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Observation of a New Mechanism Balancing Hardening and Softening in Metals

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Plastic deformation of metals refines the microstructure and increases the strength through work hardening, but this effect of deformation is counterbalanced by dynamic recovery. After large strain, the microstructure typically shows a lamellar morphology, with finely spaced lamellar boundaries connected by triple junctions. Here, we report that mechanically assisted triple junction motion is an important contributor to dynamic recovery, leading to an almost steady state. Triple junction motion replaces two boundaries by one, while maintaining the structural morphology. The observation rationalizes both a decreasing work hardening rate and the approach to a dynamic equilibrium of structural refinement at large strains.

Keywords: Microstructure, Plastic Deformation, Work Hardening, Dynamic Recovery, Triple Junction

Plastic deformation of metals by dislocation-based mechanisms refines the microstructure and increases the strength through work hardening.[1–4] This effect of deformation is counterbalanced by a simultaneous removal of structural features, and is termed dynamic recovery.[5,6] Work hardening and dynamic recovery have been studied extensively both experimentally and theoretically over the last 50 years for metals deformed to low and medium strains,[7–13] and mechanisms have been suggested that satisfactorily explain the observed mechanical behaviour. These mechanisms are based on a subdivision of the deformed structure into cells delineated by dislocation boundaries. However, with the development of new processing routes allowing metals to be deformed to very high stress and strain levels, the typical cell structure develops into a structure subdivided instead by medium and high angle boundaries (HABs) almost similar to those typically observed in an undeformed metal however, on a much finer scale.[14–18] In parallel to this structural evolution, the strength increases at a much slower rate than that observed at low and medium strains, indicating a shift in the balance between work hardening and softening by dynamic recovery. Experimentally this shift has been underpinned by observations,[14,18–21] showing that the spacing of deformation-induced boundaries decreases more rapidly at small and medium strains and less rapidly at high strains when compared with the externally imposed shape change. These observations mark a clear transition in the mechanical and structural behaviour, which at low and medium strain has been analysed in detail.[13,22] In contrast, the high strain transition behaviour has not been explored in detail since the seminal work of Langford and Cohen,[14,23,24] who suggested that dynamic recovery may take place by stress or strain-induced migration of dislocation boundaries followed by cell rotation, thereby removing boundaries between neighbouring cells. This loss of cell walls was tentatively referred to the movement of so-called *h*-junctures, a process which is activated by plastic deformation.[14,23] However, the structure that their analysis is based on consists of only dislocation boundaries with misorientation angles less than 15° and is therefore different from typical high strain structures formed by a variety of processes as rolling, torsion, forging and drawing.[20,21,25,26] Such structures at very large strains are typically subdivided by both low angle boundaries (LABs; ≤ 15°) and HABs (> 15°) showing a lamellar morphology.

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In this study we therefore focus on dynamic recovery in high strain structures, motivated by the belief that direct observations of mechanisms can contribute to the understanding of this important materials problem.

Commercial purity aluminium deformed to large strains by cold rolling has been chosen as a typical example of high strain fcc (face-centred cubic) and bcc (body-centred cubic) lamellar structures, subdivided on a fine scale by extended boundaries.[27–29] These boundaries are connected at triple junctions (see Figure S1 in Supplementary Material), whose behaviour we study in detail. To achieve this, observations are first made in selected areas of aluminium AA1050 (99.5% purity) cold rolled to true strains 2, 4 and 5.5 (i.e. 86.5–99.6% thickness reduction), characterizing the deformation microstructures by electron channelling contrast (ECC) imaging and electron backscatter diffraction (EBSD) in a scanning electron microscope (SEM). We then introduce additional deformation by cold rolling the samples and re-examining the selected areas. By this technique we observe exactly the same areas by both ECC imaging and EBSD without further polishing of the sample surface (see Supplementary Material). These two SEM techniques are complementary: ECC has a higher spatial resolution, whereas EBSD measures the orientation of each scanned point (see Figure S2 in Supplementary Material). This process of ex situ deformation followed by re-observation can be repeated several times. In this way we are able to follow the evolution of the deformation microstructure in both the longitudinal section (ND-RD), and for some specimens in the transverse section (ND-TD), where RD, ND and TD are the rolling, normal and transverse directions, respectively.

After additional deformation of the rolled aluminium, the surface roughness (both in the longitudinal and the transverse sections) is enhanced and shear banding is clearly observed in some places at the free surfaces, where the shear plane is parallel to the TD and inclined 30–40° to the RD. In our analysis, we focus on smooth regions where there is no obvious shear banding. A comparison of the microstructure at the surface between additional rolling steps showed that the positions and misorientation angles of lamellar boundaries were almost unchanged. However, a careful comparison of individual lamella showed unexpected lateral motion of triple junctions linking the lamellar boundaries. Two examples are shown in Figure 1 for the sample deformed initially to a strain of 5.5. Figure 1(a) shows the evolution of the microstructure in a selected area in the longitudinal section before and after additional rolling with thickness reductions of 5% and 20%. The triple junction indicated by red arrows migrated along the RD during additional rolling, leading to a decrease in the length of the middle lamella (indicated by black arrows) and an increase in the thickness of the original neighbouring lamellae, and after 5% additional rolling, parts of two boundaries of misorientation angle ∼20° were replaced by a single boundary of misorientation angle 4°. Examination of larger areas in the longitudinal section reveals that for the strain 5.5 Al sample, after a 5% additional rolling about 4% of the area covered an EBSD map of 180 μm^2 was swept by triple junction motion, thereby almost completely cancelling out the microstructural refinement by rolling (cancelling out 80% of geometrical reduction). Figure 1(b) shows the triple junction motion observed in the transverse section of a strain 5.5 Al sample during additional rolling with a thickness reduction of 12%.
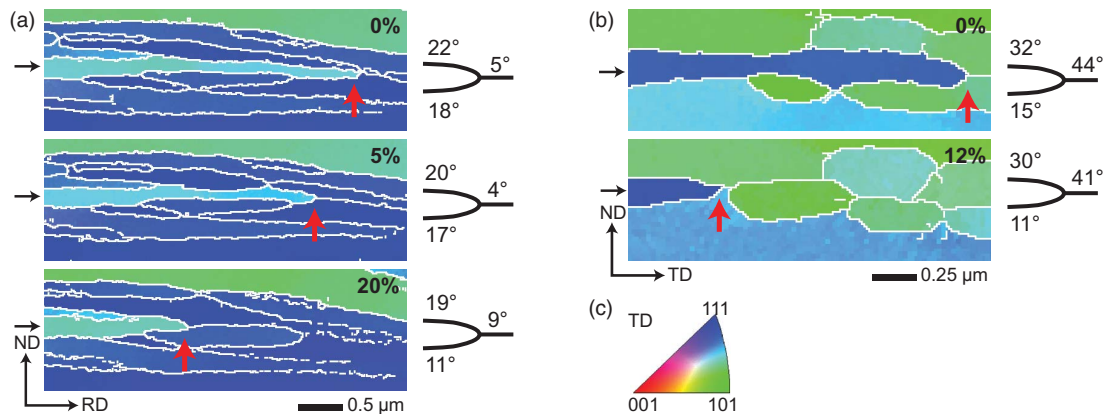
Figure 1. EBSD orientation maps showing triple junction motion observed in (a) the longitudinal section and (b) the transverse section of the strain 5.5 Al during additional cold rolling. The amounts of additional rolling are indicated within the maps. The two migrating triple junctions are marked by red arrows, and the corresponding shortening lamellae are marked by black arrows. Boundaries with misorientation angles larger than 2° are shown by white lines, and the maps are coloured according to the crystallographic orientation of the TD as coded in (c). The misorientation angles of lamellar boundaries forming the migrating triple junctions are indicated in the sketches.

Figure 2. EBSD orientation maps, with the same colour coding as in Figure 1, showing triple junction motion (red arrows) and break-up of a lamella (yellow arrow) as observed in the longitudinal section of a strain 4 Al sample during 5% additional rolling.

Triple junction motion was also observed in the strain 2 and strain 4 Al. In each case, the process reduces the rate of microstructural refinement. Besides the triple junction motion, break-up of lamellae also occurred during cold rolling. An example of break-up is shown in Figure 2, marked by a yellow arrow. The break-up of a lamella may be caused by localized shear deformation so that two lamellar boundaries meet each other (by shear banding in extreme cases). This creates a pair of triple junctions in the viewing plane, and is typically followed by migration of the triple junction pair away from each other.

From analysis of the ex situ observations in all samples, triple junction motion was found typically to take place at lamellae with relatively small lamellar spacings. For strain 5.5 Al, the average perpendicular spacing of the lamellae shortened in length by triple junction motion is $0.13\,\mu m$, whereas the average lamellar spacing is $0.24\,\mu m$, as measured by EBSD. Besides the lamellar boundary spacing, the crystal orientations (texture) of the lamellae and the misorientation angles of the adjoining lamellar boundaries may also play a role. However, analysis of the results obtained showed that the effect of these parameters on triple junction motion is not significant when compared with the lamellar boundary spacing. At a strain of 2, lamellae with non-rolling texture orientations bounded by HABs were found to be more frequently removed by triple junction motion. This may be related to the fact that these lamellae have spacings smaller than the sample average.

The ex situ observations capture triple junction motion on the sample surface. Although the in-plane migration may be not much affected by the free surface and well represents the bulk behaviour, it is appropriate to study the behaviour of triple junctions in the bulk

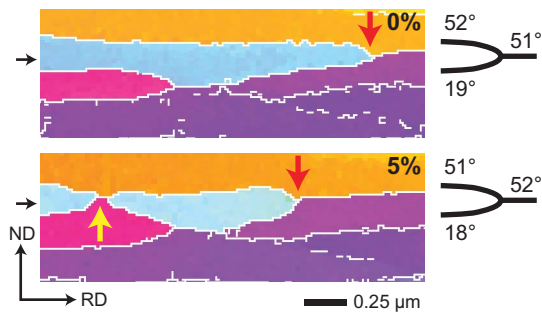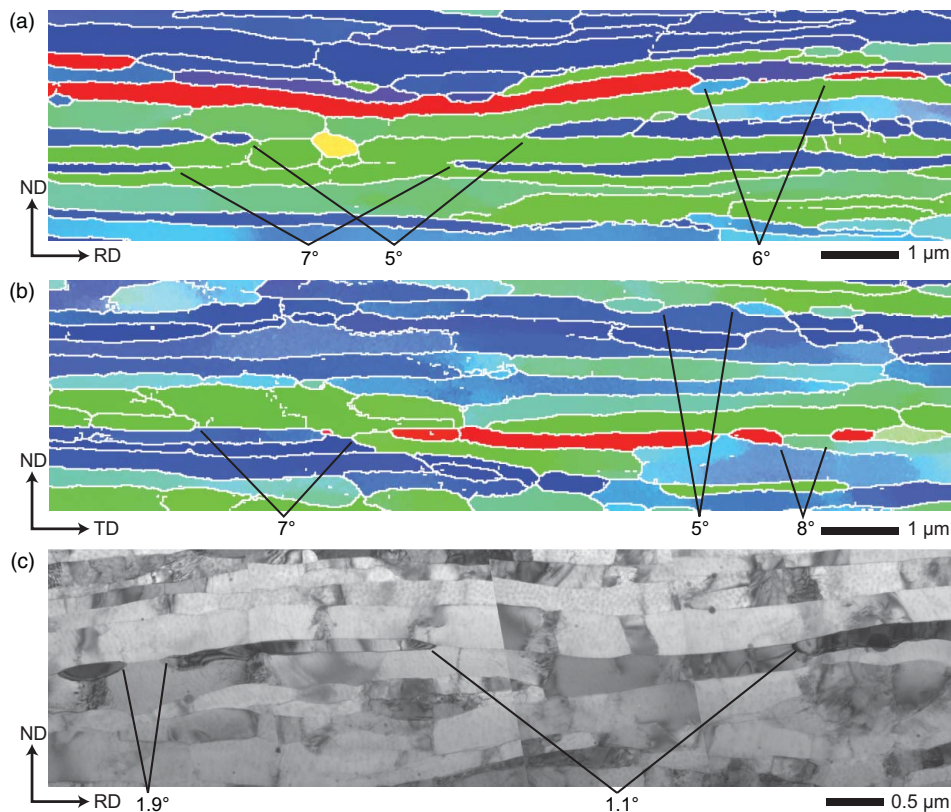Figure 3. EBSD orientation maps, with the same colour coding as in Figure 1, showing deformation structures in (a) the longitudinal section and (b) the transverse section of the Al sample deformed to strain 5.5. Separated pairs of lamellae with small misorientation angles marked indicate break-up of lamellae and triple junction motion in the bulk interior during deformation. (c) Similar observations by TEM.

interior during deformation. While direct observations are not possible at the moment, forensic evidence of triple junction motion can however be readily found in deformed samples in planes cut from the bulk interior. As shown in Figure 3(a) (viewed in the longitudinal section) and 3(b) (viewed in the transverse section), there are many pairs of separated lamellae with similar orientations. These configurations are likely a result of break-up of lamellae followed by triple junction motion in the bulk interior during deformation. The presence of such configurations was also confirmed by extensive forensic observations with transmission electron microscopy (TEM), and one example is shown in Figure 3(c). Triple junction motion may lead to the removal of an entire lamella or leave a relatively equiaxed grain. The small yellow grain in Figure 3(a) is likely a result of triple junction motion.

Triple junction motion as illustrated above has recently also been observed during annealing of highly strained aluminium, where thermally activated triple junction motion was found to be an important recovery mechanism, leading to uniform coarsening of lamellar deformed microstructures.[28] However, thermally activated triple junction motion only takes place very slowly at temperatures below 100°C [29] and thus cannot explain the present observations, where no significant temperature increase was observed during the additional cold rolling. We conclude therefore that the observed triple junction motion is primarily mechanically assisted, as stress and strain concentration are enhanced at triple junctions due to anisotropic plastic response from neighbouring lamellae. The stress and strain may reduce or overcome the energy barrier for triple junction motion and coupled boundary migration, although some thermal activation is always present. Through such motion the mismatch stress and strain around a lamella is reduced, two lamellar boundaries are replaced by one, and at the same time interconnecting boundaries and dislocations originally stored in the lamella that is removed are annihilated due to triple junction motion. As a result, this process reduces the amount of energy stored in the deformed metal, i.e. the mechanism can be classified as dynamic recovery.

After deformation to high strains, the microstructural evolution approaches a steady state [17,20] and typical features are: (i) the average lamellar boundary spacing is reduced but only to a small extent [18,26]; (ii) the average length of lamellae decreases slowly [30]; and (iii) the fraction of HABs saturates at 60–80%.[16,27] Triple junction motion during deformation clarifies these observations as follows: (i) triple junction motion provides a mechanism for removal of lamellar boundaries, cancelling out the effect of geometrical reduction (by compression and shear), resulting in an almost constant average lamellar boundary spacing; (ii) it allows a reduction in the length of lamellae, which together with break-up of lamellae

by localized shear, cancels out the geometrical elongation and leads to a slight decrease of the average lamella length; and (iii) it results in two lamellar boundaries being replaced by one, which can be an LAB as a texture effect, and which may be the cause of the relatively high fraction of LABs (20–40%) observed at large strains.[31] Due to triple junction motion in combination with other dynamic recovery processes such as dislocation annihilation in HABs, a steady state may evolve, as observed in high pressure torsion tests after a strain of 30.[17] The operation of triple junction motion is also in line with other observations. For example during cold rolling, finely spaced lamellae with orientations other than rolling texture components have the tendency to break up and disappear, with a concomitant strengthening of the texture, as for example observed in aluminium cold rolled to large strains.[27]

The present observations suggest that mechanically assisted triple junction motion is a key dynamic recovery mechanism, which can underpin the frequent observation [17–20] that both the evolution in structure and strength approach a steady-state level when a metal is deformed to high and ultrahigh strains. The extent of triple junction motion is strongly enhanced as the lamellar boundary spacing is reduced during deformation, which can be tentatively related to a reduction in the dihedral angles at lamellar boundary termination junctions.[28] Another important characteristic associated with triple junction motion is that such a motion results in a lateral movement of lamellar boundaries. Such a lateral boundary migration is in contrast to the previous suggestions, namely that lamellar boundaries migrate perpendicular to their boundary plane.[17,20] At present no theory or mechanism can rationalize the observed very rapid migration of boundaries during cold deformation, which is also observed at cryogenic temperatures.[20] Therefore, the current observations suggest that structure and properties of boundaries and junctions should be characterized in depth by conventional and high-resolution TEM, which is part of the ongoing research. The atomic mechanisms governing mechanically assisted triple junction motion are also unknown, and may differ from the mechanisms suggested to control stress-induced boundary migration in nanocrystalline materials,[32,33] where one possibility is coupled boundary migration and shear deformation.[34] However, such a coupling mechanism cannot dominate in the present case since coupling leads to triple junction motion in two possible directions, while in all cases triple junction motion is observed only to occur in one direction, namely that leading towards the removal of two connecting lamellar boundaries.

The present study has through direct observations identified dynamic recovery by triple junction motion as an important mechanism to be considered when studying the microstructural evolution and its relationship with the work-hardening behaviour of metals deformed

from medium to high and ultrahigh strains. Work hardening is typically analysed as stages, where stage IV is characterized by a small and constant hardening rate over an extended strain range. Stage IV has not been explored to a large extent,[22] but the observation of dynamic recovery by triple junction motion may be an important part of future analysis of this stage, and possibly also of later stages before the hardening rate has decreased to zero. This discovery of dynamic recovery by triple junction motion is therefore important in today's very active field of materials science and engineering with the goal of producing stronger and stronger metals and alloys by plastic deformation. By knowing more about this mechanism and how it can be reduced or suppressed, for example by introducing pinning forces from fine particles, it may be possible to design metals and process parameters in order to reduce the amount of redundant work (energy) required to refine the structure, and increase the strength, of industrial metals of tomorrow processed by plastic deformation.

Supplementary online material. A more detailed information on experiments is available at http://dx.doi.org/10.1080/21663831.2014.886308.

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