MENA 3100 TEM laboratory exercise February 2017

The exercise is divided in two parts:

1) A demonstration on the JEOL 2100F TEM instrument

2) A computer session using the software of Digital Micrograph.

The goal of part 1) is to demonstrate the relationship between the orientation of the specimen and the corresponding image of the diffraction plane and image planes and the use of selected area diffraction and objective apertures.

After the demonstration you should be able to answer the following questions:

a) How does tilting of the specimen affect the contrast seen in imaging?

b) What is the use of the selected area diffraction (SAD) aperture? What is the size of the smallest SAD aperture on our JEOL2100F instrument? Can one obtain diffraction patterns experimentally from smaller areas?

c) How does tilting of the specimen affect the intensities seen in the diffraction plane? Why can one sometime see a ring of reflections outside the central area of the diffraction pattern?

d) When do you use a small objective aperture and when do you use a large aperture? When do you get amplitude contrast and when do you get phase contrast?

e) What are thickness fringes and bending contours?

The goal of Part 2) is to give you an introduction to the use of Digital Micrograph software (commonly used to analyse data obtained from a TEM).

During part 2) you should:

a) Measure d-values (inter planar distances), compare them with tabulated values, index the diffraction spots and find the zone axis of the indexed pattern(s).

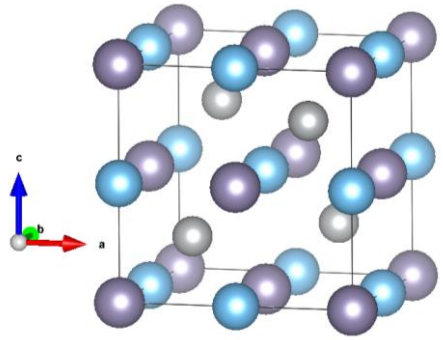
b) Reveal weak reflections on selected area diffraction (SAD) patterns.

c) Perform Fast Fourier transformation (FFT) of high resolution electron microscopy (HREM) images.

Background information:

A small selection of images from half heusler alloys, recorded on JEOL2100, is saved in the folder TEM2017MENA3100.

Phases with the half heusler type structure are cubic with a face centred Bravais lattice and F43m space group (see illustration of TiNiSn below). The lattice parameter, a, is around 6 Å depending on the elements in the unit cell.



Lattice parameters

	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)
	5.94100	5.94100	5.94100	90.0000	90.0000	90.0000
s.u.:	0.00000	0.00000	0.00000	0.0000	0.0000	0.0000

Structure parameters

			x	y	z	Occ.	U	Site	Sym.
1	Ti	Ti1	0.50000	0.50000	0.50000	1.000	1.000	4b	-43m
2	Ni	Ni1	0.25000	0.25000	0.25000	1.000	1.000	4c	-43m
3	Sn	Sn1	0.00000	0.00000	0.00000	1.000	1.000	4a	-43m

Inter planar distances (d-values) of a plane (hkl) in a cubic crystals can be calculated from: $d= a/(h^2+k^2+l^2)^{1/2}$

WEB-emaps can f.ex. be used to get tabulated d-values.

Instructions:

- Open SAD1 in DM
- Define the centre of the diffraction pattern
    - "DiffTools"
    - Locate SAD centre
        - Get image
        - Place the centre mark with the arrows and click "Manual"
- Measure d-spacings
    - "DiffTools"
    - Measure spacings and angles
        - Mark
            - Place the red and green squares on reflections on each side of the central beam (2^{nd} or third in the row from the central beam)
        - Measure
            - Replace the red and green squares to a new set of reflections and measure. Repeat until you have 3 different sets of d-calues.

    The measured d-values are given in nm.

- Compare the measured d-values with tabulated values for TiNiSn or calculated d-values.
- Index the diffraction spots measured. The sum of vectors must internally add up. Hint: Two orthogonal vectors have a dot product equal to 0.
- Find the zone axis of the pattern. Hint: A cross product of two vectors in the plane gives you a vector normal to the plane.
- If time allows, repeat for SAD2 and or SAD3.

In order to reveal weak intensities and details one can use the command "reveal weak reflections" under the DiffTool.

- Reveal weak reflections of the files "SAD1" and "SAD1 after electron radiation".

What is the major difference between the images? Could you see the differences when the contrast was not adjusted?

In many cases the smallest SAD aperture is too large to select an area of interest for a diffraction study. One way of getting alternative information is to perform a FFT analysis of a HREM image.

Open the files:

HREM_Ti05Hf05NiSn_Hf_rik and/or HREM2_Ti05Hf05NiSn_HfTi

The images are from crystals with half Heusler structure, but have Hf partly replacing Ti in the structure.

- Process
    - FFT

- Hold down Alt and mark a square on a HREM image (choose square with dotted line on the menu)
- Process
    - Live
        - FFT

Move the square around. What do you see?

If time allow, try to measure the d-values from the FFT images as you did for the SAD pattern(s).

Do you recognise the zone axes of the two crystals? What effect does Hf have on the lattice parameter of the half heusler phases?

Are there alternative ways of measuring d-values from our data?

Do you know what one should do to minimise the inaccuracy in the obtained d-values

a) when you are on the microscope? b) when you are measuring on your data?

What are the strength and weaknesses of TEM and electron diffraction techniques?