DLF
DYDAKTYCZNE LABORATORIUM FIZYCZNE

Instytut Fizyki Doświadczalnej
Wydział Matematyki, Fizyki i Informatyki
UNIWERSYTET GDAŃSKI

Experiment 38

Surface topography of materials using a scanning electron microscope

KAPITAŁ LUDZKI
NARODOWA STRATEGIA SPÓJNOŚCI

UNIWERSYTET GDAŃSKI

UNIA EUROPEJSKA
EUROPEJSKI
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I. Background theory.

1. Wave – particle duality of particles :

a) de Broglie postulate;
b) Experiments demonstrating the electron's wave properties.

2. Electron beam interactions with matter:

a) elastic and inelastic electrons scattering;
b) Auger effect;
c) secondary and reflected electrons;
d) characteristics of X-rays.

3. The principle of image magnification in an optical microscope.
4. Resolving power of microscopes.
5. Construction of scanning electron microscope (SEM):

a) electron gun;
b) electromagnetic lenses and their role in forming an electron beam;
c) microscope preparation chamber;
d) secondary electron detectors and reflectivity;
e) microscope vacuum system; turbomolecular and diaphragm pumps.

6. Operating principles of a scanning electron microscope (SEM).
7. Image formation and image contrast.
8. Using a scanning electron microscope (SEM) for metallography.
9. Technical parameters of the Hitachi TM – 1000 scanning electron microscope.

II. Experimental tasks.

1. Refer to the TM – 1000 scanning electron microscope (*Picture 1*) technical description in

Picture 1. TM – 1000 Scanning electron microscope (Hitachi) with computer

Appendix A and its instruction manual in *Appendix B.*

2. In order to stabilise the operation of the vacuum pumps, it is necessary to follow a series of start-up procedures involving the formation and breaking of a vacuum in the measuring chamber. To do this, follow the steps detailed in steps 1 – 3 of the manual in *Appendix B.*
3. After breaking the vacuum in the measuring chamber, wear gloves and place the polished alloy sample in the measuring chamber.

   The types of alloy samples to be studied will be made by the laboratory supervisor.

ATTENTION!

Bachelor and master level students may only insert a sample into the SEM with the assistance of the laboratory supervisor. Doctoral students may work on their own using the microscope's manual (*Appendix B*, steps 5 – 8).

4. In order to obtain the required vacuum in the microscope chamber, follow steps 9 – 12 of the microscope's instructions in *Appendix B*.
5. After reaching the desired vacuum, turn on the computer and then run the application TM – 1000. Continue further according to the instructions for observing and saving images with the Microsoft software TM – 1000, found in *Appendix C.*
6. Obtain sharp images of the sample surface.
7. Carry out a material contrast analysis for the polished alloy sample.
8. Associate the phases visible in the obtained images with the atomic numbers of the alloyed elements in the sample (using the "Image Mode" function described in *Appendix C*).
9. Replace the sample with the deeply etched alloy sample, strictly following the instructions in part II. of *Appendix B.*
10. Repeat the measurement procedures detailed in steps 5 – 12 of part I. in *Appendix B.*
11. Obtain sharp images of selected parts of the sample with clearly visible crystalline phases.
12. Analyse the differences in the spatial topography of the samples based on the obtained images.
13. Identify those parts of the alloy surface with clearly visible nucleation sites.
14. Identify and describe the crystallisation process of allow components using the following imaging modes: Normal, Shadow 1, Shadow 2 and TOPO (described in *Appendix C*).
15. Estimate the dimensions of the eutectic structures seen in the recorded images of the sample using the instructions in part III. of *Appendix C*.
16. Include selected images of your test samples in your report, corresponding to steps II.7. - 8. and II.12. – 14.

III. Apparatus.

1. TM – 1000 scanning electron microscope including a system of vacuum pumps.
2. Computer.
3. Set of polished and etched multi-phase material samples (Al – Si and Fe – C alloys).

IV. Literature.

1. A. Lipson, S.G. Lipson, H. Lipson – "*Optical Physics*", Cambridge University Press, 2011.
2. W. J. Croft – "*Under the Microscope. A Brief History of Microscopy*", Hackensack & London: World Scientific, 2006.
3. J. H. Moore, Ch. C. Davies, M.A. Coplan – "*Building Scientific Apparatus*", Westview Press, 2003.
4. R.P. Feynman, R. Leighton, M. Sands – "*The Feynman Lectures on Physics*", Vol. 2., Part 2., Addison – Wesley, 2005.
5. S. Flegler, J. Heckman, K.L. Klomparens – "*Scanning and Transmission Electron",* Oxford University Press, Oxford 1995.
6. H.A. Enge, M.R. Wehr, J.A. Richards – "*Introduction to Atomic Physics*", Wesley, 1981.

Appendix A

Technical specifications of the TM-1000 microscope (Hitachi)

	Items	Spec
1	Accelerating voltage	15 kV (Fixed)
2	Magnification	20-10000x (Image display magnification : 32steps (※1)) (digital zoom : 2, 4 x) Range of the maximum observation : 3.5 mm (square)
3	Electron gun Vacuum	Less than 5×10^{-2} Pa
4	Specimen chamber Vacuum	About 30～50 Pa/1～15 Pa change (Without vacuum level control)
5	Specimen size	70 mm in diameter
6	Specimen thickness	Less than 20 mm
7	Movable range of Stage	X : 15 mm, Y : 18 mm (X, Y only)
8	Electron gun	Pre-centered tungsten hairpin type
9	Diaphragm diameter	Condenser lens : 1.0ϕ ,Objective lens : 0.1ϕ
10	Condenser lens& Objective lens	Permanent magnet type + Electromagnetic type
11	Focus adjustment	Adjustment by Electromagnetic type Objective lens
12	Stigmator coil	Equipped
13	Scan switch	1 s, 10 s, Reduce, 40 s (For preservation)
14	Electron beam axis alignment	Electron gun mechanical alignment
15	Defection system	High-sensitive Semiconductor type BSE detector
16	Control method	Note PC + Specialized micro computer
17	Display	Note PC LCD 15'
18	Image data memory	HDD of PC and other recording medium
19	Frame memory	1280 × 960 pixels, 640 × 480 pixels (only image)
20	Data display	Micron marker, Micron value, date and time, image number comments
22	Auto image adjustment function	Auto focus, Auto brightness
23	Operation support functions	Raster rotation Magnification presetting (2sets available)
24	Evacuation system	Turbo molecular pump : 30 l/s Diaphragm pump : 1 m^3/h
25	Vacuum gauge	Pirani gauge
26	Frequency	50/60 Hz
27	Power Requirements	100 V AC, single phase, 50/60 Hz, 500 VA
28	Physical Dimensions	Main body : 338 (W) × 564 (D) × 513 (H) mm (58.5 kg) Control unit : 140(W) × 525 (D) × 513 (H) mm (23 kg) Diaphragm pump : 145 (W) × 256 (D) × 217 (H) mm (4.5 kg)

Appendix B

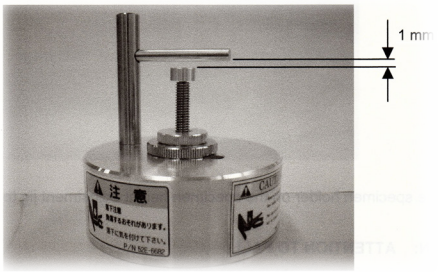
TM – 1000 microscope (Hitachi) operating instructions

I. Preparing the microscope for measurements.

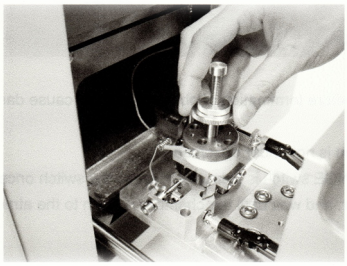
1. Turn on the microscope with the main switch (1 in *Picture 2*).
2. Wait about 3 minutes until the flickering red LED (AIR) (4 in *Picture 2*) goes out and the green LED (READY) is continuously lit. At this moment, there is a vacuum in the microscope's measuring chamber.

Picture 2. Front view of the TM-1000 microscope: 1 – main switch; 2 –specimen stage; 3 – vacuum mode button; 4 – vacuum state indicator LEDs.

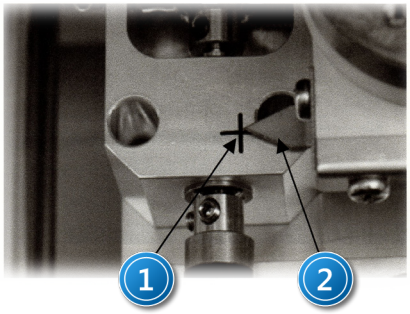
3. Press the Exchange button (3 in *Picture 2*). Again, wait about 3 minutes for the red LED (AIR) to light up – after this, the vacuum in the measuring chamber will be broken and it may be opened.
4. Wear protective gloves.
5. Prepare a measurement sample.
6. Place a sample on the sample holder and check that there is an appropriate distance between the sample surface and the lowest face of the gauge. Use the height adjustment screw (*Picture 3*) to ensure a clearance of about 1 mm.
7. **Slowly** pull out the sample stage from the measuring chamber by gripping the upper part of the front of the stage with your fingers.



Picture 3. Sample holder with height adjustment screw.



Picture 4. Installing the sample holder into the microscope's sample stage.



Picture 5. Adjusting the sample's initial position: 1 – centre; 2 – stage marker.

8. Place the sample holder into the sample stage in the measuring chamber as shown in *Picture 4* (insert the specimen holder **squarely** into the specimen stage).
9. Use the knobs (2 in *Picture 2*) on the front of the measuring chamber to set the stage marker such that it is centred on the "+" (as in *Picture 5*).
10. **Slowly** close the measuring chamber.
11. With one hand, lightly press the front of the measuring chamber and press the "EXCHANGE" button with your other hand. This will re-create a vacuum in the measuring chamber. You may release the front of the measuring chamber when the yellow "LOW" LED is illuminated.
12. Wait for the green (READY) LED to illuminate, indicating that a vacuum has been achieved. The microscope is ready for measurements. Further steps should be performed in accordance with the instructions for the microscope software in *Appendix C.*

II. Sample exchange.

1. After saving the image (according to the procedures in *Appendix C*), reduce the microscope magnification to 100x and click "Stop" (*Picture 6 in Appendix C)* in the upper-left corner of the monitor. This button sometimes does not display "Stop" and "Start" – depending on whether the electron beam is still on or off – this happens if the focussing time on the sample is longer than a few minutes – the program does this automatically to prevent damage to the sample.
2. Press the EXCHANGE button (3 in *Picture 2*). After about 3 minutes, and after the red (AIR) LED is continuously lit, the vacuum in the measuring chamber will be broken.
3. Wear protective gloves.
4. **Slowly** open the measuring chamber and remove the sample table.
5. Replace the sample and repeat the procedure in steps I. 5. – I.12. in *Appendix B.*

III. Turning off the microscope.

1. After saving the image (according to the procedures in *Appendix C*), reduce the microscope magnification to x 100 and click "Stop" (*Picture 6 in Appendix C)* in the upper-left corner of the monitor. This button sometimes does not display "Stop" and "Start" – depending on whether the electron beam is still on or off – this happens if the focussing time on the sample is longer than a few minutes – the program does this automatically to prevent damage to the sample.

   The word "Start" on the button means that the electron beam was turned off and you can continue with the microscope's shut-down procedures.

2. Press the EXCHANGE button (3 in *Picture 2*).

   After about 3 minutes, and after the red (AIR) LED is continuously lit, the vacuum in the measuring chamber will be broken.
3. Wear protective gloves.
4. **Slowly** open the measuring chamber and remove the sample table.
5. Close the chamber.

   Press the EXCHANGE button while lightly pressing the front of the chamber.

   Release pressure only when the yellow (LOW) LED is illuminated.

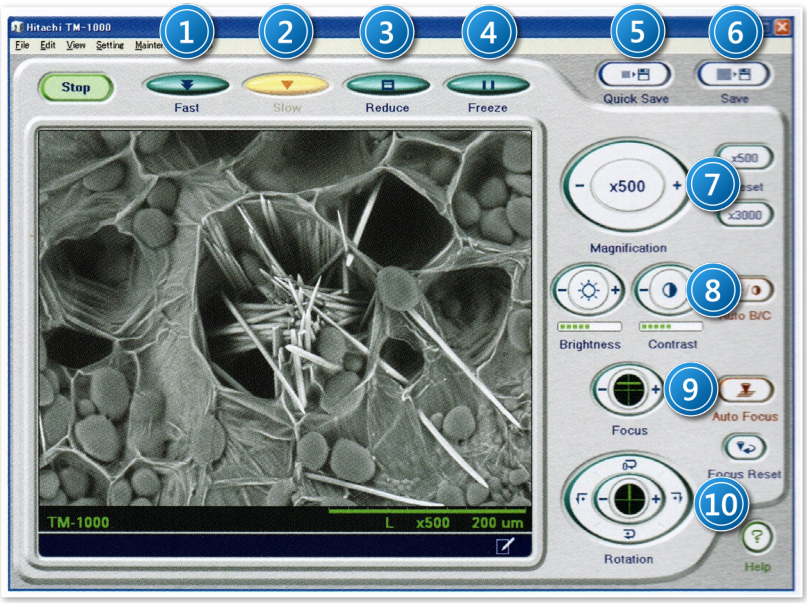
   Wait until the green (READY) LED is illuminated continuously, indicating that a vacuum has been achieved – it should take about 3 minutes.
6. Click the close button in the upper-right corner of the operating monitor window.

   A dialogue will appear – confirm closing the application by clicking “OK”.
7. Turn off the microscope’s main switch (1 in *Picture 1*).

Appendix C

Observing and saving an image

I. Observing an image.



1 – image preview mode;

2 – image saving mode;

3 – limit field of vision;

4 – freeze image;

5 – quick save;

6 – save image;

7 – zoom control;

8 – adjust brightness and contrast;

9 – focus;

10 – rotate sample stage.

Picture 6. Monitor screen with description of interface buttons.

1. Turn on the computer and start the application TM – 1000.
2. Click “Start” in the upper-left corner of the main window. Activating “Auto Start Mode” will enable the electron beam.
3. Set a magnification of x 100. The automatic functions “Auto Brightness and Contrast” and “Auto Focus” should ensure an image of the sample appears on the monitor.

   You should see a view similar to *Picture 6.*
4. Choose an area of the sample by changing the position knobs 2 in *Picture 2.* It is easier at this stage to work with a low magnification and “Fast” mode.
5. Use the automatic functions “Auto Brightness and Contrast” and “Auto Focus” (*Picture 6*) to obtain a clear image of the sample surface.

   If “Auto Focus” does not give a sharp image, you can sharpen the image manually. You should use the “Reduced Area” mode. The following procedures will help to sharpen the image:

   a) use the “Focus” button by clicking + and – to adjust the focus, or hold them down to rapidly and smoothly adjust the focus;
   b) you can use the mouse cursor to mark a chosen portion of the image and, while holding down the left mouse button, drag the mouse left or right several times to adjust the focus.
6. To accurately obtain information about the sample’s topography and material composition, you should use the image processing functions described below, starting in the mode “Slow”

(2 in *Picture 6*) which reduces the scanning speed of the electron microscope. This is the optimal mode necessary for image analysis.

From "View", select "Image Mode".

Save successive images using all four functions (image editing tools) in the "Image Mode" option:

- **Normal** – save an image using the standard (factory) BSE detector settings for the TM – 1000 microscope for maximum counts;
- **Shadow 1** and **Shadow 2** - save an image with other BSE detector positions as compared to the **Normal** mode (to the left or right of the previous position);
- **Topo –** save an image using all three of these functions to give a final image of the sample surface topography.

II. Save an image.

1. After obtaining a clear image of the sample surface, click "File" → "Image" → "Save" (or "Quick Save") to save the image. It will take about 40 seconds. The dimensions of the saved image are: 1280 x 960 pixels.

Hint

When you click "Quick Save", an image will be temporarily saved. Its dimensions will be: 640 x 480 pixels.

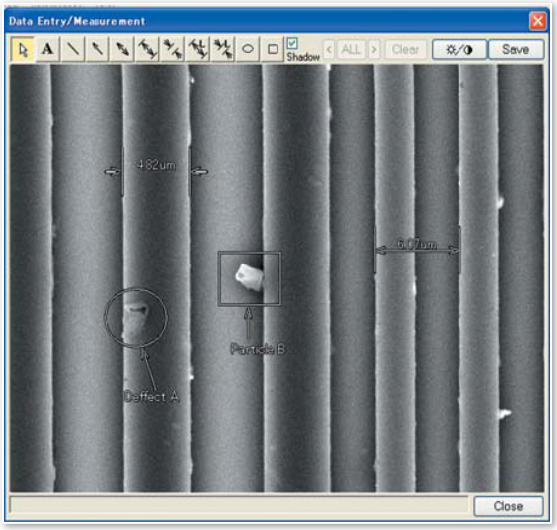
2. After saving, a dialogue will appear.

   Enter the necessary information such as name and destination and confirm by clicking "Save".
3. If you need to measure dimensions within an image, use the instructions in part III of *Appendix C*.
4. To exchange samples, follow steps II.1. – II.4. in *Appendix B*.
5. When you have completed all work with the microscope, you may shut it down by following steps 1. – 7. in part III of *Appendix B.*

III. Measuring dimensions of image elements.

Go to "Edit" → „Data/ Entry/Measurement". A dialogue appears, shown in *Picture 7,* along with a series of function buttons above the recorded image. The symbols on these buttons reflect their function in marking and sizing image elements.

If you wish to undo the current marking, use the button "Clear".



Picture 7. View of a recorded image with dimensioning toolbar buttons.