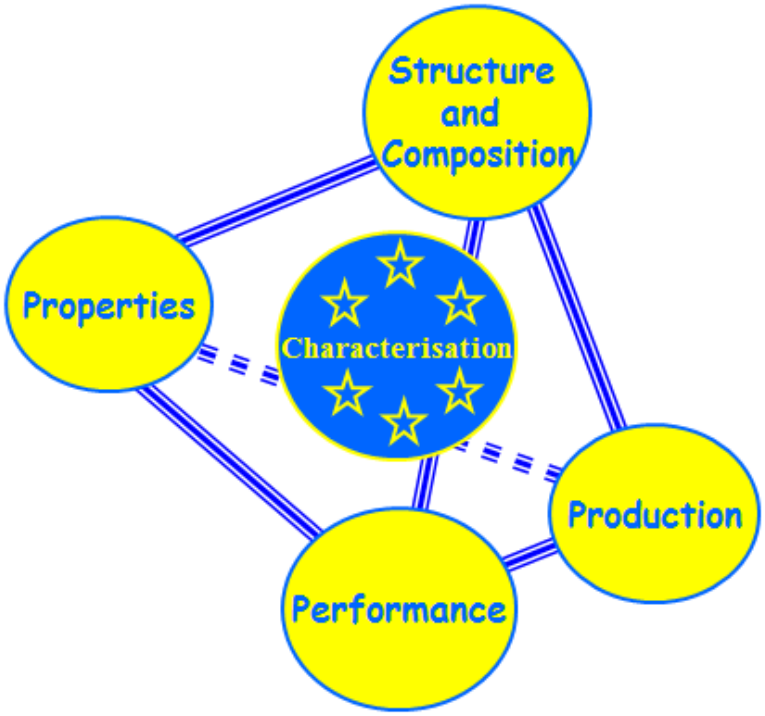
Eskisehir Technical University
Department of Materials Science and Engineering

MLZ 222
Materials Characterisation Techniques Laboratory



Spring 2023-2024

Autumn 2020-2021

Course Instructors

Prof. Dr. Servet TURAN

Assoc. Prof. Sinem BAŞKUT

Asst. Prof. Dr. Umut SAVACI

Laboratory Assistants

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Research Assist. Özlem Başak ÖZKAN KOLCUBAŞI
Research Assist. Enes DÜDEN
Research Assist. Ertuğrul İŞLEK
Research Assist. Emine ERSEZER
Research Assist. Gülseda ŞENEL

Dersin Kodu ve Adı	**:MLZ 222 Materials Characterization Techniques Laboratory**
Bölüm/Program	: **MMF-Mlz.Bil.ve Müh.Böl.-İng.**
Kullanılan Dil	: **İngilizce**
Dersi Veren	**: Prof. Dr. Servet TURAN, Assoc. Prof. Sinem BAŞKUT, Asst. Prof. Dr. Umut SAVACI,**

Dersle İlgili Görüşme Saatleri

Her Salı 14:00-15:00 arası (Ders asistanları ile kendi ilan ettikleri saatte) görüşülebilir.

Genel Amaç

Mühendislik malzemelerinin karakterizasyonu için mikroskobik ve mikroskobik olmayan tekniklerin çalışma prensipleri, sınırları ve ne tür bilgi elde edilebileceği verilerek bir mühendislik probleminin çözümünde ilgili tekniklerin hangisinin seçileceğini bilmesi amaçlanmaktadır.

Genel Yeterlilikler

Etik kurallara uyma, Öğrenmeyi öğrenme, Problem çözme

Öğretim Yöntem ve Teknikleri

Anlatım, Soru-Yanıt, Deney, Örnek Olay İncelemesi, Sorun/Problem Çözme

Dersin Koşulları

Öğrenciler düzenli olarak laboratuarlara katılmakla ve tartışmalarda yer almakla yükümlüdürler.

Öğrenme Çıktıları ve Alt Beceriler

Bu dersin sonunda öğrenci;

Farklı teknikler için numune hazırlayabilecektir.

Işık mikroskobu için neden düz numune hazırlamak gerektiğini açıklar.

Numune hazırlama kademelerini sıralar ve dikkat etmesi gereken noktaları açıklar.

İncelenmek üzere numune hazırlar.

İnce TEM numunesi hazırlar.

X- ışınları (XRD) ile numune tayini yapabilecektir.

X-ışınları difraksiyonu için numune hazırlar.

Bilinmeyen numunelerin x-ışınları difraksiyon paternlerini çözer.

X-ışınları floresan spektrometresi (XRF) için numune hazırlar.

X-ışınları floresan spektrumlarını yorumlar

Işık mikroskobu ile numune inceleyebilir.

Işık mikroskobu tekniklerini kullanır.

Numune dağlayabilir.

Taramalı elektron mikroskobu (SEM) ile elde edilen görüntüleri ve kimyasal analizleri yorumlayabilir.

Taramalı elektron mikroskobunun parçalarını ve dedektörlerin pozisyonlarını tarif edebilir.

Görüntü tekniklerini açıklar.

Kimyasal analiz tekniklerini açıklar.

Geçirimli elektron mikroskobu (TEM) görüntülerini tanımlayabilecektir.

Geçirimli elektron mikroskobu ile ne yapabileceğini tanımlar.

Difraksiyon paternlerini ve görüntüleri tanımlar.

Termal analiz cihazları (TG-DTA-DSC) ile bilinmeyen numuneleri tanımlayabilecektir.

TG tekniği ile elde edilen eğrileri yorumlar.

DTA tekniği ile elde edilen eğrileri açıklar.

DSC tekniği ile elde edilen eğrileri yorumlar.

Dilatometre eğrilerini açıklar.

Bilinmeyen numuneler için hangi teknikleri uygun olduğunu saptayabilecektir.

Bilinmeyen toz bir numuneyi nasıl tanımlayabileceğini açıklar.

Bulk haldeki bilinmeyen bir numuneyi nasıl tanımlayabileceğini açıklar.

Mikro mertebelerde hataları hangi tekniklerle çözümleyebileceğini açıklar.

Nano mertebelerde görüntüleri ve kimyasal analizi nasıl yapabileceklerini açıklar.

Laboratuar kapsamında anlatılan tekniklerin avantaj, dezavantaj ve birbirlerine üstünlüklerini sıralar.

Herhangi bir analiz için neden tek bir tekniğin çözüm olamayacağını açıklar.

Ders Kitapları

* Electron Microscopy and Analysis, PJ Goodhew, FJ Humphreys ve R. Beanland, Taylor and Francis, 2001
* Scanning Electron Microscopy and X-ray Microanalysis, J.I. Goldstein et al., Plenum Press, New York, 2003
* Handbook of Sample Preparation for Scanning Electron Microscopy and X-Ray Microanalysis, P. Echlin, Springer, 2009
* Metallographic Etching: Techn. for Metallography, Ceramography, Plastography Gunter Petzow, G. Petzow, ASM International, 1999
* Elements of X-ray Diffraction, B.D. Cullity ve S.R. Stock, Prentice Hall, 2001
* An Introduction to the Optical Microscope, S. Bradbury, Oxford University Press, 1989
* Thermal Analysis of Materials, R.F. Speyer, Marcel Dekker Inc., 1993
* Transmission Electron Microscopy: A Textbook for Materials Science, D.B. Williams ve C.B. Carter, Springer, 2009

(MLZ 222 Materials Characterization Techniques Lab)

No	Description of Experiment	Date of Experiment	Responsible Person	The Laboratory No
1	**XRD & XRF**	11-15/03/2024	Ertuğrul İŞLEK	MLZ 117
2	**Thermal Analyses**	18-22/03/2024	Ö. Başak ÖZKAN KOLCUBAŞI	MLZ/S 208
Arasınav: 25/03-6/04/2024				
3	**Sample Preparation**	15-19/04/2024	Kübra GÜRCAN BAYRAK	MLZ 120
4	**Sample Preparation**	22-26/04/2024	Enes İ. DÜDEN	MLZ 120
5	**Light Microscopy**	29/04-03/05/2024	Gülseda ŞENEL	MLZ 119
6	**SEM & Chemical Analyses**	13-17/05/2024	Emine ERSEZER	MLZ 121
7	**TEM & Chemical Analyses**	20-24/05/2024	Umut SAVACI	MLZ 121
03-13/06/2024 Dönem Sonu Sınavları				

Group E: Monday 14:00-16:00

Group F: Monday 16:00-18:00

Group B: Tuesday 11:00-13:00

Group G: Tuesday 15:00-17:00

Group D: Wednesday 17:00-19:00

Group C: Thursday 09:00-11:00

Group A: Friday 14:00-16:00

Harf Notu Nasıl Belirlenecek?

Alınan en yüksek notun kaç olduğuna, sınıftaki öğrencilerin davranışlarına, derse olan ilgilerine (derste ne kadar soru sorulduğu, ders notlarının dersten önce ve sonra ne kadar okunduğu vb) ve özellikle final sınavında öğrencilerin başarı durumuna göre alt ve üst sınırlar belirlenecektir.

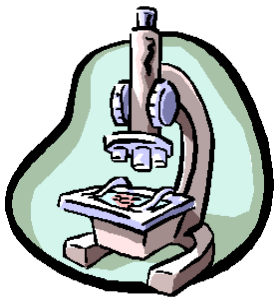
General Instructions for the Lab

1. It is extremely important that you read each experiment and basic references prior to the lab. There might be an exam for each laboratory subject before the lab session.

2. The nature of working in groups implies that there should be cooperation and discussion between members of the group and the lab instructor.

3. Students must attend each lab on the specified date in a specified group. The students is admitted to the class within the first half an hour.

Çok zevkli olduğuna inandığımız bu dersinizde hepinize BAŞARILAR dileriz…



EXPERIMENT # 1

MATERIALS CHARACTERIZATION WITH XRD

1.Objective of the Experiment

Understanding the practice of x-ray diffraction and qualitative phase analysis of an unknown sample using XRD.

2. What should you know before the experiment?

You should know;

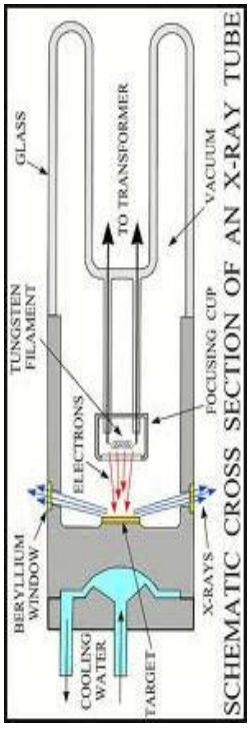
- How to generate X-rays
- The main properties of X-rays
- Derivation of Bragg law and diffraction.
- How and why to obtain monocromatic X-rays
- Determining the factors for the position (x-axis) and intensity (y-axis) of XRD pattern
- What are the other meanings of XRD pattern?
- What is the importance of structure and atomic scattering factors
- How to calculate peak intensities
- Diffraction conditions for different structures
- How to identify unknown phases with Hanawalt method

3. What will you learn during the experiment?

You will learn;

- Sample type and quantity
- Importance of sample preparation in XRD
- Which equipment parameters affect the peak intensity and position
- Why and when we need to use Si powder in XRD
- How to use XRD equipment and how to qualitatively analyse the patterns by Hanawalt method
- How to identify the patterns from amorphous or crystalline materials
- How to use the search-match (Jade) program.
- How to index XRD patterns
- How to quantify the different phases

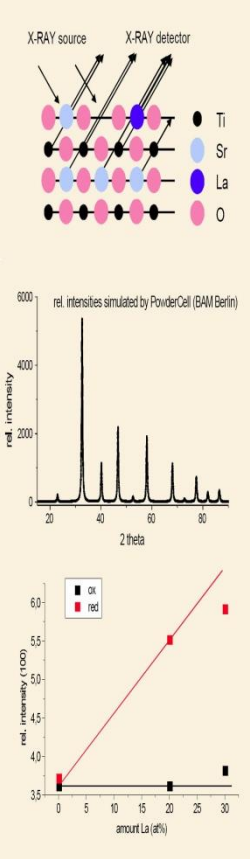
4. Schematic view of experimental procedure



If the sample to be analysed is in bulk form, then, at least one surface of the sample must be perfectly flat.

If the sample is in powder form, then, it must be less than 60 μm in size.

The sample is placed (if it is in the bulk form) or pressed
(if it is in the powder form) into the sample holder.



XRD tube voltage (for instance 40 kV) and current (for instance 30 mA) values are adjusted

Select the scan speed and θ values and start the analysis

After analysis search-match the all peaks on the pattern by using XRD software which is based on the Hanawalt

Indeks the XRD pattern and if necessary quantify the phases

5. Equipments and materials

- Powder, bulk sample and XRD sample holder
- XRD instrument (Rigaku Rint 2200) and XRD software
- Hanawalt book

6. Important points / hints for the equipments and / or results obtained from the analyses

- Powder sample particle size must be under 63 micrometer
- Sample surface must be smooth and same level as the holder
- Be careful about opening the XRD equipment door.

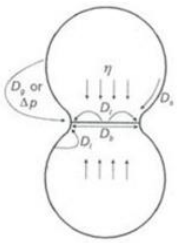
EXPERIMENT # 2

THERMAL ANALYSIS OF MATERIALS

1.Objective of the Experiment

To determine weigth loss, evaporation, oxidation, dehydration, crystal formation, polymorphic transformation by thermogravimetric and differential thermal analysis (TG and DTA) and expansion-shrinkage behaviour of materials by dilatometer with response to changing temperature.

2. What should you know before the experiment?



You should know,

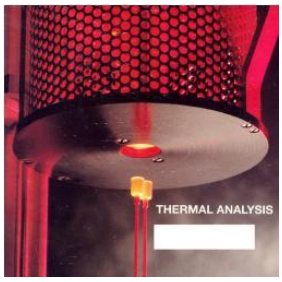
- For which information do we need to use TA instruments?
- What are the causes of weight loss or gain in materials?
- What are the causes of phase transformations in materials?
- What are the causes of volume expansion or shrinkage in materials?
- How the TA instruments work?
- How to draw theoretical curves for TA of materials containing different amount of different phases?
- What are the differences of diferent instruments in terms of information obtained ?

3. What will you learn during the experiment?

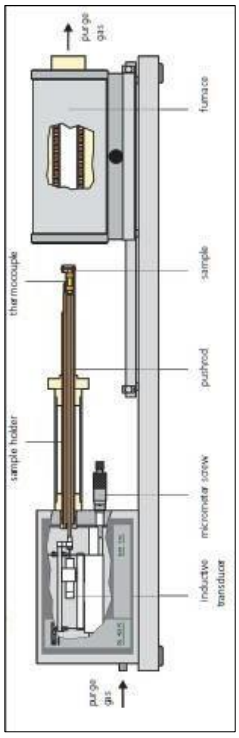
You will learn;

- How to prepare samples
- How to put samples into the instrument
- How to calibrate the instruments
- How to identify
  - mass changes
  - decomposition behaviour
  - thermal stability
  - oxidation behaviour
  - transition enthalpies
- How to identify
  - glass transitions
  - softening points
  - crystalisation temperatures
  - linear thermal expansion
  - determination of the CTE
  - sintering temperature
  - volumetric expansion
- How to calculate the amount of different phases in the mixture

4. Schematic view of experimental procedure



Instrument temp. should be between 22-24°C for starting the measurement.



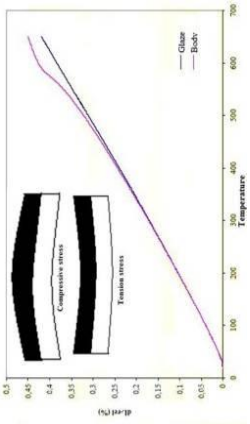
Select sample holder, thermocouple, furnace and check other settings.

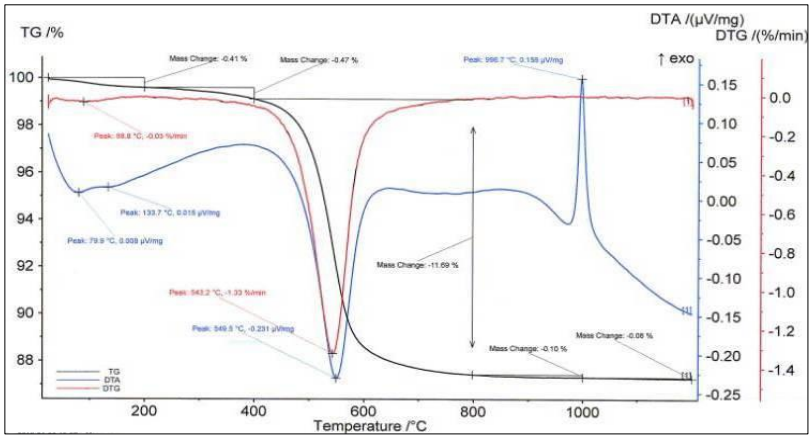
Correction should be done.

Define the measurement conditions (start & end temperature, heating rate, atmosphere must be identical with the future measurement conditions).

Samples are dried in an oven at 60°C for 12 hours.

Sample can be placed in the measuring unit and instrument can be adjusted.





5. Equipments and materials

- Samples and reference materials
- Different type of crucibles
- Sample carriers
- Simultenous thermal analyser
- Dilatometer

6. Important points / hints for the equipments and / or results obtained from the analyses

- Be very carefull about the influence of sample preparation, material homogenity, measurement condition
- Sample must be in powder form and it must be smaller than 63μm in size for STA measurement.
- Sample dimensions should be 5x5x10mm for unfired and 5x5x25mm for fired samples to make dialatometer measurement
- Crucible selection and measurement sensitivity are important
- Do not use your mobile phone during the experiment
- do not touch instrument and even the desk that instrument is placed on during the experiment
- Baseline is important
- Differential of TG curve, i.e., D-TG, is useful for interpretations

EXPERIMENT # 3

SAMPLE PREPARATION

1.Objective of the Experiment

The aim of the experiment is to learn how to prepare efficient samples and to learn the importance of sample preparation for characterisation techniques.

2. What should you know before the experiment?

You should know;

- What is the importance of characterisation in Materials Science and Engineering.
- Classification of the characterization techniques.
- What are the main stages for sample characterisation.
- Explanation of the important factors at each stages
- Why is sample preparation important.
- What are the main stages for sample preparation.
- Why is automatic preperation important.
- What are the main parameters for cutting.
- What are the mounting techniques? How can you choose the appropriate mounting technique.
- Why and when is vacuum impregnation needed.
- What are the main parameters for polishing.
- What is etching and what is it used for.
- What are the differences between light microscope sample and a TEM sample.
- What are the differences between light microscope and TEM sample preparation procedures.

3. What will you learn during the experiment?

You will learn:

- how to prepare samples for microscopical investigations (light microscopy/SEM and TEM)

4. Schematic diagrams of the experiment

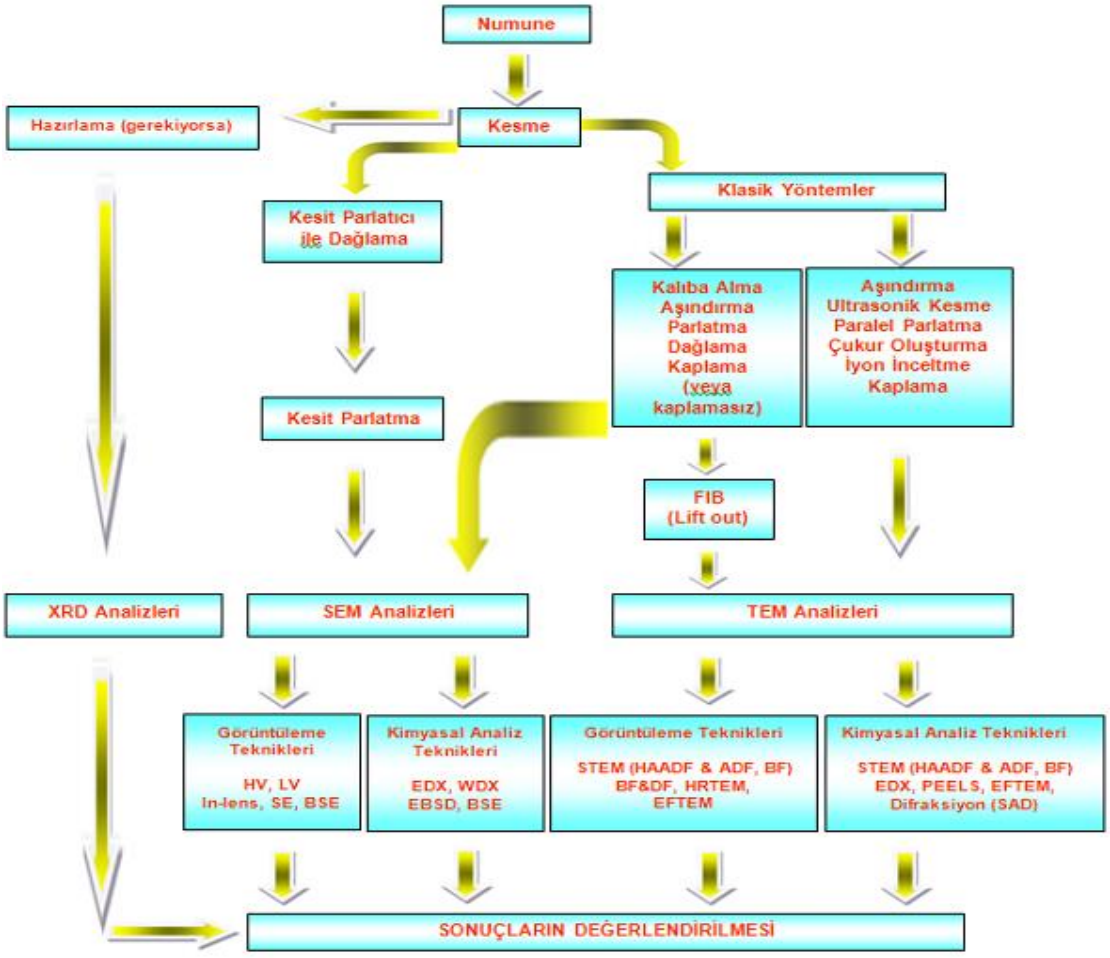


Fig.1. Schematic diagram of sample preparation stages and characterization techniques.

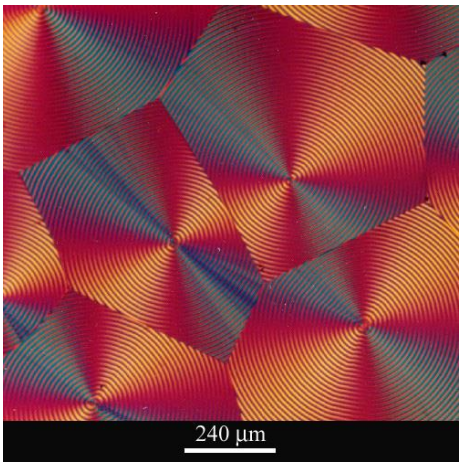
Each group would select a sample and prepare it with what they learnt during sample preperation experiment. Then, in the following experiments, they will investigate the microstructure of the un-etched and etched samples with different techniques.

5. Equipment and Materials

- Cutting equipment and cutting discs
- Hot/cold mounting equipment and consumables (Bakalite, epoxy, etc)
- Automatic or semi-automatic polisher machine
- Abrasive (SiC) papers, polishing clothes
- Wax, disc grinder and lapping films
- Ion beam thinner (IBT), Cross-polisher(CP), Ion Slicer(IS) and coating equipment

6. Important points / Hints

- You must be careful with cutting parameters not to introduce deformation to the sample.
- You must choose the appropriate mounting technique according o your procedure.
- You must be careful with the grit sequence of abrasive papers not to damage your sample.
- You must pay attention to the surface of your sample after polishing.



EXPERIMENT # 4

LIGHT MICROSCOPE

1.Objective of the Experiment

To show how to use the light microscope, inverted light microscope and stereo microscope by investigating different samples with different techniques.

2. What should you know before the experiment?

You should know;

- Resulting signals from the interaction between light and solid?
- Snell law?
- Airy discs?
- Refractive index?
- How can we see?
- How rainbow occurs?
- What is the wave length of light?
- How to calculate theoretical image resolution?
- How to increase the resolution of a light microscopes?
- Name of the aberrations that might reduce the practical resolution of the microscopes?
- Explain how the aberrations ocur and how they are

3. What will you learn during the experiment?

You will learn:

- **The use of light microscope and how to choose the right microscopy technique to match your sample and aim.**

4. Schematic diagrams of the experiment

Each group will investigate and label the microstructure of the un-etched and etched samples with different techniques.

5. Equipment and Materials

- Light Microscope (Olympus BX 60M)
- Stereo Microscope (MEIJI)
- Inverted Light Microscope (……)

6. Important points / Hints

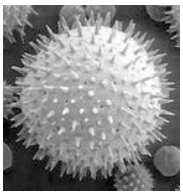
- You must recognize how to choose the right microscopy technique in which conditions.
- You must pay attention to the limitations of the technique/microscope you use.

EXPERIMENT # 5

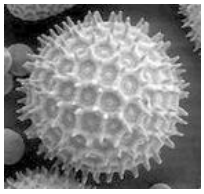
MATERIALS CHARACTERIZATION WITH SEM

1.Objective of the Experiment

To show how to use the SEM by characterizing microstructures of different samples with different techniques under different microscope parameters.



2. What should you know before the experiment?



You should know;

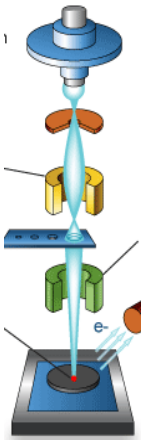
- Comparison of light with electrons
- Resulting signals from the interaction between electrons and solid and their use in microscopy
- Relative energies of SE, BSE and X-rays
- Interaction volume for different signals and importance for collection
- Name of the basic parts of SEM and their roles
- Difference between SE and BSE e- in terms of the information obtained
- Differences between different electron guns

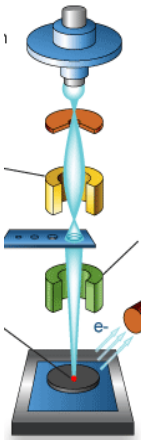
3. What will you learn during the experiment?

You will learn;

- where all the parts in the microscope
- how to obtain different images (secondary electron, backscatter electron and in lens images).
- how to select microscope parameters (accelerating voltage, working distance, aperture size) to obtain best information on polished 2D and 3D sample
- how to investigate non-conducting samples without coating
- how to adjust the microscope for best images.

4. Schematic view of experimental procedure





Select the 2D, 3D, conductor and non-conducting specimens which represent the material to be investigated.

↓

Fix the samples onto the stub or SEM holder.

↓

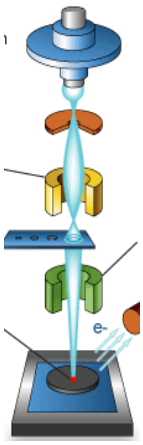
Vent the sample chamber's vacuum in SEM and place the holder into the SEM chamber.

↓

Pump the vacuum and when it reaches the requested value start the filament current

↓

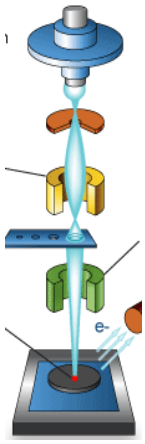
Increase the accelerating voltage



Press the imaging mode, find the sample and focus the image

Image the sample on SE, BSE and in lens modes

Carry out necessary adjustments during experiment (whobble, stigmation, brightness, contrast)

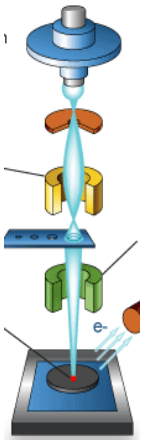


Change the microscope parameters
to show their effect on different SEM imaging modes

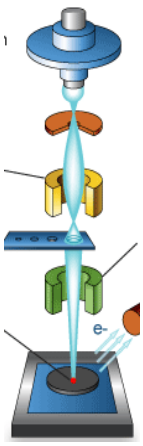
Find the non-conducting sample
to observe the charging problem in secondary electron

Select the variable pressure mode for non-conducting

Obtain images in VP mode and study appropriate conditions for VP mode



Decrease the accelerating voltage gradually and shut the SEM down



Closed the filament current

Vent the chamber and remove the sample holder and pump system vacuum

5. Equipments and materials

- Conductive and non-conductive samples
- SEM sample holder
- Carbon tape, stub and screw
- Scanning Electron Microscopy (Zeiss, SUPRA 50 VP)
- Secondary electron detector
- Backscatter electron detector
- In lens detector

6. Important points/hints for the equipments and/or results obtained from the analyses

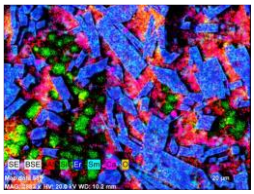
- Hold the sample holder with gloves to keep the sample and vacuum chamber from impurities and incorrect analysis
- Be very careful about damaging the gun vacuum system. You can only vent the sample chamber's vacuum system.
- During the change of the working distance, you must be in TV mode.
- Different microscope working parameters have different effects in different imaging modes.
- Interaction volume is important for different imaging and analysis modes.

EXPERIMENT # 6

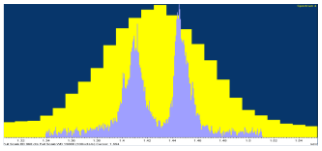
CHEMICAL ANALYSIS IN SEM

1.Objective of the Experiment

To show how to carry out chemical analysis of different samples with energy dispersive x-ray (EDX) and wavelenght dispersive x-ray (WDX) microanalysis techniques in SEM.



2. What should you know before the experiment?



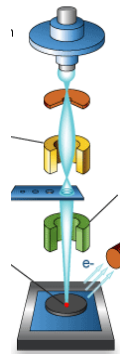
- All the "you should know and what will you learn" sections in experiment 3
- Meaning of microanalysis
- How to produce different x-rays
- What is the difference between all different type of x-rays
- What is the importance of accelerating voltage on the type and number of x-rays
- Which elements could not be detected during x-ray analysis?
- To obtain at least one signal from each element from the periodical table what is the minimum accelerating voltage?
- Interaction volume of x-ray signals for light and heavy elements and the importance of accelerating voltage on the interaction volume
- How to detect x-rays and convert it to x-ray spectra?
- What is the meaning of x and y-axis in the x-ray spectra?
- Meaning of spectral resolution and spatial resolution?
- The differences between point, line and area analysis?
- What are the advantages and disadvantages of EDX and WDX
- Diffraction and Bragg's Law
- Why do we need to use different crystals in WDX analysis

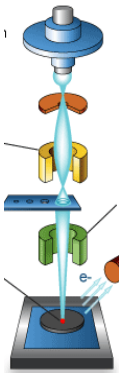
3. What will you learn during experiment?

You will learn;

- How to carry out point, line and area analysis which parameters are important for EDX analysis.
- How to map different elements
- How to quantify different elements
- How to carry out WDX analysis.
- which parameters are important for WDX analysis.
- limitations of two dedectors.
- the microscope parameters (accelerating voltage, working distance, aperture size) effect EDX and WDX analysis.

4. Schematic view of experimental procedure

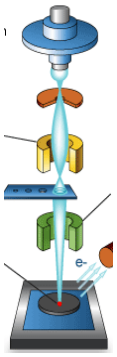




Applying chemical analysis to selected sample with point, area, linescan and mapping techniques by using EDX dedector.

Investigate the important parameters (live time, process time, dead time, acquisition rate and input count rate) for EDX analysis.

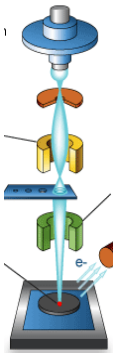
Showing the effect of microscope parameters (accelerating voltage, working distance, high current, slit size..) on EDX analysis.



See how the overlapped peak occur in EDX spectrum.

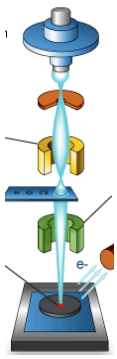
Seperating of overlapping peak by EDX-WDX combine mode.

See the effect of some parameters (scan speed, slit size..) on EDX-WDX combined analysis.



Applying mapping and line scan analysis by using WDX technique.

Comparing the results obtained from EDX and WDX



5. Equipments and materials

- Equipment and materials same as Experiment 3 and the following
- Energy dispersive x-ray dedector (EDX) (INCA Energy)
- Wavelength dispersive x-ray dedector (WDX) (INCA Wave)

6.Important points / hints for the equipments and/or results obtained from the analyses

- Each techniques have different microscope working conditions. For example, working distance should be 8mm for best EDX analysis and 15mm for best WDX analysis. (these parameters could change in different microscopes)

- Interaction volume is the important parameter in chemical analysis. If you want to obtain the chemical information from the region close to the surface you should select the low accelerating voltage.
- WDX dedector is 10X more sensitive than EDX dedector.
- Trace elements analysis (down to below % 0.01) could be obtained by using WDX technique.

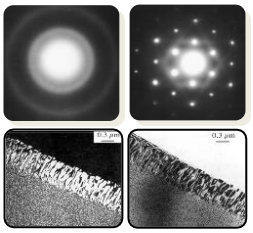
EXPERIMENT # 7

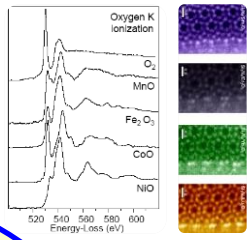
MATERIALS CHARACTERIZATION WITH TEM & CHEMICAL ANALYSES II

1.Objective of the Experiment

To show how to use the TEM by characterizing microstructures of samples with different techniques

2. What should you know before the experiment?





You should know;

- All the "you should know & what will you learn" sections in exp 3 & 4.
- What are the name of basic components of TEM and their roles?
- What are the differences between SEM & TEM?
- Which signals are most commonly used in TEM?
- How BF, DF and SAD images are formed in TEM?
- How BF, ADF and HAADF images are formed in STEM theoretically?
- What are the advantages & disadvantages of EDX versus EELS & EFTEM technique?
- What kind of information could be obtained from BF, DF, HREM images and diffraction patterns in TEM; BF, ADF, HAADF images in STEM; an EELS spectrum and an EFTEM elemental maps?

3.What will you learn during the experiment?

You will learn;

- Where all the parts in microscope?
- How to align the microscope?
- How to obtain different images (BF, DF, HREM) and diffraction patterns in TEM ?
- How to obtain different images (BF, ADF and HAADF) in STEM ?
- How to carry out chemical analyses (EDX, EFTEM and EELS)?

4.Schematic view of experimental procedure

Explanation of TEM components

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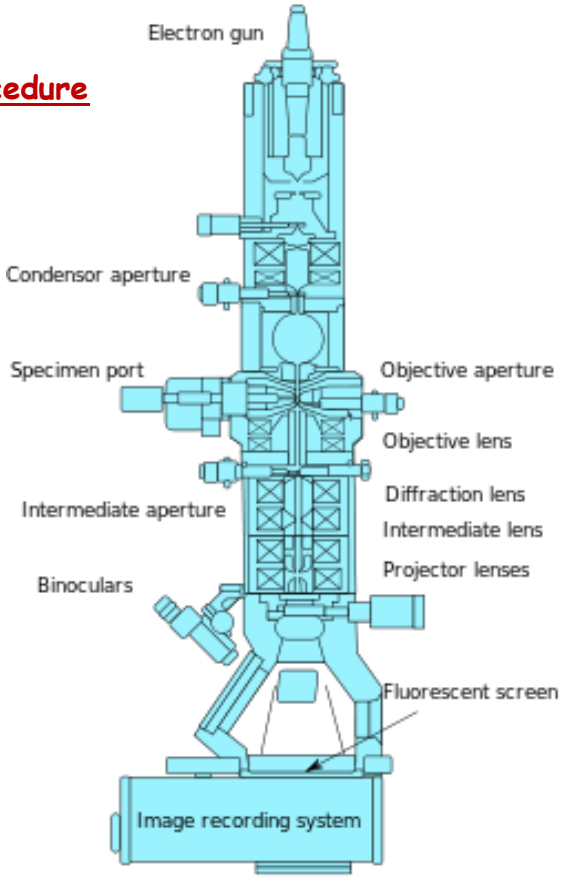
Aligning the microscope

↓

Demostration of BF, DF, HREM and diffraction pattern image in TEM

↓

Demostration of BF, ADF, HAADF image in STEM



Demostration of EFTEM Three Window Elemental mapping

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Demostration of EELS analyses both in TEM & STEM mode

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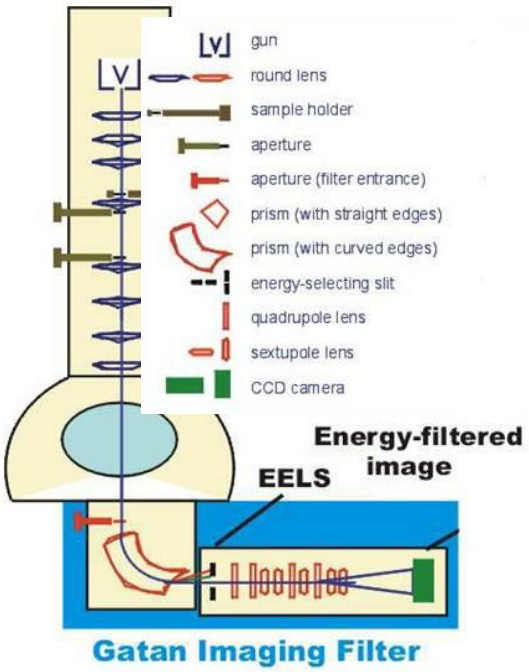
Demostration of advanced techniques (EFTEM SI & STEM SI)



5.Equipments and materials

- 200 kV field emission TEM (JEOL™ JEM-2100F) STEM
- STEM-HAADF detector (Model 3000, Fischione)
- EELS and energy filter (Gatan™ GIF Tridiem) and EDX (JEOL™ JED-2300T).
- Any sample prepared from ceramic, metal or composite material.

6. Important points / hints for the equipments and/or results obtained from the analyses

- Preliminary characterization of the sample with other techniques such as XRD, SEM
- TEM techniques should be used if you can not solve your problem with other techniques
- Thin and well prepared sample.
- Perfect alignment of the microscope

- Choosing the right TEM technique.
- Reasonable interpretation of the results