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Efficacy of Austempering Holding Time in Epoxidized-Transesterified Cotton Seed Oil Bath

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Research Article

Abstract

The influence of austempering holding time on the mechanical properties and microstructures of high carbon (AISI 1070) steel using epoxidized transesterified cotton seed oil (ETO) as quenchant has been studied. Initially, steel samples were austenitized at 850°C for 1 hour, then quenched interruptedly in an oil bath maintained at 280°C at different holding times from 15-75 minutes at regular interval of 15 minutes. Subsequently, the samples were removed and air cooled to room temperature. Mechanical properties test and microstructural examination were carried out. Obviously, except at 75 minutes austempering period; the results revealed significant enhancement in tensile strength (1580 MPa) at long holding time. Additionally, higher improvement in hardness value (430 HBN) and impact strength (12.7 J/mm) at short time was noticed. In the SEM (scanning electron microscope) images, bainitic structure (acicular), retained austenite and martensite were observed. Accordingly, 15 minutes austempering condition produced austempered high carbon steel (HCS) with the best mechanical properties.

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Keywords
Epoxidized transesterified cotton seed oil, austempering time, high carbon steel, bainitic structures, Mechanical properties, Bath

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1. Introduction

Historically, materials development tells the level of human civilizations (Stone Age, Bronze Age and Iron Age). Stone, wood, clay, and skins are the only naturally occurring materials that were available to early human civilization. As time went on, materials such as pottery and a number of metals were produced using invented techniques. This was as a result of the outstanding attributes they possess compared to those of natural materials. Additionally, it was proven that the performance of a material in service could be improved by heat treatments and by the addition of other substances (Callister and Wiley, 2006). One of the unique techniques for heat treating ferrous alloys is austempering. It is an isothermal heat treatment that develops an exceptional structure that is harder and tougher than conventional heat treatment methods. It consists of austenitizing a part to a temperature between 825-950°C, holding at this temperature for some time, depending on the component thickness, subsequently, quenching into a hot bath, holding there for a predetermined period of time between 260- 550°C then remove and air-cool (Sani and Aminu, 2021). Austempering is most typically conducted in molten nitrate/nitrite salt or lead baths. Outstanding mechanical properties results from such austempering heat treatment. However, a search for an alternative austempering medium becomes necessary due considerable limitations of molten salt and lead baths. The limitations include health hazards, scarcity, exorbitant price and eco-unfriendliness. Internationally, consideration is given to polymer solutions, vegetable and mineral oils as alternative replacement. It was reported by Okwonna *et al.*, (2017) that mineral oil (petroleum based) bath has significant cooling performance but they are toxic and non-biodegradable. Further, it has low flash point which may eventually leads to fire hazards. Vegetable oils are suggested by many scholars. Abdulhamid et al. (2017) revealed that sesame oil bath successfully austempered ductile cast iron. The study carried out by Okwonna, *et al.*, (2017) indicated that mixture of bitumen and palm kernel oil could be employed as austempering quenching media. Likewise, jatropha oil was used to austemper ductile cast iron with the resulting mechanical properties similar to that obtained in molten salt bath (Akor and Gundu, 2014). Again, potential of rubber seed oil was presented in the literature (Akor and Tuleum, 2014). Nonetheless, the challenge of vegetable oils as austempering quenching media is the occurrence of oxidative thermal decomposition at austempering temperature. Therefore, this study focuses on the use of modified vegetable oil (epoxidized transesterified cotton seed oil) while varying austempering time, so that the oxidative thermal decomposition would be minimized and thus cooling characteristics could be improved.

2. Materials and Methods

2.1 Materials

The composition of the steel involved in the investigation is 0.68%C. The oil bath filled with epoxidized transesterified cotton seed oil (ETO). Furthermore, some of the other materials include 4% picral, silicon-carbide grit papers and alumina powder.

2.2 Sample preparation

A total of 30 high carbon steel samples were prepared to ASTM A370 (ASTM, 2007); Six samples for hardness test, a pair of six samples for tensile test and then a pair of six samples for impact test.

The AISI 1070 steel samples were prepared for mechanical testing which includes tensile, impact and hardness tests. The hardness samples were subjected to microstructural analysis prior to the test. Accordingly, pair of twelve sample pieces each were produced for tensile and impact tests respectively. Six samples were as well prepared for the hardness test/microstructural analysis of the various conditions investigated. The samples were normalized to austenitizing temperature of 850ºC and holding for one hour after which it is cooled in air.

2.2 Heat treatment

Five sets of machined test samples (two of pairs of tensile and impact test and one for microstructural analysis and hardness) were placed in the furnace for heat treatment operation. The samples were austenitized at a temperature of 850°C and allowed to soak for 1 hour in the furnace. Afterwards, the samples removed from the furnace and quenched in an oil bath kept at 280 °C for 0.25 hour (15 minutes) and then taken out from the bath and air-cooled. The same procedure was repeated for the remaining sets at 0.5 hour (30 minutes), 0.75 hour (45 minutes), 1 hour (60 minutes) and 1.25 hour (75 minutes).

2.3 Tensile test

The test was carried out using Hounsfield tensometer, Type W with a capacity of 20kN and the procedure adopted was in conformity with ASTM E8 (ASTM, 2007);. Each of the samples was gripped at two ends on the tensometer and a uniaxial force is applied at a constant rate. The cell load and extensometer measured the force and extension simultaneously, until the sample finally breaks and the corresponding extension was recorded on a graph sheet. The data generated from the force extension graph sheet were used in calculating the tensile strength.

2.4 Hardness test

Hardness test was carried out on all the six hardness samples (5 austempered and 1 control). Maximum and minimum loads were 150kgf and 10kgf respectively. Three different readings were taken for each sample, with the average as the hardness value. The hardness test was carried out using an indentec universal hardness testing machine with Rockwell tester MN: 8187.5LKV model (B). The scale 'C' was used with a load of 150 kgf. The sample was placed on the anvil (flat surface) and a load was applied on the surface of the sample to obtain the hardness value; the load is applied for a standard time of 30secs. Hardness values (HRC) were read directly from the machine. An average value obtained for three indentations at different points was taken as the hardness of the samples. The corresponding brinell hardness values (HB) were also taken from the machine converter.

2.5 Impact Test

The test conducted as per ASTM E23 (ASTM, 2007); using an Izod impact testing machine with a capacity of 120ft/pounds. The sample is held on the anvil in a vertical position (cantilever test piece) with the notched facing the pendulum hammer, the pendulum was raised and gripped at the test height and the pointer of reading the impact energy value was adjusted to zero before the pendulum was released and allowed to fall freely under gravity. Upon breaking the samples, it hit the pointer of test energy value and the impact energy was recorded. This process was repeated for all the samples.

2.6 Metallography

The samples were prepared by successive steps of grinding, polishing, etching and microstructural examination. They were ground using abrasive grinding papers 220, 320, 400, 600, 800 and 1200 grit sizes to obtain a smooth shiny mirror-like surface. During the grinding, water was used as a coolant in order to prevent change in microstructure of the samples. Polishing of the samples was carried out on rotating disc universal polishing machine with a synthetic velvet polishing clothes, 1µm alumina paste was being applied to the surface of the polishing clothes. Subsequently, samples were etched with 4% picral solution by swabbing. The etched samples were then washed, clean with water and then dried. A smooth dull surface was obtained ready for microscopic examination.

Finally, the microstructures of the steel samples were visualized and snap under Scanning electron microscopy (SEM).

3. Result and Discussion

3.1 Tensile strength

After holding time of 15 min in Figure 1, the trend of the tensile strength of AISI 1070 steel progressively increases with increased austempering time and start to decreases at 75 min. Austenite undergoes a partial transformation into bainitic ferrite during the limited holding periods, and the austenite regions are not stabilized with carbon as a result. However, retained austenite transforms into bainitic ferrite, and austenitic regions are adequately stabilized during lengthy holding times. This result agrees with the findings of Ranjit et al., (2018), who works on the potential of austempering temperature and time on the mechanical properties of SAE 9260 steel. Likewise, the same findings reported by Ashwe (2014), who worked on the study of the potential of jatropha seed oil as austempering quenchant for medium carbon steel. However, this is contrary to the investigation of (Sahoo, 2012), who studied how copper addition, austempering temperature and time affect the mechanical properties of austempered ductile iron. The tensile strength results revealed that the sample austempered for 60 minutes has the peak value of tensile strength of 1580 N/mm^2, while the normalized sample has the least of 850 N/mm^2. As austempering time rises, there was corresponding decline in the amount of retained austenite and fine precipitates of cementite noticed. This is evident in the SEM images illustrated in Plate I.

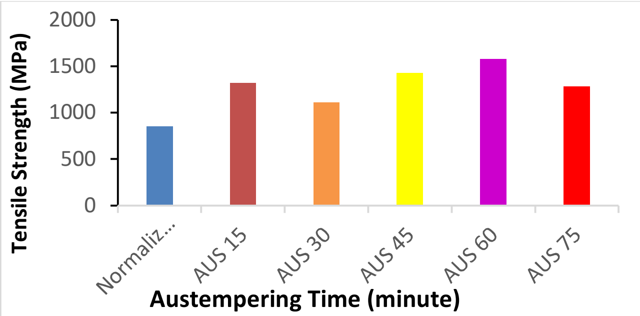


Figure 1: Tensile strength of normalized and austempered samples

3.2 Hardness

The major cause of the highest hardness value observed at 30 minutes is the presence of the brittle and hard phase martensite (Figure 2). Notwithstanding, hardness value decreases as isothermal holding time increases. This was due to the transformation of γ_{hc} to bainite (α) and ε-carbide. At longer holding time more bainite were formed in the microstructure. This result was similar with the findings of (sahoo, 2012), who researched on how copper addition, austempering temperature and time affect the mechanical properties of austempered ductile iron. In the event of the isothermal transformation, cementite precipitates are rejected from the carbon-enriched austenite films between the newly formed carbon-saturated ferrite plates. Thus, the resultant microstructure has inferior hardness values.

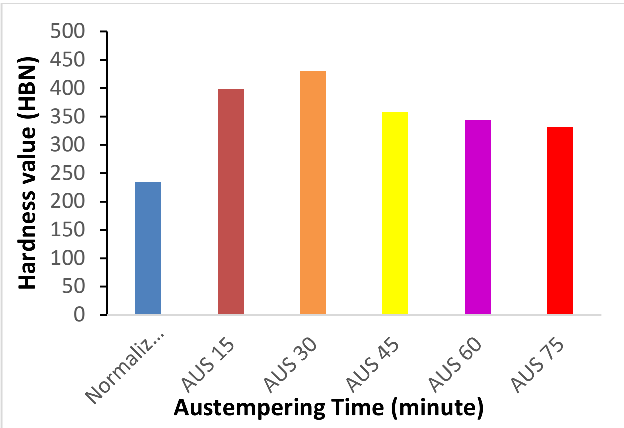


Figure 2: Brinell hardness of normalized and austempered HCS samples

3.3 Impact strength

The nature of the chart in Figure 3 clearly demonstrates the potential of austempering heat treatment on impact strength of high carbon (AISI 1070) steel samples using epoxidized transesterified cotton seed oil bath. As presented in the Figure, the impact strength significantly increases with increased austempering time. As a result, more bainite was formed.

Compared to martensite, the structure of bainite is tougher. Accordingly, it is noted that the steel toughness rises as austempering time increases, with the exception of some point where ε-carbide formation causes brittle structure. Likewise, (Reddy, n.d.) reported the same on the influence of austempering parameters on impact strength of medium carbon and high alloy steels.

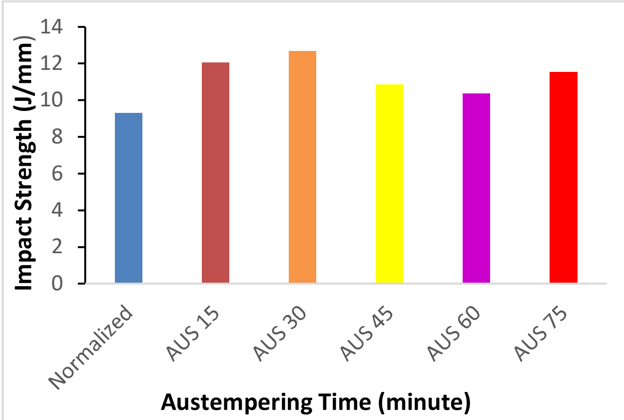


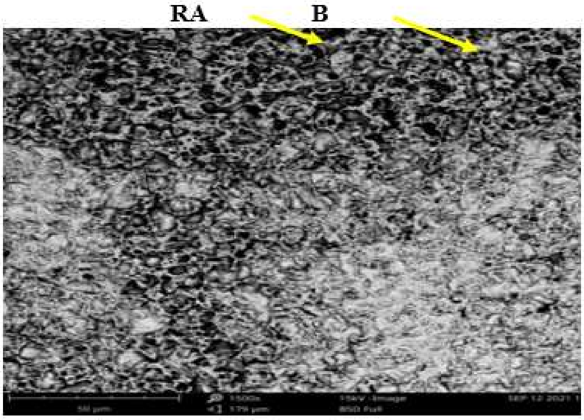
Figure 3: Impact strength of normalized and austempered HCS samples

3.4 Microstructural Examination

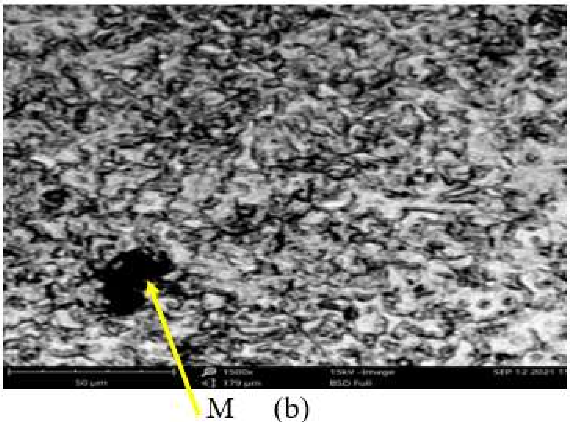
The SEM images of the HCS austempered at various holding time are depicted in Plate I. The images represent bainite (B). On the other hand,

Small dark blocks of retained austenite (RA) are visible in the dark phase, which implies martensite (M).

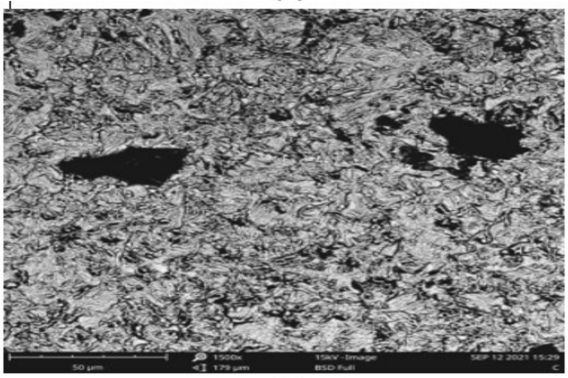
Austempering at 280ºC led to the formation of lower bainite (consist of acicular structure) and retained austenite; because cementite only precipitated inside the carbon-saturated ferrite plates at this temperature due to relatively slow diffusion. For 15 minutes austempering period, samples had a greater proportion of retained austenite than bainite and a limited amount of martensite. The percentage of bainite, however, increases as the holding period rises, whereas the amount of retained austenite falls; hence, improved in hardness and toughness at 30 minutes except at some point due to the formation of ε-carbide which led to brittleness. As the isothermal transformation completed around 30 minutes, subsequent increase in the holding time only cause mild change in the amount of bainite. Similar observation was made by Ashwe, (2014) on the possibility of jatropha seed oil as an austempering bath for medium carbon steel and again, in another study conducted by Muhammad *et al.,* (2019) on the impact of austempering variables on the mechanical properties and microstructure of AISI 4340 and AISI 4140 steels.



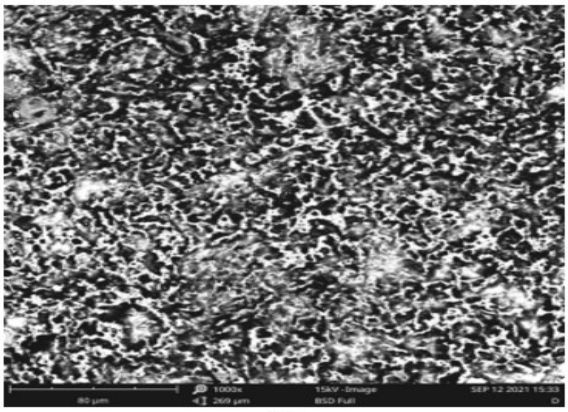
(a)



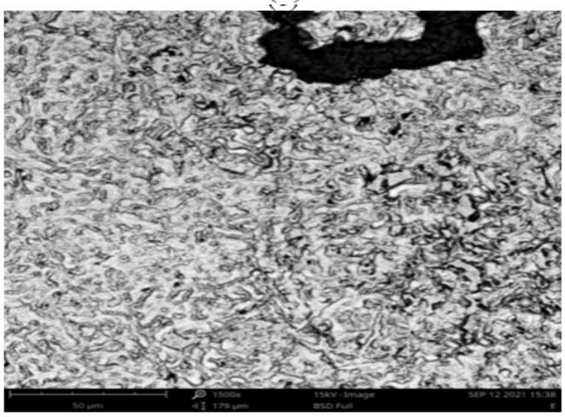
(b)



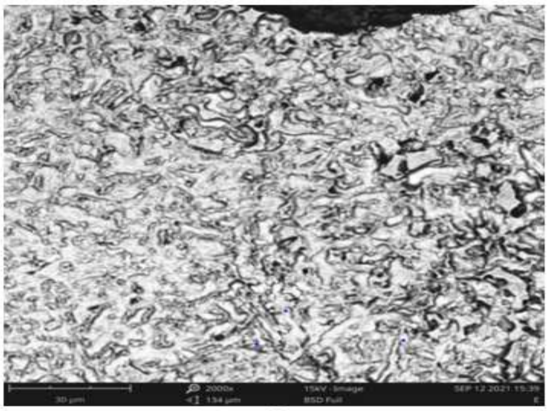
(c)



(d)



(e)



(f)

Plate I: High carbon steel sample SEM images austempered at (a) 15 min (b) 30 min (c) 45 min (d) 60 min (e) 75 min (f) 75 min.

The SEM images of the HCS austempered at various holding time are depicted in Plate I. The images represent bainite (B). On the other hand,

4. Conclusions

It can be concluded from the results presented that splendid tensile strength of HCS can be achieved if the sample is held 60 minutes in the bath. Further, of all the austempering time; major improvement in hardness value and impact strength was notice in sample austempered in 30 minutes. The overall mechanical properties of the samples austempered in 30 minutes holding is the best compared to those held in the other soaking times. Hence, austempering HCS in ETO bath at 280 °C in 30 minutes holding time gives the superior mechanical properties.

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