الجمـــهورية الجزائريـــة الديمقراطيـــة الشعبيـــة

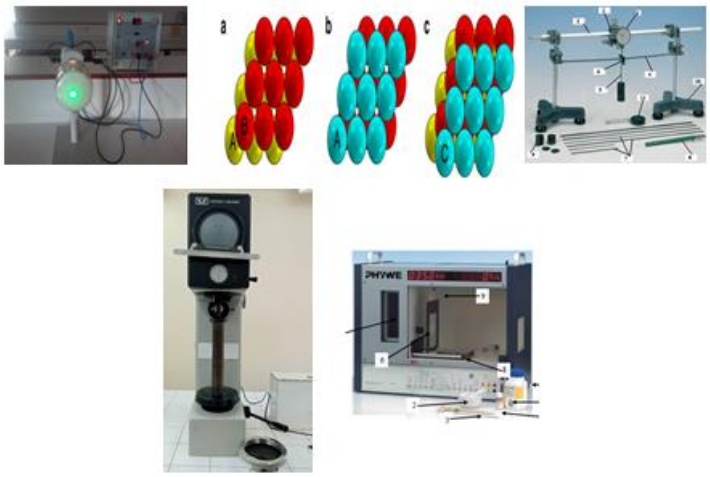
REPUBLIQUE ALGERIENNE DEMOCRATIQUE ET POPULAIRE

Ministère de l'Enseignement Supérieur et de la Recherche Scientifique
Université 8 mai 1945 Guelma
Faculté des Mathématiques et de l'Informatique et des Sciences de la Matière
Département des Sciences de la Matière

وزارة التعليم العالـي و البحث العلمي
جامعة 8 مـاي 1945 قالمـة
كليـة الرياضيات و الإعلام الآلي و علـوم المادة
قسم علـوم المادة

## POLYCOPY COURSE

# Laboratory work in Solid State Physics 1




**Designed for students of 3rd year LMD**

**Materials Physics**

**Prof. YAHI Hakima**

-2025-

# Preface

This practical work booklet is designed for third-year LMD students in Materials Physics**.** Its primary goal is to strengthen the theoretical knowledge acquired in lectures through hands-on experimental applications while developing essential skills in equipment handling and data interpretation in solid-state physics.

The experiments covered in this document focus on the following topics:

- **Atomic stacking:** Investigation of crystalline structures through the analysis of different atomic stacking arrangements.
- **X-ray diffraction:** Introduction to diffraction techniques for characterizing crystalline structures, with an emphasis on phase identification and lattice parameter determination.
- **Electron diffraction:** Study of electron diffraction phenomena using a polycrystalline graphite sample, demonstrating the wave nature of electrons.
- **Mechanical testing:** Measurement of elastic properties, including Young's modulus and Poisson’s ratio, to analyze material deformation under stress.
- **Microhardness:** Assessment of local mechanical properties through micro-indentation tests to evaluate materials' resistance to plastic deformation.

This practical program is designed to deepen students' understanding of the fundamental physical principles governing material properties while familiarizing them with essential experimental techniques. The emphasis is placed on measurement precision, rigorous data analysis, and clear result presentation.

To make the most of these practical sessions, students are expected to have a solid foundation in crystallography and a strong command of the necessary mathematical and physical tools.

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Ministry of Higher Education and Scientific Research

University 8 May 1945 Guelma
Faculty of Mathematics, and Computer Science, and Sciences of Matter
Department of Sciences of Matter

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# SYLLABUS

**Teaching Unit:** UEM
**Subject:** Laboratory work in Solid State Physics 1
**Field/Discipline/Program: SM/Physics/L3 - Materials Physics**
**Semester:** 5 **Academic year: 2024/2025**
**Credits:** 2**,** **Coefficient:** 1
**Total Weekly Hours:** 1h30min
**Course Instructor:** YAHI Hakima
**Rank:** Professor

**Objectives:**

This lab provides an educational framework for exploring the structural and mechanical properties of solid materials through practical experiments. It aims to bridge the gap between theory and practice while introducing students to modern tools and methods for material characterization.

**Program:**

- Stacking Structures
- X-ray Diffraction
- Electron Diffraction
- Mechanical Testing: Elastic (Young's Modulus, Poisson's Ratio, etc.)
- Microhardness

**Assessment:** Knowledge check & weightings

| Control | Weighting **(%)** |
|---|---|
| Final Exam | **50** |
| Report + Laboratory Work | **50** |
| Total | **100** |

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# Lab 0
# Least Squares Method for Error Calculation

# Lab 0: Least Squares Method for Error Calculation

## I. Introduction

In experimental sciences, we typically work with two sets of data, denoted {y1, y2, …, yn} and {x1, x2, …, xn}, obtained from measurements. The regression problem consists of determining a potential relationship between the x's and y's, often expressed as y = f(x).

When this relationship is linear, that is, of the form y = ax + b, it is referred to as linear regression. However, even if such a relationship exists, experimental data usually do not perfectly conform to this equation. This is due to the measurement errors inherent in the data.

To account for these uncertainties in the mathematical model, the measured values {y1, y2, …, yn} are often considered as realizations of a random variable Y, and sometimes {x1, x2, …, xn} are also considered as realizations of a random variable X. In this context, Y is called the dependent variable or explained variable, while X is referred to as the independent variable or explanatory variable.

## II. Least Squares Line

The data points (xi, yi), where i = 1, 2, …, n, can be represented as a cloud of n points, called a scatter plot, in the (x, y) plane. The centroid of this cloud can be easily calculated; it is the point with the following coordinates:

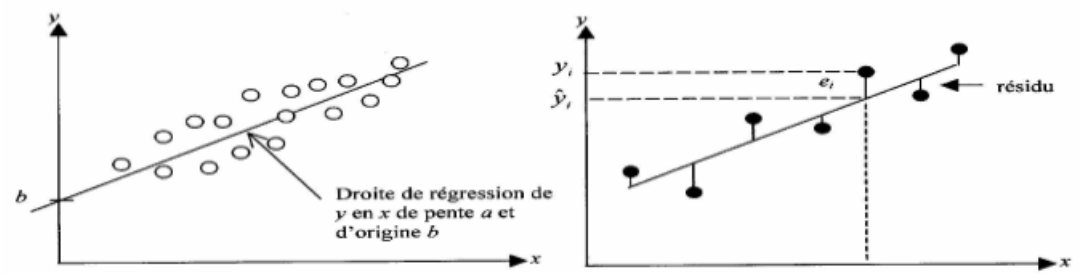
$$(\bar{x}, \bar{y}) = \left( \frac{\sum_{i=1}^{n} x_i}{n}, \frac{\sum_{i=1}^{n} y_i}{n} \right) \quad (1)$$

$\bar{x}$ *and* $\bar{y}$ represent the mean values of x and y, respectively.

Seeking an affine relationship between the variables X and Y involves finding a line that best fits the cloud of points. Among all possible lines, we choose the one with a remarkable property: it minimizes the sum of the squared differences between the observed values yi and the values predicted by the line $y_i = ax_i + b$. If εi represents this difference, also

called the residual, the principle of ordinary least squares (OLS) consists of choosing the values of a and b that minimize the following relation:

$$E = \sum_{i=0}^{n} \varepsilon_i^2 = \sum_{i=0}^{n} \big(y_i - (ax_i - b)\big)^2 \tag{2}$$




The values of a and b are determined from the following relations:

$$a = \frac{\sum_{i=1}^{n}(x_i - \bar{x})(y_i - \bar{y})}{\sum_{i=1}^{n}(x_i - \bar{x})^2} \tag{3}$$

$$b = \bar{y} - a\bar{x} \tag{4}$$

## III. Variance and Covariance

Variance and covariance are two measures used in statistics. Variance is a measure of the dispersion of data, while covariance indicates the degree to which two random variables change together. Variance is more of an intuitive concept, but covariance is mathematically defined in a way that may not seem intuitive at first glance. Often, the variance of X is used to express the value of a, $s_x^2$, and the covariance of the random variables X and Y, $cov_{xy}$ :

$$a = \frac{cov_{xy}}{s_x^2} \tag{5}$$

with:

$$s_x^2 = \frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n} \tag{6}$$

and:

$$cov_{xy} = \frac{\sum_{i=1}^{n}(x_i - \bar{x})(y_i - \bar{y})}{n} \tag{7}$$

## IV. Standard Deviation

In mathematics, the standard deviation is a positive real quantity, possibly infinite, used in probability theory to characterize the distribution of a random variable around its mean. The formula for the standard deviation is:

$$\sigma = \sqrt{\frac{\sum_{i=1}^{n} |x_i - \bar{x}|^2}{n}} \tag{8}$$

# Lab 1
# Stacking Structures

# Lab 1: Stacking Structures

## I. Principle

Study various crystalline structures on a large scale using both exploded and compact molecular models, where the atoms making up the crystal are represented as spherical balls.

## II. Objectives

- Build different types of compact and non-compact stacking's for several crystalline structures and examine their unit cells.
- Observe the various crystallographic sites in the face-centered cubic and hexagonal unit cells, and calculate the lattice parameters based on the crystallographic geometry.

## III. Theoretical Reminder

### III.1. Crystalline Lattice

A perfect crystal is a collection of particles (ions, atoms, or molecules) regularly arranged in space. This arrangement is described by:

- A crystalline lattice formed by a set of nodes,
- A basic motif, consisting of one or more atoms, that occupies the positions of these nodes.

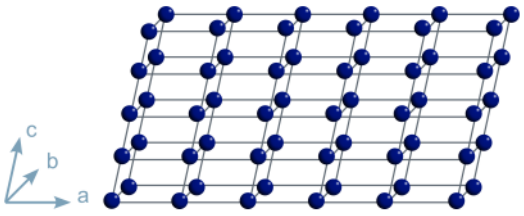



**Figure 1:** A three-dimensional crystalline lattice defined by the three primitive vectors $\vec{a}$, $\vec{b}$, and $\vec{c}$.

### III.2. Unit Cell

The unit cell is the parallelepiped defined by the three primitive vectors $\vec{a}$, $\vec{b}$, and $\vec{c}$, also known as the lattice parameters.

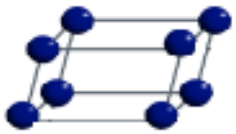


**Figure 2:** Unit cell.

### III.3. Reduced Coordinates

Since the crystalline lattice is periodic, the positions of the atoms within the unit cell are represented by the coordinates (x, y, z) such that: $0 \leq x < 1$, $0 \leq y < 1$, and $0 \leq z < 1$.

### III.4. Coordination

The coordination, or coordination number, of a given particle represents the number of nearest particles surrounding that particle.

### III.5. Multiplicity

Multiplicity **m** refers to the number of nodes belonging to the conventional unit cell.

### III.6. Crystallographic Sites

Crystallographic sites correspond to interstitial spaces between atoms. The most common are tetrahedral sites, surrounded by 4 atoms, and octahedral sites, surrounded by 6 atoms.

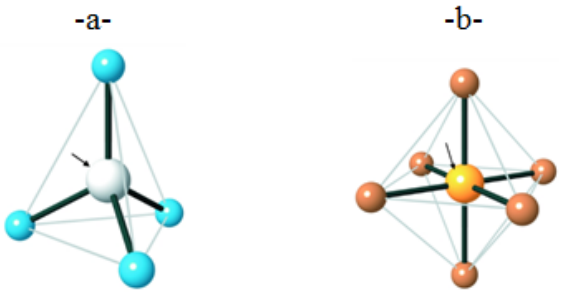



**Figure 3:** a) Tetrahedral site, and b) Octahedral site.

## II.7. Packing Density

Packing density represents the ratio of the volume actually occupied by the atoms of the motif to the total volume of the unit cell. If we assume the atoms to be spherical with radius **R**, the packing density **C** can be calculated using the following relation:

$$C = \frac{m.V_{motif}}{V_{cell}} \quad (1)$$

***m*** is the multiplicity of the unit cell.

If we assume the atoms to be spherical with radius **R**, the volume of an atom can be calculated using the following relation:

$$V = \frac{4}{3}\pi R^3 \quad (2)$$

## III.8. Density

The density **ρ** of the crystal is defined by:

$$\rho = \frac{m \times m_{motif}}{V_{cell}} \quad (3)$$

## III.9. Compact and Non-Compact Stacking's

Compact stacking refers to the arrangement of atomic spheres in space in such a way as to achieve the highest possible sphere density without any overlap between them.

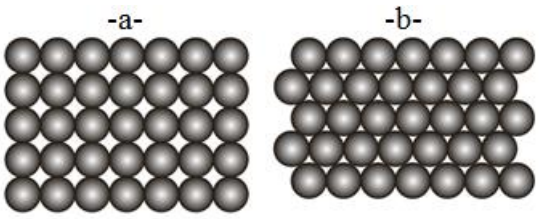



**Figure 4:** a) Non-Compact, and b) Compact Stacking's.

Compact stacking's of two types differ only in the way the tangent spheres are stacked on top of each other.

**a) Compact A-B-C stacking**

In this type of stacking, the spheres are arranged in a three-layer repeating pattern, where the third layer (C) fits into the gaps of the first layer (A), creating a repeating ABC structure.

**b) Compact A-B stacking**

In this stacking, the spheres are arranged in a two-layer repeating pattern, where the second layer (B) fits into the gaps of the first layer (A), forming an AB structure.

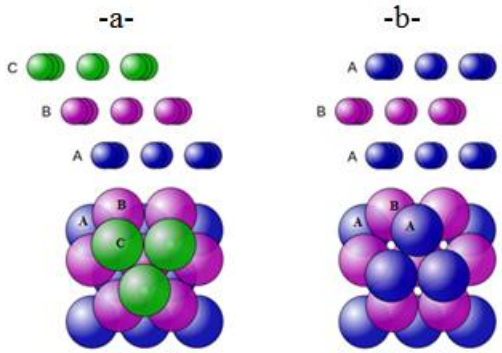



**Figure 5:** a) Compact A-B-C stacking and b) Compact A-B stacking.

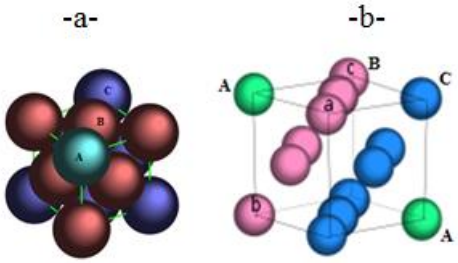



**Figure 6:** a) The closely stacked arrangement in the face-centered cubic compact model and b) The exploded model.

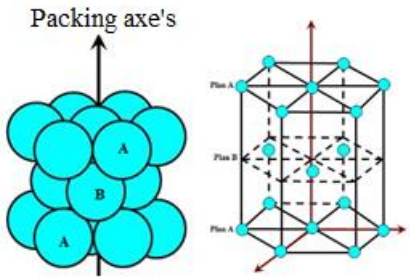



**Figure 7:** a) The closely stacked arrangement in the hexagonal compact model and b) The exploded model.

## IV. Procedure

### a) Experiment 1

- Construct the A-B-C compact stacking using the spheres provided to you,
- Construct the unit cell of the FCC lattice,
- Represent the unit cell in perspective,
- Identify the sequence of the A-B-C compact layers in the FCC unit cell,
- Indicate the direction in which these layers are stacked,
- Determine the coordination number of an atom and the multiplicity of the unit cell.
- Draw the projection of the unit cell onto the XOY plane,
- Derive the reduced coordinates of the atoms.
- Establish the relationship between the atomic radius R and the lattice parameter a.
- Observe the positions of the tetrahedral and octahedral sites,
- Represent these sites and provide their reduced coordinates.

**Application**: In the alloy with the formula $Al_xNi_yTi_z$, titanium atoms form the face-centered cubic lattice, aluminum atoms occupy all the octahedral sites, and nickel atoms occupy all the tetrahedral sites.

- **Represent the unit cell in perspective**: Illustrate the arrangement of titanium, aluminum, and nickel atoms within the FCC lattice.
- **Determine the alloy's formula**: Based on the number of atoms per unit cell and the positions of aluminum and nickel in the octahedral and tetrahedral sites, respectively, derive the stoichiometric ratio of Al, Ni, and Ti.

### b) Experiment 2

- Construct the A-B compact stacking: Use the provided spheres to form the arrangement.
- Deduce the construction of the elementary and triple hexagonal unit cells: Represent both unit cells in perspective.
- Identify the sequence of the A-B-A-B compact layers in the triple hexagonal unit cell: Indicate the direction along which these layers are ordered.
- Determine the coordination number of an atom and the multiplicity of the triple unit cell.

- Draw the projection of the unit cell onto the XOY plane: Derive the reduced coordinates of the atoms.
- Establish the relationship between the atomic radius R and the lattice parameter a.
- Establish the relationship between the lattice parameters a and c.
- Observe the positions of the tetrahedral and octahedral sites: Represent these sites and provide their reduced coordinates.

**Application**: Zinc crystallizes in a compact hexagonal structure. What is the value of the lattice parameter a, given that the atomic radius of zinc is 1.37 Å?

**c) Experiment 3**

- Construct the non-compact stacking: Use the provided spheres to form the arrangement.
- Deduce the construction of the body-centered cubic (BCC) unit cell: Represent the unit cell in perspective.
- Identify the sequence of the non-compact layers in the body-centered cubic unit cell: Indicate the direction in which these layers are ordered.
- Determine the coordination number of an atom and the multiplicity of the unit cell.
- Draw the projection of the unit cell onto the XOY plane: Derive the reduced coordinates of the atoms.
- Establish the relationship between the atomic radius R and the lattice parameter a.
- Observe the positions of the tetrahedral and octahedral sites: Represent these sites and provide their reduced coordinates.

**Application**: In the alloy with the formula $Cs_xCl_y$, chloride ions form a simple cubic lattice, and cesium ions occupy the centers of the cubes.

- **Represent the unit cell in perspective**: Illustrate the arrangement of chloride and cesium ions in the unit cell.
- **Determine the formula of the alloy**: Based on the positions of cesium and chloride ions, calculate the stoichiometric ratio of Cs to Cl in the unit cell.
- **Provide the coordination number of each ion**: Identify the number of nearest neighbors for both chloride and cesium ions.
- **Establish the relationship between the lattice parameter a and the ionic radii $R_{Cl}$ and $R_{Cs}$**: Relate the lattice parameter to the radii of chloride and cesium ions.

- **Calculate the density (ρ) of the crystal**:

Data: Molar mass of the crystal, M=168.36 g/mol, ionic radius of $Cl^-$, $R_{Cl}$=1.81 Å, ionic radius of $Cs^+$, $R_{Cs}$=1.67 Å, C=0.63, Avogadro's number, N=$6.023\times10^{23}$ atoms/mol.

# Lab 2
# X-ray Diffraction

# Lab 2: X-ray Diffraction

## I. Principle

Irradiating polycrystalline powder samples of NaCl and CsCl with X-rays and examining the resulting diffraction patterns captured on photographic films is a technique used to analyze the crystal structures of these materials.

## II. Objectives

- Capture X-ray diffraction photographs using the Debye-Scherrer method for powder samples of sodium chloride (NaCl) and cesium chloride (CsCl).
- Evaluate and assign the Debye-Scherrer diffraction rings to the corresponding crystal lattice planes.
- Determine the lattice constants of the studied samples.
- Determine the number of atoms per unit cell in each crystal structure.

## III. Theoretical Reminder

When an X-ray beam strikes a material, it is scattered by the electrons of the atoms in that material. The atomic scattering power of a single atom is represented by the atomic form factor *f*, which is approximately proportional to the number of electrons in the atom. Therefore, we have:

$$f \propto Z \tag{1}$$

where Z is the atomic number. If the material being studied has an ordered periodic structure, constructive interference occurs between the diffracted waves. The resulting diffraction is determined by Bragg's law:

$$2.d_{hkl}\,.sin\theta = n.\lambda \tag{2}$$

where n is an integer indicating the diffraction order, $d_{hkl}$ is the interplanar spacing, $\lambda$ is the wavelength of the X-rays, and $\theta$ is the Bragg angle.

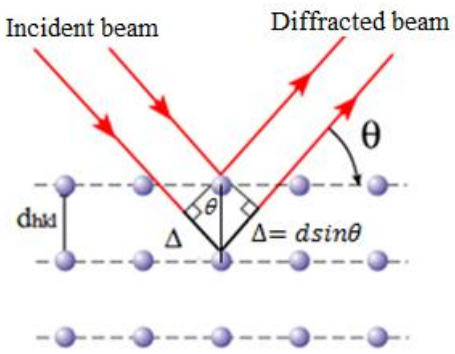



**Figure 1:** Diffraction of an X-ray beam by crystal planes

The intensity of the diffracted X-rays is proportional to the square of the structure factor $F_{hkl}$, given by the following relation:

$$F_{hkl} = \sum_j f_j e^{i2\pi(hx_j+ky_j+lz_j)} \quad (3)$$

where $x_j$,$y_j$,$z_j$ are the coordinates of an atom j in the unit cell, h, k, l are the Miller indices of the reflecting plane, and $f_j$ is the atomic scattering factor of atom j.

For a body-centered cubic (BCC) structure, the positions of the atoms in the unit cell are: (0,0,0) and (1/2, 1/2, 1/2).

- $F_{hkl}$=0 if h+k+l=2n+1 (i.e., an odd number).
- $F_{hkl}$=2$f_j$ if h+k+l=2n (i.e., an even number).

For a face-centered cubic (FCC) structure, the positions of the atoms in the unit cell are: (0, 0, 0), (1/2, 1/2, 0), (1/2, 0, 1/2), (0, 1/2, 1/2).

- $F_{hkl}$=0 if h, k, l are not of the same parity (i.e., mixed).
- $F_{hkl}$=4fj if h, k, l are all of the same parity (i.e., all even or all odd).

When monochromatic X-rays bombard a polycrystalline sample composed of randomly oriented crystallites, some of the crystallites are oriented in such a way that their lattice planes and the direction of the primary beam satisfy Bragg's law. As a result, all reflections from a particular set of lattice planes form a cone, with the axis of the cone aligned with the incident beam and the angle at the apex being 4θ. A photographic film placed

perpendicular to the primary beam will record these concentric circles as reflections, forming Debye-Scherrer rings.

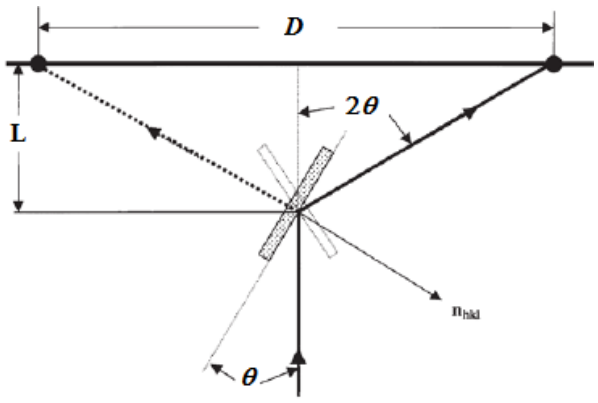



**Figure 2:** Demonstration of the diffraction of a primary X-ray beam by a photographic film placed perpendicular to the incident beam.

If the diameter of a diffraction ring is D, and L is the distance between the sample and the film, the Bragg angle is given by the following expression:

$$\theta = \frac{1}{2} arctan \frac{D}{2L} \tag{4}$$

If the sample consists of cubic crystals with a lattice parameter a, then:

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \tag{5}$$

By substituting relations (4) and (5) into relation (2), we obtain:

$$sin^2\theta = sin^2\left(\frac{1}{2} arctan \frac{D}{2L}\right) = \frac{\lambda^2}{4a^2}(h^2 + k^2 + l^2) \tag{6}$$

The diffracted rings are assigned to the Miller indices of the reflecting planes of the crystal lattice as follows: the ratios are obtained from the sum of the squares of the triplets (h, k, l), and then a correspondence is sought between these ratios and the ratios of the observed values of $sin^2\theta$ associated with the diffraction rings.

To begin, the deepest ring is assigned to the (100) plane. If this does not yield a satisfactory result, the (110) plane is tried, and so on, until a satisfactory match of the ratios is found.

## IV. Materials and Equipment

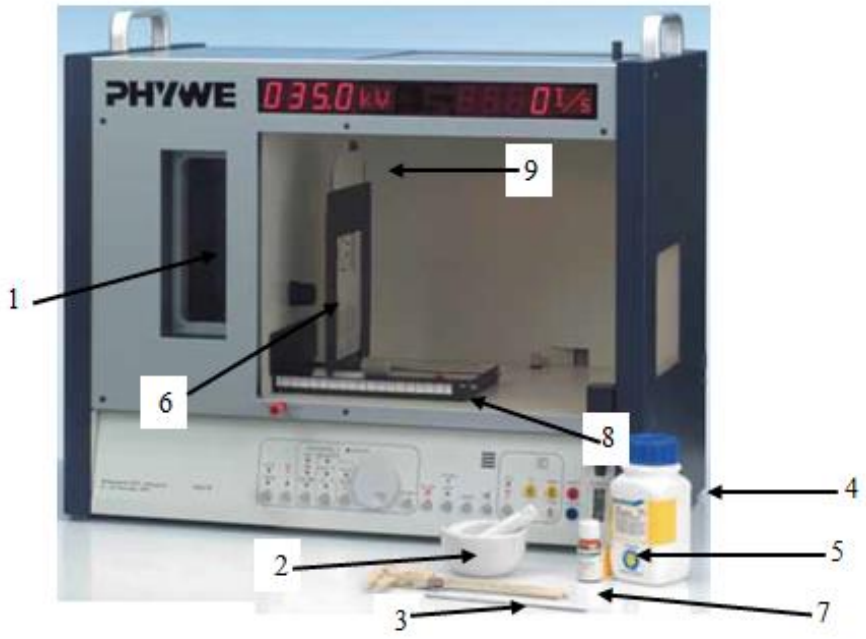



**Figure 3:** The basic X-ray apparatus with a 35 kV.

1- Mo anode drawer for X-ray apparatus

2- Mortar and pestle, 70 ml, porcelain

3- Spoon with spatula tip, length = 150 mm

4- Sodium chloride, 250 g

5- Cesium chloride, 5 g

6- Diaphragm tube with zirconium foil

7- Vernier caliper, plastic

8- Film holder

9- X-ray films, 100 × 100 $mm^2$

## V. Procedure

- The sample (the NaCl powder, then the CsCl powder) placed on a sample holder must be in the form of a fine, homogeneous powder to ensure isotropic scattering of the X-rays, and.
- Use an X-ray apparatus equipped with an X-ray source of Mo ($\lambda(K_\alpha)$=0.711Å), directed towards the sample at a controlled intensity

- A photographic film is positioned perpendicularly to the incident X-ray beam, at a known distance from the sample, and set to capture the diffraction patterns in the form of rings.
- The primary X-ray beam bombards the sample, causing diffraction of the X-rays by the crystal planes in the sample.
- After irradiating the sample, the film is developed using a specific X-ray photographic developer.
- The concentric circles observed on the film are called "Debye-Scherrer rings."
- Each ring corresponds to a reflection of the X-rays from a specific crystal plane and attributed to a particular set of crystal planes using Bragg's law and Miller indices.
- The radii of the rings are measured, and from these, the values of $sin^2\theta$ are calculated.
- These values are compared with Miller indices to identify the crystal planes which are responsible for each diffraction ring.
- The lattice constants can then be determined using Bragg's relationship.
- This allows for the characterization of the crystal structure of the studied sample.
- The interplanar distances $d_{hkl}$ of the reflecting planes are calculated from Bragg's relation.
- The Miller indices (h, k, l) are determined for each diffraction to provide a complete picture of the crystal structure of the material.

**a) Study of NaCl powder**

After taking Debye-Scherrer photographs of the sodium chloride powder samples,

- Calculate the structure factor $F_{hkl}$ for the NaCl structure, and discuss its values according to the values of the Miller indices h, k, l.
- Evaluate the Debye-Scherrer rings using the vernier caliper and assign them to the corresponding lattice planes, knowing that the distance between the sample and the film is L=32 mm+0.5 mm (the thickness of the film). The wavelength of the incident X-ray beam is λ(Kα)=0.711 Å (the average value of λ(Kα1) and λ(Kα2).
- Report the values in the following table:

| N° of the | Intensity | *D* (mm) | θ (°) | $\frac{sin^2\theta_n}{sin^2\theta_1}$ | $\frac{(h^2+k^2+l^2)_n}{(h^2+k^2+l^2)_{111}}$ | *hkl* | $d_{hkl}$(Å) | *a* (Å) |
|---|---|---|---|---|---|---|---|---|

| reflexion | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 1 | | | | | | | | |
| 2 | | | | | | | | |
| 3 | | | | | | | | |
| 4 | | | | | | | | |
| 5 | | | | | | | | |
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| 7 | | | | | | | | |

- What observations can you make about the obtained hkl indices?
- Based on these, what would the crystal structure of NaCl be?
- Calculate the NaCl lattice constant and express it as: $a_{NaCl} = a_{moy} \pm \Delta a$.
- Determine the relative error $\Delta a/a$.
- Compare your calculated value of a with the literature value: $a_{litt} = 5.639$ Å.
- Calculate the multiplicity m of the NaCl unit cell, using the following data: $\rho_{NaCl}$ = 2.16 g/cm³, molar mass of Na ($M_{Na}$ = 22.9 g/mol), molar mass of Cl ($M_{Cl}$ = 35.45 g/mol), and Avogadro's number $N_A = 6.022 \times 10^{23}$ atoms/mol.

**b) Study of CsCl powder**

After taking Debye-Scherrer photographs of the cesium chloride powder samples,

- The same questions as those asked for the NaCl powder sample, using the following data: $a_{litt}$ = 4.110 Å, $\rho_{CsCl}$ = 3.97 g.cm$^{-3}$, molar mass of Cs ($M_{Cs}$ = 132.91g/mol), molar mass of Cl ($M_{Cl}$ = 35.45 g/mol), and Avogadro's number $N_A = 6.022 \times 10^{23}$ atoms/mol.

# Lab 3
# Electron Diffraction

# Lab 3: Electron Diffraction

## I. Principle

Observation of electron diffraction on a polycrystalline graphite sample and confirmation of the wave-like nature of electrons.

## II. Objectives

- Calculating Planck's constant h.
- Verifying de Broglie's hypothesis for the electron wavelength.

## III. Theoretical Reminder

In 1924, Louis de Broglie proposed the hypothesis that particles inherently exhibit wave-like properties, with their wavelength being related to their momentum as follows:

$$\lambda = \frac{h}{p} \tag{1}$$

Where h=6.6256×$10^{-34}$ J.s is the Planck constant and λ\lambdaλ is the wavelength of the moving particle.

This equation can be transformed for electrons that have been accelerated by a voltage Ua into the following equation:

$$\lambda = \frac{h}{\sqrt{2.m.e.U_a}} \tag{2}$$

Where: λ is the wavelength of the electron, m is the mass of the electron (9.11×$10^{-31}$ kg, e is the elementary charge (1.602×$10^{-19}$ C), Ua is the acceleration voltage.

The momentum p of a particle can be determined from the relation:

$$p = m.v = \sqrt{2.e.m.U_a} \tag{3}$$

In this experiment, the wave-like behavior of electrons in a vacuum tube is demonstrated through diffraction on a polycrystalline graphite sample. Diffraction rings are observed on the fluorescent screen of the tube, surrounding a central spot along the beam's direction, with the diameter D varying depending on the accelerating voltage. These rings result from the diffraction of electrons by the lattice planes of microcrystals that meet the Bragg condition:

$$2dsin\theta = n\lambda \tag{4}$$

θ: The Bragg angle, n: the diffraction order (with n=1), d: the distance between the crystal planes.

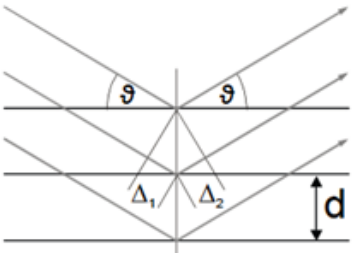



**Figure 1:** X-ray diffraction by a set of lattice planes hkl.

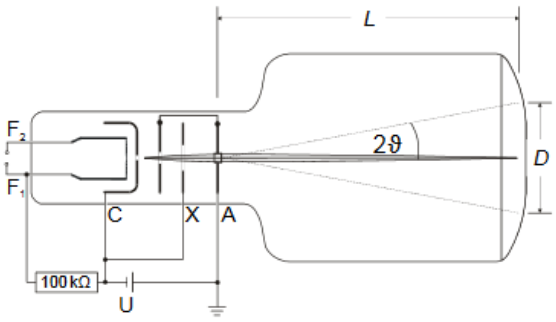



**Figure 2:** X-ray diffraction tube.

The diameter of the diffraction ring attributed to the Bragg angle θ is:

$$D = 2.L.tan2\theta \tag{5}$$

where: L is the distance between the graphite sample and the fluorescent screen, θ is the Bragg angle.

For small Bragg angles, the diffraction ring diameter D can be approximated by the following formula:

$$d = \lambda . \frac{2.L}{D} \tag{6}$$

Using the previous relations, we find:

$$D = k . \frac{1}{\sqrt{U_a}} \tag{7}$$

with

$$k = \frac{2.L.h}{d.\sqrt{2.m.e}} \tag{8}$$

Since graphite has a crystalline structure with two interplanar distances $d_1=2.13\times10^{-10}$ m and $d_2=1.23\times10^{-10}$m, two diffraction rings with average diameters $D_1$ and $D_2$ will be observed in the first diffraction order (n=1).

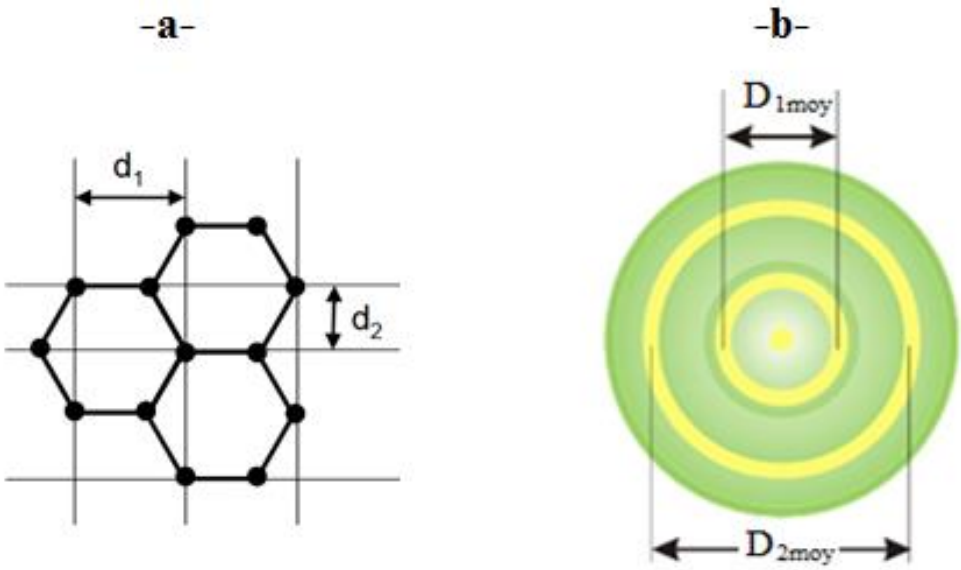



**Figure 3:** a) Interplanar distances $d_1$ and $d_2$, b) Diffraction rings.

## IV. Materials and Equipment

**Figure 4:** Experimental setup for electron diffraction.

## V. Procedure

- Apply an acceleration voltage Ua≤5 kV and observe the diffraction pattern on the fluorescent screen.
- Vary the acceleration voltage between 3 kV and 5 kV in steps of 0.5 kV and, each time, measure the inner and outer diameters $D_1$ and $D_2$ of the two diffraction rings.
- Record the measured values in the table below.

| $U_a$ (kV) | Ring 1 | | | Ring 2 | | |
|---|---|---|---|---|---|---|
| 3.0 | $D_{1int}$ (cm) | $D_{1ext}$ (cm) | $D_{1moy}$ (cm) | $D_{2int}$ (cm) | $D_{2ext}$ (cm) | $D_{2moy}$ (cm) |
| 3.5 | | | | | | |
| 4.0 | | | | | | |
| 4.5 | | | | | | |
| 5.0 | | | | | | |

- Measure the distance "L" between the graphite and the fluorescent screen.
- Calculate the experimental wavelength values $\lambda_{exp}$ and theoretical wavelength values $\lambda_{theo}$ for each diffraction ring, and record both the measured and calculated values in the table below.

| $U_a$ (kV) | Ring 1 | | | Ring 2 | | |
|---|---|---|---|---|---|---|
| 3.0 | $D_{1moy}$ (cm) | $\lambda_{1exp}$ (pm) | $\lambda_{1theo}$ (pm) | $D_{2moy}$ (cm) | $\lambda_{2exp}$ (pm) | $\lambda_{2theo}$ (pm) |
| 3.5 | | | | | | |
| 4.0 | | | | | | |
| 4.5 | | | | | | |
| 5.0 | | | | | | |

- What would you say about the obtained results?
- Plot the two curves $D_{1moy}=f(Ua)$ and $D_{2moy}=f(Ua)$ on the same graph and, from these two curves, determine the experimental values of the interplanar distances $d_1$ and $d_2$ of graphite. (Use the least squares method)
- What would you say about the obtained results?
- Calculate the experimental value of Planck's constant h.
- Conclude with interpretations.

# Lab 4
# Mechanical Testing

# Lab 4: Mechanical Testing

## I. Principle

The principle of this test is based on the bending of a simply supported flat rod under a central load. The applied force causes deflection, which depends on the material's stiffness, the flat rod's geometry, and the force magnitude. By measuring the deflection and knowing the beam's dimensions, the Young's modulus (E) of the material can be calculated.

## II. Objectives

- Determine the Young's modulus (E) of a material from the deflection of a flat rod under a known load.
- Understand the relationship between force, deflection, and flat rod geometry.
- Measure the bending stiffness of materials.
- Understand elastic deformation behavior.
- Identify the flexural properties of materials.

## III. Theoretical Reminder

A flat rod with thickness b and width a, is supported at both ends by two supports separated by the span length L. It is subjected to a force acting at its center. The deflection λ (or bending) is then expressed as a function of the Young's modulus E and the geometric properties of the flat rod, as follows:

$$\lambda = \frac{1}{4}\left(\frac{L}{b}\right)^3 . \frac{1}{a} . \frac{F_y}{E} \qquad (1)$$

Where: λ is the deflection (or bending) of the flat rod, L is the span length between the supports, b is its thickness, a is its width, $F_y$ is the applied force at its center, E is the Young's modulus of the material.

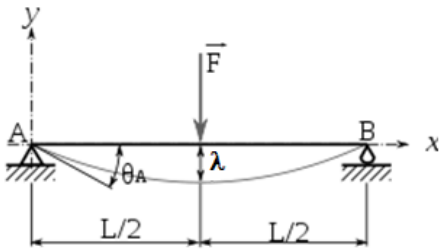



**Figure 1:** Deflection of a flat rod under applied force $F_y$.

The table below contains the theoretical values of the Young's modulus for different types of materials.

| Material | a [mm] | b [mm] | E [N.m$^{-2}$] |
|---|---|---|---|
| Steel | 10 | 1.5 | $2.059\times10^{11}$ |
| Steel | 10 | 2 | $2.063\times10^{11}$ |
| Steel | 10 | 3 | $2.171\times10^{11}$ |
| Steel | 15 | 1.5 | $2.204\times10^{11}$ |
| Steel | 20 | 1.5 | $2.111\times10^{11}$ |
| Aluminum | 10 | 2 | $6.702\times10^{10}$ |
| Brass | 10 | 2 | $9.222\times10^{10}$ |

## IV. Materials and Equipment

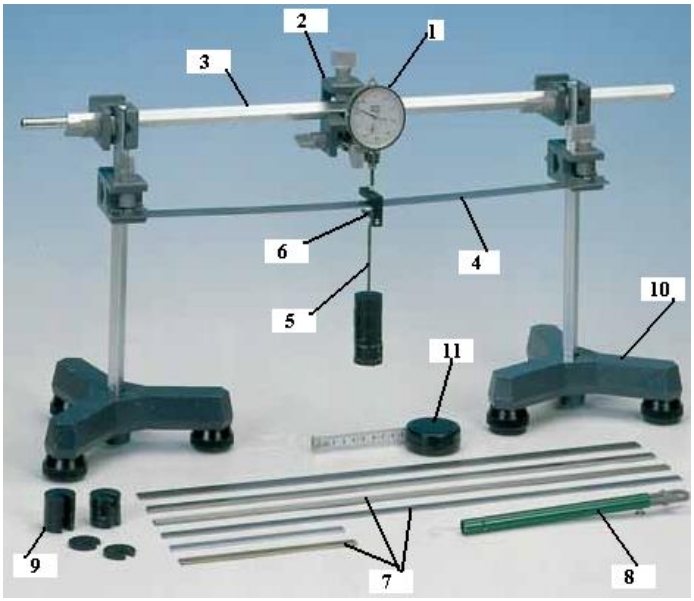
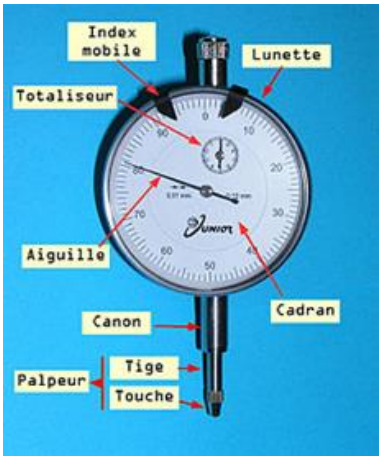



**Figure 2:** Experimental setup for mechanical testing.

1. Dial indicator, 10/0.01 mm
2. Comparator support
3. Square rod PASS
4. Flat rod
5. Weight holder for slit weights
6. Knife with clamp
7. Set of 7 flat rods
8. Dynamometer 1 N
9. Slit weights
10. Tripod PASS
11. Measuring tape, l = 2 m




**Dial indicator**

## V. Procedure

### a) Experiment 1

- Place the steel bar with a width a=10 mm and thickness b=1.5 mm on the two supports, spaced L=0.30 m apart.
- Hang different loads (masses) at its center and measure the deflection λ using the dial indicator.
- Record the measured values in a table and plot the curve λ=*f* (F).
- Determine, from this curve, the value of the Young's modulus "E".
- Conclude with interpretations.

### b) Experiment 2

- For a constant load, place in turn the steel bars of different widths a, and a constant thickness b=1.5 mm on the two supports, spaced L=0.30 m apart.
- Hang a fixed load and measure the deflection λ as a function of the width a using the dial indicator.
- Record the measured values in a table and plot the curve λ=*f* (a).
- Conclude with interpretations.

### c) Experiment 3

- Place the steel bar with a width of 10 mm and a thickness of 2 mm on the support.

- Hang a fixed load and measure the deflection λ as a function of the distance between the supports L.
- Record the measured values in a table and plot the curve λ=*f* (L).
- Determine, from this curve, the value of the Young's modulus "E".
- Conclude with interpretations.

**d) Experiment 4**

- Place, one at a time, the steel bars of different thicknesses and a fixed width a=10 on the support.
- For a fixed load and a fixed support distance, measure the deflection λ as a function of the thickness of each bar.
- Record the measured values in a table and plot the curve λ=*f* (b).
- Conclude with interpretations.

**e) Experiment 5**

- Place, one at a time, the aluminum, and brass bars with a width a=10 mm and a thickness b=2 mm on the support.
- Fix the distance between the supports and measure the deflection λ as a function of the load m.
- Record the measured values in a table and plot the curves λ=*f* (F).
- Determine, from these curves, the value of the Young's modulus E for each bar.
- Conclude with interpretations.

# Lab 5
# Microhardness

# Lab 5: Microhardness

## I. Principle

A small indenter, usually a diamond pyramid or Vickers indenter, is used to create an impression on the material's surface. The hardness value is then determined by measuring the size of the indentation and calculating it based on the applied force and indentation size.

## II. Objectives

- Highlight the hardness test.
- Be able to operate and use the durometer to measure the hardness of different materials.
- Characterize a material based on the hardness test.

## III. Theoretical Reminder

Hardness defines a material's resistance to deformation when a hard object penetrates its surface. It is typically assessed using durometers, which perform non-destructive tests widely used in manufacturing for rapid quality control of finished products. This testing method provides valuable information about a material's properties, including tensile strength, ductility, and wear resistance.

According to standards, hardness is expressed as a dimensionless number and is measured on different scales depending on the material type. It is commonly represented by the letter H, derived from the word "Hardness" in English.

The most commonly used hardness testing methods include the Brinell, Vickers, and Rockwell tests.

The Brinell test consists of pressing an indenter, usually a steel or tungsten carbide ball with a diameter D, into the material under a defined load F. Once the load is removed, two diameters, $d_1$ and $d_2$, at a 90° angle to each other, are measured on the impression left on the surface. These measurements are taken using a magnifying device and a graduated scale, accounting for the magnification factor.

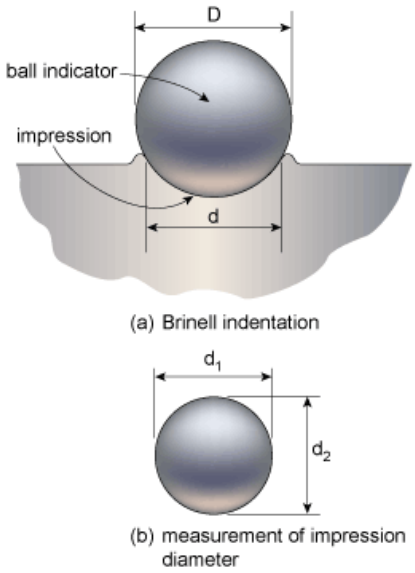



**Figure 1:** Brinell principal

The Brinell hardness number is calculated using the formula:

$$BH = 0.102 \frac{F}{\frac{\pi D}{2}\left(D - \sqrt{D^2 - d^2}\right)} \quad (1)$$

Where: *P* is the test load [N], *D* is the diameter of the ball [mm] and *d* is the arithmetic mean of the two diagonals *d1* and *d2* [mm].

The Brinell method is appropriate for hardness testing of materials ranging from soft metals (light metals with a density < 5 mg/cm³, such as aluminum, titanium, alkali metals, alkaline earth metals, lead, and zinc) to harder metals (such as steel or iron).

The principle of the Vickers test is similar to that of the Brinell test, with the only difference being the shape of the indenter. In this test, a square-based diamond pyramid indenter with a 136° apex angle is pressed into the material under a specified load F. After the load is removed, two diagonals, $d_1$ and $d_2$, are measured on the impression left on the surface. These measurements are taken using a suitable optical system.

The Vickers hardness number is calculated using the formula:

$$VH = 0.102 \frac{2Psin\left(\frac{136^\circ}{2}\right)}{d^2} = 0.189 \frac{P}{d^2} \quad (2)$$

Where: *P* is the test load [N], and *d* is the arithmetic mean of the two diagonals *d1* and *d2* [mm].

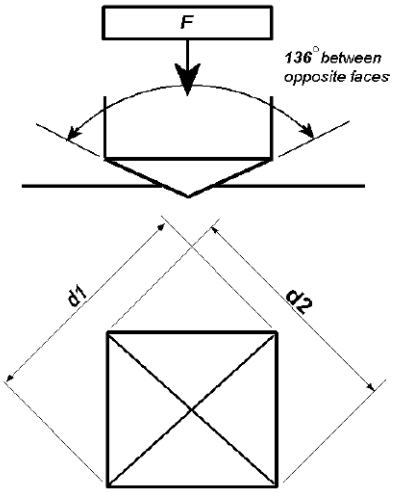



**Figure 2:** Vickers principal

The Vickers hardness test is applicable to both hard and soft materials because the measurement remains unaffected by the applied load (ranging from 49 to 981 N), thanks to the constant penetration angle. However, it requires a well-prepared surface finish, and the sample must have small dimensions. This testing method is primarily conducted in a laboratory environment and includes a specific subgroup for testing the hardness of welds.

## IV. Materials and Equipment

**Figure 3:** Experimental setup for microhardness testing.

## V. Procedure

### a) Brinell Hardness

- Place the indenter in contact with the material surface.
- Apply the force and maintain it for 10 to 15 seconds.
- Measure two diameters of the indentation, at 90° to each other. The measurement is taken using a magnifying device and a calibrated ruler, accounting for the magnification factor.

**Note:** Hardness is expressed as the ratio of the force F to the surface area S of the spherical impression made in the metal.

### b) Vickers hardness

- Polish both surfaces of each specimen.
- Clean with an alumina solution to remove burrs from the surfaces.
- If necessary, recalibrate the durometer using standard samples.
- For one face, take several measurements and calculate the average (limit to three measurements for each face) for each metal using both hardness methods.
- Present these measurements in a comparative table.
- Interpret and discuss the results obtained, and draw conclusions.

## References


1. Kittel, C. *Introduction to Solid State Physics* (8th ed.). Wiley. (2004).

2. Cullity, B. D., & Stock, S. R. *Elements of X-ray Diffraction* (3rd ed.). Prentice Hall. (2001).

3. Timoshenko, S., & Goodier, J. N. *Theory of Elasticity* (3rd ed.). McGraw-Hill. (1982).

4. Berkovitz, J. A., & Millman, P. M**.** *X-Ray Diffraction and the Identification and Analysis of Clay Minerals*. Clarendon Press. (1994).

5. Callister, W. D. *Materials Science and Engineering: An Introduction* (9th ed.). Wiley. (2018).

6. Meyers, M. A., & Chawla, K. K. *Mechanical Behavior of Materials* (2nd ed.). Cambridge University Press. (2009).