Science

3. Transmission Electron Microscopes

Microscopists Do It in the Dark

I spend a lot of time with transmission electron microscopes when I was doing my Ph.D work. I fed them liquid nitrogen and sometimes even liquid helium, cleaned their insides (including the high-voltage generators, parts that not everybody was allowed to touch), tuned and repaired their electronics, fiddled and caressed their many buttons, suffered with them through vacuum leaks, and stared down ~~their cleavage~~ a thick glass window into their insides for many hours without a break. Occasionally, when everything happened to be just right, an exciting picture was obtained that induced ecstasy.
This kind of aberrant behavior typically results from lack of girl friends. I'm thus happy to report that I had no more need to embrace electron microscopes for the last 35 years.

- It's hard to describe the frustration one experiences when after 10 hours of hard work one finally develops the photographic film that recorded the images seen on the screen (no electronic gadgetry then), just to find out that during the (10 - 20) seconds exposure time all the fine details were wiped out because the microscope wasn't quite stable.
  It is equally hard to describe the exhilaration experienced when a picture was obtained that showed something new and exciting for the very first time, something that no other human had seen before.
- As far as microscopes go, transmission electron microscopes are still my personal favorites.
  Here is a Siemens Elmiskop from around 1975, the first TEM that could resolve atomic structures on a good day. It is the type that I used for much of my post doc work.

. **Siemens Elmiscope from around 1975 (≈ DM 250.000 (€125 000))**
I found it 2011 in a museum in Dresden, Germany

The Basics of Electron Microscopes

An electron beam cannot only be described as a wave, it is also an electrical current - in contrast to X-rays or neutron beams. That means it can be bent and mutilated by magnetic and electrical fields.

Cleverly designed magnetic fields act on an electron beam almost exactly the same way a convex glass lens acts on a light beam: both lenses focus the beam onto a tiny spot. Take a lot of magnetic lenses, arrange them the same way you do it for a light microscope - bingo! You have a transmission electron microscope (TEM).
Well, not quite. There is some small print in the instructions of how to build an electron microscope. For example, everything has to be kept in a good vacuum. The room for a TEM should be free of vibrations and stray magnetic fields (a tough requirement at the levels demanded!). The high-voltage source (typically around (200 - 300) kV, but some microscopes go beyond 1 MV) and all the other power supplies need to be ultra-stable. It also pays to read the bit about "*...one should not forget that an electron microscope is essentially a huge X-ray tube. Some precautionary measures might be indicated if the life-span and healthy offspring of operators are of some concern...*".
Just kidding, There is a huge amount of lead shielding in electron microscopes and all kinds of other protective stuff.
In the end, a good TEM will set you back at least (2 - 5) Mio $. Just for comparison: for 2 Mio $ you get easily 20 - 50 very good optical microscopes.

Modern run-of-the mill TEM. Around 1.5 Mio €

Forgetting about those annoying details, the basic way to run a TEM is more or less like running an optical microscope in the transmission mode, where you look *through* your specimens:

- Illuminate the area of interest on the front-side of the specimen with the electron beam; make sure that the intensity is the same everywhere. The beam then will move through the specimen, interact somehow with whatever is in its way, and exit on the backside.
- The intensity on the backside of the specimen is no longer the same everywhere since in some parts of the specimen the electrons might have experienced different interactions with whatever is inside the specimen than in other parts.
- Magnify the intensity distribution at the backside with a system of magnetic lenses.
- Finally, let the electron beam hit a screen, where the intensity variations get turned into a picture that the eye can see.

So far it is easy. The long and short is: How do things inside the specimen interact with the electron beams? There are essentially two ways:

1. Dense parts inside the specimen, e.g. a lead containing precipitate, *absorb* or *deflect* more electrons than the matrix. The intensity on the backside of the specimen then is smaller below those regions than in other parts. The picture is essentially a kind of "shadow" projection of dense or thicker parts. It is exactly like the X-ray images your doctor takes, just on a much smaller scale. It's called absorption contrast and that's what you do

with biological samples. It is utterly boring.

2. Some parts of *crystalline* specimen *diffract* the beam differently from others, leading to changed intensities. This is highly interesting and the only way to deal with crystals.

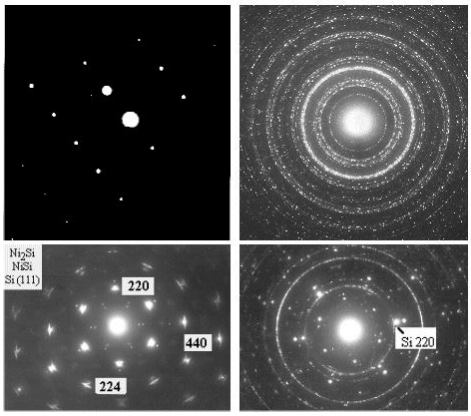
Back-bone

Dislocations

This would be a good time to look up the modules about diffraction and dislocations.
In fact, one of the modes of a TEM is doing exactly the same thing that the Laue technique does with X-rays. It produces a *direct* diffraction pattern and not a picture.
Diffraction patterns look, for example, like this:

Science Link

X-Ray diffraction



Diffraction patterns with an electron beam

Aluminum single crystal (Al)	Polycrystalline platinum silicide (PtSi)
Silicon with epitaxial nickel silicides (Si - NiSi - $NiSi_2$)	Polycrystalline nickel mono silicide (NiSi) on top of single crystalline silicon (Si).

Source: From my work

I'm not going to make you an electron microscopy expert in just 5 minutes a day, so we only note

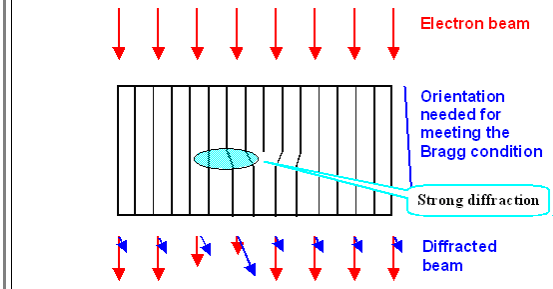
- there is a hell of a lot of information contained in those diffraction patterns, and
- they are not what we are looking for. We want "real" pictures.

We do use the information contained in those diffraction patterns, of course, but we use those diffraction pictures primarily to orient the sample in such a way that the diffraction effects are exactly what we want them to be. For this we move and tilt the specimen with a nanometer precision.

We are ready now to produce a picture of some crystal defect, some irregularity in a crystal lattice. There is not much else to see inside a crystal. We might want to image dislocations, for example.

What the microscopist typically does is:

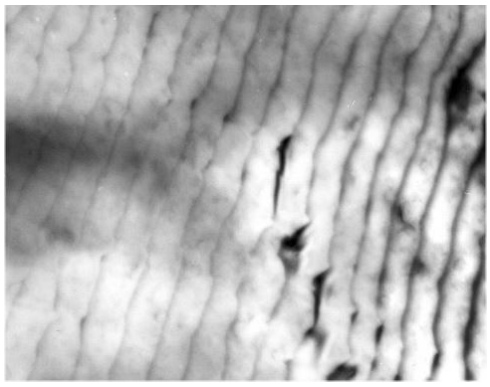
- Tilt and move the specimen until only *one* set of lattice planes diffracts the incoming beam. Parts of the incoming beam *and* one diffracted beam then will leave the sample. Owing to the wavelength of the electrons and the geometry of the whole set-up, this lattice plane will be almost parallel to the electron beam. In other words: the Bragg angle is rather small.
- Now tilt the specimen a tiny little bit *off* the Bragg angle; just enough to reduce the intensity of the diffracted beam.
- Put an aperture into the path of the electron beams leaving the sample in such a way that all diffracted beams are absorbed and can't make it down to the screen.
- Crank up the magnification to what you want. You are now going to see the dislocation as a fuzzy dark line. Why? Look at the figure below.



.Diffraction contrast from a dislocation

The actual lattice planes (black lines) are a little bit off the proper Bragg orientation (shown exaggeratedly by the blue line).

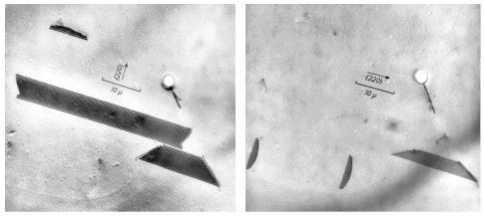
- The primary electron beams just runs through the sample, not doing much. By the way, did I mention that the samples better be very thin, just fractions of micrometers? If not, take it for granted from now on.
  Anyway, wherever we have a perfect crystal, the electron beam goes right through the sample and the screen all the way down will be bright. Around a dislocation, however (or most lattice defects for that matter), the crystal lattice is locally disturbed and deformed. As shown schematically above, in parts of the environment of the dislocation, it is bend into the proper Bragg orientation. That means that we have strong diffraction in those parts, locally generating a strong diffracted beam. This means that the primary beam now is weakened since many of its electrons now go the other way.
  With proper apertures, only the primary electrons make it to the screen, and the dislocation produces a dark line to the left of its core. This is what it looks like:



.Dislocations imaged with "fading" Bragg condition

Source: From my work

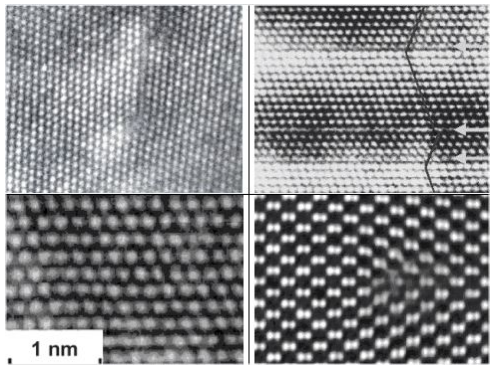
- We have a whole set of dislocations running in parallel. The specimen in this case is slightly bend and on the left-hand side only the most severely distorted regions around a dislocation is still diffracting the beam just a little bit. On the right hand side the conditions are just right for strong contrast.
  What that tells us is that dislocations (and other defects) are only visible under special circumstances, and that they look different if conditions change This is good, because it allows us to work out the nature of the defects in great detail by taking whole series of pictures under different imaging conditions.
  Look at the two pictures below. They show exactly the same region of a specimen (a tiny piece of an early integrated silicon circuit) imaged under two different diffraction conditions. What you see are stacking faults (and some other stuff) in this case. The pictures are about as different as they could be.



Stacking faults in the same region of the specimen imaged with different diffraction conditions

Source: From my work

- Doing TEM work and utilizing diffraction contrast is not for the faint of heart. Specimen preparation is rather tricky and tedious, and operating the microscope is a lengthy affair in the dark. There are also far more ways to produce an image than the simple one discussed here. Not to mention that acquiring the necessary knowledge is lengthy and tedious, especially if done without benefit of girl friend.
  - Not using diffraction contrast but utilizing atomic resolution or high-resolution TEM (***HRTEM)*** doesn't make it much better. It is fun for a while, but after you met the first few thousand atoms individually, you can't remember their names anymore and they all start to look the same



Silicon with two (edge) dislocations.	Silicon with two "twin" grain boundaries.
Perfect iron crystal.	Gallium arsenide (GaAs) with a dislocation.

Examples of high-resolution TEM pictures

Source: From my work and friends from Jülich

  - The distance between the "dots" = rows of atoms is around 0,3 nm. Note that I just as easily could have claimed that the two top pictures show iron, or that the iron picture shows silicon. Or just about anything else with a cubic lattice viewed "edge-on". Just looking at the pictures won't tell. Fortunately Materials Scientists are honest people, at least most of them most of the time.
    You need a pretty good TEM for seeing crystal lattices on the 0,1 nm scale, and you have to know how to do it. The trick in this case is not to use an aperture and to pass as many diffracted beams as possible.
- As in the case of the scanning electron microscope, the specimen emits X-rays and secondary electrons, and does other things with the electron beam that is specific to the atoms in the specimen. Recording these signals allows **analytical TEM**. You see something very small *and* you know what it is.
  - We are not yet able to identify single atoms but we are getting close. In a few more years (and with a few more millions in cost), transmission electron microscopes will be able to do whatever you fancy.
- This is particularly true if you turn your TEM into a STEM, a scanning transmission electron microscope.
  - In this case you focus the electron beam into a spot less than 1 nm in size on your specimen and scan that spot across the sample, just as in a scanning electron microscope (SEM). However. in contrast to a simple SEM, you do not (primarily) record the intensity of banged-off secondary electrons and the like, but the intensity of the beam that emerges on the *downside* of the specimen. Moreover, you may also record the energy losses of that transmitted beam, and everything else that transpires. Taking everything together you get a lot of information about the tiny (atomic-size) illuminated pixel of your sample

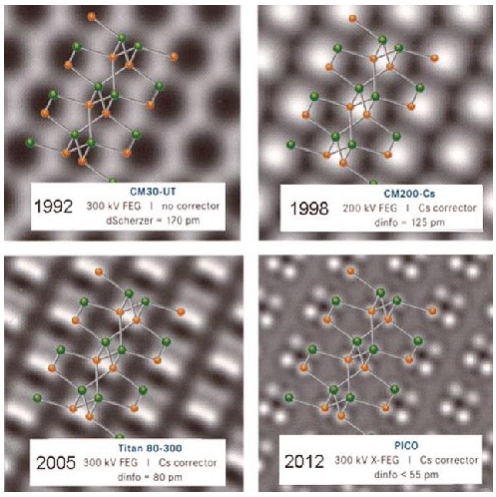
There is a *major* problem however, in looking at things with very high magnification or even atomic resolution. You only see a tiny part of your sample!

- The total volume of all specimens investigated so far is smaller than 1 mm^3 (about the size of the head of a pin!)

You will never know everything about the "inside" of your sword on an atomic level. So let's hope that the tiny little parts of whatever we do see are representative for the whole.

- Another *minor* problem is that it takes typically two advanced lecture courses just to learn the theory of electron microscopy, and at least a year or two of hands-on experience before you can call yourself an electron microscopist, plus a few millions of hard cash to get one started. So, please, pay your taxes (or send me money directly).

Finally, let me show you why electron microscopists live in exciting times. Here is a direct comparison of what happened in the last 25 years or so:



Development of Resolution for TEM

Source: "Forschung in Jülich", **1** (2012)

- The pictures show what an aluminum oxide (Al_2O_3) crystal looks like in a succession of TEM's in comparison to its schematic structure.
  The 1992 TEM had "normal" electromagnetic lenses. The 1998 and 2005 type had lenses corrected for what is called "spherical aberration" or Cs), the 2012 TEM has lenses corrected for spherical *and* chromatic aberration. Right now the research center in Jülich, Germany, is the only place that owns the newest super machine - mostly because they had a hand in its development.

Unfortunately, there is no such thing as a free lunch. While you could acquire and run a 1992 machine for around a million or less, you should be prepared to go beyond 10 million for the newest machine.

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