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Abstract

This report investigates the effects of three heat treatment processes—annealing, normalizing, and quenching—on low-carbon steel (0.05–0.25% C) at varying temperatures (900°C). The primary objective was to understand how temperature and cooling rates influence phase transformations, microstructure evolution, and mechanical properties such as hardness.

During the experiment, samples were heated to target temperatures and subjected to the respective heat treatments. Microstructural analysis was conducted using optical microscopy after metallographic preparation, and mechanical properties were evaluated through Vickers hardness testing.

The results revealed that annealing produced coarse-grained structures dominated by ferrite and pearlite with relatively low hardness values. Normalizing resulted in refined microstructures with smaller grains and moderately increased hardness due to faster air cooling. Quenching produced martensitic structures with significantly higher hardness but reduced ductility due to rapid cooling.

The discussion highlights the relationship between microstructure and mechanical properties, emphasizing the role of grain size, phase distribution, and cooling rates in determining material behavior.

This study provides a comprehensive understanding of heat treatment processes and their impact on low-carbon steel, offering valuable insights for optimizing material performance in engineering applications.

Introduction

Heat treatment is a controlled process involving heating and cooling of metals and alloys to alter their microstructure and mechanical properties. It is widely employed to optimize characteristics such as hardness, ductility, toughness, strength, and machinability. In this experiment, three fundamental heat treatment processes—annealing, normalizing, and quenching—were investigated on low carbon steel samples containing approximately 0.05–0.25% carbon.

Annealing is a heat treatment process where steel is heated to a temperature slightly above its critical temperature range, held at that temperature for a specific duration, and then cooled slowly (usually within the furnace). This slow cooling allows atoms to diffuse properly, relieving internal stresses developed during previous manufacturing processes like casting, forging, or rolling. Annealing results in a softer and more ductile material with improved machinability and formability. It also refines grain structures and homogenizes chemical composition throughout the material.

Normalizing involves heating the steel above its upper critical temperature (typically 30–50°C above), holding it briefly at this elevated temperature to achieve uniformity in the microstructure, followed by air cooling at room temperature. The faster cooling rate compared to annealing leads to a finer grain size, uniform microstructure distribution (usually pearlite and ferrite), and improved mechanical properties such as strength and toughness. Normalizing is commonly performed to eliminate structural irregularities caused by prior processing steps.

Quenching is a rapid cooling process where steel heated above its critical temperature is quickly immersed in a quenching medium such as water, oil, or brine. This rapid cooling prevents diffusion-controlled transformations like pearlite formation, resulting instead in the formation of martensite—a hard, brittle microstructure. Quenching significantly increases hardness and strength but reduces ductility and toughness. To achieve desired mechanical properties and reduce brittleness after quenching, subsequent tempering (controlled reheating) is often performed.

In this experimental study, low carbon steel samples were heated separately to temperature of 900°C. Each sample was subjected individually to annealing, normalizing, and quenching heat treatments. Following these treatments, optical microscopy was used for microstructural analysis of each sample. Additionally, Vickers hardness tests were conducted to quantitatively evaluate the effects of different heat treatments on hardness at varying temperatures.

Heat-treated low carbon steels find extensive applications across industries due to their enhanced mechanical properties. Typical applications include automotive components (gears, shafts), structural elements (beams, rails), machinery parts (bolts, fasteners), construction materials, pipelines, and various engineering components requiring specific combinations of strength, ductility, hardness, or toughness.

<u>Material</u>

The material selected for this experiment was low carbon steel, also commonly known as mild steel, with a carbon content ranging from approximately 0.05% to 0.25% C. Low carbon steel is primarily composed of iron with small amounts of carbon and minor quantities of other alloying elements such as manganese and silicon. The carbon content in low carbon steels typically ranges below 0.25%, which imparts specific mechanical properties suitable for various engineering applications.

Low carbon steel is produced primarily through basic oxygen furnaces (BOF) or electric arc furnaces (EAF), followed by casting and rolling into desired shapes and sizes. Due to its relatively low carbon content, this steel exhibits excellent ductility and malleability, allowing it to be easily formed, machined, and welded without significant risk of cracking or brittleness

Key Properties of Low Carbon Steel:

Ductility: It can be easily shaped, stretched, or bent without fracturing, making it ideal for forming processes such as bending, drawing, or stamping.

Weldability: Low carbon content minimizes the formation of cracks in heat-affected zones during welding processes, simplifying fabrication and assembly.

Density: Typically around 7.85 g/cm^3, consistent with most ferrous alloys.

Thermal Conductivity: Moderate thermal conductivity; suitable for applications where heat conduction is necessary but not critical.

Mechanical Properties: Exhibits moderate strength and toughness combined with good ductility. These properties can be further enhanced through controlled heat treatments to achieve desired hardness and strength levels.

Low carbon steel is widely used across industries due to its favorable balance between cost, mechanical properties, machinability, and ease of fabrication. Typical applications include automotive body panels, structural components (such as beams and rails), machinery parts (fasteners, bolts), pipes for low-pressure systems, construction materials, furniture frames, and general-purpose engineering components where moderate strength combined with good ductility is essential

Experimental Procedure

Heat Treatment Procedure

The experimental procedure involved heating low carbon steel samples (0.05–0.25% C) to different set temperatures—800°C, 900°C, 1000°C, and 1100°C—followed by three distinct heat treatments: annealing, normalizing, and quenching. The detailed steps for each heat treatment are described below:

Annealing

Samples were heated to their respective temperature (900°C) in furnace.

Upon reaching the desired temperature, samples were held at that temperature for approximately 30 minutes to ensure complete and uniform heating.

After soaking, the furnace was turned off, and samples were allowed to cool slowly inside the furnace to room temperature.

Normalizing

Samples were heated individually to the designated temperature (900°C) and held at these temperatures for approximately 30 minutes.

After soaking at these elevated temperatures, samples were removed from the furnace and cooled in still air at room temperature until they reached ambient conditions.

Quenching

Samples were heated separately to each of the specified temperature (900°C).

After holding at temperature for approximately 30 minutes to achieve uniform austenitization, samples were rapidly quenched in water maintained at room temperature.

Samples remained immersed until completely cooled.

Metallographic Sample Preparation

After heat treatment processes were completed, each sample underwent metallographic preparation for microstructural analysis. The following detailed procedure was employed:

Cutting and Mounting

Small sections of treated samples were cut using a precision cutting machine.

Each sample was mounted using standard mounting resin to facilitate handling during polishing.

Grinding and Polishing

The metallographic preparation involved two main stages: grinding/paper polishing followed by cloth polishing.

Grinding/Paper Polishing

Samples were initially ground on rotating abrasive papers of progressively finer grit sizes (starting from coarse grit size such as 220 grit and progressing through intermediate sizes like 400-600-800-1000, and finally up to fine grit size of about 1200-1500-2000).

During grinding, water was continuously applied as a lubricant and coolant to prevent overheating and maintain cleanliness of the sample surfaces.

Each grinding step was performed until scratches from previous grit size disappeared completely before moving to finer grit paper.

Cloth Polishing

After paper polishing, samples underwent fine polishing using a rotating polishing cloth wheel impregnated with diamond paste or alumina suspension (typically particle sizes of around 1 µm or smaller).

Polishing continued until a mirror-like surface finish without scratches or irregularities was achieved on each sample surface.

Etching

Polished samples were chemically etched using Nital solution, typically consisting of approximately 2–5% nitric acid diluted in ethanol.

Etching was done by immersing polished surfaces briefly (usually around 5–15 seconds) in Nital solution (commonly about 2–5% nitric acid in alcohol), followed immediately by rinsing with distilled water and drying carefully.

Etching selectively attacked grain boundaries and phase interfaces to reveal microstructural features clearly under microscopic examination.

Optical Microscopy

Prepared surfaces were examined under an optical microscope equipped with suitable magnification lenses.

Micrographs were captured digitally for each sample clearly showing grain structures, phases present (ferrite, pearlite, martensite), grain size distributions, and other relevant microstructural details.

Hardness Testing Procedure (Vickers Hardness Test)

Vickers hardness testing was employed to quantitatively evaluate hardness changes resulting from different heat treatments.

Procedure:

The polished metallographic samples were placed on the stage of a Vickers hardness tester.

A diamond pyramid indenter with a square base angle of 136∘136∘ was pressed into the sample surface under a standardized load (typically ranging from 10 kgf to 30 kgf depending upon material hardness).

The load was applied steadily for approximately 10–15 seconds before being released carefully.

After removing the load, indentation diagonals created on the sample surface were measured accurately using an optical microscope integrated within the hardness tester.

The average diagonal length (d) of indentation impression was measured precisely.

The Vickers hardness number (HV) was calculated using the formula:

where:

$$HV = \frac{1.8544 \times P}{d^2}$$

Figure 4.1: Formula for Hardness Number in Vickers Hardness Test

HV is Vickers hardness number,

P is applied load in kilograms-force (kgf),

d is the average diagonal length of indentation impression measured in millimeters.

Results

1) 900°C

(a) Annealing

General Observations
The sample was heated to 900°C, held at this temperature for 30 minutes, and cooled slowly inside the furnace. Annealing is characterized by slow cooling, which promotes grain growth over nucleation due to the extended time available for atomic diffusion. This results in a coarse microstructure dominated by equilibrium phases.

At 900°C, the steel initially existed in the austenitic phase (γ-Fe), as this temperature is above the upper critical temperature (A3) for low-carbon steels. Upon slow cooling, the austenite transformed into ferrite (α-Fe) and pearlite—a eutectoid mixture of ferrite and cementite (Fe_3C). The slow cooling allowed sufficient time for ferrite and pearlite grains to grow, resulting in a coarse-grained structure.

Microstructure Analysis
The microstructure observed under the optical microscope (Figure 1) reveals large equiaxed grains of ferrite (light regions) interspersed with coarse pearlite colonies (dark regions). The pearlite exhibits a lamellar structure with alternating layers of ferrite and cementite. This morphology is typical of annealed low-carbon steel and results from the slow cooling process, which favors grain growth over nucleation.

Phase Transformation: During cooling, austenite decomposed into ferrite and pearlite as per the Fe-C phase diagram. The slow cooling rate ensured that nucleation sites were limited, allowing existing grains to grow larger.

Grain Morphology: Ferrite grains are equiaxed due to recrystallization during annealing, while pearlite colonies are coarse due to extended diffusion times.

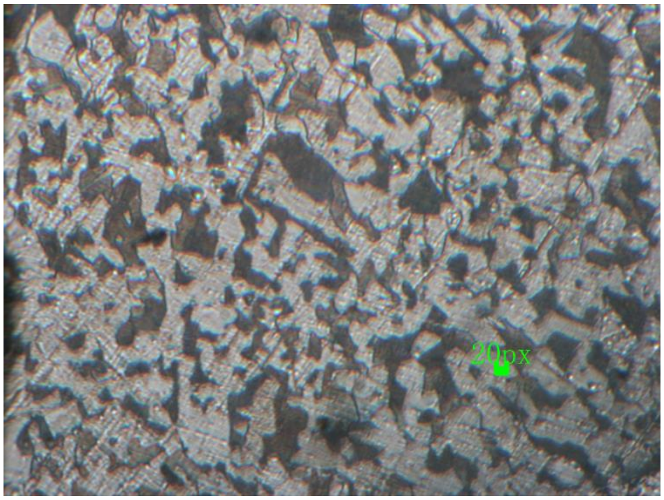

Figure 5.2.1: Microstructure of the annealed sample at 900°C (10x magnification).

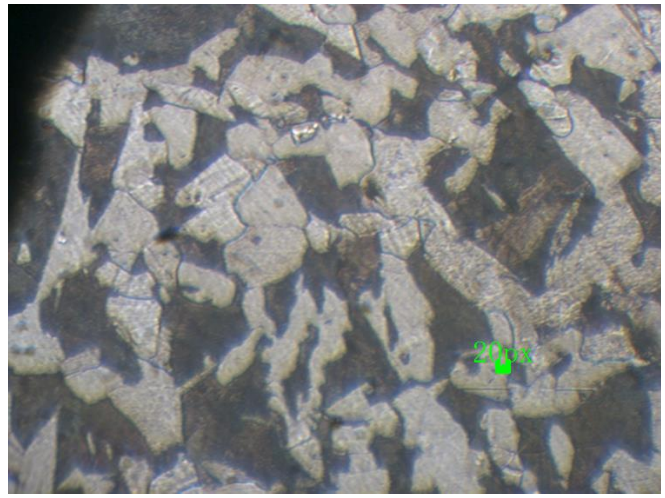

Figure 5.2.2: Microstructure of the annealed sample at 900°C (20x magnification).

Hardness Test Results

The Vickers hardness test was conducted on the annealed sample using a load of 50 gf. The average hardness value obtained was 153 HV, as shown in Figure 2. This relatively low hardness is consistent with the microstructure dominated by soft ferrite and coarse pearlite. Ferrite contributes significantly to ductility but minimally to hardness, while pearlite adds moderate hardness due to its lamellar structure.

Hardness Analysis: The slow cooling during annealing resulted in reduced dislocation density and stress relief within the grains, leading to lower hardness values compared to other heat treatments like quenching or normalizing.

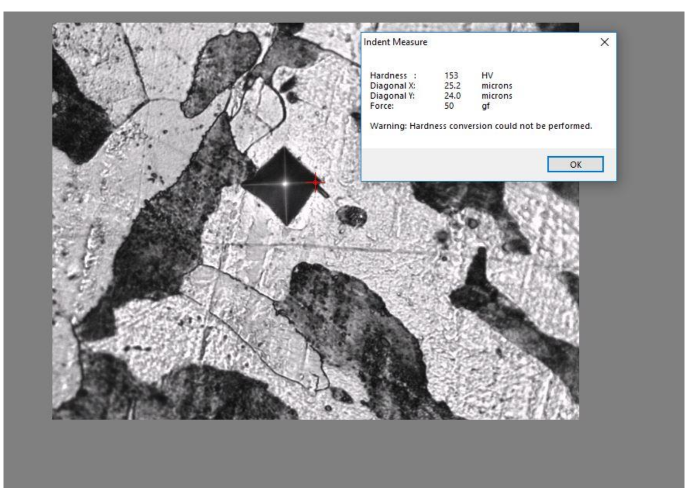

Figure 5.2.3: Vickers Hardness Test Result of annealed sample at 900°C

(b) Normalizing

General Observations
The sample was heated to 900°C, held at this temperature for 30 minutes, and then cooled in still air at room temperature. Normalizing is performed to refine the grain structure, eliminate residual stresses, and achieve a more uniform microstructure. Cooling in air results in faster cooling compared to annealing, which promotes nucleation over grain growth.

At 900°C, the steel was entirely in the austenitic phase (γ-Fe). Upon cooling, the austenite transformed into ferrite and pearlite. Due to the faster cooling rate during normalizing compared to annealing, nucleation dominated over grain growth, resulting in finer pearlite colonies and smaller ferrite grains.

Microstructure Analysis
The microstructure observed under the optical microscope (Figures 2 and 3) shows a refined structure consisting of ferrite (light regions) and pearlite (dark regions). The pearlite colonies are finer compared to those observed in annealed samples due to the increased cooling rate during normalizing. The lamellar structure of pearlite is more compact, and ferrite grains are smaller and more uniformly distributed.

Phase Transformation: During cooling, austenite decomposed into ferrite and pearlite as per the Fe-C phase diagram. The faster cooling rate during normalizing led to increased nucleation sites for ferrite and pearlite formation, resulting in finer grains.

Grain Morphology: Ferrite grains are smaller and more uniformly distributed compared to annealed samples, while pearlite colonies exhibit a finer lamellar structure due to faster cooling.

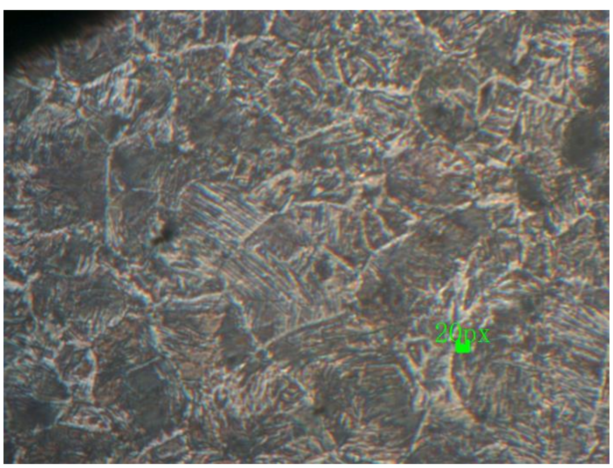

Figure 5.2.4: Microstructure of the normalized sample at 900°C (10x magnification).

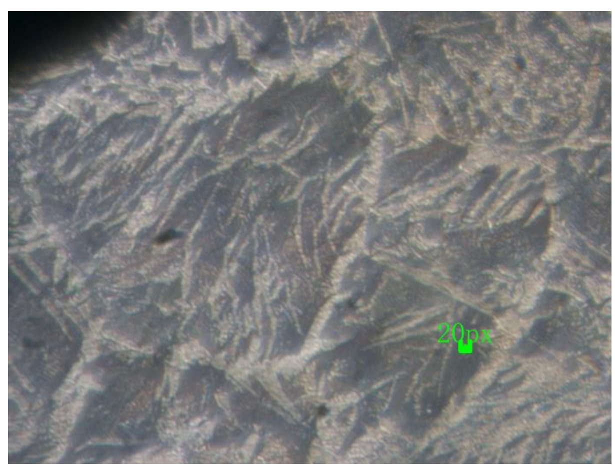

Figure 5.2.5: Microstructure of the normalized sample at 900°C (20x magnification).

Hardness Test Results
The Vickers hardness test was conducted on the normalized sample using a load of 100 gf. The average hardness value obtained was 216 HV, as shown in Figure 4. This higher hardness compared to annealed samples is attributed to the finer grain structure and compact pearlite morphology produced by normalizing.

Hardness Analysis: The faster cooling rate during normalizing increased dislocation density and refined grain size, both of which contribute to improved hardness. The presence of finer pearlite colonies also enhances hardness due to their compact lamellar structure.

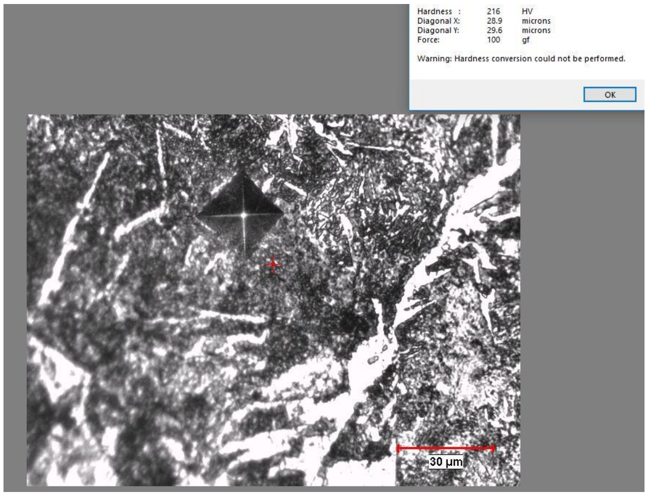

Figure 5.2.6: Vickers Hardness Test Result of normalized sample at 900°C .

(c) Quenching

General Observations

The sample was heated to 900°C, held at this temperature for 30 minutes, and then rapidly cooled by immersion in water at room temperature. Quenching is characterized by rapid cooling, which suppresses diffusion-controlled transformations and results in the formation of martensite—a hard and brittle microstructure.

At 900°C, the steel was entirely in the austenitic phase (γ-Fe). Upon quenching, the rapid cooling prevented the decomposition of austenite into ferrite and pearlite, instead transforming it into martensite. Martensite is a supersaturated solid solution of carbon in iron, formed due to the shear transformation mechanism.

Microstructure Analysis

The microstructure observed under the optical microscope (Figures 3 and 4) reveals a predominantly martensitic structure with needle-like or acicular morphology. The rapid cooling during quenching resulted in high internal stresses and dislocation density within the grains, contributing to the hardness of the material.

Phase Transformation: Austenite transformed directly into martensite due to the rapid cooling rate, bypassing the formation of equilibrium phases like ferrite or pearlite.

Grain Morphology: The martensitic structure is characterized by its fine, needle-like appearance, indicative of high hardness but low ductility.

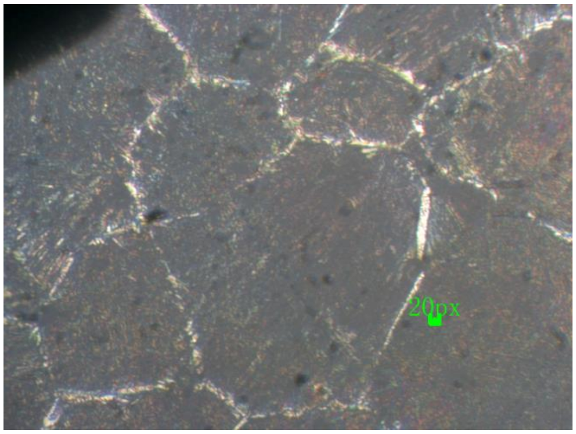

Figure 5.2.7: Microstructure of the quenched sample at 900°C (20x magnification).

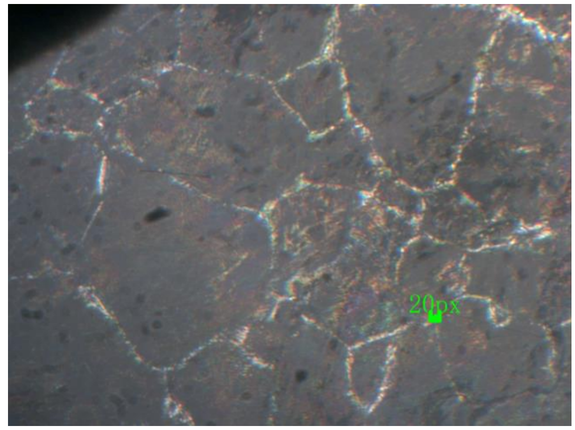

Figure 5.2.8: Microstructure of the quenched sample at 900°C (20x magnification).

Hardness Test Results

The Vickers hardness test was conducted on the quenched sample using a load of 100 gf. The average hardness value obtained was 563 HV, as shown in Figure 5. This significantly higher hardness compared to annealed and normalized samples is attributed to the martensitic structure formed during quenching.

Hardness Analysis: The martensitic structure contributes to extreme hardness due to its high dislocation density and internal stresses. However, this comes at the expense of ductility and toughness, making quenched steel brittle.

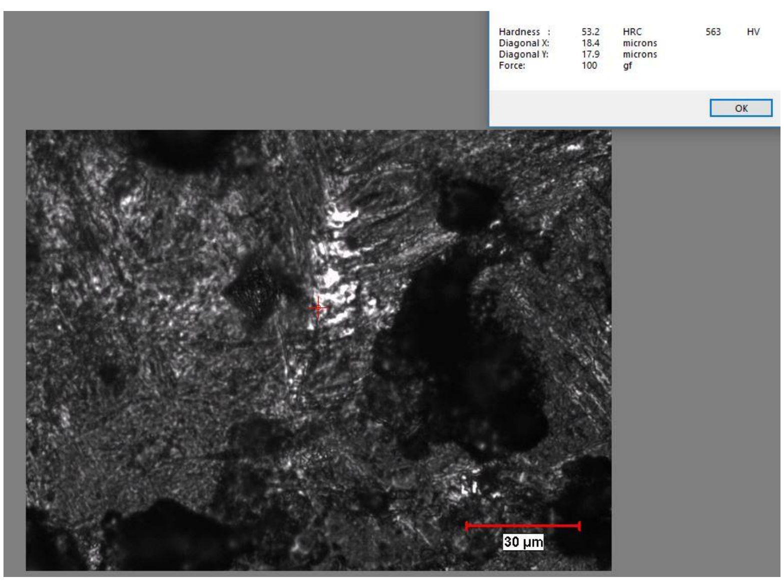

Figure 5.2.9: Vickers Hardness Test Result of quenched sample at 900°C .

Discussion

This section provides an in-depth analysis of the heat treatment results obtained at 900°C, comparing annealing, normalizing, and quenching treatments applied to low carbon steel (0.05-0.25% C). The differences in microstructure and hardness values observed between these three heat treatments offer valuable insights into phase transformation mechanisms and structure-property relationships.

Microstructural Evolution during Heat Treatments at 900°C

At 900°C, the low carbon steel was fully austenitic (γ-Fe) as this temperature is above the upper critical temperature (A3) for this composition. The subsequent cooling process—either slow furnace cooling (annealing), air cooling (normalizing), or water quenching—determined the final microstructure and properties of the material.

Annealing at 900°C

The microstructure of the annealed sample revealed large equiaxed ferrite grains (light regions) interspersed with coarse pearlite colonies (dark regions). This characteristic morphology resulted from the slow cooling process that allowed extensive atomic diffusion. During cooling, austenite decomposed into ferrite and pearlite following equilibrium phase transformations as dictated by the Fe-C phase diagram.

The slow cooling rate provided sufficient time for carbon atoms to diffuse, resulting in well-defined ferrite grains and relatively coarse pearlite colonies with widely spaced cementite lamellae. This microstructure is a consequence of growth dominating over nucleation during the prolonged cooling period. The reduced cooling rate minimized thermal stresses and allowed the material to approach equilibrium conditions, leading to a stress-free, coarse-grained structure.

Normalizing at 900°C

The normalized sample exhibited a more refined microstructure compared to the annealed sample, with smaller ferrite grains and finer pearlite colonies. The increased cooling rate during air cooling promoted nucleation over growth, resulting in more nucleation sites for ferrite and pearlite formation.

The moderate cooling rate during normalizing limited the time available for carbon diffusion, producing finer pearlite colonies with more closely spaced cementite lamellae. The ferrite grains appeared more uniform in size and distribution compared to the annealed sample. This refinement in grain structure is attributed to the faster cooling rate that restricted grain growth while still allowing diffusion-controlled transformations to occur.

Quenching at 900°C

The quenched sample revealed a dramatically different microstructure dominated by martensite, characterized by its distinctive needle-like or acicular morphology. The rapid cooling during water quenching suppressed diffusion-controlled transformations, preventing the formation of equilibrium phases (ferrite and pearlite).

Instead, austenite transformed directly into martensite through a diffusionless, shear-type transformation mechanism. This martensitic structure represents a supersaturated solid solution of carbon in a body-centered tetragonal (BCT) crystal structure. The rapid cooling trapped carbon atoms in interstitial positions, distorting the crystal lattice and creating high internal stresses within the material.

Correlation between Microstructure and Hardness at 900°C

The Vickers hardness test results revealed significant differences between the three heat treatments applied at 900°C:

Annealing: The annealed sample exhibited the lowest hardness value (153 HV) among the three heat treatments. This relatively low hardness correlates with the coarse-grained ferrite-pearlite microstructure. Ferrite, being a soft phase with low carbon content, contributes minimal hardness. The coarse pearlite with widely spaced cementite lamellae provided limited strengthening. Additionally, the slow cooling minimized dislocation density and internal stresses, resulting in a soft, ductile material.

Normalizing: The normalized sample showed an intermediate hardness value (216 HV), significantly higher than the annealed sample. This increase in hardness is attributed to the refined microstructure with smaller ferrite grains and finer pearlite colonies. The Hall-Petch relationship explains this phenomenon, where the increased grain boundary area in refined structures impedes dislocation movement more effectively, enhancing strength and hardness.

Quenching: The quenched sample demonstrated the highest hardness value (563 HV), dramatically exceeding both annealed and normalized samples. This substantial increase in hardness is a direct consequence of the martensitic microstructure. The tetragonal distortion of the crystal lattice and high dislocation density in martensite severely restrict dislocation movement, resulting in significantly increased hardness. However, this comes at the expense of ductility, making the material more brittle.

Strengthening Mechanisms at 900°C

The varying hardness values observed across the three heat treatments at 900°C can be explained through different strengthening mechanisms:

Grain Size Refinement: Comparing annealed (153 HV) to normalized (216 HV) samples, the increased hardness in the normalized sample demonstrates the effect of grain refinement. According to the Hall-Petch relationship, the yield strength (and by extension, hardness) is inversely proportional to the square root of grain size. The smaller grain size in the normalized sample increased the total grain boundary area, which effectively impeded dislocation movement.

Phase Transformation Hardening: The dramatic increase in hardness in the quenched sample (563 HV) illustrates the impact of martensitic transformation. This diffusionless transformation results in a highly strained lattice structure with significant internal stresses. The tetragonal distortion of the martensite crystal structure creates a strong barrier to dislocation movement, resulting in substantial hardening.

Dislocation Density: The cooling rate directly influenced the dislocation density in the samples. The slow-cooled annealed sample had the lowest dislocation density, the air-cooled normalized sample had an intermediate dislocation density, and the water-quenched sample had the highest dislocation density. Higher dislocation density increases hardness through dislocation entanglement and interaction, restricting further dislocation movement.

These results at 900°C clearly demonstrate how different cooling rates from the same austenitizing temperature can produce dramatically different microstructures and mechanical properties in low carbon steel through various phase transformation mechanisms and strengthening processes.

Conclusion

The detailed investigation of heat treatments at 900°C clearly demonstrated how cooling rates significantly influence the microstructure and mechanical properties of low-carbon steel (0.05–0.25% C). Annealing, characterized by slow furnace cooling, produced a coarse-grained ferrite-pearlite microstructure with the lowest hardness (153 HV), making it ideal for applications demanding high ductility and ease of machining. Normalizing, involving moderate air cooling, resulted in refined ferrite grains and finer pearlite colonies, yielding intermediate hardness (216 HV) and balanced mechanical properties suitable for structural and general engineering applications. Quenching, with rapid water cooling, led to the formation of martensite—a hard and brittle phase—with the highest hardness (563 HV), suitable primarily for applications requiring high strength and wear resistance. This experiment highlights the critical role of controlled heat treatment processes in tailoring material properties according to specific industrial requirements.

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