PRACTICAL WORKBOOK

MY-407: Design, Selection & Characterization of Engineering Materials

Name________________________

Roll No______________________

Batch________________________

Year ________________________

Department of Metallurgical Engineering

NED University of Engineering and Technology

PRACTICAL WORKBOOK

MY-407: Design, Selection & Characterization of Engineering Materials

PREPARED BY:

MUHAMMAD SAMI (PhD)

This is to certify that this practical book contains 51 pages

Approved by:

Chairman
MYD

Department of Metallurgical Engineering
NED University of Engineering and Technology

CERTIFICATE

It is certified that Mr. / Ms. ____student of class _____Batch_____, bearing Roll No. MY _____ has completed his / her coursework in **Design, Selection & Characterization of Engineering Materials (MY-407)** as prescribed and approved by the Board of Review of the Metallurgical Engineering Department.

His/her performance is reflected by the performance rubrics of his/her practical workbook. The student's overall performance will address the assigned learning attribute.

____

Course Teacher

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MY-407: DESIGN, SELECTION & CHARACTERIZATION OF ENGINEERINGMATERIALS

Experiment No. 1

Aim of the Experiment:

Safety precautions to be taken during the handling of characterization equipment.

Instructions:

1. Wash your hands before entering in the lab and wear protective clothing, such as lab coats or aprons, gloves, and eye wear. Be sure that your work area should be clean and dry.
2. Never attempt to operate any equipment without prior instruction.
3. Work in the laboratory only when a lab instructor is present, and only on authorized experiments.
4. Do not bring any unnecessary items into the lab. Do not place any personal items (purses, book bags, coats, umbrellas, etc.) on the lab table or at your feet.
5. Make sure all apparatus is supported and squarely situated on the table.
6. Do not put anything in your mouth while in the lab. Never eat, chew gum, drink, taste chemicals, mouth pipette, lick labels, smoke, or store food or drink in the lab. DO NOT bring food and drink into the laboratory.

Safety precautions in electron microscopy laboratory:

Samples and Chemicals

1. Always check the safety warnings before using any chemicals
2. Check the relevant Material Safety Data Sheets -MSDS
3. Handle samples and equipment with disposable gloves.
4. Open volatile or reactive chemicals such as aldehydes in a fume hood or well-ventilated room

Sharp Objects

Glass knives, razor blades and scalpel knives are still sharp after being used. Discard only in containers provided for that purpose, or well-sealed, and labelled cardboard boxes, so cleaning personnel will not be injured.

Equipment

Treat all fixatives with respect

1. Most vacuum evaporators do not have a safety switch to turn off power before opening the bell jar. Follow the instructions on the equipment. Never observe metal evaporation without goggles. The intense brightness can burn your retina.
2. Not more than 6 people should ever be in the SEM lab
3. Take of your shoes before entering the SEM lab
4. Always wear gloves before handling a sample or operating the equipment
5. Never lean on the table or support of the microscope
6. Do not touch equipment without permission of the supervisor
7. Follow specific safety guidelines for every particular equipment.

Radiation protection procedures:

The electron microscope is a potential source of ionizing radiation that can be dangerous. Although it complies with international safety standards it is important to keep the required set of safety procedures:

1) Understand and apply the three cardinal principles of radiation protection and control: time, distance, and shielding.
2) Analyze the hazards of each job in advance.
3) Provide safeguards against foreseeable accidents.
4) Use planned emergency procedures when accidents happen.

Electrical safety:

At a resistance of approx. 1kOhm (hand-feet) you only need a 50V tension to bring yourself into mortal danger. HT equipment components especially have potentially lethal voltages. As a good safety precaution, always expect a hazardous voltage in an unknown circuit. Only trained personnel and operators should be called in to carry out an electrical maintaince check if equipment is suspected to be malfunctioning.

Some electricity related accidents cause:

1) Loss of muscle control
2) Spasms & Involuntary movement
3) Inability to let go
4) Burns - external & internal
5) Failure of Life Support muscles: Diaphragm and breathing

Risk and hazard analysis in design:

The objective of risk and hazard analysis is to identify the level of risk and to pinpoint the parts of the system that represent the greatest risk for failure. Then, if the analysis is used properly, steps can be taken to eliminate the cause or reduce the risk to an acceptable minimum. It can be produced when actions are taken at all levels that are based on:

1. Attention to past experiences with similar systems.
2. Availability of risk information for all project personnel
3. A sound, aggressive risk and hazard analysis during all phases
4. Development of suitable corrective action and safety programs based on the analysis
5. A continuous and searching review of all phases of the program efforts

Questions

1. Name one hazard and safety measure to take while dealing with liquid nitrogen.

2. If you were put in charge of the SEM lab describe the steps you would take to ensure all safety requirement are met.

3. Describe a couple of precautions you should take while dealing with HT equipment

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Psychomotor Domain Assessment Rubric-Level P3					
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Weighted CLO (Psychomotor Score)	
Remarks	
Instructor's Signature with Date:	

Experiment No. 2

Aim of the Experiment:

Manipulate phase analysis on steel specimen via Quantitative Metallography and compare your results from microscopic techniques.

Theory:

Quantitative Metallography: After the first microscopes were created, one of the next logical questions to follow was how big a particular feature was or how much of some constituent was present. From these questions, quantitative microscopy had its roots. The next logical question to arise was how to relate observations made from two dimensional fields of view to three dimensions; this analysis is termed stereology. Initially, the procedures developed to perform stereological measurements were based on laborious time-consuming measurements. As television and computer systems were developed, and matured, powerful image Analysis Systems (I/A) were created. Today many measurements and calculations that previously required many hours to perform can be made in minutes or even micro-seconds.

"The determination of specific of microstructures using quantitative measurements on micrographs or metallographic images is called Quantitative metallography."

Techniques used for quantitative analysis:

- Phase proportions
- Point counting method
- Grain size
- Interlamellar spacing

Nomenclature:

Application of stereology has been hampered by confusion due to the use of different mathematical symbols for the same parameters.

To minimize this problem, the International Society for Stereology has promoted a standard nomenclature which is as follows:

P = Point, L = Line, A = Area, S = Surface, V = Volume, N = Number

These symbols can be combined in a number of ways to generate different symbols.

e.g., P_P represents the point fraction; that is, the fraction of grid points lying in a phase of interest. S_V represents the grain boundary surface area per unit volume. N_A is the number of particles per unit area while N_V is the number per unit volume.

1. Phase proportions:

There are three different methods of determining the amount of phases present:

Areal analysis: says that the area percent of a phase on a 2-D plane is equal to its volumetric percent, that is, $A_A = V_V$

Lineal analysis: says that the lineal fraction of test lines in a phase on the 2-D plane is equal to its volumetric percentage i.e., $L_L = V_V$
Volumetric analysis: Starting around 1930, several workers in different fields and countries showed that the percentage of points on a test grid lying in the phase of interest was equal to the volumetric percentage, that is, $P_P = V_V$

2. Point counting method:

ASTM E 562 describes the point counting procedure for determining the amount of second-phase constituents.
A grid with systematically spaced points (e.g., 10 rows of 10 equally spaced points) is superimposed over the structure, either on an eyepiece reticle or a plastic sheet placed over or behind a ground glass projection screen or on a TV monitor screen.

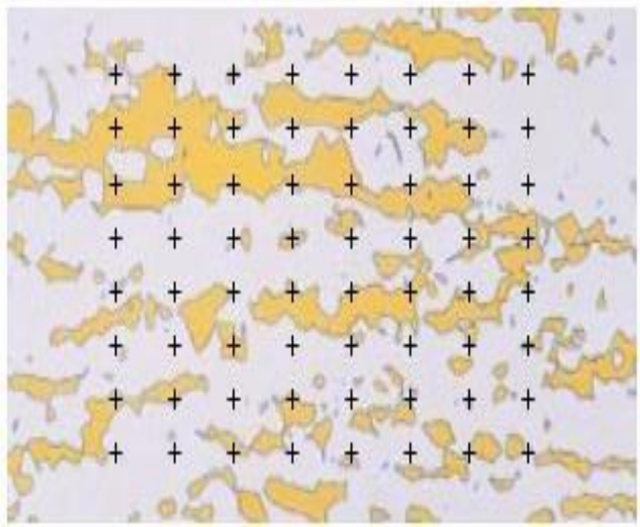

Figure1. Point CountingThe microstructure above shows the beta phase in Muntz metal (Cu-40% Zn) preferentially collared by Klemm's I reagent

3. Grain size:

There are three basic methods for grain size estimation recommended by the ASTM is:

a. Comparison Method
b. Intercept (or Heyn) Method
c. Planimetric (or Jeffries) method

4. Interlamellar spacing:

The spacing between second-phase particles, such as carbides or inclusions and pearlite in steels can affect mechanical properties and formability.

Spicing are easily assessed using a simple N_L (number of particles intercepted per unit length of test line) measurement. The mean center-to-center spacing, sometimes called σ, is simply:

$$\boldsymbol{\sigma = 1/N_L}$$

Observation:

S No.	Specimen	Chemical composition (major alloying elements)	Treatment (i.e. Annealed, hardened etc)	Phases present (i.e. binary or ternary)

Point counting (grit method)

	Phase 1	Phase2	Phase 3
Amount			

Point counting (via microscopy)

	Phase 1	Phase2	Phase 3
Amount			

Grain size measurement:

S no.	Manually	Image Analyzer
1		
2		
3		
4		
	(Mean) ∑=	(Mean) ∑=

Results:

Questions

Determine the average grain size of your sample using both intercept method. Show the measurement and calculation.

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Experiment No. 3

Aim of the Experiment:

To **operate** the various functions of Image Analyzer.

Theory:

The microscope illustrated in Figure 1 is an Olympus GX51 research microscope. This microscope represents the latest state-of-the-art design that incorporates multiple illuminators (episcopic and diascopic), analyzers and polarizers, DIC prisms, fluorescence attachments, and phase contrast capabilities. The photomicrography system is the ultimate in sophistication and performance featuring spot measurement, automatic exposure control, and zoom magnification for flexible, easy framing.

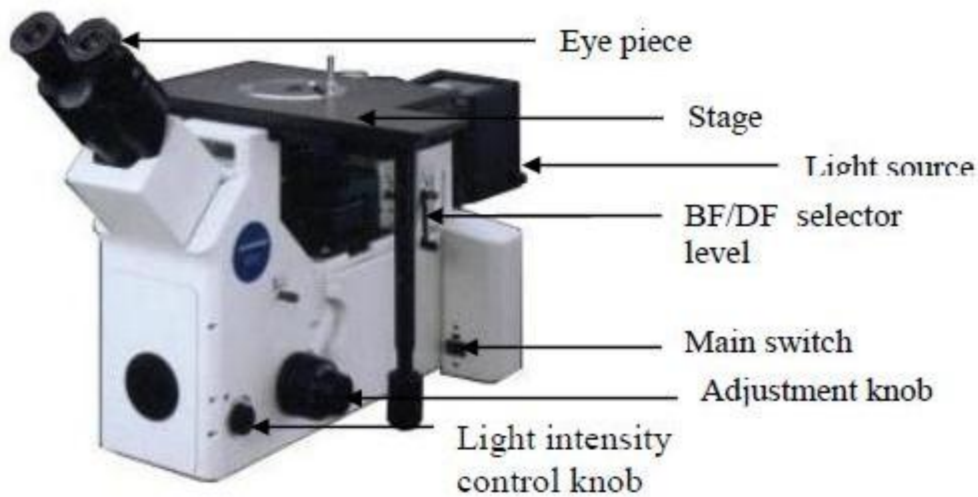


Figure 1: Image Analyzer

Operation procedure:

1. Place the specimen on the stage in such a way that the surface which is to be monitored faces down.
2. Select the required magnification powered nose piece by revolving it. This is located beneath the stage.
3. Adjust the position of stage by X-axis and Y-axis knobs, so that observer may view required portion.
4. Then set the coarse or fine adjustments for the better image quality.
5. The default setting is the Bright field, for dark field disengage the polarizer and analyzer.
6. Then select the lever to the DF option.
7. In case of DIC (Differential interface contrast), re-engage the polarizer and analyzer and insert the DIC slider.
8. The contrast can be varied by turning the prism movement knob on DIC slider.
9. If all the steps above are correct then the observer is ready to capture the snaps and analyze the microstructure
10. The image can directly by view by the eye piece or through the computer aided software on the monitor

The final analysis can be done through different steps in the software.

One of the most serious problems in microscopy is the poor contrast produced when light is passed through very thin specimens or reflected from surfaces with a high degree of reflectivity. To circumvent this lack of contrast, various optical "tricks" have been perfected by scientists to increase contrast and to provide color variations in specimens. The assortment of techniques in the microscopists bag include: polarized light, phase contrast imaging, differential interference contrast, fluorescence illumination, darkfield illumination, Rheinberg illumination, Hoffman modulation contrast, and the use of various gelatin optical filters.

Contrast in optical microscopy:

When imaging specimens in the optical microscope, differences in intensity and/or color create image contrast, which allows individual features and details of the specimen to become visible. Table 1 presents a summary of the contrast enhancing technique(s) of choice for a variety of specimens and materials that are studied with both transmitted and reflected light microscopy. This table may be used as a rough guide to approach specific imaging problems in optical microscopy.

Contrast-Enhancing Techniques

Specimen Type	Imaging Technique
Specular (Reflecting) Surface Thin Films, Mirrors Polished Metallurgical Samples Integrated Circuits **Diffuse (Non-Reflecting) Surface** Thin and Thick Films Rocks and Minerals Hairs, Fibers, and Bone Insects	Brightfield Illumination Phase Contrast, DIC Darkfield Illumination
Amplitude Surface Features Dyed Fibers Diffuse Metallic Specimens Composite Materials Polymers	Brightfield Illumination Darkfield Illumination
Birefringent Specimens Mineral Thin Sections Hairs and Fibers Bones and Feathers Single Crystals Oriented Films	Polarized Illumination
Fluorescent Specimens Mounted Cells Fluorochrome-Stained Sections Smears and Spreads	Fluorescence Illumination

Differential interference contrast (DIC):

An excellent mechanism for rendering contrast in transparent specimens, differential interference co sheared by a minuscule amount, generally somewhat less than the diameter of an Airy disk. The technique produces a monochromatic shadow-cast image that effectively displays the gradient of optical paths for both high and low spatial frequencies present in the specimen. Those regions of the specimen where the optical paths increase along a reference direction appear brighter (or darker), while regions where the path differences decrease appear in reverse contrast. As the gradient of optical path difference grows steeper, image contrast is dramatically increased. Among the chief imaging advantages of differential interference contrast microscopy is that, unlike darkfield or phase contrast, the image of smaller specimen features is not obscured by adjoining regions having large optical gradients.

Polarized illumination:

The polarized light microscope is designed to observe and photograph specimens that are visible primarily due to their optically anisotropic character. In order to accomplish this task, the microscope must be equipped with both a polarizer, positioned in the light path somewhere before the specimen, and an analyzer (a second polarizer), placed in the optical pathway between the objective rear aperture and the observation tubes or camera port. Image contrast arises from the interaction of plane-polarized light with a birefringent (or doubly-refracting) specimen to produce two individual wave components that are each polarized in mutually perpendicular planes. The velocities of these components are different and vary with the propagation direction through the specimen. After exiting the specimen, the light components become out of phase, but are recombined with constructive and destructive interference when they pass through the analyzer.

Darkfield illumination:

Dark field microscopy (dark ground microscopy) describes microscopy methods, in both light and electron microscopy, which exclude the unscattered beam from the image. As a result, the field around the specimen (i.e. where there is no specimen to scatter the beam) is generally dark. In optical microscopy, darkfield describes an illumination technique used to enhance the contrast in unstained samples. It works by illuminating the sample with light that will not be collected by the objective lens, and thus will not form part of the image. This produces the classic appearance of a dark, almost black, background with bright objects on it.

Advantages & disadvantages:

Dark field microscopy is a very simple yet effective technique and well suited for uses involving live and unstained biological samples, such as a smear from a tissue culture or individual water-borne single-celled organisms. Considering the simplicity of the setup, the quality of images obtained from this technique is impressive.

The main limitation of dark field microscopy is the low light levels seen in the final image. This means the sample must be very strongly illuminated, which can cause damage to the sample. Dark field microscopy techniques are almost entirely free of artifacts, due to the nature of the process. However the interpretation of dark field images must be done with great care as common dark features of bright field microscopy images may be invisible, and vice versa.

While the dark field image may first appear to be a negative of the bright field image, different effects are visible in each. In bright field microscopy, features are visible where either a shadow is cast on the surface by the incident light, or a part of the surface is less reflective, possibly by the presence of pits or scratches. Raised features that are too smooth to cast shadows will not appear in bright field images, but the light that reflects off the sides of the feature will be visible in the dark field images.

Brightfield illumination:

Bright field microscopy is the simplest of all the optical microscopy illumination techniques.Sample illumination is transmitted (i.e., illuminated from below and observed from above) white light and contrast in the sample is caused by absorbance of some of the transmitted light in dense areas of the sample. Bright field microscopy is the simplest of a range of techniques used for illumination of samples in light microscopes and its simplicity makes it a popular technique. The typical appearance of a bright field microscopy image is a dark sample on a bright background.

The light path of a bright field microscope is extremely simple; no additional components are required beyond the normal light microscope setup. The light path therefore consists of:

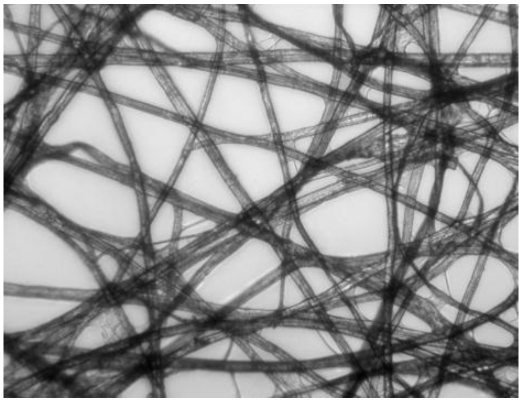
1. Transillumination light source, commonly a halogen lamp in the microscope stand.
2. Condenser lens which focuses light from the light source onto the sample.
3. Objective lens which collects light from the sample and magnifies the image.
4. Oculars and/or a camera to view the sample image.

Bright field microscopy typically has low contrast with most biological samples as few absorb light to a great extent. Stains are often required to increase contrast which prevents use on live cells in many situations. Bright field illumination is useful for samples which have an intrinsic color, for example chloroplasts in plant cells.
Bright field microscopy is a standard light microscopy technique and therefore magnification **is** limited by the resolving power possible with the wavelength of visible light.

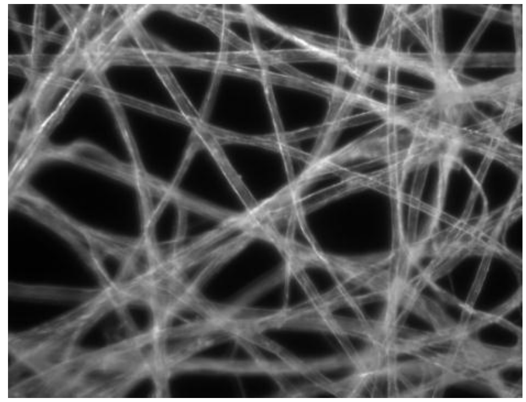
Advantages & disadvantages:

- Simplicity of setup with only basic equipment required.
- Very low contrast of most biological samples.
- Low apparent optical resolution due to the blur of out-of-focus material.

- The sample has to be stained before viewing. Therefore, live cells cannot be viewed.



Bright field illumination



Dark field illumination

Questions

1. Define the following terms:

   Magnification ____

   ____

   ____

   ____

   Numerical Aperture ____

   ____

   ____

   ____

   Resolution ____

   ____

   ____

   Parfocal ____

   ____

   ____

2. What is the use of immersion oil? How it can increase the magnification?

   ____

   ____

   ____

3. What is the function of the following parts of the microscope:

   ✓ Iris Diaphragm ____

   ✓ Condenser ____

   ✓ Coarse adjustment knob ____

   ✓ Ocular lens ____

Experiment No. 4

Aim of the Experiment

Operate a stereo microscope **under supervision** to measure the surface features of different samples.

Theory:

The stereo microscope uses two separate optical paths with two objectives and two eyepieces to provide slightly different viewing angles to the left and right eyes. In this way it produces a three-dimensional visualization of the sample being examined. The stereo microscope is often used to study the Fracture surfaces of solid specimens and to carry out close inspection of the failed part.

Stereoscopes with specially-equipped illuminators can be used for dark field microscopy, using either reflected or transmitted light. Great working distance and depth of field here are important qualities for this type of microscope.

Both qualities are inversely correlated with resolution: the higher the resolution (i.e. the shorter the distance at which two adjacent points can be distinguished as separate), the smaller the depth of field and working distance. A stereo microscope has a useful magnification up to 50×.

Figure 1: Stereo Microscope

Observations:

Observe the given surface at various magnifications and give your remarks, also attached relevant pictures on the given space which was taken from Stereo microscope:

Comments:

____

____

____

____

____

____

____

Stereo Images to be paste here

Results:

____

____

____

Questions

1. Stereomicroscopes have………………… ocular lens.
2. Stereomicroscope produces ………... dimensional images.
3. Stereo microscopes use ……………………………. light from the object being studied, compared to the ………………………………… light that is used by compound light microscopes.
4. What is the role of stereomicroscope for fractography?

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Experiment No. 5

Aim of the Experiment:
Operate X-ray fluorescence spectrometer **under supervision** to determine the chemical composition.

Apparatus:
Portable XRF, Standard Calibration Block, XRF Stand
Test Sample: Prepared test sample of student ‘s choice.

Theory:
X-ray fluorescence (XRF) spectrometry is an elemental analysis technique with broad Application in science and industry. XRF Spectrometry is used to identify elements in a substance and quantify the amount of those elements present. The XRF is widely used for |elemental analysis and |chemical analysis, particularly in the investigation of metals, glass, ceramics, polymer, composite, food, rocks minerals, building materials, and forensic science.

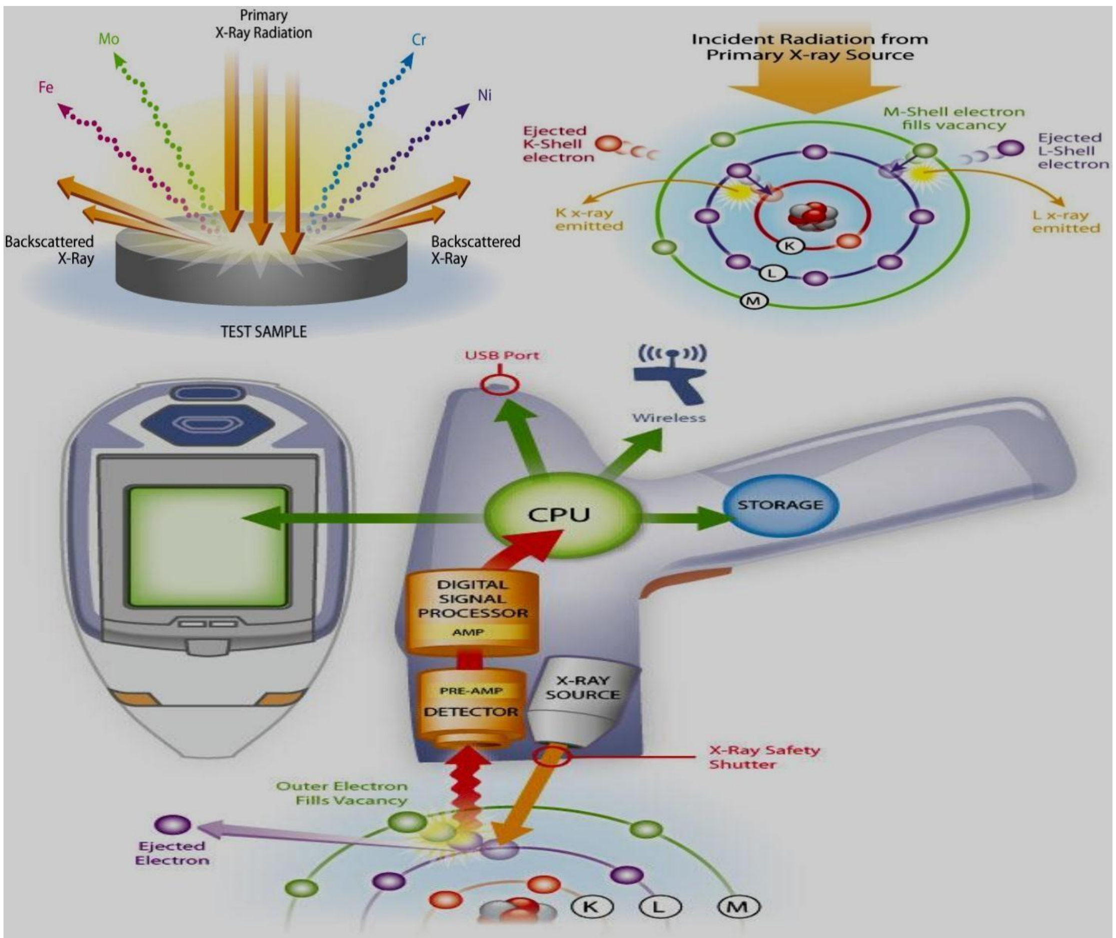

Figure 1: Working Mechanism of X-ray fluorescence (XRF) spectrometer.

Fundamentals of x-ray fluorescence spectroscopy:

XRF is based on the principle that individual atoms, when excited by an external energy source, emit X-ray photons of a characteristic energy or wavelength. By counting the number of photons of each energy emitted from a sample, the elements present may be identified and quantified. The X-ray fluorescence principle is depicted in |Figure 1. An inner shell electron is excited by an incident photon in the X-ray region. During the de-excitation process, an electron is moving from a higher energy level to fill the vacancy. The energy difference between the two shells appears as an X-ray, emitted by the atom. The X-ray spectrum acquired during the above process reveals a number of characteristic peaks. The energy of the peaks leads to the identification of the elements present in the sample (qualitative analysis), while the peak intensity provides the relevant or absolute elemental concentration (semi-quantitative or quantitative analysis).

A typical XRF spectroscopy arrangement (|Figure 1) includes a source of primary radiation (Usually a radioisotope, an electron beam or an X-ray tube) and an equipment for detecting

The secondary X-rays:

The XRF method is widely used to measure the elemental composition of materials. Since this method is fast and non-destructive to the sample, it is the method of choice for field applications and industrial production for control of materials. Depending on the application, XRF can be produced by using not only x-rays but also other primary excitation sources like alpha particles, protons or high energy electron beams.

XRF parts:

MMD EDXRF consists of a battery operated miniature X-ray tube, a high-resolution silicon pin detector for measuremnt of characteristic x-ray energy, and a IPAQ handheld computer for calculations, results and operator interface as shown in Figure.

Figure 1: Portable XRF gun

Procedure:

1. Place a battery in the analyzer.
2. Power on the Analyzer Press the ON/OFF button on the back of the analyzer.
3. Power on the iPAQ (Button located in upper right hand corner of iPAQ)
4. Select Innov-X from the start menu located in the upper Left hand comer of iPAQ screen.
5. Read the radiation safety notice and acknowledge that you are a certified user by pressing Start.
6. Select the desired analysis mode {i.e., Analytical Alloy, Analytical Vacuum (for light elements Si, Al, Mg).
7. The instrument will undergo a one minute hardware initialization period.
8. Standardize the instrument with the 316 Stainless Steel mask standard. Standardize the instrument every 4 hours or as directed by the display.
9. Release the software trigger lock and analyze a sample of known composition, in order to verify the correct operation of the analyzer.
10. When standardization is complete, remove the standardization clip.
11. Analyze samples of unknown composition.

Observations:

Sample No.	Reading No.	Elements (wt.%)									

Results:

____

____

____

Questions

1. If the absorption of electromagnetic radiation by matter results in the emission of the radiations of the same or longer wavelength for a long or a short time, the phenomena is termed as ____________________________________

2. The measurement of intensity of fluorescent X-rays provides a simple and ____________ way of _____________ analysis.

3. The energy of the emitted X-rays depends upon the _________ of the atom and their intensity depends upon the __________

4. Which of the following is Mosely's equation if 'C' is the speed of light, 'a' is a proportionality constant, 'σ' is a constant which depends on electronic transition series, 'Z' is the atomic number and 'λ' is the wavelength?
   a) $C\lambda = a(Z-\sigma)^2$
   b) $C/\lambda = a(Z-\sigma)^2$
   c) $C(Z-\sigma)^2 = a\lambda$
   d) $C(Z-\sigma)^2 = a/\lambda$

5. In X-ray fluorescence spectrometer, the relationship between the excitation intensity and the intensity of fluorescence does not depend on
   ____
   a)Spectrum of the incident radiation
   b) Angle of radiance
   c) Molecular weight
   d) Incident angle

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NED University of Engineering & Technology
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Course Code and Title: ____

Psychomotor Domain Assessment Rubric-Level P3					
Skill Sets	Extent of Achievement				
	0	1	2	3	4
Equipment Identification Sensory skill to ***identify*** equipment and/or its component for a lab work.	Not able to identify the equipment.	--	--	--	Able to identify equipment as well as its components.
Equipment Use Sensory skills to ***demonstrate*** the use of the equipment for the lab work.	Doesn't demonstrate the use of equipment.	Slightly demonstrates the use of equipment.	Somewhat demonstrates the use of equipment.	Moderately demon strates the use of equipment.	Fully demonstrates the use of equipment.
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Laboratory Session No.______________ Date: ______________________

Weighted CLO (Psychomotor Score)	
Remarks	
Instructor's Signature with Date:	

Experiment No. 6

Aim of the Experiment:

Operate X-ray Powder Diffractometer **under supervision** to characterized different phases.

Theory:

The X-ray diffractometer is the primary instrument used for the measurement of the intensities of diffracted X-rays. The purpose of today's laboratory is to help you establish a basic familiarity with the operation of the diffractometer.

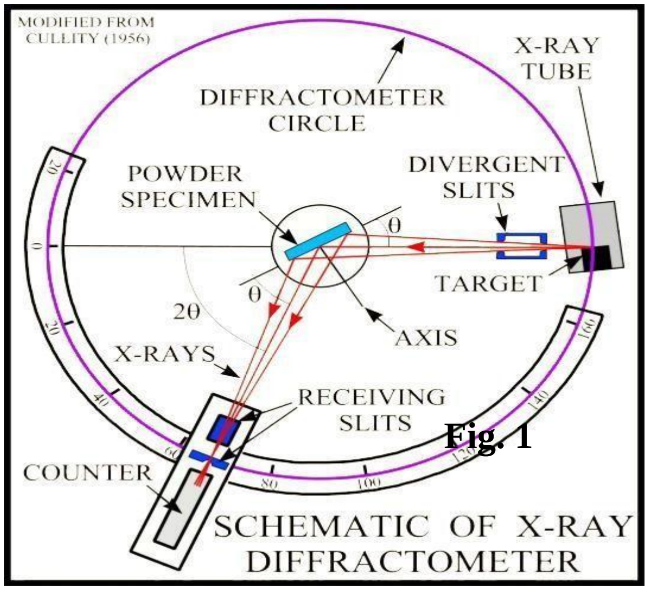

Figure 1: X-Ray Diffractometer

Construction:

The mechanical aspects of the instrument are shown in Figure 1a while parts are labelled in Figure 1. A specimen C in the form of a flat plate is supported on a table *H*, which can be rotated about an axis 0 perpendicular to the plane of the drawing. The X-ray source is S, the line focal spot of the target *T* of the X-ray tube; S is also normal to the plane of the drawing and therefore parallel to the diffractometer axis O. X-rays diverge from this source and are diffracted by the specimen to form a convergent diffracted beam which comes to a focus at the slit *F* and then enters the counter G. *A* and *B* are slits that define and collimate the incident and diffractedbeams. The receiving slits and counter are supported on the carriage *E*, which may be rotated about the axis 0 and whose angular position 2θ may be read on the graduated scale *K*. The supports *E* and *H* are mechanically coupled so that a rotation of the counter through *2x* degrees is automatically accompanied by rotation of the specimen through *x* degrees. This coupling ensures that the angles of incidence on, and reflection from, the flat specimen will always be equal to(me another and equal to half the total angle of diffraction. The counter may be driven by a stepper motor in fixed increments about the diffractometer axis or slewed to any desired angular position.

X-RAY detector:

Mounted on the diffractometer is a scintillation counter, which consists of an inorganic crystal that gives off light (scintillates) when struck by an X-ray photon, and a photomultiplier tube that converts the light pulses into electrical pulses. The pulses sent to a rate meter, which indicatesthe average count rate by smoothing out the succession of pulses and converting it into a steady current. These current pulses given to the electronic circuitry and computer for processing.

The diffractometer used for this lab is also equipped with a diffracted beam monochromator to eliminate interference from fluorescent radiation from the sample. The combination of a PHA and a diffracted beam monochromator is very effective at lowering the background intensity level, thus greatly improving the quality of the diffraction data. The diffractometer is fully computer automated, thus simplifying data collection in that the completed scan can be output asa simple ASCII file suitable for later analyses.

Diffractometer parts:

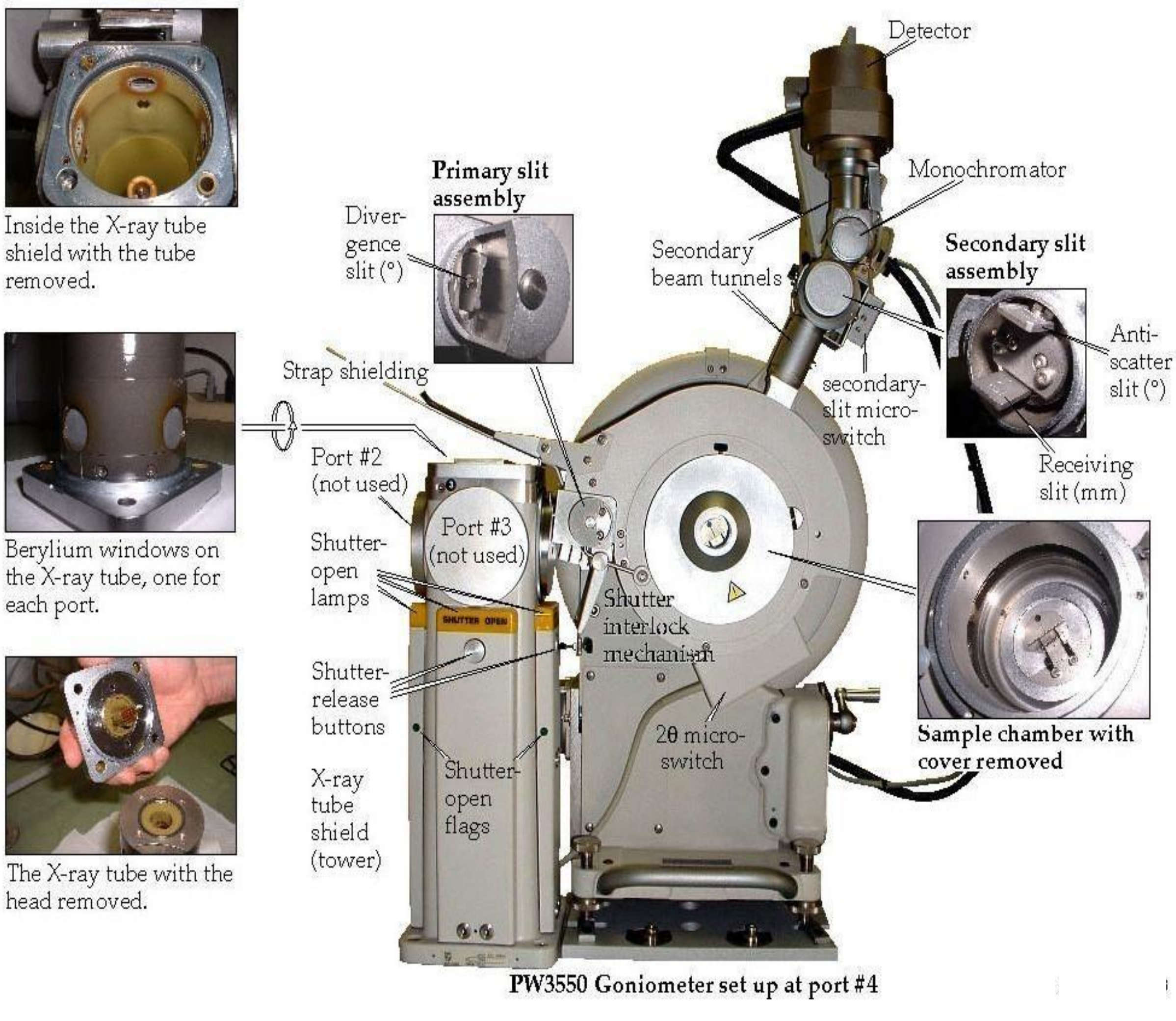

Figure 2: Diffractometer parts

Questions

1. What is the principle behind X-ray crystallography?

2. Why do we observe peaks of different heights in the XRD pattern?

3. How can you determine the crystal plane corresponding to a particular peak in the XRD pattern?

4. Why are peaks corresponding to some planes missing or not observed in the XRD pattern?

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Laboratory Session No._____________ Date: _____________________

Weighted CLO (Psychomotor Score)	
Remarks	
Instructor's Signature with Date:	

Experiment No. 7

Aim of the Experiment:

Manipulate XRD scan to determine the lattice parameter and identify the Bravais lattice using analytical technique.

Peak indexing

Consider the following XRD pattern for Aluminum, which was collected using CuKα radiation. Index this pattern and determine the lattice parameters.

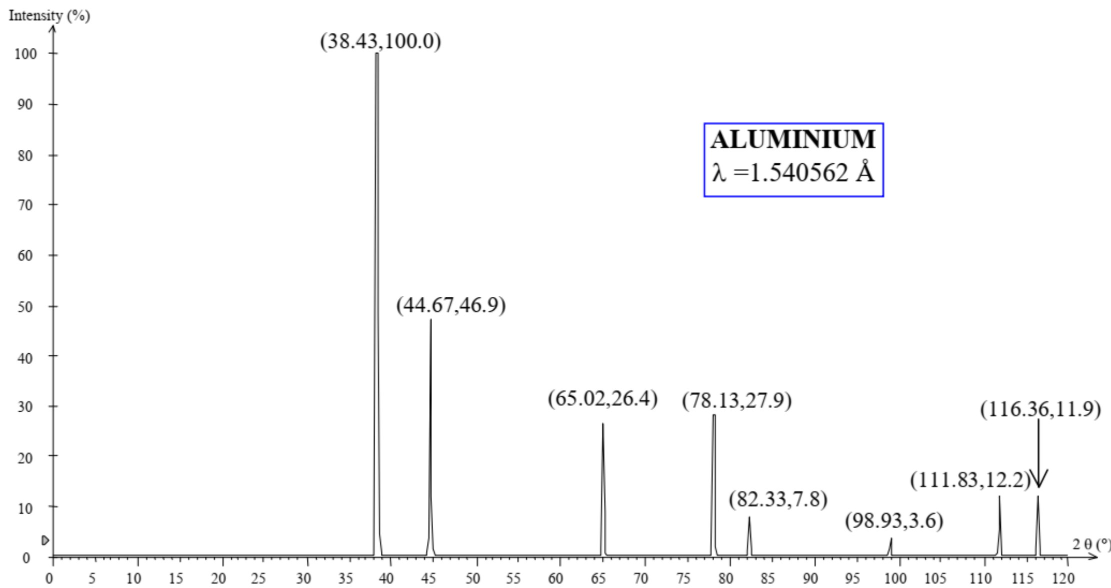

Figure 1: XRD pattern with some characteristic peaks

Steps:

(1) Identify the peaks and determine sin 2θ.
(2) Calculate the ratio $\sin^2\theta/\sin^2\theta_{min}$ and multiply by the appropriate integers.
(3) Select the result from (2) that yields $h^2 + k^2 + l^2$ as an integer.
(4) Compare results with the sequences of h k l values to identify the Bravais lattice.
(5) Calculate lattice parameters.

Observation:

Peak No.	2θ	$\sin^2\theta$	$1\times\dfrac{\sin^2\theta}{\sin^2\theta_{min}}$	$2\times\dfrac{\sin^2\theta}{\sin^2\theta_{min}}$	$3\times\dfrac{\sin^2\theta}{\sin^2\theta_{min}}$	$h^2+k^2+l^2$	hkl	a (Å)
1	38.43							
2	44.67							
3	65.02							
4	78.13							
5	82.33							
6	98.93							
7	111.83							
8	116.36							

Calculations:

Results:

Questions

1. What are the relations between interplanar spacing and miller indices of the planes for different lattice types?

Cubic

____

____

____

____

Rhombohedral

____

____

____

____

Orthorhombic

____

____

____

____

Triclinic

____

____

____

____

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Course Code and Title: ____

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Laboratory Session No.______________ Date: ______________________

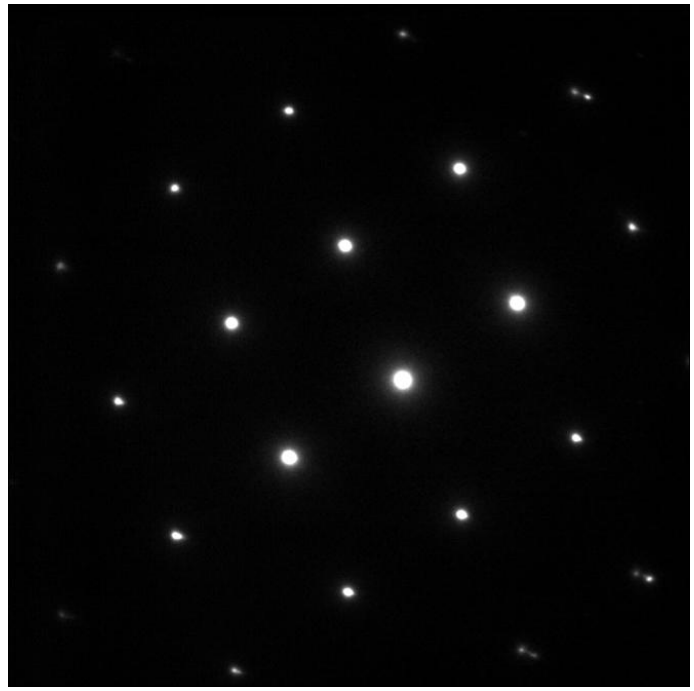
Weighted CLO (Psychomotor Score)	
Remarks	
Instructor's Signature with Date:	

Experiment No. 8

Aim of the Experiment:

Manipulate SAED pattern for single crystal material (Spot type) and index it using any freeware e.g. FIJI

Consider the following Selected Electron Diffraction (SAED) pattern for TiNiCu alloy, which was collected using diffraction constant of 440Å*Px. Index this pattern and determine the lattice parameters and its miller indices (i.e. hkl planes).



Theory:

In TEM analysis, the type of SAED pattern observed can provide insights into the nature of the material being studied. The appearance of spot-type or ring-type patterns depends on various factors, including the sample's crystal structure, thickness, and orientation.

Spot-Type SAED Pattern:

A spot-type SAED pattern is typically observed when analyzing a thin single crystal sample or a small region within a polycrystalline material. In this pattern, distinct spots are visible, representing diffraction from individual crystal planes. The spots are sharp and well-defined, indicating a high degree of crystallographic order. The presence of spot-type patterns suggests a more uniform crystal orientation and minimal grain boundaries within the analyzed area.

Ring-Type SAED Pattern:

A ring-type SAED pattern is commonly observed in polycrystalline materials or thick samples with multiple crystal orientations. In this pattern, diffraction rings or arcs are observed instead of discrete spots. The rings result from the overlapping of diffraction spots from various crystal planes and orientations present in the material. The ring-type pattern indicates a mixture of crystal orientations and the presence of grain boundaries. The size, shape, and intensity distribution of the rings provide information about the texture, grain size, and preferred orientation within the material.

It's important to note that the interpretation of SAED patterns requires careful analysis, and the presence of spot-type or ring-type patterns alone does not provide a complete understanding of the material. Additional characterization techniques and analysis are often required to determine the crystal structure, grain boundaries, and other material properties accurately.

Derivation of $r_{hkl}\, d_{hkl} = \lambda L$

From the diagram,

$r/L = \tan 2\theta$

In electron diffraction, the angle θ is small so that we can make the following approximations:

$\tan 2\theta \approx 2\theta$ (with θ in radians),

$2\theta = r/L$ ➔ $\theta = r/2L$

The Bragg condition (which is true for the sets of variables that produce diffraction spots) states that:

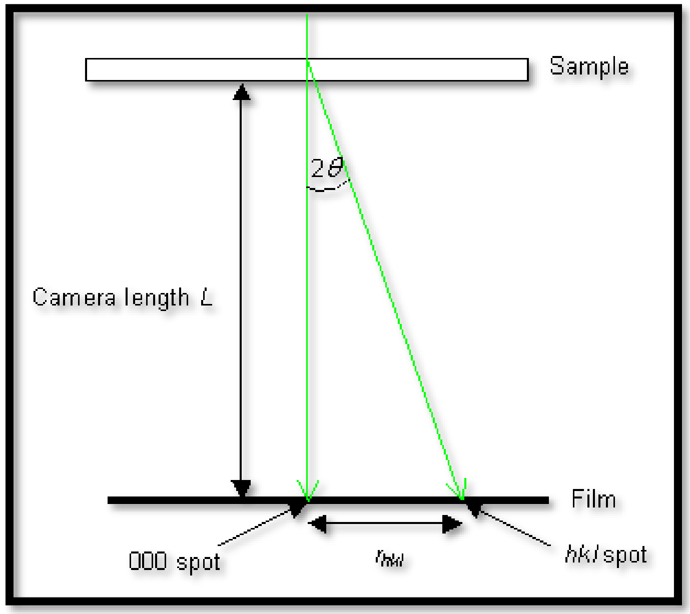
$\lambda = 2\, d_{hkl} \sin\theta$ ➔ $\lambda = 2\, d_{hkl}\, \theta$

$\lambda = 2\, d_{hkl}\, (r/2L)$ ➔ $\lambda L = r_{hkl}\, d_{hkl}$

So, $d_{hkl} \propto 1/\, r_{hkl}$

In other words

$r_1/r_2 = d_2/d_1$ ➔ $d = a / \sqrt{h^2 + k^2 + l^2}$



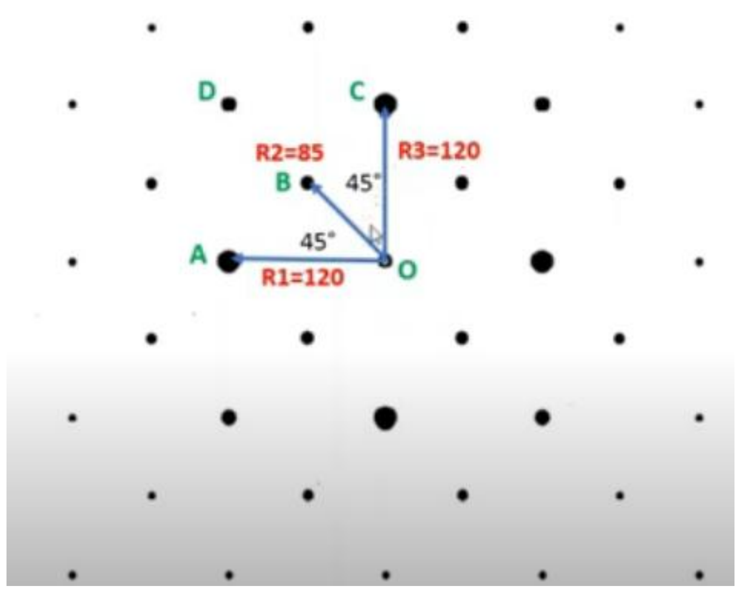
$\mathbf{r_1/r_2 = \sqrt{h_1^2 + k_1^2 + l_1^2} / \sqrt{h_2^2 + k_2^2 + l_2^2}}$ ➔ **Eqn (1)**

The angle between two lines can be determine from the following; equation:

$\mathbf{\cos\theta = (h_1 h_2 + k_1 k_2 + l_1 l_2) / (\sqrt{h_1^2 + k_1^2 + l_1^2} * \sqrt{h_2^2 + k_2^2 + l_2^2})}$ ➔ **Eqn (2)**

STEPS:

1. Locate and identify the central bright diffraction spots in the SAED pattern.
2. Select several well-defined and distinct spots for indexing.
3. Measure r_1, r_2 and r_3 and angles between them as mentioned in Fig using any freeware.
4. Calculate their ratios i.e. r_1/r_2, r_3/r_2, and r_3/r_1.
5. Now determine their ratios as per eqn (1) by putting standard hkl values from Table 2 to get a similar ratio as calculated in step 4 (by hit and trial method).



6. To validate the results of step 5. Determine the angle between two lines i.e. r_1 , r_2 or r_3 from eqn (2), this should match the angle determined from the given SAED pattern (step 3).

Table-1. Conditions of forbidden and allowed reflections (h k l) of common crystal structures (F: the Structure Factor).

Bravais Lattice	Forbidden reflections	Allowed reflections	F	Number of lattice points per cell	Example Compounds
Primitive Cubic	None	Any h, k, l	f	1	α-Po
fcc	h, k, l are mixed odd and even	h, k, l are all odd or all even	4f	4	fcc metals, GaAs, NaCl-rocksalt, ZnS-zincblende
bcc	h + k + l is odd	h + k + l is even	2f	2	bcc metals
fcc	h, k, l are mixed odd and even; or, all even and h + k + l ≠ 4n	As fcc, but if all even and h + k + l ≠ 4n, then absent (n is integer)			Si, Ge, Sn - diamond cubic
Base centered		h, k and l all odd or all even	2f	2	
	h + k + l is odd				bct
Primitive Hexagonal	h + 2k = 3m and l is odd	All other cases			Hexagonal closed packed (hcp) metals
Hexagonal close-packed (hcp)				Reflection example	
		h + 2k = 3n with l odd	0	0001	
		h + 2k = 3n with l even	2f	0002	
		h + 2k = 3n ± 1 with l odd	f3	01-11	
		h + 2k = 3n ± 1 with l even	f	01-10	

Table-2. Allowed list of ($h^2 + k^2 + l^2$) for cubic crystal structures

Forbidden numbers	Primitive, P	Face Centered, F	Body Centered, I	Corresponding *hkl*
	1			100
	2		2	110
	3	3		111
	4	4	4	200
	5			210
	6		6	211
7				
	8	8	8	220
	9			221, 300
	10		10	310
	11	11		311
	12	12	12	222
	13			320
	14		14	321
15				
	16	16	16	400

Table-3. Relative reciprocal lattice Spacing for Face Cubic and Diamond Cubic lattices

Relative Reciprocal Lattice Spacings for Face-Centered and Diamond Cubic Lattices[a]

	111	200	220	311	331	420	422	511	531
111	1•								
200	1.155	1							
220	1.63•	1.41	1•						
311	1.92•	1.66	1.17•	1•					
222	2.00	1.73	1.225	1.045					
400	2.31•	2.00	1.415•	1.21•					
331	2.52•	2.18	1.54•	1.31•	1•				
420	2.58	2.235	1.58	1.35	1.027	1			
422	2.85•	2.45	1.73•	1.48•	1.124•	1.096	1•		
333,511	3.00•	2.60	1.84•	1.57•	1.19•	1.16	1.06•	1•	
440	3.27•	2.83	2.00•	1.71•	1.30•	1.217	1.156•	1.09•	
531	3.42•	2.96	2.09•	1.785•	1.36•	1.32	1.21•	1.14•	1•
442	3.46	3.00	2.12	1.81	1.38	1.34	1.225	1.157	1.014
620	3.66•	3.16	2.24•	1.91•	1.45•	1.42	1.29•	1.22•	1.07•
533	3.79•	3.28	2.32•	1.98•	1.503•	1.47	1.34•	1.26•	1.11•
622	3.82	3.32	2.34	2.00	1.52	1.48	1.355	1.28	1.12
444	4.00•	3.47	2.45•	2.09•	1.59•	1.55	1.415•	1.33•	1.17•
711,551	4.12•	3.57	2.52•	2.15•	1.64•	1.595	1.458•	1.374•	1.207•

[a]Asterisks indicate spacings for diamond cubic lattice.

Table-4. Interplaner angle (in degrees) in Cubic crystals between planes of the form $h_1k_1l_1$ and $h_2\,k_2\,l_2$

	$\{h_1k_1l_1\}$						
$\{h_2k_2l_2\}$	100	110	111	210	211	221	310
100	0 90						
110	45 90	0 60 90					
111	54.7	35.3 90	0 70.5 109.5				
210	26.6 63.4 90	18.4 50.8 71.6	39.2 75.0	0 36.9 53.1			
211	35.3 65.9	30 54.7 73.2 90	19.5 61.9 90	24.1 43.1 56.8	0 33.6 48.2		

Observation:
Observations from Freeware of spot type SAED pattern (mention proper units)

S No.	r_1	r_2	r_3	θ_1	θ_2	r_1/r_2	r_3/r_2	r_3/r_1
1.								
2.								
3.								

Calculations:

Results:

Paste indexed SAED pattern here

Bonus Marks:

Miller indices of the Zone axis is ____

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Laboratory Session No.______________ Date: _______________________

Weighted CLO (Psychomotor Score)	
Remarks	
Instructor's Signature with Date:	

Experiment No. 9

Aim of the Experiment:
Manipulate SAED pattern for polycrystalline material (ring type) and index it using any freeware e.g. FIJI

Consider the following Selected Electron Diffraction (SAED) pattern for Aluminum alloy, which was collected using a diffraction constant of 440Å*Px. Index this pattern and determine the lattice parameters.

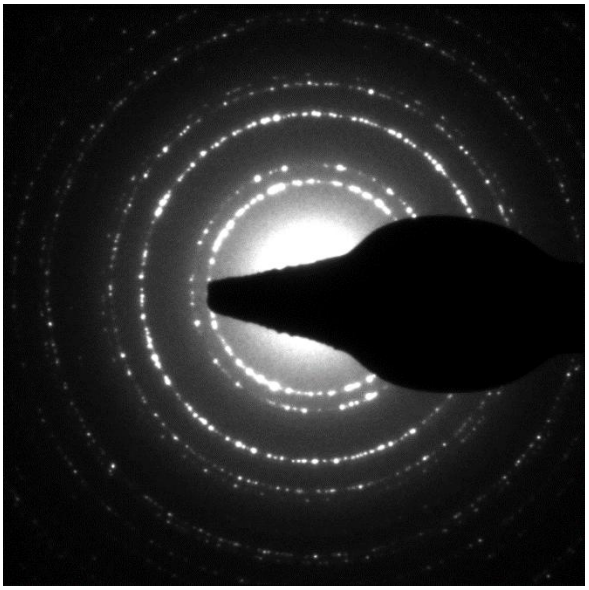

Figure 1: Ring type SAED pattern

Theory:

Refer to Practical 8

Steps:

1. Locate and identify the central bright diffraction ring in the SAED pattern.
2. Select several well-defined and distinct rings for indexing.
3. Measure radii of the rings i.e. r_1, r_2, r_3…… r_n using any freeware.
4. Calculate d-spacing by multiplying r values if it's in nm unit.
5. In order to obtain hkl values a standard JCPDS card is required.

Table-1 Miller indices of Diffracting planes, and allowed and forbidden reflections

{hkl}	$\Sigma[h^2 + k^2 + l^2]$	FCC	Diamond cubic	BCC
{100}	1	-	-	-
{110}	2	-	-	110
{111}	3	111	111	-
{200}	4	200	200	200
{210}	5	-	-	-
{211}	6	-	-	211
	7	-	-	-
{220}	8	220	220	220
	9	-	-	-
{310}	10	-	-	310
{311}	11	311	331	-
{222}	12	222	-	222
	13	-	-	-
{321}	14	-	-	321
	15	-	-	-
{400}	16	400	400	400
	17	-	-	-
{411}	18	-	-	411
{330}	18	-	-	330
{331}	19	331	331	-
{420}	20	420	-	420
	21	-	-	-
{332}	22	-	-	332
	23	-	-	-
{422}	24	422	422	422
	25	-	-	-
{431}	26	-	-	431
{511}	27	511	511	-
{333}	27	333	333	-
	28	-	-	-
	29	-	-	-
{521}	30	-	-	521
	31	-	-	-
{440}	32	440	440	440

Observation:

S No.	1/2r (nm)	1/r (nm)	r (nm)	d-spacing (A°)	(hkl)

Calculations:

Results:

Paste indexed SAED pattern here

Questions

1. Diffused continuous and thick rings indicate ______________________________materials, whereas bright dotted rings indicate ___________________________materials and randomly dotted rings (almost unclear and overlapped rings) indicate ____________________ materials.

2. The resolving power of TEM is derived from ____.
3. The cathode of transmission electron microscope consists of a ____________________________.
4. During TEM, a vacuum is created inside the ____.
5. Describe the ZAF correction in detail, and discuss how the correction is carried out.

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NED University of Engineering & Technology
Department of____________________Engineering
Course Code and Title: ____

Psychomotor Domain Assessment Rubric-Level P3					
Skill Sets	Extent of Achievement				
	0	1	2	3	4
Equipment Identification Sensory skill to ***identify*** equipment and/or its component for a lab work.	Not able to identify the equipment.	--	--	--	Able to identify equipment as well as its components.
Equipment Use Sensory skills to ***demonstrate*** the use of the equipment for the lab work.	Doesn't demonstrate the use of equipment.	Slightly demonstrates the use of equipment.	Somewhat demonstrates the use of equipment.	Moderately demon strates the use of equipment.	Fully demonstrates the use of equipment.
Procedural Skills ***Displays*** skills to act upon sequence of steps in lab work.	Not able to either learn or perform lab work procedure.	Able to slightly understand lab work procedure and perform lab work.	Able to somewhat understand lab work procedure and perform lab work.	Able to moderately understand lab work procedure and perform lab work.	Able to fully understand lab work procedure and perform lab work.
Response Ability to ***imitate*** the lab work on his/her own.	Not able to imitate the lab work.	Able to slightly imitate the lab work.	Able to somewhat imitate the lab work.	Able to moderately imitate the lab work.	Able to fully imitate the lab work.
Observation's Use ***Displays*** skills to use the observations from lab work for experimental verifications and illustrations.	Not able to use the observations from lab work for experimental verifications and illustrations.	Slightly able to use the observations from lab work for experimental verifications and illustrations.	Somewhat able to use the observations from lab work for experimental verifications and illustrations.	Moderately able to use the observations from lab work for experimental verifications and illustrations.	Fully able to use the observations from lab work for experimental verifications and illustrations.
Safety Adherence Adherence to ***safety*** procedures.	Doesn't adhere to safety procedures.	Slightly adheres to safety procedures.	Somewhat adheres to safety procedures.	Moderately adheres to safety procedures.	Fully adheres to safety procedures.
Equipment Handling ***Equipment care*** during the use.	Doesn't handle equipment with required care.	Rarely handles equipment with required care.	Occasionally handles equipment with required care.	Often handles equipment with required care.	Handles equipment with required care.
Group Work ***Contributes*** in a group based lab work.	Doesn't participate and contribute.	Slightly participates and contributes.	Somewhat participates and contributes.	Moderately participates and contributes.	Fully participates and contributes.

Laboratory Session No.______________ Date: ______________________

Weighted CLO (Psychomotor Score)	
Remarks	
Instructor's Signature with Date:	

Experiment No. 10

Aim of the Experiment:

Manipulate CES software for the selection of materials for an engineering application.

Students are required to attach instruction sheet and rubric assessment sheet along with the report.