AFM Observation of Slip Line Evolution in Nb Single Crystal

J. Veselý,1,2 D. Charrier,2 M. Cieslar,1 J. Bonneville,2 and C. Coupeau2
1Charles University, Faculty of Mathematics and Physics, Prague, Czech Republic.
2Université de Poitiers, Poitiers, France.

Abstract. Thanks to the AFM, the traces (steps) left on the surface by passage of only a few dislocations can be studied. The surface can be observed during in-situ deformation at room temperature or post-mortem analysis of samples deformed at elevated temperatures can be performed. In this work, preliminary experimental results on Nb single crystal are presented.

Introduction

Utilizing the AFM to study steps created by emerging dislocations their behaviour can be observed at nanometer scale. After the successful application of this method to Ni3Al [Coupeau and Bonneville, 2007] we turn to the relatively well known bcc niobium [Votava, 1968; Evans and Schwenk, 1970; Louchet and Kubin, 1975; Reed and Arsenault, 1976; Chang et al., 1983; Wasserbich and Novák, 1985] in this study.

We would like to employ the resolution offered by the AFM to see in the bigger detail the cross slips of the screw dislocations that lead to the wavy slip bands characteristic of bcc metals [Votava, 1968].

Experimental procedures

Oriented single crystalline rod of Nb was supplied by the Goodfellow company. The sample with dimensions 4.98 × 2.43 × 1.83 mm3 was cut by diamond wire saw. Deformation axis was oriented along ⟨001⟩ with observed face normal to the ⟨110⟩ direction. Proper orientation (within 4°) was verified by Laue back reflected X-ray pattern. Observed surface was polished to the 1.4 nm RMS roughness (evaluated from 50 × 50 µm2 area). Final polishing was carried out using alkaline colloidal silica with 50 nm grain size (Logitech SF1 Polishing Fluid) and lasted several hours.

In-situ deformation experiments were conducted in contact mode of Veeco Dimension 3100 scanning probe microscope. Custom built compression device as described by Coupeau et al. [1998] was used. This apparatus allows to follow in-situ by AFM the evolution of surfaces under increasing strain (or stress).

The piezo ceramic actuators have limited extensions, therefore sample was firstly brought to the yield point by hand screw. From this point deformation continued by ramping up the voltage on piezo. After constant voltage increase the deformation was stopped and the AFM images were recorded. These interruptions lasted several tens of minutes, since multiple images of different magnifications were taken. During this time considerable relaxation on sample as well as on piezo elements occurred.

The purpose of the apparatus is to create the slip lines to be observed. It is by no means a perfect compression device. Therefore only limited information about the strain is available: it increases monotonously with each measurement.

Total plastic strain of about 3% was evaluated from sample dimensions after testing.
Experimental results

Few AFM images taken from the sequence of about 20 images recorded during deformation are shown in figure 1. These are depicted in error signal mode that provides better insight on the surface features. Two different kinds of slip lines were observed: straight lines, appearing white in error signal image and curved lines appearing black. Straight slip lines make an angle of 54° with the deformation axis and correspond to the \langle 111 \rangle \{110\} slip systems with a Schmid factor of 0.41 (see diagram in figure 2). Slip of \langle 111 \rangle \{112\} and \langle 111 \rangle \{123\} slip systems (with maximal Schmid factor) would manifest by lines at 90° and 76° respectively. However such lines were not observed, although Schmid factors of their slip systems are higher (0.47 and 0.46 respectively).

From the AFM height data we could plot profiles across selected slip lines to see evolution of the step with increasing deformation (figure 3). Several tens of steps were analyzed. Typical step is 3–5 nm high after 3% of plastic deformation and grows continuously during the course of deformation (although occasional saturation of growth as well as stopped and restarted growth was observed). No significant difference was found between the evolutions of two kinds of slip lines. Step resulting from the emergence of one dislocation at free surface, can be calculated from the projection of Burgers vector $b$ on the surface normal. For $b = \frac{\sqrt{3}}{2}[111]$ and Nb lattice constant $a = 0.330 \text{ nm}$ we get elementary step height of 0.233 nm. This corresponds to about 15 dislocations forming a typical step at the end of deformation.

Discussion

Due to the symmetrical orientation of deformation axis, slip should occur for all four \langle 111 \rangle Burgers vectors. However, \langle 111 \rangle and \langle 11\overline{1} \rangle lie in the observed plane and thus slip would not create the step on the surface. The lateral distortion of pre-existing steps from other slip systems would occur, but the typical amount of dislocations moving per slip line is not enough for this effect to be noticeable at the scale of our observations.

In the AFM error mode images black and white lines correspond to the steps of different

![Figure 1. Evolution of slip line structure during the compression test error signal AFM mode. Images (from left to right) correspond to the increase of global plastic strain from yield point to about 3%. Compression axis is horizontal. All images show approximately the same location at the same magnification.](image)

![Figure 2. Geometry of crystal orientation showing the slip systems \langle 111 \rangle(\overline{0}10), \langle 111 \rangle(\overline{0}11), \langle 1\overline{1}1 \rangle(101) and \langle 11\overline{1} \rangle(011).](image)
Figure 3. Evolution of the step profile with progressing deformation. Original raw data were filtered with $3 \times 3$ median filter to remove noise. Measurement number 0–23 correspond to increasing global plastic strain from 0 to about 3%. Baseline of individual profiles is arbitrarily shifted.

directions (up/down). From this point of view it is surprising that straight lines represent step in only one direction and therefore were created by dislocations with one Burgers vector, although slip on both symmetric Burgers vectors ([111] and [11\overline{1}]) should result in the both up-steps and down-steps (black and white lines).

Perhaps small misalignment from exactly symmetric orientation of the crystal could cause differences in behaviour of dislocations with different Burgers vectors.

This method provides wealth of quantitative information since measured height field is available from each AFM image. However, sophisticated data analysis methods have to be devised in order to extract physically relevant information. The preparation of the extremely smooth surface is important in order to reliably discern elementary steps. Finally, the solid link between the surface phenomena (observed by in-situ by AFM) and processes in the bulk (responsible for plastic behaviour of material) needs to be established. To address this issue surface observations will be supplemented with post-mortem TEM analysis.

Summary

Slip lines corresponding to the $\langle 111 \rangle \{110 \}$ slip systems were observed on the surface of deformed Nb. Steps created after 3% of plastic strain, are related to the emergence of about 15 dislocations. Conventional AFM offers enough out-of-plane resolution to study steps created by single dislocation. However, quality of the initial sample surface (roughness) seems to be a crucial factor. More detailed study of Nb as well as more complicated bcc based intermetallic alloys (FeAl and Fe$_3$Al) is under way.

Acknowledgments. This work is supported by the Charles University Grant Agency under Contract 23510. J. Veselý gratefully acknowledges the scholarship of French government.

References


