Practical 2P4
Introduction to AFM Analysis

What you should learn from the practical:

Science:
This practical has three parts. In the first you will learn about the basic operation of an atomic force microscope using a standard sample.

In the second you will study with the AFM the RMS values of roughness on the best polish you can achieve on a metallographic specimen.

In the third you will learn how to analyse thermal grooving, which is covered in the Surfaces and Interfaces lecture course.

Practical Skills:
You will learn how to operate an atomic force microscope, and how to analyse data from AFM images.

Safety Considerations:
The AFM has a laser which could potentially affect your vision if looked at directly. To avoid this, never rotate the scanner head.
Overview:

Day 1:
- Learning the basic operation of the AFM and observing.
- Polishing silver sample, and analysing rms surface roughness values on polished surface.

Overnight:
- Annealing silver sample.

Day 2:
- Observation of thermal grooving in silver sample.

Day 3:
- Further microscopy if required.

The AFM:
The AFM is a form of scanning probe microscope developed in the mid 1980s. It works by scanning an extremely fine probe on the end of a cantilever across the surface of a material, profiling the surface by measuring the deflection of the cantilever. This allows a 3D profile of the surface to be produced at magnifications over one million times, giving much more topographical information than optical or scanning electron microscopes. Its limitation is that the surface to be observed needs to be very flat or the tip will crash into the ‘hills’ as it is scanned.
The microscope can run in two modes, contact and close contact. Contact mode scans the probe across the surface, keeping a constant force between tip and sample, maintained by a feedback control. The amount of movement required to keep the constant force is then used to create the image. Close contact mode, often called tapping mode, uses a vibrating cantilever. Simple height data can be obtained from the changes in Z-axis displacement, but phase data can also be obtained. There is a phase difference between the measured signal and the drive signal, caused by interactions between probe and material. A 'phase image' can be formed using this data, which will indicate regions of different composition and/or phase in the material.

**NB. You will be using contact mode only. The use of tapping mode is much harder to learn.**

**Useful Reference:**
http://www.lot-oriel.com/pdf_uk/all/pni_tutorial_uk.pdf

**Experimental Procedure**

**Preparation:**
You are given a silver sample to grind and polish. It is important to polish it very well as small scratches will appear very large in the AFM, and the sample should be ultrasonically cleaned between different grades of polish. Finally use colloidal silica solution to produce an even smoother surface finish. Check this finish in the optical microscope, before using the AFM to look at what a ‘good’ optical polish really means in terms of surface topography.
Once the sample is polished it needs to be annealed overnight at $850^\circ C$. You will need a demonstrator to help with using the furnace.

**Microscopy:**

[1] Operation of the AFM

The Junior Demonstrator will show you how to use the AFM, but you will as a group need to spend at least an hour getting familiar with how to align it. To help with this, you should use the test sample which is an etched silicon surface with very clear features.

- The first result that you need to include in your write-up is a 2D image of the sample surface in an imaging mode that you think reveals the surface structure most clearly, and a measurement of the height of the etched features.

[2] Observation of surface structure of polished silver

Take at least 5 images of different areas of the polished Ag surface at magnifications suitable for showing the surface features.

- Select one of these for your write-up, and explain what the image shows.
- Use the AFM software to calculate the root mean square roughness of at least 5 areas of the sample, and comment on the average value you obtain.
Observation of Thermal Grooving

Aim:

To investigate the phenomenon of thermal grooving in silver and to calculate the relative grain boundary and surface energies.

Thermal grooves are found where grain boundaries intersect the surface of a material due to the excess interfacial energies associated with the surfaces and grain boundary. As with any triple junction it is at equilibrium when the forces associated with the interfaces and their surface tensions must balance. In the case of a grain boundary intersecting a perfectly flat surface this is clearly not the case. Therefore to make the forces balance thermal grooves are formed. This process is described in the Surface and Interfaces course, and the key equation that describes the equilibrium situation is

\[ \sigma_{gb} = 2\sigma_s \cos(\theta/2) \]

Method:

First check the annealed sample under an optical microscope. You should be able to see thermal grooves under low magnification. Now place the sample in the AFM and follow the instructions in the appendix. It is often hard to accurately position the tip over the exact place you want to scan, so there is a bit of trial and error involved. If your surface is very flat you can do a wider scan (50 \( \mu \)m). On the wide scan image, you can select the region you want to look at more closely by clicking and dragging with the mouse. Then right click this area and click on the scan button to scan the selected region. The 3D profile should confirm whether you have actually scanned a
thermal groove. You will be able to tell if it is a thermal groove because of its characteristic shape of a groove with raised material on either side. A groove will also appear on the Z(HGT) image as a darker line with lighter regions on either side.

You can now use the line scan over the boundary to measure the value of $\theta$ in the equation above and then work out the ratio of surface energy and grain boundary energy. Do this on at least 3 grain boundaries – more if possible.

**Write Up:**
Include:
- Brief description of what you did.
- Pictures of the surfaces you scanned, with any features labelled.
- Outline the mechanisms involved in thermal grooving.
- Give the result of the thermal groove calculations.
- Comment on the benefits/drawbacks of the AFM compared with an optical microscope in the assessment of surface roughness.

**Questions you should answer:**
- Why does the sample need to be annealed before significant thermal grooving is observed?
- How would you get absolute values of grain boundary and surface energies?
- What differences can you see between optical and AFM micrographs?
Appendix A

Using the AFM:

THE PROBE IS EXTREMELY DELICATE AND NEW TIPS COST 25 POUNDS TO REPLACE! TO AVOID DAMAGING TIPS NEVER MOVE THE PROBE USING THE STAGE CONTROLLERS OR REMOVE THE COVER PROTECTING THE AFM STAGE WHEN THE TIP IS IN CONTACT! ALWAYS CLICK RETRACT TIP WHEN SCANNING IS COMPLETE.

– A demonstrator will turn on the PC and AFM for you.
– Click mode and select contact mode.
– Click align laser and use the knobs on the far left of the AFM stage to position the red dot at the top of the green area and centred on the vertical line. If at this stage the scale to the right has not reached the minimum level, then either the laser isn't aligned, or the tip needs replacing. If this is the case ask a demonstrator.
– Ensuring the tip is high enough place the sample in the microscope. Make sure you don't touch the probe with the sample, because even a very slight knock will break it.
– Click stage and move the probe down until the tip is about 1mm above the sample. Do this by eye. Now focus on the sample and choose the area you with to scan. Choose an area free of dirt and scratches, as a speck of dust or deep scratch could be enough to break the tip.
– Once you have chosen the area to scan focus back on the cantilever, place the protective cover over the stage and click tip approach.
– Now select scan sample. For the feedback controls, set the gain to 6, the proportional to 10, the integral to 10 and the differential to zero. Now select the desired scan size and click scan.

– The probe will now scan over the sample and an image will form on screen. You can view two different images, so select the Z(ERR) image on the right. If you want you can try different values of proportional and integral to try and obtain a sharper image.

– To zoom in on a specific region of the sample, click and drag a square on the image then right click the square. Select OK on the message that appears and click scan sample.

– Now select image processing. There are a variety of options available. Z(ERR) will give you the best picture, but no 3D view, whereas Z(HGT) and Z(SEN) will provide good 3D views.

– When you have finished with the image processing, select retract tip, raise the stage to the top and remove the sample.
Appendix B

Image Processing:

- Click image processing on the tool bar.
- Select the acquisition channel you want. Choose Z(HGT) for a 3D view, or to obtain a cross section, and select Z(ERR) if you want to look at a sharp surface image.
- Firstly click the plane correction tab to open the plane correction screen. Select polynomial plane correction and click apply.
- To see a 3D profile click the 3D tab. You can rotate the image by holding down the right mouse button and moving the mouse.
- To acquire a cross sectional profile click the line profile tab. Select the oblique option, and drag the mouse over the area you want a cross section of, in your case a thermally grooved grain boundary.
- Use “print screen” button to save images as picture files for reports.
Instructions for the Demonstrator Regarding Operation of the AFM

The aim of this sheet is to instruct you in the general use of the atomic force microscope and instruct you how to perform procedures that the student won't be expected to do.

General operation:
Switch on the PC (AFM login – password nanor), open the SPM cockpit then switch on the AFM. Wait for the green light to appear at the bottom of the screen.

Click the mode tab and select EZ mode then click start on the EZ mode toolbar. Now select linearise and follow the on-screen instructions. Check that the graph shows a linear region over an x distance of 150-200um.

Click select mode and select contact mode.

Now you can load a sample. Click the up tab to raise the Z motor until you have enough clearance and then insert the sample on a puck.

The next step is to align the detector. Click on align laser and make sure the laser is on. Using the knobs on the AFM to the left of the stage try to position the red dot so it is at the top of the green shaded region, centred on the Y-axis. In this position the scale to the right should be well above the minimum. If this is not the case you will either have to manually align the laser or change the tip.
The controls to manually align the laser are situated to the right of the stage on the AFM. Firstly you will need to focus on the cantilever and then, using the knobs on the AFM and looking at the video monitor, align the laser on the tip of the cantilever. Now go back to the align laser section and repeat the above steps. If you now have a reading above minimum go to the next step. If you don't then you will need to replace the tip, which is covered later.

Now that the laser is aligned go to the stage controls and, monitoring it by eye, move the tip down until it is about 1mm from the surface. Now focus on the sample. You can now choose the area you want to scan, preferably away from dirt and scratches. Once you have chosen an area, focus on the cantilever and click tip approach. After a short time a message saying complete should appear. If an error message appears, you should click retract tip and repeat the laser alignment procedure.

Once the tip is in contact you can scan the sample. The scanner controls should be set as follows:

- Scan size: experiment dependent
- Scan range: 2Hz
- Resolution: 256
- Scan angle: 0
- Acq. Channels: 4
- Topography gain: 1x
Set the feedback controls to
Setpoint: 0
Gain: 6
Proportional: 10
Integral: 10
Derivative: 0

It is important for the derivative to be set to zero in contact mode as non-zero values will create a poor image.

Select Z(SEN) and Z(ERR) channels from the drop down menus beneath the two displays then click start scan.

Once the scan has completed you can then select image processing.

- Select the acquisition channel you want. Choose Z(HGT) for a 3D view, or to obtain a cross section, and select Z(ERR) if you want to look at a sharp surface image.
- Firstly click the plane correction tab to open the plane correction screen. Select polynomial plane correction and click apply.
- To see a 3D profile, click the 3D tab. You can rotate the image by holding down the right mouse button and moving the mouse.
- To acquire a cross sectional profile click line profile tab. Select the oblique option, and drag the mouse over the area you want a cross section of.
- Use “print screen” button to save images as picture files for reports.
Replacing a tip:
You need to replace the tip if:

a) The laser is aligned, but the scale to the right of the red dot alignment reads below the minimum value.
b) No image is obtained when the tip is down and a scan is initiated.

Removing a tip:
Firstly you need to remove the sample and puck from the stage. Next click the stage control tab and click change tip. The scanner will then move to the top of its range. Now move the focus to the top of its range. Click align laser and switch the laser off to prevent any potentially hazardous exposure.

Turn the probe exchange knobs on the side of the scanner head away from you until you can slide it towards you. Slide it towards you and rotate it through 90 degrees.

Using tweezers, grasp the metal substrate ate the top of the probe and rotate the tweezers so the cantilever side of the substrate lifts up off the magnetic mount first.

Put the probe down on the magnetic strip in the probe box so the side opposite the cantilever comes down first. Now gently lower the cantilever side. Make sure you keep new probes and potentially damaged probes separate.
Installing a tip:
Use tweezers to nudge a probe so that the substrate extends over the edge of the magnetic strip in the box.

Grasp the substrate and slowly rotate the tweezers so the side with the cantilever lifts off the magnetic strip first.

Place the probe onto the magnetic mount so the side of the substrate opposite the cantilever fits into the L shaped retainer.

Using the tweezers push the substrate flush against the L.

Now rotate the scanner head back into position, slide it back towards the stage until you feel resistance and then rotate the probe exchange knobs back towards you.

Looking after the microscope:
The tips are very fragile and expensive to replace so a lot of care must be taken while operating the AFM. To increase the lifetime of the tips the following precautions should be followed:

- Always make sure there is adequate clearance between tip and sample when removing or inserting samples as even lightly touching the tip is likely to break it.
- Never use the stage controls when the tip is in contact.
- Never touch the scanner head when the tip is in contact. To prevent this put the protective cover over the stage before you select tip approach.
- Avoid scanning rough areas with dirt/large scratches etc.
- Stay away from the manual tip up/down button in the scan sample section.
- When you leave the microscope always replace the protective cover.