EXPERIMENT NO 10

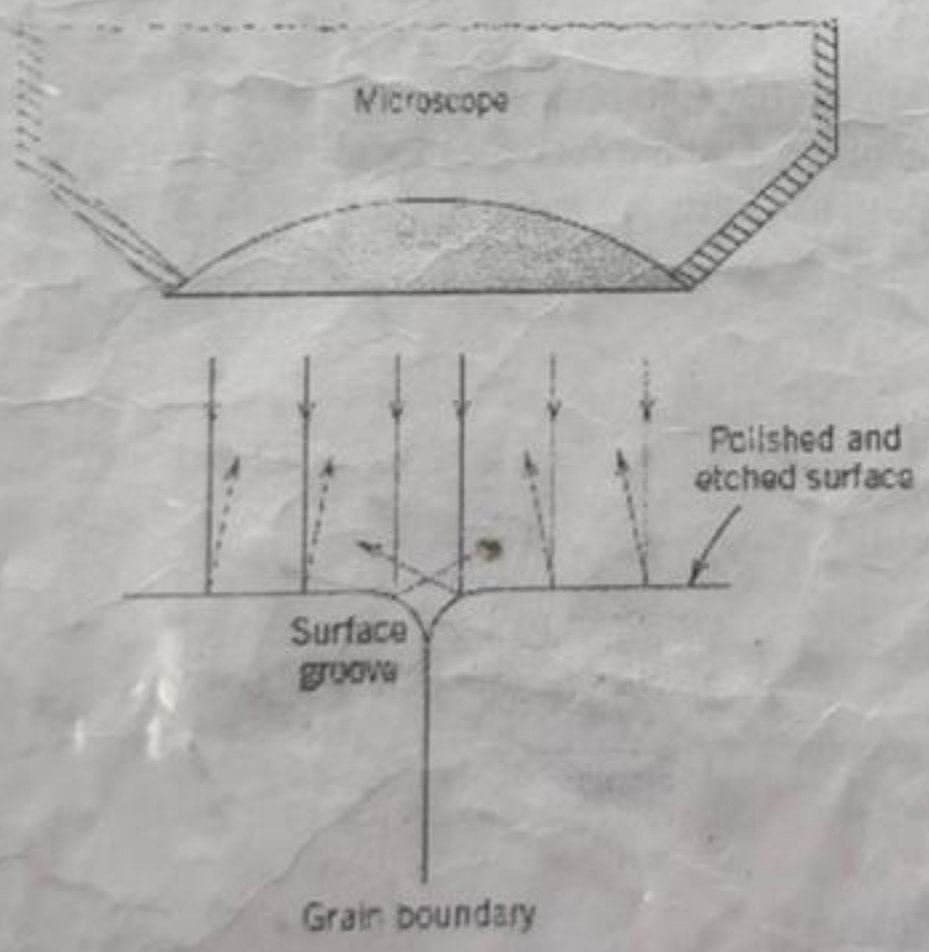
OBJECTIVE: TO PREPARE TWO METALLIC SPECIMENS FOR METALLOGRAPHIC EXAMINATIONS AND MEASURE THEIR GRAIN SIZE.

REQUIREMENTS: Sample, grinder, emery paper of grit vary from 100 to 800, alumina powder and diamond paste, ethyl alcohol, distilled water, dryer, mounting machine, metallurgical /optical microscope, etc.

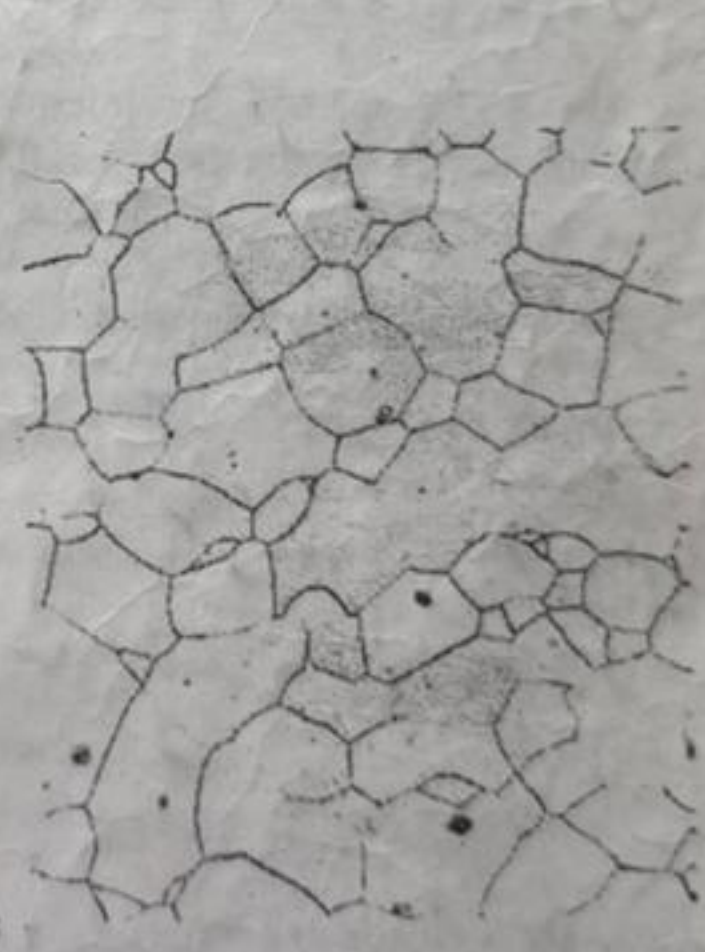
THEORY:

For polycrystalline materials many properties depend on grain size .The micro structural study features observed through microscopes can provide information regarding grains, their morphology and distribution and phases in the material. Some of the technique which have developed for observing the features are optical microscopy, electron microscopy, field-ion microscopy, field emission microscopy, X-ray microscopy, and electro-probe microanalysis. Of these, optical microscopy is by far the most important, since the equipment is relatively inexpensive and easy to handle.

In order to observe the structure in optical microscope, the specimen surface is polished and subsequently etched with appropriate reagents before microscopic examination. In a polished specimen, the etching not only delineates grain boundaries, but also allows the different phases to be distinguished by difference in brightness of grains. Differences in contrast may result from differences in light absorption characteristics of the different phases.



a)



b)

Figure:

a) Section of a grain boundary and its surface groove produced by etching; the light reflection characteristics in the vicinity of the groove are also shown.

b) Photomicrograph of the surface of a polished and etched polycrystalline specimen in which the grain boundaries appear dark.

Etching results in preferential attack or preferential staining of the surface. The

preferential attack is electrochemical corrosion. Grain boundaries are often anodic to the bulk metal in the interior of the grain and so are etched away preferentially and delineated staining is produced by deposition of solid enchant product on the specimen surface. This is formed by chemical reaction between enchant and the specimen. Under the favorable conditions the use of proper enchant enables the identification of constituents. Micro structural examination can provide quantitative information about the following parameters:

- The grain size of specimens
- The amount of interfacial area per unit volume
- The dimensions of constituent phases

For grain size measurement, the grains along a line, circle, or within a known area are counted. From the study of actual grain shapes it has been found that, while grains do not actually posses a regular or idealized forms, the truncated octahedron is a reasonable approximation for equaxed grains. From the analysis of the distributions of sections in space occupied by truncated octahedral, the ASTM system gives the following relationship:

$$n = 2^{N-1}$$

Where, 'n' is the number of grains per square inch at 100X and
'N' is the ASTM grain index number

In the ASTM method, the microstructure or the photomicrograph of a given polycrystalline specimen at a magnification of 100X is compared with a set of standard micrographs or idealized polygons as can be seen in the microscope. The charts are indexed for varying from 1 to 8.

Formula for grain size determination:

At 100X

$$\text{area of one grain} = \frac{1\ \text{inch}^2}{n} = \frac{(2.5)^2\ \text{cm}^2}{n}$$

$$\text{Radius of one grain} = \sqrt{\frac{\text{Area}}{\Pi}}$$

So, **Actual Grain size** = (Diameter at 100X)/100

EXPERIMENTAL PROCEDURE:

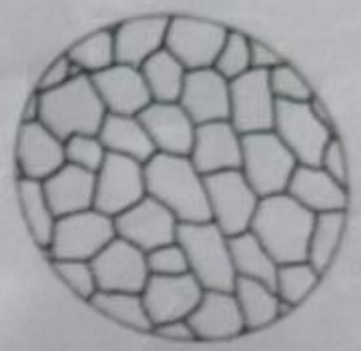
Specimen preparation:
Grinding: An unprepared specimen may have sharp edges and many scratches. Any burn on the edges of the specimen should be removed by means of a coarse grinding paper or a file. In order

Grain size standard diagram for steel austenite (100x)

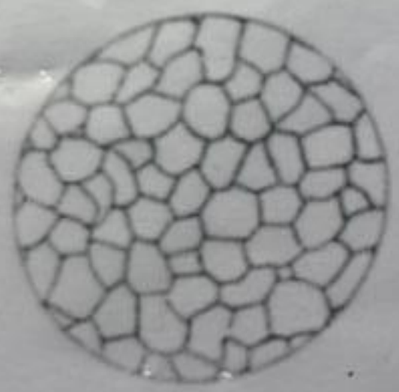
Grain size number 1

Grain size number 2

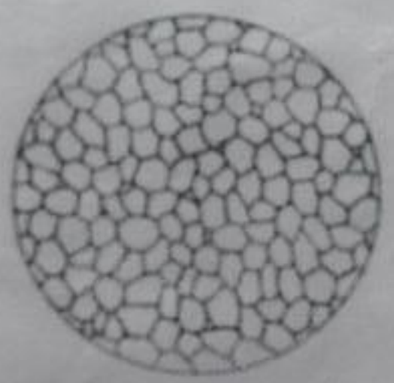
Grain size number 3



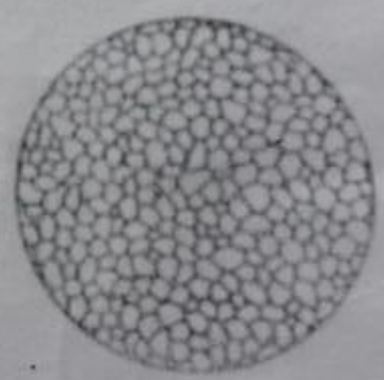
Grain size number 4



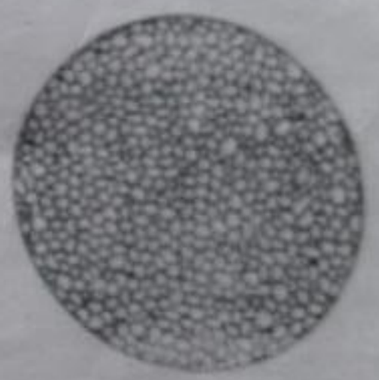
Grain size number 5



Grain size number 6



Grain size number 7



Grain size number 8

to remove scratches, the specimen is rubbed on emery papers. Initially a coarse grade paper is used for this purpose. The paper is placed on a smooth hard surface such as a glass sheet and then specimen is gently rubbed backward and forward against it until only scratches due to the particular paper can seen to cover the surface. Then the specimen is ground on the next finer emery paper in such a direction that the new set of parallel scratches are at right angle to the previous set, so that the removal of previous grinding marks is easily observed. This procedure is repeated till the finest emery paper has been used.

Polishing: The specimen is polished by holding it against a horizontal rotating disk. The disk is covered with a velvet cloth which has very fine particle such as alumina (Al_2O_3) or Fe_2O_3 (600 nm – 1 μm size) embedded in it. During polishing the velvet cloth is supplied with these particles in the form of an aqueous suspension at regular intervals. These abrasive particles rub against the specimen and produce a very smooth surface. During this process the surface must attain such a good polish as to resemble a mirror.

Etching: Before etching, the specimen should be thoroughly washed with water and dried with alcohol. The specimen, with its polished side upwards should be immersed in the etching solution contained in a small porcelain dish. The specimen surface should be examined from time to time and the specimen is removed from the enchant when grain structure is visible to the unaided eye. After etching, the specimen is thoroughly washed with water and then dried with alcohol.

Microstructural Examination: Before placing he specimen on the microscope stage, fix the specimen on a glass slide with plasticine and level the etched surface with the help of leveling device

OBSERVATIONS:

i) Calculate the number of grains per square inch of each specimen.
ii) Measure the grain size of both specimens and report it.