

## Chapter 8 Introduction to Metallographic Study

### Capítulo 8 Introducción al Estudio Metalográfico

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## Abstract

The quality assurance of materials in every process depends on materials science and engineering, since the characterization of materials is a fundamental process, for their control and innovation, as well as their proper functioning. In the synthesis of new materials, their properties depend not only on the type of raw material used (metal, polymer, ceramic, composite material), but also on the morphology acquired in the synthesis, structure, microstructure, etc. of the material obtained, hence the importance of metallographic characterization. Metallography is the science that deals with the microscopic study of the structural characteristics of a metal or an alloy, to study the microstructure, inclusions, as well as the thermal treatments to which the material has been subjected, in order to determine if the material complies with the specifications established in the Standards applicable to the design requirements for a specific use. The objective of this study was to present the basic techniques for the preparation of specimens to be evaluated metallographically, addressing the following topics: Generalities, Atomic structure: Nucleation and atoms, Crystal structure: Perfect crystals and crystals with imperfections. Substructure: Subgrains and other cellular structures, Microstructure: Grains of single metallic phases and configuration arrangements of alloys with multiple phase systems, Texture. Macrostructure. and Metallographic practice applicable to all metals. It is concluded that this study lays the foundations for subsequent and specific metallographic studies of ferrous and non-ferrous alloys.

## Quality Assurance, Characterization, Microstructure, Properties

### Resumen

El aseguramiento de calidad de los materiales en todo proceso depende de la ciencia e ingeniería en materiales, ya que la caracterización de los materiales es un proceso fundamental, para su control e innovación, así como del buen funcionamiento de los mismos. En la síntesis de nuevos materiales, las propiedades de los mismos dependen no solamente del tipo de materia prima utilizada (metal, polímero, cerámico, material compuesto), sino también de la morfología adquirida en la síntesis, estructura, microestructura, etc. del material obtenido, de ahí la importancia de la caracterización metalográfica. La metalografía es la ciencia que se encarga del estudio microscópico de las características estructurales de un metal o de una aleación, para estudiar la microestructura, inclusiones, así como los tratamientos térmicos a los que haya sido sometido el material, con la finalidad de determinar si el material cumple con las especificaciones establecidas en las Normas aplicables a los requisitos de diseño para un uso específico. El objetivo de este estudio consistió en dar a conocer las técnicas básicas para la preparación de especímenes a evaluar metalográficamente, abordando los siguientes temas: Generalidades, Estructura atómica: Nucleación y átomos, Estructura cristalina: Cristales perfectos y cristales con imperfecciones. Subestructura: Subgranos y otras estructuras celulares, Microestructura: Granos de fases metálicas sencillas y arreglos de la configuración de aleaciones con sistemas de fases múltiples, Textura. Macroestructura. y Práctica metalográfica aplicable a todos los metales. Se concluye que este estudio sienta las bases para estudios metalográficos posteriores y específicos de aleaciones ferrosas y no ferrosas.

## Aseguramiento de Calidad, Caracterización, Microestructura, Propiedades

### 1. Introduction

The quality assurance of materials in every process depends on materials science and engineering, since the characterization of materials is a fundamental process, for their control and improvement, as well as their proper functioning. Now, the development and innovation of materials requires knowledge of the characteristics of the material, in order to choose the most suitable for a specific application. In the synthesis of new materials, their properties depend not only on the type of raw material used (metal, polymer, ceramic, composite material), but also on the morphology acquired in the synthesis, structure, microstructure, etc. of the material obtained, hence the importance of metallographic characterization. Metallography is responsible for the study of the structure, chemical composition and physical properties of metals and their alloys, for which it uses Microscopy, Polarimetry, and Radiography, studying the surface of metal specimens previously polished and subsequently attacked with chemical solutions. To reveal the microconstituents under study. Due to the great importance that metallography has for carrying out metallographic tests and problem solving in the industry, it is of great interest to know the basic techniques for the preparation of specimens to be evaluated, hence the relevance of this study.

The chapter has been compiled into 8 sections, which will serve as an auxiliary tool for students of Materials Engineering, Chemical Engineering, Industrial Engineering, and in general for all those people who, within the field, perform functions related to Quality Assurance of Metallic Materials, as well as for those who are interested in the Metallographic Study and wish to solve practical problems. Topics are addressed as follows:

## **Section 1. Generalities**

### **1.1 Historical Background**

Metallography began more than 200 years ago. One of the first initiators was R. A. F de Reaumur (1683-1757) who gave indications on how to differentiate steel gates through macroattack. At that time, its macro-development was carried out without the help of optical means.

Another initiator was S. Rimman (1) who by means of acid attack differentiated authentic Damascus steels from imitations, he wrote in 1774 “The attack is an appropriate means of which one can be valid to distinguish iron and steel gates in terms of differences in hardness, homogeneity and non-homogeneity”.

N. G. Sefstrom (2) in 1825 found that acid etching was a good means of seeing the homogeneity of the rolled iron structure.

The development of the structure of ground and polished iron is due to A. de Widmanstätten (3) 1808, who observed structural characteristics of iron, non-annealed gray iron and hardened steel which is known as Widmanstätten'sche structure.

In 1887 C.H. Sorby (4) reported the printing test on ground steels treated with strong acids. These age-old methods were macroetches that could be seen with the naked eye and did not require a mirror-polished specimen. Later use of the microscope required better surface preparation of the specimen and a special etching medium to differentiate metals.

Especially in the years 1870 to 1880 a series of microetching methods for iron and steel were used by the pioneers of metal microscopy. H. C. Sorby (5), A. Martens (6), and F. Osmond (7), who used dilute solutions of iodine in alcohol as etching media.

Years later the attack methods were further improved. Other researchers who also took part in these studies were:

H. Weddings (8), E. Hayn (9), H. Le Chatelier (10), I. E. Stead (11), W. Ischewsky (12), and Kourbatoff (13).

Day by day the era marked a new path, new methods of analysis that are innovated every day.

Metallography as a classic method is used to verify the development of structures in metallic alloys, mirror polishing metallic surfaces called metallographic specimens and observing them in an incident light microscope (14).

The structure of metals comprises very extensive features of varying degrees of complexity. In increasing order these magnitudes are:

- a) Atomic structure: Nucleation and atoms
- b) Crystal structure: Perfect crystals and crystals with imperfections
- c) Substructure: Subgrains and other cellular structures
- d) Microstructure: Grains of simple metallic phases and configuration arrangements of alloys with multiple phase systems.
- e) Texture
- f) Macrostructure.

## Section 2. Atomic Structure: Nucleation and Atoms

Atom. It is the basic unit that can intervene in a chemical combination. It is made up of subatomic particles, of which the most important are electrons, protons and neutrons. Electrons are negatively charged particles that are found in energetic places known as *rempe*s or orbitals. Its mass is  $9.1 \times 10^{-28}$  g. Protons are positively charged particles found in the atomic nucleus and whose mass is  $1.67 \times 10^{-24}$  g. Neutrons are electrically neutral particles, found in the nucleus and having a mass slightly greater than that of protons  $1.675 \times 10^{-24}$ g. The number of protons in the nucleus of an element is known as the atomic number (Z). The number of protons and neutrons present in the nucleus of an atom of an element is known as the mass number. When measured in amu (atomic mass units), referred to the mass of a carbon 12 atom), it is called atomic mass (A). Some elements have more than one atomic mass, depending on the number of neutrons in their nucleus. These atoms are called isotopes (elements with the same atomic number but different masses). The atomic weight of an element is the average of the masses of the natural isotopes expressed in atomic mass units (amu).

### 2.1 Atomic models

At the beginning of the 20th century, Bohr proposed a planetary model to explain the atomic structure: in the center of the atom was the nucleus where the protons and neutrons are and surrounding said nucleus, the electrons rotated distributed in shells or energy levels. The closer they were to the nucleus, the less energy they had. This model did not explain some experimental results and therefore, in the late 1920s, Schrödinger and Heisenberg proposed a quantum mechanical model. Heisenberg said that it is impossible to know exactly the position and speed of an electron at a given time (Principle of uncertainty), so statistical regions of greater electronic probability (*rempe*) were described that define the possible position of an electron at a given time. moment. These regions are also known as atomic orbitals and have some sublevels. The position of an electron can be defined by 4 quantum numbers: **n**, **l**, **m** and **s**.

#### Bohr's postulates

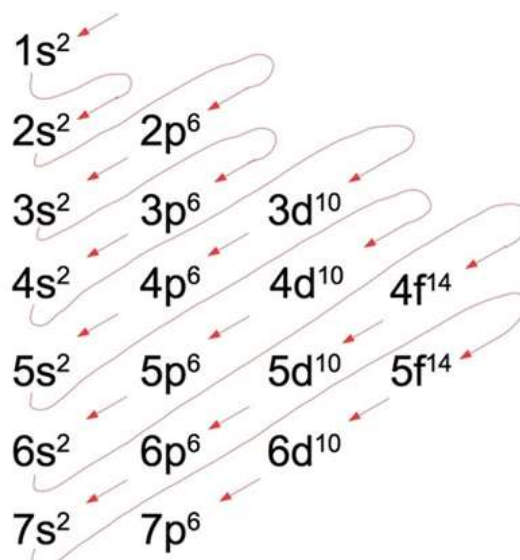
1. The electron revolves around the nucleus in a fixed set of allowed orbits, called stationary states: in them it rotates without absorbing or emitting energy.
2. Only those orbits in which the angular momentum of the electron is an integer multiple of  $h/2\pi$  are allowed.
3. Electrons can jump from one allowed orbit to another by absorbing (if the final orbit is farther from the nucleus) or emitting (if the final orbit is closer to the nucleus) energy in the form of electromagnetic radiation. The Bohr model of the atom was unable to explain the following: The spectra of atoms more complex than the hydrogen atom. The variation of the intensity of the spectral lines, the arrangement and distribution of the electrons in various orbits, the fine structure of the spectral lines of hydrogen. And the effects of Stark and Zeeman.

### 2.2 Electronic configuration

The arrangement of the electrons in the various atomic orbitals is known as the electron configuration and it follows some basic rules:

1. An orbital cannot have more than two electrons, which must rotate in opposite directions.
2. Electrons do not gather in an orbital if there is another available with the same energy. There is an order in which the orbitals are filled and it is described in Figure 1. Following the direction indicated by each of the diagonals, the order of filling of the sublevels in the respective levels is determined.

**Figure 1** Arrangement of electrons in the various atomic orbitals.



Source: <https://www.shutterstock.com/es/search/configuraci%C3%B3n-electr%C3%B3nica>

Electronic configuration examples: Helium (He<sub>2</sub>: 1S<sup>2</sup>), Neon (Ne<sub>10</sub>: 1S<sup>2</sup> 2S<sup>2</sup> 2 P<sup>6</sup>), Krypton (Kr<sub>36</sub>: 1S<sup>2</sup> 2S<sup>2</sup> 2P<sup>6</sup> 3S<sup>2</sup> 3P<sup>6</sup> 4S<sup>2</sup> 3d<sup>10</sup> 4P<sup>6</sup>). Thompson model. In 1904, before protons and neutrons were discovered, J.J. Thompson proposed a model for the atomic structure.

### Thompson model

Sir Joseph Thompson (1856-1940). This English physicist at the age of 28 had the honor of being elected as a member of the Royal Society. In the same year he became Cavendish Professor of Experimental Physics at Cambridge. In 1897, he announced that he had identified cathode rays as streams of negatively charged particles, which he called corpuscles; which were later called electrons. He received the Nobel Prize for Physics in 1906. His model was referred to by others as "raisin pudding". According to this reasoning, an atom must have enough of a positive charge somewhere to neutralize the negative charges of the electrons present.

### Rutherford's model

Many features of Thompson's model were examined and criticized, for example, some of them leading to certain predictions regarding the behavior of very thin metallic foils against atomic-sized projectiles, such as alpha particles. The streams of alpha particles come from uranium or radium, in bundles initially called alpha rays, because no one knew their nature it was necessary to give them a name. Ernest Rutherford identified alpha rays by defining them as atomic-sized particles with a mass of 4 amu and a charge of 2+.

Rutherford proposed that all of an atom's mass and all of its positive charge were concentrated in a small, dense bundle at the center of the atom (the nucleus). Surrounding this nucleus at some distance from it were the electrons that made the particle electrically neutral, although he did not explain how they could be outside and not be attracted to the nucleus.

### 2.3 Types of Bonds

Chemical bonds are the forces that hold atoms together in compounds. They are divided into several classes, depending on the properties of the compounds. The three main types are:

- 1) Ionic bonds, formed by the transfer of one or more electrons from one atom or group of atoms to another.
- 2) Covalent bonds that appear when one or more pairs of electrons are shared between two atoms. Both of these types of links are extreme and all links have some of them. Compounds in which the ionic character predominates are called ionic compounds; those in which the covalent character predominates are called covalent compounds.

- 3) Metallic bonds are found in solid metals such as copper, iron, and aluminum. In metals, each atom is bonded to several neighboring atoms, the bonding electrons are relatively free to move through the three-dimensional structure forming an electron cloud or sea of electrons, which give rise to typical metallic properties, such as high conductivity, electric and brightness.

### Section 3. Crystal structure: Perfect crystals and crystals with imperfections.

Imperfections in crystals include defects such as atomic impurities, interstitial voids and aggregates, line defects (dislocations), and area defects (paired interfaces, subboundaries, and grain boundaries).

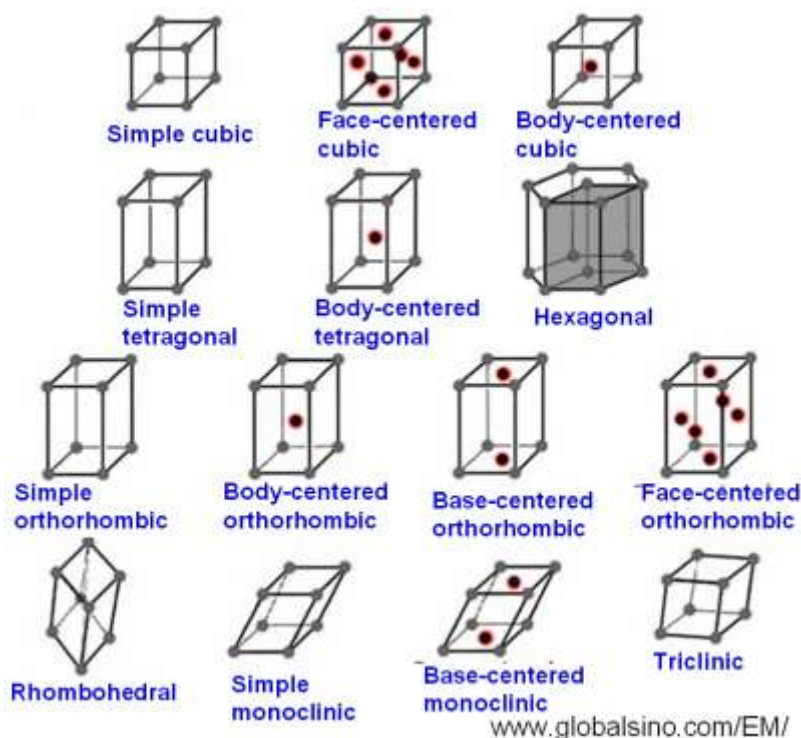
A common solid contains an order of  $10^{23}$  atoms/cm<sup>3</sup>. To communicate the spatial arrangements of the atoms in a crystal, it is clearly not necessary to specify the position of each atom. Two complementary methodologies will be explained to describe in a simple way the three-dimensional arrangements of the atoms of a crystal. They will be referred to as the lattice and base concept and the unit cell concept, concepts linked to the principles of crystallography. An atom consists of a nucleus of protons and neutrons surrounded by electrons, but for the purpose of describing the arrangements of atoms in a solid, we will visualize the atoms as rigid spheres, similar to ping pong balls, starting with the concept of network and base.

#### 3.1 Network

A network is a collection of points called network points, which are arranged in a periodic pattern such that the neighborhoods of each point in the network are identical. A network is a purely mathematical construction whose extension is infinite. A network can be one-dimensional, two-dimensional, or three-dimensional. In one dimension there is only one possible network: It is a line of points separated from each other by an equal distance. A group of one or more atoms located in a particular way with respect to one another and associated with each lattice point is known as a motif or base.

The base must have at least one atom, but it can contain many atoms of one or more types. To obtain a crystal structure, the base atoms must be placed at each lattice point (i.e., the crystal structure will be equal to the lattice plus the base). There are five different ways to arrange the points in two dimensions, so that each point has identical neighborhoods: Therefore there are five two-dimensional networks. There are only 14 unique ways to arrange the points in three dimensions. These unique three-dimensional arrangements of lattice points are known as Bravais lattices, named after Auguste Bravais (1811-1863), who was one of the first French crystallographers. Figure 2 shows Bravais networks. The 14 Bravais lattices Figure 2, are grouped into seven crystal systems, which are known as cubic, tetragonal, orthorhombic, rhombohedral (trigonal), hexagonal, monoclinic, and triclinic.

Figure 2 Bravais networks



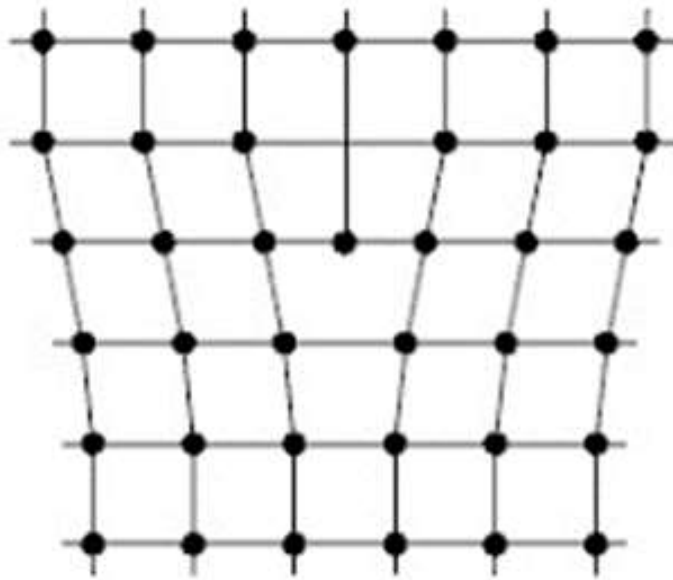
### 3.2 Imperfections in the crystals

The arrangement of atoms or ions in engineering materials contains imperfections or defects, defects that have a large effect on the properties of the materials. There are 3 basic types of imperfections: point defects, linear defects (dislocations), and surface defects.

#### 3.2.1 Point Defects

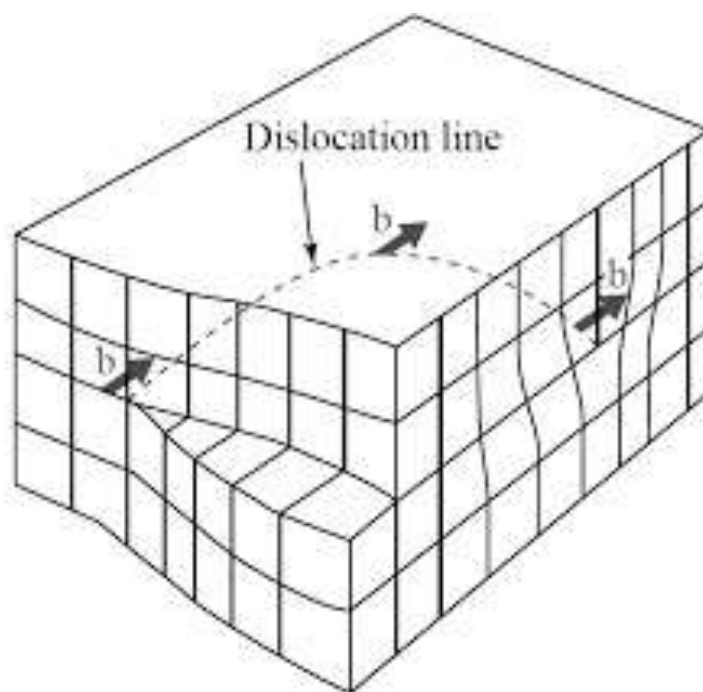
They are localized disturbances in the ionic or atomic arrangements of a crystal structure. The disturbance affects a region involving several atoms or ions; Examples of such imperfections are dislocations (misplaced atoms), vacancies, interstitial atoms, substitutional atoms: Figures 3, 4, 5, 6, 7.

**Figure 3** Edge Dislocation

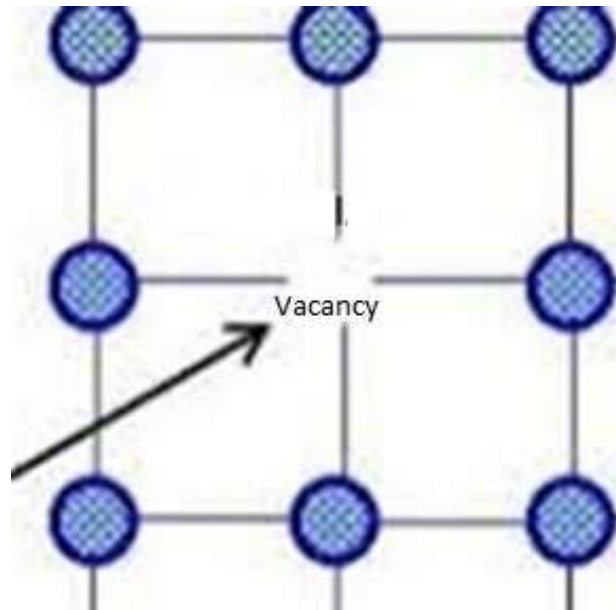


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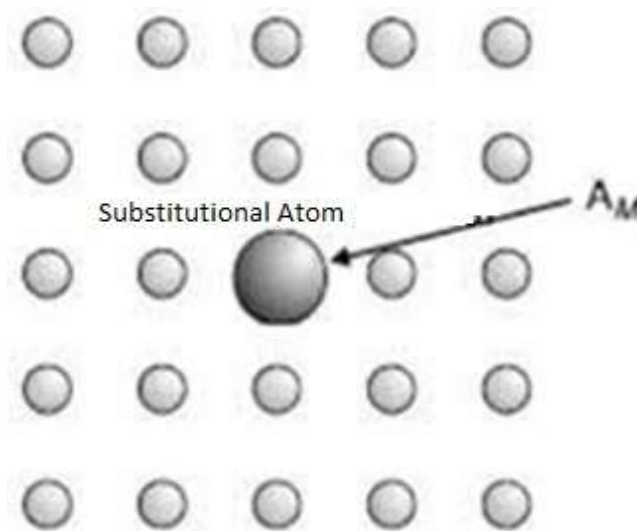
**Figure. 4** Mixed dislocation



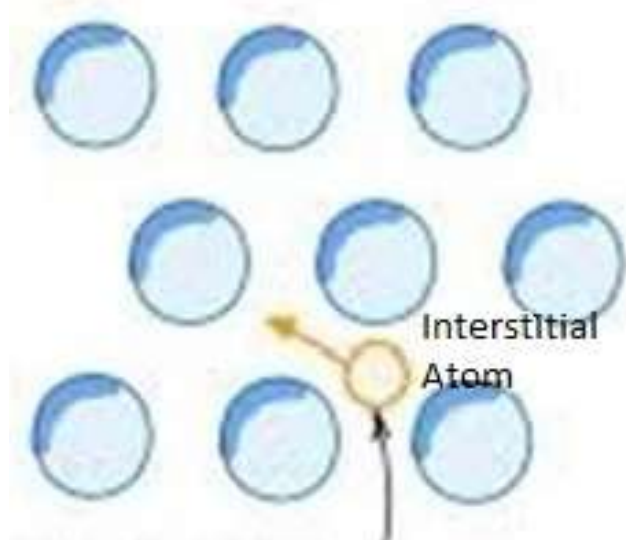
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**Figure. 5** Vacancy

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**Figure. 6.** Substitutional Gold Atom

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**Figure 7** Interstitial atom

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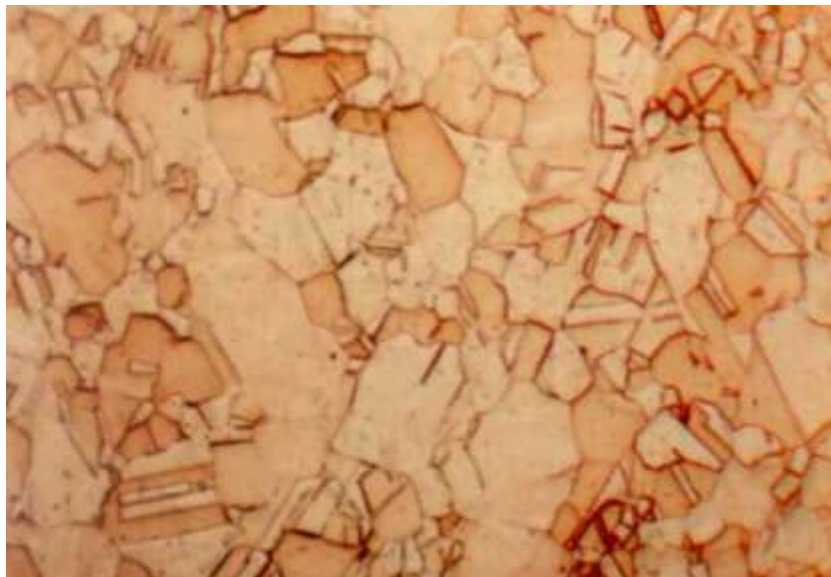


These imperfections can be caused by the movement of atoms or ions as they gain energy by heating during material processing or by the intentional or unintentional introduction of impurities. Impurities are usually elements or compounds contained in raw materials or during processing. For example, silicon crystals grow in quartz crucibles whose impurity is oxygen. In the case of dopants, they are elements or compounds that are added intentionally, in known concentrations, at specific places in the microstructure, with a desired beneficial effect on properties or processing. In general, the effect of impurities is detrimental, while in the case of dopants it is beneficial to obtain desired properties for a specific use of the material.

#### Section 4. Substructure

Subgrains and other cellular structures. Subgrains or cellular structures are formed by means of subboundaries (boundaries with small angles). The simplest of these boundaries consists of periodically spaced dislocations, dislocations can form cellular structures particularly in structures resulting from deformation. All kinds of imperfections in crystals can be found in single crystals or between grains of polycrystalline metals. Twins are an example of this. A twin is the symmetric grouping of identical crystals. The symmetry can be mirror with respect to the twin plane or by the rotation of its elements around the twin axis by  $60^\circ$ ,  $90^\circ$ ,  $120^\circ$  or  $180^\circ$ . A plastic deformation is produced on an annealed copper sample, performing a microstructural analysis represented in figures 8, 9, twins are observed. Twins are microstructurally identified as narrow subgrains with fairly rectilinear and parallel edges dividing the initial single crystal.

**Figure 8** Twins within the grains of the copper matrix, due to deformation. Metals Handbook



**Figure. 9.** Strain twins in a pure zinc crystal. Metals Handbook



## Section 5. Microstructure

Grains of single metallic phases and configuration arrangements of alloys with multiple phase systems. Imperfections in crystals include defects such as atomic impurities, holes (vacancies), and interstitial aggregates, as well as substitutional atoms, linear defects (dislocations), and area defects (paired interfaces, subboundaries, and grain boundaries). The grain structure of single-phase polycrystalline metals is the most salient feature in the microstructure.

### 5.1 Pairs

They are imperfections that can originate during the development process; example: during annealing of cold-worked metal, or during grain deformation.

### 5.2 Structure with multiphase

It is that configurational arrangement of two or more phases to form a multifaceted structure, which highlights certain microstructural characteristics.

## Section 6. Texture

It combines the strengths of lattice oriented crystallography with the microstructural strengths of grain structure. In a metal that has a preferred texture or orientation, the crystal takes over the grains and they are arranged in a correlated and organized manner.

Usually the metallic material is a polycrystal composed of a lot of crystal beads. When the grain orientation of a polycrystal is concentrated around a certain reference plane (or direction) of a macroscopic material, it is called preferred orientation, and texture is the preferred orientation of polycrystals. In a broad sense, the phenomenon that the grain orientation deviates from the random distribution in the polycrystal can be called texture.

In metallic materials, the existence of texture phenomena is universal. The external temperature field, electromagnetic field, strain field, and anisotropy within the crystal can cause texture. For example, the preferred grain orientation during deformation is slip surface, crystal slip, and moment effect during stretching. Industrial materials commonly have casting texture, deformation texture, recrystallization texture and phase change texture, among which deformation texture and recrystallization texture are most studied.

### 6.1 Description of crystal orientation and common types of texture.

The so-called crystal orientation refers to the three crystal axes (such as [100], [010], [001] axis) in a given reference coordinate system (such as rolling direction RD, lateral TD, and normal ND in rolling plate). When the orientation of the crystal is actually described, different reference frames are established due to different deformation conditions. For example, for the most common rolling deformation, the three reference frame axes are generally set to the rolling direction (RD) and the rolling surface. The direction (ND) and the transverse direction of the rolled sheet, that is, the direction perpendicular to the rolling direction (TD), assuming an orientation, is expressed as (110) [112], indicating the (110) plane of the unit cell at this time, parallel to the rolling surface, the [112] direction is parallel to the rolling direction.

The type of texture mainly depends on the nature of the metal and the processing method, etc. Among them, there are rolling texture, drawing texture and the like. Rolling texture is the texture that occurs during rolling deformation. It is characterized in that a certain crystal plane {hkl} of each grain is parallel to the rolling surface, and one direction is parallel to the rolling direction. The tread texture is usually expressed as {hkl}. Unidirectional stretching and pattern deformation cause a certain direction of the polycrystalline grains to be parallel to the stretching or pattern direction. The texture thus formed is called silk texture, also called fiber texture, parallel to stretching.

Or the glass orientation of the drawing direction. The presence of texture is universal in metallic materials. The essence of texture is that many grains are not distributed in a random orientation, which naturally leads to anisotropy in material properties. The effect of texture on material properties is studied to better utilize the texture in the material and to be able to regulate the related properties of the material.

## **Section 7. Macrostructure**

The macrostructure of metals and alloys deals with those characteristics on a larger scale than that of the constituents of the microstructure (14).

A macrostructure comprises gas holes or porosities in molten metal which originate during solidification, such as flow lines in forging which originate during the deformation process.

Flow lines in the forged can be caused by elongated inclusions or by inhomogeneities in the alignment of the shape of the grains.

### **7.1 Origins of the structure**

The structural characteristics of metals and alloys are produced by:

- a. transformations in which one or more phases are converted into one or more new phases.
- b. Mechanical processes
- c. Thermal processes or
- d. Diffusion processes (14).

Cold working is a typical mechanical process. The annealing of a cold worked metal is the main example of a thermal process. The fundamental principles that govern these principles are under the jurisdiction of Physical Metallurgy (15).

The transformations and processes that result in typical structures encompass basic mechanical characteristics. The transformation that produces solidification structures and solid-state transformation structures encompasses several mechanisms, the most important of which are: diffusion, nucleation, and growth; some more complex mechanisms are handled in martensitic and bainitic transformations (15).

The basic mechanical deformation includes mating and displacement and annealing processes, mainly recrystallization and grain growth processes by the mechanism of polygonization, nucleation, grain boundary growth and migration.

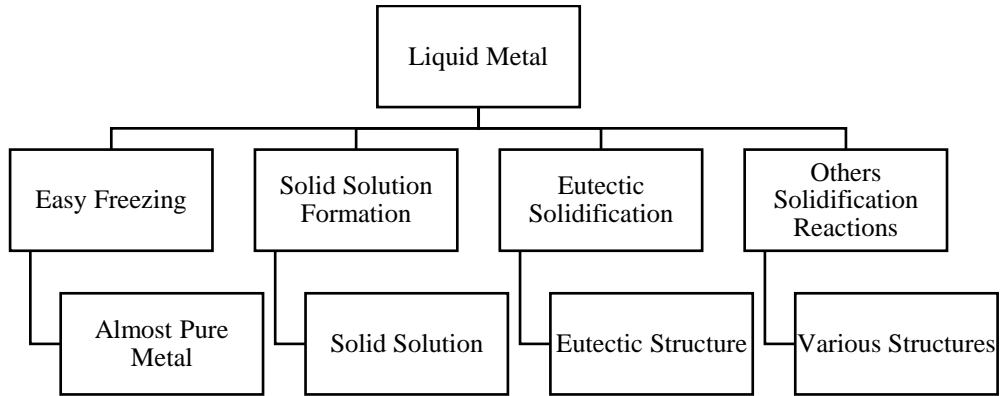
### **7.2 Single phase microstructure**

Micrographic examination aims to reveal the structure of the metal and its non-metallic inclusions through observation through the optical microscope.

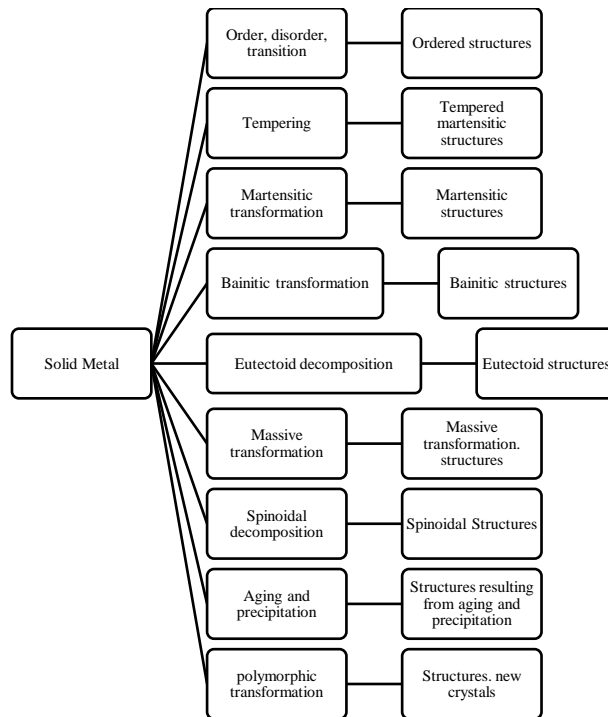
The definition of the microstructure of metallic materials is of overriding importance in the characterization of their composition and properties.

The main types are: solidification structures, solid state transformation structures, and annealing deformation structures, which are shown schematically in diagrams 1, 2, 3 (14, 20).

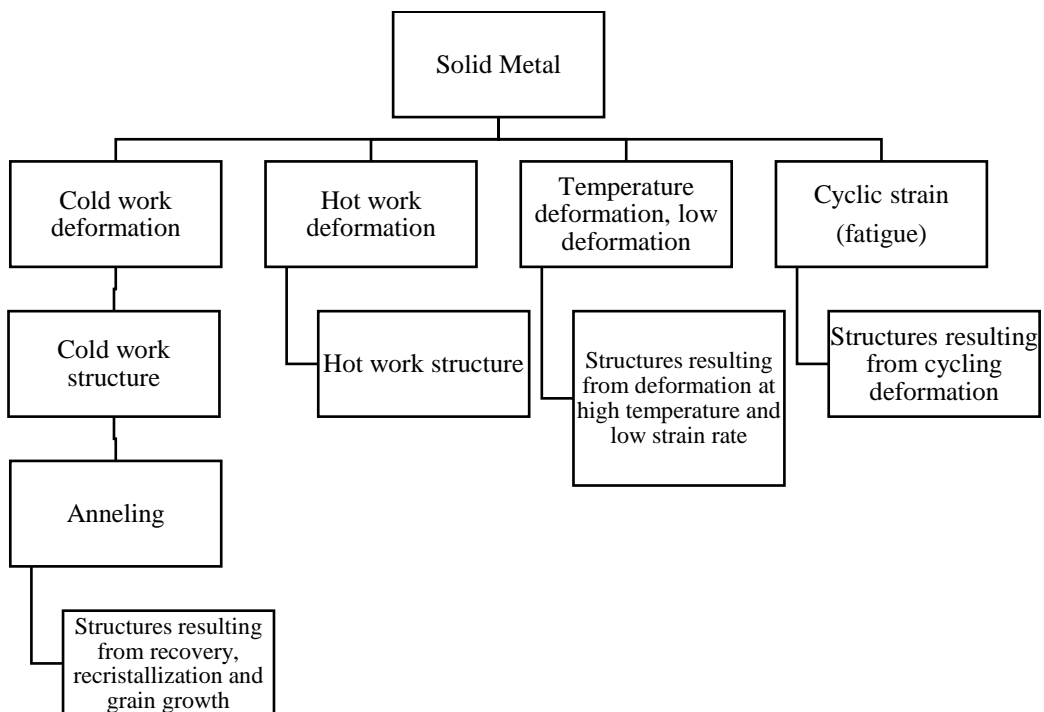
**Diagram 1 Solidification Structures**



**Diagram 2 Solid State Transformation Structures**



**Diagram 3 Annealing Deformation Structures**



The most notable structural features of single phase metals and alloys such as: grain structure and substructure are discussed below:

### 7.2.1 Grain structure

The grains are small crystals called crystallites that form a three-dimensional aggregate. The main characteristics of a grain structure are: grain size and anisotropic grain shape.

### 7.2.2 Types of grain structures

Typical grain structures include the following: affected structure, columnar structure, equiaxed grain structure, mature grain structure, deformed grain structure, inhibited recrystallization structure, and duplex grain structure (14, 18).

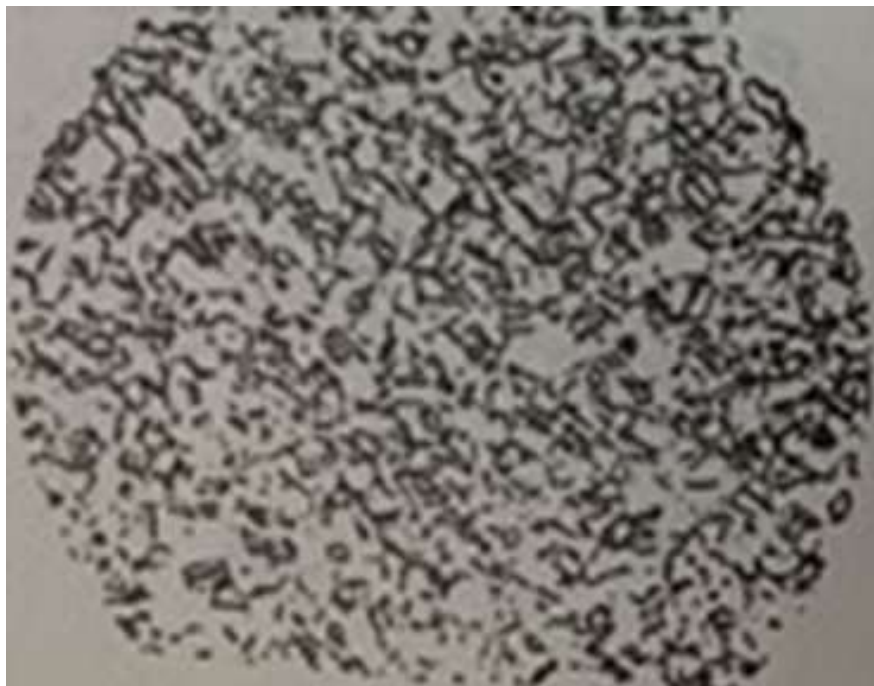
#### 7.2.2.1 Structure affected

This type of structure is formed when grains grow to clump together or affect each other producing broken interfacial features. This type of structure is very rarely seen because the interfaces are generally flattened while the specimen is still at high temperatures. Affected grains are seen after secondary recrystallization.

#### 7.2.2.2 Columnar structure

It is formed by processes in which there is unidirectional growth, especially during solidification, and also by aggregation processes including diffusion accompanied by a solid-state transformation. A columnar structure is shown in figures 10, 11 (14, 20).

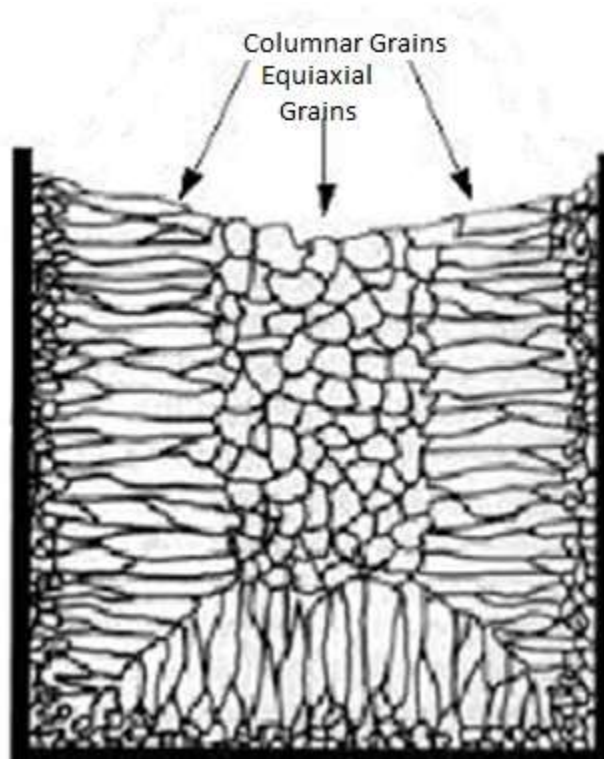
**Figure 10** Columnar structure Metals Handbook



#### 7.2.2.3 Equiaxed grain structure

This type of structure is formed in severe processes such as solidification and recrystallization Figure 12.

**Figure 11** Columnar and equiaxial grains



*Source: Own Elaboration*

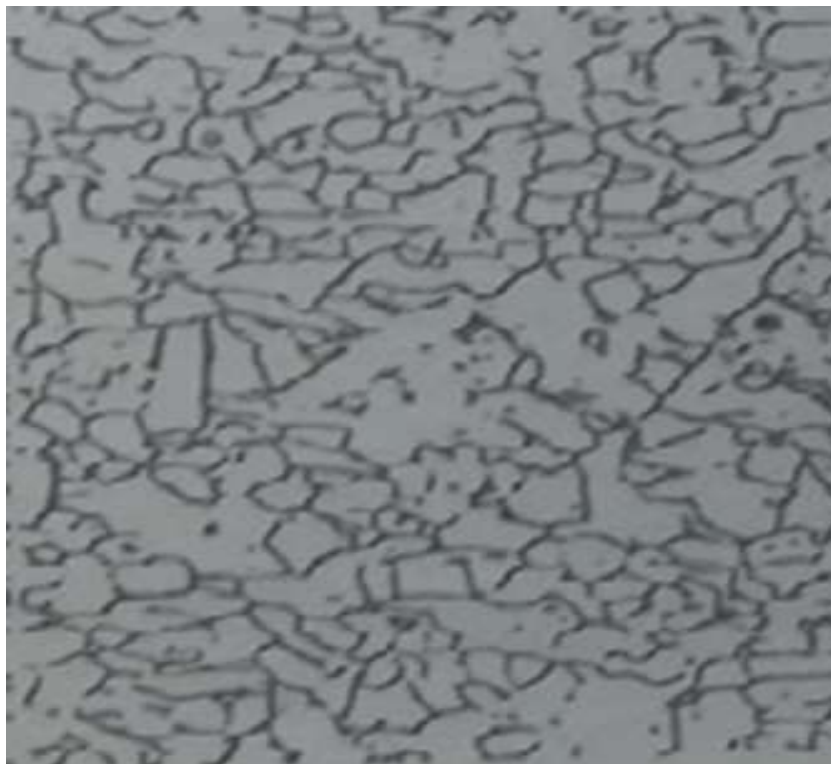
#### 7.2.2.4 Mature grain structure

It forms when interfaces (i.e. those resulting from grain affection) adjust under the force of surface tension action.

#### 7.2.2.5 Deformed grain structure

It is the result of cold working. In this structure the shape of the grains is anisotropic (Figs. 12-17).

**Figure 12** Steel 1008 to 250X, with 10% of reduction. Metals Handbook



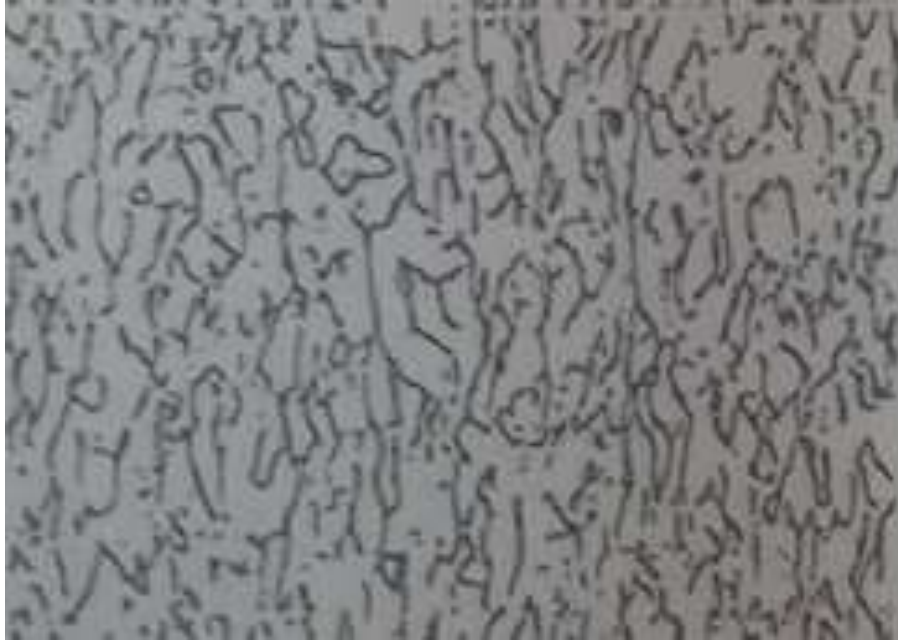
**Figure. 13** Steel1008 a 250X, with 20% of reduction. Metals Handbook



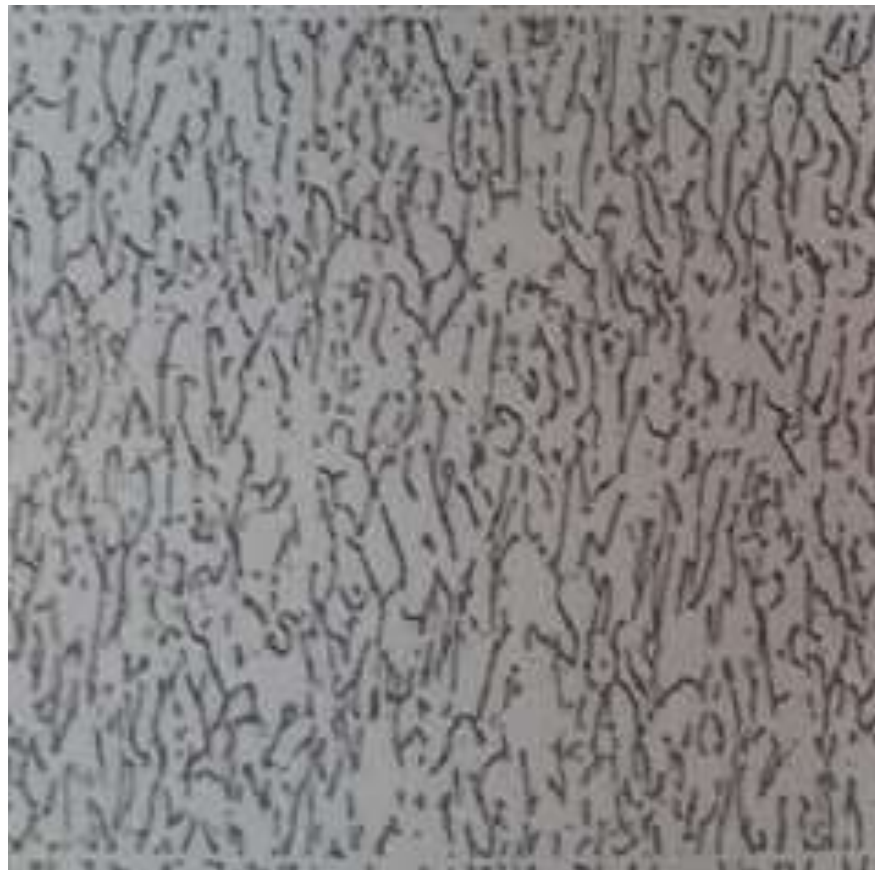
**Figure 14** Steel 1008 a 250X, with 30% of reduction. Metals Handbook



**Figure. 15** Steel 1008 a 250X, with 40% of reduction. Metals Handbook



**Figure. 16** Steel 1008 to 250X, with 50% of reduction. Metals Handbook





**Figure 17** Steel 1008 to 250X, with 60% of reduction by cold rolled. Metals Handbook



#### 7.2.2.6 Inhibited recrystallization structure

This type of microstructure is formed when the particles of secondary phases arrange themselves in a well-defined shape, preventing the movement of grain boundaries and imposing their specific shape on the resulting recrystallized structure (micrographs 18, 19, 20).

**Figure 18** Low carbon Steel to 100X. Coarse ferrite. Grain 5. Metals Handbook



**Figure. 19** Low carbon Steel to 100X. Grain 7. Metals Handbook



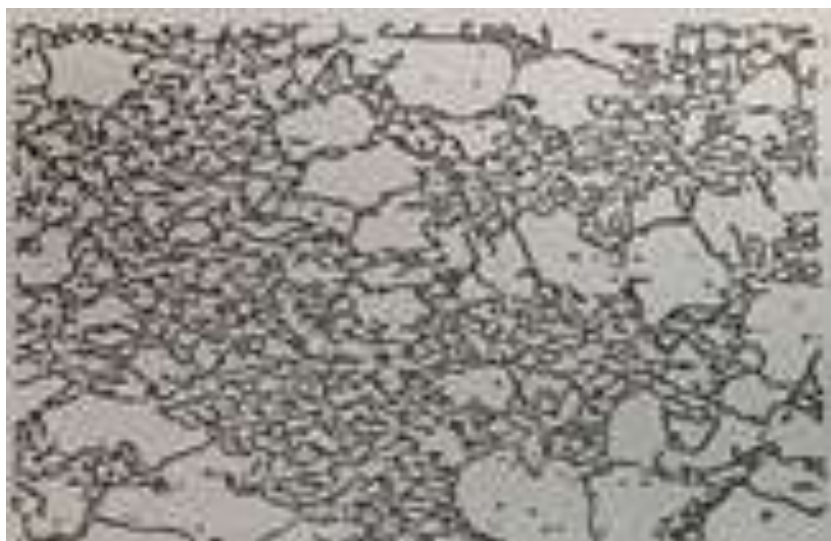
**Fig. 20** Low carbon Steel to 100X. Grain 9. Metals Handbook



#### 7.2.2.7 Duplex grain structure

It is one that consists of discrete regions with larger or smaller grain sizes (bimodal grain size distribution (Fig. 21).

**Figure. 21** Steel with 0.013% of carbon to 100X. Dúplex ferrite. Metals Handbook



### 7.2.2.8 Three-dimensional grain structure

The grain structure exists in three dimensions. In a typical structure when two grains are separated by an interface, three interfaces join along a line or edge, and four edges join at a join point. These joints can be connected in countless ways without structural symmetry or exact repetition of details.

### 7.3 Grain shape

The grain shape for some purposes can be approximated to that of a sphere when the grains are equiaxial Fig. 22 Similarly non-equiaxial grains can be represented by ellipsoids. (14, 18).

**Figure. 22** Equiaxed grains. Brass 260 to 75X. Size of grain 0.035 mm annealing to 482°C. Metals Handbook



## Section 8. Metallographic Practice Applicable to All Metals

### 8.1 Methodology

It is worth mentioning that many of the steps described here for the preparation of specimens for metallographic study can also be used for other types of metallographic tests such as: electron microscopy, microhardness test, quantitative measurement of structure constituents and microtest analysis. electronics.

A properly prepared sample is one that meets the following requirements:

- a. Representative sample. The size of the sample will depend on what you want to evaluate.
- b. Sectioning, encapsulation (which is optional) can be done cold with resin and catalyst, or hot by means of a pressure encapsulator.
- c. Polished. Free from scratches, pinholes, and liquid stains or surface oxidation, this operation should be done as much as necessary to minimize distortion or flow of metal on the surface, caused by mechanical deformation, and thereby allow the true microstructure to be revealed by etching. later. It must be done in such a way that non-metallic inclusions are considered intact.

In general, the preparation of samples for metallographic analysis requires 5 operations, which are described below:

### **8.1.1 Sectioned**

It is the operation by which a piece to be tested is sectioned to have a representative sample for the metallographic study, according to the characteristics that are to be evaluated; for example: for a deformation study, two sections are generally required; one perpendicular and one parallel to the major axis of the deformation direction. In the case of failed parts, they can be studied to find the cause of the failure; Selecting a specimen that intersects the origin of the fault zone, if the origin can be identified on the surface of the part, depending on the type of fault it may be necessary to take several samples from the fault area and from areas adjacent to the fault.

The sectioning operation can be done in three main ways:

#### **8.1.1.1 Due to Fracture**

Fracture tests can be done by means of controlled fractures, which can be produced by means of impact tests or tensile tests where the location of the fracture can be controlled by marking the material.

#### **8.1.1.2 By Sawing**

It is the oldest method used in the metallographic laboratory to section a material using a manual hacksaw. Saw blades are generally made of hard steel and are used to cut materials whose hardness is less than that of the saw blade or serrote. It is necessary to use oil or water-soluble oil as the cutting fluid to minimize frictional heating, which can soften or harden the tooth or age the microstructure of the specimen below the cut surface.

#### **8.1.1.3. Cut with abrasive disc**

Most of the cutting methods currently used in various laboratories is sectioning using abrasive disc cutting machines. All abrasive wheel cuts should be made wet using either water flow or a coolant (water soluble oil), which should flow directly into the cutting machine.

### **8.1.2 Mounting of Specimens**

The main purpose of mounting the specimens for the metallographic study is to facilitate their handling, especially those that, because they are so small in size, cannot be handled properly for their preparation and study. Another additional benefit of mounting is the ease of being able to identify by name, alloy number or laboratory code for storage, writing on the surface of the specimen without damaging the sample.

#### **8.1.2.1 Compression mounting**

This is the most common method of mounting and is done by heating and pressing with molding materials such as bakelite, dialkyl phthalate resins, and acrylic resins. Bakelite and phthalate are thermosets and acrylic resins are thermoplastics. Both thermoset and thermoplastic materials require heating and pressure during the molding cycle, but after curing, assemblies made from thermoset materials can be ejected from the mold at the maximum molding temperature, whereas thermoplastics remain molten at the maximum molding temperature, for which it is necessary to allow it to cool under pressure before expelling the sample from the mold.

#### **8.1.2.2 Materials for cold assembly**

These materials are classified as polyesters, epoxies and acrylics, it is worth mentioning that these materials need a hardener (catalyst). The mixture of resin and hardener is prepared in the proportion that the manufacturer recommends, the mixture is made and poured into the mold which can be of any desired size or shape, according to the needs of each particular laboratory. Sometimes you can make your own molds with aluminum foil around blocks of wood of the desired size, since the aluminum foil can easily come off, it can also be prepared with silicone rubber.

### 8.1.3 Abrasion

It is one of the most important operations in sample preparation. During roughing, the operator must eliminate mechanical damage to the surface of the specimen due to cutting, since even if sectioning is done carefully, severe damage to the surface results. Damage can be removed by prolonged roughing. Roughing is completed by polishing the surface through a sequence of operations with progressively finer abrasives, 40 to 150 grit size abrasives generally being considered as rough abrasives and 180 to 600 grit sizes as rough abrasives. fine abrasives.

### 8.1.4 Polished

It is the final step to produce a flat, scratch-free, mirror-like surface (a necessary condition for subsequent metallographic interpretation, either qualitative or quantitative).

The polishing technique should not introduce foreign structures, such as contaminating metals, holes, comet tails or stains.

#### 8.1.4.1 Mechanical polishing

This term is often used to describe the wide variety of final polishing procedures, including the use of cloth covered discs, and suitable polishing abrasives (alumina, diamond paste, magnesia). The disks have a rotating and vibrating movement, and the specimens are held manually or with automatic devices that allow polishing more than one specimen at the same time. Polishing must be done in a dust-free area, separate from the sectioning, assembly and grinding area, any contamination of a cloth by abrasive particles carried by a preceding operation or by dust, dirt or other material should not be tolerated. There are automatic polishers in which several specimens can be prepared automatically at the same time.

##### 8.1.4.1.1 Movement of the specimen

The specimen is held in one hand, or both, depending on the operator's preference, and rotated in the direction opposite to the rotation of the disk. In conclusion, the specimen continuously moves back and forth between the center and the corner of the disc, thereby ensuring distribution of the abrasive and uniform wear of the polishing cloth.

Some metallurgists use a small rotation while moving the specimen from the center to the corner of one side of the disk. The main reason for sample rotation is to prevent the formation of comet tails, a polishing defect that is illustrated in Figure 23.

**Figure. 23.** Steel 12L14 without attack to 100X. It shows Comet tails resulting from directional polishing. Metals Handbook



#### **8.1.4.1.2 Polishing pressure**

In general, the applied pressure is determined by experience, the specimen must be held firmly in the initial polishing steps with a certain pressure, which decreases proportionally as polishing progresses. For soft metals the required pressure is less.

#### **8.1.5. Washing and drying**

The sample should preferably be washed with running water, dried with methanol or any other alcohol that does not leave a residue, and finally dried in a stream of hot air. Alcohol in general can be used for washing when the abrasive used is not soluble in water. The above cleaning precautions must be strictly followed.

#### **8.1.6 Attack**

Metallographic attack comprises all the procedures used to reveal particular structural characteristics of a metal under study, which cannot be observed in the polished material alone. Examination of a polished material before etching is recommended as it may reveal some important aspects such as porosity, cracks and non-metallic inclusions. In some non-ferrous alloys the grain size can be revealed adequately, only with a well polished surface without etching, using polarized light, because the etching obscures the grain boundaries, whereas in other applications etching is necessary to reveal the grain structure.

The attacker is used for phase identification, pore attack, and for orientation studies. The principle of multiphase attack in alloys is based on attack preference (different solution proportions of the phases in the attack), or preferential staining of one or more phases, to differentiate discrepancies in chemical composition or orientation. However, in pure metals or in single-phase alloys, preferential attack is primarily a result of differences in grain orientation.

Before attack, all specimens must be inspected to verify that the polishing has been correct and thus avoid the presence of scratches, pores, polishing reliefs, comet tails, and inclusions caused by polishing. Chemical etching is carried out by immersion of the sample in the etching solution until the required structure is revealed. Said attack is carried out in Petri dishes or in other suitable containers provided with loose covers to prevent excessive evaporation of the solvent, especially in those solutions that contain alcohol.

Glass containers can be used for all attackers except those solutions containing Hydrofluoric Acid. For these, the container must be made of polyethylene or some other suitable material, with the help of tweezers or some other means of handling; the surface of the specimen is submerged on the attacker with some agitation to ensure that the attacker is in contact with the specimen at all times. During the attack most metals lose their brilliance thereby indicating that the attack has been made. In practice, one can guess by the degree of darkness of the surface when the attack should be considered finished. It is worth mentioning that the attack can also be carried out with a cotton saturated with the attack solution, passing it over the polished surface. Once the attack is complete, the surface is washed with running water and then with alcohol, dried with a current of hot air (like that of a manual hair dryer).

##### **8.1.6.1. Attack for macrostructure (Macroattack)**

Macroscopic examination differs from microscopic examination because it uses very low magnifications and is used for the investigation of defects and structure in a larger area, while for microscopic examination small areas are used and are observed at higher magnifications. The macroetching technique is used to reveal solidification structures, flow lines, segregations, changes in structure due to welding, general distributions, size of inclusions, porosity, ingot defects and manufacturing defects, as well as fissures or cracks. It is important for the researcher to be aware that macroetching can greatly exaggerate the size of differences and defects, which can lead to misinterpretations of the actual condition of the material, for which experience is needed to make decisions. Special care must be taken to control the attack time.

When the roughness of the surface of the specimen is high, a strong attack is necessary, instead a moderate attack should be used for surfaces that have a fine finish such as that obtained with a 400 grit abrasive. If after macro etching there are rough lines this indicates that a finer roughing should have been used. Virgin metal exposed to attack is generally susceptible to oxidation, so it is necessary to protect it with a finish of either clear oil, glycerol, or clear lacquer if surface preservation is to be desired.

#### **8.1.6.2 Attack for microstructure (microetching)**

When a sample has been properly prepared, microscopic examination will clearly reveal structural features such as grain size, segregation, shape and size, phase distribution, and inclusions that may be present. The microstructure also reveals the type of heat treatment the metal has received. The proper attack is just as important, as are the steps of sampling, sectioning, mounting, roughing and polishing.

For most applications the following attack rules should be followed.

- a. Attack for a fair time. This time should be sufficient to reveal significant details of the microstructure, as excessive etching will exaggerate or erase fine details of the material's structure.
- b. If you want to have an additional contrast as for micro photography, this should be obtained by means of photographic techniques or (in some metaloscopes sets of lenses are integrated which give the desired contrast), instead of using an over attack which we it is convenient.
- c. When a specimen has been insufficiently etched it should be re-polished to remove the etching from the surface (repeating the last polishing step is usually sufficient) and re-etched, as re-etching without re-polishing generally produces poor results.
- d. Low magnification examination generally requires a higher etching than that required for higher magnification examination.
- e. After etching the surface of the specimen shall not come into contact with anything. It should be examined or photographed immediately before it rusts or becomes contaminated.

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#### **Conclusions**

1. This chapter will serve for the Engineering student to acquire the fundamental knowledge to carry out the preparation of specimens for the metallographic characterization of materials, which is directly related to their physical and chemical properties.
2. Quality assurance in all manufacturing processes requires a metallographic study to determine compliance with the parameters established in the regulations applicable to each type of material, hence the importance of metallography.
3. This introductory study to Metallography will serve as the basis for other specific studies later.

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