<u>Practical Workbook</u>

MM-201: Physical Metallurgy

Name_________________________

Roll No_______________________

Batch_________________________

Year ________________________

Department of Metallurgical Engineering
NED University of Engineering and Technology,
Karachi-75270, Pakistan

Practical Workbook

MM-201: Physical Metallurgy

Prepared by

Dr. Waseem Khan & Prof. Dr. Ali Dad Chandio
(Assistant Professor and Chairman MYD)

This is to certify that this practical book contains _____38________ pages.

Approved by:

Chairman
MYD

Revised July 2024

Department of Metallurgical Engineering
NED University of Engineering and Technology,
Karachi-75270, Pakistan

<u>CERTIFICATE</u>

It is certified that Mr. / Ms. ____student of class _____Batch_____, bearing Roll No. MY _____ has completed his / her coursework in **MM-201: Physical Metallurgy** as prescribed and approved by the Board of Review of the Metallurgical Engineering Department.

His/her performance is reflected by the performance rubrics of his/her practical workbook.

The student's overall performance will address the assigned learning attribute.

____

Course Teacher

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Experiment No. 1

Aim of the Experiment: Operate metallographic machines **under supervision** to reveal the microstructure of carbon steel by standard metallography procedure.

Apparatus & Material:
Abrasive cut-off machine, mounting machine, grinding machine, polishing machine, metallurgical microscope, etchant and carbon steel specimen.

Theory:

Procedure:

1- Cutting the carbon steel specimen into appropriate size using abrasive cut-off machine.
2- Mounting the specimen in epoxy using hot mounting machine or cold mounting process.
3- Grinding specimen using 200, 400, 600, 800 and 1000 grit SiC paper followed by cloth polishing using 5 µm Al_2O_3 slurry.
4- Etch using proper chemical etchant.
5- Look at the specimen in the metallurgical microscope.

Observation:

Before Etching	**After Etching**
Magnification:	**Magnification:**
Specimen:	**Specimen:**
Chemical Composition:	**Chemical Composition:**
Microstructure Details:	**Microstructure Details:**
	Etchant:
	Etching Time:

Questions

1- The purpose of the abrasive cut-off machine is to

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2- What is the purpose of etchant?

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3- Differentiate low-carbon steel and high-carbon steel?

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F/OBEM 01/05/00

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

Psychomotor Domain Assessment Rubric-Level P3					
Skill Sets	Extent of Achievement				
	0	1	2	3	4
Equipment Identification Sensory skill to ***identify*** equipment and/or its component for a lab work.	Not able to identify the equipment.	--	--	--	Able to identify equipment as well as its components.
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Laboratory Session No. _____________ Date: _____________________

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

Experiment No. 2

Aim of the Experiment: Operate metallographic machines **under supervision** to reveal the microstructure of cast iron by standard metallography procedure.

Apparatus & Material:
Abrasive cut-off machine, mounting machine, grinding machine, polishing machine, metallurgical microscope, etchant and cast iron specimen.

Theory:

Procedure:

1- Cutting the cast iron specimen into appropriate size using abrasive cut-off machine.
2- Mounting the specimen in epoxy using hot mounting machine or cold mounting process.
3- Grinding specimen using 200, 400, 600, 800 and 1000 grit SiC paper followed by cloth polishing using 5 µm Al_2O_3 slurry.
4- Etch using proper chemical etchant.
5- Look at the specimen in metallurgical microscope.

Observation:

Before Etching	**After Etching**
Magnification: **Specimen:** **Chemical Composition:** **Microstructure Details:**	**Magnification:** **Specimen:** **Chemical Composition:** **Microstructure Details:** **Etchant:** **Etching Time:**

Questions

1- Name different type of cast iron

2-Compare Gray cast iron and SG cast iron?

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NED University of Engineering & Technology

Department of ____________________ Engineering

Course Code and Title: ____

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Remarks	
Instructor's Signature with Date:	

Experiment No. 3

Aim of the Experiment: Operate metallographic machines **under supervision** to reveal the microstructure of non-ferrous alloy by standard metallography procedure.

Apparatus & Material:
Abrasive cut-off machine, mounting machine, grinding machine, polishing machine, metallurgical microscope, etchant and non-ferrous alloy specimen.

Theory:

Procedure:

1- Cutting the given alloy specimen into appropriate size using abrasive cut-off machine.
2- Mounting the specimen in epoxy using hot mounting machine or cold mounting process.
3- Grinding specimen using 200, 400, 600, 800 and 1000 grit SiC paper followed by cloth polishing using 5 µm Al_2O_3 slurry.
4- Etch using proper chemical etchant.
5- Look at the specimen in the metallurgical microscope.

Observation:

Before Etching	**After Etching**
Magnification: **Specimen:** **Chemical Composition:** **Microstructure Details:**	**Magnification:** **Specimen:** **Chemical Composition:** **Microstructure Details:** **Etchant:** **Etching Time:**

Questions

1- Name different type of copper and aluminum alloy with their applications

2- Draw the unit cell of aluminum and copper.

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Remarks	
Instructor's Signature with Date:	

Experiment No. 4

Aim of the Experiment: Operate relevant instruments **under supervision** to reveal macroscopic features of the given specimen using the macro-etching method.

Apparatus & Material:
Grinding machine, polishing machine, etchant, tongs, cotton, gloves, goggles and specimen.

Theory:

Procedure:

1- Cutting the given specimen into appropriate size using abrasive cut-off machine.
2- Grinding specimen using 200, 400, 600, 800 and 1000 grit SiC paper followed by cloth polishing using 5 µm Al_2O_3 slurry.
3- Etch using proper etchant.
4- Look at the specimen in microscope at low magnification 20X or unaided eye.

Observation:

<u>Before Etching</u>	**<u>After Etching</u>**
Magnification: **Specimen:** **Chemical Composition:** **Microstructure Details:**	**Magnification:** **Specimen:** **Chemical Composition:** **Microstructure Details:** **Etchant:** **Etching Time:**

Questions

1- What is the difference between macro and micro examination?

2- What are the major types of etching?

3- What happens to the structure when etchant is applied?

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Instructor's Signature with Date:	

Experiment No. 5

Aim of the Experiment: Operate metallographic machines **under supervision** to reveal the microstructure of the given specimen using tint etching.

Apparatus & Material:
Abrasive cut-off machine, mounting machine, grinding machine, polishing machine, metallurgical microscope, tongs, cotton, gloves, goggles, specimen and tint etchant.

Theory:

Procedure:

1- Cutting the given specimen into appropriate size using abrasive cut-off machine.
2- Mounting the specimen in epoxy using hot mounting machine or cold mounting process.
3- Grinding specimen using 200, 400, 600, 800 and 1000 grit SiC paper followed by cloth polishing using 5 µm Al_2O_3 slurry.
4- Etch using proper tint etchant.
5- Look at the specimen in the metallurgical microscope.

Observation:

Before Tint Etching	**After Tint Etching**
Magnification: **Specimen:** **Chemical Composition:** **Microstructure Details:**	**Magnification:** **Specimen:** **Chemical Composition:** **Microstructure Details:** **Tint Etchant:** **Tint Etching Time:**

Questions

1- What is the tint etchant for stainless steel, mild steel and brass?

2- What happens to the structure when tint etchant is applied?

F/OBEM 01/05/00

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Experiment No. 6

Aim of the Experiment: Manipulate the cooling curves of the Pb-Sn alloy using the thermocouple.

Apparatus & Material:
Melting furnace, thermocouple, crucible, tongs, cotton, gloves, goggles and Pb-Sn alloy (common solder).

Theory:
In this experiment, you will measure the cooling curve of Pb-Sn as a part of your study on determination of phase diagram of a simple eutectic alloy system. $Pb_{38.1}Sn_{61.9}$ alloy sample (solder alloy) is heated to about 200^0C to obtain fully molten alloy. The temperature of the sample is monitored using a thermocouple during cooling of the alloy to room temperature. The temperature of the sample is plotted as function of time. The sample temperature drops continuously as function until it reaches 183^0C. At 183^0C, the sample undergoes solidification by forming eutectic phase mixture from liquid, $L \rightarrow \alpha+\beta$ (refer to Pb-Sn binary phase diagram in Figure 6.1). The sample temperature remains constant until the whole liquid solidifies and then the sample temperature is dropped again. In real situation, cooling curve looks a bit different from the above scenario. In fact, solidification does not begin at 183^0C, because nucleation of solid phases from the liquid requires undercooling (drop in temperature) to provide sufficient driving force (change in volume free energy, G). Therefore, the temperature of the sample drops slightly below 183^0C before the solidification begins. As soon as solidification begins, latent heat is released into the sample. This leads to heating up of the sample and therefore, sample temperature increases a bit from the temperature at which solidification begins. Thereafter, the temperature remains constant until whole liquid solidified. Then sample temperature drops again. By measuring the solidification temperature of the sample, you can measure the solidus temperature of the eutectic alloy and this data can then be used to construct the phase diagram. By doing such experiments using alloys of different compositions, we can obtain the phase diagram.

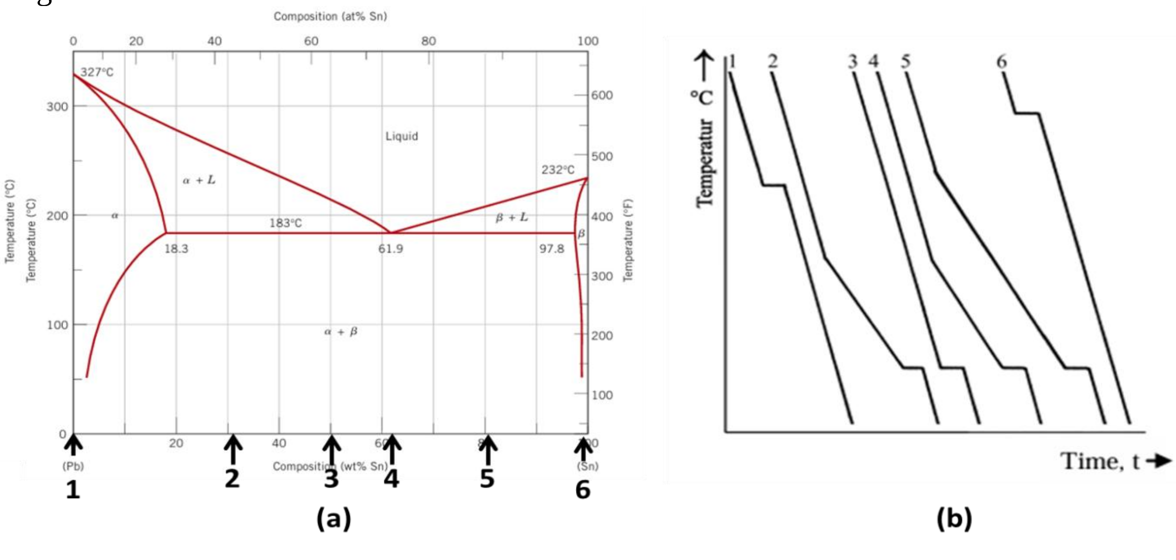

Figure 6.1: (a) Pb-Sn phase diagram and **(b)** Typical cooling curves for the alloys shown in (a)

Figure 6.1 indicates how the phase alloy diagram can be constructed using a set of cooling curves obtained for different alloys compositions ranging from 0wt.% Sn to 100wt.% Sn in Pb-Sn alloys. It is evident that these simple experiments can provide us insight into the alloy phase diagram! For the purpose of the experiment, we shall use a thermocouple of K-type. The thermocouple is a widely used type of temperature sensor. The basic principle of thermocouples is based on the fact that when two dissimilar metals are joined at two ends, a predictable voltage will be generated between the two ends if two ends are kept at different temperatures. The voltage can be related to the difference in temperature between the measuring junction and the reference junction (connected to the measuring device). The selection of the optimum thermocouple type (alloys used in their construction) is based on application temperature, atmosphere, and required length of service, accuracy and cost. In this experiment, you will use a K type Chromel(+)/ Alumel(-) thermocouple, which is suitable for measurement of temperature in the range of -200^{0}C to 1200^{0}C in air with precision of $\pm$ 15K.

Procedure:

The experimental steps as follows:

1. Heat the alloy inside a furnace to 200^{0}C at a predetermined rate (10K/min.) and hold liquid for 10 minutes.
2. Place a thermocouple (Chromel-Alumel K-type) to determine the temperature using voltage. Cool the liquid alloy at a determined rate (10K/min.).
3. Record the temperature of the sample during heating and cooling as function of time.
4. Plot the change the temperature of the sample (T) with time (t).

Observation:

Questions

1- Regardless of temperature, what is the maximum amount of tin that can be dissolved in lead (α phase) and what is the maximum amount of lead that can be dissolved in tin (β Phase)?

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2- Cite the phases that are present and the composition of each phase:

i. Co = 10wt.%Sn, 90wt.%Pb, 200^0C.

ii. Co = 30wt.%Sn, 70wt.%Pb, 180^0C.

iii. Co = 90wt.%Sn, 10wt.%Pb, 180^0C.

iv. Co = 62wt.%Sn, 38wt.%Pb, 190^0C.

Experiment No. 7

Aim of the Experiment: Manipulate the cooling curves in order to construct phase diagram of the Pb-Sn alloy.

Apparatus & Material:
Melting furnace, thermocouple, crucible, tongs, cotton, gloves, goggles and Pb-Sn alloy.

Theory:
In the last experiment, you have measured the cooling curve of a Pb-Sn eutectic alloy. In this experiment, you will measure the cooling curve of Pb-Sn hypo-eutectic and hyper-eutectic alloy as a part of your study on determination of phase diagram of a simple eutectic alloy system. Hypo-eutectic and hyper-eutectic (as shown in Figure 6.1) alloy sample is heated to about 300^0C to obtain fully molten alloy. The temperature of the sample is monitored using a themocouple during cooling of the alloy to room temperature. The temperature of the sample is plotted as function of time. The sample temperature drops continuously as function until it reaches to about 250^0C, at which solidification of Pb-rich α-phase begins for hypoeutectic alloy and to about 200^0C, at which solidification of Sn-rich β-phase begins for hypereutectic alloy. A careful look at the phase diagram will tell you that α-phase and β-phase do not have a fixed solidification temperature. Rather solidification of the α-phase and β-phase begins at temperature of ~250^0C and ~200^0C and ends at solidus temperature (183^0C). Therefore, at 250^0C the primary α-phase starts forming, the slope of the cooling curve changes. The new cooling curve progresses further until the temperature reaches 183^0C or eutectic temperature. At 183^0C, the sample undergoes solidification by forming eutectic phase mixture from liquid, L → α +β (Refer to Pb-Sn binary phase diagram in Figure 6.1). The sample temperature remains constant until the whole liquid solidifies and then the sample temperature is dropped again. The resulting cooling curve shows the two stages of solidification with a section of reduced gradient where a single phase is solidifying and a plateau where eutectic is solidifying. By taking a series of cooling curves for the same system over a range of compositions the liquidus and solidus temperatures for each composition can be determined allowing the solidus and liquidus to be mapped to determine the phase diagram.

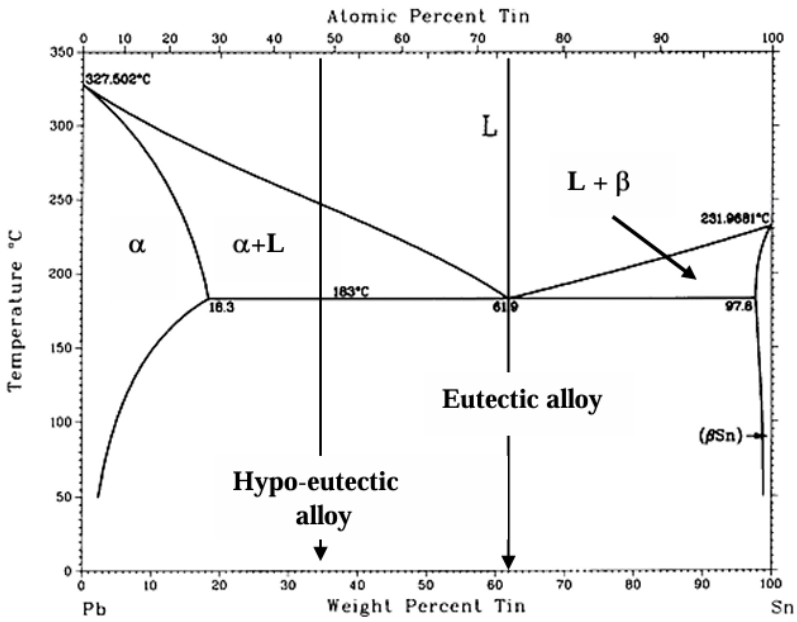
Procedure:
1. Heat the alloy inside a furnace to 300^0C at a predetermined rate (10K/min.) and hold the liquid for 10 minutes.
2. Place a thermocouple (Chromel-Alumel K-type) to determine the temperature using voltage. Cool the liquid alloy at a determined rate (10K/min.).
3. Record the temperature of the sample during heating and cooling as function of time.
4. Plot the change the temperature of the sample (T) with time (t).

Observation:

Questions

1- A lead–tin alloy of composition 20wt% Sn–80wt% Pb is slowly heated from a temperature of 150 ^{O}C.

(a) At what temperature does the first liquid phase form?
(b) What is the composition of this liquid phase?
(c) At what temperature does complete melting of the alloy occur?
(d) What is the composition of the last solid remaining prior to complete melting?



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1- What are the common applications of lead-tin alloy?

Experiment No. 8

Aim of the Experiment: Operate metallographic machines **under supervision** to reveal the microstructures of as cooled Pb-Sn alloy specimens by standard metallography procedure.

Apparatus & Material:
Abrasive cut-off machine, mounting machine, grinding machine, polishing machine, metallurgical microscope, etchant and Pb-Sn alloy specimens.

Theory:
In this experiment, standard metallography practice will be used to reveal the microstructures of as cooled Pb-Sn alloy specimens obtained in experiment # 6 and 7. The phase diagram of lead-tin binary eutectic alloy is represented below in Figure 8.1. The lead-tin alloy phase diagram shows different phases at different temperature and composition.

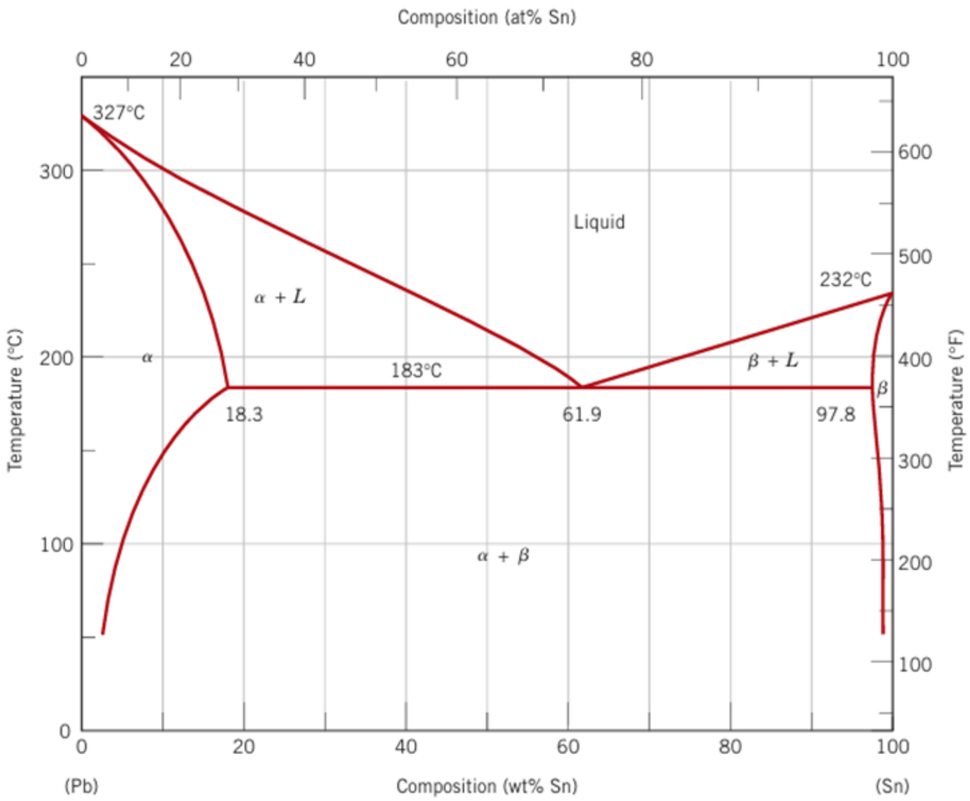

Figure 8.1: Lead-Tin alloy phase diagram.

Procedure:

1- Cutting the Pb-Sn alloy specimen into appropriate size using abrasive cut-off machine.
2- Mounting the specimen in epoxy using hot mounting machine or cold mounting process.
3- Grinding specimen using 200, 400, 600, 800 and 1000 grit SiC paper followed by cloth polishing using 5 µm Al_2O_3 slurry.
4- Etch using proper chemical etchant.
5- Look at the specimen in metallurgical microscope.

Observation:

Questions

1- If an alloy composed of 30wt% Sn and 70 wt% Pb is slowly cooled from liquid to 185 ^oC, what phases would be present, what is their composition and how much of each phase will be present?

2- Sketch the appearance of the microstructure for question # 1 labeling and describing the various constituents?

Experiment No. 9

Aim of the Experiment: Manipulate grain size of given specimen by Hyen intercept method.

Apparatus & Material:
Photomicrograph, ruler, pencil and specimen.

Theory:

Procedure:
A number of the prepared metallographic specimens having different grain sizes shall be provided to the students. The microstructure of the specimens shall be projected on the projection screen of the microscope at a magnification of 100X. The grain size as viewed on the projection screen of the microscope shall be compared with ASTM standard grain size charts and reported as the ASTM grain size number.

ASTM Comparative Method.

The ASTM standard grain size chart are indexed from 00 to 10, each index number representing some mean number of grains per square inch at a magnification of 100x according to the following relation.

$$\mathbf{N = 2n-1}$$

Where,
N= mean number of grains per square inch at 100X.
n = ASTM grain size number of index.

The method is essentially one of the comparisons in which the image of the microstructure projected at a magnification of 100X, or a photograph of the structure at the same magnification is compared with the graded standard grain size charts. By trial and error method a match is secured and the number corresponding to the index number graded in similar manner and it is customary in such cases to report grain size in terms of numbers.

ASTM grain size index

Reading No	Length of line (mm)	Number of grains cut
1	50	
2	50	
3	50	
4	50	
5	50	
6	50	
7	50	
8	50	
9	50	
10	50	

Total length of line = 50 X 10 = 500 mm

Total number of grains cut ____________

Average grain size = 500/ Total number of grain cut

Actual grain size = average grain size/100= ________________

Result:

Experiment No. 10

Aim of the Experiment: Manipulate grain size and phase analysis of given specimen by Image J software.

Tool & Material:
Mounting machine, grinding machine, polishing machine, etchant, metallurgical microscope, Image J software and specimen.

Theory:

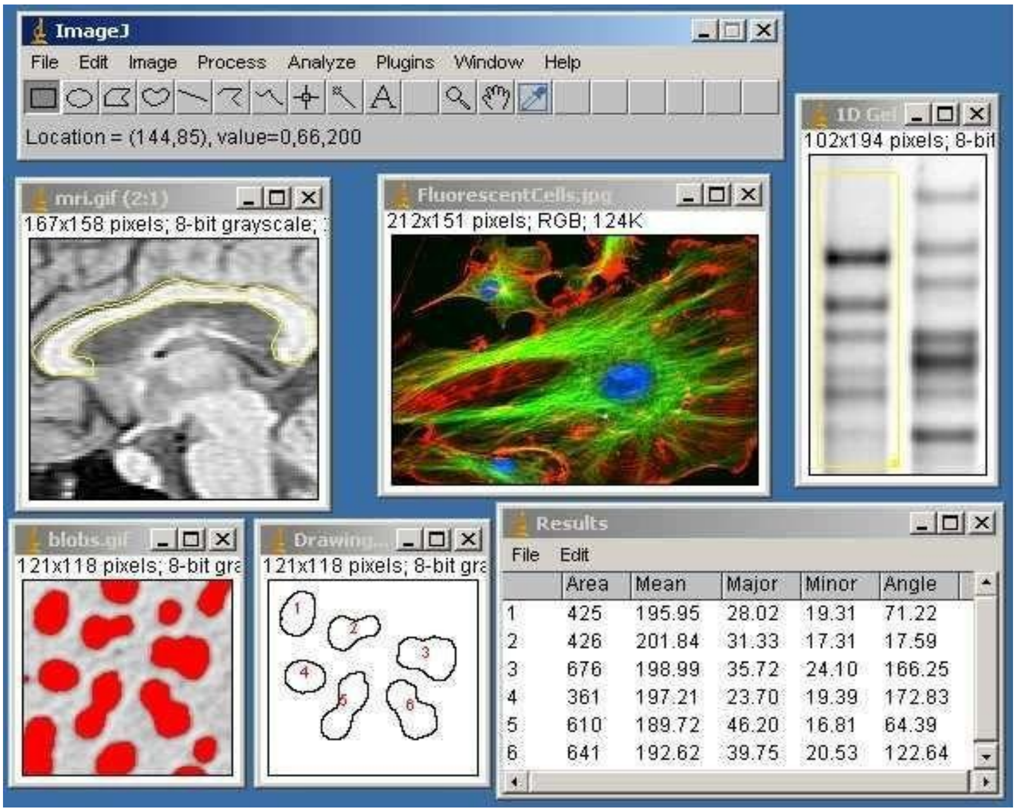

Figure 10.1: Image J Software.

Procedure:

1- Cutting the given specimen into appropriate size using abrasive cut-off machine.
2- Mounting the specimen in epoxy using hot mounting machine or cold mounting technique.
3- Grinding specimen using 200, 400, 600, 800 and 1000 grit SiC paper followed by cloth polishing using 5 µm Al_2O_3 slurry.
4- Etch using proper chemical etchant.
5- Look at the specimen in metallurgical microscope.
6- Capture the micrograph and import the micrograph in Image J software.
7- Use Image J software tools to calculate grain size and phase.

Observation:

Questions:

1- What is meant by phase?

2- Highlight the uses of Image J software.