Epoxidized-Transesterified Cotton Seed Oil for Temperature-Dependent Austempering Process

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*Abstract—*Temperature dependent austempering of high carbon steel using epoxidized-transesterified cotton seed oil (ETO) was examined. Five sets of samples were heated to 850 °C and held for one hour and then quenched in an oil bath of ETO at 250 °C for one hour. The same procedure was performed on the remaining samples, which were austempered at 270 °C, 290 °C, 310 °C, and 330 °C. Next, mechanical property tests were conducted. The austempered samples were then analyzed for microstructure using a scanning electron microscope (SEM). The results indicate that tensile strength and hardness decrease with increasing temperature, while impact strength improved with rising temperature. It was observed that 270 °C is the best austempering temperature, as it produces austempered samples with the best combination of mechanical properties.

*Keywords—*Epoxidized-transesterified cotton seed oil, austempering temperature, high carbon steel, bainitic structures.

I. INTRODUCTION

USTEMPERING is an isothermal heat treatment that can A ^{USTEMPERING} is an isothermal heat treatment that can
serve as a substantial replacement to conventional hardening treatments. In this process, the ferrous alloy component is heated to the austenite region and then cooled rapidly to a predetermine temperature above the martensite start (M_s) , aiming to transform the austenite into bainite instead of martensite [1]. Quenchant is the liquid medium usually employed in austempering and conventional quenching treatment to achieve rapid cooling. There are many quenchants available with different relative importance and area of applications. The standard quenching media for austempering of ferrous alloys is a molten salt bath. However, it can break down at high temperatures, increasing the risk of hazards. Lead baths are also used, but they carry a high risk of lead poisoning and are relatively expensive due to additional cleaning costs, as lead metal tends to adhere to the surface of the components treated. Petroleum and vegetable oils have been utilized as quenchants [2]. Nonetheless, the film or nucleate boiling heat transfer that the petroleum-based oils demonstrate impairs them, leading to lower-temperature cooling rates. This behavior is not present in vegetable oils, where convective cooling controls the heat transfer [3]. Compared to other quenchants, vegetable oils cool more rapidly [4], making them relatively efficient for austempering heat treatment. However, at the predetermine temperature for austempering, a series of breakdown products can develop in vegetable oils due to poor thermal-oxidative stability. This consequently affects the quenching performance of the oil and

has remained a problem for their widespread use in austempering treatment [5]. Altering the molecular structure of the oils presumably subdues these drawbacks. Modifying cotton seed oil through chemical means can be achieved by transesterification and/or epoxidation processes. In the event of epoxidizing transesterified cotton seed oil; the end product would be ETO. Hence, an attempt has been made in the present investigation to explore the potential of ETO in the austempering treatment of high carbon steel.

II. MATERIALS AND METHOD

A. Materials

The study involved the use of high carbon steel (HCS), ETO and 4% picral. The carbon content of HCS is 0.68%.

B. Sample Preparation

Six sets of machined test samples (prepared to standard mechanical test sizes and shapes) were placed in the furnace for normalizing heat treatment operation. Here, the samples were heated in the furnace to 850 °C and soaked for one hour, after which it is cooled in air.

C. Heat Treatment

Five out of the six sets of samples were heated to the austenitizing temperature of 850 ℃ and allowed to soak for one hour in the furnace (holding time). Subsequently, a set of the samples was taken from the furnace and quenched in an ETO bath maintained at a preselected temperature of 250 ℃ for an hour and then taken from the ETO bath and air-cooled. The same technique is again followed to achieve the austempering of the samples at 270 °C, 290 °C, 310 °C, and 330 °C. The last set of the samples are now kept aside as the control.

D.Tensile Test

In conducting this test, a Hounsfield testometer type "W" with maximum load of 20 kN was used, following the ASTM E8 standard. Each sample was gripped at both ends on the tensometer, and a force was applied at a constant rate. Readings of force and extension were measured simultaneously by using a load cell and an extensometer, respectively, until the sample ultimately broke. The corresponding extension was recorded on a graph sheet. The tensile strength was then calculated by taking the ratio of the breaking load and the cross-sectional area of the fractured sample. Each test was carried out three times, and the average was recorded to ensure the reliability of the generated data.

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E. Hardness Test

An indentec universal hardness testing machine with Rockwell tester MN: 8187.5LKV model (B) was used for this test. The C scale was employed, with maximum and minimum loads were 150 kgf and 10 kgf, respectively. A sample was placed on the anvil, and a load was applied on the surface of it to obtain the hardness value; the load is applied for 30 seconds. The hardness values were then read directly from the machine. An average value was obtained for three indentations at different points, which was taken as the hardness of the sample. The same technique employed for the remaining samples.

F. Impact Test

An Izod impact testing machine was employed for this test. Samples with dimensions $75 \times 10 \times 10$ mm were used, and the testing machine had a capacity of 120 ft/pounds. The readings were taken directly from the machine, indicating the energy absorbed by each sample. The sample was placed in the path of the applied load, which was released and allowed to move under the influence of gravity. This process was repeated for all the samples. Three tests were conducted for each test condition, and the average was taken to ensure the accuracy of the data recorded.

G.Microstructural Examination

Samples were prepared through a series of consecutive

steps involving grinding, polishing, and etching. The samples were ground using abrasive papers with grit sizes ranging from 150 to 1200. During polishing, a 1 µm of alumina paste was applied to the disc. Subsequently, the samples were etched with 4% picral solution by swabbing with cotton soaked in the etchant. After etching, the samples were dried. Finally, the microstructures of the samples were captured using SEM.

III. RESULT AND DISCUSSION

A. Tensile Strength

Fig. 1 shows that tensile strength of HCS decreases successively with rising austempering temperature. The maximum strength is attained by the sample austempered at 250 °C. Reference [6] explains that a mixed bainiticmartensitic microstructure is obtained at lower austemperings temperature, while martensite significantly enhances the steel's strength due to its superior strength compared to bainite. Additionally, the simultaneous formation of martensite and bainite from the earlier austenite grains introduces plastic restrictions in the bainite, resulting in increased strength in the steel. Similarly, Sandeep [7] observed that the tensile strength of austempered ductile iron rises with lower temperatures, with peak strength achieved at $250 \degree C$.

Fig. 1 Tensile strength of normalized and austempered samples

B. Hardness Value

In Fig. 2, the hardness value shows a slight increase from 250 °C to 270 °C, attributed to the precipitation of carbide phases in the bainite. However, as the austempering temperature rises, hardness values of HCS gradually decrease. It is noteworthy that upper bainite forms at higher temperatures and is free from carbides [8]. Moreover, during isothermal transformation, cementite precipitates from the high-carbon austenite films between the ferrite plates, resulting in a microstructure with lower hardness values. Furthermore, the ferrite itself is devoid of carbides. Similarly,

Dalwatkar et al. [9] reported higher hardness values for highsilicon steels austempered at lower temperatures.

C. Impact Strength

From Fig. 3, the energy absorbing capacity of the austempered HCS significantly increases from $250 \degree C$ to $270 \degree C$ due to the enhanced formation of bainte in the C due to the enhanced formation of bainite in the microstructure. However, it drops considerably at 290 °C, after which it increases progressively. This decrease is due to the formation of ε-carbide precipitate just after bainite formation. The maximum toughness was observed at 330 °C. Generally, the austempered samples exhibit superior toughness compared

to their normalized counterparts. This finding aligns with the work of Sandeep [7], which reported an increase in the

toughness of ductile cast iron with higher austempering temperatures.

Fig. 3 Impact strength of normalized and austempered samples

Fig. 4 Plate I: HCS sample SEM image (a) normalized; austempered at (b) 250 °C, (c) 270 °C, (d) 290 °C, (e) 310 °C, (f) 330 °C

D.Microstructural Examination

The SEM analyses in Fig. 4 (a) reveal the structure of normalized HCS. The microstructure shows spots predominantly consisting of ferrite within thin platelets of pearlite. The lack regions indicate pearlite, while the white regions represent ferrite. Figs. 4 (b)-(f) illustrate the SEM images of various austempered HCS samples, revealing a bainitic structure composed of separate narrow films of retained austenite (black) in supersaturated laths of ferrite (grey) with minimal martensite present. The sample austempered at 270 °C exhibits the largest proportion of martensite, which accounts for its highest hardness. As the austempering temperature increases, the fraction of retained austenite in the microstructure rises, and both ferrite and austenite become progressively coarser. This coarsening explains the higher impact strength observed in the samples austempered at elevated temperatures. At lower austempering

temperatures, the nucleation rate is higher, resulting in a finer microstructure. Conversely, at higher temperatures, the nucleation rate decreases, leading to a coarser microstructure. Elevated temperatures result in a transformed microstructure of upper bainitic ferrite with a substantial amount of retained austenite. Similarly, Dalwatkar et al. [9] indicated that higher austempering temperatures lead to a bainitic structure with a greater content of retained austenite.

IV.CONCLUSIONS

Based on the result obtained, it could be concluded that ETO has the potential to successfully austempered HCS. The austempering temperature significantly effects the mechanical properties of austempered HCS. Lower austempering temperatures demonstrate a bainitic structure characterized by fine laths of ferrite with narrow films of retained austenite and minimal martensite. In contrast, higher temperatures result in

upper bainitic ferrite with a larger proportion of retained austenite. Consequently, the best combination of mechanical properties for austempered HCS is achieved by austempering at 270 °C. Therefore, ETO can effectively enhance the mechanical properties of HCS through the austempering treatment.

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