Microstructural study of quenched EN 31 steel in biodegradable oils

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***Abstract*: The effect of quenching at biodegradable oils in various proportions on EN 31 steel is studied in the present work. This steel is austenitized and is quenched in different media of biodegradable oils; groundnut oil, neem oil, and its combinations. Micro structural analysis is carried find the effect of quenching medium on EN-31 steel. In this experiment mainly 4 samples of EN 31 Steels are used.ie, pure neem oil, pure groundnut oil, 60:40 ratios of both. Finally, observed that the hardness of the sample with 100% groundnut oil has highest & combination of 60% neem oil and 40 % groundnut oil has less. The martensite concentration is also reveals the same in metallographic observation.ie, pure groundnut oil has higher concentration of martensite of about 34.68% & sample of 60% neem oil and 40% groundnut oil has lesser concentration of 9.37%. Martensite concentration increases the hardness is also increases. So the Microscopic study as well as Rockwell hardness test reveals the same result.**

***Index Terms*: EN-31, Austenitization, Martensite, Biodegradable, Rockwell hardness test.** *(Key words)*

I. INTRODUCTION

Microstructural properties can be altered by heating the metal to definite temperature and then allowing it to cool at suitable rate. This heating and cooling of metals is termed as heat treatment. Heat treatment is done not only in metals but also in ceramics and composite materials. The purpose of heat treating is to improve the mechanical properties of steel. The electrical conductivity, corrosion and thermal conductivity are also altered during the process of heat treatment. The beneficial changes that occur in the microstructure do not take place during the heating process, but during the cooling or quenching from high temperature to low temperature [1]. Hence the quenching medium depends on the hardenability of a particular alloy, the selection of thickness and shape, and the cooling rate needed to achieve desired microstructure [2]. The commonly used quenching medium include water, oil, brine, and synthetic solution. Water quenching is faster and economical. But it has a drawback of forming cracks or effecting dimensional changes due to high cooling rate and oil does not induce enough hardness. This creates a need of a suitable quenching medium. Hence, biodegradable oil was selected, because they are non toxic, environmental friendly and less expensive. Groundnut oil and neem oil are biodegradable oils used in this work. The blend of both oils in various proportion of 100% neem oil,100% groundnut oil, 40% neem oil and 60% groundnut oil, 40% groundnut oil and 60 % neem oil. In this present work the Ni-Cr-Mo steels (EN 31) in a blend of groundnut oil and neem oil. Steel is defined as an alloy of iron and carbon with the carbon content up to about 2% wt., with a vast application almost in every part of our life. EN 31 is a popular grade of alloy steel which is widely used in automobile industry for production of axle, roller bearings, shear blades, spindle , forming , molding dies, ball bearings, spinning tools, beading rolls, punches and dies, shafts, studs, bolts, used in high stress and with a large cross section. It includes aircraft and general engineering applications for propeller or gear components. The typical composition of EN 31 steel is as shown Table 1. The properties of the carbon steels vary with their composition and microstructure, which are dependent on the alloying elements present in the steel and to the kind of heat treatment process it is subjected to. Steel is heated to the austenitization temperature and then cooled at a faster rate to avoid ferrite or pearlite transformation and allow the formation of bainite and martensite to obtain maximum hardness and strength. The factors that the critical cooling rate depends on specific heat capacity and thermal conductivity of the steel as well as the quenchants in addition to quench-bath temperature and agitation. The effect of hardness on the steel is determined by conducting Rockwell hardness test and tensile test by using universal testing machine. Microstructure study is carried out using optical microscope.

Elements	Specification Requirement (wt. %)
Carbon	0.90-1.20
Silicon	0.10-0.35
Maganese	0.30-0.75
Sulphur	<0.04
Phosphorus	<0.04
Chromium	1.00-1.60
Iron	Remaining

Table 1: Chemical composition of EN 31 steel

II. EXPERIMENTATION

1. ROCKWELL HARDNESS TEST

The Rockwell hardness test method consists of indenting the test material with a diamond cone or hardened steel ball indenter. In this work, cone shaped indenter is used. The indenter is forced into the test material under a preliminary minor load F_0 (Fig.1) usually 10 kgf. When equilibrium has been reached, an indicating device, which follows the movements of the indenter and so responds to changes in depth of penetration of the indenter, is set to a datum position. While the preliminary minor load is still applied an additional major load is applied with resulting increase in penetration (Fig.1). When equilibrium has again been reach, the additional major load is removed but the preliminary minor load is still maintained. Removal of the additional major load allows a partial recovery, so reducing the depth of penetration (Fig.1). The permanent increase in depth of penetration, resulting from the application and removal of the additional major load is used to calculate the Rockwell hardness number.

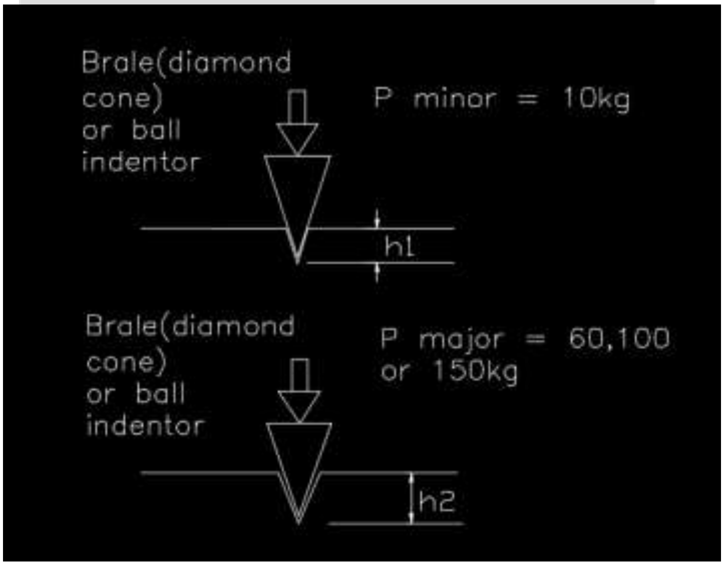


Fig 1: Principle of Rockwell hardness test

2. MICROSTRUCTURAL STUDY USING OPTICAL MICROSCOPE

The microstructural study is carried out by optical microscope in 500 resolutions. Steel are polished by using emery paper of grit 320, 400, 600, 800, 1200, 1500, 2000.followed by polishing using diamond paste on rotating linen disc and finished with polishing on velvet cloth using white kerosene as the suspension medium. These samples were etched with 2% nital (nitric acid and ethanol) and dried in air. The etched samples were used to get the microscopic views studied using optical microscope at a magnification of 500×.

Fig 2: Optical microscopy

III. METHODOLOGY

1. Sample preparation

The sample EN 31 steel of length 48mm and 16 mm diameter is cut into 4 pieces of length 12 mm from one end of the sample using a power hacksaw, while cutting with the power hacksaw suffient amount of coolant is provided to the power hacksaw. So the cutting temperature does not affect the material structure.

2. Annealing

The austenitization is take place about 1000°C.all the specimens placed in the electrical furnace at some distance apart from each other and it requires about 2.5 Hours to Reach the Desired Temperature. The toggle is the tool used for placed out red hot specimen.

3. Soaking

The temperature of the furnace must be held constant during the soaking period, since during this period that rearrangement of the internal structure of the steel takes place. Length of the soaking period depends upon the type of steel and the size of the part. Naturally heavier parts require longer soaking to ensure equal heating throughout. As a general rule a soaking period of 30 minutes to one hour is sufficient for the average heat treating operation. Here after reaching the desired temperature, the samples were soaked about 30 minutes.

4. Quenching

The Quenching is the rapid cooling of the material or specimen. In this work the biodegradable oils, groundnut oil and neem oil were taken in a known proportion. This method was followed for four combinations with ratio 60:40, 40:60, also pure oils. Here after the soaking period, the samples are taken out one by one from the furnace with help of tong and dipped in five different sand pots consisting of different proportions of quenchant. Each pot is designated depend on the proportion of quenchant contained in it. Once each sample is sudden quenched in each pot, it is made to lie inside the pot for about 3 to 4 minutes. The temperature of quenchant after quenching rises to about 50°c.

Specimen code	Groundnut oil (wt. %)	Neem oil (wt. %)
A	0	100
B	100	0
C	60	40
D	40	60

Table 2: Quenchants used in heat treatment

5. Removing Scales

The scales present in the samples were removed in order to get true surface so that polishing work can be performed. The scales are formed by chemical composition of iron or other elements contained in steel with oxygen from air or from the atmosphere of electric furnace.

6. Rough grinding

The first step in grinding is to make the surface absolutely flat, in order to make it suitable for viewing at higher magnification through microscope. This can be done using file, rotating grinding wheel or a motor driven energy belt. If a file is used, it is preferable to keep the file stationery and to rub the specimen fixed and filing in the conventional way. Again, care must be taken to ensure not to get the specimen heated.

7. Intermediate and fine grinding

This is carried out using a sequence of emery papers of progressively finer abrasive grit sizes. Grit sizes of 180, 220, 280, 320, 380, 400, 600, 1200, 1500 and 2000 are usually used for this purpose. The emery paper held on a hard flat surface like a glass plate and the specimen rubbed against it with reasonable pressure. Rubbing should be done only in one direction, preferably away from the operator. After grinding on every paper, the specimen as well as the hands of the operator are thoroughly washed and dried. the specimen is first ground on 180 emery paper so that new scratches due to present grinding are produced at right angles to those were produced during the previous filing or grinding. Grinding on 180 papers is continued till all the previous scratches are disappeared. Specimen is then washed and dried. The same procedure is repeated with 220, 280, 320, 400, 600, 1200, 1500 and 2000 grit papers one after the other. While transferring the specimen to next finer grade of paper, the specimen should be turned through 90°c and ground. This procedure is repeated till the finest emery paper has been used and the specimen would be apparently scratch free.

8. Advanced polishing

Here the specimen once polished manually is bought on to the double disc. Double disc polishing machine consist of two disc rotated by a motor. Emery paper with 2000 grit is attached to the polishing disc to aid the polishing. The specimen is placed against the rotating disc by firmly holding it with the hand. Here the entire polishing takes places without using any lubricant. The material is polished until all the scratches are removed and a mirror finished surface is obtained.

9. Etching

Before being etched the specimen can be viewed for inclusions, slags, flakes etc. The mechanism of polishing is such that it leaves a flowed or amorphous layer of metal on the surface. Unless the layer is removed, the true structure will not be revealed. Etching is done on a polished specimen to remove the flowed layer produced by polishing. An etchant is used to dissolve this layer and remove it so that the microstructural features are visible under microscope.

The principle of etching is based on the preferential staining or preferential dissolution of one or more phases present in the microstructure. This happens primarily due to the differences in chemical composition and secondly due to differences in orientation of grains. The atoms oriented along the region between two adjacent grains will dissolve easily than other atoms. This would expose the grain boundary and it can be clearly seen under a microscope. Before etching the specimen must be absolutely clean, otherwise it will stain during etching. In most cases after an initial washing in running water, the surface is swabbed with cotton wool dipped in a soap solution and again washing in running water will be enough. After the final washing the surface should be dried immediately by a hot air dryer. The polished specimen is etched by using the etching medium (4% nitric acid and 96% ethanol). The material will be etched few to several seconds. After the etching process the material is quickly transferred into running water to wash away the etching reagent. Then the material is dried using a hot air blower. After drying properly, the specimen is taken out for the microstructure analysis.

10. Microstructure Analysis

Microstructure is the very small scale structure of material, defined as the structure of prepared surface of material is revealed by a microscope. Here the processed specimen is placed in an inverted microscope. The microscope used was of as metrology which uses metallurgical image analysis software (MIAS). The software is totally automated and provides analysis of microstructure. the microstructure of material can strongly influence physical properties such as strength, toughness, ductility, hardness, etc. the specimen is placed over a table on microscope which can be moved with the knob provided the side of microscope to get the desired microstructure. Focusing knob is provided to focus the image of microscope. By pressing the snap button take and store the picture of microstructure at each position. In order to discriminate the phases of steel select the required microstructure and click on phase button, enter the name and select a colour that distinguishes each phase. Similarly mark the name and identification colour of all phases. Once every phase is marked, click on the run button to get the percentage details of each phase and click on the report button to get detailed percentage composition represented with the help of pie chart along the original microstructure image and phase distinguished image.

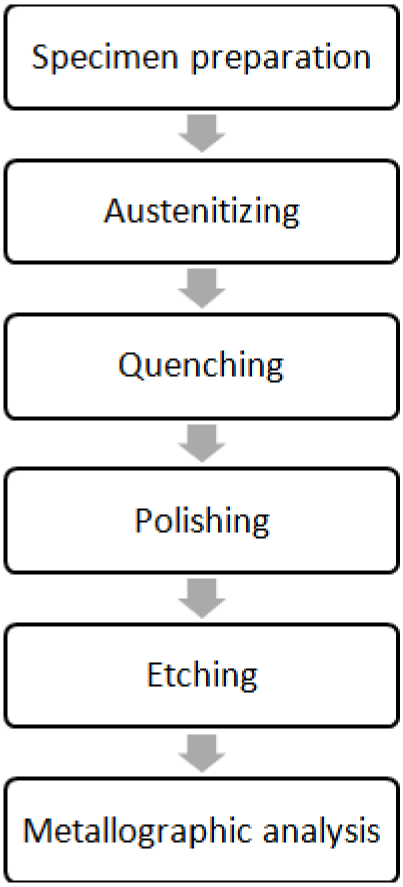


Fig 3: Flow chart of process

IV. RESULTS AND DISCUSSION

1. Hardness test results

The hardness test results of the samples are shown in the table. Rockwell hardness number of EN 31 for various heat treatment is presented in the table indicate the increase of hardness of steel samples due to heat treatment and also shows the percent composition of different elements in microstructure. Hence the amount of martensite determines the hardness of the material.

Nomenclature	Hardness number (HRC)	Retained austenite (RA) %	Martensite (M) %
A	57	83.44	16.56
B	60.9	65.32	34.68
C	56.8	87.58	12.42
D	52.6	90.63	9.37

Table 2: Result comparison

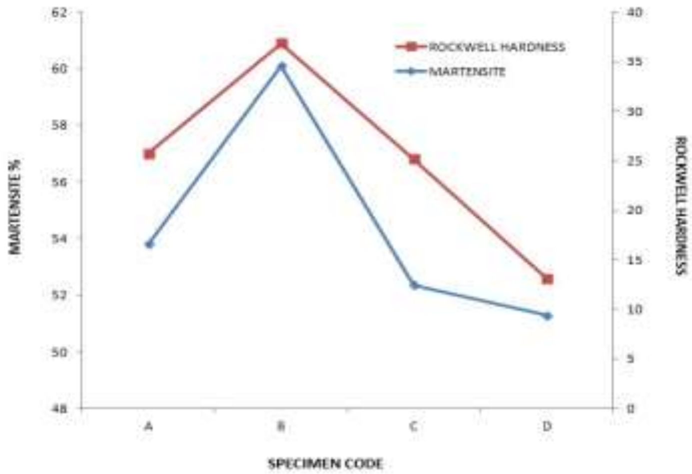


Fig 4: Relationship between percentage of martensite and Rockwell hardness

2 .Microstructural analysis

In the result from fig: 5 to fig: 12 can see the microscopic views of EN 31 Steel at various quenching condition. The matrix mostly consists of martensite which is the reason for hardness.[5] The structure consist of untempered martensite (dark, needle like structure) and large amount of retained austenite (light constituent).The carbide particles are less which indicates poor precipitation levels.

EN 31 A

Fig 5: The microscopic view of martensite and retained austenite in EN 31 steel quenched in 100% of neem oil.
In this structure the uniform distribution of martensite with medium concentration. The light yellow colour retained austenite is situated in the intermediate of them. As the above theory [5] this has medium level hardness.

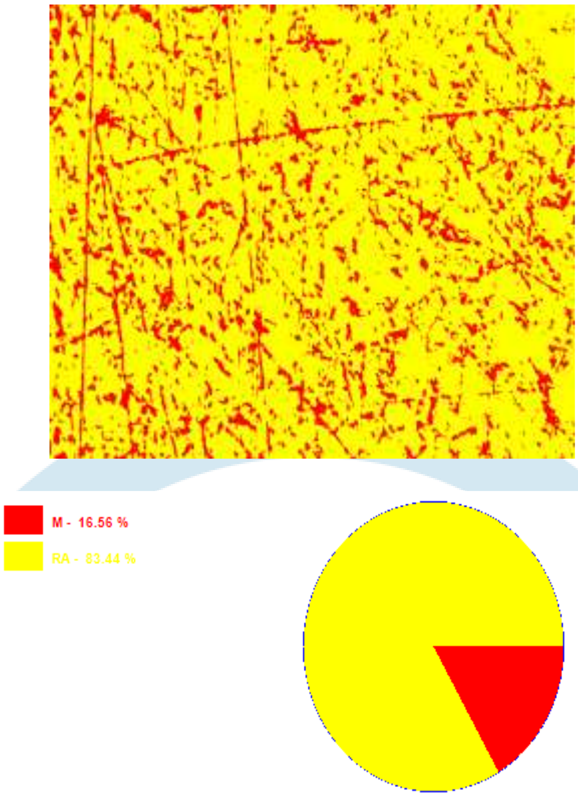


Fig 6: Phase transformation of retained austenite and martensite. It contains 16.56% of martensite & 83.44% of retained austenite.

EN 31 B

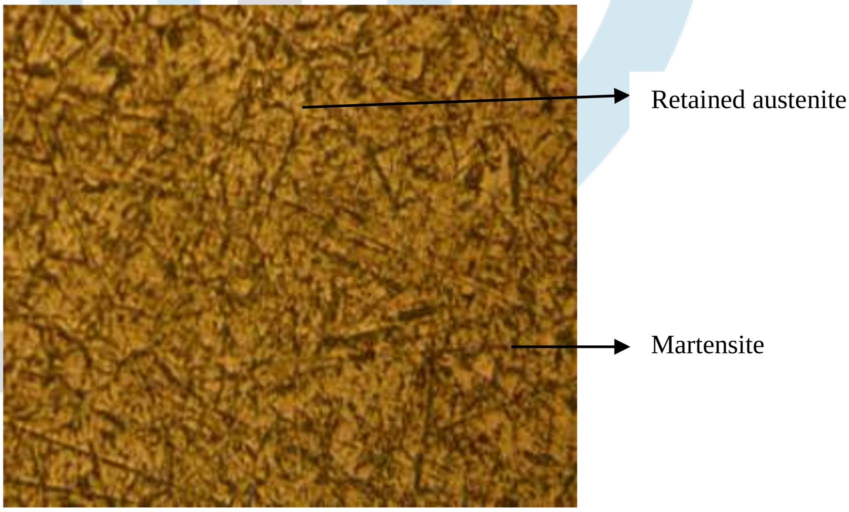
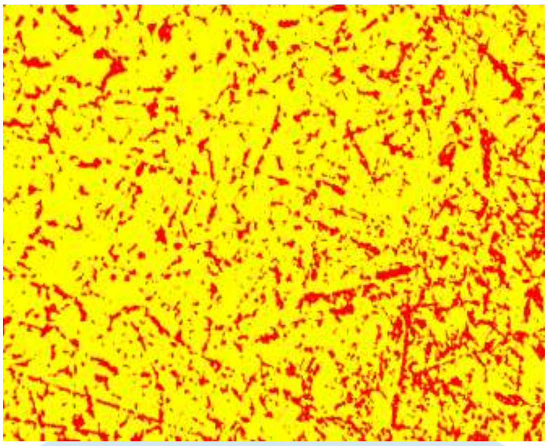


Fig 7: The microscopic view of martensite and retained austenite in EN 31 steel quenched in 100% of groundnut oil. Here the distribution of martensite is higher and non uniformity of the martensite at certain intervals.

In this the martensite distribution is higher with uniform distribution. The retained austenite is very less amount. The need like martensite is concentrated with all regions. So, it has comparatively high hardness.



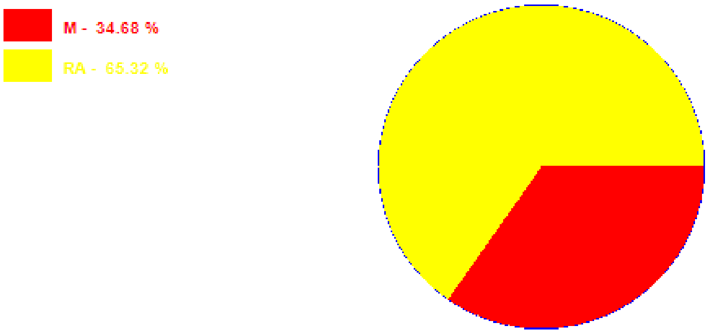


Fig 8: Phase transformation of retained austenite and martensite. It contains 34.68% of martensite & 65.32% of retained austenite.

EN 31 C

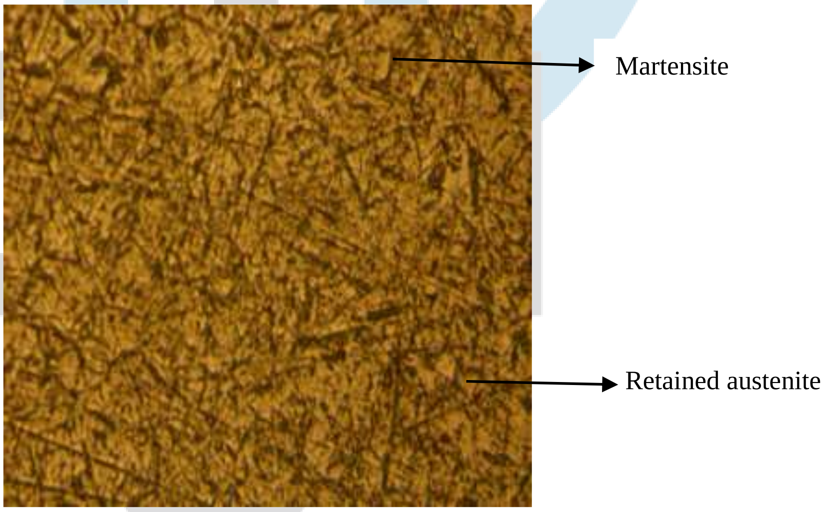
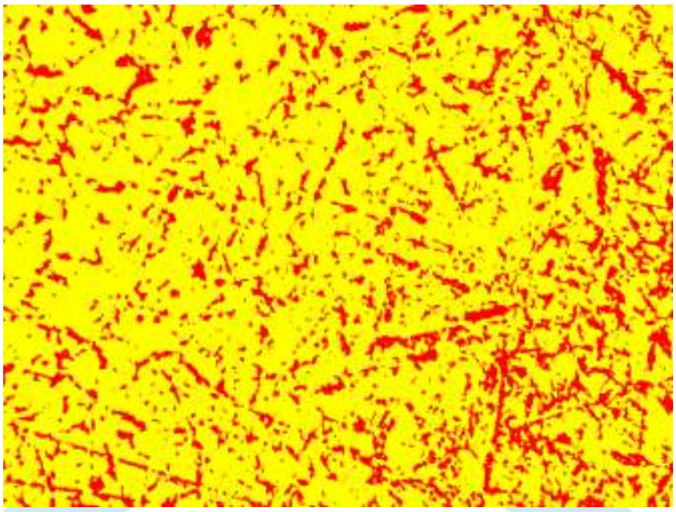


Fig 9: The microscopic view of martensite and retained austenite in EN 31 steel quenched in 60% of groundnut oil & 40% Neem oil.

In this the martensite concentration is medium level. The retained austenite is uniformly distributed over the concentration of martensite. So it has medium range of hardness.



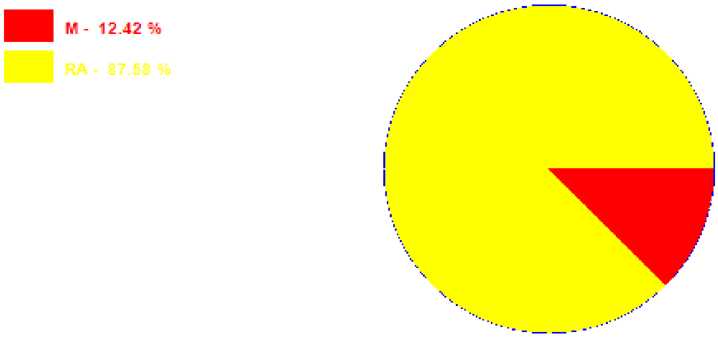


Fig 10: Phase transformation of retained austenite and martensite. It contains 12.42 % of martensite & 87.58 % of retained austenite.

EN 31 D

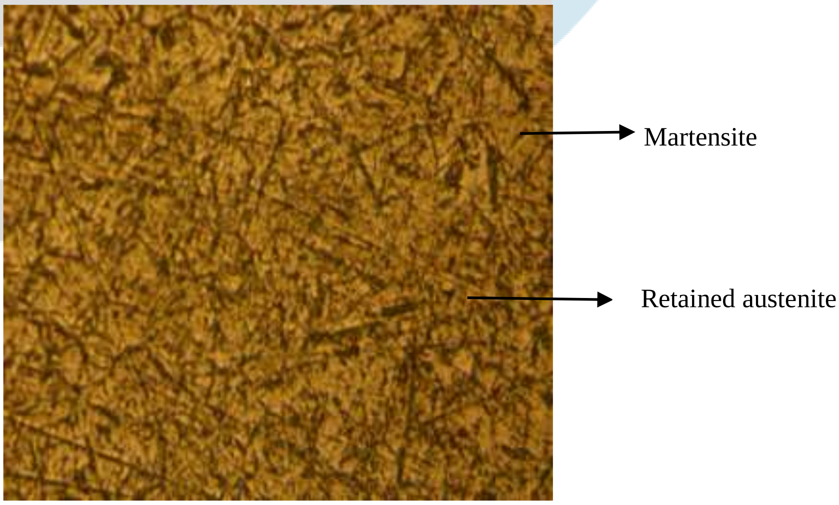


Fig 11: The microscopic view of martensite and retained austenite in EN 31 steel quenched in 40% of groundnut oil & 60% Neem oil.

From this microscopic observation it is conclude that the martensite or needle like structure is very less. The concentration of light yellow region or retained austenite is high. So the less amount of hardness for this specimen.

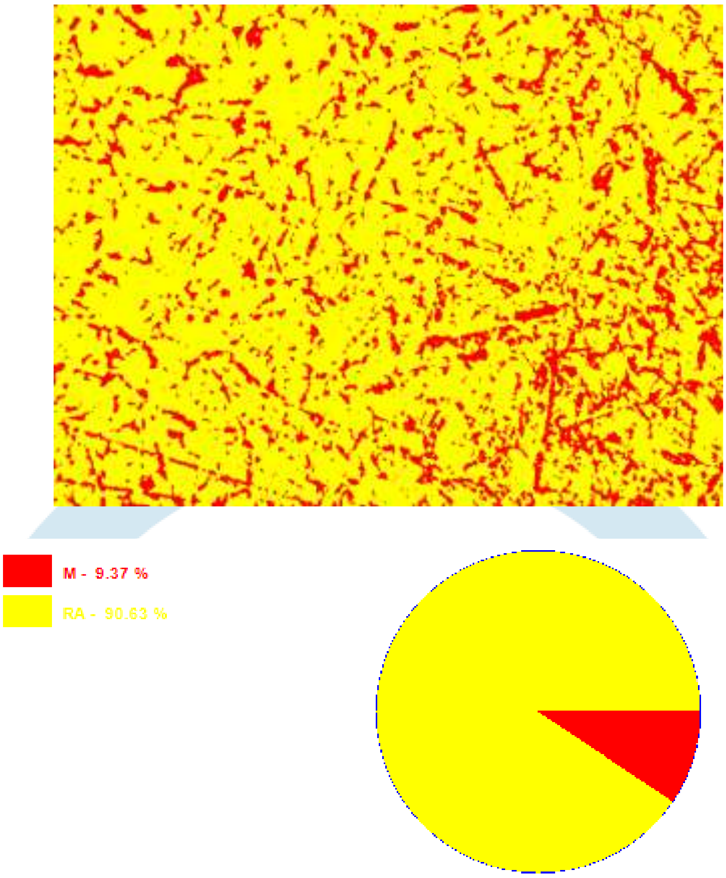


Fig 12: Phase transformation of retained austenite and martensite. It contains 9.37% of martensite & 90.63 % of retained austenite.

V. CONCLUSION

From this study, determines that the sample B has highest hardness (61 HRC).i.e., sample B has quenched with 100% groundnut oil. It is also found that maximum percentage of martensite of about 34.68% (Metallographic observations).Hence the increase in percentage of martensite increases the hardness of the sample. The lowest hardness was obtained with the sample D (52.6 HRC) and it has less percentage of martensite about 9.37%.

The reason for increase in hardness when marten site phase increase is due to the body centered tetrahedral cubic structure of martensite as it blocks the movement of carbon atom.

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