DISLOCATION / PRECIPITATE INTERACTION MECHANISMS IN 6xxx ALUMINIUM ALLOYS

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Abstract. Post-mortem transmission electron microscopy (TEM) investigations have been carried out to characterize the dislocation-precipitate interaction mechanisms of a compression-tested 6061-T6 aluminium alloy. The study was complemented by an approach combining image analysis by high resolution TEM which gives a direct measurement of the strain field around precipitates that is further introduced in the simulation of the dislocation propagation.

1. Introduction

Among aluminium alloys that have found application in automotive structures, as they offer an attractive combination of strength, formability and corrosion resistance, there are aluminium alloys of the 6xxx and 5xxx series. Because of the ever-increasing demand for high strength, low-cost materials, investigation of processing-microstructure relationships is strongly required. Heat treatable 6xxx aluminium alloys are of special interest for they offer hardening possibilities that lead to specific properties. More generally, if the understanding of the deformation mechanisms in the large plastic strains regime are essential for the reliability of constitutive laws that aims at the prediction of material behaviour during complex sheet metal forming processes, it is also clear that dislocation-precipitate interaction mechanisms in the domain of small deformations are needed to access the observed hardening.

In aluminium alloys improvement of the mechanical properties is classically obtained by the nanoscale precipitation produced by the decomposition of the supersaturated solid solution during ageing. The hardening effects result from dislocation interaction with the nano-sized precipitates acting as obstacles to the dislocation motion. The improvement of the metallurgical process and the use of heat treatable aluminium alloys as structural materials are then strongly linked to the understanding of the underlying dislocation/precipitate interaction mechanisms.

The metallurgical process that produces precipitates in heat treatable aluminium alloys is well documented. Basically it is a two steps procedure: first a supersaturated solid solution is produced by heating the material at a temperature where the phase diagram exhibits a maximum solubility, generally an eutectic temperature, followed by rapid quenching at room temperature. This step is then followed by an ageing procedure consisting in maintaining the sample at a room temperature (natural ageing or T4) or a higher temperature generally around 200°C (artificial ageing or T6) where a hardness peak is observed. This process produces precipitates evolving from the so-called Guinier-Preston zones to coarse incoherent precipitates (β) when the equilibrium is reached. The hardness peak is for the most part due to the presence of fine intermediate (coherent/semi-incoherent) precipitates (β′/β″) which harden the matrix upon a subsequent deformation process.
During the interaction process a dislocation pinned by the precipitates under applied stress will bypass or shear the precipitates. Actually, coherent precipitates are surrounded by an elastic field that results from lattice misfit with the matrix. This strain field interact with that associated with the dislocations. As a result, when the stress is high, dislocation loops are created that contribute to raise the overall dislocation density leading to the hardening effect. The mechanism underlying the bypass effect is either the Orowan process (bypass in the glide plane) or a climb process (bypass out of the glide plane). The parameters determining the deformation mechanisms are essentially the precipitate characteristics: morphology, size, density, structure, composition and the precipitate/matrix orientation relationships. Unfortunately, most of them are unknown for alloys of commercial interest since property optimisation lead to numerous solute additions and to supersaturated solid solution exhibiting complex decomposition sequences.

To access the deformation mechanisms of dislocation/precipitate in aluminium alloys, an approach avoiding approximations linked to the determination of the elastic compliances of the precipitate and the matrix is needed. To this end this study presents three complementary approaches carried out to tackle the deformation mechanisms in heat treatable 6xxx aluminium alloys. The study starts with the description and analyses of the dislocation/precipitate interaction by post-mortem TEM under weak beam conditions. Secondly a new approach combining image analysis and simulation of the dislocation motion is carried out. This is justified by the very fine scale and the large density of the hardening precipitates which strongly limits the possibility of imaging the dislocation/precipitate interaction. The analysis of HRTEM images allows a direct measurement of the strain field around precipitates. This is further introduced in the simulation of the dislocation propagation and allows to figure out which of the bypass or precipitate shearing mechanism is actually operating for different precipitates.

2. Results and discussion.

2.1. As-processed material. The geometry of precipitates in heat treatable aluminium alloys has been studied by means of High Resolution Transmission Electron Microscopy [1, 2]. In the T6 state, a large density of precipitates is observed (10^{15} – 10^{17} particles/cm^3 depending on solute contents). The precipitates show two morphologies: laths and rods with typical sizes. Figure 1 below shows the microstructure of the 6061-T6 alloys. In accordance with previous observations, it consists in a high density of fine precipitates (β’, β”) having specific orientation with the crystallographic axes. This projection along the [001] zone axis shows that the precipitates are oriented along the <002> directions of the aluminium matrix. Dislocations (D) are seen pinned by the non-equilibrium precipitates ((β’/β”) as well by coarse equilibrium β precipitates. But as it will be shown below, the efficiency in hindering the dislocations motion is due for the most part by non-equilibrium precipitates. It is difficult looking at this picture to state about the geometry of the precipitate. Actually the precipitation state in commercial alloy is very complex and display quite different scale : namely submicrometric and nanometric. Figure 2 shows the submicrometric precipitates which are usually imaged by Bright Field images in 6xxx alloys, these precipitates are equilibrium phases resulting from the addition of solutes in order to control the grain size and the recrystallization behavior. Indeed nanoscale precipitates can be imaged in 6xxx alloys only with HREM or Dark field images taken along a (001) zone axis. However, it is well established that the hardening precipitates in 6xxx alloys are laths and rods [1, 2]. These two morphologies are further illustrated in this paper by the Fig. 3b and HREM images in Fig. 4a and 5a. Moreover, the information given by HRTEM on the precipitate structure, though
incomplete, clearly shows that the precipitates exhibit a structure strongly departing from that of the matrix. Despite of the absence of a simple relation with the matrix lattice, these precipitates cannot be considered as incoherent since a strain field always surrounds the precipitates.

![Figure 1](image)

**Fig. 1.** a) Dark field image showing the general microstructure of as-processed 6016-T6 aluminium alloy. Notice the high density of fine precipitates that have probably nucleated at dislocation (D). Projection along [001] zone axis. b) Bright field TEM micrograph showing the submicronic precipitates also called dispersoids.

2.2. Deformed sample. After processing samples (3x3x8 mm³) for compression tests where prepared and strained at a strain rate of 10⁻⁴ s⁻¹ up to 2% total elongation. Figure 3a and b below show the induced deformation microstructure. Figure 3a contains some striking features of the dislocation/precipitate interaction mechanisms. Most of de dislocations are pinned by the nanosized precipitates (strong contrast P in the picture). At some place the dislocation contrast vanishes due to the interaction of its strain field with that of the precipitates. Numerous dipoles (A, and B) are also seen. This observation strongly suggests the occurrence the bypass mechanism by the cross slip of screw segments. As a consequence numerous prismatic dislocation loops are seen in the microstructure as it is shown in figure 3a (top left). Segments labeled i and j show an initial stage of the bowing out of a dislocation segment that lead to profusely observed dislocation dipoles. It should be noted that despite of a close investigation no concentric loops as expected by the operation of an Orowan-like bypass mechanism was seen. So it can be conclude here that the only visible deformation mechanism operating here is the bypassing by cross slip. It was not possible to image in the same time precipitate and dislocation. Nevertheless Fig. 3b, which is a weak beam image of a recovered sample, shows a lath-like precipitates. A shear offset (arrow) can be seen on the lath. It is then possible that both the bypass and shearing mechanisms occur during the deformation in the alloy studied here.
Fig. 2. Weak beam micrograph of the tested compression sample (projection along the [111] axis). Some striking features can be seen, namely dislocation dipoles (A, B), nanosized precipitates (P) and bowing out of dislocation segments (ij).

Fig. 3. a) Numerous prismatic dislocation loops as the consequence of the operation of the bypass mechanism. Notice the alignment and size of the loops with that of precipitate. Weak beam image parallel to (001). b) traces of laths and rods imaged by dark field taken along the [100] zone axis in the deformed sample showing traces of shearing

At a first sight the observation of extensive dislocations bypassing precipitate as the major mechanism is in disagreement with that were claimed elsewhere [3-5]). Indeed, because of the small size of the precipitate diameter and the pseudo coherency they develop with the matrix, dislocations are expected to shear the precipitates. In fact, either in the post mortem study [3] as well as the in situ observation [4, 5] shearing was not directly observed. On the other hand, stress strain curves carried out on AA6056 and AA6063 alloys show at room temperature a moderate but significant hardening [3,4]. Such behaviour is indeed not very consistent with precipitate shearing as the major mechanism. The moderate hardening would be indeed more consistent with the activation of several mechanisms such as shearing of laths and by passing of rods. However the difficulties of investigating in details the interaction of dislocation with
2.3. The strain field effect. The formation of loops is usually associated to large and/or incoherent precipitates. We are clearly here is the opposite case: small precipitates rather coherent with the matrix. In fact, as shown by the high deformation contrast on TEM images of 6xxx alloys, the strain field is remarkably strong with respect to the small precipitate size. This unusual behaviour suggests to investigate more closely the effect of the strain field on the propagation of dislocations using the combination of two recent developments in quite distinct fields: HREM image analysis and simulation of dislocation propagation. Since the so-called geometrical phase method is able to give the strain field created by a particle in the matrix [6], this experimental strain field can be further introduced in the simulation of the dislocation propagation [7]. The simulation uses the equations of the dynamics of dislocations, including short range and long range interactions and allows for any dislocation character. The combination of image analysis and simulation provide then a tool to examine the dislocation behaviour when approaching the precipitates. Such tool is particularly appropriate to our case since no hypothesis on the structure composition or the elastic properties of precipitates has to be used, the strain field created by the precipitates being the experimental one given by direct measurement. The geometrical phase method is based on the ability of HREM to provide an atomic resolution projection of the structure. The image of a crystalline area can be decomposed in a set of periodic components g. In a perfect lattice, the phase of a g-component is constant, while in a lattice distorted by a displacement field \( u(x,y) \), the phase is \( -2\pi g \cdot u(x,y) \). This property is a direct result of image formation in HREM. In each point of the phase map, the intensity is proportional to the displacement \( u(x,y) \), the scaling factor is simply the grey level full range for a fringe spacing. The method, which can be described as a numerical moire, is either a quantitative and a high accuracy tool. The phase images are here obtained following the method given by [6]. Its possibilities and limits of the method regarding the extraction of strain field of nanoscale precipitates have been examined in details [8].

Fig. 5 is an HREM image of the Al matrix and a lath shape precipitate in a 6056 Al alloy. Fig. 5b and 5c are the phase images for two Fourier components \( g_1 \) and \( g_2 \) selected from the FFT of the area (insert in Fig. 5a). As mentioned above, these phase maps simply correspond to the displacement fields \( u_1 \) and \( u_2 \). Notice that since \( g_1 \) and \( g_2 \) are not colinear, the phase maps allows for a complete determination of the displacement field in the plane of the micrograph but no information on the component of \( u \) perpendicular to the image. However, for precipitates elongated along one direction, which is hopefully the case for hardening precipitation in 6xxx alloys, the displacement field is very small, if not zero, along this direction, and thus the method leads to a complete determination of the displacement field. Finally, the strain and stress tensors components can be further derived from the displacement fields using the usual equations of elasticity. It is worth noting that the area studied by image analysis is typically 10-20 nm large, in order to be able to define a reference in an undeformed zone.
As expected for a lath, the deformation effect is stronger along the direction parallel to the width than to the lath length. Note that the longest lath direction is here parallel to the beam axis. A remarkable property of the image analysis is that it is able to measure very small strain field. The point was illustrated with simulated images of weakly deformed matrix [7]. Fig. 6a is a HREM image of a rod shape precipitate seen edge on, Fig. 6b and 6c are the related displacement maps. The strain field is much stronger than in the lath case, besides since the precipitate has a very uneven shape, the displacement maps do not have particular symmetry. This is a typical illustration of the interest for direct measurements of the strain field to study nanoscale microstructures of metastable particles.

By simple derivation of phase maps and using the elastic constant of the matrix the stress field is derived. The interaction of dislocations with each type of precipitate (lath or rod) can be then specifically addressed.
2.4. Simulation of dislocation propagation in the experimental strain field. The influence of stress fields on the mechanical properties can only be precisely taken into account by simulating its influence on the dislocations behaviour during deformation. In particular, the relative small stress field around laths compared with the surrounding stress field around a rod appears to be at the origin of a difference on the dislocation behaviour when bypassing the precipitates.

![Figure 6](image1)

Figure 6. Comparison of the simulated movements of a 1/2<110> dislocation under a constant applied stress in the vicinity of a rod or of a lath. The dislocation has been drawn for successive positions corresponding to a same constant interval of time. Notice that the shearing of the lath starts at the small end of the lath, which corresponds to the lowest stressed area of the matrix near the surface of the lath.

Fig. 7 shows the simulated movement of a gliding a/2<110> dislocation in the vicinity of a lath and should be compared with the behaviour of the same dislocation undergoing the same external stress but in the vicinity of a precipitate with a rod shape. In the case of a rod shape, the acting stress on the dislocation, that is the sum of the external stress and of the residual stress, is not sufficient for the dislocation to attain the precipitate, thus implying that the precipitate can only be by-passed by formation of a dislocation loop, the so-called Orowan process. In the case of a lath, as the residual stress is slightly smaller than around a rod, the dislocation is not stopped by the surrounding stress field and can shear the precipitate.

For this calculation, and in order to simplify the simulation of the shearing of the lath, two assumptions were made, both based on the fact that the cross section of the lath is small: (i) as the crystallography of the precipitate is unknown, it has been decided to consider that there is no change in Burgers vector of the dislocation nor glide plane while shearing, and (ii) the stress field inside the precipitate has been taken constant and equal to the value of the stress at the interface between the matrix and the precipitate. Notice that these two assumptions are not very important in the sense that the main result of the simulations is to show that the residual stress field is not sufficient to prevent the dislocation to attain the interface of a lath-shaped precipitate.

3. Conclusion

Deformation mechanisms in heat treatable 6xxx aluminium alloys are particularly difficult to study because of the density, scale and complex morphology of the precipitates which in addition consist of metastable unidentified phases. Therefore direct observations are necessary for the understanding of dislocation/precipitate interaction. TEM Dark field imaging of
deformed samples indicated either the formation of numerous small dislocation loop and the shearing of lath precipitates. Using HRTEM image analysis and simulation of dislocation propagation, we have analysed the impact of the precipitate strain field on the mechanisms. It comes out that the strain field is responsible for the bypassing of the rod while the shearing of lath is allowed. According to these first results, it seems very appropriate to investigate nanoscale precipitation hardening by the combination of HRTEM and simulation of dislocation propagation.

**References**