Nanoscale AFM and TEM observations of elementary dislocation mechanisms and implications for material science.

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Abstract of Doctoral Thesis

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### Authors publications

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1 Introduction

Transmission electron microscopy (TEM) is a traditional technique for the study of dislocations in the bulk material. On the other hand, the atomic force microscopy (AFM) has been so far only rarely applied to the study of dislocations emerged on the surface of deformed material. The correspondences of features generated on the surface to the traditional understanding of dislocation mechanisms are not yet well established. Therefore, the aim of this work is to compare and find correspondences in the observations of dislocation mechanisms by AFM and TEM.

The Fe–Al intermetallics were chosen as the material to be studied. This system has been studied for a long time, however, some of its peculiarities such as yield stress anomaly are still not satisfactorily explained.

The iron rich part of phase diagram is dominated by bcc based compounds with varying degree of order [1]. The bcc structure (α-iron) contains two equivalent positions in the middle and in the corner of the cubic cell. In the B2 structure these two sites are occupied differently. D0₃ structure at ideal 3:1 stoichiometry is a superstructure of alternating bcc and B2 cells. The smallest unit cell corresponds to a 2×2×2 stack of 4 bcc and 4 B2 unit cells.

Deformation of bcc metals is controlled by thermally activated creation and migration of kinks [2, 3] on \( b = \frac{1}{2}(111) \) screw dislocations. Peierls stress of these dislocations is particularly high because their core is supposed to be split on multiple planes [4, 5].

In B2 structure the \( b = \frac{1}{2}(111) \) dislocation creates an antiphase boundary. A pair of \( b = \frac{1}{2}(111) \) dislocations is needed to restore the chemical order. In D0₃ structure four \( \frac{1}{4}(111) \) dislocations (equivalent to \( b = \frac{1}{2}(111) \) in bcc due to double lattice parameter) are needed to restore the order and two different kinds of APB are formed between them (fig. 1).

![Figure 1: Structure of perfect superdislocation in D0₃ lattice.](image)


3. **Experimental methods**

By itself, the step stretching experiment is lethal to the sample. However, if the step stretching experiment is performed in a controlled manner, it can lead to a significant increase in the deformation of the sample. This increase is accompanied by a significant increase in the stress of the sample. As the deformation increases, the stress of the sample also increases.

It is important to note that the step stretching experiment is not a safe procedure. The sample must be carefully monitored and controlled to ensure that the deformation does not exceed safe limits. In addition, the sample must be cooled to maintain its structural integrity.

The strength of the metals decreases monotonically with increasing temperature. In order to investigate the deformation behavior of the metals under different conditions, the step stretching experiment is performed at various temperatures and pressures. The deformation behavior is then studied and characterized.
parameterized by the height \( h \), position \( p \) and half-width \( w \) superimposed on a parabolic background (coefficients \( a \), \( b \), \( c \)):

\[
H(x) = \frac{h}{2} \tanh \left( \frac{x-p}{w} \right) + ax^2 + bx + c
\]

Parameters \((h, p, w, a, b, c)\) are obtained independently for every scan-line segment. Variations of the step shape along the slip line are thus determined. To suit the final application, resulting dependencies of height, width and position can be further manipulated. The fact that individual scan-line segments are fitted independently contributes to the robustness of the method.

### 2.2 Stereo reconstruction and Kikuchi line fitting

Often it is of interest to describe in three dimensions the dislocation structures observed in TEM. Tomography methods applied to dislocations \([17, 18]\) are experimentally challenging even with the state of the art equipment. Here a less demanding method is developed. Only a handful of projections (at least two) are needed to reconstruct the dislocation geometry if one dimensional nature

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**References**


3.1. Deformation

3 Results

of assessed directions.

of matrix between set of vectors detected from multiple parts and get
transformation (19) and inverse transformation (20) is used to do the rotation
transformation. The detected and analyzed triplets are deformed using inverse
matrix and then transformed using matrix rotation. However, this work
determined the orientation from a reference frame, the camera position, and
in principle the rotation matrix. A proper projection can be read

get equations (19) can be solved in the least squares sense.

projection from (19) space are assumed into a space of the
position projection (20) space are assumed into a space of the
inconsistencies with low numbers. This means for coordinates of these
interactions on multiple projections must be function and
point recognition on multiple projections that are interactions and
point recognition. Displacements are modeled as straight lines between

4 Conclusion

Dispersion of plane mismatches, parallel was then explained using the geometric model based on
plane stress (1:0.75 strain order). In both the AVG and the
the plane stress strain order. AVG can be observed as discontinuities in the
stresses. This can be observed as discontinuities in the
ages. During the image acquisition (< 10 min the stress in
aging was preferably stopped in order to acquire AVG images.
Displacements were preferably stopped in order to acquire AVG images.
Recorded strains on curves are shown in figures 20 a.

in single, generally good agreement was found between

Next the TFA was used to process firstly the deformations.

The strain energy resisted to plane stress.

The AVG is shown. However, high showed change of ship

where AVG was 100 adjusted. Moreover, AVG showed change of ship

the AVG after the deformation. Summarize of

parts that are used as deformation.

ship crosses in position to cross slip in AVG (14) ↓AVG

since the record of the interaction are not introduced in this work,

principal applications of the interaction are not introduced to the

ALGO showed extension strip and added at the ends a form the

ALGO showed extension strip and added at the ends a form the

strain energy resisted to plane stress.

Since calculated for AVG to ALGO and for ALGO were detected in.
3.5 Carbides

Particles of $\kappa$-AlFe$_3$C carbide phase were found in all three investigated alloys (fig. 11). Carbide particles have rod-like morphology. They often form clusters with particles pointing in different directions. Figure 14 shows orientations of long axes of some 50 particles folded into the standard orientation triangle. It can be seen that the orientation of long axis of carbide particles with respect to the FeAl matrix is not random. An analytical model looking for the directions of best match in moire patterns of overlapping lattices of matrix and carbide can satisfactorily explain the distribution of orientations of particle axes.

Figure 3: Deformation curves (a–c) and yield stresses (d) of Fe–Al$_{13}$, Fe–Al$_{18}$ and Fe–Al$_{20}$ deformed in compression. Yield stresses were determined using 0.2% strain offset method.

Figure 14: Orientations of long axis of carbide particles with respect to FeAl matrix. Data points were determined from the 3D reconstruction of stereo images. Blue line is a result of carbide orientation model.
substrates were deformed.}
3.4 Fe–Al<sub>40</sub>

TEM of undeformed Fe–Al<sub>40</sub> specimen showed presence of square dislocation loops around carbide particles (fig. 11). These were formed by the excess vacancies eliminated during low temperature annealing.

Loops were determined to lie in the planes of \{100\} type with edges along directions of (100) type. This geometry of straight dislocation segments is suitable for the reconstruction of 3D structure. Figure 12 shows particles represented by their long axes together with dislocation lines. It can be seen that segments of square dislocation loops on different \{100\} planes tend to encircle the particles. Figure 13 shows the directions of dislocation line segments. Clear dominance of (100) directions is observed.

Figure 5: Post-mortem images of bow tie structures at two different magnifications. Parallelogram in (b) shows the region selected for further analysis using step fitting method (fig. 6).

(a)  
(b)  

Figure 6: Detailed analysis of a single bow tie.

(a)  
(b)  
(c)  

Figure 11: Square dislocation loops around impurity particles. \( g = (220), d \approx [001] \).
fully at V5A peak compressive (1
crystallography directions. Slip deformation bands become finer and
finesse, while incorrect deformation bands give rise to
imperfections. While incorrect deformation bands appear discrete on X-ray
crystals, different directions which they appear on X-ray crystal
clearly show the slip bands corresponding to (101) slip planes on these three samples (101), 200 and 700°C. The secondary plane was 20° away from the primary plane in (111) plane (Fig. 2a). A prominent slip band parallel to (101) plane (Fig. 2b), in Cr, Al, from slip bands form at temperatures below V5A peak show a
Figure 2: Secondary reduced pole figure showing the directions

the primary slip bands. The band shows the secondary slip bands created from the lip of
Oh, a propagation of the respective primary slip observations. The 

AFM observation of deformed Fe-Al° revealed numerous slip bands

3.3. Fe-Al°
and (211) planes are observed at 800 K. At 900 K only very faint structure is visible (fig. 8e).

![Images](image1.png)

(a) Room temperature  (b) 500 K  (c) 700 K  (d) 800 K  (e) 900 K

Figure 8: AFM images of Re-Al23 samples deformed at selected temperatures.

TEM of a sample deformed at 900 K showed homogeneous distribution of dislocations. Observations performed in thicker areas showed interconnected network of interacting dislocations. Three dimensional structure of a part of this network was reconstructed (fig. 9).

![Images](image2.png)

Figure 9: Images used to reconstruct the 3D model overlayed by the view of the model itself. Line colors are based on the segment crystallographic direction (fig. 10).

Some insight into the nature of the dislocations can be gained from their line directions. These are determined readily from the 3D model. Their distribution is shown in the figure 10, that also serves as the color key for figure 9.