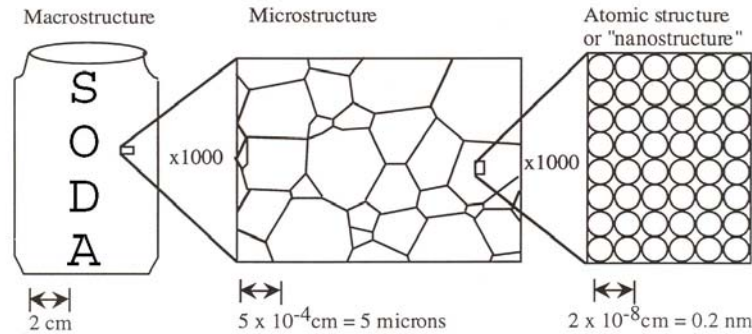


Microstructure is a term that is used broadly within materials science and engineering to describe the structural features within a solid that are larger than atoms, but smaller than the external dimensions of a solid. Loosely, we can define three levels of structural order: the macrostructure is the external form of the solid or components within the solid that are visible to the naked eye and easily manipulated by hand. The length scale for the macrostructure of a material is usually in the centimeter range (10^{-2} m) or larger. The atomic structure refers to the arrangement of the individual atoms in the solid and the length scale here is in the nanometer range (10^{-9} m). What is in between is the microstructure – it refers to features such as defects and grain boundaries that occur at an intermediate length scale and are usually measured in micrometers, or for short, microns (10^{-6} m). This is the length scale that most closely relates to the processing and properties of a solid, so microstructural features are of great interest to engineers.

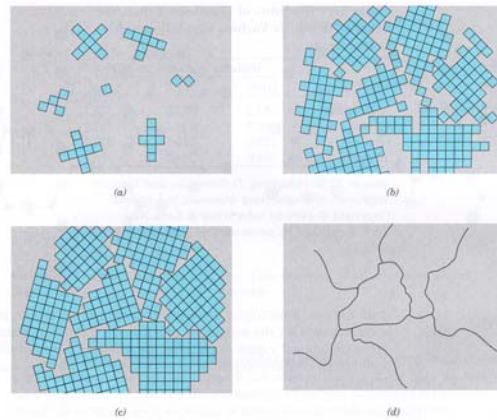


I. Grain Size

Most materials are composed of millions of individual crystals, or grains, which nucleate during the formation of a solid and grow independently of one another. Within each individual grain there is a systematic packing of atoms. However, each grain has a different orientation, so as the grains grow from the liquid, they will meet. And the region where grains meet in a solid is called the grain boundary. There is a disturbance in the atomic packing. When a material is polished to a mirror-like finish and subsequently immersed in an acidic solution (this is called etching, which is something we will do in a later lab), the grain boundary regions are preferentially attacked. When viewed under a light microscope, the grain boundaries reflect light at different angle from the grain interiors, and thus appear dark in contrast.

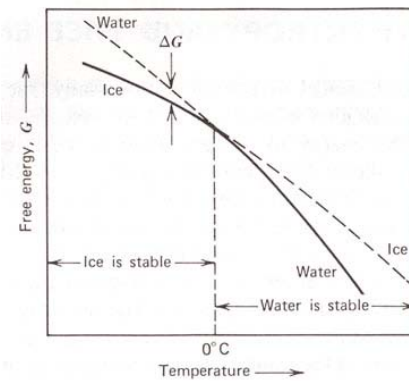
The schematic diagrams below show the various stages in the solidification of a polycrystalline material; the square grids depict unit cells. (a) Small crystals form or nucleate. (b) Growth of the crystals; the obstruction of some grains that are adjacent to one another is also shown. (c) Upon completion of solidification, grains having irregular shapes have formed. (d) The grain structure as it would appear under the microscope, dark lines are the grain boundaries.

The term polycrystalline refers to any material which is composed of many individual grains. Some materials are actually used in their single crystal state: large silicon crystal ingots are grown and sliced into thin wafers for integrated circuits. Nickel alloys are grown for aircraft engine turbine blades. However, most engineering materials are polycrystalline in nature, and control of grain size is an important concern in a wide array of processing operations, ranging from the sintering of ceramics to the rolling of steel. One example of a material property that is dependent upon grain size is the strength of a material: it might make logical sense to you that as grain size is reduced, the material becomes stronger (there are more grains and more grain boundaries to travel). Note that strength is expressed in units of stress – a more detail definition will follow.



II. Dendrite Arm Spacings

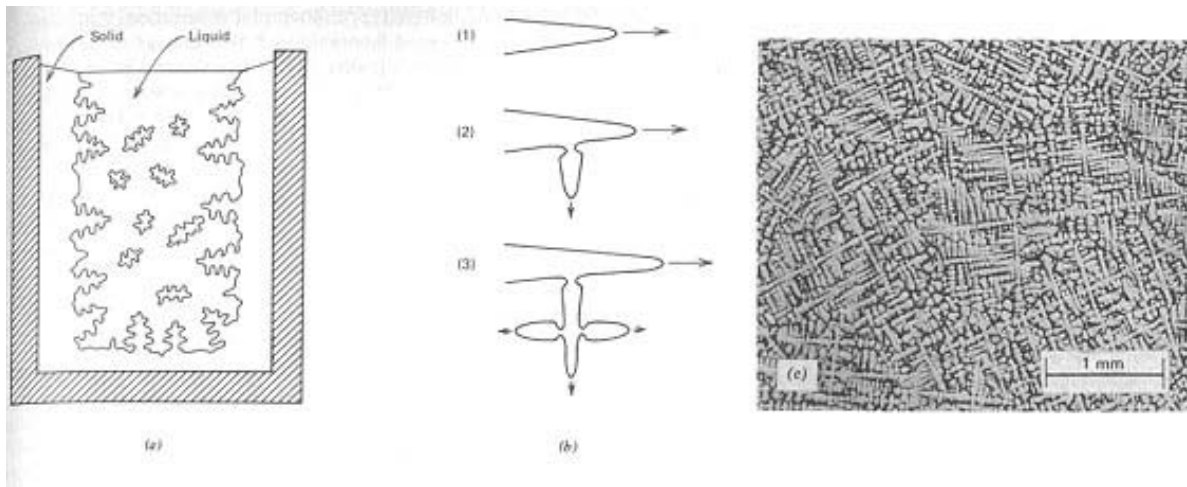
Most engineering materials are melted and solidified from the liquid state during the manufacturing process. This includes everything from solders on circuit boards to hip implants. Solidification is a material transformation familiar to everyone. Snow is a form of solid water that results from rapid cooling. Snowflakes, when viewed with a powerful microscope or sometimes just on your car windshield on a cold day, take on an interesting structure made up of tree-like forms called dendrites. The term “dendrite” comes from the Greek word “dendros”, which means “tree.” Except in extreme conditions, materials tend to solidify from their liquid state “dendritically”. The size, shape, and orientation of the dendrites help determine the strength and durability of steel, aluminum, and superalloys used in the production of automobiles and airplanes. Because virtually all industrially important alloys solidify from a molten state by dendritic processes, enhancing the basic understanding of dendritic solidification may help improve industrial production techniques.



Free energy as a function of temperature for crystalline (ice) and liquid (water) forms of H₂O. Below 0°C, the equilibrium melting temperature, and at atmospheric pressure, the free energy of ice is lower than that of water; therefore, ice is the stable phase. Above 0°C water is the stable phase. At any temperature the difference in free energy, ΔG , is represented by the vertical separation between the curves; at the equilibrium melting point the free energies are equal and both ice and water are stable.

Because most materials that contain more than one element do not have one melting point, but rather a range of temperatures over which they solidify, they gradually go from all liquid to a mixture of liquid+solid to fully solid as the temperature decreases. In the liquid+solid portion of the process, the solid assumes a dendritic shape so that we have a collection of solid dendrites surrounded by liquid. As cooling continues, the branches of the solid “tree” continue to grow until all liquid is consumed. <http://liftoff.msfc.nasa.gov/shuttle/usmp4/images/clips/idge.mov> As the dendrite arms, or “tree branches” continue to grow, their

composition changes. This process of changing composition during freezing/cooling is called segregation. Thus, when we etch our material, different regions with different chemical compositions that develop due to segregation have a different contrast when viewed under the microscope. This contrast allows us to measure the dendrite arm spacings in the material. The spacings between the main trunks of the trees are the primary dendrite arm spacing, while the spacing between the side branches is the secondary dendrite arm spacing. The dendrite arm spacings are dependent on the cooling rate during solidification.



III. Phase Volume Fractions

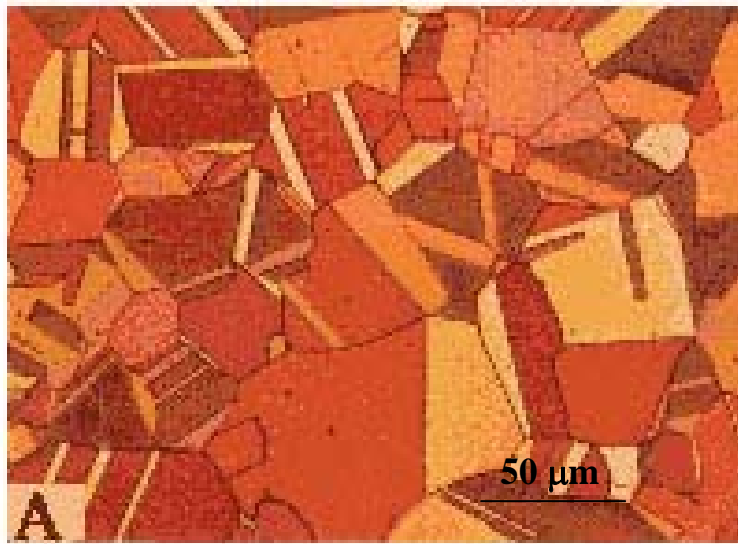
Sometimes materials can be quenched from the liquid state, instead of slowly cooled. In this case, we obtain an amorphous microstructure that is entirely differently from one that is a result of solidification. The photograph below is a scanning electron microscope picture of an amorphous alloy consisting of Fe-Zr-B-Cu metals. (Most engineering materials are not composed of a single element or compound, but rather of a complex mixture of a number of elements. A substance which contains a mixture of elements is referred to as an alloy.) The matrix is amorphous, but upon reheating, small crystallites of Fe being to nucleate out of the matrix. The volume fraction of crystallites that form, which element forms first, and the size distribution of these small crystallites all relate to the mechanical, electrical, and magnetic properties of the material. Controlling nucleation also gives us an idea of how we can control material properties.

Your Mission Today:

I. Grain Structure

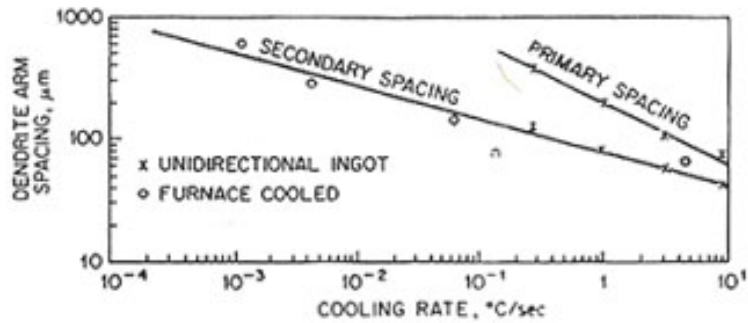
A sample of brass (70%Cu-30%Zn) has been cold-rolled with a 10% reduction in thickness. Following rolling, it is annealed in a furnace at 800°C for 1 hour. The sample was then mounted, polished, and etched to reveal the grain structure.

- (a) Taking the copied microscope photograph of the sample of brass, draw four lines randomly across the photographed section. You may wish to do this on an overlaying transparency sheet. Find the sum of the lengths, given the length scale of the photograph.
- (b) Count the total number of grains, N, crossed by the lines. Outlining individual grains prior to counting may be useful. Do not include twins in your definition of grains (they are mechanical deformations *within* grains as a result of the rolling). Any grains that are only partially contained in the viewing sections around the borders should be counted as ½.
- (c) Calculate the average grain size, D, in microns, as $D = L / N$.

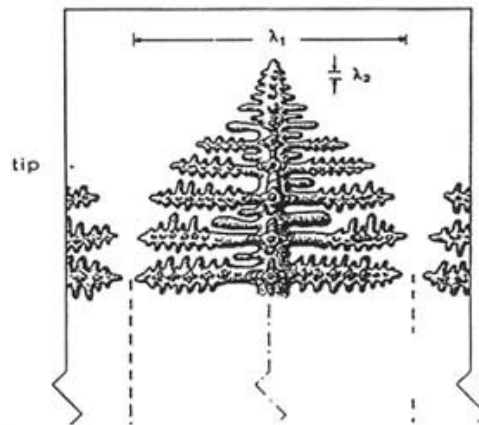
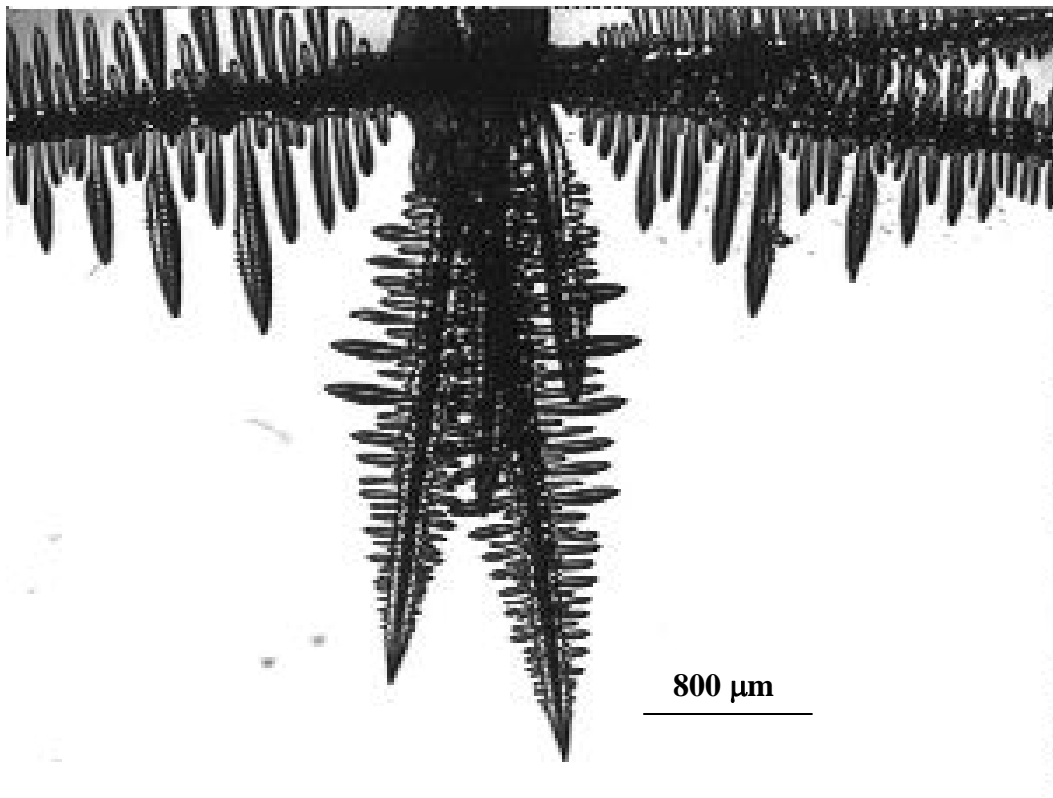


II. Dendrite Arm Spacing

- (a) Sketch the dendrite shown on page 5 in your lab notebook. Remember that you are viewing a 2-D projection of a 3-D phenomena.
- (b) Measure the distance between the pair of primary dendrites.
- (c) Measure the distance between 10 pairs of secondary dendrites. Determine the mean and median spacing of the secondary dendrites. Based on the graph of dendrite arm spacing vs. cooling rate, what is the approximate cooling rate of this alloy.



The relationship between dendrite arm spacing and cooling rate.



Schematic of a dendrite and the associated primary (λ_1) and secondary (λ_2) spacings.

III. Measurement of Volume Fraction by Point Counting

- Taking the SEM photograph on page 6, draw a small area and calculate the area A .
- Count the number of Fe crystallites that have nucleated out of the amorphous matrix, N , within this area. You may wish to overlay a transparency to do this. Determine the number of particles per unit area, $W = N/A$.
- Measure the diameters of several particles within the region of interest. Calculate an average particle diameter, and then an average particle area, P .
- Determine the volume fraction VF as: $VF = W \times P$.

