

EXPERIMENT

(A) MICROSTRUCTURE OF METALS AND ALLOYS (B) QUANTITATIVE METALLOGRAPHY

THEORY: When two or more elements combine in such a way that the elements are mixed on an atomic scale, the product is called an alloy or solid solution. Generally a solid solution consists of a single phase. The element with the highest quantity in the solution is called the solvent and the rest of the elements are known as solutes. Unlike pure metals, alloys generally freeze over a range of temperature. During solidification of an alloy, this feature introduces compositional inhomogeneity, which is reflected in the microstructure.

Columnar Grains : Macrostructure of Pure Zn

When a melt is poured into a container which is at a much lower temperature than the melt, large undercooling occurs in the regions where the melt comes into contact with the container walls. The liquid is cooled below its freezing temperature and the nucleation of the solid phase occurs adjacent to the walls.

The formation of these solid crystals releases the latent heat of fusion and the degree of supercooling is reduced in the liquid adjacent to these crystals. Consequently no fresh nuclei form in the center of the mold and the growth of the crystals formed near the mold wall continues inwards. The direction of growth of the crystals is opposite to the direction of the heat flow from the center of the ingot to the container walls.

This results in long columnar crystals perpendicular to the walls. Sidewise growth is inhibited by neighboring grains, so the growing grains become columnar in shape. Fig 8.10. The grains in the columnar zone have different orientations from each other because they are differently oriented in the plane normal to the heat direction. Later, grains may begin to form within the interior of the melt, and grow approximately equally in all directions. Such grains are called equiaxed.

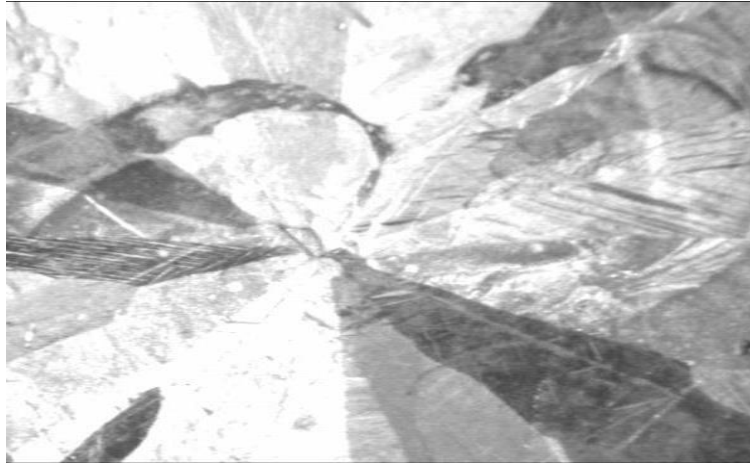


Fig 8.10 Long Columnar grains of Pure Zinc

Dendritic Growth: Microstructure of Cu-Ni

The initial stage of solidification of an alloy is similar to that for a pure metal. During the period of columnar growth, however, the solid phase develops protuberances. This is because the solute atoms are rejected during the formation of the solid phase and the liquid in the immediate vicinity of the solid- liquid interface is enriched with solute.

This liquid has a lower freezing temperature. Since the interface can be assumed to be at equilibrium and the actual temperature gradients quite small, the liquid ahead of the interface is supercooled and the supercooling increases with the distance from the interface. As a result, any solid protuberance ahead of the plane interface grows more rapidly than the plane interface since the liquid solidifies more readily with increased supercooling.

The resulting branched crystal often has the appearance of a miniature pine tree and is accordingly called a **dendrite** after the Greek word dendrites meaning “of a tree”. The direction in which these spikes grow is crystallographic and is known as the dendritic growth direction. The direction of dendritic growth depends on the crystal structure of a metal. (Fig8.1). The phase diagram of Cu-Ni is shown in Fig.8.2.

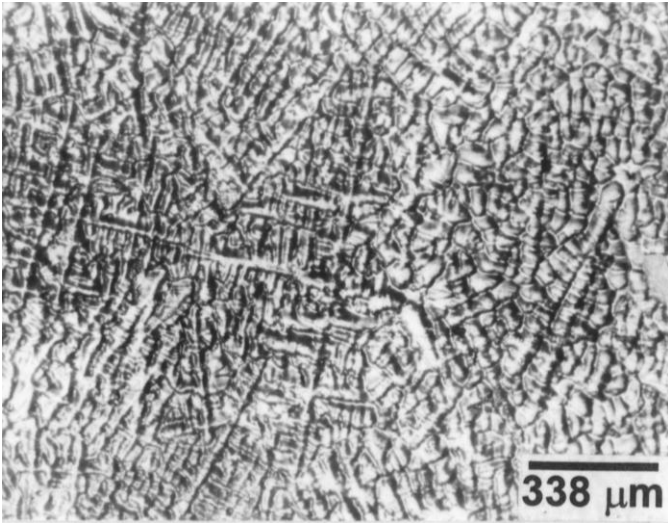


Fig.8.1 Dendritic microstructure

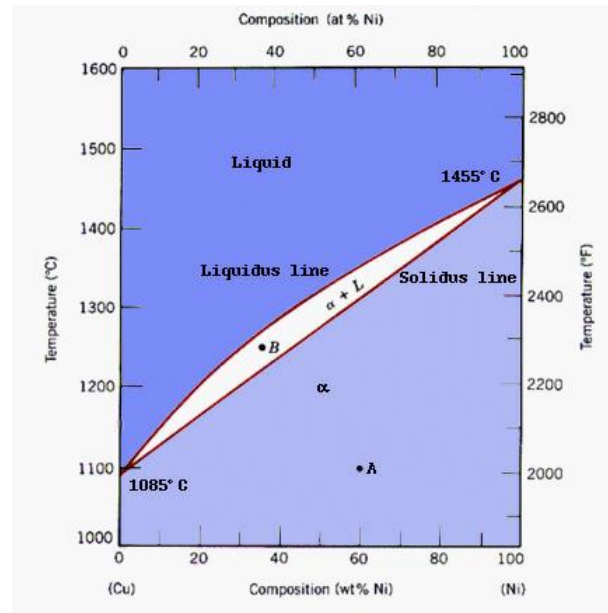


Fig.8.2 Cu - Ni Phase diagram

The region above liquidus line is liquid and below solidus line is solid and the intermediate region is called the mushy zone where liquid and solid co-exist. From phase diagram it is obvious that as the composition of solute nickel increases the freezing temperature is increasing and at temperature higher than 1455⁰C (M.P. of Ni) it is totally liquid for any composition of nickel. At temperature below 1085⁰C (M.P.Cu) the alloy is totally solid and only a single phase is obtained at room temperature for any composition of nickel in copper. Copper and nickel form in the solid state a continuous series of fcc mixing crystals. The strength of copper-nickel alloys rises with the Ni content; at about 60% Ni the maximal value is obtained at a still good toughness; the breaking stress there is more than 40%. Much used Cu-Ni alloys are: Cupronickel, Cu with 10-30% Ni and about 1%Fe.

Two phase structure:

(i) Eutectic structure: Microstructure of Cu-P

In eutectic structure reaction a liquid phase transforms into a mixture of two solid phases with different compositions and crystal structures.



on cooling, the melt with composition C in Fig 8.5 forms a two phase microstructure with crystals of α and β . The initial formation of a crystal causes the surrounding liquid to be super saturated in β with the result that the condition are favourable form the nucleation and growth of a β crystal next to the α crystal already formed. The Microstructure of Cu/8.4 wt % P alloy shows the grains composed of colonies of fine eutectic structure A lamellar pattern consisting of crystals of copper rich solid solution (dark) and copper phosphide (Cu_3P) (white) is observed. (Fig. 8.4)

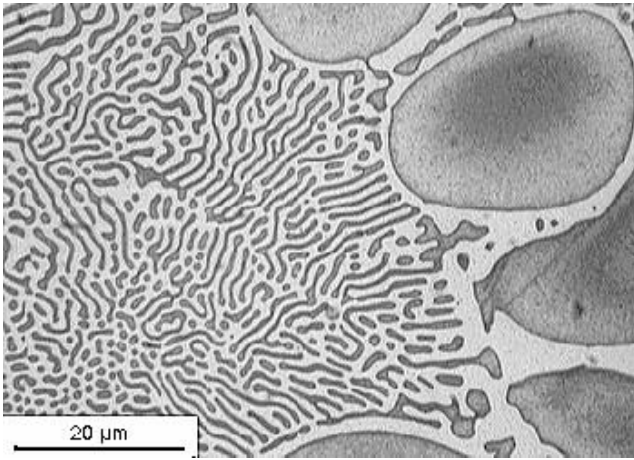


Fig 8.4 Microstructure of Cu-4.5wt%P

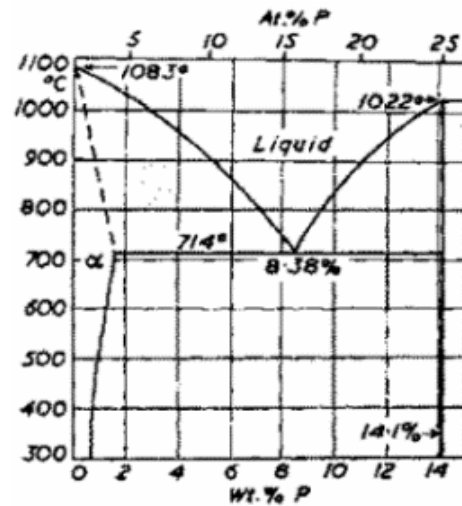


Fig 8.5 Cu-P Phase diagram

(ii) Peritectic Structure: Microstructure of Cu-Zn

In peritectic reaction a solid and a liquid phase react to produce another solid phase.



If the melt composition C in is cooled below temperature T_p , the product phase β starts to form at the interface between α and liquid. The growth of this phase is dependent on the diffusion of atoms on one side of β to the other side. Since diffusion is slow in solid state, equilibrium microstructures are rarely obtained in peritectic systems. The microstructure shows crystals of copper rich solid solution (light etching), partly surrounded by β crystals (dark) are observed. β crystals have formed by peritectic reaction Fig 8.6. The phase diagram of Cu-Zn is shown in Fig.8.7.



Fig 8.6 Microstructure of Cu-Zn

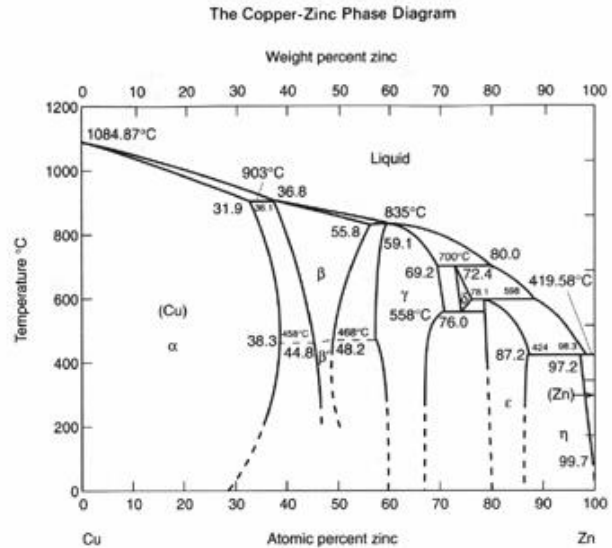


Fig 8.7 Phase diagram of Cu-Zn

Polycrystalline Structure: Microstructure of Cu

Most metals are commonly found in polycrystalline form. That is, the material is composed of many small regions, called grains that have different orientations of the crystal structure. Between the grains are grain boundaries, which are regions of extreme disorder in the crystal structure. Due to this disorder acids and other etchants typically attack (react with) the grain boundary material first. This provides a method for making the grain boundaries visible in an optical microscope. The image below (Fig 8.8) shows some typical grains in the polycrystalline copper discussed here. The strength of a polycrystalline metal increase as the grain size decreases; this is known as the Hall-Petch effect, after the scientists who first studied it.

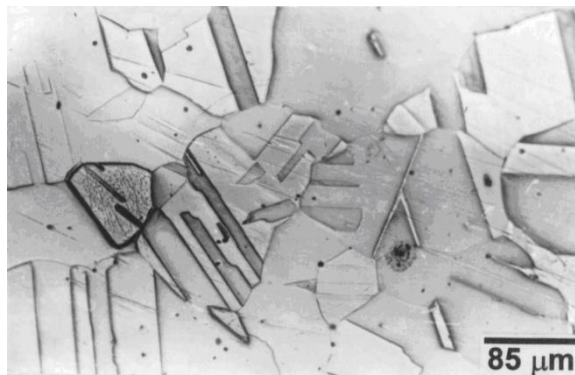


Fig 8.8 Microstructure of Copper

(a) Quantitative Metallography : Measuring Volume Fraction

Volume Fraction, Areal Analysis

1. Trace the structure onto paper, weight the paper, cut out the parts containing the phase of interest and weigh these parts. The weight fraction equals the area fraction of phase in this section. The average of a number of area fraction measurements gives one the volume fraction of phase.
2. Etch the specimen to color each phase differently. Use a computer to measure the area of each phase (color). The average of number area fraction measurements is equal to the volume fractions of phases.

Volume Fraction, Lineal Analysis

Using lines uniformly distributed but randomly oriented, the average length of the line segments crossing the selected phase is equal to the area fraction of the phase viewed in the planar section. The average of number area fraction measurements is equal to the volume fractions of phases.

Volume Fraction, Point Count

The fraction of a number of points which fall on the selected phase is equal to the area fraction of that phase. The average of a number of area fraction measurements is equal to the volume fractions of phases.

RECOMMENDED METHOD

Measurement of Volume Fraction of Phases Using the Systematic Point Count Method

This method (Fig 8.9) is based on the fact that the average fraction of points which fall on grains of a particular phase is equal to the area fraction of the phase in the polished section and that the average of these area fractions is equal to the volume fraction of the phase. This method entails selecting a suitable array of points, overlaying it on the specimen and counting the points which fall on the selected phase, selecting a new area and counting again, and again until the desired error is obtained.

The procedure is as follows:

- The most efficient method will be that which requires the least effort per observation.
- Using a reticule grid and observing the polished sample is more efficient than using a micrograph.
- A square array of points is simple, symmetric and easy to obtain.
- The optimum number of points in the grid large to minimize the effort of changing views (or micrographs) and minimum to reduce the effort of counting. .

- Select an area on the specimen where counts are to be made. This selection should be made at random or per a predefined pattern so as to minimize bias.
- Count each point which falls on the selected phase.
- Assign a count value of $\frac{1}{2}$ to points which fall on inter phase boundaries.
- Tabulate the Results
- Compute the mean
- Divide these by the number of points in the grid and multiply them by 100. This gives the results in terms of the volume percent of phase.

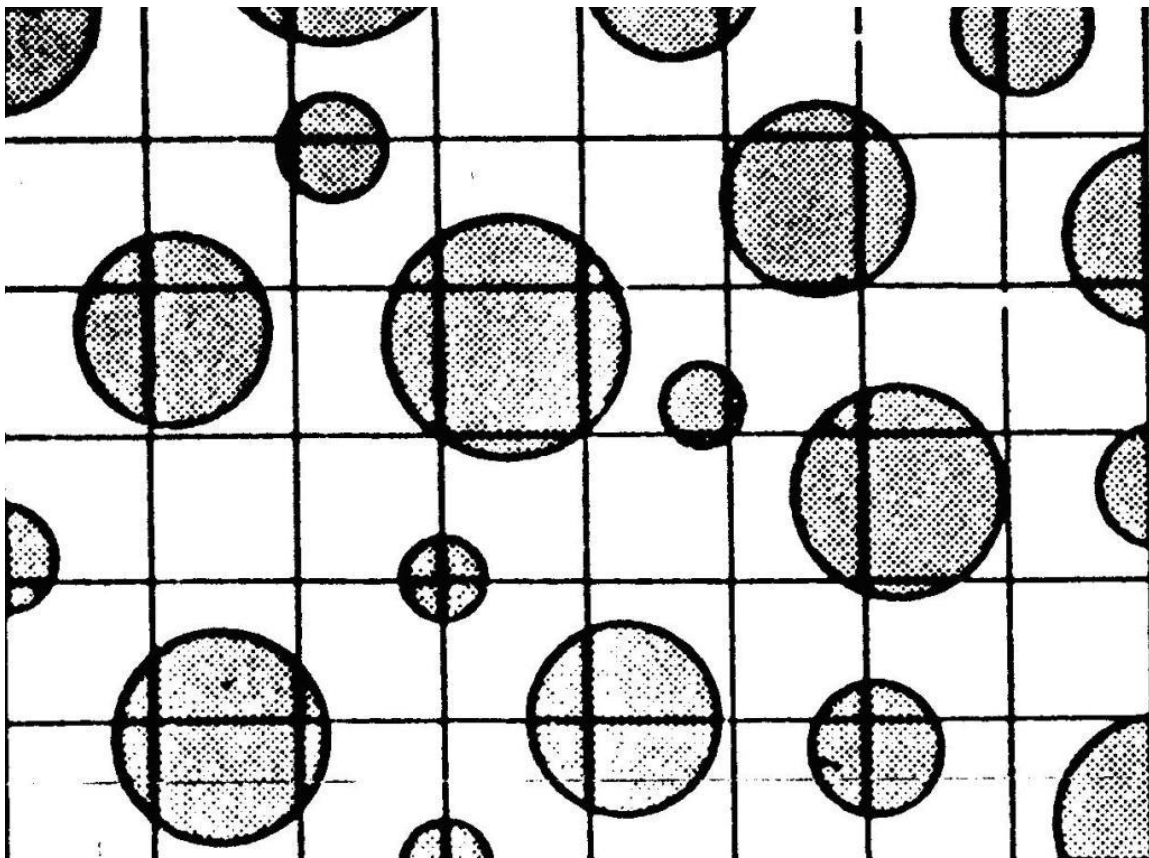


Fig 8.9. Point count method