In-situ TEM study of dislocation patterning during deformation in single crystal aluminum

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Abstract. The evolution of dislocation patterns in single crystal aluminum was examined using transmission electron microscopy (TEM). In-situ tensile tests of single crystals were carried out in a manner that activated double slip. Cross slip of dislocations, which is prominent in all stages of work hardening, plays an important role in dislocation motion and microstructural evolution. In spite of the limitations of in-situ straining to represent bulk phenomena, due to surface effects and the thickness of the samples, it is shown that experiments on prestrained samples can represent the early stages of deformation. Transition between stage I and stage II of work hardening and evolution during stage III were observed.

1. Introduction

Patterning of dislocations into cell structures is common to many metals during plastic deformation during stage III of work hardening. The extensive research devoted to this phenomenon has revealed many of its characteristics [1-5].

The first stage of work hardening is characterized by dislocation glide, referred to as easy glide, where only one slip system is active. The transition between stage I and stage II of work hardening is believed to evolve due to cross slip of dislocations as a result of different barriers for dislocation glide, eventually causing the formation of dislocation tangles [1-3]. The tangles form dislocation cells by dislocation’s rearrangement into self-screening structures, in order to minimize the stored energy per unit length of the dislocation lines. Stage III is known as dynamic recovery by the increase of dislocations within cell boundaries, increasing misorientation between adjacent cells and a decrease in cell size [1, 6]. In all stages of work hardening cross slip plays an important role in the dislocation’s rearrangement into different patterns [5]. The ability of the dislocations to cross slip depends on the stacking fault energy and the activation energy for cross slip determines the ease at which typical dislocation patterns will form [7-8].

In this research in-situ transmission electron microscopy experiments were performed in order to study mutual interactions between dislocations and the collective behavior of dislocations to form typical patterns during deformation. Moreover, the validity of in-situ TEM experiments relative to bulk experiments was discussed.
2. Experimental

High purity (99.999%) aluminum single crystals [011] discs were cut into dog-bone shaped samples. The samples were prestrained in an Instron type machine to different strains (10-15%). The samples were prestrained in an orientation that assured the activation of two slip systems in \(<221>\) directions [4]. From these samples 5.50x1.68mm² plates were cut and polished to 100µm. Final thinning was achieved by jet electro-polishing in a solution of 70% methanol and 30% HNO₃ at a temperature of \(-15\)°C.

In-situ tensile tests were performed in a JEOL-1200EX microscope equipped with a double tilt stage \((x=\pm30°, y=\pm10°)\). A detailed description of the experimental setup is given in [9].

3. Results

The initial microstructural evolution following low levels of strain, shown in figure 1, consisted of a stage I to stage II transition microstructure. The dislocations observed were mostly easy glide dislocations. The dislocations were jogged and amongst them some dislocation loops were noticeable. Figure 1(a) shows the typical microstructure of a sample prestrained to 10% in the \([\bar{2}\bar{2}1]\) direction. This direction was chosen in order to activate only two slip systems. The dislocations were characterized using invisibility criterion in two beam conditions as \(\frac{1}{2}[\bar{1}\bar{1}0]\) and \(\frac{1}{2}[101]\). The tensile axis (TA) is marked on the micrograph.

Figure 1. Transition between stage I and stage II of work hardening. Black arrows show the direction of dislocation movement. Slip traces are circled in the micrographs. The area denoted A is the same in all micrographs. (a) Dislocations parallel to the foil surface. (b) The dislocations cross slip and begin to accumulate the form of a tangle. (c) Some of the dislocations cross slipped into the tangle. (d) Dislocation tangle formed at area denoted A.
When force was applied, the dislocations bow out in $\Sigma 1$ trace direction and cross-slip towards the area denoted as A in Figure 1 (same area in all frames) to form a tangled dislocation boundary. Pre-existing dislocation loops and dislocations act as pinning points and barriers for dislocation movement. The direction of the movement and the cross slip can be observed via the slip traces (circled on the micrographs).

Figure 2 shows the evolution of a dislocation boundary by the cross slip of dislocations and the accumulation of dislocations within a cell boundary at stage III of work hardening in single crystals. The sample was prestrained to 13% in the $[\Sigma 2]$ direction (TA) and the dislocations were characterized as $\{\frac{1}{2}[110]\}$ and $\{\frac{1}{2}[10\overline{1}]\}$. As force is applied, dislocations cross slip towards the boundary denoted as A on figure 2(a), from both sides of the boundary. The activation of two slip systems is noticeable in the two sets of slip traces inside the cells. A change in contrast between the upper cell and the lower cell in figure 2 is observed with the increase of strain, comparing figures 2(a) and 2(c), as a result of the misorientation between the two volume elements. These processes continue until the volume of the cell is depleted of dislocations.

![Figure 2](image)

**Figure 2.** Evolution of a dislocation boundary at stage III of work hardening. Black arrows show the direction of dislocation movement. Area denoted A is the same area in all micrographs. (a)-(d) frames showing the depletion of the cell’s volume from dislocations into the cell’s boundary by cross slip of dislocations. Slip traces are circled in the micrographs.

In both cases, it is seen that cross slip of dislocations is necessary for the formation of typical dislocation patterns. The processes observed in the in-situ experiments conform to the common belief that cross-slip of dislocations is necessary for the formation of typical dislocation patterns.
4. Discussion
In-situ tensile experiments were performed on [011] single crystal aluminum in <221> orientation in order to study dislocation interactions and the formation and evolution of typical dislocation patterns. Furthermore, the possibility of imaging bulk deformation processes in thin TEM samples was examined. The samples were prestrained prior to in-situ experiments in order to form an initial microstructure that minimizes surface effects. In-situ tensile experiments show the transition between stage I and stage II of work hardening by the cross-slip of dislocations to form dislocation tangles and cross slip of dislocations in stage III to form dislocation cells. This evolution was extensively modeled and experimentally observed, mostly in post-mortem observations of deformed samples [1-8].

In order to get a comprehensive understanding of dislocation interactions, dynamic behavior during deformation processes and correct sequence of events during microstructural evolution, post-mortem observations are not merely enough, and in-situ observations are essential to get a full picture. However, there are key limitations to in-situ TEM experiments that make the dynamic processes observed questionable in whether they reflect bulk behavior [10-12]. These limitations emanate from the limited thickness of the sample and surface effects. In spite of the above mentioned limitations, we have shown that the microstructural evolution of thin prestrained TEM samples is similar to that of bulk samples. The transition between stage I and stage II of work hardening occurs via the cross slip of gliding dislocations. Furthermore, the evolution of the cellular structure is by the depletion of the cell’s interior.

5. Conclusion
In-situ tensile experiments can contribute to the understanding dislocation interactions and microstructural evolution sequence during deformation. We have shown that dislocation cross slip is essential for the transition between stage I and stage II of work hardening, in the formation of tangled dislocation boundaries and the depletion of cell’s interior during stage III. The processes observed in in-situ experiments conform to the common knowledge and modeling concerning dislocation pattern formation and evolution in bulk samples.

6. References
[1] Kuhlmann-Wilsdorf D 1989 Mat. Sci. Eng. A 113 1
[2] Kuhlmann-Wilsdorf D 1987 Mat. Sci. Eng. 86 53
[3] Sevillano J Aernoudt E 1987 Mat. Sci. Eng. 86 35
[4] Kawasaki Y 1994 Strength of Materials (ed. Oikawa et al. The Japan Institute of Metals) 187
[5] Jackson P J 1985 Prog. Mater. Sci. 29 139
[6] Swann P R 1963 Electron Microscopy and Strength of Crystals (ed. G Thomas and J Washburn, Interscience, NY,) 131
[7] Landau P, Shneck R Z, Makov G, Venkert A 2007 J. Mat. Sci. 42 9775
[8] Landau P, Shneck R Z, Makov G, Venkert A 2009 Mat. Sci. Eng. in press
[9] Dlabacek Z, Gemperle A, Gemperlova J 2004 Proceeding of the 13th European Microscopy Congress (ed. D. Schryvers, J.-P. Timmermans) 1 417
[10] Fujita H 1967 J. Phys. Soc. Japan 23 1349
[11] Tabata T, Yamanaka S, Fujita H 1978 Acta Metall. 26 405
[12] Hirsch P B, Howie A, Nicholson R B, Pashley D W , Whelan M J 1965 Electron Microscopy of Thin Crystals (Butterworth & co., London, England) 78

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