Abstract of Doctoral Thesis

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Nanoscale AFM and TEM observations of elementary dislocation mechanisms

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Contents

1 Introduction ........................................... 6
2 Experimental methods ................................. 7
   2.1 Step fitting method ................................ 7
   2.2 Stereo reconstruction and Kikuchi line fitting . 8
3 Results .................................................... 9
   3.1 Deformation ........................................ 9
   3.2 Fe–Al_{20} ........................................... 11
   3.3 Fe–Al_{28} ........................................... 13
   3.4 Fe–Al_{40} ........................................... 17
   3.5 Carbides ........................................... 19
4 Conclusion ................................................ 20

References ............................................... 21

Authors publications .................................... 24
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 the thermally activated creation and migration of kinks [2, 3] on \( b = 1/2\langle111\rangle \) 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 \( b = 1/2\langle111\rangle \) dislocation creates an antiphase boundary. A pair of \( b = 1/2\langle111\rangle \) dislocations is needed to restore the chemical order. In D0₃ structure four \( 1/4\langle111\rangle \) dislocations (equivalent to \( b = 1/2\langle111\rangle \) 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-url)
Strength of bcc metals decreases monotonously with increasing temperature as the thermally activated nucleation of kinks on $\frac{1}{2}[111]$ screw dislocations becomes more efficient [2]. Curiously though, ordered Fe–Al alloys exhibit a temperature range where their yield stress increases with temperature. This gives rise to a peak in the yield stress occurring at about 800 K [6]. Quite a few mechanisms [7–9] responsible for YSA were suggested, however it is still not conclusively explained [10–12].

2 Experimental methods

Single crystals of iron with 20, 28 and 40 at. % aluminium were grown using Bridgman method [13], oriented using Laue X-ray diffraction and cut to the $2\times2\times5$ mm$^3$ samples. These were then deformed in compression at room temperature. Samples of Fe–Al$_{28}$ and Fe–Al$_{40}$ were deformed also at temperatures in the range of yield stress anomaly. Room temperature deformation was done in an apparatus that enabled in-situ observation of the evolution of surface by atomic force microscope [14]. Samples deformed at elevated temperatures were investigated by AFM after the deformation. Dislocation structures in deformed samples were then investigated in transmission electron microscope. Figure 2 shows the deformed sample and an area observed by AFM as well as slices extracted for observation by TEM.

2.1 Step fitting method

Numerical processing is required in order to take full advantage of quantitative height information present in the AFM images. Image needs to be leveled, the scatter of scan-lines has to be corrected, then the profile across the step is extracted. AFM images are usually processed interactively [15]. That negatively affects reproducibility of the analysis [16]. When the sequences of in situ images are analyzed it is essential to analyze the successive images of the same feature in exactly the same manner. Therefore, automated method of analysis of individual steps imaged by AFM is presented: In order to determine parameters describing the shape of the step, every scan-line segment is fitted by the step function
Figure 2: The location of in-situ AFM observation area (green) and five slices taken to prepare TEM specimens (red). The images of the edges of three slices taken after they were cut were matched to the image of whole sample. Two missing slices were already polished and prepared for TEM observation. Optical image from AFM microscope showing the cantilever while scanning was matched to the image as well.

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 \((h, p, w, a, b\) and \(c\)) 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 its one dimensional nature
is considered. Dislocations are modeled as straight lines between points recognizable on multiple projections such as junctions and intersections with foil surfaces. Equations for coordinates of these points projected from 3D space are assembled into a system of linear equations. This can be solved in the least squares sense.

In principle the rotation matrix for a given projection can be determined from the goniometer angles. However, in this work we determine the orientation from Kikuchi line patterns using the method akin to EBSD. Kikuchi lines are detected using Hough transform [19] and Kabsch algorithm [20] is used to fit the rotation matrix between set of vectors derived from Kikuchi pairs and set of assigned hkl directions.

### 3 Results

#### 3.1 Deformation

Recorded stress-strain curves are shown in figures 3a–c. In-situ deformation was periodically stopped in order to acquire AFM images. During the image acquisition (≈ 5–10 min) the stress in the system relaxed. This can be observed as disturbances on the stress-strain curve. Figure 3.1 shows temperature dependence of yield stress (0.2% strain offset). In both Fe–Al_{28} and Fe–Al_{40} the yield stress anomaly with maximum around 800 K was observed.
Figure 3: Deformation curves (a–c) and yield stresses (d) of Fe–Al$_{20}$, Fe–Al$_{28}$ and Fe–Al$_{40}$ deformed in compression. Yield stresses were determined using 0.2% strain offset method.
3.2 Fe–Al$_{20}$

Wavy steps in the direction that roughly follows the plane of maximum Schmidt factor were observed on the surface of Fe–Al$_{20}$ specimen deformed in-situ in AFM (fig. 4). TEM investigation of the same specimen showed slip bands of high dislocation density separated by dislocation free areas. On multiple occasions slip band bifurcations were observed. Dislocation free areas are sometimes crossed by the “bridges” – tiny slip bands composed of well resolved individual dislocations.

![AFM images of wavy slip bands in Fe–Al$_{20}$](image_url)

(a) (b)

Figure 4: Post-mortem AFM images of wavy slip bands in Fe–Al$_{20}$.

Another Fe–Al$_{20}$ specimen deformed in-situ showed peculiar dislocations traces with a narrow middle part that spreads towards the sides (fig. 5). One of the structures (fig. 5b) well separated from the others was analyzed using the step fitting method. Numerical analysis gave more detailed insight into the actual shape of the bow tie (fig. 6).
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).

Figure 6: Detailed analysis of a single bow tie.
3.3 Fe–Al$_{28}$

AFM observation of deformed Fe–Al$_{28}$ revealed massive slip bands on (101) plane as well as finer slip lines on (211) plane. In-situ observations (fig. 7) of a propagating tip of massive primary slip band showed fine secondary slip lines being emitted from the tip of the primary slip band.

![Slip Bands](image)

Figure 7: Excerpts from a sequence showing secondary slip lines emitted from the tip of a primary slip band.

Slip bands formed at temperatures below YSA peak show huge primary slip bands parallel to (101) plane (fig. 8a–c). At room temperature cross slip from these planes to (211) was observed. At 500 and 700 K the secondary plane was (110). These three samples also showed very weak slip lines on (101) plane that correspond to different Burgers vector since they appear dark on AFM error images. With increasing temperature deformation gets more dispersed. Initially massive and separated slip bands become finer and more homogeneously distributed. Slip character changes substantially at YSA peak temperature (fig. 8d): thin slip lines on (101)
and \((\overline{2}11)\) planes are observed at 800 K. At 900 K only very faint structure is visible (fig. 8e).

Figure 8: AFM images of Fe–Al\textsubscript{28} 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).

![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).](image)

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.
Figure 10: Symmetry reduced pole figure showing the directions of segments of a dislocation network extracted from a 3D model. Marker size is proportional to segment length. Color coding matches figure 9.
3.4 Fe–Al$_{40}$

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

![Image of dislocation loops](image.png)

Figure 11: Square dislocation loops around impurity particles. $g = (220)$, $d \approx [00\bar{1}]$.

Loops were determined to lie in the planes of $\{100\}$ type with edges along directions of $\langle 100 \rangle$ 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 $\langle 100 \rangle$ directions is observed.
Figure 12: 3D reconstruction of dislocations (red) and long axes of carbide particles (black). Planes of roughly planar dislocations are shown in red (001), green (010) and blue (100).

Figure 13: Directions of dislocation line segments (red) and directions of long axes of carbide particles (black) in stereographic projection. The later are also shown folded into standard orientation triangle in the figure 14.
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 moiré patterns of overlapping lattices of matrix and carbide can satisfactorily explain the distribution of orientations of particle axes.

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.
4 Conclusion

Single crystals of Fe–Al$_{20}$, Fe–Al$_{28}$ and Fe–Al$_{40}$ were deformed in-situ under the AFM. Evolution of slip line structures was captured on the sequences of AFM images: wavy slip bands in Fe–Al$_{20}$, cross slip at the tip of the slip band in Fe–Al$_{28}$ and homogeneous fine slip lines in Fe–Al$_{40}$. Another differently oriented sample of Fe–Al$_{20}$ showed peculiar slip lines spread at the ends in a bow tie like shape. This shape was analyzed in detail using the original method. Further applications of this method are not mentioned in this work, since the work focuses on Fe–Al system. However, elsewhere [21] it was used on several examples from microtwins in LaAlO$_3$, through slip traces in niobium to cross slip in Ni$_3$(Al, Ta).

Fe–Al$_{28}$ and Fe–Al$_{40}$ were also deformed at elevated temperatures and observed in AFM after the deformation. Surface of Fe–Al$_{40}$ was too oxidized, however Fe–Al$_{28}$ showed change of slip line structure related to yield stress anomaly.

Next the TEM was used to precisely identify the dislocations found in samples. Generally good agreement was found between AFM and TEM observations validating the AFM as relevant tool to study dislocations.

A technique of construction of 3D representations of dislocations without the prohibitive difficulties of tomography was developed. 3D models of dislocation structures found in Fe–Al$_{28}$ and Fe–Al$_{40}$ were created. The method is in fact applicable to any 1D structures and the directions of axes of carbide particles present in the alloys were determined as well. Distribution of orientations of these particles was then explained using the geometrical model based on minimisation of lattice mismatch.
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