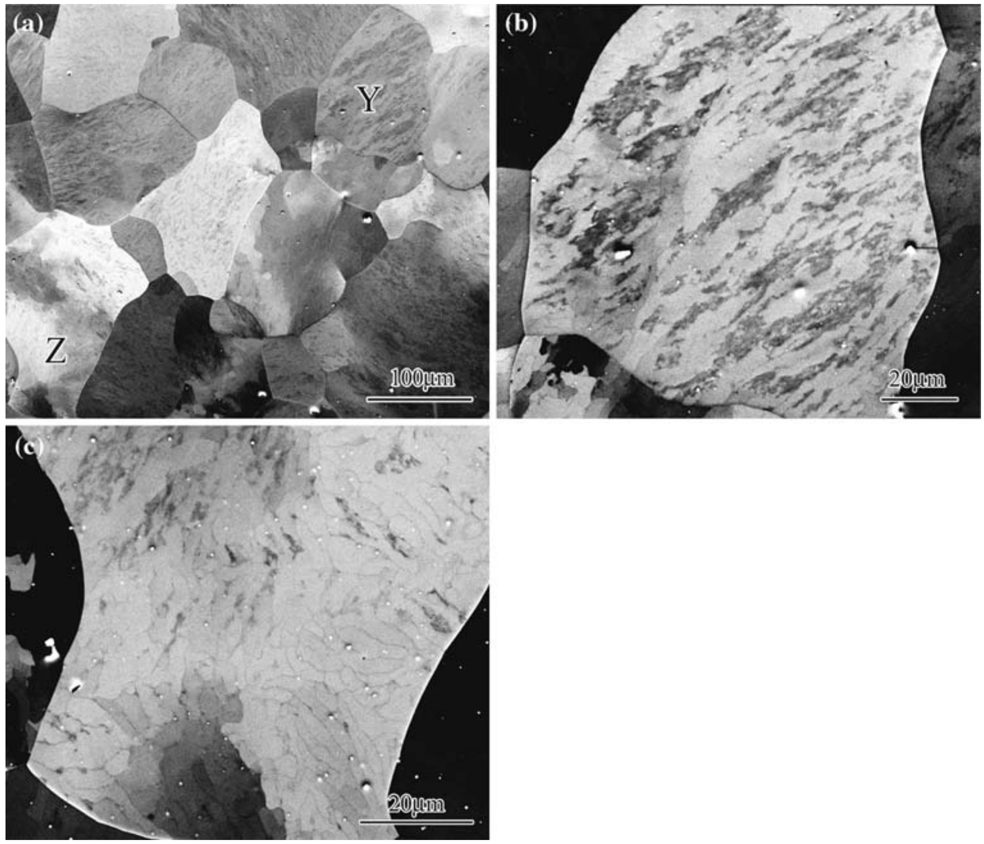

Fig. 3 SEM/BEI/ECCI micrographs taken from sample cycled to 4×10^5 cycles without failure at $\varepsilon_{max} = 0.2\%$: **a** showing loop patch structure developed within most of grains; **b** mark Y in (**a**) showing detail loop patches; and **c** mark Z in (**a**) showing large dislocation cells and dislocation walls

interior. Therefore, the incompatible stress is essential to accommodate the small dislocation cells that are formed near the grain boundary at a cyclic strain that slightly exceeds the endurance limit.

Majumdar et al. [1] indicated that the fatigue failure is preceded by the significant growth of grain-boundary cracks over and above those at inclusions and the ferrite grain body. This finding suggests that dislocation structures in the grain interior differ from those close to grain boundaries. Many studies have demonstrated that the dislocation cells always develop in front of the crack tip in most metals [10–12, 17–19]. Additionally, Awatani et al. [19] further indicated in an experiment on crack propagation in α-iron, the size of the dislocation cell apparently decreases as the distance from the side of the crack decreases. This result demonstrates that the cracks prefer to grow into a region that contains smaller dislocation cells than one that contains larger dislocation cells. Hence, the aggregation of small dislocation cells along grain boundaries and the triple junction of grains which are observed in this experiment, provide a more appropriate structure for crack growth than do large dislocation cells, such mechanism are schematized in Fig. 6. Consequently, grain boundaries are better sites for fatigue cracking than inclusions when cyclic deformation is close to the endurance limit.

Conclusions

1. Fatigue failure does not occur when the strain amplitude is controlled below $\varepsilon_{max} = 0.2\%$. As the cyclic strain was increased above $\varepsilon_{max} = 0.25\%$, the secondary cyclic hardening occurred prior to fatigue failure. Hence, $\varepsilon_{max} = 0.2\%$ in this study is a strain that is very close to the endurance limit.
2. Dislocation structures are mainly composed of loop-patch structure while cycling just below fatigue endurance limit. Additionally, a few large dislocation cells can be found near the grain boundary; hence, loop-patch structure and large dislocation cells have no significant effect on fatigue failure.
3. While cyclic strain slightly exceeds the endurance limit, the small dislocation cells tend to develop near grain boundaries and triple junction of the grains. These small dislocation cells provide a more appropriate structure for crack growth than do large dislocation cells.

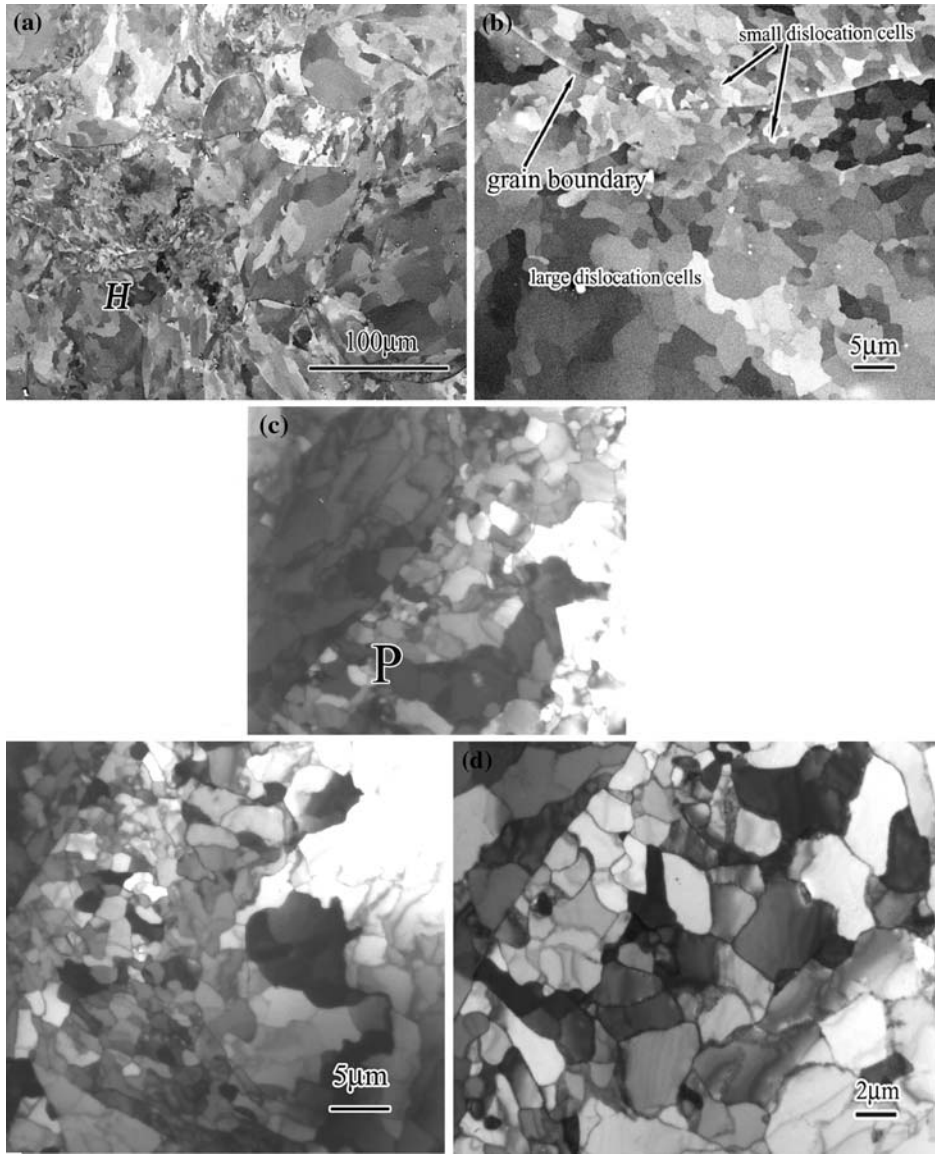

Fig. 4 Microstructure of IF steel cyclically deformed at $\varepsilon_{max} = 0.25\%$ and cycled to failure: **a** SEM micrograph observation under BEI/ECCI mode showing all grains are occupied by either large dislocation cells or small dislocation cells; **b** mark H in (**a**) showing small dislocation cells prefer to form along the grain boundaries than large dislocation cells; **c** similar morphologies shown in TEM micrograph for comparison; and **d** mark P in (**c**)

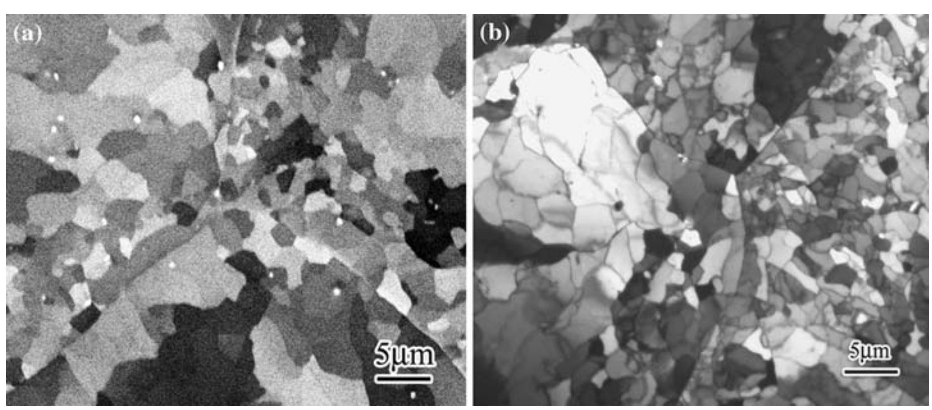

Fig. 5 Microstructure of IF steel cyclically deformed at $\varepsilon_{max} = 0.25\%$ and cycled to failure: **a** SEM micrograph observation under BEI/ECCI mode showing many small dislocation cells developed near the triple junction of the grains; and **b** similar morphologies shown in TEM micrograph for comparison

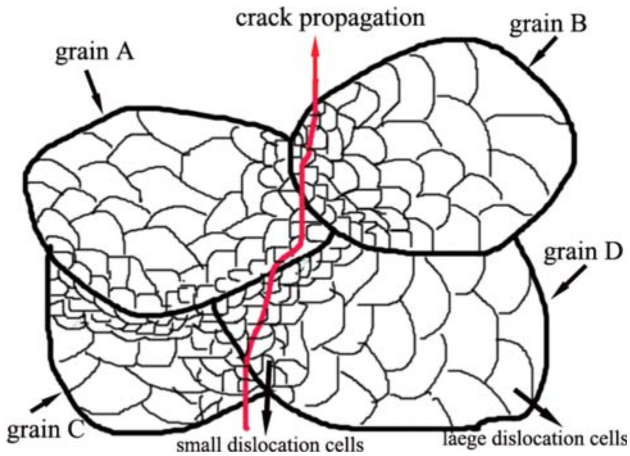

Fig. 6 Schematic diagram shows the relationship between crack propagation and dislocation structures

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