# PRACTICAL WORKBOOK

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



Name	 	 
Roll No	 	 
Batch		 

**Department of Metallurgical Engineering** NED University of Engineering and Technology

# PRACTICAL WORKBOOK

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



## **PREPARED BY:**

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This is to certify that this practical book contains \_\_\_\_\_ pages.

Approved by:

Chairman MYD

**Department of Metallurgical Engineering** NED University of Engineering and Technology

# **CERTIFICATE**

It is Certified that Mr. / Miss _	
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his/her course work in the subject of \_\_\_\_\_\_

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His/her performance is reflected by index/contents of his/her practical workbook. This overall performance of the student is Excellent/Very Good/Good (satisfactory)/Not Satisfactory

**Course Teacher** 

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# **PRACTICAL 1**

**OBJECTIVE:** Safety precautions to be taken during designing, selection and characterization of materials.

#### **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. <u>DO NOT</u> 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 labeled 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 the 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

## EXERCISE

Q1. Name one hazard and safety measure to take while dealing with liquid nitrogen.

Q2. If you were put in charge of the SEM lab describe the steps you would take to ensure all safety requirement are met.

Q3. Describe a couple of precautions you should take while dealing with HT equipment

# **PRACTICLE 2**

**OBJECT:** To perform 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 Counting The 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  $\sigma$ , is simply:

$$\sigma = 1/N_L$$



## **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	Phase3
Amount			

# **POINT COUNTING (via microscopy)**

	Phase 1	Phase2	Phase3	
Amount				

## **GRAIN SIZE MEASUREMENT:**

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

## **CONCLUSION:**

## **EXERCISE**

Q1. Write down the procedure of point counting method you performed.

Q2.What is the effect of Grain size on mechanical properties? (I.e. UTS, hardness and toughness)

Q3. Explain the kinetics of phase transformation w.r.t Gibbs free energy.

## PRACTICAL 3

**OBJECT:** To study the operation and various functions of **IMAGE ANALYSER**.

## **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.



Fig.1

## **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**

## **DIFFERENTIAL INTERFERENCE CONTRAST (DIC):**

An excellent mechanism for rendering contrast in transparent specimens, differential interference

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			

contrast (DIC) microscopy is a beam-shearing interference system in which the reference beam is

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 waterborne 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

# EXERCISE

Q1. Define the following terms w.r.t their usage in microscopic techniques;

- a) Dark field (DF)
- b) Differential interference contrast (DIC)
- c) Polarization
- d) fluorescence illumination

## **PRACTICAL 4**

**OBJECTIVE:** To Study Ductile and Brittle fracture using Stereo Microscope.

## **THEORY:**

Brittle fractures and ductile fractures are two of the best known failure modes. The factors that control both brittle and ductile fracture revolve around the energy that must be provided to extend the fracture by a microscopic distance and the amount of elastic strain energy that is concurrently made available by that microscopic crack extension. If the elastic strain energy being released exceeds the energy required for crack extension then we have spontaneous fracture.

The crack stops growing either when it reaches the end of the part (the part breaks), or the energy required for crack extension exceeds the strain energy being released by that same crack extension and we have crack arrest. This happens, for example when a crack grows through an area under tensile stress and then stops when it runs into an area of stress that is reduced or compressive.

**Ductile materials** - extensive plastic deformation and energy absorption ("toughness") before fracture.

Brittle materials - little plastic deformation and low energy absorption before fracture.

## SOME CHARACTERISTICS OF BRITTLE FRACTURE:

- 1. There is no gross, permanent deformation of the material; fracture surface is smooth and shiny.
- 2. The surface of the brittle fracture tends to be perpendicular to the principal tensile stress although other components of stress can be factors.
- 3. Characteristic crack advance markings frequently point to where the fracture originated.
- 4. The path the crack follows depends on the material's structure. In metals, transgranular and intergranular cleavage are important.



Fig1: Brittle fracture in mild steel

### SOME CHARACTERISTICS OF DUCTILE FRACTURE:

- 1. There is permanent deformation at the tip of the advancing crack that leaves distinct patterns.
- 2. As with brittle fractures, the surface of a ductile fracture tends to be perpendicular to the principal tensile stress, although other components of stress can be factors.
- 3. In ductile, crystalline metals and ceramics it is microscopically resolved shear stress that is operating to expand the tip of the crack.
- 4. The fracture surface is dull and fibrous.
- 5. There has to be a lot of energy available to extend the crack.



### **STEREO MICROSCOPE:**

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 threedimensional 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  $50 \times$ .

There are two major types of magnification systems in stereo microscopes. One is fixed

magnification in magnification is set of objective degree of other is zoom or magnification, continuously magnification Zoom systems magnification auxiliary increase total factor. Also, both fixed and varied by



which primary achieved by a paired lenses with a set magnification. The pancreatic which are capable of a variable degree of across a set range. can achieve further through the use of objectives that magnification by a set total magnification in zoom systems can be changing eyepieces.

Fig 3. Stereo Microscope

## EXERCISE

- 1. Describe crack propagation for ductile and brittle materials.
- 2. Explain why brittle materials are much less strong than possible theoretically
- 3. Observe the brittle fracture surface at 10x, 20x and 40x magnifications on stereo microscope and write down your comments.
- 4. Observe the ductile fracture surface at 10x, 20x and 40x on stereo microscope and write down your comments.

# PRACTICAL 5

# **OBJECTIVE:** To study different parts of **Scanning Electron Microscope** (SEM) and its

various operation modes.

## **INTRODUCTION:**

In scanning electron microscopy, (SEM) an electron beam is scanned across a sample's surface. When the electrons strike the sample, a variety of signals are generated, and it is the detection of specific signals which produces an image or a sample's elemental composition. The three signals which provide the greatest amount of information in SEM are the secondary electrons, backscattered electrons, and X-rays.



Fig 1: Shows the signals generated by the interaction of electron with matter

Two main parts of the SEM are the electronic console (operation unit) and the electron optical column. The console provides the switches and knobs for adjusting the focus, magnification and image intensity on viewing and photography screens. The column is where the beam is generated, focused on to a small spot, and scanned across the specimen to create signals that control the intensity of the image on the viewing screen [refer to figure 2 & 3].



top of the column where electrons are emitted from a hot tungsten filament and accelerated down an evacuated column. The three gun components are the filament, the wehnelt, which controls the number of electrons leaving the gun, and the anode, which accelerates the electrons to a selectable

voltage between 1 - 30 kV. A vacuum is necessary because electrons can travel only short distances in air.



Fig ;4 Conventional tungsten hairpin filament electron gun



Fig 5: LaB<sub>6</sub> filament

## **ELECTRON LENSES:**

Three electron lenses are used to demagnify the electron beam to a small spot about  $1\mu m$  in diameter. The condenser lens is located closest to the electron gun and the final or objective lens is located closest to the specimen. The objective lens moves the smallest spot formed by the beam up and down in space (working distance, WD) to meet the specimen surface, which is a focused condition.



**G SYSTEM:** 

The image is formed by pushing the beam across the specimen surface in a regular manner in synchronism with a beam scanning within the computer monitor on the console. Scan coils, used to push or deflect the beam, are located within the objective lens.



**Fig 9: scanning coils** 

## **OBJECTIVE APERTURE:**

A foil with a small hole (~100  $\mu$ m), located above the objective (final) lens. Its function is to limit the angular width of the electron beam to reduce aberrations and to improve depth-of-field in the image.

## **CORRECTION COIL:**

Corrects for astigmatism - a beam aberration caused by slight imperfections in the lenses or by the electric field associated with specks of dirt, causing elongated foci to form at slightly different focal lengths.

## **SPECIMEN CHAMBER:**

This large evacuated space below the objective lens contains the specimen stage along with electron signal detectors and a line to the vacuum pumping system.

## **ENERGY-DISPERSIVE X-RAY SPECTROSCOPY (EDS):**

Interaction of an electron beam with a sample target produces a variety of emissions, including xrays. An energy-dispersive (EDS) detector is used to separate the characteristic x-rays of different elements into an energy spectrum, and EDS system software is used to analyze the energy spectrum in order to determine the abundance of specific elements. EDS can be used to find the chemical composition of materials down to a spot size of a few microns, and to create element composition maps over a much broader raster area. Together, these capabilities provide fundamental compositional information for a wide variety of materials.

## **WORKING OF EDS:**

EDS systems include a sensitive x-ray detector, a liquid nitrogen dewar for cooling, and software to collect and analyze energy spectra. The detector is mounted in the sample chamber of the main instrument at the end of a long arm, which is itself cooled by liquid nitrogen. The most common detectors are made of Si(Li) crystals that operate at low voltages to improve sensitivity, but recent advances in detector technology make availabale so-called "silicon drift detectors" that operate at higher count rates without liquid nitrogen cooling.

An EDS detector contains a crystal that absorbs the energy of incoming x-rays by ionization, yielding free electrons in the crystal that become conductive and produce an electrical charge bias. The x-ray absorption thus converts the energy of individual x-rays into electrical voltages of proportional size; the electrical pulses correspond to the characteristic x-rays of the element.



Fig 10: Typical layout of EDS dectector

### **VACUUM SYSTEM:**

Vacuum is produced by an oil diffusion pump backed by a mechanical pump. In the diffusion pump a stream of hot oil vapor strikes and pushes air molecules toward a mechanical pump that expels them from the system. A mechanical pump and valve system are used to reevaluate the system because a diffusion pump only operates after a vacuum is created.

### EXERCISE

- Q1. What is the difference between electron and optical microscope?
- Q2. Write the principle of electron generation in Scanning Electron Microscope?
- Q3. Why Conductive coating is necessary for polymeric materials?
- Q4. Explain the phenomenon of sputter coating?
- Q5. What is the difference between EDS and WDS?
- Q6. Which type of signal is useful for EDS analysis?
- Q7. Basic working principle of EDS detector?
- Q8. Draw the schematic diagram of EDS & WDS System?

# **PRACTICAL 6**

**OBJECTIVE:** Study of Ductile and Brittle fracture using Scanning Electron Microscope (SEM)

### **INTRODUCTION:**

We are already familiar with properties of ductile and brittle materials. In this experiment we will examine the fracture surfaces at higher magnification using Scanning Electron microscope (SEM). SEM plays an important role in failure analysis because it provides us information at very high magnification and higher depth of field.

The Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM instead of a light microscope.

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time. The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification. Preparation of the samples is relatively easy since most SEMs only require the sample to be conductive. The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in research areas today.



Fig: Micrographs of brittle fracture



Fig: Micrographs of ductile fracture

## **CHARACTERISTICS OF A SEM:**

#### **Magnification:**

is the ratio of scanned area to the display area. For most SEM: 10-200,000x (or more).

Useful Magnification: ~20,000x

#### **Resolution:**

Theoretical Limit of Resolution: 50 Å (5 nm) Practical Resolution: 200 Å (20 nm) The resolving power of the SEM depends primarily on the effective beam diameter of the probe (spot size). For two points having a inter-point distance, d, to be resolved, spot size must be smaller than d.

### **Depth of Field:**

Depth field of the SEM is the greatest among microscopes: 20 mm at 10X 5 µm at 10,000X Large depth of field is a great advantage for keeping in focus all parts of a rough Topography, but a compromise must be made between field depth and resolution.

## **PROCEDURE:**

A detailed explanation of how a typical SEM functions follows (refer to the diagram below):



Fig: Schematic of SEM

- 1. The "Virtual Source" at the top represents the electron gun, producing a stream of monochromatic electrons.
- 2. The stream is condensed by the first condenser lens (usually controlled by the "coarse probe current knob"). This lens is used to both form the beam and limit the amount of current in the beam. It works in conjunction with the condenser aperture to eliminate the high-angle electrons from the beam
- 3. The beam is then constricted by the condenser aperture (usually not user selectable), eliminating some high-angle electrons
- 4. The second condenser lens forms the electrons into a thin, tight, coherent beam and is usually controlled by the "fine probe current knob"
- 5. A user selectable objective aperture further eliminates high-angle electrons from the beam
- 6. A set of coils then "scan" or "sweep" the beam in a grid fashion (like a television), dwelling on points for a period of time determined by the scan speed (usually in the microsecond range)

- 7. The final lens, the Objective, focuses the scanning beam onto the part of the specimen desired.
- 8. When the beam strikes the sample (and dwells for a few microseconds) interactions occur inside the sample and are detected with various instruments
- 9. Before the beam moves to its next dwell point these instruments count the number of interactions and display a pixel on a CRT whose intensity is determined by this number (the more reactions the brighter the pixel).
- 10. This process is repeated until the grid scan is finished and then repeated, the entire pattern can be scanned 30 times

### **OBSERVATION:**

- 1. Examine the brittle fracture surface at very high magnifications i.e. at above 2000x. Give comments about your observation.
- 2. Examine the ductile fracture surface at very high magnifications i.e. at above 2000x. Give comments about your observation.
- 3. How can you explain the different fracture surface appearances?

# **PRACTICLE 7**

**OBJECT:** To study different parts of **Transmission Electron Microscope** (TEM) and its various operation modes.

### **THEORY:**

**Transmission electron microscopy** (**TEM**) is a microscopy technique whereby a beam of electrons is transmitted through an ultra thin specimen, interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen; the image is magnified and focused onto an imaging device, such as a fluorescent screen, on a layer of photographic film, or to be detected by a sensor such as a CCD camera.TEMs are capable of imaging at a significantly higher resolution than light microscopes, owing to the small de Broglie wavelength of electrons. This enables the instrument's user to examine fine detail—even as small as a single column of atoms, which is tens of thousands times smaller than the smallest resolvable object in a light microscope. TEMs find application in cancer research, virology, materials science as well as pollution, nanotechnology, and semiconductor research.

### **COMPONENTS:**

A TEM is composed of several components, which include a vacuum system in which the electrons travel an electron emission source for generation of the electron stream, a series of electromagnetic lenses, as well as electrostatic plates. The latter two allow the operator to guide and manipulate the beam as required. Also required is a device to allow the insertion into, motion within, and removal of specimens from the beam path. Imaging devices are subsequently used to create an image from the electrons that exit the system.



## VACUUM SYSTEM:

To increase the mean free path of the electron gas interaction, a standard TEM is evacuated to low pressures, typically on the order of  $10^{-4}$  Pa. The need for this is twofold: first the allowance for the voltage difference between the cathode and the ground without generating an arc, and secondly to reduce the collision frequency of electrons with gas atoms to negligible levels, this effect is characterized by the mean free path. TEM components such as specimen holders and film cartridges must be routinely inserted or replaced requiring a system with the ability to re-evacuate on a regular basis. As such, TEMs are equipped with multiple pumping systems and airlocks and are not permanently vacuum sealed.

The vacuum system for evacuating a TEM to an operating pressure level consists of several stages. Initially a low or roughing vacuum is achieved with either a rotary vane pump or diaphragm pumps bringing the TEM to a sufficiently low pressure to allow the operation of a turbo molecular or diffusion pump which brings the TEM to its high vacuum level necessary for operations

## **SPECIMEN STAGE:**

TEM specimen stage designs include airlocks to allow for insertion of the specimen holder into the vacuum with minimal increase in pressure in other areas of the microscope. The specimen holders are adapted to hold a standard size of grid upon which the sample is placed or a standard size of self-supporting specimen. Standard TEM grid sizes is a 3.05 mm diameter ring, with a thickness and mesh size ranging from a few to 100  $\mu$ m. The sample is placed onto the inner meshed area having diameter of approximately 2.5 mm. usual grid materials are copper, molybdenum, gold or platinum. This grid is placed into the sample holder which is paired with the specimen stage. A wide variety of designs of stages and holders exist, depending upon the type of experiment being performed. In addition to 3.05 mm grids, 2.3 mm grids are sometimes, if rarely, used. These grids were particularly used in the mineral sciences where a large degree of tilt can be required and where specimen material may be extremely rare. Electron transparent specimens have a thickness around 100 nm, but this value depends on the accelerating voltage.





Fig: A single axis tilt sample holder for insertion into a TEM goniometer

The electron gun is formed from several components: the filament, a biasing circuit, a Wehnelt cap, and an extraction anode. By connecting the filament to the negative component power supply,

electrons can be "pumped" from the electron gun to the anode plate, and TEM column, thus completing the circuit. The gun is designed to create a beam of electrons exiting from the assembly at some given angle, known as the gun divergence semi angle,  $\alpha$ . By constructing the Wehnelt cylinder such that it has a higher negative charge than the filament itself, electrons that exit the filament in a diverging manner are, under proper operation, forced into a converging pattern the minimum size of which is the gun crossover diameter.



Fig: Cross sectional diagram of an electron gun assembly, illustrating electron extraction

## **ELECTRON LENS:**

Electron lenses are designed to act in a manner emulating that of an optical lens, by focusing parallel rays at some constant focal length. Lenses may operate electrostatically or magnetically. The majority of electron lenses for TEM utilize electromagnetic coils to generate a convex lens. For these lenses the field produced for the lens must be radially symmetric, as deviation from the radial symmetry of the magnetic lens causes aberrations such as astigmatism, and worsens spherical and chromatic aberration. Electron lenses are manufactured from iron, iron-cobalt or nickel cobalt alloys, such as <u>Permalloy</u>. These are selected for their magnetic properties, such as magnetic saturation, hysteresis and permeability.



Fig: A TEM split pole piece design lens

## **APERTURE:**

Apertures are annular metallic plates, through which electrons that are further than a fixed distance from the optic axis may be excluded. These consist of a small metallic disc that is sufficiently thick to prevent electrons from passing through the disc, whilst permitting axial electrons. This permission of central electrons in a TEM causes two effects simultaneously: firstly, apertures decrease the beam intensity as electrons are filtered from the beam, which may be desired in the case of beam sensitive samples. Secondly, this filtering removes electrons that are scattered to high angles, which may be due to unwanted processes such as spherical or chromatic aberration, or due to diffraction from interaction within the sample.

## **IMAGING METHODS:**

Imaging methods in TEM utilize the information contained in the electron waves exiting from the sample to form an image. The projector lenses allow for the correct positioning of this electron wave distribution onto the viewing system. Different imaging methods therefore attempt to modify the electron waves exiting the sample in a form that is useful to obtain information with regards to the sample, or beam itself. i.e.

#### 1. Contrast formation

Bright Field Diffraction Contrast Electron Energy Loss Phase Contrast

- 2. Diffraction
- 3. Three-dimensional

### **SAMPLE PREPARATION:**

Sample preparation in TEM can be a complex procedure. TEM specimens are required to be at most hundreds of nanometers thick, Preparation of TEM specimens is specific to the material under analysis and the desired information to obtain from the specimen. As such, many generic techniques have been used for the preparation of the required thin sections.

#### **EXERCISE**

- Q1. What is the difference between SEM and TEM?
- Q2. Which type of material used for filament in Transmission Electron Microscope?
- Q3. Why vacuum is necessary for Transmission Electron Microscope?
- Q4. Why diffusion pump needs backing of rotary pump?
- Q5. Why vacuum level is higher near electron gun?
- Q6. Write down the working principle of TEM
- Q7. What limits the resolution of a perfect optical microscope?

## **PRACTICAL 8**

**OBJECTIVE:** Determination of Chemical Composition by X-ray Fluorescence Spectroscopy (XRF).

## **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 lelemental analysis and lchemical analysis, particularly in the investigation of metals, glass, ceramics, polymer, composite, food, rocks minerals, building materials, and forensic science.



## 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.



## **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.

## **RESULTS:**

Sample	Reading	Elements (wt.%)								
INO.	1NO.									

## **OBSERVATIONS:**

### EXERCISE

Q1: Determine the Grade of your Sample?

Q2: Write down the XRF working principal in your own word.

Q3: How many numbers of element can be detected by an XRF?

# **PRACTICLE 9**

#### **OBJECTIVE:** Demonstration of various Parts and functions of an X-RAY POWDER

#### DIFFRACTOMETER.

#### **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.



## **MECHANICAL 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 diffracted beams. The receiving slits and counter are supported on the carriage E, which may be rotated about the axis 0 and whose angular position  $2\theta$  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 indicates the 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 as a simple ASCII file suitable for later analyses.

## **DIFFRACTOMETER PARTS:**



### EXERCISE

Q-1: List out Parts of a powder diffractometer and their functions.

Q-2: Describe the working principle of a powder diffractometer in your own words.

Q-3: Make a good, full-page sketch schematic drawing of the diffractometer you are using. Show the following parts:

- i. X-Ray Source
- ii. Specimen
- iii. Detector
- iv. Scatter and Divergence Slits
- v. Scatter and Receiving Slits

Q-4: You have given following mounting materials for your powder sample:

- i. Quartz slide
- ii. Aluminum Plate
- iii. Glass Slide
- iv. Double sided tape
- v. Bakelite Plate

What materials will you select for x-ray diffraction analysis and why?

# **PRACTICAL 10**

#### **OBJECTIVE:** Metal characterization by **Wet Chemical Analysis** (Titration Method).

## **APPARATUS:**

- An overflow dissolving vessel, divided by an overflow baffle into at least two chambers,
- Means for maintaining a constant stream of solvent flowing successively through the chambers of said overflow dissolving vessel, and
- Metering conveyor scale means for continuously metering a measured amount of a solid sample to be analyzed to the overflow dissolving vessel.

## **THEORY:**

To cater the needs of the metal industry where the range of metals and its alloys has no limits, different chemical testing facilities for testing wide range of metals and its alloys which includes identification and estimation of different elements are design, to provides accurate & precise results based on methods traceable to National and International standard test procedures.

#### WET CHEMICAL ANALYSIS:

Wet Chemical analysis excludes all techniques that use instrumentation for quantitative analysis i.e. Gravimetric (in which a chemical species is determined by weighing) and Tetrameter (which involves volume measurement of a liquid reactant) are two procedures that we use in laboratory to perform classical chemistry.

Most classical wet chemical methods can accommodate comparatively small amounts of a sample in diverse shapes or forms. It can also be applied to represent the gross chemistry of moderately inhomogeneous material sample.

Wet chemical analysis plays on important role in many other analytical applications including identification of elements in ferrous & non-ferrous materials i.e. ammonia, nitrogen, "Chloride, "Chromium, "Cyanide ,dissolved Oxygen, "Fluoride ,"Nitrate ,"Phenols ,"Phosphate ,"Phosphorus ,"Silica ,"Sulfate & "Sulfide.

# EXERCISE

Q1. Write the procedure of Wet Chemical Analysis which you performed

Q2. What chemical species were you able to identify? What can be assessed about the grade of steel you have been?

Q3. Name a drawback and advantage of characterization through Wet Chemical Analysis.

# **PRACTICAL 11**

**OBJECTIVE:** Designing and selection of **Aero-Engine** Materials

## **THEORY:**

Hotter, Stiffer, Stronger, Lighter... Where does the aero-engine go next?



#### The Past Shows The Future Will Be Hotter:

The figure on the left shows that the remarkable improvements in aero-engine performance have come about because the materials designer has been able to provide the engineer with materials can be used at hotter temperatures. Higher engine temperatures are needed so that the engines can run more efficiently, while weight reductions require stiffer, stronger, and lighter materials

#### If You Can't Stand The Heat!

The hot parts of the engine require materials which can operate at 1000°C, the cooler parts at 600°C. Furthermore, the environment is very harsh chemically and mechanically, with very large forces generated by the high rotational speeds and even the possibility of birds being sucked into the engine! The maximum service temperature chart (on the right) is a useful way of identifying new possibilities for materials development. By drawing lines at 600°C and 1000°C it is possible to identify the materials classes which might be suitable in this case, namely metals and ceramics. At the present time titanium and nickel alloys are used for the low and high temperature parts.



#### WHY NOT CERAMICS?

To see why ceramics, which appear to have the best high temperature properties, are not used in aero-engines it is helpful to consider other properties which are important.

Poor toughness is the main reason why ceramics have not been successfully introduced into the majority of engines, but research continues.

#### THE TRICKS METALLURGICAL ENGINEERS PLAY ...... ???

Metallurgical engineers have worked hard to increase the operating temperature of today's alloys:

• Turbine blades are 'grown' as single crystals, because these are more resistant to creep

(gradual changes in dimensions under stress and temperature);

- Current nickel super-alloys contain expensive alloying elements such as Hafnium and Rhenium in order to increase their high temperature performance;
- Turbine blades have little networks of holes to air-cool the blade surface





### **CERAMIC COATINGS - THE WAY AHEAD?**

While ceramics cannot yet be used for major components, Zirconia (ZrO) coatings are being used to increase the operating temperatures. These coatings can operate at much higher temperatures and protect the metal from chemical attack. Oxides (e.g. ZrO, Al2O3) are often used for high temperature coatings. Combined, the effects of air cooling and ceramic coatings mean that the combusting gases can reach temperatures over 1600°C - higher than any metal can work.

### EXERSISE

- Q1: What type of coatings used in Jet Engine? Specify the locations and justify your selection?
- Q2: What is Thermal barrier Coating (TBC)?
- Q3: Select materials for Different parts of the following Jet engine?



# **PRACTICAL 12**

### **OBJECTIVE:** Design and Selection of Materials for the Automotive Engine.

## **THEORY:**

### How an engine works:

The engine needs its input to generate power -- air and fuel. Now each engine has "cylinders" in which "pistons" slide through along with a complex layout of pipes that pump fuel in, valves that regulate the input and output of fuel/air and a way to transfer the reciprocating movement of the piston to a rotating motion. All of this is just about the Engine itself. For the automobile to run, this power generated by the engine has to be transferred to the wheels and a whole slew of mechanisms and systems will kick -- the clutch and the transmission systems will transmit this power to the differential (which rests on one of the axles) which ultimate transfers it to the wheels.

Coming back to the engine, the core of the engine is formed by the solid all metal (now-a-days, it's all aluminum) Engine Block. We have the pistons reciprocating within the cylinders with valves appropriately timed and regulating the flow of the air/fuel mixture; the connecting rods connecting the piston to the crankshaft which rotates a rather heavy flywheel. The kinetic energy obtained at the flywheel is regulated and transferred with the help of the clutch and the aiding transmission system to the two driving wheels (front or rear or both). I am aware that everything has been pounded and made to sound too simplistic, but that was to give you an idea as to how the engine works and how it fits into the overall scheme of things. As you unravel the mystery of the automobile, it starts to become clearer.





## Fig. Internal Combustion engine

#### Engine's Cylinder Block

The engine's cylinder block has always been made with grey cast iron or aluminum alloys. Now-a-days, most of the smaller car engines and the like are made using aluminum alloys while the larger ones -- for trucks, commercial vehicles and huge marine engines, still employ the cast iron types.

Grey cast iron is so called due to the evident grey appearance when fractured. Carbon is present within the interiors of the grey cast iron which gives it that color. The important point to note is that this carbon, present as flakes of graphite, gives the material great resistance to wear and tear and gives it better machinability. It also brings about better corrosion resistance.

Aluminium alloys have been gaining a lot of prominence due to the fact that their strength is almost on par with the heavier cast iron and has good casting properties to enable building the engine blocks with ease. They do have better corrosion properties have resistance the kind of temperatures experienced within the engine while it is functioning.

Aluminium has a high thermal conductivity and hence is able to dissipate heat quicker than cast iron. Also, it leads more thermal efficiency, cooler running engines and are lighter thereby improving the overall vehicle's operative characteristics.



Fig. Al Cylinder Block

The composition of the Grey Cast Iron is 3.5% carbon, 2.5% silicon and 0.65% manganese. Carbon (thanks to the graphite flakes, as mentioned earlier) also provides good lubrication characteristics, provides toughness and strength. The aluminum on the other hand has about 11% silicon, 0.5% manganese and 0.4% magnesium.

**Cylinders:** The cylinders are bored right into the cast iron or aluminum block and are finely honed to perfection (the insides). A well-designed and manufactured cylinder must be uniform across its diameter but can vary in its diameter by about 0.0005 in (0.012mm) -- that's the maximum room for mistake.

**Cylinder Sleeves:** These are commonly thought of as separate surfaces or liners for the pistons to come into contact with instead of the cylinder bores themselves. These can be made of a separate material like steel which can last longer and give the engine more life and allow for bringing down the costs of production.



Fig. Cylinder Sleeves

#### The Crankcase:

This is the part of the engine block that is separately bolted to the underside of the block, covering the crankshaft. The crankcase is used for holding the engine oil that is partly used for lubrication for the entire engine. The lower part of the crankcase is also called as oil pan and is made either with cast iron, aluminum or pressed steel.



Fig.The Crankcase

#### Cylinder head:

On top of the finely machined surface of the Cylinder block, there sits another cast iron or aluminum made —cylinder Headl -- this is the part of the engine that acts like a lid for the block; the cylinders and also provides space for the valves to be seated and the also the camshaft which opens and closes these valves (Overhead Camshaft). A cylinder head also comes complete with the lubrication holes drilled into it as an extension from the cylinder block. The cylinder head also contains tappets and other mechanisms.



Fig. Cylinder head

#### **Constructional materials for the Pistons:**

As mentioned above pistons do the dirty work of actually taking the brunt of the force of explosion arising of the combustion of the fuel and passes it onto the crankshaft (the big, heavy part of an engine that rotates due to the movement of the piston). It takes a tremendous amount of pressure (about 1000 Psi) notwithstanding the severe heat that it has to take. Now, when designing pistons, the weight is a serious determining factor. Imagine the scenario-on one hand you would need the pistons to be able to pick up all that heat and pressure, but on the other hand, you still want it light. Material sciences come to the rescue again with aluminum leading the pack for the choice with its favorable strength-to-weight ratio; the fact that it is easily machinable, has a great thermal conductivity (can transfer heat quickly) and most importantly, it is light weight, aluminum is the choice material for making pistons today.



## EXERSISE

Q1: Select materials with reason for different locations of following Car and its parts?

