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International Journal Of Modern Engineering Research (IJMER)

Effect of Subzero Treatment on Microstructure and Material Properties of EN24 Steel

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Abstract: *Cryogenic treatment of steels has been widely used for enhancing mechanical properties like hardness, toughness and stable metallurgical structure. Application such as gears, kicker rods, bolts are made of medium carbon alloy steels like EN-24 steel. In these applications, percentage of retained austenite has considerable effects on the life of the material. A comparative study on conventionally heat-treated (CHT) and shallow cryogenic treated (SCT) EN-24 steel was done to evaluate the effect of shallow cryogenic treatment (SCT) on hardness, toughness and the amount of retained austenite present in the structure of EN24 steel. The microscopic structure of cryogenic treated EN24 steel revealed the formation of carbides, both primary and secondary carbides. An estimated amount of 15% retained austenite after CHT tempered condition was less than 2% after SCT tempered condition. Tensile test fractography of subzero treated (SCT) specimen revealed ductile fracture. The maximum hardness observed in case of SCT tempered samples was 415BHN, 15% increase from CHT tempered samples. The maximum impact strength observed in case of SCT tempered samples was 240kJ/m2, 11% increase from CHT tempered samples. Further SCT tempered samples, tempered at 650°C resulted in ductility increase by 55% as compared to CHT tempered samples without sacrificing hardness.*

Index Terms: *EN24 Steel, hardness, shallow cryogenic treatment, retained austenite, tensile test fractography.*

I. Introduction

Cryogenic treatment is an added heat treatment technique over conventionally heat-treated materials. It is an effective method to improve the engineering performance of steels [1] - [2]-[3]-[4]. Cryogenic treatments are carried out in two aspect, the shallow cryogenic treatment (SCT) conducted at temperature between -75°C (198°K) and -90°C (183°K), the deep cryogenic treatment (DCT) conducted at temperatures below -195°C [8]. The commonly used cryogenics are liquid nitrogen (-195.8°C), liquid helium (-269°C), liquid hydrogen (-252.9°C), methane (-161.5°C) and solid carbon dioxide (-78°C). Cryogenic treatment increases the mechanical properties of cutting tool materials, die materials and bearing materials such as hardness, toughness, tensile strength, wear resistance and corrosion resistance [3]-[7]. The lifetime of material under mechanical wear, get affected by the retained austenite present in it [1]-[7]. In working conditions there may be chances for micro structural transformations of retained austenite into martensitic structure which causes parts to break due to sudden brittleness. This investigation revealed the fact that the retained austenite present after conventional heat treatment is reduced substantially by lowering the quenching temperature up to subzero temperature without sacrificing hardness and toughness of the heat treated material.

II. Literature Review

Cryogenic treatment reduces percentage of retained austenite into tempered martensitic structure thereby increasing material resistance to wear [2]-[7]. Cryogenic treatment not only helps in microstructural transformations (retained austenite to martensite) but also helps in improving the metallurgical structure [5]-[7]. For high carbon steels the retained austenite transformation to martensite structure, require deep cryogenic treatment [1]-[2]-[7]. In this investigation, the shallow cryogenic treatment (SCT) on EN24 medium carbon steel was conducted and the effect of SCT on material hardness and toughness was investigated. EN24

(817M40T–Molybdenum steel) is a high tensile alloy steel known for its wear resistance and used for high strength applications. EN24 steel is surface-hardened to enhance wear resistance by induction hardening or nitriding processing. Application includes propeller or gear shafts; connecting rods; aircraft landing gear components etc.

The EN24 specimens are kept in a sealed insulated container containing solid carbon dioxide known as dry ice for a period of 24 hours. The temperature was maintained at -76°C (197°K). The directions followed during the investigation are ***a)*** retained austenite during conventional heat treatment (CHT) process transforms to martensitic structure after shallow cryogenic treatment (SCT) and ***b)*** increase in material hardness as an effect of martensitic transformation imparted into the material due to cryogenic treatment.

III. PROCESS METHODOLOGY

A. Specimen preparation

Specimens were prepared as per ASTM standards from annealed EN24 alloy steel bar with nominal material composition of C-0.35%, Mn-0.45%, Si-0.11%, Cr-0.91%, Ni-1.31%, Mo-0.21%. For comparative study, two sets (CHT and SCT) of specimens with four samples under each treatment were prepared for standard laboratory tests.

B. Conventional heat treatment

The specimens are first austenized at a temperature of 850°C and quenched in a gas-carburizing furnace with neutral proportions of one-inch one hour soak condition. The specimens treated are free of quench cracks imply a little or nil residual stress. After confirming, the as quenched hardness, specimens are either double tempered (2T) or triple tempered (3T). The specimens are soaked for 90 minutes duration at temperatures of 450°C and 650°C followed by air-cooling at room temperature as shown in Fig. 1.

C. Shallow Cryogenic treatment

Shallow Cryogenic treatment is a supplement treatment to conventional heat treatment and it involves three stages. *Stage-1*: Conventional heat treatment (CHT) and quenched, *Stage-2*: Subzero treatment (SCT at -78°C) and *Stage-3*: Subzero treatment (SCT) followed by double or triple tempering process at temperatures 450°C and 650°C and cooled down at room temperature. Subzero treatment is carried in an insulated container with solid CO_2 at a temperature of -78°C and then bringing it to room temperature followed by tempering as shown in Fig. 1.

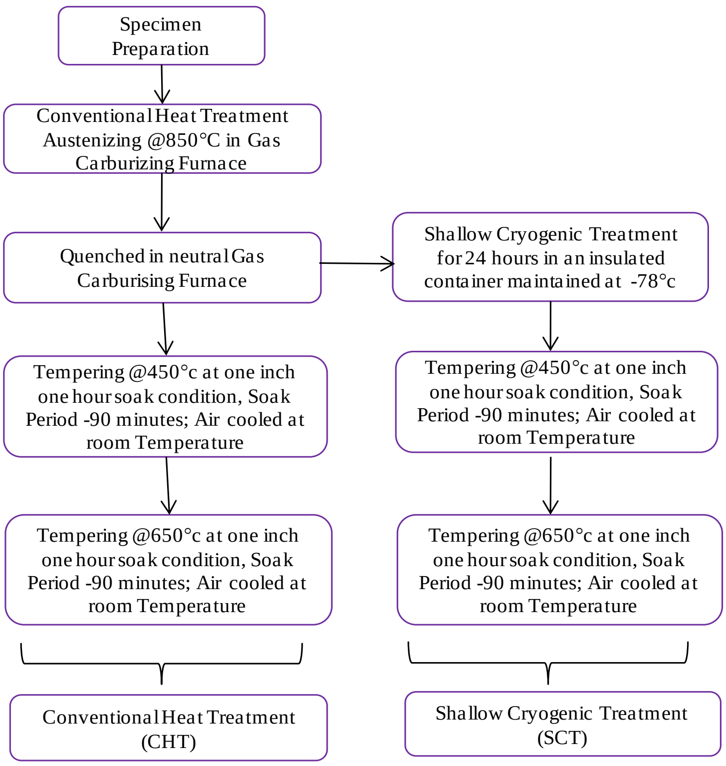

Fig. 1. Process flow chart

IV. RESULTS AND ANALYSIS

A. Hardness Test

Hardness testing is widely used for material evaluation due to its simplicity and low cost relative to direct measurement of many properties [9]. Hardness testing does not give a direct measurement of any performance properties, hardness correlates with material strength, wear resistance and other mechanical properties [9]. The annealed EN24 Steel sample has an equivalent hardness of 217BHN and the average hardness of the CHT quenched samples was 363BHN. The maximum hardness observed in case of SCT tempered samples were 415BHN. There is a considerable increase in the hardness value of the subzero treated samples as compared to conventionally heat treated and annealed EN24 Steel. The average value of hardness is tabulated for reference in Table 1.

Table 1. Hardness test Results

Sl. No	Specimen	Hardness (BHN)	Micro Vickers hardness (HRC)
1	Annealed	217	239
2	CHT Tempered	363	388
3	SCT Tempered	415	465

B. Impact Test

The impact test was performed as per ASTM A370 (standard test method and definitions for mechanical testing of steel products) to study the effect of cryogenic treatment on the toughness of EN-24 steel. The most common test is the Charpy V-notch impact test, in which the standard specimen is struck opposite the notch by a heavy falling pendulum. The toughness is expressed in terms of the kinetic energy absorbed by the fracture. The result of tests revealed that conventional heat treated and quenched samples absorbed 17.5 joules of energy whereas subzero tempered samples absorbed 24 joules. The average value of toughness is tabulated for reference in Table 2.

Table 2. Toughness test Results

Sl. No	Description of samples	Impact value (Joules)	Impact Strength (KJ/m2)
1	CHT and quenched	17.5	175
2	CHT and tempered	21.50	215
3	SCT and tempered	24.00	245

C. Tensile test

Tensile test was performed on six different samples of EN24 steel. The test was done on annealed, CHT quenched, CHT tempered at 450°C, SCT quenched, SCT tempered at 450°C and SCT tempered at 650°C. The SCT samples are tempered under two different temperatures, 450°C and 650°C to understand its elastic and elasto-plastic elongation. The tensile test was carried out according to ASTM A370-05. The test specimen dimensions are presented in Fig. 2.

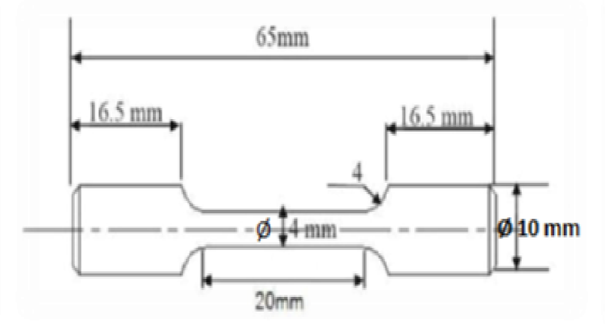

Fig. 2 Tensile Test Specimen

From Table 3 it has been observed that the tensile strength of subzero treated samples is more than that of the conventionally quenched and annealed samples. Tempering after subzero treatment at 450°C has lesser elongation, greater ultimate strength, greater yield strength than tempering at 650°C. The experimental results reveal that it is optimum to use tempering temperature scale at 450°C.

Table 3. Tensile Test Results

Sl. No	Specimen	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)	Reduction (%)
1	Annealed specimen	849.95	759.04	15.60	59.04
2	CHT Quenched	2177.99	2095.58	10.00	39.88
3	CHT Tempered @450°C	1580.16	1517.81	7.20	21.36
4	Subzero Quenched	2248.55	2158.45	10.40	37.83
5	Subzero Tempered @450°C	1587.31	1550.29	4.80	23.44
6	Subzero Tempered @650°C	1184.33	1098.79	11.20	29.56

D. Graphical Analysis

Percent elongation is the ability of the material to flow plastically before facture. Even though there is no proportional relationship between ductility and load carrying capacity of the material under elasto-plastic region, yet the material of low percent elongation can withstand greater loads. From Table 3, SCT tempered EN24 steel has least percent elongation in elasto- plastic region; it means that it can withstand greater loads compared to CHT tempered EN24 steel. SCT tempered EN24 steel, tempered at 450°C has lesser elongation, greater ultimate strength, greater yield strength and high ductility when compared to CHT tempered EN24 steel. The stress strain curve for different material treatment condition is presented in Fig. 3.

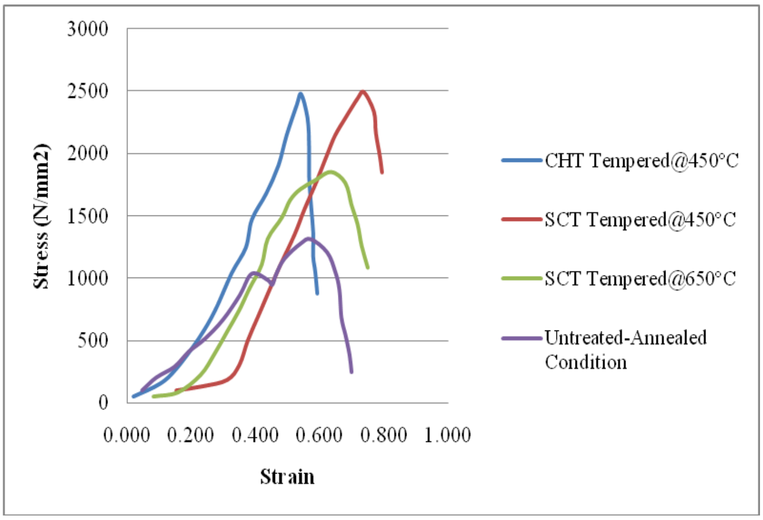

Fig. 3. Stress strain curves –Comparative Trend

E. Tensile Test Fractography analysis using SEM

Broken tensile test samples were tested for fractography analysis. Test Sample made with cut section diameter of 10 mm and length 20 mm molded using Bakelite. The specimen are ground progressively with finer SiC water proof papers from 120 to 1000 grit to produce polished flat surface. The surface was etched using 2% Nital and cleansed using alcohol. In CHT quenched specimen presences of voids are visualized. More over the grain refinement is very small it means that the particles are coarse and load withstanding capacity will be less.

Fractography test of Annealed EN24 material as shown in Fig. 5, exhibits ductile fracture mode with cracks and micro voids. The annealed material under heavy loads develops voids in the structure that prolongs and collapse.

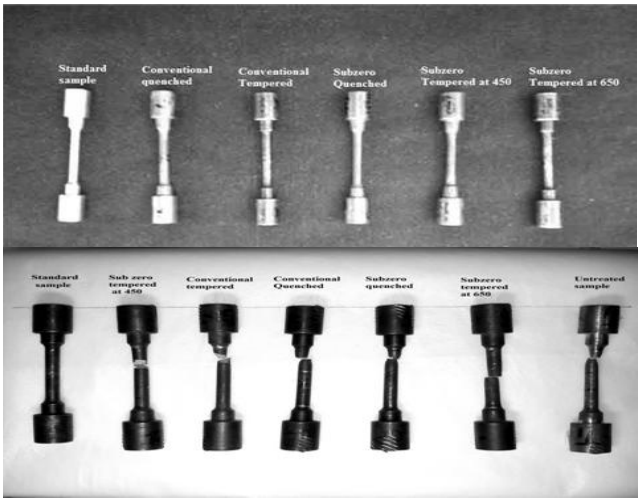

Fig. 4. Specimens before and after tensile testing

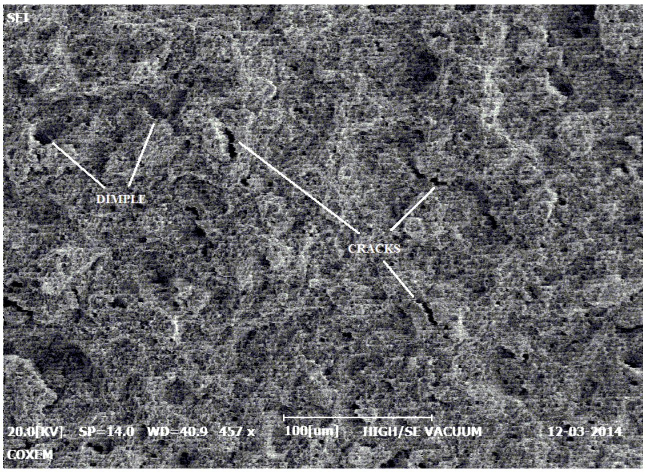

Fig. 5. SEM Fractography of Annealed EN24 material.

Fractography test for CHT tempered material as shown in Fig. 6, exhibits ductile fracture mode. It means that due to elongation the material reduces its diameter and after yielding failure takes place. Dimples and hot tear phenomenon are clearly seen in the surface, observed uneven breakage due to unevenly scattered carbides.

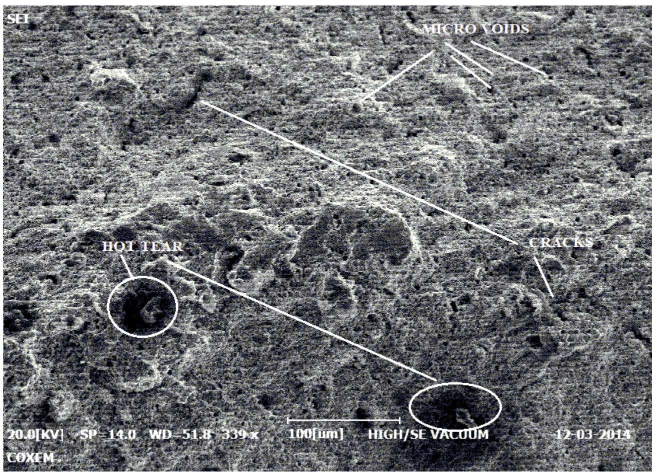

Fig. 6 SEM Fractography of CHT Tempered EN24 material

Fractography test of SCT Tempered EN24 material as shown in Fig. 7, exhibits micro cracks, small in size when compared to CHT quenched samples. The presence of dimples are negligible hence even fracture is visible in the picture. Plastic flow prior to fracture was observed as shown in Fig.8. The secondary carbides observed in the microstructure of SCT Tempered at 450°C as shown in Fig.11, increases the wear resistant property of the material.

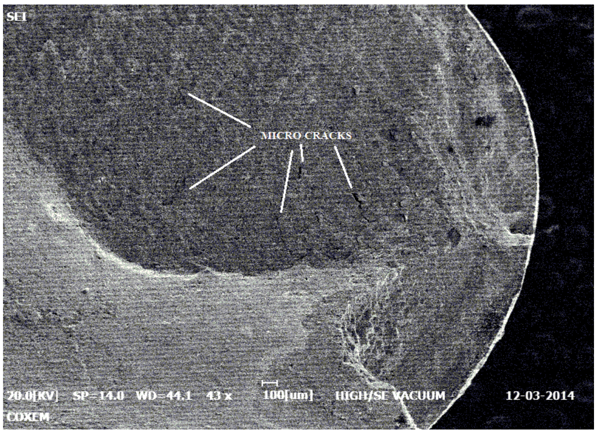

Fig 7. SEM Fractography of SCT Tempered EN24 material

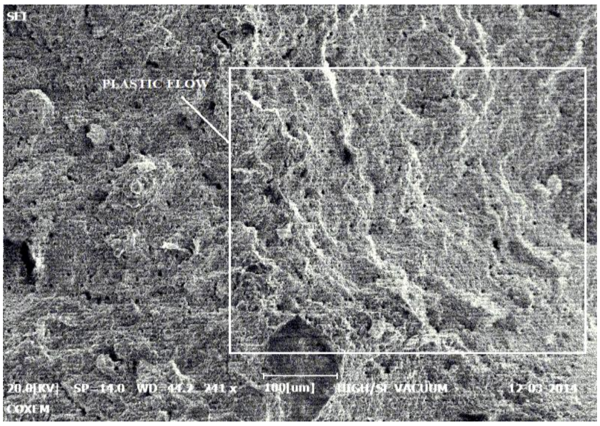

Fig 8. SEM Fractography of SCT Tempered EN24 material Showing Plastic Flow behavior before fracture

F. Microscopic study

The test samples for micro examination grounded using different grades of emery papers (200, 400, 800, and 1200) then polished using diamond paste. It is cleansed using etchant Nital 2% and allowed to dry. The microstructure was examined using Optical Microscope. Fig.9 shows the microstructure of annealed EN24 Steel revealing fine pearlite matrix structure.

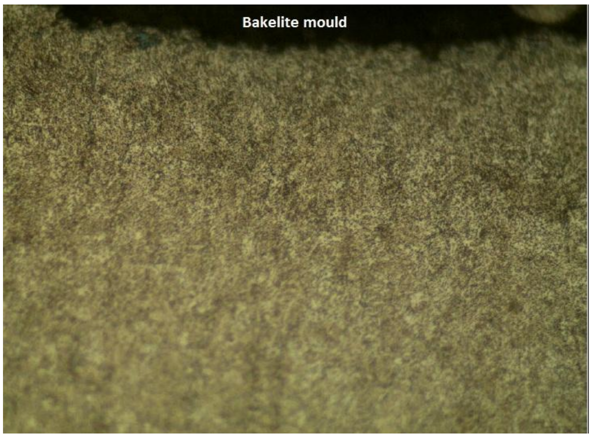

Fig 9. Microstructure of annealed En-24 steel, Mag: 500X

The microstructure of steel at conventionally heat treated condition as shown in Fig.10 reveals an estimated amount of the retained austenite was 15%. The microstructure in Fig.10 where more of white spots (retained austenite) present is responsible for the decrease in the hardness value of the specimen.

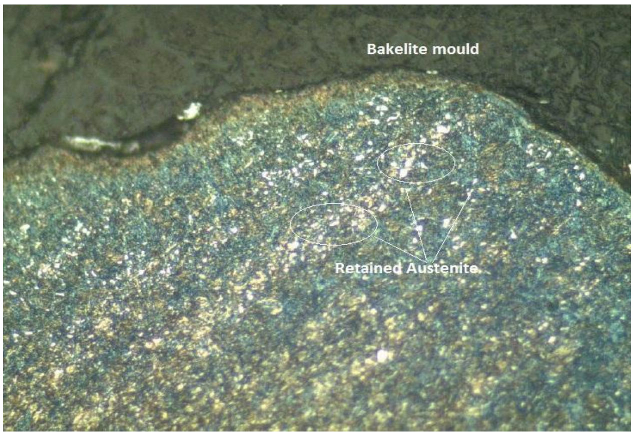

Fig 10. Microstructure of CHT and quenched En-24 steel

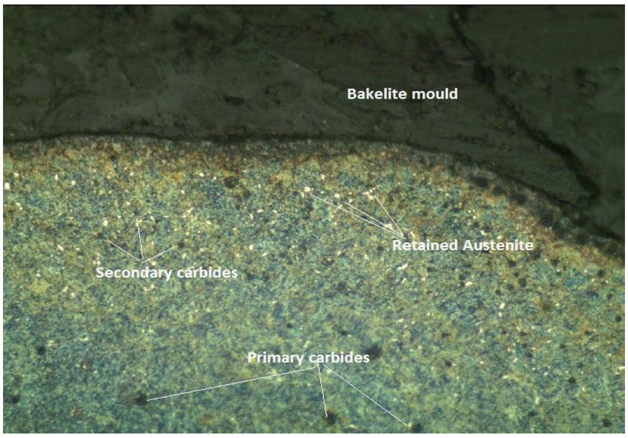

Fig 11. Microstructure of SCT Tempered at 450°C

For subzero treated sample the retained austenite present was observed to be less than 2%. The retained austenite found as shown in Fig.11 are scattered and the presence of secondary carbides on the surface are evident. Secondary carbides formed mainly due to transformation of retained austenite particles to temper martensitic.

V. CONCLUSIONS

The comparative study made on the effect of cryogenic treatment on EN-24 steel revealed the following:

- A substantial improvement in hardness from 363BHN for conventionally heat treated (CHT) material to 415BHN for subzero treated (SCT) material.
- A substantial improvement in toughness of material from 17.5 Joules in case of annealed sample to 24 Joules in case of SCT sample.
- A substantial improvement in ultimate tensile strength in case of SCT EN24 steel with decrease in percentage elongation. The tensile strength and yield strength of SCT tempered samples depicted marginal improvement than CHT tempered samples, tempered at 450°C, but percentage elongation for SCT tempered samples reduced by 50% compared to CHT tempered samples.
- The Tensile test fractography study indicates the mode of failure was ductile in each case.
- Presence of retained austenite from 15% in case of conventionally heat treated condition has been reduced to less than 2% in case of subzero treatment.

The mechanical testing of shallow cryogenically treated EN24 carbon steel material exhibited increase in mechanical properties like hardness, toughness and substantial reduction in percentage of retained austenite.

VI. ACKNOWLEDGEMENT

The authors acknowledge the services M/s.Micro- Hard Processing-Heat Treatment, Ambattur, M/s.Microlab, Ambattur Industrial Estate, and St. Joseph's College of Engineering, OMR, Chennai, Tamilnadu, to encourage us to carry out a part of this research.

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