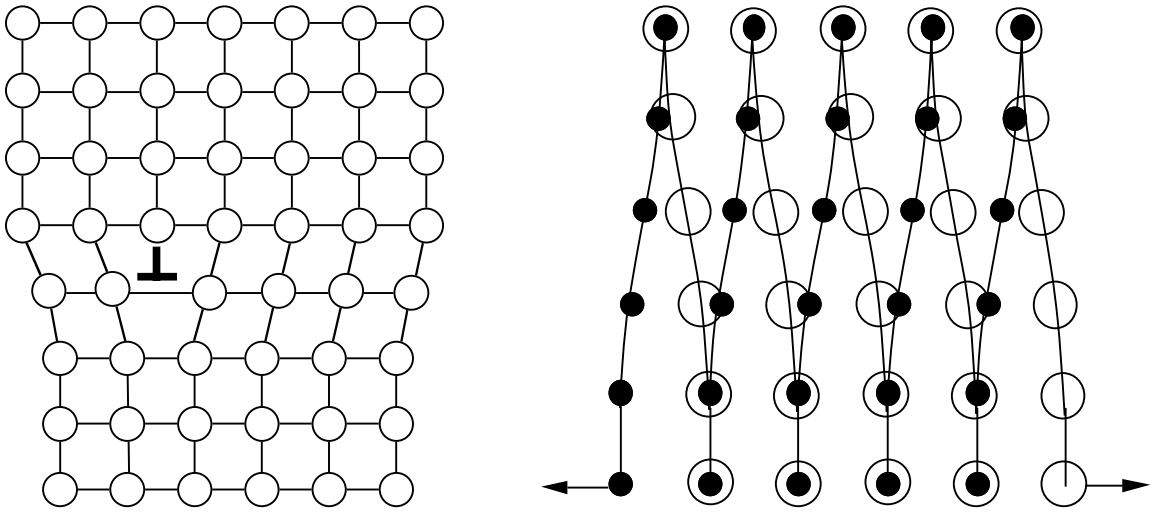
Lab 2
Polygonization in Rock Salt
27-202
Fall 1999

Objective

The objectives of this lab are to experimentally observe dislocations in rock salt, to increase their density by plastic deformation, to observe polygonization, to determine the slip system, and to determine the size of dislocation free domains.

Introduction

Dislocations are important defects because they influence rate at which crystals grow, as well as the mechanical, electrical, and optical properties of crystals. The two limiting cases for dislocations (edge and screw) are shown in Fig. 4. While a small population of dislocations normally occurs in all crystals, the population can be greatly enhanced by plastic deformation.



(a) (b)

Figure 4. The atomic structure of the (a) edge dislocation and the (b) screw dislocation. In (b) the smaller filled circles are in the same plane (above the paper) and the larger open circles are in a different plane (below the paper).

Because these defects have dimensions on the atomic scale, direct observation is difficult. However, it is possible to locate dislocations by forming visible etch pits at the

point where dislocations intersect the crystal surface. Using this method, we will examine the arrangement of dislocations in as received crystals, in crystals after plastic deformation, and in the deformed crystals after annealing. This lab is based on the experiment originally described by C.L. Bauer in: C.L. Bauer, "Polygonization of Rock Salt", Trans. Metall. Soc. of AIME 223 [4] (1965) 846-847.

Background

The atomic displacements near a dislocation are a source of internal stress. In the diagram of the positive edge dislocation shown in Fig. 4 (a), it is clear that the upper half of the crystal, where the extra half plane has been inserted, is under compression and the bottom half of the crystal is under tension. The stress fields that result from the compressive and tensile forces around different dislocations interact with one another. If the dislocations are free to move, we expect them to take up positions that minimize their elastic energy. There are two types of dislocation motion, glide and climb. When a dislocation moves by glide, as illustrated in Fig. 5, it remains in the same slip plane. This process, called conservative motion, can occur at relatively low temperatures. Climb, on the other hand, is a nonconservative motion that involves the creation and migration of point defects. Thus, it is temperature activated and occurs only at higher temperatures. This process is illustrated in Fig. 6.

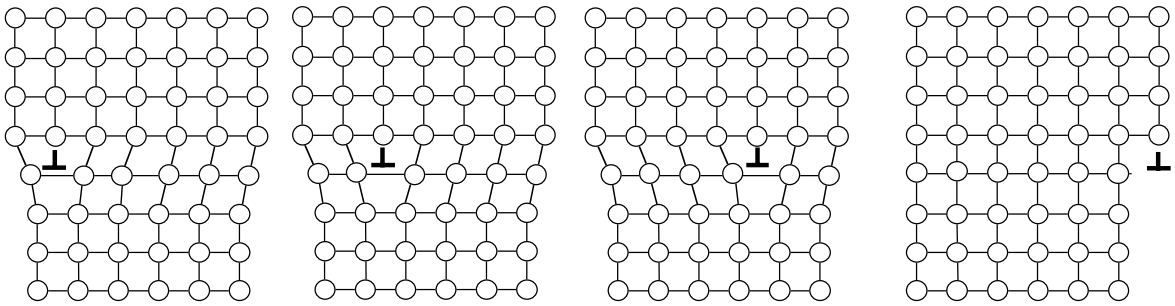


Figure 5. Dislocation glide. An edge dislocation moves to the right and eventually exits the crystal.

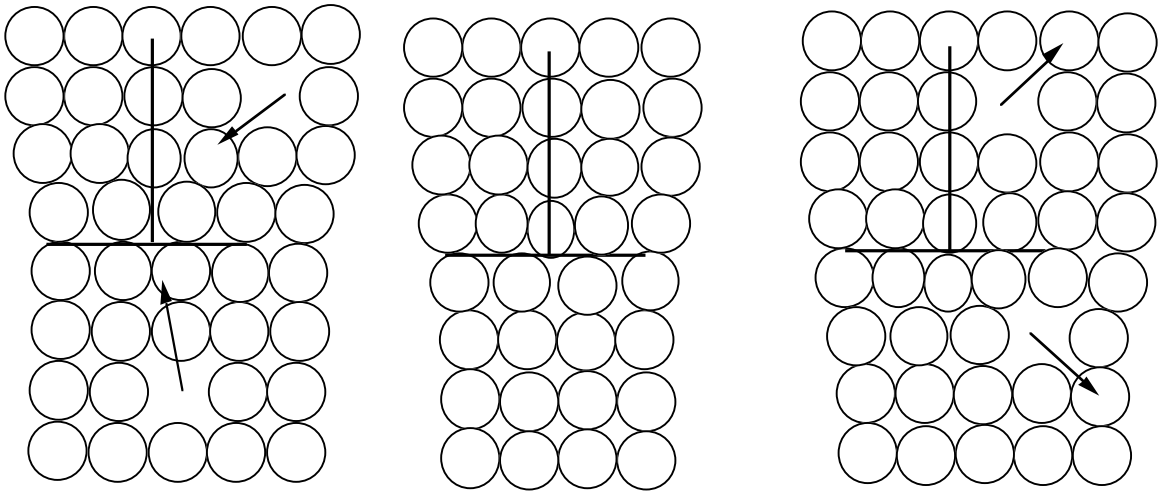


Figure 6. Dislocation climb. In (b) the edge dislocation is centered. By adsorbing vacancies it can climb in the positive direction, as shown in (a). By emitting vacancies it can climb in the negative direction, as shown in (c).

Equilibrium configurations of dislocations of the same and opposite signs have been determined theoretically and the results are summarized in Fig. 7. In arriving at these configurations, it was assumed that only glide was possible. A somewhat different result occurs if climb is active. For example, consider the attractive interaction of two edge dislocations of opposite sign (if the edge dislocation shown in Fig. 9.1 is a positive dislocation, then a negative edge dislocation is one in which the extra half plane is inserted from below). If these two dislocations are permitted to climb towards each other, they annihilate.

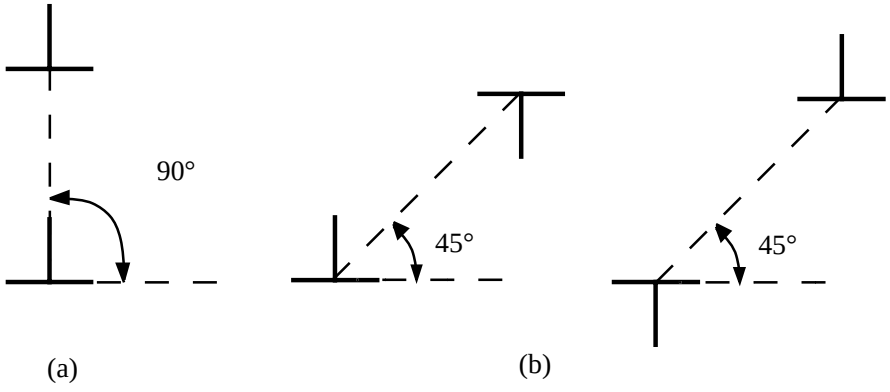


Figure 7. The equilibrium configuration of edge dislocations free to glide (a) of the same sign, (b) of opposite sign.

We will determine the positions at which dislocations intersect the cleaved surface of a crystal by examining etch pits. An example of such pits is shown in Fig. 8. During dissolution, atoms from near the dislocation are removed at different rate from those far from the dislocation. This process, which results in the formation of an etch pit, occurs because of the bond strain near the dislocation (atoms in the strained positions are more easily removed) and the impurities that concentrate near the dislocation. In practice, producing visible etch pits can be difficult since it is important that the removal rates from the ideal and defective areas occur in the proper ratio.

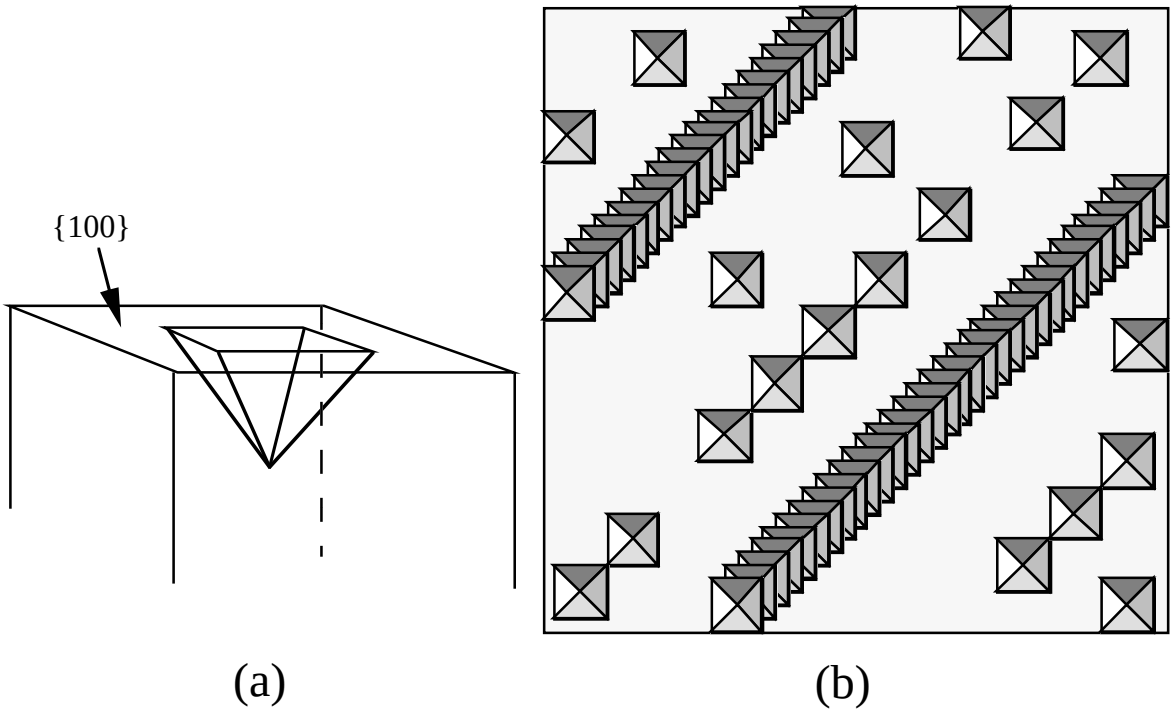


Figure 8. Dislocation etch pits. (a) a three dimensional view of a pit with square pyramidal facets. (b) Projection showing schematic distribution on the surface.

In our experiment we will use single crystals of rock salt (NaCl). Not only is the observation of etch pits simplified in this material, but it is also easily cleaved and deformed.

Procedure

1) The first thing to do is to cleave a piece of your crystal that will serve as your "as received" specimen. To cleave a {100} face, a razor blade should be aligned along the

long axis of the crystal and given an impact. It is probably best if you see this demonstrated once by a lab assistant before trying it for yourself.

2) After putting your cleaved piece aside, you must deform the remaining crystal. Although rock salt is usually considered brittle, it is a rather simple matter to bend it under water. So that it is bent to a known curvature, deform it around the radius of a submerged watch glass (or other suitable object). This should be done slowly and gently.

3) After deforming the crystal, cleave off another piece and set it aside. Divide the remaining crystal and anneal one piece 450 °C and another at 680 °C. These should be annealed for at least two hours. While waiting, you should take the opportunity to examine the as received and deformed specimens.

4) Etch the as received sample in the Ferric Chloride etching solution (four grams of ferric chloride per liter of glacial acetic acid). Immerse the crystal in the solution for about 2 to 7 minutes, remove and air dry. Examine the cleaved, etched surface under the optical microscope and look for etch pits. Recorded at least one photograph of the specimen. Repeat this process for the deformed crystal and characterize, as quantitatively as possible, the change in the etch pit density.

5) After the other two samples have been annealed for two hours, remove them, etch and record your microscopic observations. How did the population and arrangement of the dislocations change after each treatment? Can you account for these changes?

6) Determine the directions of the boundaries and the density of dislocations along three boundaries.

7) Make sure that you measure the field of view in the microscope, so that you can put scale bars on your images.

Report

We will use the same format that we used for the first lab; please refer to the notes on pp. 8-10 regarding the content of each section. The following questions are intended to guide the specific content of your report.

Abstract

In less than 100 words, describe what you have done as well as the most important data and/or results and/or conclusions. In this lab, you should include observed dislocation densities, boundary directions, the slip system, the domain size, and the misorientation.

Introduction

What methods can be used to observe dislocations?
How is the dislocation density increased in a crystal?
What do you expect to happen during annealing?
Why do we use single crystals?

Procedure

How did you cleave the samples?
How do you bend the samples?
How were they etched?
How were they annealed?
What type of microscopy did you use?

Results

Your results section should include images of the cleaved surface before etching, after etching, and the annealed surface after etching.
What are the most apparent features?
Along which crystallographic directions were the features observed?
[Remember that all images must have scale bars]

Discussion

How does each process affect the dislocation density?
What is the dominant slip system in NaCl?
What is dislocation density along the boundaries?
What is the misorientation across the boundaries?