An approach of precipitate/dislocation interaction in age-hardened Al-Mg-Si alloys : measurement of the strain field around precipitates and related simulation of the dislocation propagation

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Abstract. TEM study of deformed samples complemented by a new approach combining image analysis and simulation of the dislocation motion have been carried out to study the precipitate /dislocation interaction in Al-Mg-Si alloys (AA-6XXX). The analysis of HRTEM images allows a direct measurement of the strain field around precipitates and is further introduced in the simulation of dislocation propagation. In the case studied here, the simulation indicates that for an applied stress close to the yield stress, dislocations' motion in the matrix occurs by both the by-pass of precipitates through the activation of the Orowan mechanism and the shearing of precipitates. This is in agreement with TEM observations on deformed samples showing numerous dislocations loops along with laths shearing

Introduction

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 such as the T6 tempering. Actually the hardening effects (both the hardening rate and the increase in the yield strength) result from the interaction of dislocations and the nanoscale precipitates which act as obstacles to the dislocation motion. Hence for the improvement and the use of heat treatable aluminium alloys as structural materials it is a challenge task to understand the precipitate/dislocation mechanisms that lead to the macroscopic behaviour.

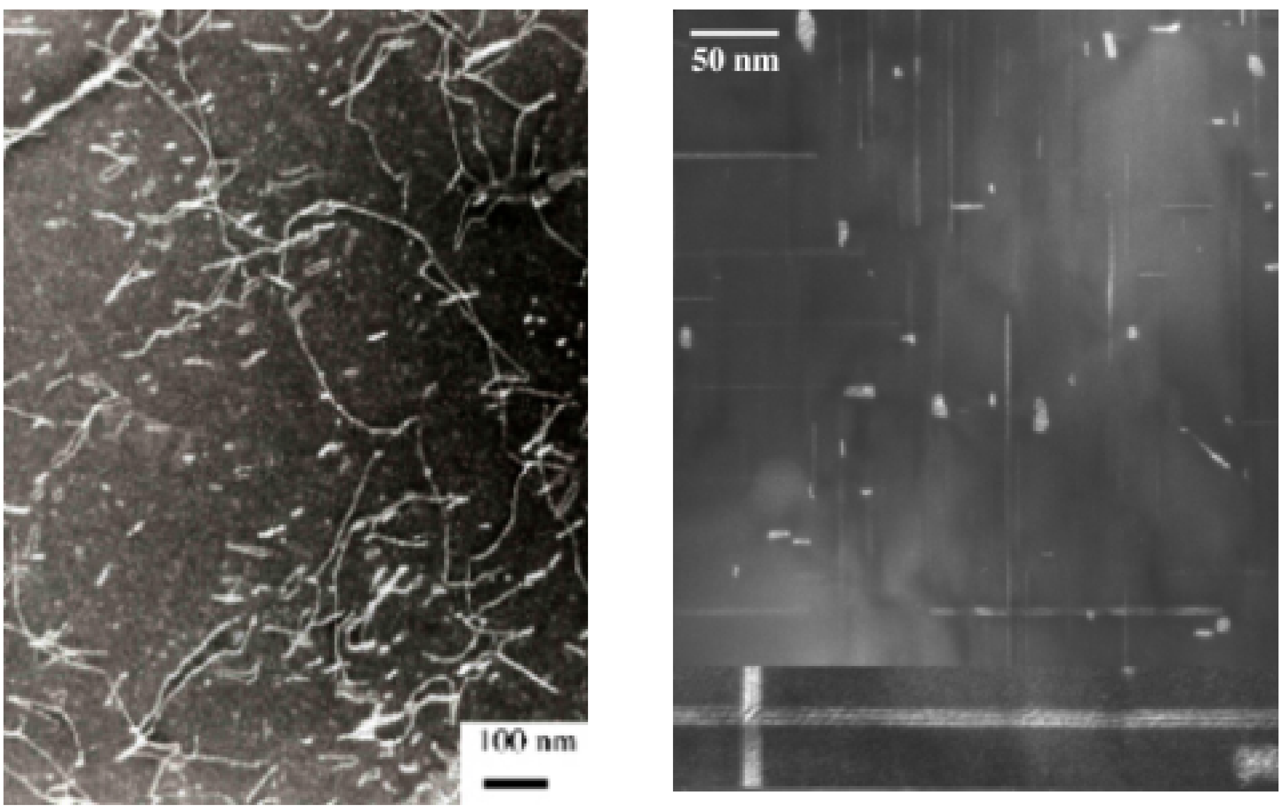
It is well known that during the interaction process dislocations pinned by the precipitates under the increasing of the applied stress will finally shear or by-pass the precipitates and depending on the case a macroscopic hardening effect will or not be observed. The parameters determining the deformation mechanisms are essentially the precipitate characteristics: morphology, size, density, structure, composition and precipitate/matrix orientation relationships. Unfortunately, most of these parameters are unknown for alloys of commercial interest since property optimisation lead to numerous solute additions and hence to supersaturated solid solution exhibiting complex decomposition sequences.

The decomposition sequence in the Al-Mg-Si alloys has motivated many works. Most of them were carried out by means of High Resolution Transmission Electron Microscopy (HRTEM) ([1, 2] and references there in). In the T6 state, a large density of precipitates is observed: 10^{15} –10^{17} particles /cm^3 depending on solute contents. The precipitates shows two morphologies: laths and rods, the typical size are $\Phi \sim 5$ nm, L ~ 50 nm for the rods and, L ~ 100 nm, l ~ 10 nm, e ~ 5 nm for the laths. The information given by HRTEM on the precipitate structure, though incomplete, still clearly shows that the precipitates do have a complex structure which strongly departs from that of the matrix. Despite of the absence of a simple relation with the matrix lattice, the precipitates cannot be considered as incoherent with the matrix since a strain field always surrounds the precipitates.

Actually, there are only few works on deformation mechanisms in AA 6XXX alloys [3, 4, 5]. The present paper is aimed at clearing up the dislocation/precipitate interaction in AA 6XXX alloys : it consists in a TEM study of deformed alloys complemented by a new approach combining the measurement of strain field around the precipitates and the simulation of the dislocations' movement through the measured strain field.

TEM study of deformed 6061 alloys

The precipitate/dislocation interaction in AA 6XXX alloys is particularly difficult to study because of the high density and the small size of the precipitates. Depending on the solute content, the density of precipitates can vary from an alloy to another for similar precipitate size. Therefore to limit the overlapping effect on the images, we have chosen an alloy for which the heat treatment leads to a moderate precipitate density. Following the heat treatment samples were compression tested up to 2% engineering deformation at room temperature and at a strain rate of 10^{-4} s^{-1}. Samples for TEM analysis were prepared using standard techniques and observations carried out under dark field conditions to tackle the precipitate-dislocation interaction mechanism(s).
The weak beam dark Field image in Fig. 1a shows the overall dislocation structure following 2% deformation: dislocations are extensively pinned all along by precipitates making it difficult for them to move as it can be seen by numerous bowing out segments.



(a) (b)

Fig. 1 : a) Weak beam images of the deformed alloy. Note the numerous small elliptical loops. b) Traces of laths and rods imaged by dark field taken along the (100) zone axis in the deformed sample. Some laths shows traces of shearing (see insert at the bottom of Fig. 1b at glancing angle)

The striking feature here is the high density of small and elongated dislocation loops just as large as the rod-like precipitates. This observation suggests that dislocations actually bypass precipitates through an Orowan like process. The shape of the loops might depend on the process (climb or cross-slip) as reported elsewhere [5]. More large loops (see for example Fig. 1a) and dislocation dipoles were also profusely seen. As for the possibility of precipitate shearing process during straining the imaging conditions under weak beam did not allow to visualise dislocation and the precipitates in the same time. Nonetheless, high magnification weak beam studies were carried out to investigate the structure of the precipitate after straining. As illustrated by the insert in Fig. 1b, , some laths exhibit traces of shearing after deformation. The rate at which precipitate shearing occurs and the quantification of the mechanism was not measured and investigated. Nevertheless, we do think that it occurs only locally compared to the bypassing process as discussed below. However, shearing offset on laths is clearly evidenced but the proportion of which seems to be low.

Because of their small diameter and the pseudo coherency, the precipitates are expected to be sheared the dislocations. This was more claimed than observed. In situ experiments or post mortem analysis did not directly show a such mechanism [3, 4, 5]. Moreover, the actual observation of extensive dislocations bypassing precipitates as the major mechanism is in disagreement with shearing as the major one but in accordance with the hardening effect seen in the stress-strain curves. However the difficulties of investigating in details the interaction of dislocation with nanoscale precipitates motivates to carry out the complementary approach developed in the next section.

Analysis of the strain field effect on the dislocation propagation

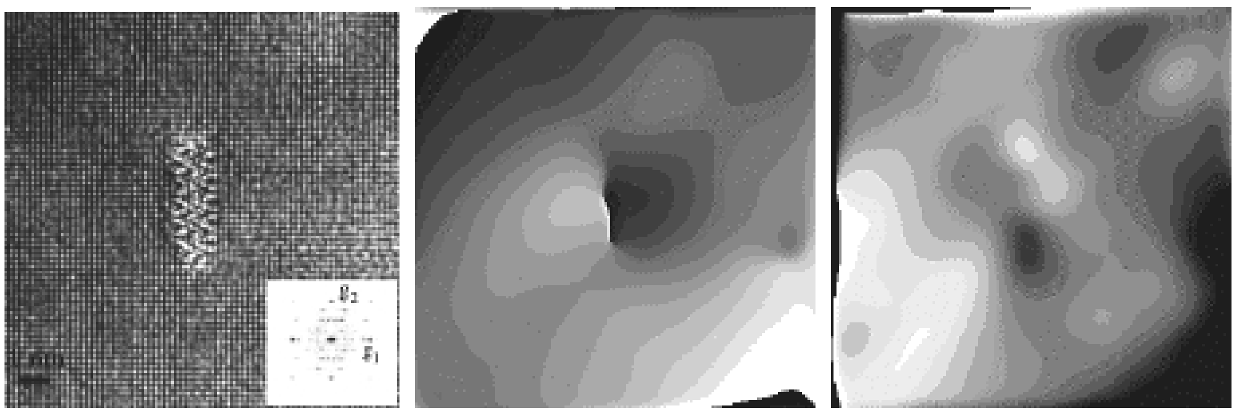
The formation of loops is usually associated to large and/or incoherent precipitates. We are clearly here in the opposite case : small precipitates rather coherent with the matrix (for an illustration see insert in Fig. 4). In fact, as shown by the high deformation contrast on TEM images of 6XXX alloys, 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: HRTEM 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.

Measurements of strain field from HRTEM images

The geometrical phase method is based on the ability of HRTEM 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 $-2\pi g.u(x,y)$. This property is a direct result of image formation in HRTEM. 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 “moiré”, is either a quantitative and a high accuracy tool. The phase images are here obtained following the method given by Hytch *et al.* [6]. The possibilities and limits of the method regarding the extraction of strain field of nanoscale precipitates have been examined in details in [8].

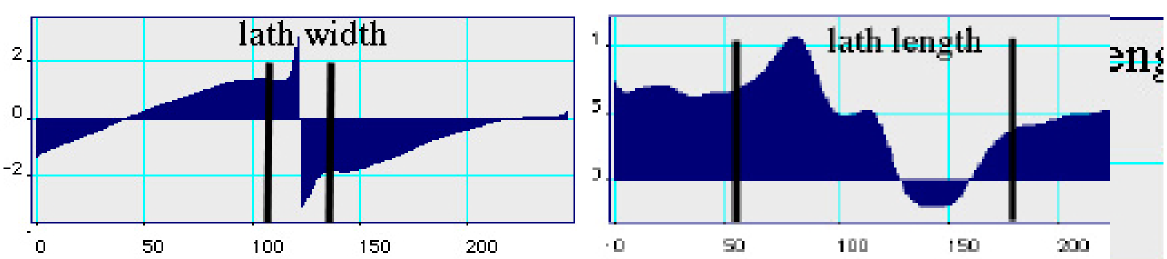
Fig. 2 is an HRTEM image of a lath shape precipitate in a AA6056-T6 Al alloy, the longest lath dimension being parallel to the beam axis. Fig. 2a and 2b are the phase images for two Fourier components g1 and g2 selected from the FFT of the area (insert in Fig. 2). As mentioned above, these phase maps simply correspond to the displacement fields u1 and u2. Notice that since g1 and g2 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 AA-6XXX-T6 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. 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



(a) (b) (c)

Fig. 2: a) HRTEM image of a lath precipitate embedded in the Al matrix (AA6056-T6) . b) Phase map obtained with the g1 selected vector c) Phase map obtained with the g2 selected vector

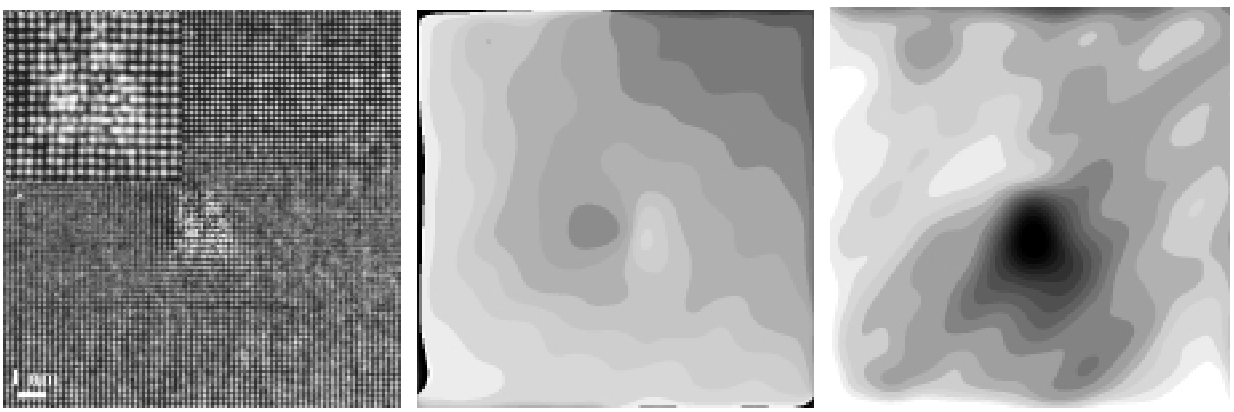
In Fig. 3, the profiles of the displacement illustrate the ability of the method to quantify the experimental strain field. Fig. 3a (resp. 3b) gives the displacement profile extracted from the map phase from Fig. 2b (resp. 2c) along horizontal (resp. vertical) lines running across the precipitates. On the profile, the phase ranging on $(-\pi, \pi)$ plotted is linearly related to the selected plane spacing (here $2\pi = 0.2$ nm). The phase rapidly oscillates when crossing the precipitate, this variation has no meaning and is simply due to the fact that the precipitate spatial frequency do not match the one selected to build the phase map.



(a) (b)

Fig 3: a) (resp. b) Profile of the displacement taken along a horizontal (resp. vertical) line cutting the lath. The phase range between $(-\pi, \pi)$ which is linearly corresponding to a displacement 0.2 nm.

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], it is here illustrated by the profiles measuring the displacement component along the lath length and width.
Fig. 4a is a HRTEM image of a rod precipitate seen edge on, Fig. 4b and 4c are the related deformation maps. The strain field is here 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.



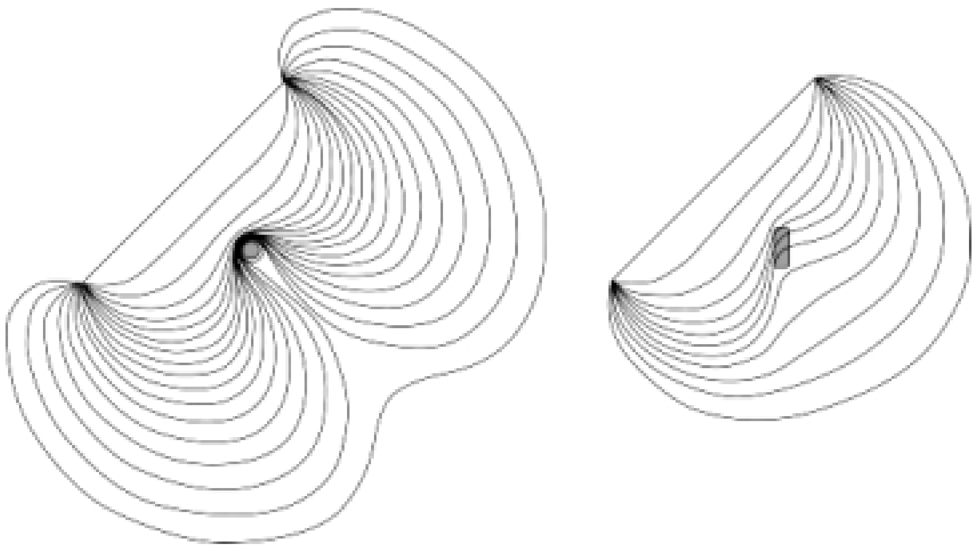
(a) (b) (c)

Fig. 4 : a) HRTEM image of a rod precipitate embedded in the Al matrix (AA6056-T6). As illustrated by the insert, the precipitate structure is quite ill defined and the matrix is highly deformed by the rod precipitate. b) Phase map obtained for a g1 direction c) Phase map obtained for the g2 direction. Fig. 4c clearly shows the asymmetry of the displacement field due to the rod.

By simple derivation of phase maps and using the elastic constant of the matrix the stress field is derived. The interaction of dislocations with lath or rod can be then specifically addressed.

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.



(a) (b)

Fig. 5 : Comparison of the simulated movements of a 1/2<110> dislocation under a constant applied stress in the vicinity of a rod a) or of a lath b). The dislocation has been drawn for successive positions corresponding to a 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. 5 compares the simulated movement of a gliding Ω<110> dislocation in the vicinity of a rod or of a lath under the same applied stress. 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.

Summary

Deformation mechanisms in AA 6XXX-T6 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 by passing 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 TEM study of deformed samples and the combination of HRTEM to simulation of dislocation propagation.

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