NTNU Norges teknisk-naturvitenskapelige universitet
Fakultet for naturvitenskap og teknologi
Institutt for materialteknologi

Weeks 6, 7 & 8, 2020

TMT4301 Materialkarakterisering: SCANNING ELECTRON MICROSCOPY

Lab 1a: depth of filed
Specimen: Fracture surface of aluminum (Z = 13).

Image the fracture surface at low magnification (about 50×). Make an image at short (10mm) and long (40 mm) working distance (WD). Apply small (30µm) and big (120 µm) apertures at both WDs Try to make that most of the sample is in focus. Write down the most important parameters: Objective lens aperture, working distance and magnification.
Suppose d_p= 8 nm. Calculate the theoretical depth of field with the selected operational parameters. Apply the electron beam divergence (α) data in the table.

Lab 1b: resolution
Specimen: Tin particles on flat substrate

What are the most important parameters and how will you set these to obtain good resolution in the SEM?
The sample should be investigated at high magnification (> 50.000X). Take micrographs of the sample at short and long WD, and write down the important operation parameters, i.e. magnification, aperture, working distance, probe current and acceleration voltage.
The absorbed current (i_{AE}) can be measured and the probe current (i_p) may be calculated by:

$$i_p = i_{SE} + i_{BSE} + i_{AE}$$

Calculate the theoretical resolution (ex. aberrations) by using the selected operating parameters. Apply the absorbed current to calculate the probe current. Assume i_{se} as 10% of i_p, and find i_{BSE} from the lecture notes. The resolution dp=3nm when the probe current is 30 pA, working distance is 10mm, aperture is 30µm, high voltage 20kV (normal current mode).

Lab 2: Z-contrast imaging and element identification (EDS).
Specimen: Flat polished multiphase metallic sample.

Image the specimen with Z-contrast at 20kV (select an area with more than 2 phases).
Determine the elements in each phase by using X-ray microanalysis (spot mode).
Explain the relationship between intensity and chemical composition of the different phases in the Z-contrast micrograph.
Reduce the acceleration voltage to 5kV and acquire another X-ray spectrum. Compare these 2 spectra from 5kV and 20kV from the same specimen region. What differences do you see? Explain the reason for this.

Task: Each group shall write a short report (including Lab 1 + Lab 2) with caculations, electron images and x-ray spectra within 7 days after Lab 2 was finished. Send the report to stud.ass. Ulrik Aalborg Eriksen ulrikaer@stud.ntnu.no , copy to Jarle Hjelen Jarle.hjelen@ntnu.no

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Beam divergence angle *(α in mrad) at FEG normal current mode*

Working Distance	10mm		40mm	
Apertures	30 micron	120 micron	30 micron	120 micron
Accelerating Voltage 10 kV	4.64	27.8	1.83	11.1
Accelerating Voltage 20 kV	4.21	25.1	1.91	9.88

Beam divergence angle *(α in mrad) at FEG HC mode*

Working Distance	10 mm		40 mm	
Apertures	30 micron	120 micron	30 micron	120 micron
Accelerating Voltage 10 kV	1.46	5.84	0.58	2.32
Accelerating Voltage 20 kV	1.22	4.88	0.48	1.92