MM208
PHASE TRANSFORMATION AND HEAT TREATMENT LAB

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Entry Number:- 2023MMB1420
Group Number:- 4
Temperature at which heat treatment performed:- 1100°C

Index:-

Introduction

Heat treatment of metals is a regulated process consisting of heating and cooling of metals to modify their microstructure and mechanical characteristics. It is done to increase hardness, strength, ductility, toughness, or machinability. The treatment process usually includes heating the metal to a defined temperature (which may be greater than its critical point), keeping it at the same temperature, and cooling at a regulated rate. General heat treatment processes involve annealing (softening), normalising (grain structure refinement), quenching (hardening), and tempering (tempering for brittleness reduction). These processes control phase transformations, grain size, and carbon diffusion to attain required properties for use in manufacturing and engineering.

Heat treatment of metal is extensively performed in industries for improving the mechanical properties and suiting them to a particular requirement. Its applications involve providing hardness, strength, ductility, and also resistance to wear, corrosion, and impact. For example, it is employed in the motor vehicle industry to make strong engine parts and chassis components, in aerospace to fabricate high-stress applications such as jet engines, and in machinery equipment to enhance the lifespan of heavy-duty machines. Heat treatment also alleviates brittleness, eases internal stresses due to production operations such as welding or machining, and enhances machinability.

Heat Treatment Methods performed:-

1. Annealing- In this process, the sample is heated to a specific temperature and then let it cool down slowly on its own by letting it stay in the furnace only. Very gradual cooling of sample takes place. It is usually performed to reduce the hardness of the material and increases its ductility thereby making it easier to bend the material without cracking. Also it reliefs internal stress in the material and improves its overall mechanical properties.
2. Quenching- In this process, the sample is heated to a specific temperature and then it is rapidly cooled down by dipping it in mediums like oils, water, salt solutions and suitable polymers. In this process the material’s austenite phase gets transformed to martensite phase so this process is usually done to enhance material’s hardness, strength and resistance to wear and tear.
3. Normalising- In this process, the sample is heated to a specific temperature and then it is allowed to cool down in the presence of air. It is usually done to obtain a homogeneous microstructure by removing micro-structural non-uniformities and structural imperfections. It also reduces the material’s excessive hardness and increases the overall material ductility, stability and toughness.

Brief introduction of process followed:-

For our heat treatment we were give a cylindrical rod of mild steel sample which we cut into 4 different pieces of same cross sections and of equal lengths. One sample was chosen as our reference sample and heat treatment processes of normalisation, quenching and annealing were performed on other 3 samples. Firstly 3 of our samples were heated to a temperature of 1100°C in a furnace followed by heat treatment methods of annealing, quenching and normalisation by letting the samples to cool down at different rates. Then sample preparation was done by sample polishing on emery paper followed by cloth polishing. Once polishing was completed and all scratches were removed, etching was done by dipping our sample in 5% Nital solution for 4-5 seconds to enhance the sample’s grain boundaries to be able to view its microstructure under microscope. The microstructure analysis was done under the optical microscope followed by microhardness test using Vicker’s test method of our samples to compare their physical hardness and strength.

Material Used

Material Name: Mild Steel (Iron+Carbon)
Composition: 0.05% - 0.25% Carbon

Mild Steel is a mixture of Iron and low percentage of carbon. Mild Steel is made by smelting iron ore in a blast furnace where carbon is introduced into the process and all other impurities are eliminated. One of the most commonly used means by which this is done is through a mixture of iron ore and carbon containing coal. After coal and iron ore are mined from the ground, they are smelted together in a blast furnace. After being melted, the two are then transferred to another furnace to incinerate any impurities with which they might have been contaminated, and to make any other changes to the chemical makeup of the mild steel. Changes are also made at this stage to create the required chemical composition to enable the production of mild steel. This steel is then cooled into an ingot (slab). The ingot is then cut to the desired size by either a "Hot Rolling" or "Cold Rolling" process. Mild steel is not an alloy, its just a simple mixture of iron and carbon and so there are no major traces of chromium, molybdenum, or other alloying elements.

Properties of mild steel:-

1. Strength:- as low carbon content is present in mild steel it is generally not that hard compared to other steels.
2. Malleability:- mild steel is highly formable and ductile because its microstructure consists of ferrite and pearlite, making it softer than high-carbon steels.
3. Corrosion resistance:- Mild steel has low resistance to corrosion and rusts pretty easily on exposure to moisture. Also, it is highly reactive and so it is incompatible with strong acids.
4. Magnetism:- Mild steel is magnetic in nature due to presence of high iron content in it.

Applications of mild steel:-

1. Construction material:- As mild steel is highly malleable, its metals sheets are used as roofing material and in pipes used to transport water and gas through structures.
2. Tools:- Due to high affordability and versatility of mild steel, it is often used in tools ranging from pliers to wrenches.
3. Electronic appliances:- As it has high resistance to heat and physical stress, it is used to make electronic appliances like ovens and refrigerators.
4. Containers:- As it has high capacity to resist deforming, it is used to make storage containers like tanks, drums, bins and cans to store and transport food and liquids.
5. Art and decor:- As it has high malleability, it is used ornamate and decorate iron gates, railings and other furniture along with its use to make public sculptures and showpieces.

Experimental Procedure

Heating of sample:- We heated 3 of our samples to a temperature of 1100°C in a muffle furnace and we let them stay at this controlled and constant temperature for about 20-25 minutes.

Heat treatment methods:-

Annealing:- One sample was left to gradually cool down in the furnace only by switching off the furnace

Quenching:- One sample was rapidly cooled down by dipping it in water

Normalising:- One sample was let to gradually allowed to cool in air till room temperature was obtained.

Sample preparation (Polishing):-

Paper Polishing: Removal of any scratches or deformities from the surface of all 4 samples by paper polishing on emery/grit papers of different of different grit sizes of 120, 200, 400, 800, 1200, 1500, 2000. It is done by rubbing the sample in one direction on different papers starting from the lowest grit size and increasing it gradually one paper after the other. On changing every paper sample is rotated at 90° to ensure removal of all possible scratches more efficiently.

Cloth polishing: Polishing the sample on metallographic sample polishing machine. The sample is gently held over a rotating polishing cloth to remove any final scratches and simultaneously adding alumina and water to the polishing cloth of sample polishing machine to fill any final scratches by reducing friction between cloth and sample.

Etching of sample:-

Etching of all 4 samples was done in a 5% Nital solution which is prepared by mixing 5ml of nitric acid (HNo3) in 95ml of ethanol by dipping the sample in the solution for 4-5 seconds and then instantly washing it with water and then drying it. This is done to enhance the microstructure of the sample as the solution selectively reacts with the high energy sites of grain boundaries thereby highlighting them in the microstructure.

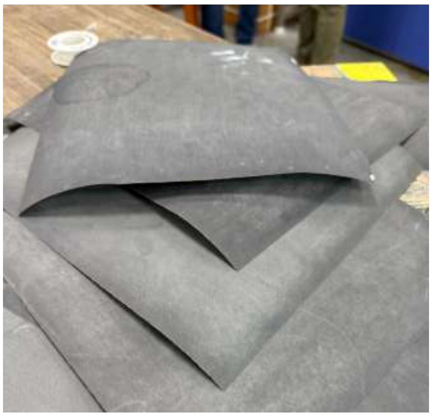
Microstructure analysis:-

We analysed the microstructure of all 4 samples one by one under an optical microscope under the magnifications of 10x, 20x and 30x. The sample was place on the screen on the stage and magnification of objective lenses was varied and coarse and fine adjustments were made to focus the lens on the sample and then the microstructure was viewed using microscopy camera on the computer screen.

Hardness testing:-

Microhardness tester was used to test the hardness of all 4 samples using the Vicker hardness test and record their hardness values. The sample was placed on the stage of the machine followed by adjusting the sample placement using Hand wheel and then impact was made on the sample using the impactor to measure material's hardness digitally.

Apparatus used:-



Grit Paper

Cloth Polishing

Paper Polishing

Microhardness Tester
(Indentation making)

Microhardness Tester

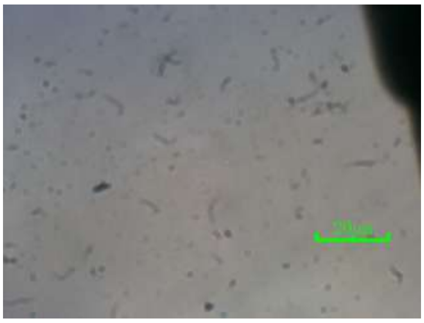
Optical Microscope

Etchant Preparation (Nital)

Results

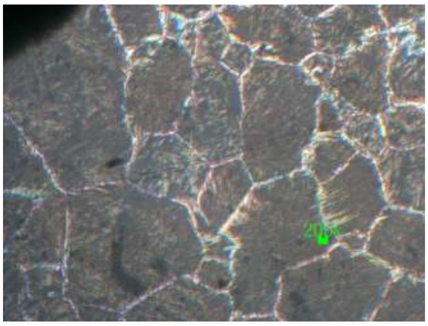
Microstructure:-

1. Reference sample:



10x

2. Quenched Sample:



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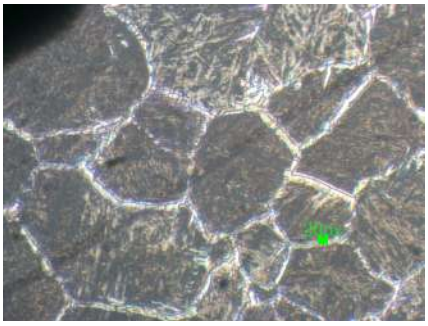

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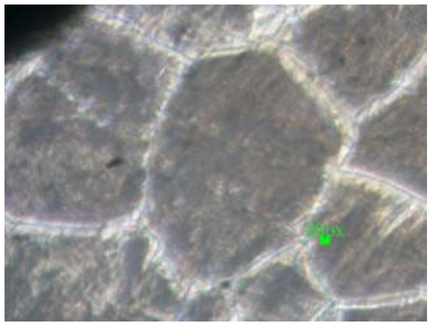
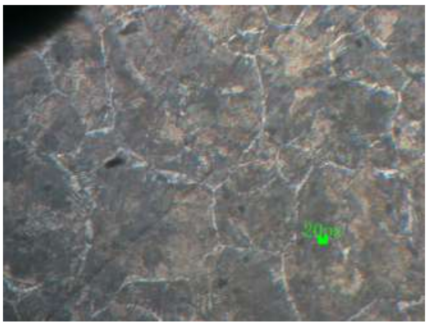


30x

3. Normalised Sample:



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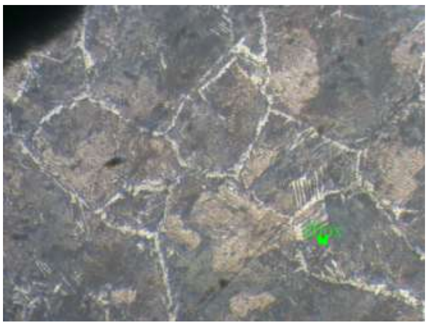
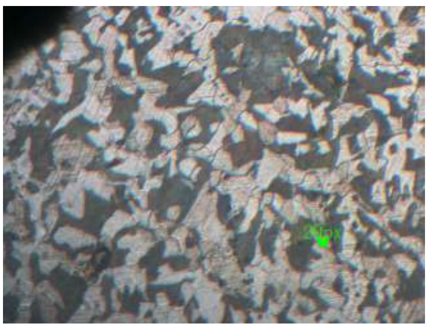


20x

30x

4. Annealed Sample:



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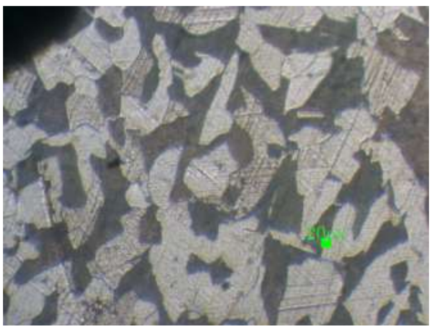
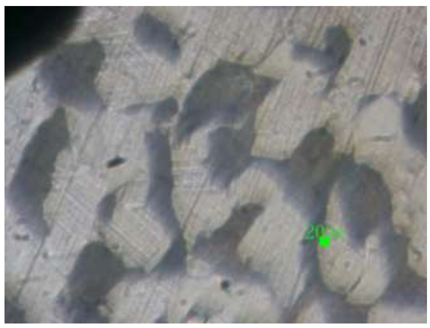


20x

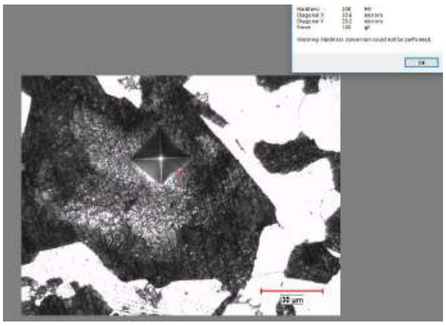


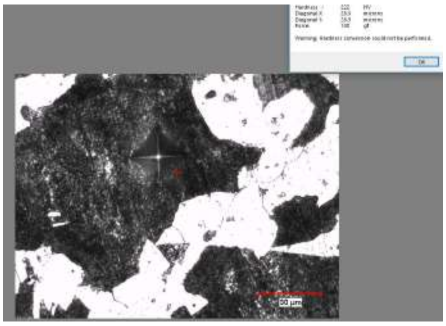
30x

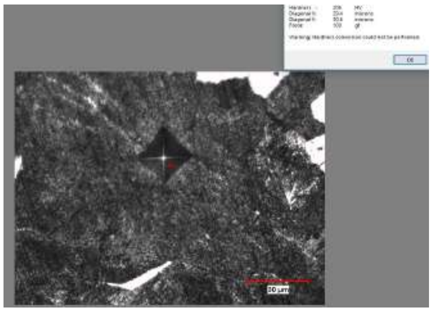
Hardness values:-

1. Annealed Sample:

Average Hardness Value = 212 HV







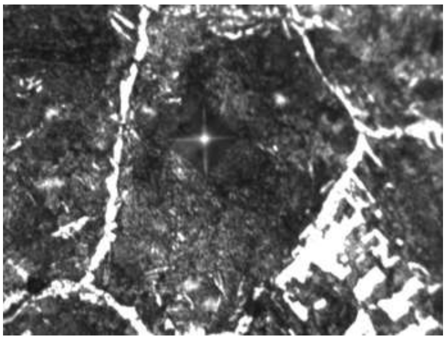
208 HV

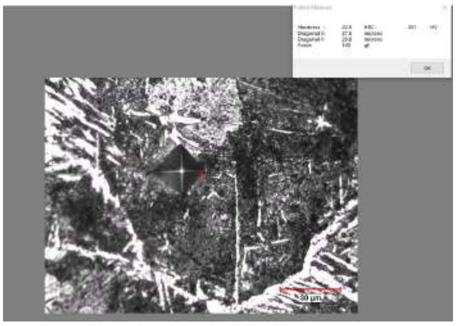
222 HV

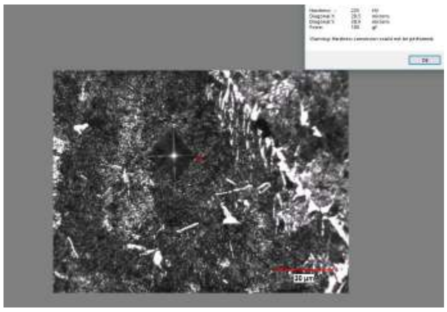
206 HV

2. Normalised Sample:

Average Hardness Value = 238 HV







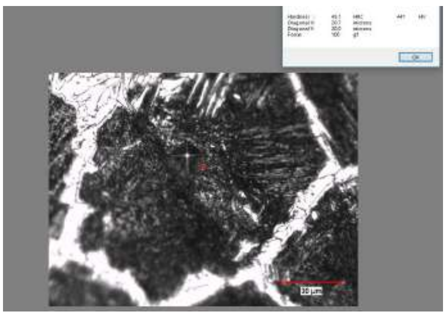
238 HV

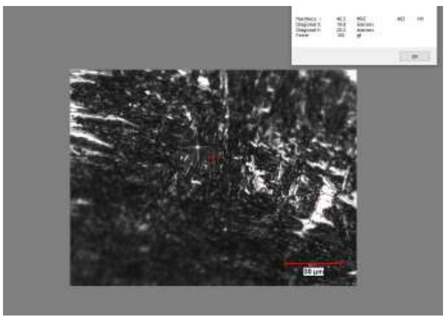
251 HV

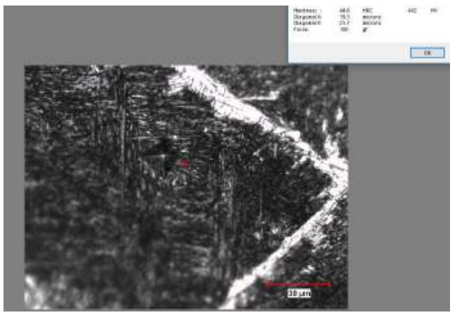
225 HV

3. Quenched Sample:

Average Hardness Value = 450.66 HV







447 HV

463 HV

442 HV

General Observations

Microstructure:

1. For the microstructure of reference sample, mostly ferrite phase is present with a very few dark spots of pearlite phase present in it.
2. For quenched sample, we can see mostly light coloured grains that are shaped Needle-like or plate-like appearing of martensite phase.
3. For normalised sample, we can see light coloured, large and more rounded grains of fine ferrite with a few dark spots of pearlite present on it. Pearlite phase has alternate layers which are of ferrite and cementite.
4. For annealed sample, we can see light coloured, large and more rounded grains of coarse ferrite with a few dark spots of pearlite present on it. Pearlite phase has alternate layers which are of ferrite and cementite.

It can be observed that from annealing to normalising the grains got more fine and well shaped and from normalising to quenching we obtained a new hard phase of ferrite hat is martensite phase. Also The hardened samples contained martensite structures while tempered samples contained granular form of fine ferrite and cementite.

Hardness test:-

1. For annealed sample, average hardness value is 212 HV
2. For normalised sample, average hardness value is 238 HV
3. For quenched sample, average hardness value is 450.66 HV

It can be observed that the rate at which the steel cools to room temperature during the full anneals has a significant impact on the microstructure and room-temperature tensile properties. Increasing the tempering temperature is causing the ductility of steel grade to increase.

It can be observed as we increase the cooling rate that is from annealing to normalising and from normalising to quenching the hardness of our sample increases.

Significance of doing experiment at 1100°C:-

Temperature at which heat treatment is done also affects the microstructure of the sample obtained. For our experiment we performed heat treatment at 1100°C. At higher temperatures, more grain growth takes place if cooling is slow, leading to coarser grains in annealed or normalised steels compared to incomplete phase transformation at lower temperatures as some ferrite and pearlite may remain in the microstructure along with partially transformed austenite.

Also the hardness of sample increases on cooling from higher temperatures as martensite forms due to suppressed carbon diffusion compared to incomplete austenitization which leaves behind softer phases like ferrite or pearlite in case of lower temperatures.

Discussions

1. Reference sample:- The reference sample has only two phases of light coloured ferrite with dark spots of pearlite present on it. A few spots of dark coloured perlite are present as the sample given to us is of mild carbon steel, so the percentage of carbon in our sample is pretty much less thereby decreasing the presence of the pearlite phase too in our microstructure.

2. Annealed sample:- The annealed sample has large round grains of ferrite with a few dark grains of pearlite present on it. As in annealing the material is slowly cooled down, the nucleation of new grains sites is minimised thereby allowing the existing grains to grow into large coarse grains. Also, this slow cooling allows sufficient time for carbon atoms to diffuse evenly thereby forming coarser grains. These coarser grains reduces internal stresses and softens the material thereby improving overall ductility but decreases hardness. Therefore it has the lowest hardness value when compared to the other 2 samples.

3. Normalised sample:- The normalised sample has finer grains of ferrite with a few dark spots of perlite phase present on them. As cooling rate oil faster as compared to annealing, grain growth is limited resulting in finer grain sizes. This also happens because faster cooling increases nucleation sites for new grains during the transformation from the austenite phase to new phases of ferrite and pearlite. These finer grains compared to coarser grains in annealed sample lead to improvement in strength and hardness while maintaining sample's ductility. Therefore it has its hardness value in between that of annealed and quenched samples.

4. Quenched sample:- The quenched sample has a metastable phase present instead of equilibrium phases of ferrite and pearlite which is because of rapid cooling of the sample. This rapid cooling suppresses the process of carbon diffusion thereby forming martensite phase only. Martensite phase has carbon atom trapped inside its lattice thereby making it very hard and of the highest strength but it is most brittle due to internal stresses caused by lattice distortion. Therefore it has the highest hardness value among all the 3 samples.

The micro-structural examination confirmed that the annealed sample contained a microstructure of pearlite, compared to the hardened samples which contained a high percentage of martensite combined with ferrite.

For maximum ductility and minimum toughness, annealing is done to provide satisfactory properties. Normalisation treatment is done to yield greater tensile strength and hardness compared to annealed samples. Quenched sample possessed highest tensile strength and hardness along with lowest ductility and impact strength compared to other heat treated samples.

Conclusion

Through this lab experiment, we performed different heat treatment methods of annealing, quenching and normalisation for a given mild steel sample to observe the changes that take place in our sample's microstructure and hardness values. We heated our samples to a given critical temperature of 1100°C followed by sample preparation. On obtaining the desired samples we did etching in Nital solution and then observed each of the 4 samples under optical microstructure. It was observed that the reference sample has majority ferrite phase with small spots of pearlite phase; the annealed sample had coarse grains of ferrite with a few dark spots of pearlite phase; and normalised sample had finer grains compared to annealed sample. Finally quenched sample had the formation of metastable state of martensite due to rapid cooling. These microstructures also explained the hardness values of our samples which were then measured practically using Vickers hardness test. Quenched sample had maximum hardness, followed by the normalised sample and then finally the annealed sample.

Altogether by performing this experiment, we learned about various heat treatment techniques by practically performing them and their related consequences on our given sample. We verified the theoretically expected results with the practical results obtained on performing the experiment.

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