Quantitative Comparison of Grain Structure in Heat Treated, Nital-Etched Mild Steel and TMT Bars

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Abstract

Steels have microstructures such as ferrite, pearlite, and martensite, which directly influence properties like hardness, strength, and thermal conductivity. The impact of specific heat treatment procedures on mild steel and TMT bars, such as normalizing and quenching in mediums like water, ice, and oil, lead to changes in these microstructures, resulting in variations in mechanical and thermal properties. Nital reagent used for etching steel and other ferrous materials reveals their microstructures. Optical microscopy and image analysis technique is particularly effective in determining the amount or composition of different phases within a heat treated sample. Ice quenching is one that has the maximum (i.e., 85.05) percent of martensite in a mild steel rod, which signifies higher hardness making it suitable for applications where high strength is critical but, ductility is less important. Similarly, water quenched Thermex TMT bar shows a maximum (i.e., 11.73) percent of ferrite, which shows higher ductility as compared with mild steel rod and other TMT rods that makes it better for applications where deformation occurs, whereas normalizing in mild steel results in 73.37% of pearlite which suggests good balance between strength and ductility making it suitable for components that undergo cyclic loading. The conclusion shows the relevance of looking for the proper heat treatment techniques in order to obtain the best mechanical properties for various steels used in wide range of applications.

Keywords: Heat Treatment, Microstructure, Nital, Normalizing, Quenching

1. Introduction

A metal is a substance that has high electrical conductivity, possesses magnetic properties, is malleable, ductile, lustrous, and can form alloys with other metals. An alloy is a substance formed from the combination of two or more metals in unique proportion. Alloys can also be formed from combination of metals and other elements. Steel is the alloy of iron and carbon: a metal and a non-metal. Properties of such alloys can be studied under different environments and conditions using various methods one of which is heat treatment. Heat treatment is the process of altering physical and sometimes chemical properties of a material to achieve desired mechanical properties. Using different heat treatment processes and cooling rates, phases of steel and their composition can be altered to achieve various mechanical properties like ductility, hardness, yield strength, tensile strength and impact resistance (Kandpal et al., 2020).

The primary phases in steel that determine its performance are percentage composition of ferrite, pearlite, and martensite. Ferrite is a soft, ductile phase with a Body-Centered Cubic (BCC) structure (Khurmi and Sedha, 2008). Pearlite is very fine plate like or lamellar mixture of ferrite and cementite. Martensite is hard and brittle phase formed by the rapid quenching of austenite (Shackelford, 2015). Some of the widely used heat treatment processes for steel are annealing, normalizing, quenching and tempering. Normalizing is a heat treatment process applied to carbon and alloy steels, involving heating above the critical temperature followed by air cooling to refine grain structure, enhance strength, hardness and machinability. Quenching is a heat treatment process where hot metal is rapidly cooled, usually in water or ice or oil, to harden it and increase its strength (Kakani and Kakani, 2016).

Several articles have shown that normalized steel has higher ultimate tensile and yield strength compared to anneal steel (Senthilkumar and Ajiboye, 2012). The mechanical properties of mild steel can be changed and improved by various heat treatments for applications. The annealed steel, which mainly has a ferrite structure, has the lowest tensile strength and hardness values and the highest ductility values compared to hardened and tempered steel (Sultana et al., 2014). After normalizing, the strength of TMT steel increases, while the grain size decreases. Additionally, the proportion of pearlite increases, and the proportion of ferrite decreases (Saikot, 2024). The best annealing process for carbon steel ASTM 285 Gr. C is at 860°C with a soaking time of 30 minutes inside the furnace, and this process does not lead to the loss of elements. With increasing the temperature of the annealing process, the strength of ASTM 285 Gr. C decreases until it reaches around 350 MPa at 1000°C. Carbon steel ASTM A285 Gr. C loses elements when it is normalized by cooling in air (Ahmed, 2020).

Dahiwade et al. study the influence of steel hardness during hot rolling processes by annealing and normalizing. They observed that after normalization, the hardness of the steel specimen is higher than that found in annealing because the cooling rate is faster in normalizing rather than in annealing. In contrast, the uniform steel specimen microstructure includes finer grains relative to the structure of annealed steel (Dahiwade et al., 2014). By using low carbon steel, At-Qawabah et al. studied the influence of different annealing temperature on the mechanical characteristics, microstructure, micro hardness, and impact toughness. They used several temperatures annealing regimes: 820, 860, 900 and 940 ˚C. The impact energy has been found to be increased as the temperature of the annealing increases; the average is 22.5 percent that has been reached at 900 °C (Qawabah et al., 2012).

In summary, this discussion is about how the heat treatments like normalizing and quenching impacts the properties of steel. This study focuses on the effect of different heat treatment methods on mild steel rod (sample 1) and comparison of properties of Thermex TMT bar (sample 2) and TMT bar (sample 3) on those heat treatments. The percentage composition of Martensite, Ferrite and Pearlite is observed on subjecting all the sample specimens to quenching and normalizing. There has not been a significant research report on effect of different heat treatments on Thermex TMT bar (sample 2) and TMT bar (sample 3). This investigation focuses on analyzing the properties of these steels giving a better understanding of their compositions.

2. Materials and Methodology

2.1 Materials, Equipment, and Analytical Tools

2.1.1 Test Specimen

In this study, the test specimens of mild steel bar (sample 1), Thermex TMT bar (sample 2) and TMT bar (sample 3) with diameter 20mm and length 25mm were considered for heat treatment, etching and microscopic analysis.

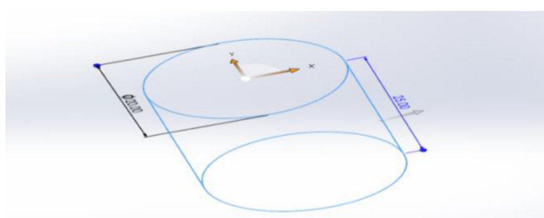

Figure 1: Model of Sample Specimen

2.1.2 Equipment and Instruments Used

A furnace is used to provide controlled heating and cooling environment for heat treatment of the sample specimen. Labpol Duo Twin Grinder contains a dual wheel setup which allows for both rough grinding and fine polishing of sample specimens. HandiMet 2 Roll Grinder is a grinder with four steps grinding stations and controlled water flow which provides a high-quality surface finish to the sample specimen. An optical microscope uses visible light and a system of lenses used to generate a magnified image of the sample which is used to study microstructure of different metals and alloys. The equipment and instruments used in the investigation is shown in figure 2.

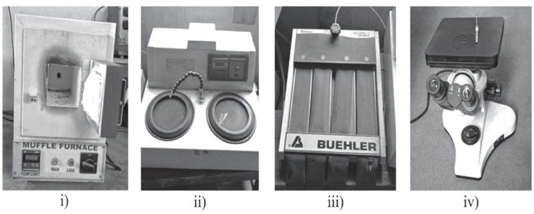

Figure 2: Equipment and Instruments Used, i) Muffle Furnace, ii) Labpol Duo Twin Grinder, iii) HandiMet 2 Roll Grinder, and iv) Optical Microscope

2.1.3 Etching Reagents and Chemicals

The chemical used for etching is Nital reagent. It is a mixture of 10% nitric acid and 90% ethanol. It is used for etching steel and other ferrous materials which reveals their microstructures for examination under a microscope.

2.1.4 Microstructure Imaging and Analysis Tool

ImageJ software is used for analysis of microstructural images. It is an open-source image processing software developed by Wayne Rasband at the NIH (Rasband & Contributors).

2.2 Methodology

This study aims to determine the proportion of martensite, ferrite and pearlite in the heat treated sample specimens using optical microscopy, etching techniques and image analysis method. To achieve this objective the investigations includes several methods to be followed starting with pre-investigation and going through heat treatment, investigation and analysis.

In Pre-investigation phase the materials were accumulated and samples of required dimensions were prepared. Then in heat treatment phase the samples were heated at 850°C and held at that temperature for 35 minutes. Normalizing, and quenching treatment (in ice, water and oil) was done with the samples. The samples were polished and then etched using Nital reagent before microscopic observation. The etching time for the samples was found by performing hit and trial. Microstructural image of the samples as seen in the optical microscope was captured for analysis. The phase compositions of martensite, ferrite and pearlite were determined using image analysis method with the help of imageJ software. The obtained results were then interpreted to draw a conclusion and finally the documentation of the investigation and study was done. The methodological flowchart of the tasks performed from the beginning to the end of the research is shown in figure 3.

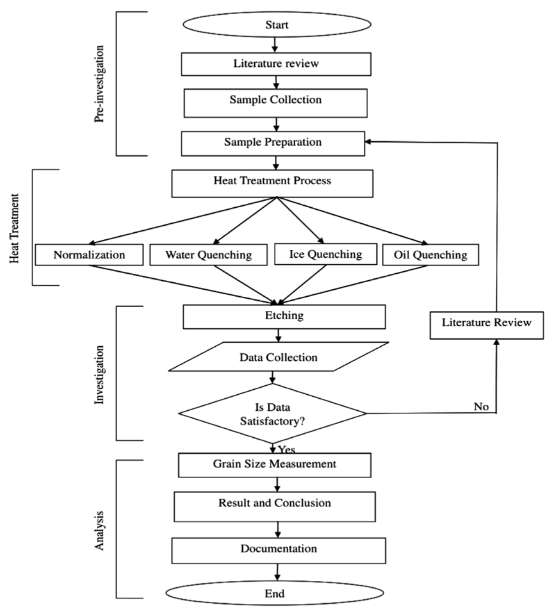

Figure 3: Methodological Flowchart

3. Result and Discussion

The sample specimens were heat treated at 850^{o} C with soaking time of 35 minutes and observed under optical microscope after etching with Nital reagent. The percentage of martensite, ferrite and pearlite in the sample were calculated through image analysis method using imageJ software.

3.1 Results

3.1.1 Mild Steel Bar (Sample 1)

The microstructural images of mild steel bar obtained from optical microscope is shown in figure 4 and results obtained from image analysis is shown in table 1.

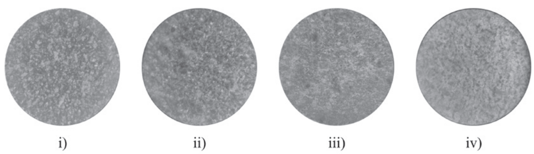

Figure 4: Microstructure of Mild Steel Bar for i) Water Quenching, ii) Ice Quenching, iii) Oil Quenching and iv) Normalizing

Table 1: Grain Measurement of Mild Steel Bar

S. N.	Heat Treatment Process	Martensite (%)	Ferrite (%)	Pearlite (%)
1.	Water Quenching	66.67	2.10	31.23
2.	Ice Quenching	85.05	8.43	6.52
3.	Oil Quenching	37.78	9.96	52.26
4.	Normalizing	23.05	3.57	73.37

3.1.2 Thermex TMT Bar (Sample 2)

The microstructural images of Thermex TMT bar obtained from optical microscope is shown in figure 5 and results obtained from image analysis is shown in table 2.

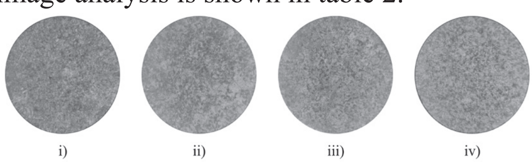

Figure 5: Microstructure of Thermex TMT Bar for i) Water Quenching, ii) Ice Quenching, iii) Oil Quenching and iv) Normalizing

Table 2: Grain Measurement of Thermex TMT Bar

S. N.	Heat Treatment Process	Martensite (%)	Ferrite (%)	Pearlite (%)
1.	Water Quenching	22.03	11.73	66.24
2.	Ice Quenching	73.16	0	26.83
3.	Oil Quenching	36.5	0.41	63.09
4.	Normalizing	28.55	6.33	65.11

3.1.3 TMT Bar (Sample 3)

The microstructural images of TMT bar obtained from optical microscope is shown in figure 6 and results obtained from image analysis is shown in table 3.

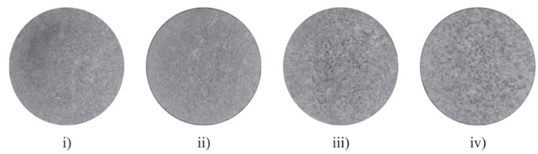

Figure 6: Microstructure of TMT Bar for i) Water Quenching, ii) Ice Quenching, iii) Oil Quenching, and iv) Normalizing

Table 3: Grain Measurement of TMT Bar

S. N.	Heat Treatment Process	Martensite (%)	Ferrite (%)	Pearlite (%)
1.	Water Quenching	58.11	9.02	32.87
2.	Ice Quenching	76.25	0	23.75
3.	Oil Quenching	44.11	0	55.89
4.	Normalizing	32.41	3.37	64.21

3.2 Discussion

The obtained microstructure compositions of martensite, ferrite, and pearlite for mild steel, Thermex TMT, TMT bar shows noticeable differences based on the heat treatment processes. In mild steel, water quenching resulted in 66.67% martensite, 2.10% ferrite, and 31.23% pearlite, indicating a rapid cooling rate, leading to a hard yet brittle structure. Ice quenching further increased the martensite content to 85.05%, making it extremely hard but likely to brittleness. In contrast, oil quenching showed a balanced microstructure with 37.78% martensite and 52.26% pearlite, suggesting a slower cooling rate. Normalizing produced the softest structure with only 23.05% martensite and a high pearlite content of 73.37%.

For Thermex TMT bar, water quenching yielded 22.03% martensite, 11.73% ferrite, and 66.24% pearlite, showing a relatively softer structure than mild steel. Ice quenching has increased martensite composition of 73.16%, with no ferrite, showing a strong but brittle microstructure. Oil quenching resulted in 36.5% martensite and 63.09% pearlite, making it slightly softer than ice-quenching but suitable for applications requiring moderate hardness. Normalizing produced 28.55% martensite and 65.11% pearlite, resulting in a predominantly ductile structure.

In TMT bar, water quenching produced 58.11% martensite, 9.02% ferrite, and 32.87% pearlite, similar to mild steel, indicating high hardness with some toughness. Ice quenching yields 76.25% martensite and 23.75% pearlite, making it the hardest among all three materials. Oil quenching led to 44.11% martensite and 55.89% pearlite, representing a balanced microstructure, while normalizing showed 32.41% martensite and 64.21% pearlite, providing a tough and ductile structure.

The determination of microstructure percentages may have uncertainties due to slight variations in the heat treatment processes, such as minor changes in quenching duration and cooling rates, which can impact the final phase compositions. Additionally, inconsistencies during polishing or etching with Nital can affect the visibility of grain boundaries under the optical microscope. The use of ImageJ software for quantitative analysis may introduce errors due to pixel threshold settings and manual boundary identification, which could lead to slight variations in phase proportions.

4. Conclusion

The experiment was done in the metal samples of mild steel and TMT bars that are heat treated at 850°C, followed by cooling techniques, normalizing and quenching (ice, water, and oil). The heat treated sample was then polished and etched using Nital reagent. The microstructural image was taken using an optical microscope, and image analysis was done to find out the composition of different phases specifically martensite, ferrite and pearlite in the heat treated samples using ImageJ software. Ice quenching in mild steel resulted in the highest martensite composition which signifies higher hardness and lower ductility. Normalizing the samples yielded similar pearlite compositions in all samples greater than 60% which indicates better ductility. This study highlights the critical role of heat treatment parameters and cooling mediums in determining the phase composition of metals. Future experiments could be done in other temperatures between 760^{o}C and 950^{o} C and with other etching reagents like Picral, Vilella's reagent, Klemm's reagent, or Marble's Reagent to further understand the microstructure of metals.

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