EXPERIMENT

RECOVERY, RECRYSTALLIZATION AND GRAIN GROWTH GRAIN SIZE MEASUREMENT BY QUANTITATIVE METALLOGRAPHY

AIM: To report microstructural changes in a cold worked single phase alloy during its annealing and grain size determination of recrystallized alloy by quantitative metallography.

THEORY:

A. Recovery, Recrystallization and Grain Growth:

Metals can be plastically deformed. i.e. they will not come back to their original shape and size when the load causing their deformation is removed. Thus, we can roll a metallic strip to reduce its thickness. Similarly, there are many other metal working operations which are industrially used to make components and parts of many engineering devices from metallic materials.

A small percentage of the energy spent in plastically deforming a metal remains stored in its structure and increases its internal energy. This increment in the internal energy is associated with the generation of lattice defects such as vacancies, interstitials, dislocations and stacking faults during plastic deformation of the crystal. On a microscopic scale the distribution of these defects, however is very inhomogeneous. For example, bulk of the deformed crystal consists of relatively dislocation-free regions (called sub-grains or cells) separated by boundary regions of high dislocation density in which many dislocations are arranged as tightly packed tangles There may be many of such cells in each grain of the material. If the deformation occurs at a low enough temperature so that the crystal has very little thermal energy to recover from such a structure, dislocation tangles are retained in its structure and we refer to such a structure as the cold worked structure. What do you think will happen when such a structure is heated? Obviously, the crystal acquires thermal energy and with its help several thermally activated mechanisms, such as atomic diffusion begin to operate. Heating of a cold-worked (or cold-deformed) structure at a given temperature for a given period of time is generally referred to as annealing. Phenomena which occur during annealing are recovery, recrystallization and grain growth.

RECOVERY:

The first effect of reheating previously cold-worked metal is reduction of internal stress without noticeable change of mechanical properties. Since this effect is not evident microscopically, the importance of the recovery process has not been widely appreciated by engineers. Recovery in a cold-worked structure occurs when the material is held at relatively lower temperatures for a given time interval. It involves changes in the number and distribution of point defects and dislocations present in the cold-worked structure. Since the temperature of annealing is low during recovery, structural changes in the material occur by the clustering of point defects like vacancies and interstitials and the migration of point defects to dislocations, grain boundaries and/or external surfaces of the crystal. If the annealing is carried out at some what higher temperature even dislocations (which are less mobile than point defects) may gain appreciable mobility and move by glide and climb to relieve some of the internal strain accumulated in the structure. Some of the dislocations may rearrange themselves while some others may get annihilated by mutually canceling each other. Thus during the process of recovery of a cold-worked material the dislocation density of cells gets reduced and dislocation walls around these cells become more sharply defined. Small changes in hardness which are usually observed during later stages of recovery are due to decrease in the number of point defects and dislocations.

RECRYSTALLIZATION:

Recrystallization entails the complete formation of new and unstrained metal grains, with simultaneous elimination of previously cold-worked and distorted metal. Recrystallization is very evident because of its marked effect on mechanical properties. Strain hardening and work strengthening which resulted from cold working are largely eliminated by recrystallization and ductility is simultaneously restored. The microscopical appearance of cold-worked metal is also greatly changed by recrystallization.

When annealing is carried out at even higher temperatures recrystallization occurs. During the recrystallization process new strain-free grains crystallize in the earlier cold-worked structure. The free energy of the system is lowered by these newly formed strain-free grains. In an aggregate of deformed metal crystals, there are regions where the· atomic structure is most highly disarranged, and such regions exhibit the strongest tendency for realignment into an unstrained condition and the strongest desire for recrystallization. These regions are at grain boundaries, at slip planes or twinning planes, at internal discontinuities, regions of stress concentration caused by inclusions, and elsewhere with the crystal where internal strain is high. These new grains grow at the expense of the deformed structure until the whole matrix is consumed. This process occurs either by migration of original grain boundaries or by sub-grain growth. The recrystallized matrix has much lower dislocation density than the deformed material. The lowest temperature at which strain-free recrystallized grains appear in the structure of the previously cold-worked structure is termed as the recrystallization temperature of that material. The recrystallization temperature is influenced by the grain size, the severity of cold-work, strain given during plastic deformation, the presence of solute atoms and the second phase in the structure.

Greater prior cold working increases the number of recrystallization nuclei and ensures finer grain size after complete recrystallization. Greater plastic deformation also creates greater misalignment at grain boundaries and regions of internal strain, a factor which allows recrystallization at lower temperature. The recrystallization temperature is lower because less thermal agitation is necessary to obtain sufficient energy for the atoms to become rearranged by recrystallization.

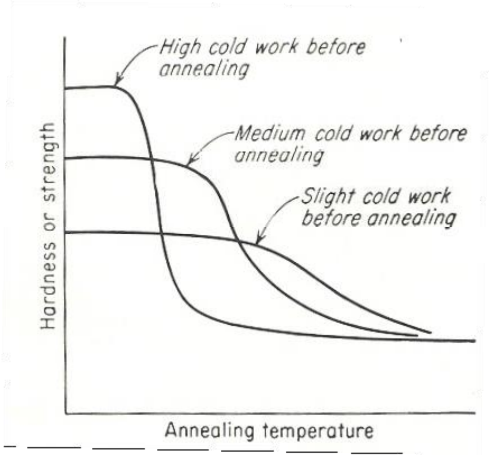
Finer initial grain size results in more strained regions in plastically deformed metal, which in turn causes finer grain size in the fully recrystallized metal. Nucleation and growth of new unstrained crystals are time and temperature dependent. For a given amount of plastic deformation, the rate of nucleation and the growth rate of the newly formed grains are more rapid at higher temperatures.

Recrystallization occurs by the continual formation of new unstrained grains and their growth until all the strained metal is replaced by unstrained material. The recrystallization temperature is defined as the temperature at which recrystallization is complete, but it is usually much more satisfactory to think in terms of the range of temperatures between the first appearance of a new grain and complete recrystallization. This is called the recrystallization temperature range.

It is not possible to cite particular recrystallization temperatures, since recrystallization depends upon several variables. Increased plastic deformation allows recrystallization at a lower temperature, as plotted in Fig. l2-l. The time of annealing also has an effect. Increased time allows recrystallization at lower temperature.

(Fig.l2-2). Coarse grained metal is not work-hardened as much by a given amount of deformation as is finer-grained metal and will not have as great an urge to recrystallize.

Therefore the recrystallization temperature will be higher for coarse-grained metal



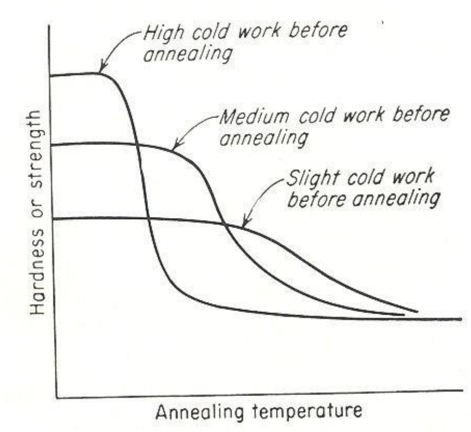


Fig l2.l Effect of prior cold work on recrystallization temperature

Fig l2.2 Effect of annealing time on recrystallization temp of cold worked metal

Grain Growth or Coalescence

When cold-worked metal is held above the recrystallization temperature after recrystallization is complete, growth of some of the recrystallized grains at the expense of other grains occurs, a process called grain growth or coalescence. This is a digestion process in which some grains increase in size while others get smaller and finally disappear, resulting in a larger average grain size and a smaller total number of grains.

Grain growth occurs because in doing so the total area of grain boundaries reduces and lowers, in turn, the grain boundary energy of the system.

Because engineering properties of metals are related to grain size, control of grain growth is an important feature of the metallurgy of cold working and annealing. Control of grain size of cold-worked metal during heat treatment depends, to a large measure, upon control of prior history of the material, but a fine-grained structure can be coarsened by coalescence during annealing. Attempts to shorten the annealing time after cold working by utilizing a high furnace temperature may result in excessive coalescence. Overloading furnaces so that some parts of the charge are overheated before the entire charge is adequately softened may also result in excessive coalescence in the overheated portions.

Why do some crystals grow during coalescence while others become smaller and disappear? Grain-boundary migration is the fundamental feature that governs this condition, although the driving force for boundary migration is not completely known. When the boundary between, two metal grains is perfectly plane, it has little tendency to migrate, and there is little tendency for one grain to digest its neighbor. However, boundaries that are not in plane appear to migrate toward the center curvature of the boundary. This tends to make large grains larger small grains smaller until they disappear. Also, there may be a tendency for grains to grow in certain crystallographic directions preferentially to other directions.

A complete analysis of grain-boundary migration is not within the scope of engineering metallurgy, but this brief statement of possible mechanisms is included as a means of evaluating the effects of various working and heating cycles on boundary migration. Purer metals or alloys coalesce extensively, while less pure material retains fine grain size for longer annealing periods or at higher annealing temperatures. All metals, or even all heats of the same metal, do not develop similar grain sizes during forming and reheating processes, and do exhibit differences in subsequent operations and significant variation in final properties.

The Cold-work-Anneal Cycle

From the preceding discussion it is evident that cold working hardens and strengthens metals at the expense of decreased ductility. However, when cold working is excessive, the metal may fracture before reaching the desired size and shape. To avoid such defects, the total deformation is produced in several steps with intermediate anneals to soften the hardened metal and to regain lost ductility so that further deformation is possible. This process of repeated cold working and annealing is called the cold-work- anneal cycle, which is illustrated for specific conditions in Fig. l3-20.

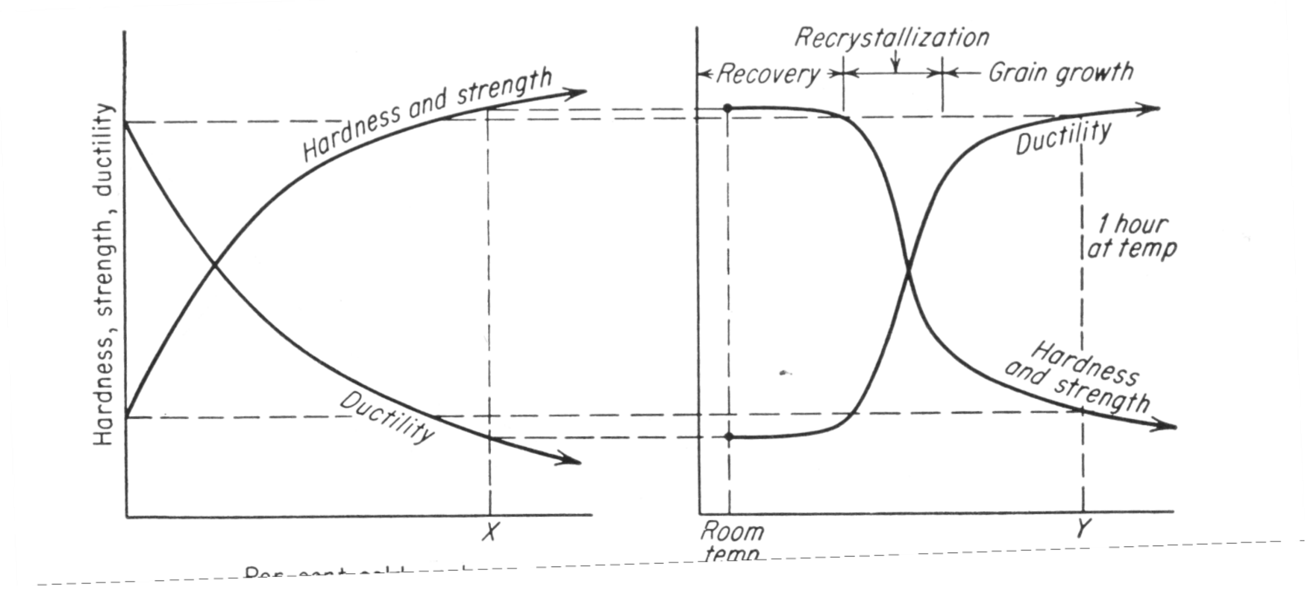


Fig l3-20. Cold –worked anneal cycle.

Metal is hardened and strengthened by cold deformation and ductility is decreased. Before the metal fractures, cold working is stopped as at X. If metal which has been previously deformed to point X is reheated for 1 hr at a temperature Y, the original ductility and strength will be returned. To obtain immediate properties the stock is annealed when sufficiently oversize so that the desired properties will be developed during a final working operation.

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Grain growth occurs because in doing so the total area of grain boundaries reduces and lowers, in turn, the grain boundary energy of the system. In simple situations the occurrence of grain growth can be expressed in terms of the following relationship;

$$D_t -- D_0 = Kt^n$$

Where D_t is the average grain diameter after the annealing time of t, Do is the initial grain diameter before grain growth starts, K and narc constants. Usually a value of n is estimated to be l/2.

Grain Size Measurement by Quantative Metallography:

From the study of actual grain shapes it has been found that while grains do not actually possess regular or idealized forms the truncated octahedron is a reasonable approximation for equiaxed grains. For the quantitative estimate of the average grain size of a single-phase equiaxed grain structure the grains along a line, circle or within a known area are counted. In Jeffries' Planimetric Method a circle surrounding a known area is superimposed on the microstructure as shown in Fig.l2.4.The grains cut by the circumference. n_c and grains completely within the circle n_i are counted. Half of the number of grains intersected by the circumference is added to the number of completely included grains to get the number of equivalent whole grains within the circle. Thus

$$n_{eq} = (½).n_c + n_i$$

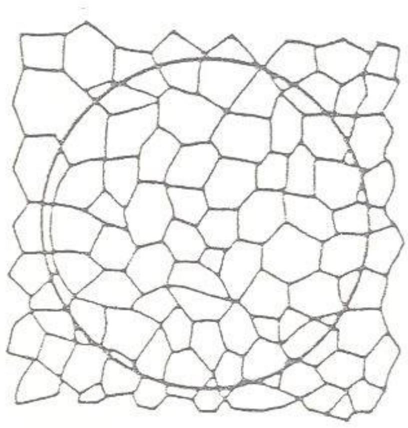

Fig l2.4 Grains size measurement by Jefferies Planimetric method

From the analysis of the distributions of sections in space. occupied by truncated octahedra. The ASTM (American Society of Testing Materials). System gives the following relationship

$$Na = 2^{N-l}$$

where n is the number of grains per square inch at l00x and N = ASTM grain size index number. In the ASTM method the microstructure of a given single-phase polycrystalline specimen when observed at a magnification of l00X is compared with a set of standard photomicrographs.. The charts are indexed for N varying from l to 8.

EQUIPMENT:

Metallurgical microscope, A calibrated eyepiece and Six polished and etched specimen of a single phase material of cold rolled and annealed samples

PROCEDURE:

(l) Observe the microstructures of Sample No.l-6. Note the difference in the shape of grains in each cold rolled and recrystallized sample. Note the absence of any recrystallization activity in Sample No.2 and formation of a few recrystallized grains Sample No.4. Also observe the difference in the grain structure of Sample Nos.5 and 6

(2) Calculate the ASTM grain size for sample 6.

REPORT:

(l) Schematic diagram of micro structures of Sample Nos.l - 6.

(2) Mention the etching reagent used.

QUESTIONS:

l. What factors are expected to raise the recrystallization temperature of a metal or alloy? Why?

2. If we hold the cold worked sample in the furnace for a very very long time at a temperature above its recrystallization temperature {will the grain growth continue or will it stop at some stage? Explain.

3. During recrystallization where do new grains will first form? Why?

4. Why is metal able to plastically deform under a given load, while glass breaks