RESEARCH PROJECT

PAPER TITLE: MICROSTRUCTURAL CHARACTERIZATION OF THE SINGLE-PHASE HIGH STRENGTH STEEL

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CHARACTERIZATION

When used in materials science, refers to the broad and general process by which a material's structure and properties are probed and measured. It is a fundamental process in the field of materials science, without which no scientific understanding of engineering materials could be ascertained. The scope of the term often differs; some definitions limit the term's use to techniques which study the microscopic structure and properties of materials, while others use the term to refer to any materials analysis process including macroscopic techniques such as mechanical testing, thermal analysis and density calculation.

CHARACTERIZATION OF MATERIAL

WHAT IS CHARACTERIZATION OF MATERIALS?

1. Characterization refers to the process by which a material's structure and properties are determined.
2. It is a fundamental process without which no scientific understanding of engineering materials could be established.

WHAT IS ITS SIGNIFICANCE?

1. It helps to increase the different degree of understanding of materials showing different properties and behaviour.
2. It helps to analyse and optimise the materials.

CHARACTERIZATION TECHNIQUES

Characterization techniques are essential tools in materials science that allow scientists and engineers to probe and measure the properties of various materials. These techniques provide valuable information about the chemical, physical, mechanical, and electrical characteristics of materials. Here are some key aspects of characterization techniques:

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1. *Microscopy Testing*

- Microscopy involves probing and mapping the surface and sub-surface structures of materials. It uses various types of radiation (such as photons, electrons, and ions) or physical probes to gather data about a sample's structure on different length scales.
- Common microscopy techniques include:

Optical microscopy: Uses visible light to observe samples.

Stereo microscope

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Optical microscopy, also known as **light microscopy**, is a fundamental technique used to closely examine samples by magnifying them using visible light and a system of lenses. Let's delve into the details:

1. **Basic Principles**:
   - ❖ **Light Source**: Visible light is either transmitted through or reflected from the sample.
   - ❖ **Lenses**: A single lens or multiple lenses (in compound microscopes) magnify the image.
   - ❖ **Detection**: The resulting image can be directly observed through the eyepiece, captured on a photographic plate, or digitally imaged.

2. **Components of an Optical Microscope**:
   - ❖ **Objective Lens**: The primary lens closest to the sample, responsible for magnification.
   - ❖ **Eyepiece (Ocular)**: The lens through which the viewer observes the magnified image.
   - ❖ **Stage**: Where the sample is placed for examination.
   - ❖ **Light Source**: Illuminates the sample.
   - ❖ **Condenser**: Focuses and directs light onto the sample.
   - ❖ **Diaphragm**: Controls the amount of light passing through the sample.
   - ❖ **Focus Adjustment**: Allows precise focusing on different layers of the sample.
   - ❖ **Mechanical Stage**: Enables movement of the sample for scanning.
   - ❖ **Coarse and Fine Focus Knobs**: Adjust focus rapidly or finely.
   - ❖ **Camera Port**: Used for digital imaging.

3. **Sample Illumination**:
   - ❖ **Bright Field Illumination**: Light passes through the sample from below, providing contrast.
   - ❖ **Dark Field Illumination**: Light comes from around the objective lens, highlighting edges and surface irregularities.
   - ❖ **Polarized Light**: Reveals crystal orientation in metallic objects.
   - ❖ **Phase-Contrast Imaging**: Enhances contrast by emphasizing refractive index differences.

4. **Magnification and Resolution**:
   - ❖ Optical microscopes achieve magnifications up to around 1000x due to the limited resolving power of visible light.

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❖ Larger magnifications are possible but do not reveal additional details of the object.
❖ Alternatives like electron microscopes (e.g., scanning electron microscopy) achieve much greater magnifications.

In summary, optical microscopy remains a powerful tool for examining biological specimens, materials, and various samples in research, education, and industry.

Scanning electron microscopy (SEM): Provides high-resolution images of surfaces.

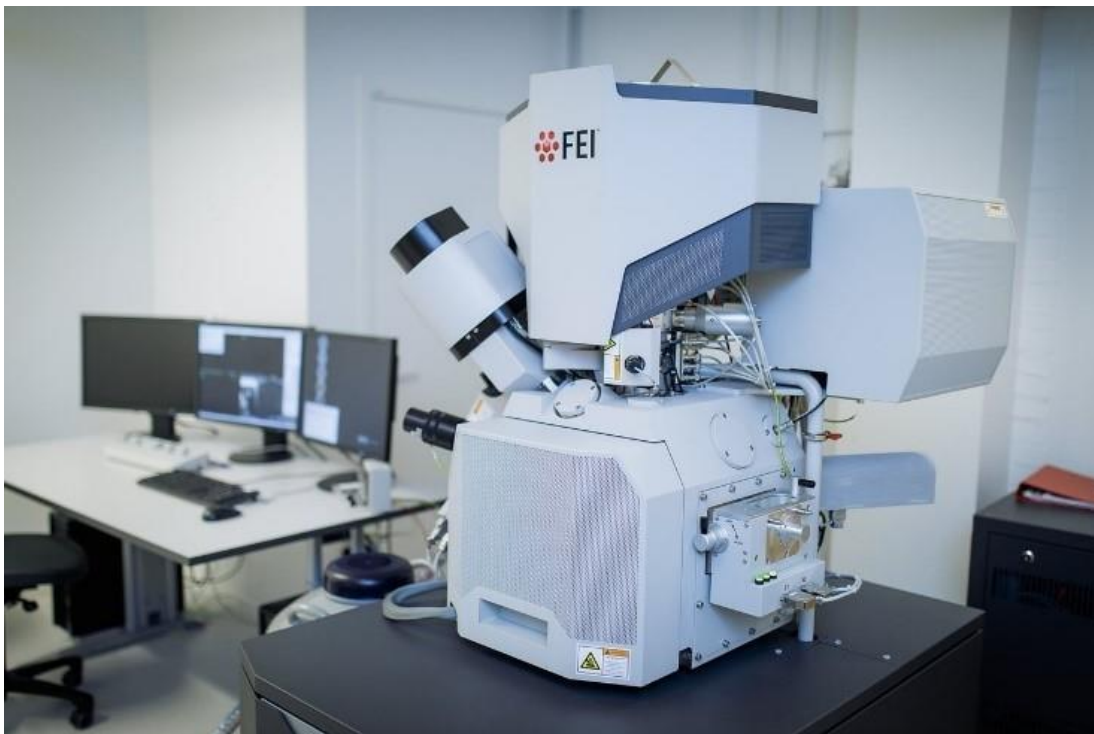**Fig. Scanning Electron Microscope**

1. Basic Principles
❖ SEM is a type of electron microscope that produces detailed images of a sample's surface.
❖ Unlike optical microscopes that use visible light, SEM relies on a focused beam of electrons.

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❖ Here's how it works:
1. An electron beam scans the sample in a raster pattern.
2. As the beam interacts with the sample, secondary electrons are emitted from atoms excited by the electron beam.
3. These secondary electrons are detected using a secondary electron detector (such as the Everhart–Thornley detector).
4. The position of the beam and the intensity of the detected signal are combined to create the SEM image.

2. Parts of a Scanning Electron Microscope (SEM)

❖ **Electron Source**:
  ➤ Produces the electron beam (usually from a filament).
  ➤ The beam is then focused on the sample surface using a set of lenses in the electron column.
❖ **Sample Chamber and Stage**:
  ➤ Holds the sample during imaging.
  ➤ The stage allows precise positioning of the sample.
❖ **Electron Column**:
  ➤ Contains electromagnetic lenses that focus and control the electron beam.
  ➤ Includes condenser lenses, objective lenses, and scanning coils.
❖ **Secondary Electron Detector**:
  ➤ Detects secondary electrons emitted from the sample surface.
  ➤ Provides contrast information for imaging.
❖ **Vacuum System**:
  ➤ SEM operates in a high vacuum to prevent electron scattering.
  ➤ Some SEMs allow imaging in low vacuum or wet conditions.

3. Applications of SEM

❖ **Materials Science**: Studying material surfaces, defects, and microstructures.
❖ **Nanotechnology**: Investigating nanostructures and nanoparticles.
❖ **Biology**: Imaging biological samples (cells, tissues, etc.).
❖ **Geology**: Analysing mineral structures and rock surfaces.
❖ **Forensic Science**: Examining trace evidence and forensic samples.

4. Advantages of SEM

❖ High resolution (better than 1 nanometre).
❖ Detailed surface information.

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❖ Versatility (imaging various materials).

5. Limitations of SEM
❖ Surface sensitivity (only top few nanometres).
❖ Sample must be conductive or coated.
❖ Vacuum requirements.

In summary, SEM provides valuable insights into material surfaces, making it an indispensable tool in scientific.

Transmission electron microscopy (TEM): Allows imaging of thin sections of materials.

Fig. Transmission Electron Microscope

1. Principle of Transmission Electron Microscopy (TEM)
The working principle of TEM is similar to that of a light microscope, but with a crucial difference: TEM uses a beam of electrons instead of visible light. Here's how it works:

1. **Electron Beam**:
   ➢ In TEM, a beam of high-energy electrons is generated using an **electron gun** (usually heated tungsten or LaB6 filament).

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➤ These electrons are accelerated to high voltages (typically 60-120 kV).

2. **Interaction with the Specimen**:
   ➤ The electron beam is focused onto a **thin specimen** (usually less than 100 nm thick) placed in the path of the beam.
   ➤ As the electrons pass through the specimen, they interact with its atoms and structures.

3. **Image Formation**:
   ➤ The transmitted electrons form an **image** based on their interactions with the specimen.
   ➤ The image is then magnified and focused onto an imaging device (such as a fluorescent screen, photographic film, or a digital detector).

4. **Resolution**:
   ➤ Electrons have a much shorter wavelength than visible light (about 0.005 nm compared to hundreds of nanometres).
   ➤ This short wavelength allows TEM to achieve **exceptional resolution**—up to 1000 times better than light microscopes.
   ➤ TEM can reveal details at the **atomic scale**, including individual columns of atoms and crystal structures.

5. **Contrast Mechanisms**:
   TEM provides various contrast mechanisms:
   ➤ **Mass-thickness contrast**: Differences in specimen thickness or density.
   ➤ **Z contrast**: Variation in atomic number (composition).
   ➤ **Crystallographic contrast**: Reveals crystal structures.
   ➤ **Phase contrast**: Detects quantum-mechanical phase shifts caused by individual atoms.
   ➤ **Spectrum imaging**: Measures energy loss by electrons.

2. Applications of TEM

TEM is a powerful analytical tool used in various scientific fields:
➤ **Materials Science**: Characterizing materials at the nanoscale.
➤ **Biology**: Studying cellular structures, viruses, and biomolecules.
➤ **Nanotechnology**: Investigating nanoparticles and nanomaterials.
➤ **Virology**: Visualizing viruses and their components.
➤ **Semiconductor Research**: Analysing semiconductor devices.
➤ **Paleantology and Palynology**: Examining fossil structures.

3. Advantages and Limitations

- ❖ **Advantages**:
  - ➢ High resolution (down to atomic levels).
  - ➢ Detailed structural information.
  - ➢ Versatility (multiple operating modes).
  - ➢ Reveals composition and crystallography.
- ❖ **Limitations**:
  - ➢ Requires thin specimens.
  - ➢ Complex sample preparation.
  - ➢ Vacuum environment.
  - ➢ Beam damage to sensitive samples.

In summary, TEM is an essential tool for nanoscience, providing unprecedented insights into the smallest structures of matter.

Fourier transform infrared spectroscopy (FTIR):

Analyses molecular vibrations.

Fourier Transform Infrared Spectroscopy (FTIR) is a powerful analytical technique used to obtain an infrared spectrum of absorption or emission from a solid, liquid, or gas. Let's explore the principles behind FTIR and its applications:

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1. Principle of FTIR Spectroscopy

❖ FTIR hinges on measuring how different **chemical bonds** in a molecule absorb infrared light.

❖ Each type of bond and its environment exhibit specific absorption patterns, providing a unique **molecular fingerprint**.

❖ Rather than shining a monochromatic beam of light (composed of only one wavelength) at the sample, FTIR shines a beam containing **multiple frequencies of light** simultaneously.

❖ The sample absorbs different combinations of frequencies, yielding a series of data points.

❖ A computer then works backward using a **Fourier transform** to infer the absorption at each wavelength.

❖ The process involves:

➤ Starting with a **broadband light source** containing the full spectrum of wavelengths.

➤ Passing the light through a **Michelson interferometer**, which periodically blocks and transmits different wavelengths due to wave interference.

➤ Collecting an **interferogram**—a plot of intensity versus time.

➤ Applying the Fourier transform to convert the raw data into the actual **infrared spectrum**.

2. Advantages of FTIR

❖ Non-destructive technique: Samples remain intact.

❖ Precise measurement without external calibration.

❖ Rapid data collection (scans every second).

3. Applications of FTIR

❖ **Identifying Functional Groups**:

➤ Determines the presence of organic and inorganic compounds based on their absorption patterns.

➤ Useful in chemistry, materials science, and pharmaceutical analysis.

❖ **Quality Control and Purity Assessment**:

➤ Ensures product consistency and purity.

➤ Detects impurities or deviations from expected compositions.

❖ **Biomolecular Research**:

➤ Studies proteins, nucleic acids, lipids, and carbohydrates.

➤ Provides insights into biological structures and interactions.

❖ **Environmental Monitoring**:
  ➢ Analyses pollutants, gases, and atmospheric components.
  ➢ Helps assess air and water quality.

In summary, FTIR spectroscopy offers a powerful tool for understanding molecular structures and compositions.

X-ray diffraction (XRD): Determines elemental composition and chemical states.
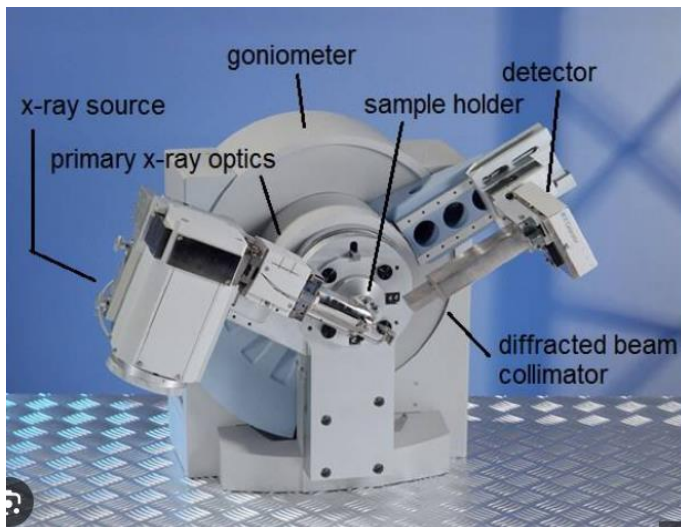
X-ray Diffraction (XRD) is a powerful analytical technique primarily used to identify the atomic and molecular structure of a crystal. The basic principle behind XRD is the diffraction of X-rays by the orderly array of atoms in a crystal, which produces a pattern characteristic of the crystal structure.

1. Principles of XRD

❖ *Bragg's Law*: The fundamental principle that underpins XRD is Bragg's Law, which relates the angle of incidence of X-rays to the spacing between crystal planes. It is given by the equation:

$$n\lambda = 2d\sin\theta$$

where n is an integer (the order of the diffraction),
λ is the wavelength of the incident X-ray,
d is the distance between the crystal planes, and
θ is the angle of incidence.

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❖ *Diffraction Pattern*: When X-rays are directed at a crystal, they are diffracted in specific directions determined by the crystal's structure. The resulting pattern of diffracted rays can be detected and analyzed to infer the crystal structure.

2. Components of an XRD Instrument

❖ *X-ray Source*: Typically, a copper (Cu) target is used to produce X-rays at a characteristic wavelength (Cu Kα radiation, λ ≈ 1.54 Å).
❖ *Sample Holder*: The crystalline sample is placed in the path of the X-rays.
❖ *Detector*: This captures the diffracted X-rays and measures their intensity and angle of diffraction.
❖ *Goniometer*: This is a precision instrument that allows the sample and detector to be rotated to various angles.

3. Procedure

❖ *Sample Preparation*: The sample must be ground to a fine powder to ensure that many crystals are randomly oriented.
❖ *Data Collection*: The X-ray source irradiates the sample, and the detector measures the intensity of the diffracted X-rays as a function of the angle 2θ.
❖ *Data Analysis*: The collected data is used to generate a diffraction pattern, typically a graph of intensity versus 2θ. Peaks in the diffraction pattern correspond to the planes in the crystal lattice.

4. Applications

❖ *Phase Identification*: XRD can identify the phases present in a material by comparing the diffraction pattern to standard reference patterns.
❖ *Crystallinity*: XRD can determine the degree of crystallinity in a sample.
❖ *Lattice Parameters*: It can measure the lattice parameters and symmetry of a crystal.
❖ *Stress Analysis*: XRD can be used to measure residual stresses within a crystalline material.
❖ *Texture Analysis*: XRD can study the orientation distribution of crystallites within a polycrystalline sample.

5. Advantages

❖ *Non-destructive*: XRD does not alter the sample.
❖ *Quantitative*: Provides detailed information about crystal structure, phase composition, and other structural properties.
❖ *Versatile*: Applicable to a wide range of materials, including metals, minerals, polymers, and ceramics.

6. Limitations

❖ *Sample Requirement*: The sample must be crystalline.

- ❖ *Complexity*: Interpretation of XRD patterns can be complex and requires expertise.
- ❖ *Size Limitation*: May not be suitable for very small or very thin samples due to weak diffraction signals.

Overall, XRD is an essential tool in materials science, chemistry, geology, and various other fields for analyzing crystalline materials and understanding their structural properties.

1. *Macroscopic Testing*

- ❖ Macroscopic techniques study bulk properties of materials. Some examples include:
  - ➢ **Mechanical testing**: Evaluates tensile strength, hardness, toughness, and other mechanical properties.
  - ➢ **Differential thermal analysis (DTA)**: Measures heat flow during temperature changes.

Mechanical testing is a crucial aspect of materials science and engineering. It involves evaluating the mechanical properties of materials to understand their behaviour under various conditions. Let's explore different types of mechanical testing methods:

1. **Tensile Testing**:
   - ❖ **Purpose**: Determines material properties such as tensile strength, yield strength, and modulus of elasticity.
   - ❖ **How it works**: A sample is subjected to axial tension until it fractures. The stress-strain curve provides valuable information.
   - ❖ **Application**: Used for quality control, material selection, and design validation.
2. **Compression Testing**:
   - ❖ **Purpose**: Measures compressive strength and deformation behaviour.
   - ❖ **How it works**: The sample is compressed along its axis until failure occurs.
   - ❖ **Application**: Assessing materials used in columns, pillars, and structural components.
3. **Flexural Testing (Bend Test)**:
   - ❖ **Purpose**: Evaluates the bending strength and stiffness of materials.

- ❖ **How it works**: A sample is subjected to bending forces, and the resulting deformation is measured.
- ❖ **Application**: Assessing materials for beams, bridges, and other structures.

4. **Hardness Testing**:
   - ❖ **Purpose**: Determines a material's resistance to indentation or scratching.
   - ❖ **Methods**: Common techniques include Vickers, Brinell, and Rockwell hardness tests.
   - ❖ **Application**: Assessing wear resistance, material selection, and heat treatment effectiveness.

5. **Impact Testing**:
   - ❖ **Purpose**: Measures a material's toughness and ability to absorb energy during sudden loading.
   - ❖ **Methods**: Charpy V-notch and Izod tests are commonly used.
   - ❖ **Application**: Assessing materials for safety-critical components.

6. **Fatigue Testing**:
   - ❖ **Purpose**: Determines a material's resistance to cyclic loading.
   - ❖ **How it works**: The sample undergoes repeated loading cycles until failure.
   - ❖ **Application**: Designing components subjected to repetitive stress.

7. **Fracture Mechanics Testing**:
   - ❖ **Purpose**: Investigates crack propagation and fracture behaviour.
   - ❖ **How it works**: Analyses the critical stress intensity factor (K_{IC}) and fracture toughness.
   - ❖ **Application**: Assessing materials in critical structures.

8. **Creep Testing**:
   - ❖ **Purpose**: Studies material deformation under constant load at elevated temperatures.
   - ❖ **How it works**: The sample experiences long-term deformation.
   - ❖ **Application**: Evaluating materials for high-temperature applications.

9. **Shear Testing**:
   - ❖ **Purpose**: Measures shear strength and deformation.
   - ❖ **How it works**: The sample is subjected to shear forces.
   - ❖ **Application**: Assessing materials in riveted joints, adhesives, and welds.

10. **Peel Testing**:
    - ❖ **Purpose**: Evaluates the bond strength between two adhered materials.
    - ❖ **How it works**: Measures the force required to peel them apart.
    - ❖ **Application**: Assessing adhesive bonds.

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Mechanical testing ensures the safety, reliability, and performance of materials in various applications. Whether it's designing a bridge, selecting materials for aerospace components, or ensuring product quality, mechanical testing plays a vital role.

Differential Thermal Analysis (DTA) is a thermoanalytic technique widely used in material science, chemistry, and engineering to measure temperature differences between a sample and a reference under controlled conditions. Let's explore the basic principles and applications of DTA:

1. Principle of Differential Thermal Analysis (DTA)

❖ In DTA, the material under study and an inert reference undergo identical thermal cycles (i.e., the same cooling or heating program).
❖ The key parameter monitored is the **temperature difference (ΔT)** between the sample and the reference.
❖ As the sample experiences phase transitions (such as melting, crystallization, or sublimation), the heat absorbed or emitted by the chemical system is determined.
❖ The DTA curve (or thermogram) plots this differential temperature against time or temperature.

2. Instrumentation and Measurement

❖ **Sample Holder and Thermocouples**:
  ➢ A DTA setup includes a sample holder, thermocouples, sample containers, and a ceramic or metallic block.
  ➢ Two thermocouples are connected to a voltmeter:
  ➢ One thermocouple is placed in an inert material (e.g., α-alumina).
  ➢ The other is placed in the sample being analyzed.
  ➢ When the sample undergoes a phase transition, there is a brief deflection of the voltmeter due to latent heat incorporation.
❖ **Inert Environment**:
  ➢ DTA is performed in an inert environment using gases like helium or argon.
  ➢ These gases do not react with the sample or reference.

2. Applications of DTA

❖ **Phase Diagrams**:
  ➢ DTA helps determine phase transitions, such as glass transitions, melting points, and crystallization.

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➢ It provides data on transformations occurring in the material.

❖ **Heat Change Measurements**:
   ➢ DTA quantifies the enthalpy change associated with phase transitions.
   ➢ The area under a DTA peak corresponds to the enthalpy change.

❖ **Decomposition Studies**:
   ➢ DTA is used to study decomposition reactions in various atmospheres.
   ➢ It provides insights into stability and thermal behaviour.

❖ **Industrial Applications**:
   ➢ DTA finds applications in the pharmaceutical, food, and cement industries.
   ➢ It helps characterize materials and assess their suitability for specific processes.

In summary, Differential Thermal Analysis (DTA) is a valuable tool for understanding thermal behaviour, phase.

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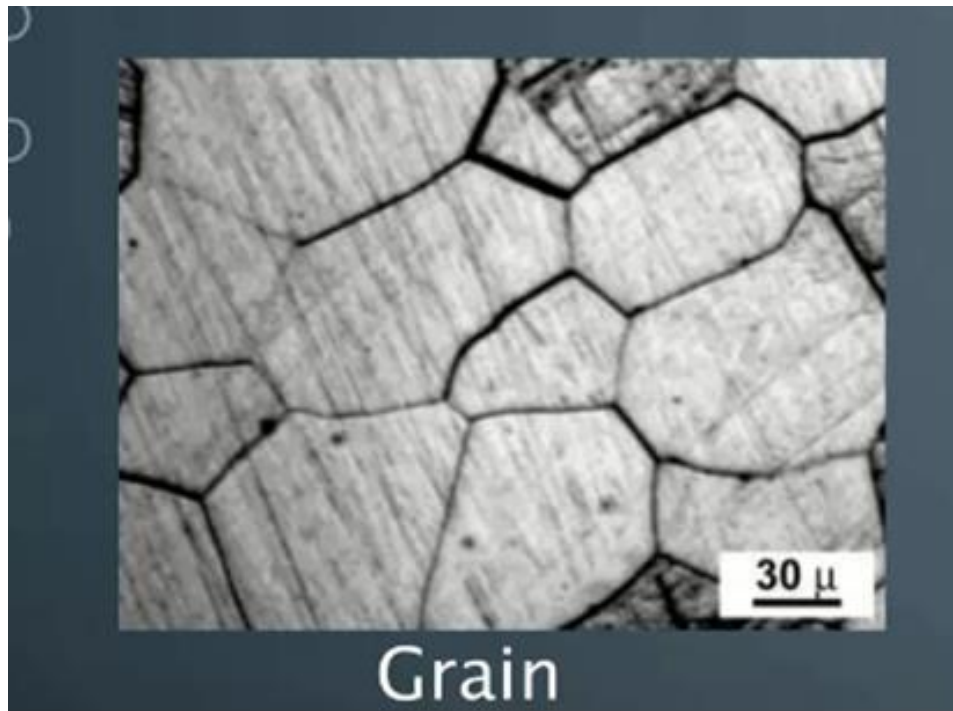
STRENGTHENING MECHANISMS OF METALS AND ALLOYS

✓ WHY STRENGTHENING IS NEEDED……….

- ❖ In pure state of some metals, the strength is not sufficient for specific engineering works.
- ❖ All the metals have movable internal dislocations due to stresses associated with the forming process.
- ❖ Movement of these dislocations cause plastic deformation.
- ❖ By avoiding the movement of dislocations, metals can be strengthened.

✓ Variation of deformation process with temperatures. [Tm is the absolute melting point of the metal]

- ❖ T > 0.5 Tm – Hot working
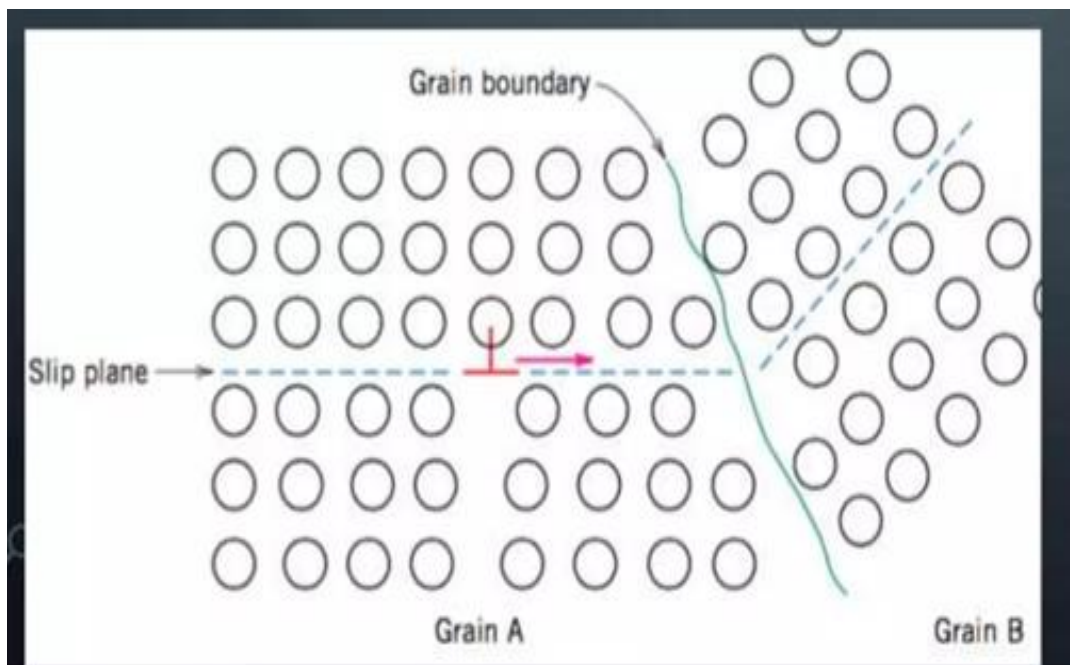- ❖ T ~ 0.5 Tm - Warm working
- ❖ T < 0.5 Tm – Cold working

✓ MAIN TYPES OF STRENGTHENING METHODS:

1. Grain-size reduction
2. Solid-solution alloying
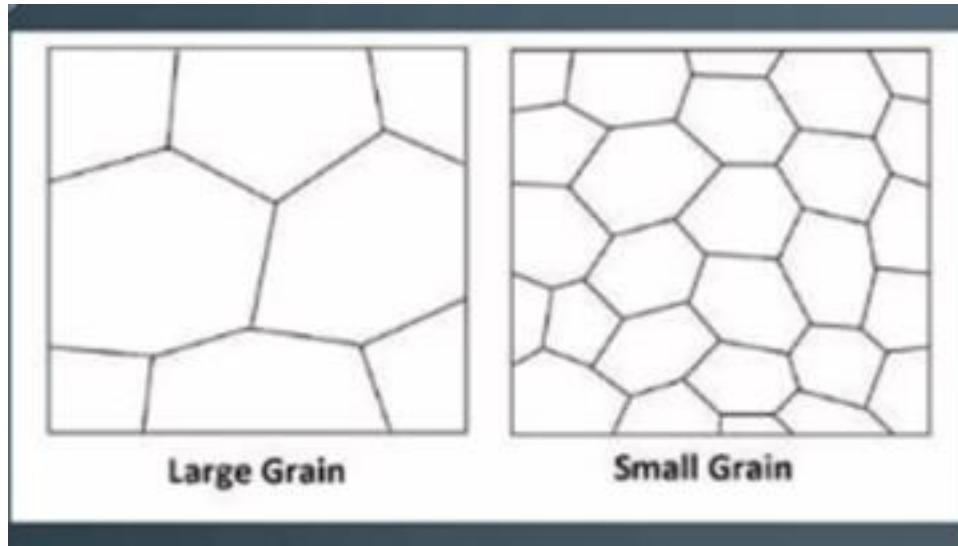3. Strain hardening (work hardening or cold working)
4. Annealing of deformed metals

GRAIN SIZE REDUCTION: -

Grain

❖ Grains are not well oriented in polycrystalline materials as single crystals.

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❖ Barrier to dislocation movement. [Changes or stops the path of the dislocations]

Large Grain   Small Grain

❖ By increasing the number of grains, the number of barriers to the dislocations also increases. Therefore, by reducing grain size, the number of barriers can be increased.

❖ High- angle grain boundaries block slip and increases strength of the materials. Because the pressure of the slip is stopped by exerting much force by large angles.

HALL – PETCH EQUATION

$$\sigma_{yp} = \sigma_o + \frac{k}{\sqrt{d}}$$

where,

σ_{YP} = *Yield strength* σ_0 = *constant for a particular material*

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k = constants for a particular material d = is the average grain diameter

❖ Grain size(d) varying parameters are Rate of solidification and Appropriate heat treatment.
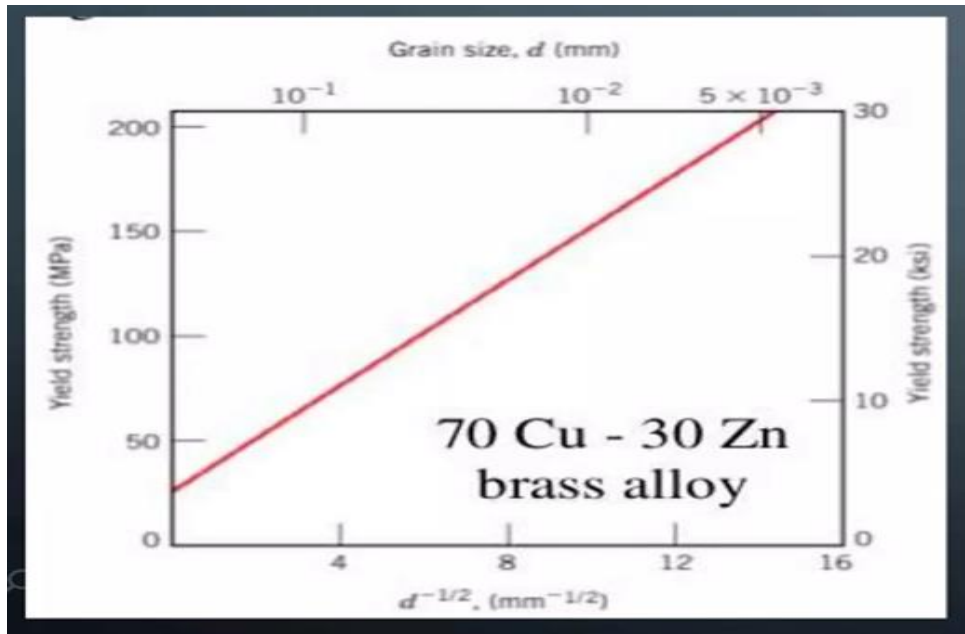
❖ According to the graph, the yield strength is increased with the reduction of grain size. Because of reduction, the number of grains are increased.

GRAIN SIZE ANALYSIS

ASTM grain size number – The ASTM grain size number, G, was originally defined as:

$$N_{AE} = 2^{G-1}$$

Where N_{AE} is the number of grains per square inch at 100X magnification. To obtain the number per square millimetre at 1X multiply by 15.50

Grain – That area within the confines of the original boundary observed on the two-dimensional plane of polish or that volume enclosed by the original boundary in the 3D object.

METHODS OF GRAIN SIZE ANALYSIS

1. COMPARISON METHOD – The comparison procedure does not require counting of either grains intercepts or intersection but as the name suggests involves comparison of the grain structure to a series of graded images, either in the form of a wall chart, clear plastic overlays or an eyepiece reticle.

2. PLANIMETRIC METHOD – The planimetric method involves an actual count of the number of grains within a known area. The number of grains per unit area N_A, is used to determine the ASTM grain size number G.

3. INTERCEPT METHOD- The intercept method involves an actual count of the number of grains intercepted by a test line or the number of grain boundary intersections with a test line, per unit length of test line, used to calculate the mean linear intercept length $\bar{l}$, $\bar{l}$ is used to determine the ASTM grain size number G.

COMPARISON METHOD

The comparison procedure shall apply to completely recrystallized or cast materials with equiaxed grains. The estimation of microscopically determined grain size should usually be made by direct comparison at the same magnification as the appropriate chart. Good judgement is necessary for accurate result.
Grain size estimation shall be made on three or more representative areas of each specimen section.

When the grains are of a size outside the range covered by the standard photographs, or when magnification of 75X or 100X are not satisfactory, other magnifications may be employed for comparison.
If the grain size is reported in ASTM numbers, it is convenient to use the relationships:

$$Q = 2 \log_2 (M/M_b)$$
$$= 6.64 \log_{10} (M/M_b)$$

Where Q is a correction factor that is added to the apparent micro-grain size of the specimen, as viewed at the magnification, M, instead of at the basic magnification, M_b (75X or 100X), to yield the true ASTM grain size.

PLANIMETRIC METHOD

In a planimetric method inscribed a circle or rectangle of known area (usually 5000mm^2 simplify the calculation). On a micrograph for the ground glass screen of the metallograph, select a magnification which will give at least 50 grains in the field to be counted.

Count the number of grains within this area. The sum of all grains included, completely within the known area plus one half the number of grains intersected by the circumference of the area. Gives the number of grains within the area.

If this number is multiplied by the jeffries multiplier f, the product will be the number of grains per square millimetre.

The number of grains per square millimetre at 1X, N_A, is calculated from:

$$N_A = f\,(N_{inside}+(N_{intercepted}/2))$$

Where f is the Jeffries' multiplier, N_{inside} is the number of grains completely inside the test circle and $N_{intercepted}$ is the number of grains that intercept the test circle. The average grain area A is the reciprocal of N_A, that is $1/N_A$.

Ignoring the four corner grains, a count is made of N_{inside}, the grains completely within the box, and of $N_{intercepted}$, the grains intercepted by the four sides of the box now becomes :

$$N_A = (M^2/A)\,(N_{inside} + 0.5N_{intercepted} +1)$$

Where M is the magnification, A is the test figure area in mm^2.

INTERCEPT METHOD

The intercept method involves an actual count of the number of grains intercepted by a test line or the number of grain boundary intersections with a test line per unit length of the test line, used to calculate the mean lineal intercept length, $\bar{l}$, $\bar{l}$ is used to determine the ASTM grain size number, G.

Estimate the average grain size by counting (on the ground glass screen, on the photomicrograph of a representative field of the specimen, a monitor or on the specimen

itself) the number of grains intercepted by one or more straight lines sufficiently long to yield at least 50 integers.

An intercept is a segment of test line overlaying one grain. An intersection is a point where a test line is cut by a grain boundary.

Either may be counted, with identical results in a single phase material.

When counting intercepts, segments at the end of a test line which penetrate into a grain are scored as half intercepts.
When counting intersections, the end points of a test line are not intersections and are not counted except when the end appears to exactly touch a grain boundary, when ½ intersection should be scored.

An intersection apparently coinciding with the junction of three grains should be scored as 1, 1/2.

The effects of moderate departure from an equiaxed structure may be eliminated by making intercept counts on a line array containing lines having four or more orientations.
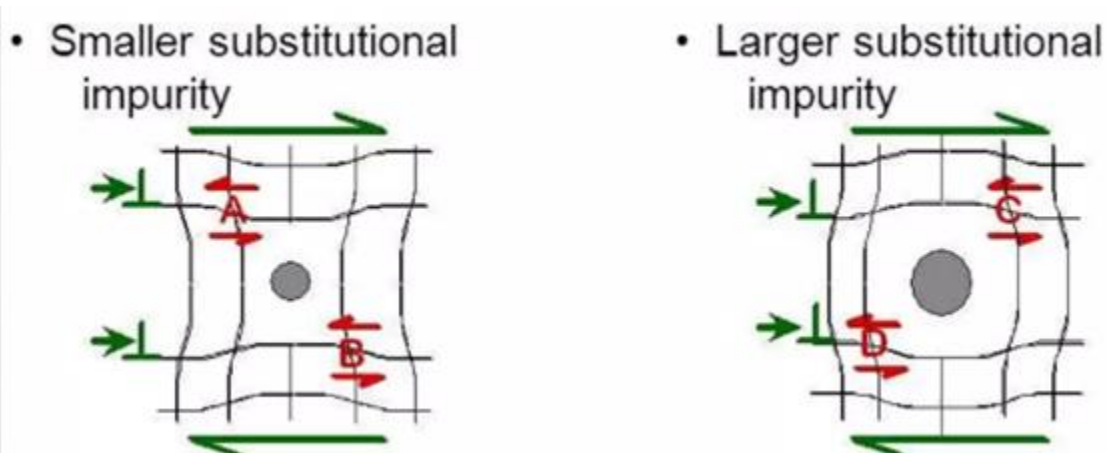
SOLID – SOLUTION ALLOYING: -

Substitutional solid soln.   OR   Interstitial solid soln.
(e.g., Cu in Ni)   (e.g., C in Fe)
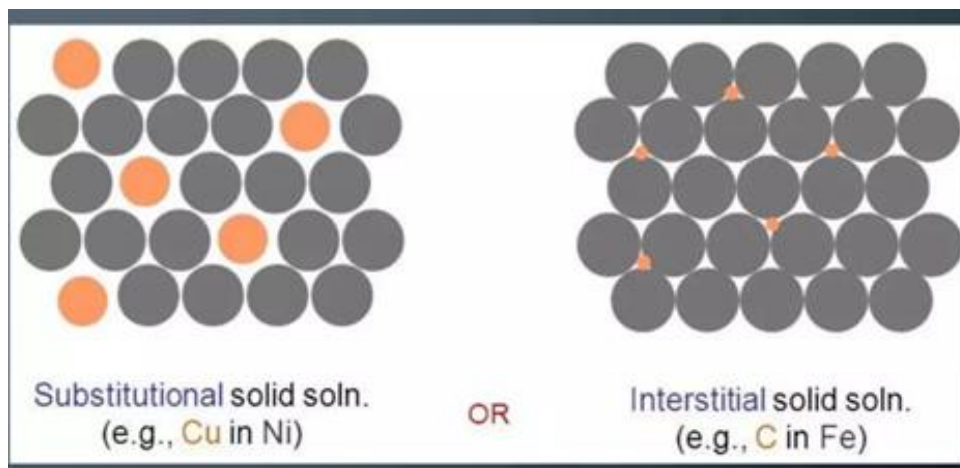
❖ The technique works by adding atoms of one element (the alloying atom) to the crystalline lattice of another element (the base element) forming a substitutional or interstitial solid solution.

❖ **SUBSTITUTIONAL** – Distributed impurity atoms are similar in size of the host metal atoms. Maintain the ductility of the initial pure metal.

❖ **INTERSTITIAL** – Some of interstices in metal structure occupied by smaller atoms. More rigid than the primary metal.

❖ Small impurities mostly concentrate near to act as a barrier to the dislocation.

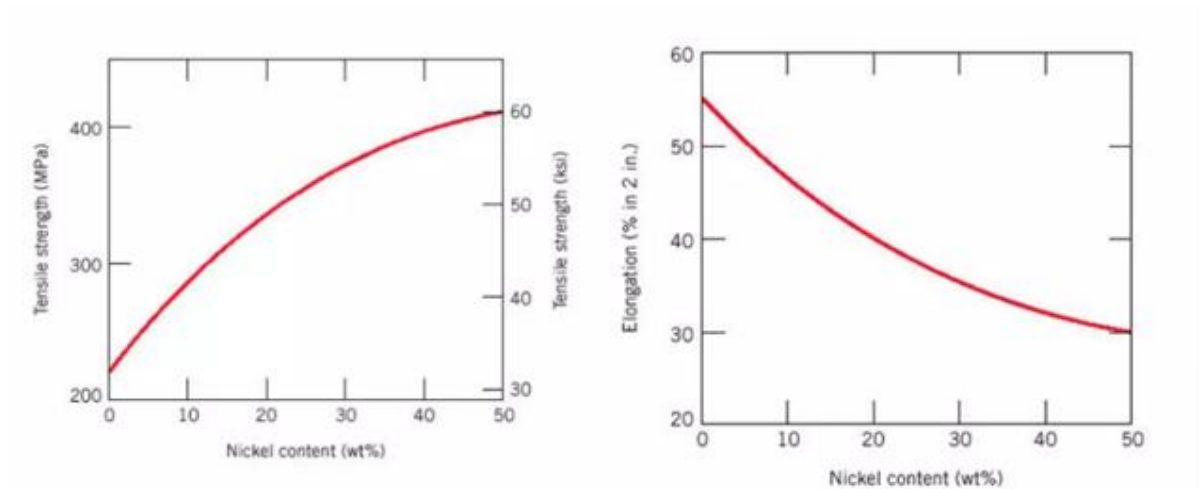❖ These impurities act as an obstacle to moving dislocations and minimize the propagation of the dislocations.

(a)
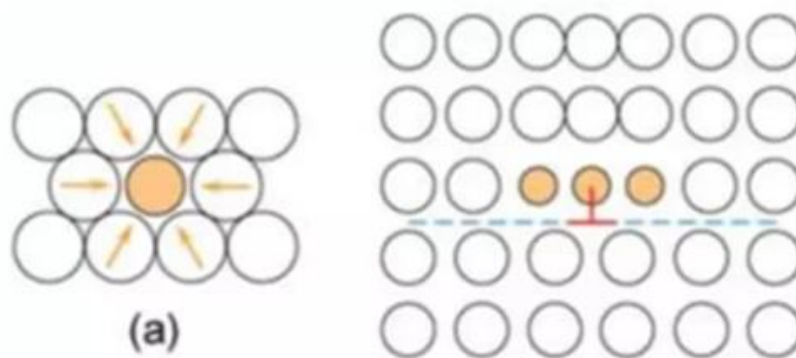
❖ This increases the yield strength of the material.

• Smaller substitutional impurity

• Larger substitutional impurity

❖ Solid solution strengthening depend on:
  ➢ Concentration of solute atoms.

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➤ Shear modulus of solute atoms.
➤ Size of solute atoms.
➤ Valency of solute atoms (for ionic materials).

STRENGTHENING BY STRAIN HARDENING (WORK HARDENING OR COLD WORKING):-

❖ Plastic deformation of ductile materials increases the strength of the material at very low temperature than its melting point.
❖ Strengthening of a metal or polymer by plastic deformation is called as work hardening or strain hardening.
❖ This strengthening occurs because of dislocations movements and dislocation generation within the crystal structure of the material.
❖ The reason for strain hardening is the increase of dislocation density with plastic deformation.
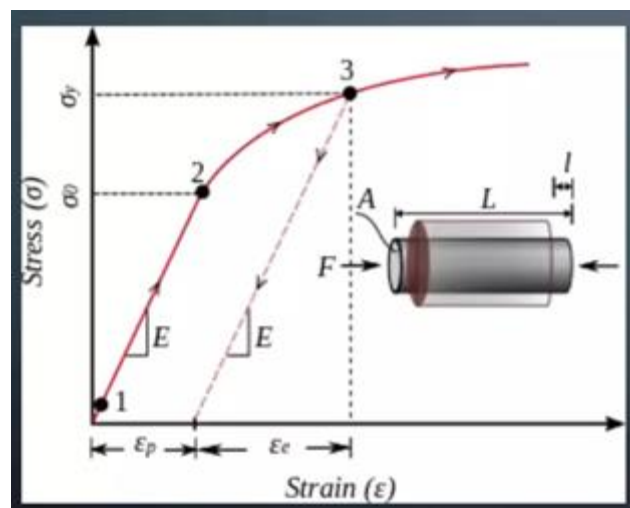
ϵ_e = recoverable elastic strain
ϵ_p = inelastic strain
$\sum_0$ = stress at initial yield
$\sum_y$ = stress at new yield

$$\sum_0 < \sum_y$$

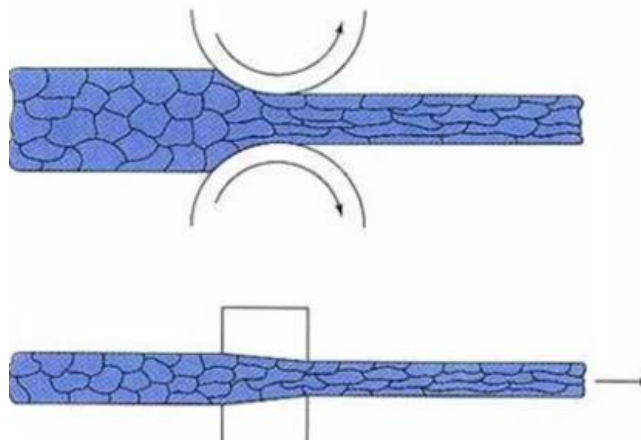❖ The average distance between dislocations decreases and dislocations start blocking the motion of each other.

$$\%CW = \left(\frac{A_o - A_d}{A_0} \right) \times 100$$

A_0 = Initial cross-sectional area
A_d = new cross-sectional area
$CW_\% $ = Cold work percentage

❖ In addition to maximizing dislocation density, work hardening changes the following parameters of the materials ~ Hardness increase, Yield strength increases, Ductility decreases (material becomes more brittle), Grain become directional.
❖ Conducting temperature is very low compared to melting point temperatures (~1/3 of melting temperatures).

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ANNEALING OF DEFORMED METALS: -

❖ After cold working, most of metals are in high energy state.
❖ For restoration to the initial state of the metal, annealing process can be carried out by heat treating.
❖ Ductility is very low after the cold working, but hardness is higher.
❖ Main steps in annealing are: -
    1. Recovery
    2. Recrystallization
    3. Grain growth

RECOVERY: -

1. The strain fields around dislocations cause them to interact (exert force on each other). If dislocations are in the same plane, they attract/annihilate when they have opposite signs. Therefore, dislocation cancel out and their contribution to the stored energy is removed.

2. Steps of recovery are: -
    ❖ Heating
    ❖ Increased diffusion
    ❖ Enhanced dislocation motion
    ❖ Decrease in dislocation density by annihilation
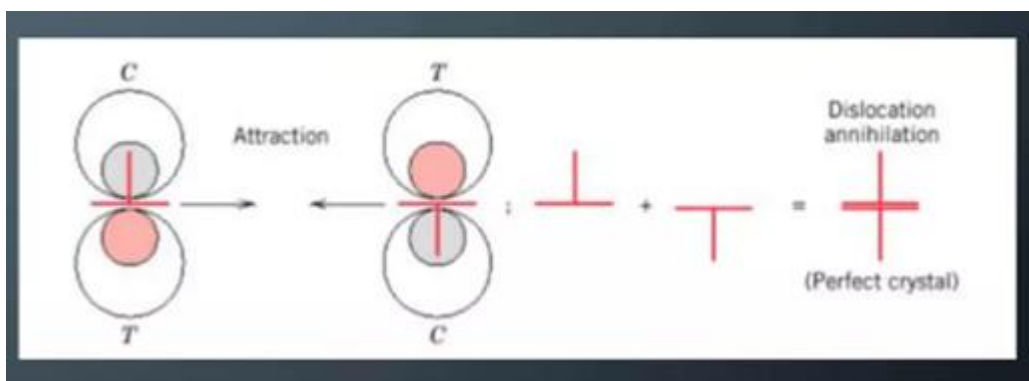   ❖ Relieve of the internal strain energy
    ❖ Recovered material

FIG: RECOVERY

RECRYSTALLIZATION: -

1. Even after recovery, the grains can be strained. These strained grains of cold- worked metal can be replaced, upon heating, by strain – free grains with low density of dislocations.
2. Simply the regeneration of new grains can be observed.
3. Recrystallization Temperature: The temperature at which the process is complete in one hour. It is typically 1/3 to ½ of the melting point temperature.

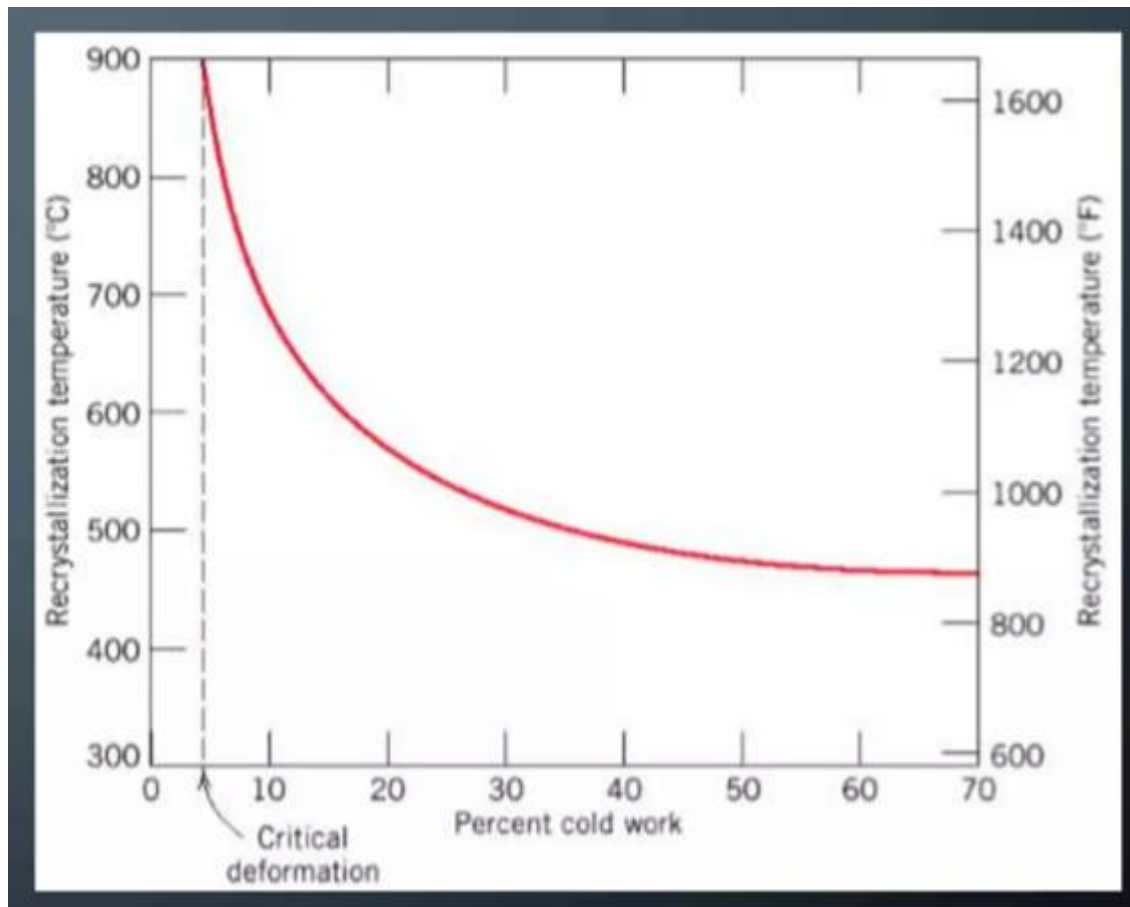**Fig: RECRYSTALLIZATION GRAPH**

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GRAIN GROWTH: -

1. Growth of the generated grains in recrystallization state, by maintaining at annealing temperature, then further grain growth occurs.
2. Driving force causes reduction of the total grain boundary area and hence, big grains grow at expense of the small ones.
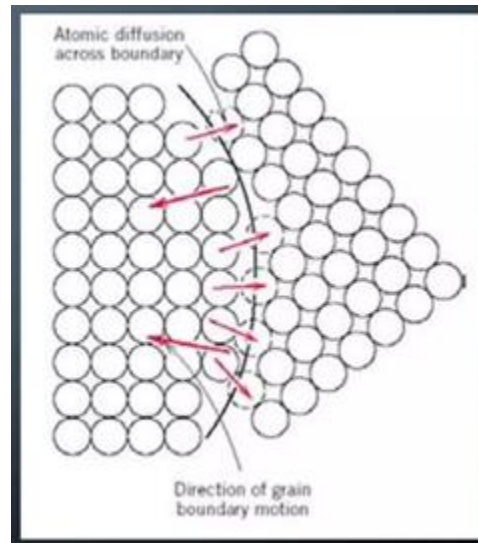3. Boundary motion occurs by short range diffusion of atoms across the grain boundary.

Fig: GRAIN GROWTH

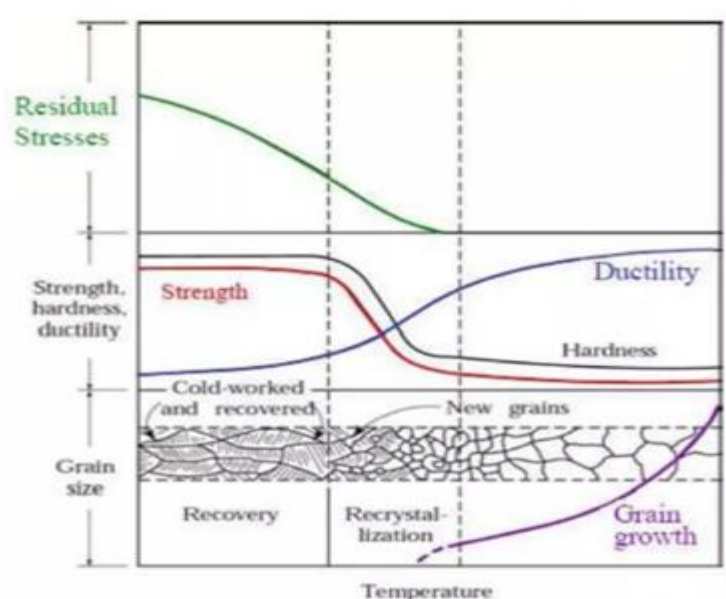4. Change of properties with the steps of the annealing.

Fig: CHANGE OF PROPERTIES WITH THE STEPS OF THE ANNEALING SHOWN IN GRAPH

STRUCTURE OF INTEGRATED STEEL PLANT
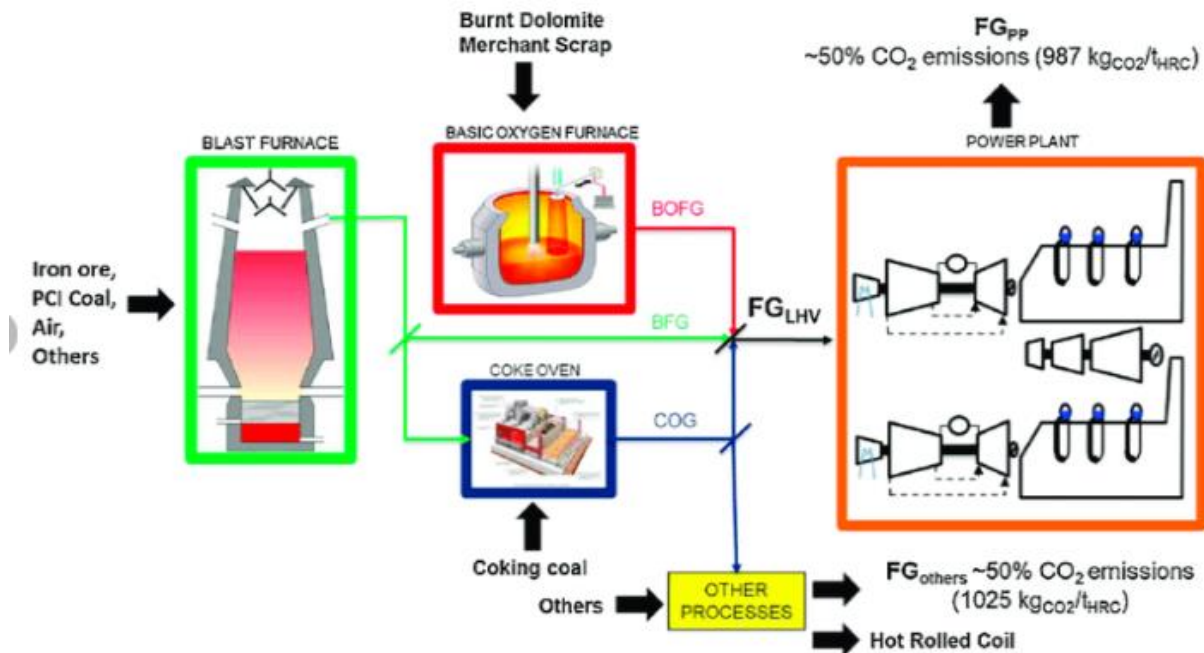
FIG: - Schematic representation of integrated steel plant

First Integrated Steel Plant in India was setup by Tata Iron and Steel Co. at Tatanagar and it was followed by Indian Iron and Steel Co. at Asansol.

As the power sector, there is a massive demand for steel also to meet up the infrastructural growth. Apart from the giants like SAIL, RINL, TISCO other comparatively small companies have started production of steel to cope up the market demand. Most of the major steel plants have their captive power units for uninterrupted supply of power.

Integrated Steel Plant

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What is Integrated Steel Plant (ISP)?

Steel plants which produce iron from iron ore or scrap by following different process to finish steel products like bars of different diameters, a structural section of varying shape and size, pipes are known as Integrated Steel Plants.

Some of the major ISPs are Vizag Steel Plant, Bhilai Steel Plant, Durgapur Steel Plant etc.

Major structures in Steel plant

The following components or structures play an important role in steel plants and production.

1. Blast Furnace
2. Steel Melting Shop/ Basic Oxygen Furnace
3. Stove / Soaking Pit Foundation
4. Hot Strip Mill
5. Cold Rolling Mill
6. Continuous Casting Plant
7. Silicon Treatment Plant.

Let's delve into each of these components or structures and their roles in steel plants and production:

1. **Blast Furnace:**
   - ❖ **Role**: The blast furnace is a key component in the ironmaking process. It's where iron ore, coke, and limestone are melted and chemically transformed into molten iron (pig iron).
   - ❖ **Process**: Iron ore, coke (carbon), and limestone are charged into the furnace from the top. Hot air (blast) is blown into the furnace from the bottom, causing combustion of coke to produce heat. The oxygen in the air reacts with the carbon in the coke to produce carbon monoxide, which reduces the iron ore to molten iron. The limestone acts as a flux, reacting with impurities in the iron ore to form slag, which floats on top of the molten iron.

2. **Steel Melting Shop/ Basic Oxygen Furnace (BOF)**:
   - ❖ **Role**: The steel melting shop, which may include a Basic Oxygen Furnace (BOF), is where molten iron from the blast furnace is further refined into steel.
   - ❖ **Process (BOF)**: In the Basic Oxygen Furnace, oxygen is blown into the molten iron to oxidize impurities such as carbon, silicon, and phosphorus. This process

removes impurities and adjusts the carbon content to produce steel of desired quality and composition.

3. **Stove / Soaking Pit Foundation**:
   - ❖ **Role**: Stoves or soaking pits are used in the steelmaking process to preheat air before it enters the blast furnace or other furnaces.
   - ❖ **Process**: Cold air is passed through the stoves or soaking pits, where it absorbs heat from hot combustion gases. Preheating the air reduces the amount of fuel required to achieve the high temperatures needed in the blast furnace or steel melting shop.

4. **Hot Strip Mill**:
   - ❖ **Role**: The hot strip mill is where hot-rolled coils produced in the steelmaking process are further processed into flat-rolled steel products such as sheets and coils.
   - ❖ **Process**: Hot-rolled coils are passed through a series of rolling stands to reduce their thickness and achieve the desired dimensions. The steel may also undergo additional treatments such as pickling, annealing, and tempering to improve its mechanical properties and surface finish.

5. **Cold Rolling Mill**:
   - ❖ **Role**: Cold rolling mills process hot-rolled coils or sheets at room temperature to produce cold-rolled steel with tighter tolerances and improved surface finish.
   - ❖ **Process**: Hot-rolled coils or sheets are passed through a series of rolling stands at room temperature to reduce their thickness and improve their surface finish. Cold rolling also imparts strength and hardness to the steel.

6. **Continuous Casting Plant**:
   - ❖ **Role**: The continuous casting plant is where molten steel is cast into semi-finished products such as slabs, blooms, or billets.
   - ❖ **Process**: Molten steel from the steel melting shop is poured into a water-cooled mold, where it solidifies into a continuous strand. The strand is then cut into desired lengths to produce semi-finished products, which are further processed in rolling mills.

7. **Silicon Treatment Plant**:
   - ❖ **Role**: The silicon treatment plant is used to add silicon to steel, which enhances its strength and wear resistance.

❖ **Process:** Silicon is added to molten steel in controlled amounts using specialized equipment such as ladle or tundish injection systems. The addition of silicon modifies the steel's microstructure, improving its mechanical properties.

These components and structures work together seamlessly in an integrated steel plant to transform raw materials into high-quality steel products used in various industries.

Steel Production Process

Integrated steel plants are fascinating facilities that transform raw materials into finished steel products.

Let me provide with an overview of the process from mining to finished results.

1. **Mining and Raw Materials:**
   ❖ The process begins with mining iron ore, which is the primary raw material for steel production.
   ❖ Other raw materials include coal, limestone, and scrap metal.

2. **Ironmaking:**
   ❖ Iron ore is smelted in a blast furnace to produce molten iron.
   ❖ The blast furnace process involves reducing iron oxide (Fe_2O_3) to iron (Fe) using coke (carbon)and hot air.

3. **Steelmaking:**
   ❖ The molten iron from the blast furnace is then converted into steel through various processes.
   ❖ Basic oxygen furnaces (BOFs) and electric arc furnaces (EAFs) are commonly used for steelmaking.

4. **Casting and Solidification:**
   ❖ The liquid steel is cast into large rectangular blocks called slabs.
   ❖ These slabs are then cooled and solidified.

5. **Hot Rolling:**
   ❖ Slabs are reheated and passed through a hot strip mill.

- ❖ In the hot strip mill, the steel is rolled into coils or sheets at high temperatures.
- ❖ This process improves the mechanical properties and shape of the steel.

6. **Pickling and Cold Rolling:**
   - ❖ Pickling removes any scale or oxides from the hot rolled steel.
   - ❖ Cold rolling reduces the thickness of the steel by passing it through a series of rolling stands at room temperature.
   - ❖ Cold – rolled steel has improved surface finish, flatness, and tighter thickness tolerance.

7. **Finishing Operations:**
   - ❖ After cold rolling, the steel can undergo additional processes such as annealing, galvanizing, or coating.
   - ❖ Annealing improves the material's properties, while galvanizing provides corrosion resistance.
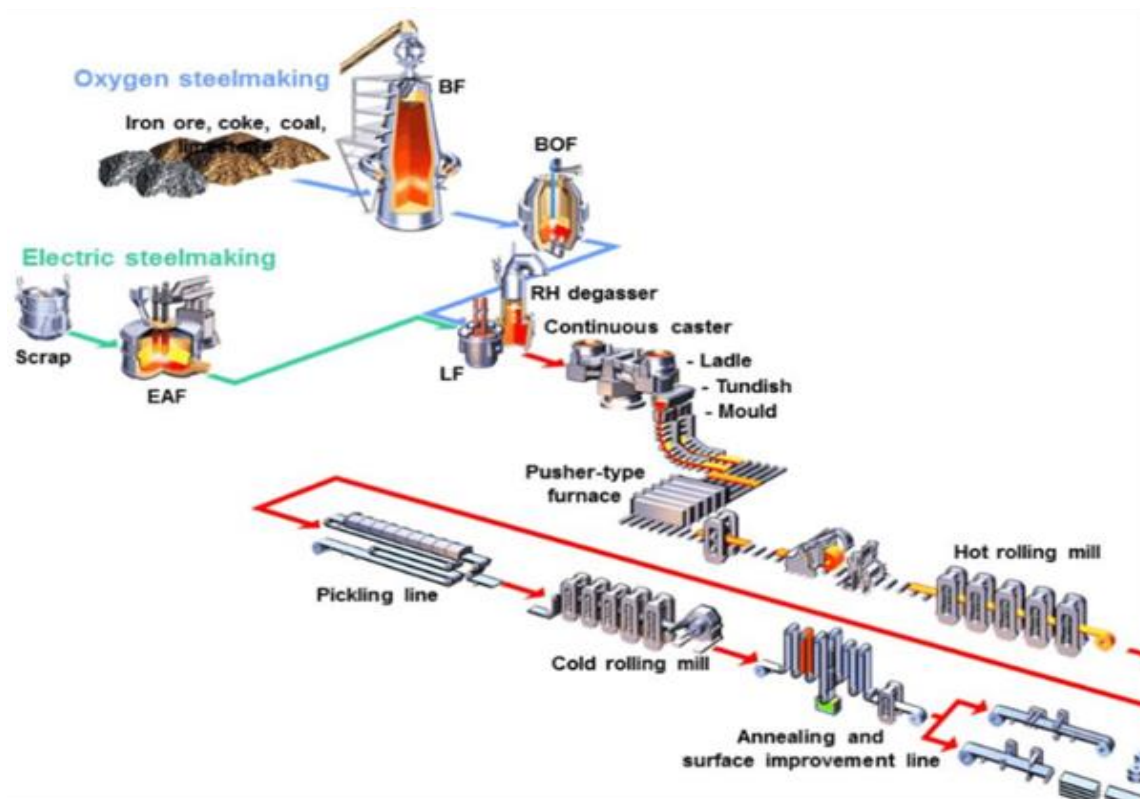
Fig: - Schematic diagram of the integrated steel manufacturing process.
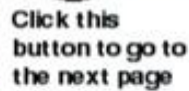
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Fig: - Schematic diagram of the integrated steel manufacturing process.

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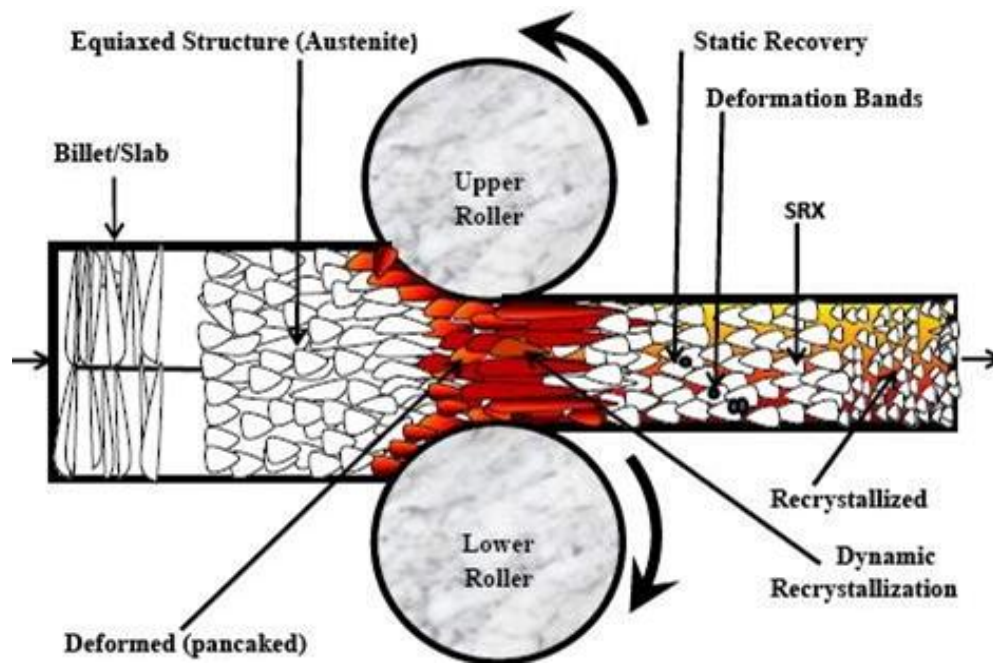
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THERMOMECHANICAL PROCESSING DURING HOT ROLLING
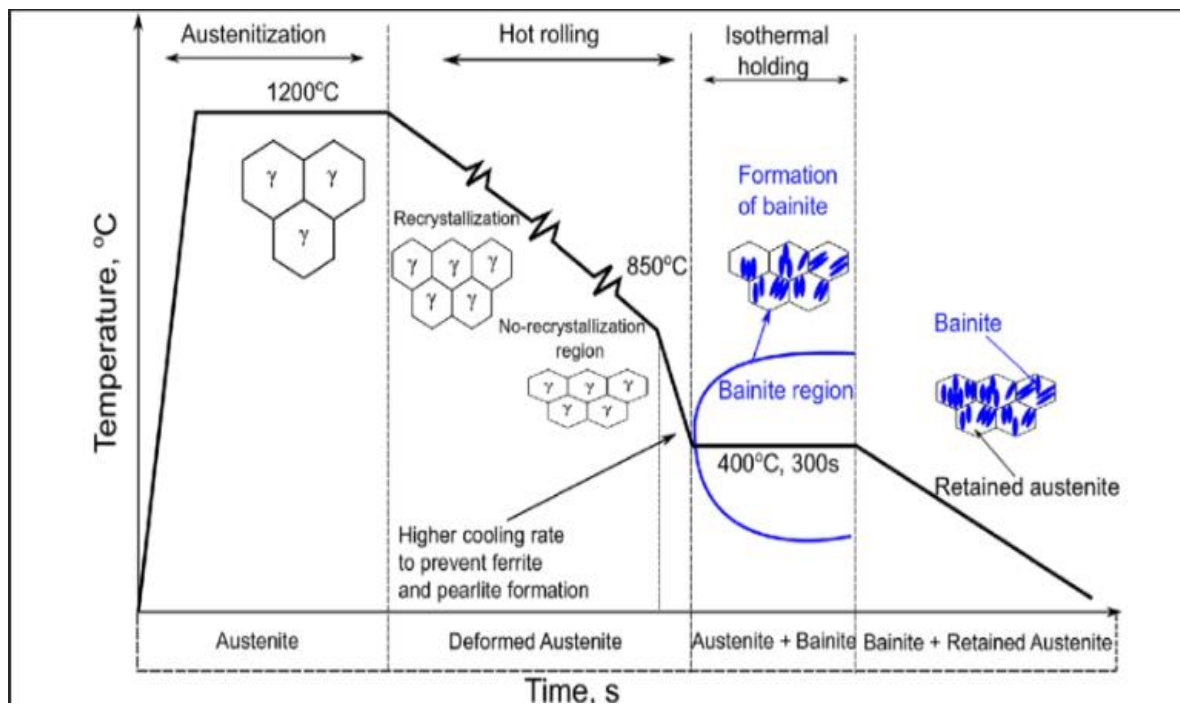
During hot rolling the **microstructure and mechanical properties of the material change with its thermomechanical state**, determined by composition, percent reduction, strip thickness, strip speed, and heat transfer.

Microstructural Phenomena: -

During hot rolling, material undergoes significant microstructural and mechanical changes influenced by factors such as composition, reduction percentage, strip dimensions, speed, and heat transfer. Steel is typically rolled at 850–1200 °C in its austenite phase, whereas aluminum alloys are processed at 350–500 °C. Each rolling pass induces strain, increasing dislocation density and work hardening while elongating grains along the rolling direction. This process triggers dynamic and static softening mechanisms: dynamic recrystallization in steels during roughing mill stages and static recovery followed by recrystallization between passes. In steel, niobium alloying retards recrystallization, promoting finer grain size and enhancing mechanical properties like yield and tensile strength after cooling. Aluminum alloys, however, rely solely on statically recrystallized grain size post-rolling for their mechanical properties without subsequent transformation.

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Thermo-mechanical processing during hot rolling is a critical step in shaping metals. Let's dive into the details: -

1. **Objective of Hot Rolling:**
   - ❖ To lower the strength of a metal and improve its plasticity, allowing for significant thickness reductions.
   - ❖ To achieve the proper starting point phase for controlling transformation during hot rolling.
   - ❖ To utilize metallurgical mechanisms like recovery and recrystallization, softening the steel during deformation for more reduction.
   - ❖ To refine as-hot rolled properties through precipitation mechanisms that don't occur at room temperatures.
   - ❖ To eliminate inherent casting defects by providing sufficient thickness reduction.

2. **Basic Objectives of Hot Rolling:**
   - ❖ Geometry / Size: Producing customer-specific sizes and geometries (thickness, width, length, weight, flatness, straightness, crown, edge drop).
   - ❖ Metallurgical Properties: Controlling precipitation of certain elements and grain refinement.
   - ❖ Internal Soundness: Improving compactness and internal soundness.
   - ❖ Surface: Achieving a semi-uniform surface appearance free of defects.

3. **Geometry Control:**
   - ❖ Optimize the reduction sequence (number of passes and reduction per pass) to achieve final strip geometry.

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- ❖ Consider metallurgical and equipment constraints (e.g., thickness, width, flatness, straightness, profile).

4. **Equipment Constraints:**
   - ❖ Rolling cannot occur outside typical mill electrical and mechanical limits.
   - ❖ Key operating variables include entry/exit thickness sequence, roll radius, rotational speed, and tension forces.
   - ❖ Deformation behaviour of the metal being rolled (rheology) also plays a role.

5. **Thermo-Mechanical Controlled Processing (TMCP):**
   - ❖ TMCP combines well-defined deformation operations and heat treatment in a single stage.
   - ❖ It controls the microstructure of rolled steel, producing desired external qualities and acceptable mechanical properties

Remember, hot rolling is a fascinating interplay of temperature, mechanical deformation, and time, shaping metals into useful forms.

THERMOMECHANICAL PROCESSING DURING COLD ROLLING

Let's explore the fascinating world of cold rolling and its impact on metal properties: -

1. **Cold Rolling Overview:**
   - ❖ Cold rolling is an industrial process that strengthens metal by changing its shape without using heat.
   - ❖ Unlike hot rolling (which occurs at high temperatures), cold rolling happens at room temperature or slightly below the metal's recrystallization temperature.
   - ❖ The process involves passing the metal through rollers, compressing it to achieve specific properties.

2. **Process Steps:**
   - ❖ Hot Rolling: The initial step involves hot rolling, where steel is rolled at temperatures above 900°C (its recrystallization temperature).
   - ❖ Pickling: After hot rolling, the metal is "pickled" to remove scale formed during the process.
   - ❖ Cold Rolling: The cooled steel is then passed through cold rollers at room temperature. This process is called annealing.

❖ Annealing Benefits:
- ➤ *Relieves stress.*
- ➤ *Increases yield strength and hardness.*
- ➤ *Reorients grain structure and creates flaws, leading to microstructure hardening.*

3. **Cold Rolling Mill:**
- ❖ Thickness gauges monitor the steel as it exits the rollers.
- ❖ Reversing mills allow the steel to be pushed back through the rollers until the desired thickness is achieved.
- ❖ Multi-stand mills use several pairs of rollers to gradually reduce thickness.
- ❖ Cooling agents (oil or water) keep the rollers and metal cold and lubricated.

4. **Resulting Metal:**
- ❖ Cold-rolled steel typically has a thickness between 0.12 mm and 2.5 mm.
- ❖ Applications include lightweight vehicle panels, drink cans, and more.
- ❖ Modern cold rolling mills can produce millions of tons of cold-rolled steel annually.

Remember, cold rolling transforms metal properties, resulting in smoother surfaces, increased hardness, and precise dimensions.

EXPERIMENT PERFORMED

1. **AIM: -**
   - ❖ Preparation and study of the Micro Structure of pure metals High Carbon Steel.

2. **OBJECTIVES:** -
   - ❖ To learn the preparation of specimen for microscopic observation.
   - ❖ To understand what Microscopy is, and how it can be used to observe Microstructure of metals.

3. **THEORY: -**
   - ❖ **Eutectoid**: A eutectoid system occurs when a single-phase solid transforms directly to a two- phase solid.
   - ❖ **Hypereutectoid**: Hypereutectoid systems exist below the eutectoid temperature.
   - ❖ **Hypoeutectoid**: Hypoeutectoid systems exist above the eutectoid temperature.
   - ❖ **Ferrite**: Body-centered cubic iron or an iron alloy based on this structure.
   - ❖ **Austenite**: Face-centered cubic iron or an iron alloy based on this structure.
   - ❖ **Delta iron**: The body-centered cubic phase which results when austenite is no longer the most Stable form of iron. Exists between 2802 and 2552 degrees F has BCC lattice structure and is magnetic.

   - ❖ *Classification of Steel*: The steels are classified by various methods and each method is based on a definite criteria as follows:
     1. Amount of carbon
        - ✓ Low carbon steels (0.008 - 0.3%C)
        - ✓ Medium carbon steels (0.30 - 0.60%C)
        - ✓ High carbon steels (0.60 - 2.00%C)
        - ✓ Amount of alloying elements and carbon
     2. Amount of deoxidation
     3. Method of manufacture
     4. Form and use

4. **PROCEDURE: -**

The preparation of metallic or other materials for microscopic examination and microstructural characterization is in principle very simple. There are four basic processes that we will need to become familiar with: sample cutting and sectioning, metallographic mounting, Surface grinding and surface polishing.

A. *Sample Cutting and Sectioning*

Sectioning

Sectioning involves extracting a manageable specimen from a larger sample while preserving its microstructure using an abrasive cut-off machine. This machine features an abrasive cutting wheel or saw, complemented by a secure vice for stabilizing the work piece. The primary focus of this procedure is to reduce heat generation during cutting. To achieve this, some cut-off saws are outfitted with a water-cooling system. This setup effectively safeguards the sample from thermal damage, ensuring the integrity of its properties throughout the sectioning process.

B. *Mounting*

For samples larger than approximately 25 mm square, mounting is unnecessary as they can be polished without support. However, smaller samples in our laboratory utilize two primary mounting techniques for easier handling and to prevent damage to polishing equipment. The predominant method involves hot-mounting with Bakelite, a thermosetting plastic compound that encapsulates the specimen. Alternatively, a cold-mounting process using room temperature curing epoxy is reserved for heat-sensitive samples. Bakelite, available in various colors, facilitates straightforward sample identification through color coding.

The procedure for Hot mounting process

Here is the step-by-step process for encapsulating specimens in Bakelite:

1. Place the sample face down on the small piston inside the press and lower it into the cylinder by partially opening the front valve.
2. Pour approximately three tablespoons of Bakelite over the sample and gently position the top of the press without fully tightening it; engaging all screw threads is sufficient.
3. Plug in and activate the cylindrical heater (indicated by a red light), surrounding the mold to reach 135°C to 150°C. Close the valve and pump up the cylinder using the lever. As the Bakelite heats, it will flow to fill voids, causing pressure to drop; maintain consistent pressure.
4. Once pressure stabilizes, maintain 150°C for 5-7 minutes to complete curing. Maintain pressure throughout heating and cooling.

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5. Remove the heater and allow the mold to cool for 6-8 minutes with a cooling collar.

6. Release pressure by cracking the valve, unscrew the mold top (which may stick due to Bakelite), and slowly push the sample out using the cylinder.7. Clean any residual Bakelite from the mold surfaces after marking the sample on its back.

The procedure for Cold Mounting process

The following process is used to room temperature curing epoxy process:

1. Apply mold release agent to mold. Place specimen in the mold.

2. Mix epoxy powder and bonding liquid in 1:2 ration in a cup.

3. Pour into mold... wait for 10 minutes.

4. Eject the mold.

C. *Sample Surface Polishing*

The goal of the surface polishing is to end up with a planar cross section of sample free from scratches or disturbed metal introduced by the cutting and sectioning.

Grinding

To begin sample preparation, ensure a flat surface using a water-cooled abrasive grinder. Once flat, thoroughly wash the sample. Proceed to the hand lapping station equipped with graded abrasive papers (starting with 240 grit). Keep water flowing uniformly over the abrasives. With firm, consistent pressure, move the specimen back and forth until only 240 grit scratches remain uniformly. Wash the sample and hands, then advance to 320 grit, repeating the process for 400 and 600 grits, verifying after each step that only corresponding scratches are present.

Rough Grinding

Before starting the first polishing stage, ensure the sample is washed with water. Begin by applying water to the leftmost wheel and activating its motor, gently cleaning it with your fingers. Next, apply a small amount of AI203 abrasive slurry to the wheel, where the abrasive particles are 5 micrometers in diameter. Hold the sample firmly and place it on the wheel, moving it in circular motions against the wheel's rotation with moderate, even pressure to maintain flatness. After several minutes, pause to create parallel scratches for assessing previous grinding damage removal. Examine under a microscope to verify uniform scratch size. Repeat these steps on the middle wheel using 0.3 micrometer A1203 abrasive in water suspension.

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Final Polishing

Following the final polishing stage on the right polishing wheel, utilizing a 0.05 micrometer A1203 abrasive in water suspension, the sample achieves a remarkably smooth surface, sensitive even to oils and finger contact, which can cause larger scratches than the abrasive. From this point forward, avoid touching the sample surface. The subsequent step involves etching to reveal the microstructure. Use a cotton swab and petri dish for this process, applying the etchant gently and allowing it to stand briefly before rinsing with water and methanol. Examine the specimen under a microscope, adjusting etching duration as needed for optimal microstructure visibility.

D. *Etching*

Grains cannot be seen without etching. Cracks, pores and defects are observed without etching. Etchant reacts with atoms and dissolves them. Atoms at grain boundaries dissolve quickly. Dissolved grain boundaries appear dark.

Steps:

1. Apply enchant to polished surface for some time
2. Rinse with distilled water

Enchants:

1. 50/50 HCl: equal parts hydrochloric acid (HCl) mixed with water.
2. Alcoholic Ferric Chloride: 5 grams FeC13; 2 ml concentrated HCl acid; 95 ml methyl alcohol.
3. Aqueous Ferric Chloride: 10 grams FeC13; 20 ml concentrated HCl acid; 80 ml water.
4. Ammonia/Hydrogen peroxide: 1-part strong ammonia; 1-part hydrogen peroxide; 2 parts water; FRESHLY MADE.
5. Mixed acids: 95 ml water; 1.5 ml concentrated HCl acid; 2.5 ml concentrated nitric (HN03) acid; 0.5 ml hydrofluoric (HF) acid.
6. 2% Nital: 2 ml concentrated HN03; 98 ml methyl alcohol.

Procedure To Use Optical Microscope &SEM To Obtain Micrograph

- ❖ **Setup the Microscope**
  - ➢ Ensure the microscope is clean and properly adjusted.
  - ➢ Begin with the lowest magnification objective lens
- ❖ **Placing the Sample**
  - ➢ Place the prepared and etched steel sample on the microscope stage.
- ❖ **Focusing**
  - ➢ Use the coarse and fine focus adjustments to bring the surface into sharp focus.
  - ➢ Start with the low magnification objective to get an overall view and then switch to higher magnifications (e.g., 20x, 50x, 100x) for more detailed observations.
- ❖ **Adjusting Illumination**
  - ➢ Adjust the light intensity and condenser aperture to optimize contrast and resolution.
- ❖ **Observation**
  - ➢ Examine the grain structure at various magnifications.
  - ➢ Take note of the grain size, shape, and any other notable microstructural features (e.g., inclusions, phases).
  - ➢ Capture images using a camera attached to the microscope for documentation and analysis.

Measure the hardness value of steel using a Vickers hardness testing machine

- ❖ *Preparation*
  - I.   **Sample Preparation**: Ensure the steel sample has a smooth, flat surface. The surface should be free from oxides, scale, and any surface deformations that could affect the accuracy of the test.

  - II.   **Polishing**: For accurate results, polish the surface to a mirror-like finish. Start with coarse abrasives and progress to finer ones.
  - III.   **Cleaning**: Clean the sample to remove any debris or oils.
- ❖ *Setting Up the Machine*
  - I.   **Calibration**: Ensure the Vickers hardness testing machine is calibrated. Check the machine's manual for specific calibration procedures.

    II.   **Selecting the Indenter**: A diamond pyramid indenter is typically used for Vickers hardness testing.

❖ *Performing the Test*

    I.   **Mount the Sample**: Place the prepared sample on the stage of the hardness testing machine.

    II.   **Focus the Microscope**: Use the microscope on the testing machine to focus on the area of the sample where you want to make the indentation.

    III.   **Apply the Load**: Select an appropriate test load (measured in grams or kilograms). Common loads range from 1 kgf to 100 kgf, but the specific load depends on the sample material and thickness.

    IV.   **Make the Indentation**: Activate the machine to apply the selected load through the diamond indenter for a specific duration (usually 10-15 seconds).

❖ *Measuring the Indentation*

    I.   **Unload**: After the specified dwell time, the machine will automatically release the load.

    II.   **Measure the Diagonals**: Use the machine's microscope to measure the diagonals of the resulting indentation. The Vickers hardness value is calculated based on these diagonal measurements.

    III.   **Calculation**: The Vickers hardness (HV) is calculated using the formula: $HV = (1.8544 \times F)/d^2$ *Where*: F is the applied load in kilograms-force (kgf), d is the average length of the diagonals in millimeters (mm)

❖ *Recording Results*

    I.   **Average Multiple Indentations**: For accurate results, perform multiple indentations on different areas of the sample and calculate the average hardness value.

    II.   **Document Findings**: Record the hardness values, the load applied, the dwell time, and any other relevant details in a report.

❖ *Tips for Accurate Measurement*

    I.   Ensure the sample is securely clamped to prevent movement during the test.

    II.   Perform the test in a controlled environment to avoid temperature variations and vibrations.

    III.   Regularly check and calibrate the machine to maintain accuracy.

    IV.   Ensure the indentations are well-spaced to prevent interaction between adjacent impressions.

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Measure the grain size of steel sample

- ❖ *Prepare the Image*
    - I. **Capture Image**: Capture a clear micrograph of the sample with visible grains. Ensure the image has good contrast.
    - II. **Open Image**: Launch ImageJ and open your image by going to **File > Open.**
- ❖ *Image Processing*
    - I. **Convert to Grayscale**: If your image is coloured, convert it to grayscale via **Image > Type > 8-bit.**
    - II. **Enhance Contrast**: Adjust the image contrast using **Image > Adjust > Brightness/Contrast** to clearly distinguish the grain boundaries.
    - III. **Thresholding**: Set the threshold to highlight the grains using **Image > Adjust > Threshold.** Adjust the sliders until the grains are well defined in red. Click **Apply**.
- ❖ *Analyze Particles*
    - I. **Analyse Particles**: Go to **Analyse > Analyse Particles**. In the dialog box, set the size and circularity range appropriate for your grains. Check Display results and Show outlines options.
    - II. **Measure Grain Size**: Click **OK** to analyse the particles. The results window will show measurements for each detected grain.
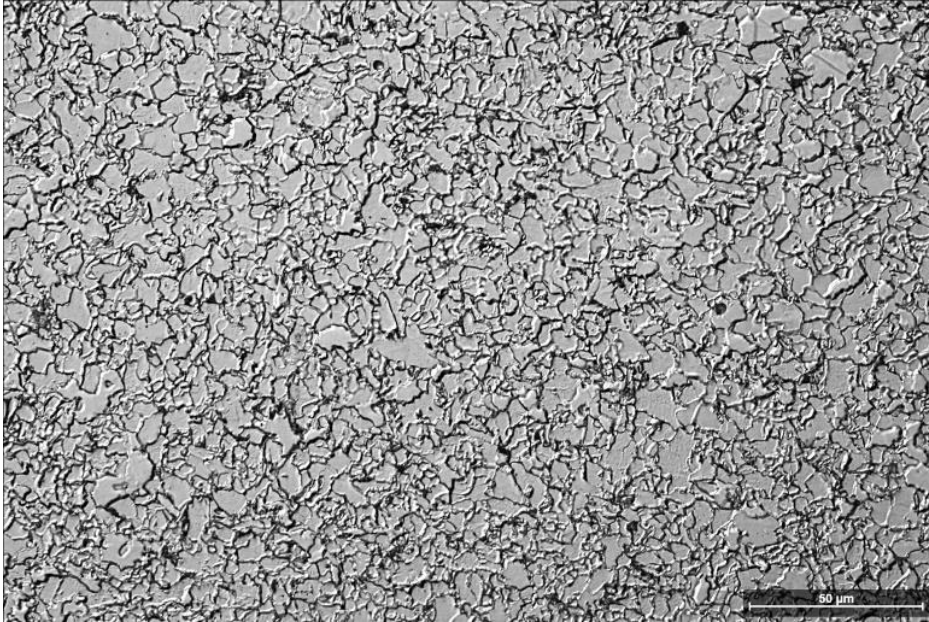- ❖ *Calculate Grain Size*
    - I. **Determine Mean Grain Size**: In the results window, focus on the **Area** column. Calculate the average area of the grains.
    - II. **Convert to Grain Diameter**: If needed, convert the area to an equivalent grain diameter using the formula: Diameter=$2 \times \sqrt{area}/\sqrt{pie}$
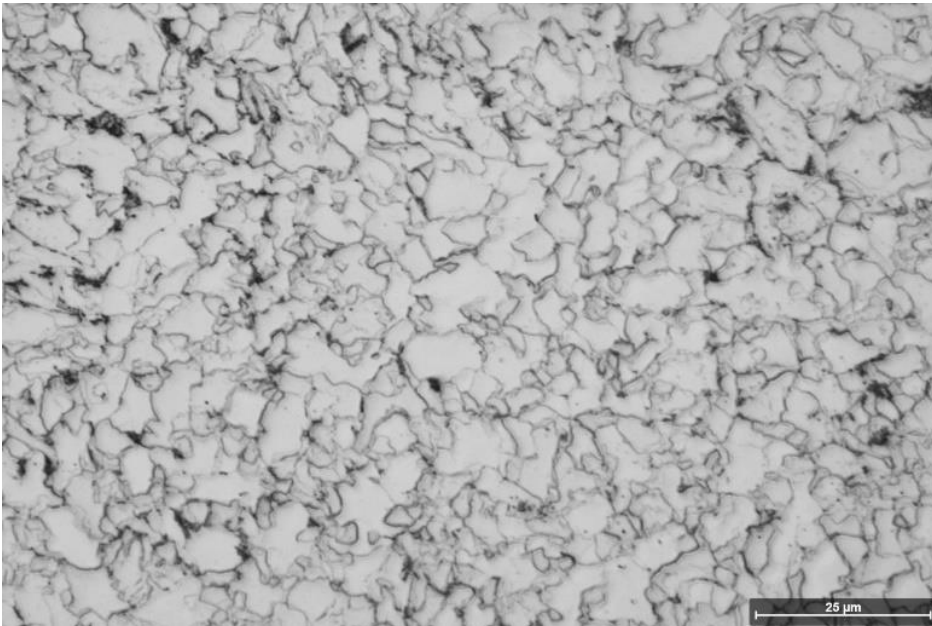
5. Metallographic Observation

Observe microstructure, Place specimen on metallography and adjust magnification, focus and position s adjust microscope
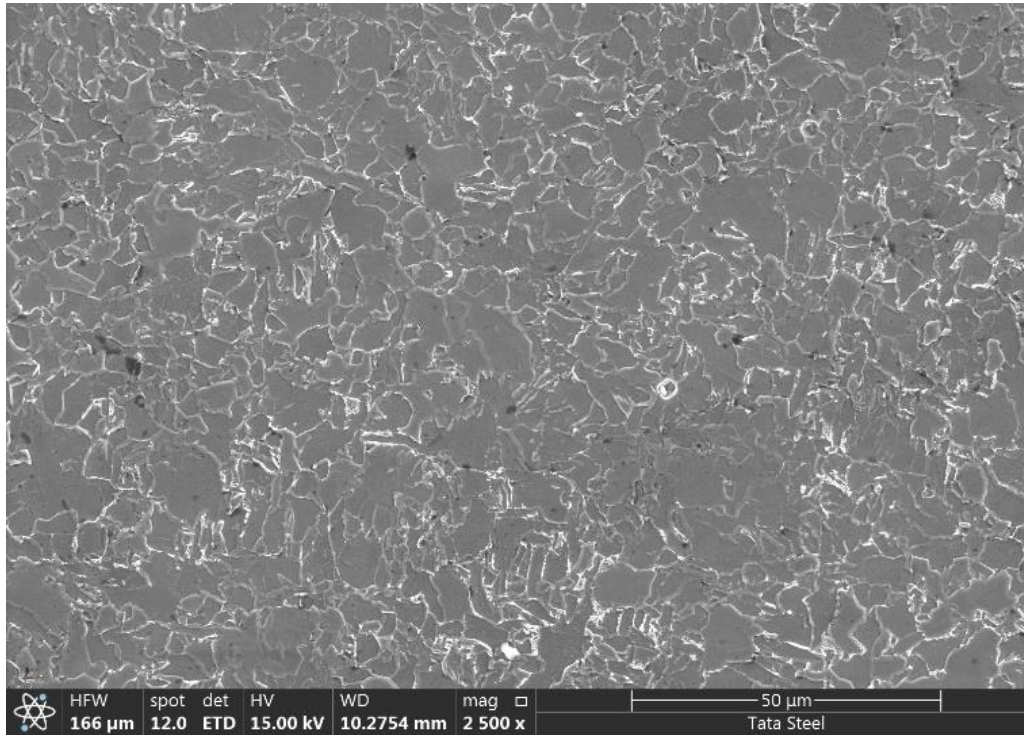High magnification - to study phases and Low magnification -to study grain size.
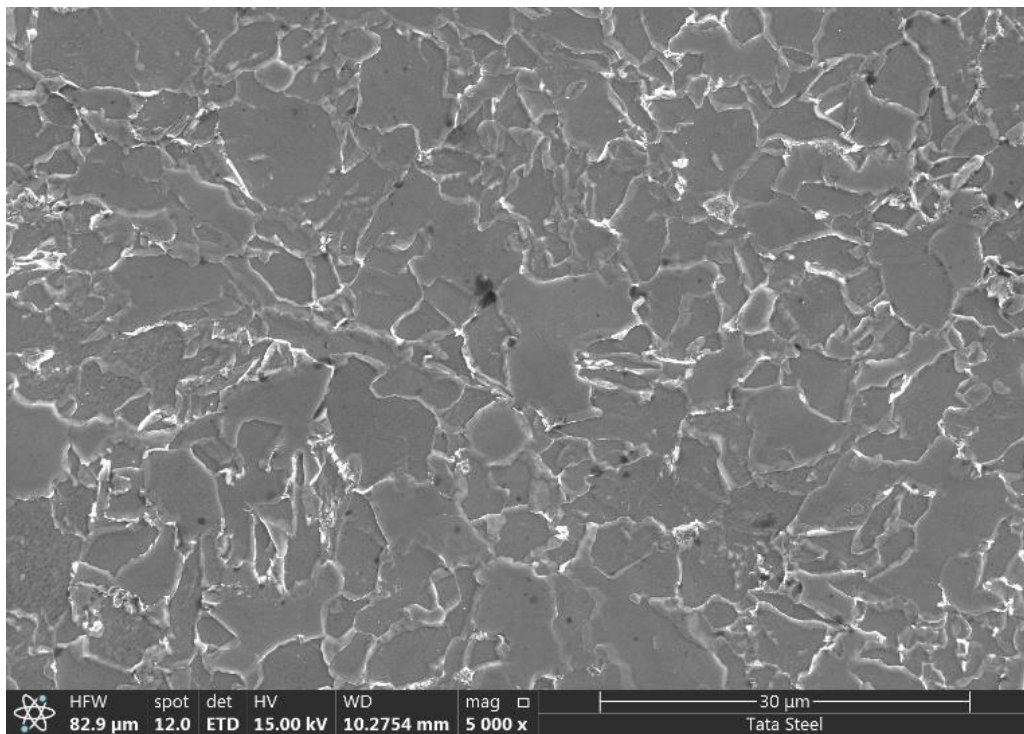
SAMPLE: STEEL A

500X

1000X

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UNDER SEM:

| HFW | spot | det | HV | WD | mag □ | 50 μm |
| 166 μm | 12.0 | ETD | 15.00 kV | 10.2754 mm | 2 500 x | Tata Steel |

2500X

| HFW | spot | det | HV | WD | mag □ | 30 μm |
| 82.9 μm | 12.0 | ETD | 15.00 kV | 10.2754 mm | 5 000 x | Tata Steel |

5000X

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SAMPLE: STEEL B

500X

1000X

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UNDER SEM:

2500X

HFW	spot	det	HV	WD	mag ☐	50 µm
166 µm	12.0	ETD	15.00 kV	10.0088 mm	2 500 x	Tata Steel

5000X

HFW	spot	det	HV	WD	mag ☐	30 µm
82.9 µm	12.0	ETD	15.00 kV	10.0088 mm	5 000 x	Tata Steel

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THE HARDNESS VALUE OF DIFFERENT SAMPLE

HARDNESS VALUE(HV)	STEEL A	STEEL B
POINT1	215	282
POINT2	202	295
POINT3	194	275
POINT4	198	293
POINT5	192	285
POINT6	204	287
POINT7	198	282
POINT8	223	277
POINT9	207	279
POINT10	204	280
AVG: -	203.7	283.5

DETERMINATION OF GRAIN SIZE[PIXEL/MICROMETER] OF THE GIVEN SAMPLE:

STEEL A	6.838	9.306	7.188	6.194	6.426	7.66	6.184	9.888	**AVG:**7.461
STEEL B	9.211	5.957	5.49	7.242	7.773	5.813	6.713	6.412	**AVG:**6.826

RESULTS:

- ➢ The average hardness of sample (Steel A) is: 203.7HV
- ➢ The average hardness of sample (Steel B) is: 283.5HV
- ➢ The average grain size of sample (Steel A) is: 7.461 pixels/micrometer
- ➢ The average grain size of sample (Steel B) is: 6.826 pixels/micrometer

Increasing grain size decreases strength and hardness. Thus, Steel A has less hardness compared to Steel B. So, grain size of Steel A is more than Steel B.

DISCUSSIONS AND CONCLUSION:

The above shown micrographs show a polycrystalline structure with clearly defined grain boundaries. The grain boundaries are more visible and detailed in the higher magnification micrograph. The contrast between grains suggests the presence of different phases or differences in etching response. Possible phases present are **Ferrite** (α-Fe) generally appears lighter in colour and forms the matrix in many steels**. Pearlite** consists of alternating layers of ferrite and cementite and can appear as a lamellar or fine structure within the grains. **Cementite** (Fe_3C) often appears darker and can form discrete particles or layers within pearlite. **Austenite** (γ-Fe) typically retained austenite appears as a brighter phase due to different etching characteristics. **Martensite** appears as a needle-like or plate-like structure and is usually darker due to the etching process.
The absence of large second-phase particles suggests a well-distributed microstructure, potentially indicating a single-phase material or fine distribution of secondary phases.

These observations can help in understanding the microstructure and identifying any treatments or processes the steel has undergone, such as annealing, quenching, or tempering.

The following conclusions we have come to after observing different micrograph under different magnification is that, Steel A sample consist of ferrite as well as pearlite structure. And Steel B sample has ferritic structures in it.

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