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Dislocation structures in nickel during high temperature low cycle fatigue at large cumulative strains

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The development of the dislocation arrangement during high temperature low cycle fatigue in nickel polycrystals was investigated. Special attention was paid to dislocation structures in the vicinity of grain boundaries. It was found that a defined cell structure develops during progressing cyclic deformation. The motion of grain boundaries associated with high temperature cyclic deformation leads to a dislocation structure gradient in the wake of the boundaries. These structure gradients are suspected to be prone to setting off nucleation of dynamic recrystallization. Upon strain path changes the fatigued structure was observed to become gradually superimposed by a new cell structure without prior decomposition.

1. Introduction

The dislocation structure during monotonic deformation at elevated temperatures has been frequently shown to develop from a more random arrangement to a tangled cell structure and eventually to a subgrain structure with well recovered subgrain boundaries after large strains [1–3]. However, the development of a dislocation cell structure is not confined to large monotonic strains. Despite small strain amplitudes, a defined cell structure was also observed to develop during high temperature low cycle fatigue (HTLCF) of various materials [4–6].

High temperature deformation also comprises other microstructure developments besides cell formation. Grain-boundary migration during HTLCF has been frequently reported [7, 8], and recently it was even reported that grain-boundary migration may also cause dynamic recrystallization (DRX) during cyclic deformation. When grain boundaries move, they consume the dislocations which they encounter in the swept volume, except for very special crystallographic arrangements, where dislocations can pass the boundary [9]. In transmission electron microscopy (TEM) investigations on specimens cycled at elevated temperatures, however, dislocation free regions were never observed in the vicinity of grain boundaries despite unambiguous evidence of grain-boundary migration from optical microscopy. However, distinct differences in the dislocation structure in the grain interior and near grain boundaries were obvious. It is the objective of this paper to show that these differences are due to different development stages of the dislocation structure and that they actually can be utilized as a probe for structural changes, in particular to monitor grain-boundary migration during deformation.

2. Experimental procedure

Cylindrical fatigue specimens with a gauge section of 24.5 mm length and 12.6 mm^2 cross section were machined from 99.99% pure nickel rods. Strain controlled cyclic deformation was carried out on a computerized INSTRON mechanical testing machine at a strain rate of $1.5 \times 10^{-4}\,sec^{-1}$, corresponding to cyclic frequencies between 4×10^{-3} and $1.3 \times 10^{-2}\,sec^{-1}$, depending on cyclic strain amplitude. The deformation temperature was always 600° C (0.5 T_m, T_m is the melting temperature), and the tests were conducted in a reducing atmosphere of 90% N_2 and 10% H_2 to avoid oxidation. TEM samples were prepared by sectioning the deformed samples perpendicular to the stress axis, thinning down mechanically and finally jet polishing to perforation in a solution of 950 ml acetic acid and 50 ml perchloric acid. All TEM investigations were carried out in a HITACHI H800 electron microscope at 200 kV operating voltage.

3. Results and discussion

Figure 1 shows the cyclic hardening curve of samples deformed at 600° C with a total strain amplitude $\Delta\varepsilon = 0.5\%$ and cycle frequency 1.3×10^{-2} Hz. The corresponding microstructures are given in Figs 2 to 5. After 10 cycles the development of a cell structure is evident (Fig. 2). The grain boundary in Fig. 2b reveals heavy activity in terms of steps, curvature and considerable dislocation content in the boundary. In contrast to the cell interior (Fig. 2a), the cell structure in the vicinity of the boundary is only poorly developed with incomplete cell wall sections and frequent debris in the cell interior. After 40 cycles, the cell structure in the grain interior is now very well developed and much more distinct than after 10 cycles, although far from being uniform (Fig. 3). There are areas with narrow, condensed cell walls and essentially dislocation free cell volumes (Fig. 3a), but other areas comprise much less orderly arranged dislocations (Fig. 3b).

The dislocation structure after 160 cycles (Fig. 4) is

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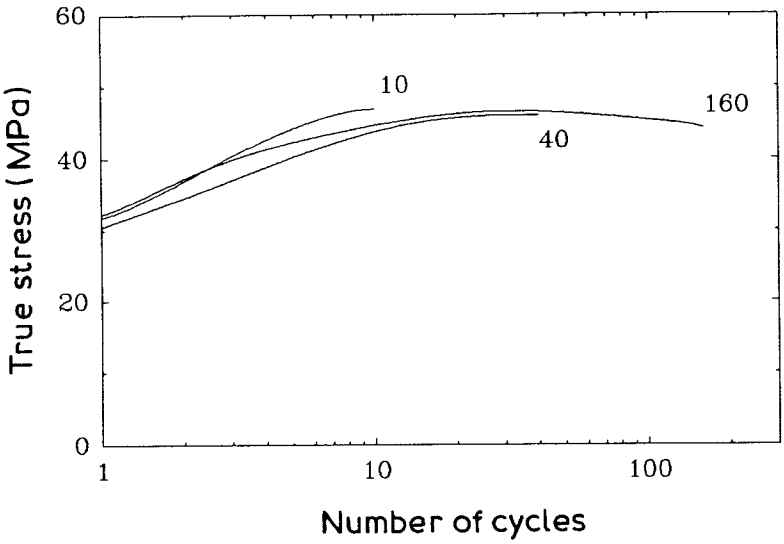
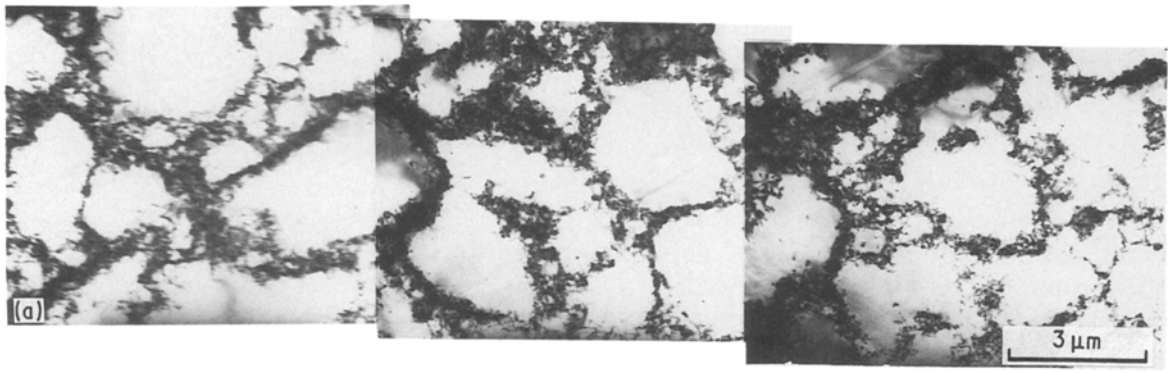


Figure 1 Cyclic hardening curve of nickel tested at 600° C with 0.5% total strain amplitude.



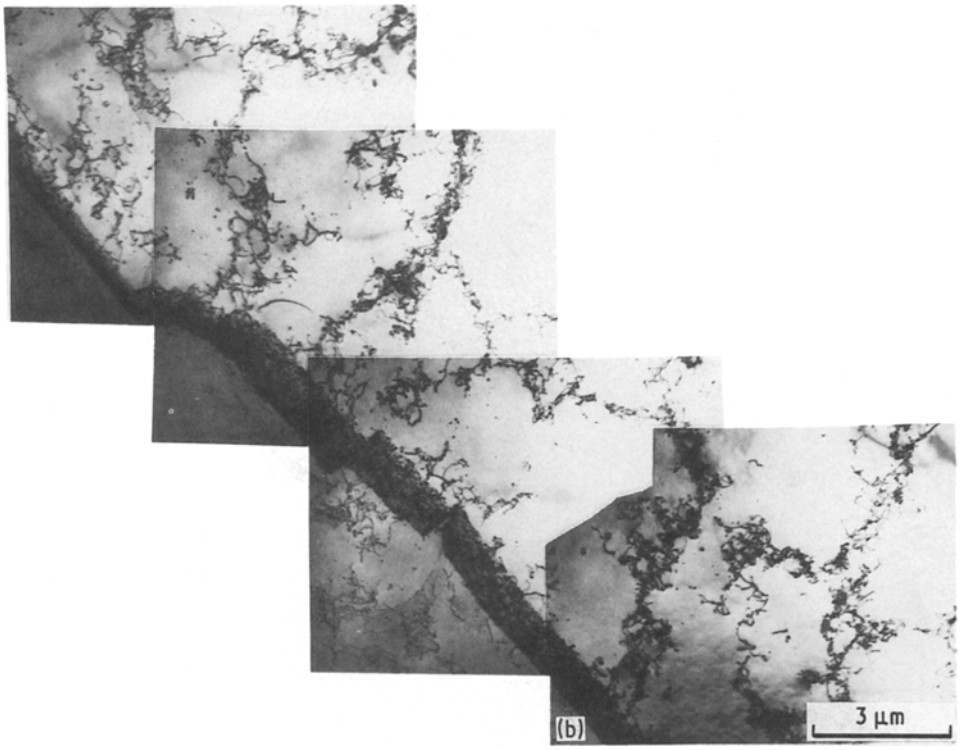


Figure 2 Microstructure of nickel after testing at 600° C with 0.5% total strain amplitude for 10 cycles. (a) grain interior, (b) around a grain boundary.

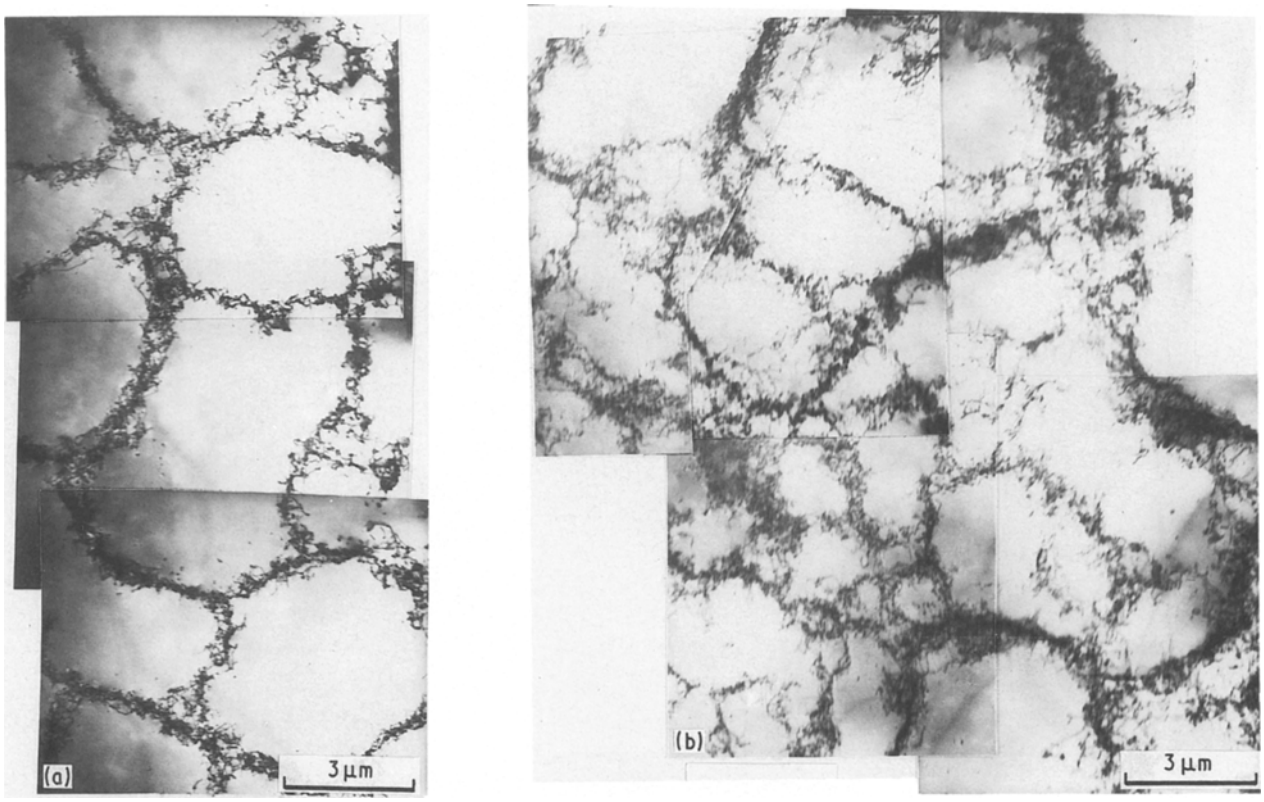


Figure 3 Microstructure of nickel after testing at 600° C with 0.5% total strain amplitude for 40 cycles. (a) and (b) different areas in the same specimen. Note difference in structure.

akin to the structure after 40 cycles. Most dislocations are arranged in constricted cell walls, enclosing regions completely denuded of dislocations, but locally patches of less orderly dislocation patterns are apparent. The area around a grain boundary is shown in Fig. 5. The boundary curvature indicates recent grain boundary motion. Far away from the grain boundary the cell structure is very pronounced and well recovered. In close proximity to the boundary, however, the dislocation structure is quite different, namely very little organized and only incipient of cell formation. Obviously the structure in the wake of the boundary corresponds to a very early state of cell structure development and thus, has to be interpreted as the rebuilding of a cell structure which was eliminated during grain-boundary migration.

It is noted that the poor recovery of the cell structure is not due to simply its proximity to the grain boundary. There are regions where a clean cell structure extends right to the boundary (e.g. Fig. 6b). This is in agreement with observations that some grain boundary migrate very far, while others do not migrate at all [10]. It is also interesting with regard to grain-boundary motion during HTLCF that the dislocation

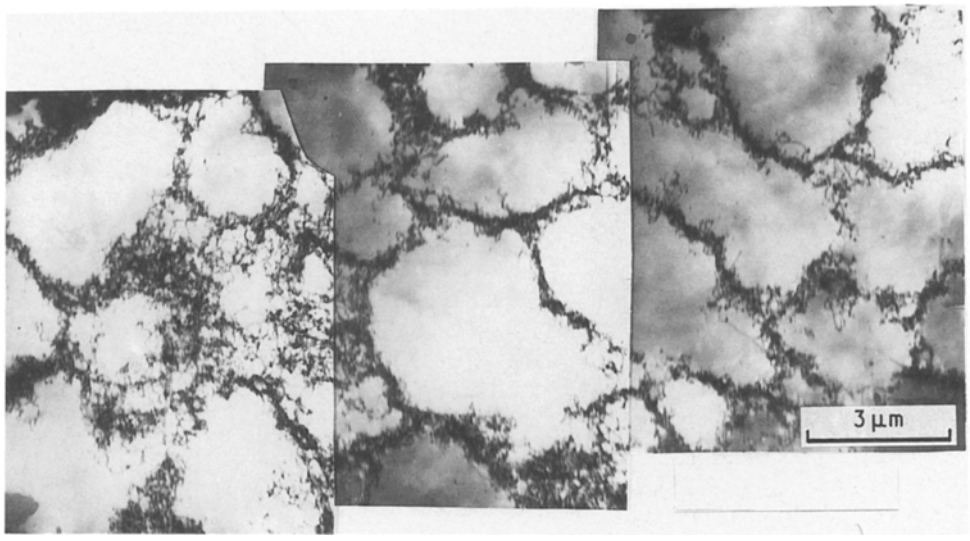


Figure 4 Dislocation structure in the grain interior of nickel after testing at 600° C with 0.5% total strain amplitude for 160 cycles.

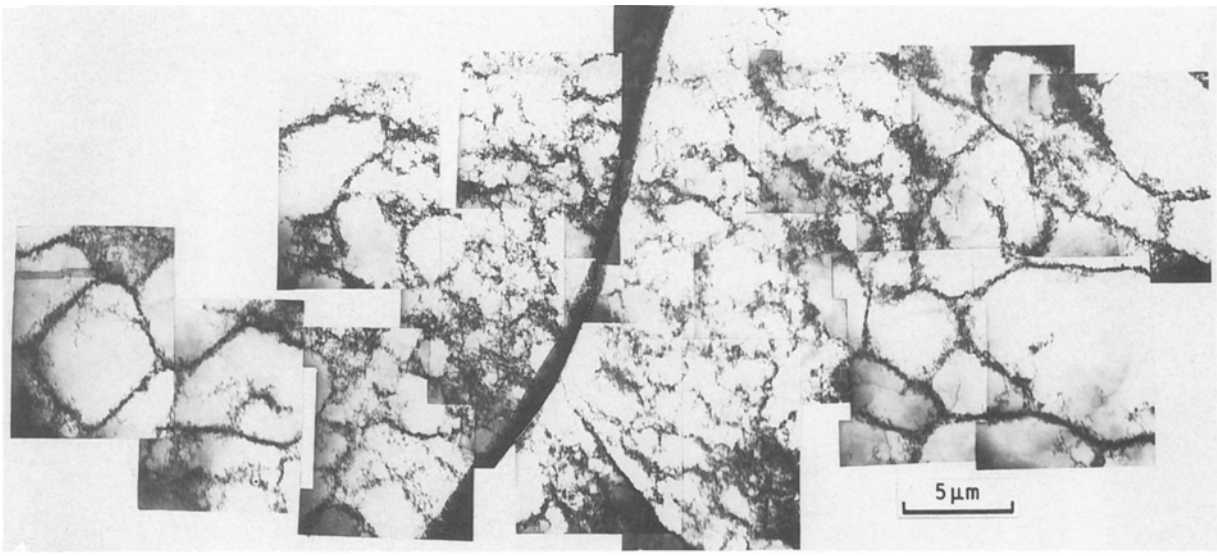
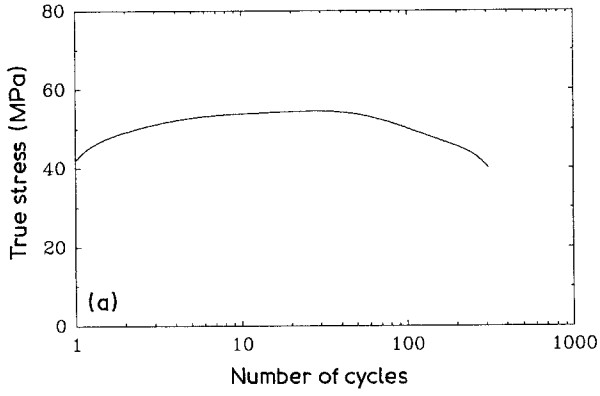
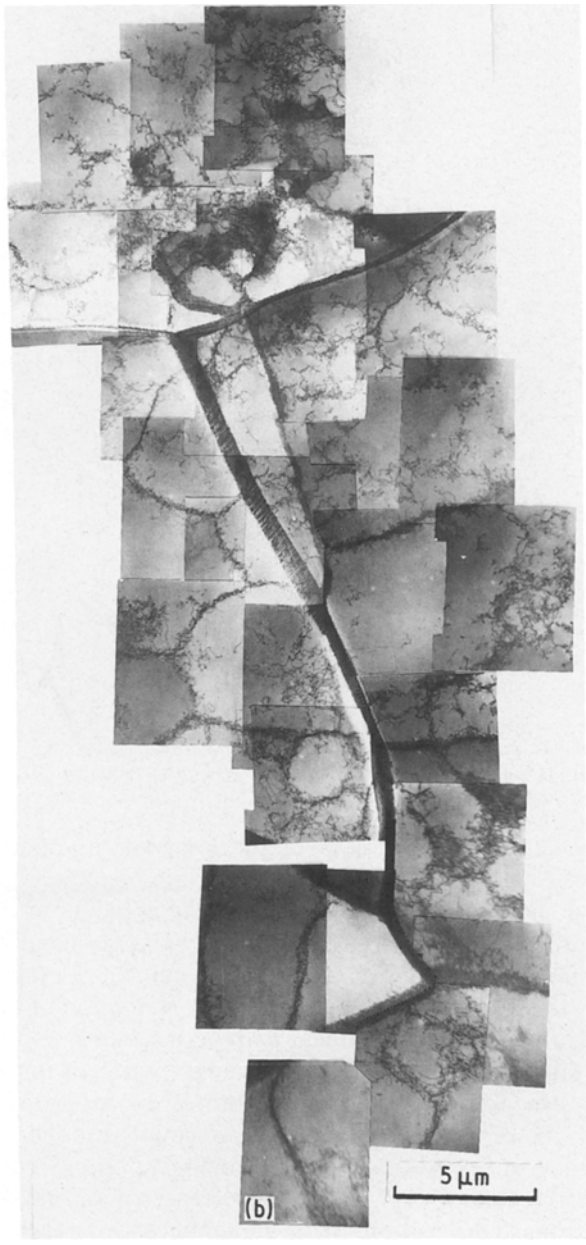


Figure 5 Dislocation structure around a grain boundary of nickel after testing at 600° C with 0.5% total strain amplitude for 160 cycles.





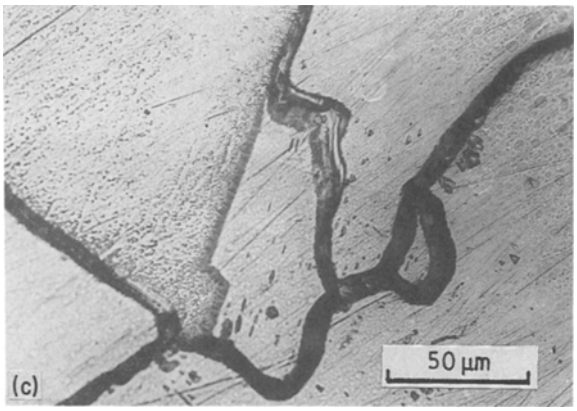
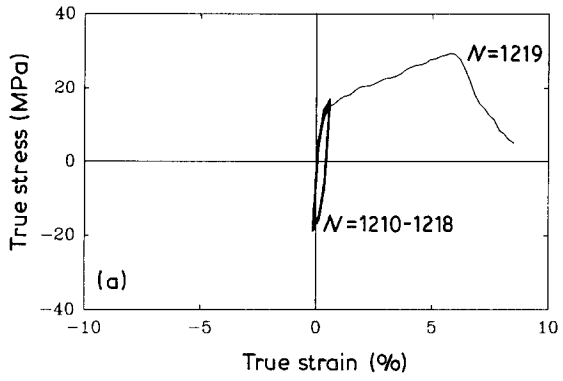


Figure 6 (a) cyclic hardening curve, (b) dislocation structure around a grain boundary, and (c) optical micrograph of nickel tested at 600° C with 1.7% (a, b) and 0.5% (c) total strain amplitude.



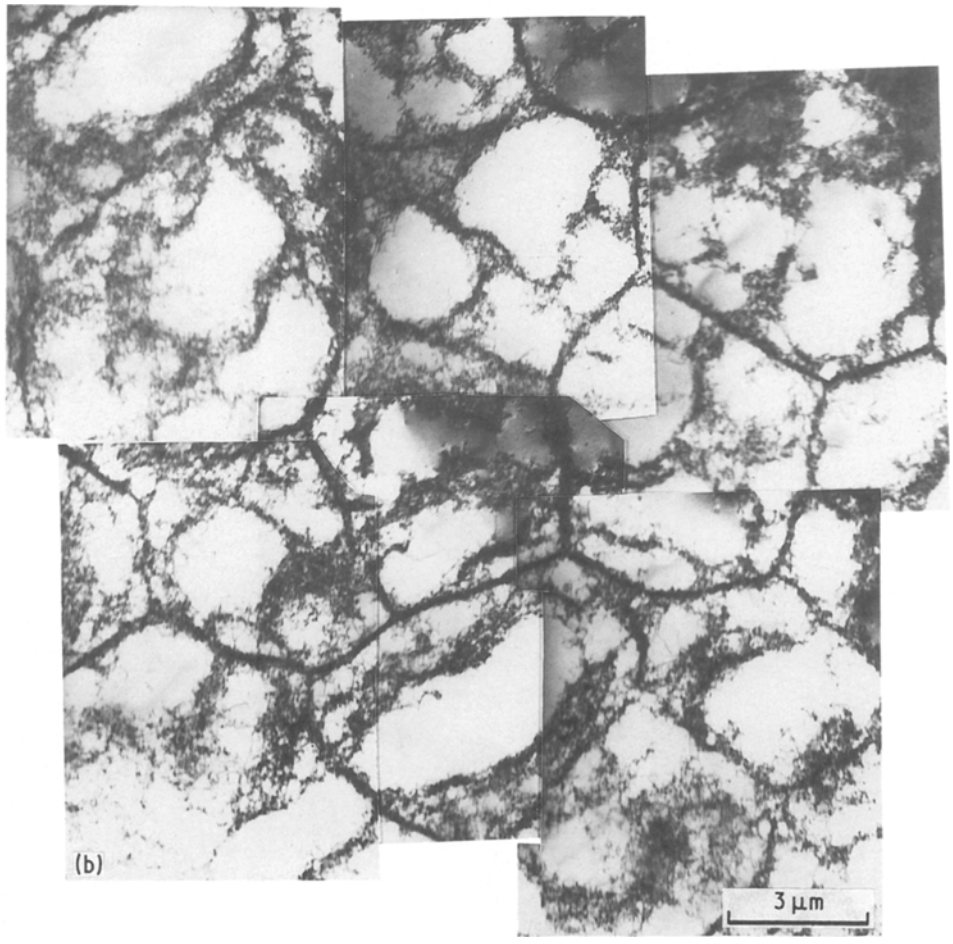


Figure 7 (a) True stress against true strain curve during cycles 1210-1218 and (b) corresponding dislocation structure of nickel tested at 600° C with 0.5% total strain amplitude. The test was terminated during the extended tensile cycle no. 1219.

structure on both sides of the boundary is poorly recovered. This indicates that the grain boundary did not move in only one direction, as apparent from surface investigations, but seems to have oscillated or changed directions during the large number of cycles.

In essence we conclude from the observation of the structural gradient along a path perpendicular to the grain boundary, that boundary migration leads to the destruction of the previously formed dislocation cell structure, which subsequently is rebuilt through all states of cell development. By analogy, one may conjecture that the patchwise occurrence of disorderly arranged dislocations in the grain interior may also be the result of dynamic changes in the microstructure owing to concurrent deformation.

The dislocation density and arrangement gradients produced by grain-boundary migration during cyclic deformation may actually be the cause for the recently reported DRX phenomena during HTLCF [6, 11]. The microstructure in Fig. 6b is due to nickel polycrystal deformed at a large strain amplitude, namely $\Delta\varepsilon = 1.7\%$, to 300 cycles. The cyclic hardening curve (Fig. 6a) indicates the occurrence of DRX and grain-boundary migration as apparent from the distinct maximum of the flow curve [6]. The vicinity of the vertical boundary in Fig. 6b comprises areas with more recovered cell arrangements (left to the boundary, especially at the bottom) and more disorderly arranged dislocations (to right of boundary, especially at the top). In the latter area a twin is created at a

triple junction (most likely during the migration of the vertical grain boundary to the left) and the steps in the twin boundary indicate migration activity. Also interesting is the area next to the junction of the twin boundary with the horizontal boundary. A small grain seems to have developed there right adjacent to a heavily tangled dislocation arrangement. The resemblance of this arrangement with nucleation phenomena observed in dynamically recrystallizing nickel during HTLCF is obvious (Fig. 6c), and supports the hypothesis that DRX phenomena are due to the structure gradients in the wake of moving grain boundaries. This argument would also comply with the observation that DRX does not occur during HTLCF of single crystals [12].

Of particular interest with regard to the stability of dislocation structures is their accommodation of strain path changes, especially if such changes lead to a different steady state dislocation arrangement. Figure 7 gives an example that dislocation rearrangements due to changes of strain path can occur without disruption of the previously established structure. After 1218 cycles at 600° C and $\Delta\varepsilon = 0.5\%$ deformation was continued only in tension to $\varepsilon = 8\%$ (actually incidentally due to malfunction of the machine control). The maximum of the tensile flow curve indicates the occurrence of large scale DRX at 6% tensile strain (Fig. 7a). The dislocation structure after this additional tensile strain (in the unrecrystallized volume, of course) reveals the superposition of two cell structures. The well recovered cell structure was produced during cyclic deformation and is still retained, while a new cell structure, still much less recovered, is generated within the old structure. The new cell structure tends toward a smaller cell size than the fatigued structure, because of the higher flow stress [3, 4, 6]. No disruptions or discontinuities of the previous structure are apparent, rather both structures seem to be able to coexist, and gradually the former cell walls become incorporated in a new homogeneous arrangement. Hence, Fig. 7b reveals two different stages of dislocation cell structure development and indicates that the specimen actually retains a "temporal memory" of its strain history in terms of its dislocation arrangement, until a new structure is fully developed.

4. Conclusions

The development of the dislocation arrangement during HTLCF of nickel polycrystals was investigated, with special emphasis on the dislocation structures in the vicinity of grain boundaries. The following results were obtained.

1. With progressing cyclic deformation a cell structure develops; the cell boundaries become increasingly condensed and the cell interior become free of dislocations.

2. Dislocation structures near grain boundaries indicate that the migration of grain boundaries destroys the previously established cell structure and that a new cell structure is generated through all development stages involved.

3. Grain boundary motion during cyclic deformation leads to a gradient in dislocation density and arrangement on a path away from the boundary. This gradient seems to provide potential nucleation sites for DRX during HTLCF.

4. Strain path changes do not lead to sequential destruction and rebuilding of the cell structure. Rather the new cell structure is continuously superimposed to the former structure until a homogeneous structure is attained.

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