MECHANICAL CHARACTERSIATION AND MICROSTRUCTURE ANALYSIS OF TRIPLE PHASE MEDIUM CARBON STEELS

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CONTENTS

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ABSTRACT

Medium carbon steels are a widely utilized set of metals in the production industry. Various mechanical components such as gears, spindles, shafts etc. are made from medium carbon steels. Owing to its importance in the industry, the project undertaken aims to harness and utilize various advantageous properties from the different phases of steels namely; Ferrite, Bainite, Martensite, Austenite, Pearlite. The steel specimens will be subjected to Heat Treatment processes in order to enhance and investigate properties in question, such as Hardness, tensile strength and brittleness. The specimen was to be Normalized by heating to 900 degrees Celsius and then cooled in air; after a soaking period of 2 hours. The previously normalized specimen will then be heated at a temperature of 750 degrees Celsius, and be allowed to soak for 2 hours which will predictably create a Ferrite + Austenite dual phase. The specimen is then Austempered in a pre-heated salt bath ($NaNO_2$ + $NaNO_3$) in another furnace at 350 degrees Celsius; the end product of which will give us Ferrite + Bainite dual phase. Lastly, the specimen is then rapidly Quenched in water after which the third and final Martensite phase is obtained. The microstructure will then be determined by Scanning Electron Microscopy and hardness testing will be done on the Rockwell Hardness Testing Machine.

1. INTRODUCTION

Medium carbon steels display superior strength properties and are important members in the low alloy category. These steels have a carbon content between 0.30 and 0.60%, and manganese between 0.60 and 1.65% and show a positive response to all types of heat treatment methods. The project undertaken, aims to compare properties like Hardness, and experimentally investigate and analyze the Microstructures of triple phase ***EN19, EN8 and EN24 steels.***

1.1 EN19:

Untreated, mass produced EN19 steel has good ductility, tensile strength and shock resistance thus finding its way into a myriad of engineering applications such as machine tools and automobile component. Despite all of the positive properties, it still displays relatively low wear resistance and hence, preferably has to be heat treated in order to optimize its properties to further maximize its potential and safety in application. Its typical applications involve:

- General purpose axles
- Gears
- Bolts and studs
- Spindles

Chemical	Composition
Fe	96.86%
C	0.38%
Si	0.21%
Mn	0.91%
Cr	1.04%
Ni	0.23%
Mo	0.23%

1.2 EN8:

EN8 is another commonly used medium carbon steel, which displays improved strength and ductility over mild/ Low carbon steel. EN8 finds its way into innumerable applications owing to its property of being readily machinable in any condition.

EN8 steels are generally used in original as bought, untreated condition; but we aim to further heat treat its specimen in order to make it viable for producing components with enhanced wear resistance. Its typical applications involve:

- Shafts
- General axles
- Gears
- Bolts and studs
- Spindles

Chemical	**Composition**
Fe	98.6%
C	0.4%
Si	0.2%
Mn	0.75%
P	.05%
S	.0005%

1.3 EN24:

EN24 is a nickel-chromium-molybdenum combination medium carbon steel which offers high tensile steel, with superior ductility and relatively better wear resistance characteristics. With relatively good impact properties at low temperatures, EN24 is also suitable for a variety of Heat Treatment procedures. Its typical applications involve:

- High strength shafts
- Drill bushings
- Gears
- Retaining rings
- Punches
- Dies

Chemical	**Composition**
Fe	95.79%
C	0.38%
Si	0.22%
Mn	0.62%
Cr	1.21%
Ni	1.44%
Mo	0.27%
P	0.03%
S	0.04%

Comprehending the relations between structural and mechanical properties of highly versatile steels is crucial in order to further design and develop various high-strength, multi-phase steels. In the project, we aim to carry out a comparative study and define the structure-property relationships of EN8, EN19 and EN24 steels with Heat and Triple Phase Treatment.

2. LITERATURE REVIEW

The purpose of this study, was an attempt to co-relate our project with previously written works in order to formulate prospective results, and to establish a base line of expectations for the heat treatment of steels in order to obtain triple phase. Triple phase Heat treatment of prominent medium carbon steels is a well sought-after branch of metallurgy, which aims to harness and optimise properties and strengths from different phases of steel into a single compound structure. Medium carbon steels are the most commonly used steels for various mechanical components and hence it is only fitting that we try and find the most optimum combination of phases in order to boost its utility in the industry.

Volume fraction of the phases, has first and foremost the greatest effect on various mechanical properties of the steel like hardness, tensile strength and ductility. In a study, steel bars were heat treated by being austenitized at 900 °C for 1 hour followed by inter-critical annealing at 740 °C for 100 min. Lastly, they were quenched in a salt bath maintained at a temperature of 300 °C, and then subsequently held at different times to obtain triple phase microstructures with 34 vol.% fraction ferrite and various martensite (or bainite) contents. Results of the tensile and hardness tests showed that with increasing martensite volume fraction, yield strength, ultimate tensile strength and hardness increase, hence showing a correlation to higher martensite volume fraction and higher strength of ferrite and higher residual stresses. The increment of martensite volume fraction also increased ductility. In another study observing ferrite-pearlite-martensite triple phase in medium carbon steels, various step-quenching heat treatment cycles were carried out at 650 °C for different holding times, followed by subsequent water quenching after re-austenitization of the specimens at 860 °C for 30 min. The ferrite-pearlite-martensite triple phase samples consisting of more than 70% martensite were associated with a higher level of mechanical properties compared to those of the ferrite-martensite DP samples; as observed by scanning electron microscopy with X-ray dispersive spectroscopy. The combination of these two studies show that martensite volume fraction is an advantageous quantity to achieve due to superior mechanical properties. Also, the combination of a pearlite triple phase along with ferrite-martensite, only complemented the hardness and created a specimen with better mechanical properties than dual phase.

Temperature resistance and cryogenic ability is another important aspect of treated steels which has been experimented on in the past. After heat treating a low Mn medium carbon steel, and obtaining a triple phase microstructure of 75% ferrite, 15% bainite and 10% retained austenite, specimens of this steel with triple phase structure were tensile tested at temperature range of 25–450 °C. Stress–strain curves showed serration flow at temperature range of 120–400 °C and smooth flow at the other temperatures. All the obtained stress–strain curves at various temperature ranges showed discontinuous yielding at all testing temperatures. This study showed that both yield and ultimate tensile strength decreased with increasing temperature, but there exists a temperature region (120–400 °C) where a reduction of strength with increasing temperature is retarded or even slightly increased. The variation in the mechanical properties with temperature was correlated to the effects of dynamic strain aging, high temperature softening, bainite tempering and austenite-martensite transformation during deformation. Certain studies such as this shed light and put an emphasis on strain responses on different operational temperatures of the steels.

Another chain of thought was to experiment the advantages and disadvantages of uninterrupted quenching as opposed to step quenching. This research intended to keep with our study by experimenting operational temperatures. This study stemmed from the need to store and transport volatile fluids with high vapour pressure at cryogenic temperatures such as -40 Celsius. To ensure ambient and cryogenic temperature toughness, the microstructure of the second phase needs to comprise predominantly fine-grained lower bainite or fine-grained lath martensite, or mixtures thereof. It was deemed preferable to substantially minimize the formation of embrittling constituents such as upper bainite, twinned martensite and martensite-austenite in the second phase. While martensite adds mechanical properties it also has an adverse effect by substantially increasing brittleness and hence showed be avoided when dealing with large temperature ranges and cryogenic temperatures. In order to complement brittleness, tensile strength is a very important property in medium carbon steels which needs to be throttled and controlled very closely. Referring to a study which was along similar lines as our current study; AISIh4140 steel specimens were heated at 850˚C for 1 hour. They were then placed at 720˚Chfor 3 min and transferred into a salt bath with different temperatures of 380, 400, 420 and 450˚C for 4 min and finally were quenched in water. Tensile test revealed that increasing the salt bath temperature also commonly known as Austempering temperature decreased yield strength, ultimate tensile strength and elongation. Fractography of tensile specimens by stereo microscopes showed, with increasing austempering temperature, the fracture surface changed from ductile to brittle. In addition to this, by studying a similar research on effect of tempering and heat treatment on microstructures of medium carbon steels where tempering was performed for triplehphase microstructures at 250, 450 and 650ºC forh90 min, microstructural studies and mechanical tests such as tensile and hardness showed continuously reduction of yield strength and ultimate tensile strength, couple by an increase of elongation with increasing tempering temperature. The fracture surfaces of tensile tested specimens showed more ductile behaviour at 650C tempering temperature which was in agreement with other mechanicalhproperties and studies.

In conclusion, these correlations between tempering temperatures and mechanical properties such as tensile strength, yield strength, hardness and ductility were crucial in order to streamline the current research and establish a baseline as per which our project would be undertaken. We have found that it is essential to balanced martensite phase with either bainite or pearlite phase, in order to reduce the high brittleness which martensite adds to the structure. We can also derive that in order to achieve suitable tensile strength we need to throttle the austempering temperatures very closely and prevent using temperatures which are too high to avoid the risk of continuously reducing yield strength and ultimate tensile strength.

3. OBJECTIVES

To perform triple phase treatment of EN8, EN19 and EN24 steels.

- Mechanical Characterization of obtained Triple Phase steel
  - Hardness Testing

- Microstructure analysis by Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) analysis.

4. METHODOLOGY

- We begin by procuring the materials in focus and processing them using the required CNC or Lathe operations into 3 specimens upon which Hardness testing and Microstructure analysis will be carried out.

- The specimens will further be subjected to Heat Treatment processes in order to enhance and investigate the properties in question.

- The first process to be carried out is *Normalizing*. Normalizing refers to the process of heating a metal at high temperatures; generally, near its recrystallization temperature, allowing it to soak in the same temperature under furnace conditions for an appropriate period of time and then removing it from the furnace and allowing it to cool under atmospheric conditions. The properties of normalized materials are slightly inferior to those of annealed materials because of the creation of multiple cooling zones with varying cooling rates owing to the atmospheric cooling; hence creating an aspect of inconsistency through the material; which can be tinkered with through further heat treatment.

- In this project, the specimen will be Normalized by heating to 900 degrees Celsius and then cooled in air; after a soaking period of 2 hours. Predictably, austenization will take place through this process.

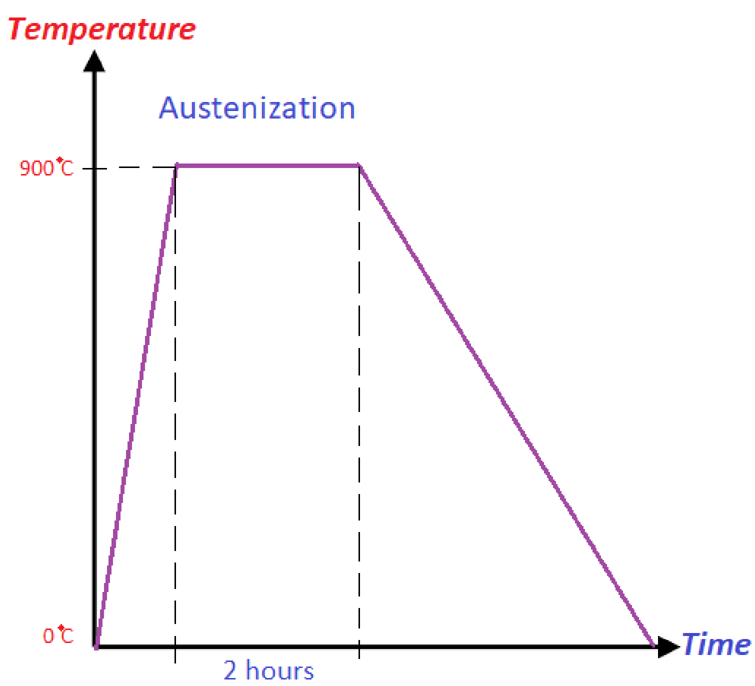

Fig 1. Austenization TT Graph

- Next, we will be proceeding by heating the previously normalized specimen at a temperature of 750 degrees Celsius, and allowing it to soak for 2 hours which will predictably create a *Ferrite + Austenite* dual phase

- The specimen is then transferred to a pre-heated salt bath ($NaNO_2$ + $NaNO_3$) in another furnace at 350 degrees Celsius. This process is also referred to as *Austempering*, the end product of which will give us *Ferrite + Bainite* dual phase. Austempering is a heat treatment process for medium-to-high carbon ferrous metals which is used to enhance material properties namely strength, toughness, shock resistance and ductility.

- After removing it from the furnace, the specimen is then rapidly *Quenched* in water after which the third and final phase called *Martensite* is obtained. The quenching process greatly enhances the materials hardness and abrasive resistance.

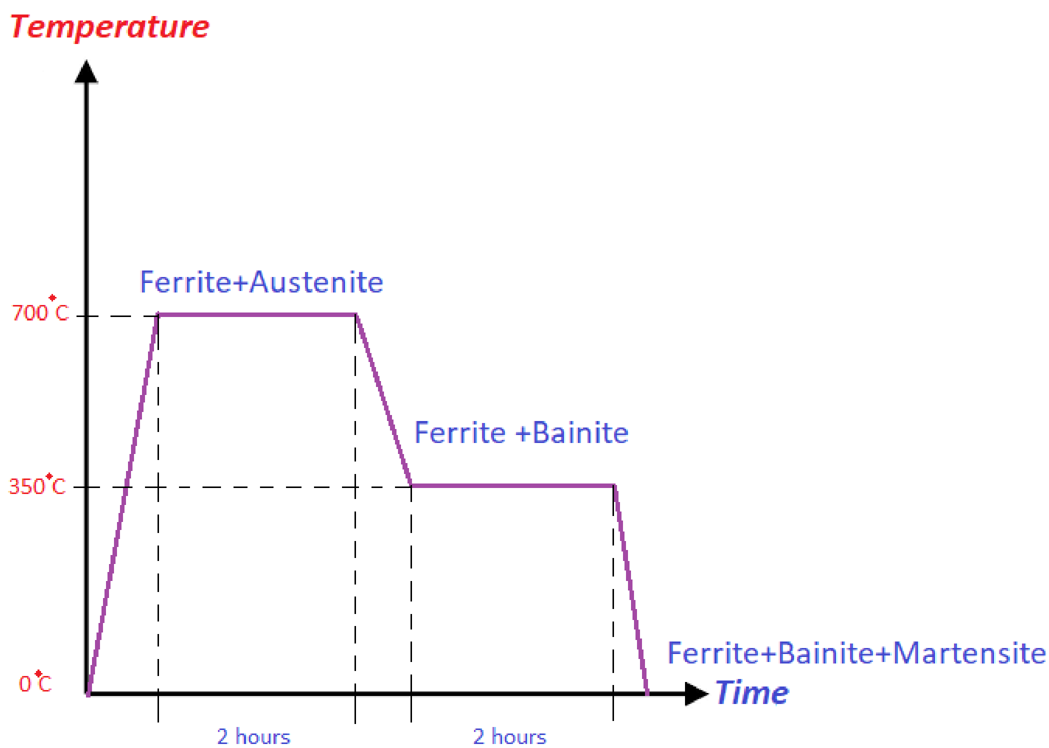

Fig 2. Triple Phase TT graph

- The above stated procedure in its entirety is called ***Triple Phase Treatment***, and the end product obtained, will exhibit a *Ferrite + Bainite + Martensite* triple phase.

- Once triple phase specimen preparation is completed, the following mechanical characterization tests are carried out:

    1. **Hardness Test (Rockwell test)**
    2. **SEM Analysis (Scanning electron microscopy)**

4.1 Specimen preparation:

In order to carry out Triple Phase analysis, we were required to prepare 3 hardness specimens of EN8, EN19 and EN24 each of dimensions 15mm in diameter and 10mm in length. The Steps involved are as follows:

- Material was ordered from Hi-Tech Sales Corporation in Mangalore. We received 1m rods of each of the materials 25mm in diameter.

- The rods were first cut into half meter segments using the Bench Cutter.

- The half meter segments were then loaded on the lathe for Turning down to the required 15mm diameter.

- After the diameter was reduced, the rod was once again processed on the bench cutter in order to cut it into 12mm length segments, leaving 1mm allowance on each side.

- The 12mm specimens were then Faced on the lathe machine and brought down to the required 10mm length

- Hence 9 specimens, 3 each for EN8, EN19 and EN24 were obtained through this process.

Fig 3. EN8 specimen

Fig 4. EN 19 specimen

Fig 5. EN 24 specimen

4.2 Rockwell Hardness Testing:

Rockwell hardness testing is a general method fordmeasuring the bulk hardness of metallic and polymer materials. Although hardness testing does not give a direct measurement of any performance properties, hardness of a material correlates directly with its strength, wear resistance, and other properties.

It is an indentation testing method, where the indenter is either a conical diamond or a hard steel ball. Different indenter ball diameters from 1/16 to 1/2 in. are used depending on the test scale.
To start the test, the indenter is placed upon the control surface of the the sample at a prescribed minor load. A major load is then applied and held for a set time period. The force on the indenter is then decreased back to the minor load. The Rockwell hardness number is calculated from the depth of permanent deformation of the indenter into the sample, i.e. the difference in indenter position before and after application of the major load. The minor and major loads can be applied using dead weights or springs. The in enterdposition is measured using an analog dial indicator or an electronic device with digital readout.

The various indenter types combined with a range of test loads form a matrix of Rockwell hardness scales that are applicable to a wide variety of materials. Each Rockwell hardness scale is identified by a letter designation indicative of the indenter type and the major and minor loads used for the test. The Rockwell hardness number is expressed as a combination of the measured numerical hardness value and the scale letter preceded by the letters, HR. For example, a hardness value of 80 on the Rockwell A scale is reported as 80 HRA.

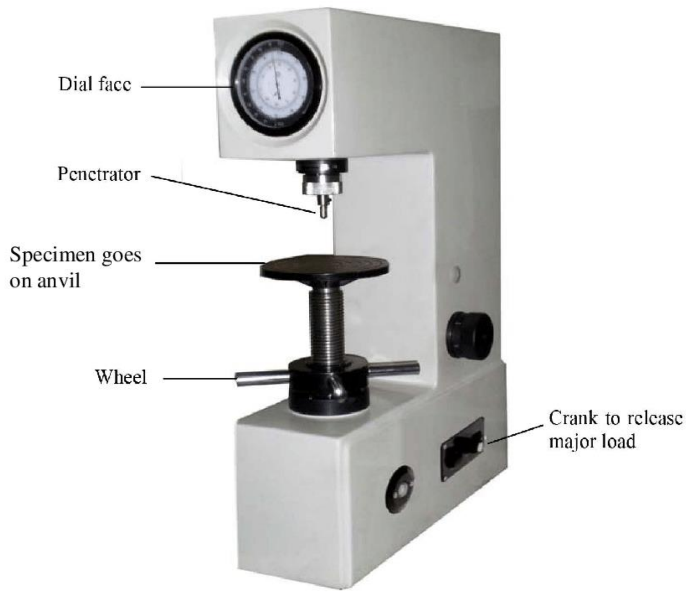

Fig 6. Rockwell Hardness Testing Machine

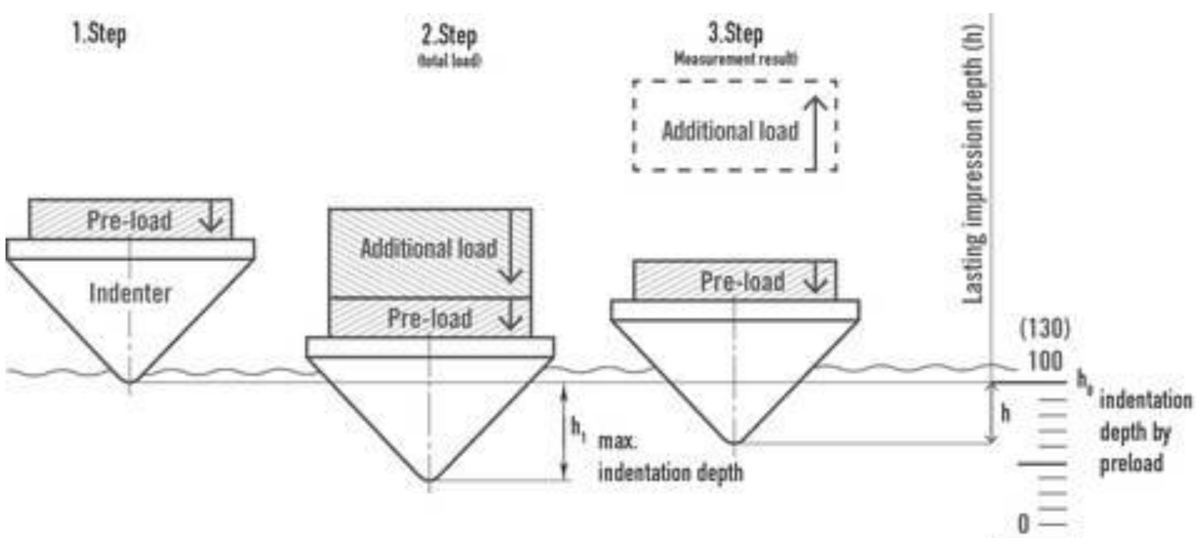

Fig 7. Rockwell Hardness Testing procedure

4.3 Scanning Electron Microscopy (SEM Analysis)

Scanning electron microscopy (SEM) is a method for high-resolution imaging of surfaces. The SEM uses electrons for imaging, much as a light microscope uses visible light. The advantages of SEM over light microscopy include much higher magnification (>100,000X) and greater depth of field up to 100dtimes that of light microscopy.

The SEM generates a beam of incident electrons in an electron column above the sample chamber. The electrons are produced by a thermal emission source, such as a heated tungsten filament, or by a field emission cathode. The energy of the incident electrons can be as low as 100 eV or as high as 30 keV depending on the evaluation objectives. The electrons are focused into a small beam by a series of electromagnetic lenses in the SEM column. Scanning coils near the end of the column direct and position the focused beam onto the sample surface. The electron beam is scanned in a raster pattern over the surface for imaging. The beam can also be focused at a single point or scanned along a line for x-ray analysis.

To create an SEM image, the incident electron beam is scanned in a raster pattern across the sample's surface. The emitted electrons are detected for each position in the scanned area by an electrond etector. The intensity of the emitted electron signal is displayed as brightness on a display monitor and/or in a digital image file. By synchronizing the position in the image scan to that of the scan of the incident electron beam, the display represents the morphology of the sample surface area. Magnification of the image is the ratio of the image display size to the sample area scanned by the electron beam. The incident electrons cause electrons to be emitted from the sample due to elastic and inelastic scattering events within the sample’s surface and near-surface material. High-energy electrons that are ejected by an elastic collision of an incident electron, typically with a sample atom’s nucleus, are referred to as backscattered electrons. The energy of backscattered electrons will be comparable to that of the incident electrons. Emitted lower-energy electrons resulting from inelastic scattering are called secondary electrons. Secondary electrons can be formed by collisions with the nucleus where substantial energy loss occurs or by the ejection of loosely bound electrons from the sample atoms. The energy of secondary electrons is typically 50 eV or less.

Scanning electron microscopy has various secondary methods and modes of procuring an image as follows;

Secondary Electron Imaging - This mode provides high-resolution imaging of fine surface morphology. Inelastic electron scattering caused by the interaction between the sample's electrons and the incident electrons results in the emission of low-energy electrons from near the sample's surface. The topography of surface features influences the number of electrons that reach the secondary electron detector from any point on the scanned surface. This local variation in electron intensity creates the image contrast that reveals the surface morphology.

Backscatter Electron Imaging - This mode provides image contrast as a function of elemental composition, as well as, surface topography. Backscattered electrons are produced by the elastic interactions between the sample and the incident electron beam. These high-energy electrons can escape from much deeper than secondary electrons, so surface topography is not as accurately resolved as for secondary electron imaging. Higher atomic number material appears brighter than low atomic number material in a backscattered electron image. The optimum resolution for backscattered electron imaging is about 5.5 nm.

Variable Pressure SEM - Traditionally, SEM has required an electrically-conductive sample or continuous conductive surface film to allow incident electrons to be conducted away from the sample surface to ground. If electrons accumulate on a nonconductive surface, the charge buildup causes a divergence of the electron beam and degrades the SEM image. In variable-pressure SEM, some air is allowed into the sample chamber, and the interaction between the electron beam and the air molecules creates a cloud of positive ions around the electron beam. These ions will neutralize the negative charge from electrons collecting on the surface of a nonconductive material. SEM imaging can be performed on a nonconductive sample when the chamber pressure is maintained at a level where most of the electrons reach the sample surface, but there are enough gas molecules to ionize and neutralize charging. Variable pressure SEM is also valuable for examination of samples that are not compatible with high vacuum.
Field Emission SEM (FESEM) - SEMs that use a thermal emission source (i.e., tungsten filament) to generate the electron beam are generally adequate for most samples and provide satisfactory resolution at magnifications up to about 100,000X. However, for high resolution and high magnification imaging a cold field emission (FE) gun provides the best resolution available for SEM. The cold FE gun extracts electrons from the FE cathode by applying a strong electrical field close to a very sharp tip. This method of electron extraction results in a higher electron yield and a smaller beam size, which thus provides a brighter signal with better resolution.
The useful magnification for FESEM imaging ranges up to 500,000X. A second advantage of FESEM is that high resolution imaging can be performed with very low accelerating voltages. At low voltage, very fine features are more readily observed and many non-conductive materials can be examined without applying a conductive coating. Low voltage FESEM examination is ideal for imaging nano-materials, polymers, and thin films.

5. CONCLUSION

The triple phase structure of medium carbon steels has greatly advantageous properties as compared to dual phase steels and superior hardness and ductility. The study shows us that martensite volume fraction is a crucial factor in determining brittleness of material and that the austempering temperature should not be held too high, in order to get superior tensile strength. Through further experimentation along similar lines, the usage and adaptability of medium carbon steels can be greatly enhanced in the industry by heat treatment and tempering it to produce superior mechanical properties.

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