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Effect of different austenitizing temperatures on the impact strength and hardness of ductile iron austempered in sesame oil bath

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Abstract:
Heat treatment operations that require hardening of components by quenching, those quenching media such as water, brine, oil may sometimes be too drastic, hence resulting to inferior mechanical properties and defects such as cracking and distortion. Such defected components are eventually rejected, resulting to economic waste. However, in order to impart good mechanical properties, especially strength and toughness and simultaneously to avoid cracking and distortion, many researchers suggested that other heat treatment method like austempering in molten salt bath or hot oil bath could be used. But unfortunately, salt bath furnace is known to be very costly. Therefore, this study aimed at the effect of different austenitizing temperatures on the impact strength and hardness of ductile cast iron austempered in hot sesame oil (Sesanum indicum) bath. Samples of ductile cast iron were machined to standard dimensions for impact strength test, hardness test and microstructural analysis prior to the austempering process. Sesame oil was used as hot quenching bath for the austempering process. The samples were normalized and thereafter, austempered in hot Sesame oil bath at 300°C for periods of 1,2,3,4 and 5 hours from austenitizing temperatures of 850°C and 910°C. Mechanical properties (impact strength and hardness) of the test samples were evaluated. The test results obtained showed that the highest hardness value of 403HVN was obtained from samples austenitizing at 910°C. Maximum impact strength of 157 kJ/m^2 was also obtained from samples austenitizing at 910°C. Microstructures of the samples were analysed using scanning electron microscopy (SEM). The microstructure, which consist of graphite nodules embedded in a matrix of ausferrite (acicular ferrite and carbon enriched austenite) was observed.

Keywords: *Ausferrite, Austempering, Ductile iron, Quenching, Sesame oil*

1. Introduction

Since the discovery of nodular (ductile) cast iron in 1948, no other new type of cast iron has been discovered, hence austempered ductile cast iron (ADI) is a relatively new engineering material with exceptional combination of mechanical properties and marked potential for numerous applications [1, 2]. The attractive properties of ADI are due to its distinct and unique microstructure, which consists of fine acicular ferrite with carbon enriched stabilized austenite (ausferrite) [3]. Metals and alloys develop requisite properties by heat treatment which plays a critical role in achieving appropriate microstructure that imparts the desired characteristics in a given material [4, 5]. Hence, the study of heat treatment is of great significance [6].

Akor and Tuleum [7] pointed out that, in the middle of the 19th century, more attention was directed to develop the art and science of producing, working and treating of metals to suit various areas of applications to improve man's comfort. The final properties of an alloy are not only due to its chemical composition, but also due to its metallurgical history. It's possible to use different treatments so as to change the properties of an alloy; thus, finding the easiest way of heat treatment and optimum treatment conditions is of fundamental importance in achieving the desired properties [8, 9, 10]. Isothermal heat treatment is most typically carried out in nitrate/nitrite salt bath due to the advantages they offer compared to others, although a lot of attention is given today to vegetable oils, minerals oils and polymer solutions [11]. Generally, quenchants are grouped into two categories namely: those maintained at room temperature and those maintained at higher temperatures (250-450°C) [12]. The room temperature quenchants are regarded as the most popular, common and conventional or traditional type of quenchants. Few examples include; water, brine, oil (mineral or vegetable), caustic soda solution, etc [11, 13]. Those quenchants maintained at higher temperatures are used for interrupted quenching heat treatment processes.

Examples of these types of quenching media are molten salt bath, molten lead bath and hot oil bath [14]. Ductile iron is a family of cast iron with free carbon existing as spherical graphite [15]. Compared to other kinds of cast irons it has the best mechanical properties which makes it a very attractive material. The heat treating of ductile iron by austempering produces the austempered ductile iron with outstanding mechanical properties that can be varied over a wide range by changing the heat treatment parameters [16, 17]. Austenitizing and austempering temperatures and time are factors that affect the final properties of ADI [7, 18, 19]. Depending on various parameters (composition of the alloy, cooling rate, section size, etc), austenite may transform on cooling to lower temperature phases such as: ferrite, pearlite, bainite or martensite. The presence of these transformed phases and their structure is important in deciding the resultant properties in the metal/alloy [11, 20].

The resulting microstructure, known as "Ausferrite", consists of unique matrix of fine acicular ferrite with carbon enriched stabilized austenite while, the matrix of conventional ductile iron is a mixture of pearlite and ferrite [21]. Austempered ductile iron offers a design engineer the best combinations of low cost, design flexibility, good machinability, high strength to weight ratio and good toughness, wear resistance and fatigue strength [22]. Austempered ductile iron has a microstructure containing spheroidal graphite embedded in a matrix of bainite (ferrite and austenite). The resulting microstructure of ADI provides properties that are superior to conventional ductile iron, cast and forged aluminium and many cast and forged steels [22, 23]. Many researchers reported on the suitability of polymer solution, hot bitumen, mineral and vegetable oils as quenching medium for austempering of nodular cast iron. These findings minimized the need for the molten salt and lead baths.

2. Methodology

2.1 Materials

The materials used in this research work include: Ductile (Nodular) cast iron samples, Sesame oil, polishing powder, water, grinding papers, polishing cloth, cotton wool, copper piece, nitric acid and ethanol. The equipment used include: Oil bath, electrically heated muffle furnace, optical Metallurgical Microscope (OM), Scanning Electron Microscope (SEM), lathe machine, polishing machine, Hounsfield balance impact testing machine and Vickers hardness testing machine.

2.2 Methods

The ductile cast irons (rod form) were machined to standard impact, hardness and microstructural analysis samples. The machined samples were initially normalized by heating to a temperature of 900^{o}C and soaked for one hour and then air-cooled. 15 samples were loaded into the furnace, heated to 850°C, soaked for one hour, then removed and quenched in hot Sesame oil bath at 300°C. After one hour, the first set of samples (one hardness and two impact test samples) were removed from the hot Sesame oil bath, cooled in air. Another set of samples were removed after 2, 3, 4 and 5 hours respectively and cooled in air. The same process was repeated as described above for austenitizing temperature of 910^{o}C.

2.3 Hardness test measurement

The hardness test samples were prepared to standard dimension as specified in ASTM A370 [24]. The test method adopted is ASTM E18. A diamond indenter was used to indent the surface of the test specimen by the application of static load of 30kgf, which was maintained for fifteen minutes. Both diagonals of the impression were measured using a lower power graduated microscope. The mean value of the diagonal length was used in the determination of the hardness number. The procedure was repeated for 3 different points of the test piece and the average value was recorded.

2.4 Impact Test

The impact test samples were machined to standard dimensions as specified in ASTM A370 [24]. The test method adopted is ASTM E23.The samples were machined to 8mm diameter and 45mm length with a 1mm deep V-notch. Before the test, the pendulum was set to a potential energy position of 162.75J. The test specimen was griped vertically in a vice, the trigger was released and the registering pointer of the quadrant scale will indicate the energy absorbed in Joules to break the specimen. The energy absorbed in breaking the sample was recorded and the same was repeated for other test pieces.

2.5 Microstructural examinations

All the specimens (both untreated and heat-treated) were prepared for optical microscopic examination. The specimens were ground on a water lubricated silicon carbide abrasive papers of 180, 240, 320, 400 and 600 grit sizes. Polishing was carried out on 15cm rotating discs of a *Metaserv* universal polishing machine with synthetic velvet polishing clothes impregnated with 1µm Alumina paste. The specimens were then etched with 2% Nital solution using the swabbing method with cotton wool soaked in the etchant. The microscopic examinations were carried out on M100 optical metallurgical microscope and the microstructures obtained were captured with in-built camera. SEM was carried out as well for the quenched samples.

3. Results and Discussion

The chemical composition of the nodular cast iron used is given in Table 1. The Physicochemical properties of the Sesame oil used is shown in Table 2. Fourier Transform Infrared Spectroscopy (FTIR) Analysis. The Fourier transform infrared spectroscopy (FTIR) analysis of the Sesame oil before and after heat treatment are shown in Figure 1 and 2.

Table 1. Chemical composition of the as-received ductile cast iron

Element	**C**	**Si**	**Mn**	**P**	**S**	**Cu**	**Ni**	**Mo**	**Mg**	**Fe**
Content (%)	3.81	2.70	0.26	0.06	0.042	0.32	1.85	0.2	0.04	Balance

Table 2. Physico-chemical properties of the Sesame oil used

Iodine Value (g/100g)	**Acid Value (mgKOH)**	**Peroxide Value (mgKOH/g)**	**Saponification Value (mgKOH)**	**Density (g/cm^3)**	**Viscosity 25.5^{o}C(cp) Before Aust.HT**	**Viscosity 25.5^{o}C(cp) After Aust.HT**	**Smoke Point (^{o}C)**	**Flash Point (^{o}C)**
113.2	0.5	7.7	189	0.914	40.2	48	223	391

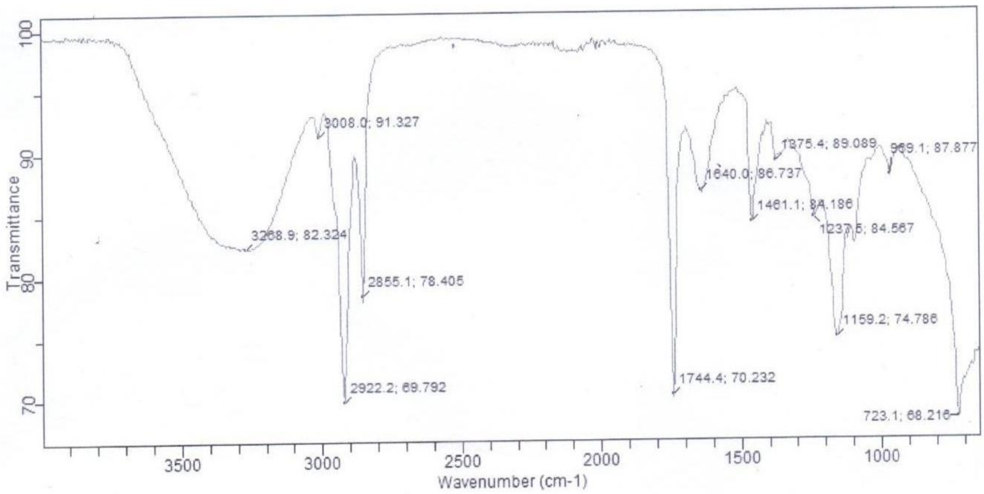

Figure 1: FTIR of the Sesame oil before heat treatment.

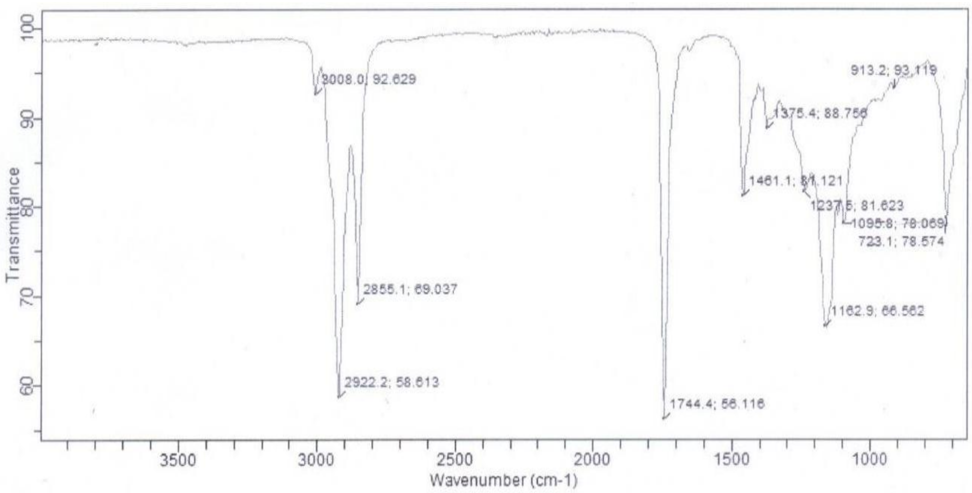

Figure 2: FTIR of the Sesame oil after austempering heat treatment

Figures 1 and 2 show Fourier transform infrared spectroscopy (FTIR) of the Sesame oil before and after austempering heat treatment. It was observed that some peaks (functional groups/radicals) were re-configured after the austempering heat treatment. This is attributed to the fact that some peaks 1095.8, 1162.9, 1237.5 and 1461.1 cm^{-1} representing functional groups and indicating the class of compounds (Ethers, Esters and Alkyls) before heat treatment changed to 1159.2, 1237.5, 1375.4, 1461.1 and 3268.9 cm^{-1} indicating the class of compounds (Alcohol, Alkanes, Amides and phenol) may be responsible for the result obtained.

3.1 Impact Strength

Figure 3 indicate the effect of austenitizing temperatures on the impact strength of ductile cast iron samples austempered in sesame oil bath. From Figure 3, it can be seen that the effect of austenitizing two different temperatures of 850^{o}C and 900^{o}C on the impact strength of ductile cast iron samples austempered in hot Sesame oil bath at 300^{o}C for 1-5hours.

The results clearly show that, as the austempering time increase, impact strength of the samples increased until it reaches 3 hours. This increase may be due to formation of stage 1 austempering reaction which might have caused large fraction of austenite to transform to ausferrite structure. The decrease in impact strength by samples austempering at 4 and 5 hours, could be attributed to the brittleness set into the samples because of the start of stage II austempering reaction. These observations are in line with what Ause [11] established.

3.2 Hardness

Figure 4 indicate the effect of austenitizing temperatures on the hardness of ductile cast iron samples austempered in sesame oil bath.

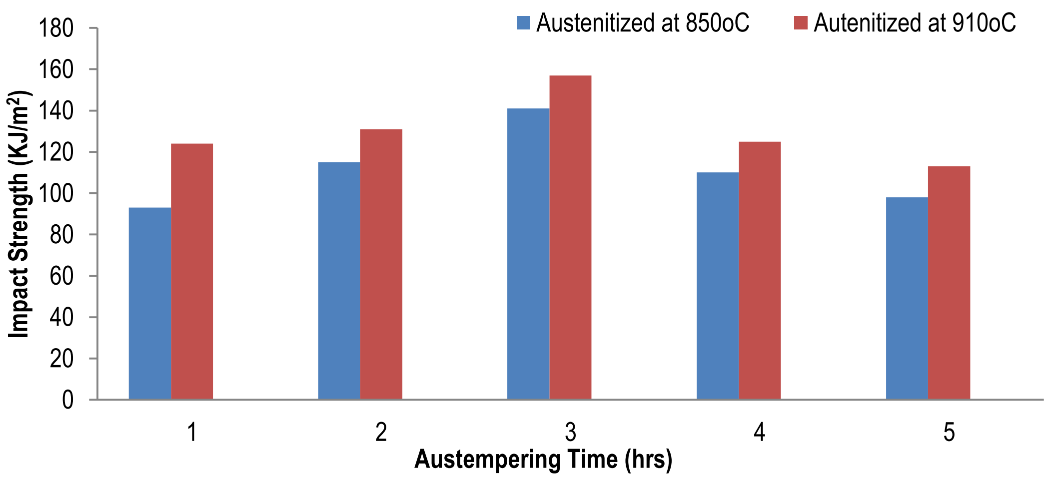

Figure 3: Profile of impact strength of the ductile cast iron samples austenitized at 850ᵒC and 910ᵒC austempered in sesame oil at 300ᵒC.

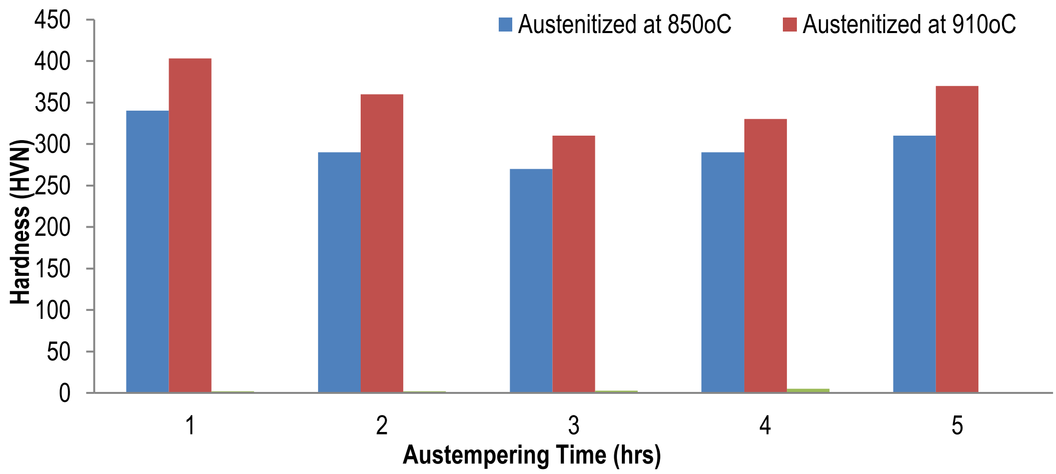

Figure 4: Hardness Profile of the ductile cast iron samples austenitized at 850ᵒC and 910ᵒC austempered in Sesame oil at 300ᵒC.

Figure 4 illustrate the effect of austenitizing temperatures (850ᵒC and 900ᵒC) on the hardness of ductile cast iron samples austempered in hot Sesame oil bath at 300ᵒC for 1-5hours. Hardness values were found to be high at initial austempering times but decreased to a minimum at 3 hours and then increased as the austempering time was increased. The higher hardness values obtained at the austempering time of 1hour may be due to the insufficient time for reasonable austempering reaction to have occurred before the sample was removed from the hot sesame oils bath, leading to high proportion of retained austenite. The increase in of hardness at austempering times of 4 and 5 hours may be attributed to the presence of ε-carbide formed during stage II reaction. This observation is similar to the samples austempered in molten salt bath as reported by Tun and Lwin [15]. An increase in hardness as the austenitisation temperature was increased from 850ᵒC to 910ᵒC was observed, and the reason for this could be attributed to the enlargement of austenite grains which improves hardenability [23].

3.4 Microstructure

The SEM images of the ductile cast iron samples austenitized at 850ᵒC and austempered in sesame oil bath at 300ᵒC for 1-5hours is shown in plate I to V. Also, plate VI to X show the SEM images of tested ductile cast iron samples austenitized at 910ᵒC and austempered in sesame oil bath at 300ᵒC for 1-5hours.The structure of Plate I show ausferrite structures and retained austenite. The structure in Plate II show ausferrite structures and retained austenite. The structure in Plate III show ausferrite structures. The structures in Plate IV show ausferrite structures. The structure in Plate V show ausferrite structures. The structure in Plate VI shows ausferrite structure and retained austenite. The structure in Plate VII shows ausferrite structure and retained austenite. The structure in Plate VIII shows ausferrite structure. The structure in Plate IX shows ausferrite structure. The structure in Plate X shows ausferrite structure.

The microstructures of the samples austempered in hot Sesame oil bath at 300°C for 1-5hours from austenitizing temperatures of 850°C and 910°C. The SEM images (Plate I to X) show that the proportion of ausferrite structures increase with increase in austempering time. This could be attributed to the progressive setting in of stage I austempering reaction. This trend clearly explained the high toughness obtained for samples austempered up to 3 hours.

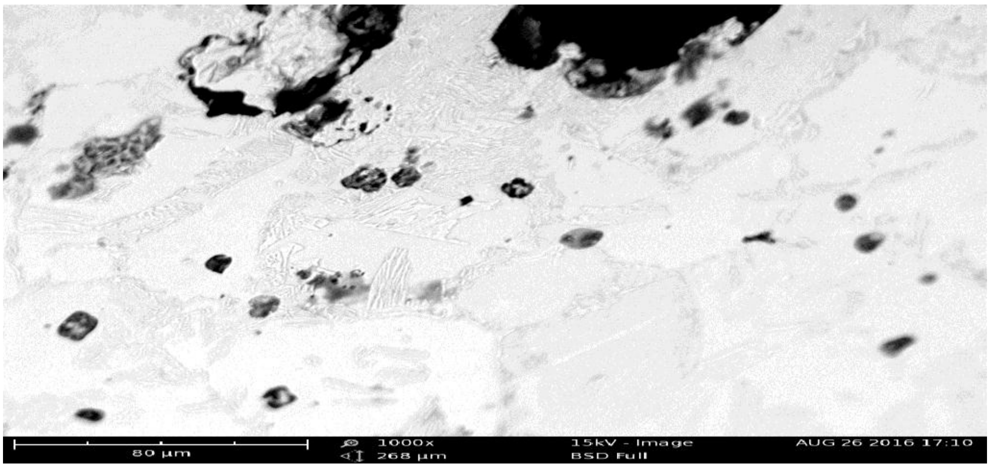

Plate I: SEM image of sample austenitized at 850°C and austempered in hot Sesame oil bath at 300°C for 1hour.

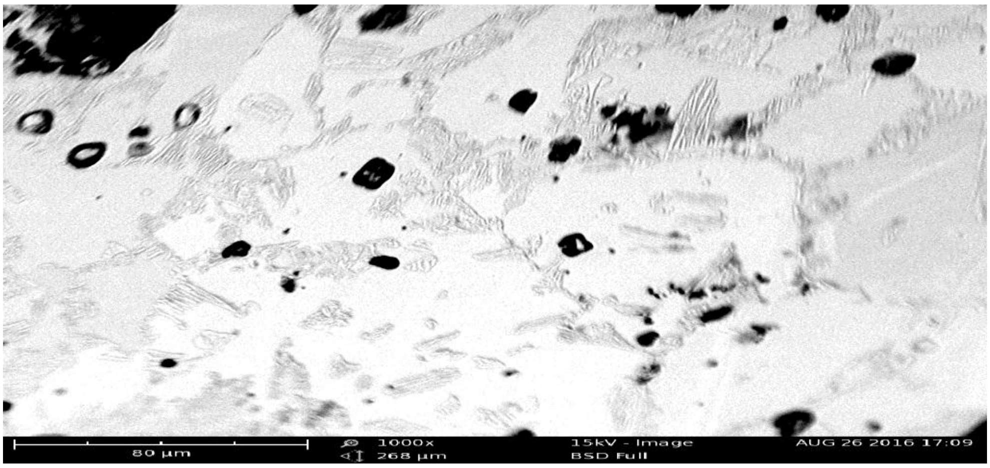

Plate II: SEM image of sample austenitized at 850°C and austempered in hot sesame oil bath at 300°C for 2hour.

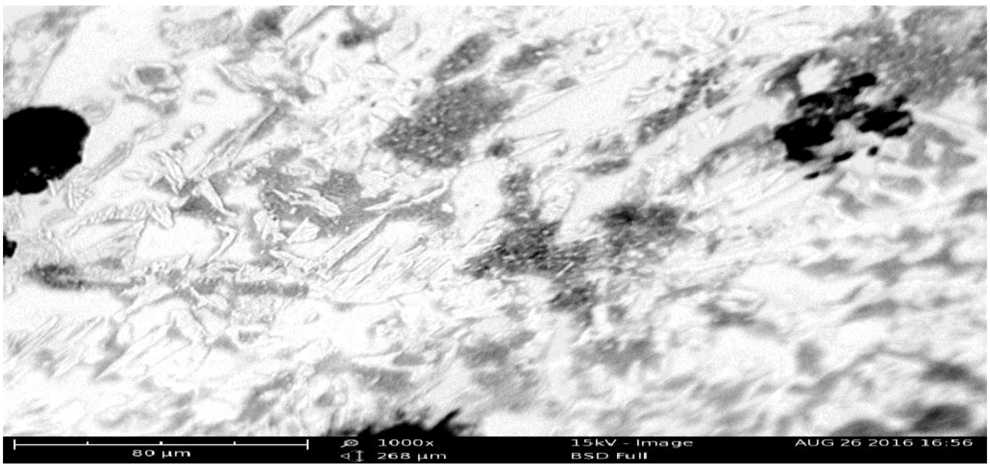

Plate III: SEM image of sample austenitized at 850°C and austempered in hot Sesame oil bath at 300°C for 3hour.

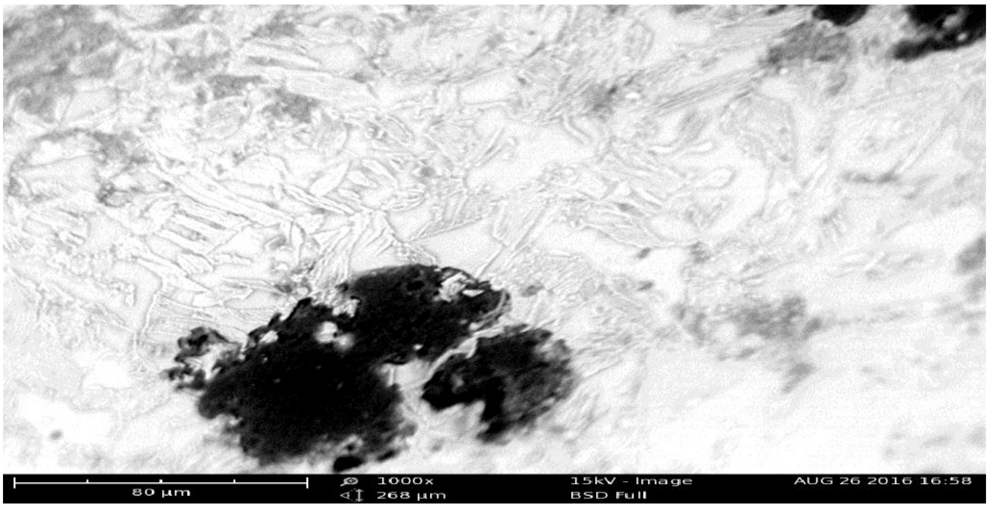

Plate IV: SEM image of sample austenitized at 850°C and austempered in hot Sesame oil bath at 300°C for 4hour.

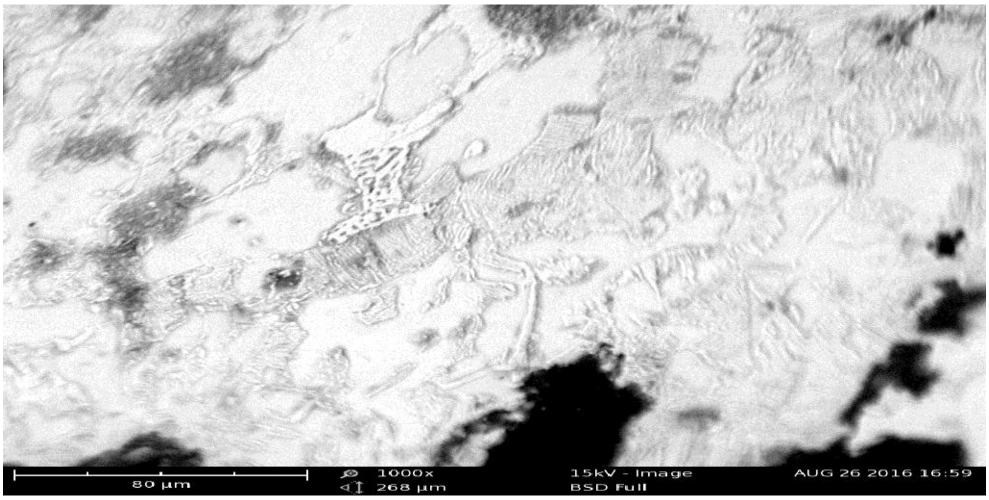

Plate V: SEM image of sample austenitized at 850°C and austempered in hot Sesame oil bath at 300°C for5hour.

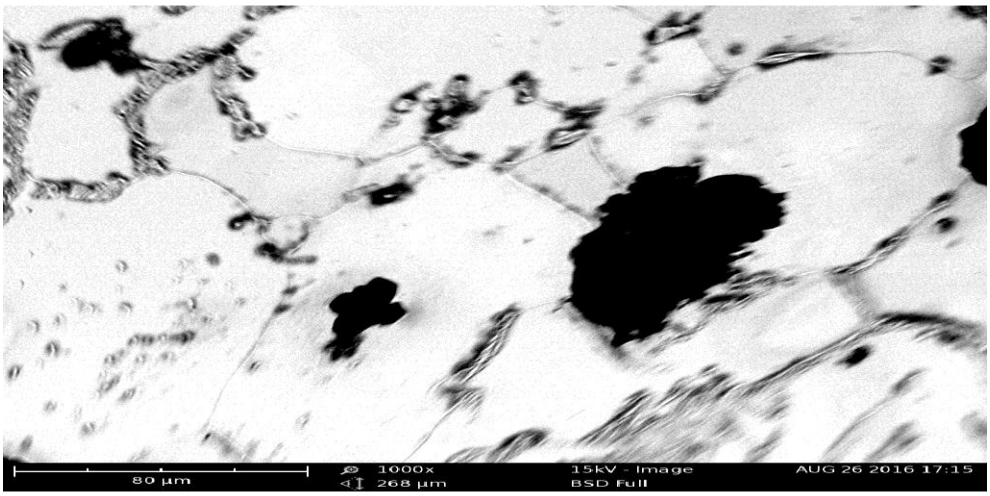

Plate VI: SEM image of sample austenitized at 910°C and austempered in hot Sesame oil bath at 300°C for 1hour.

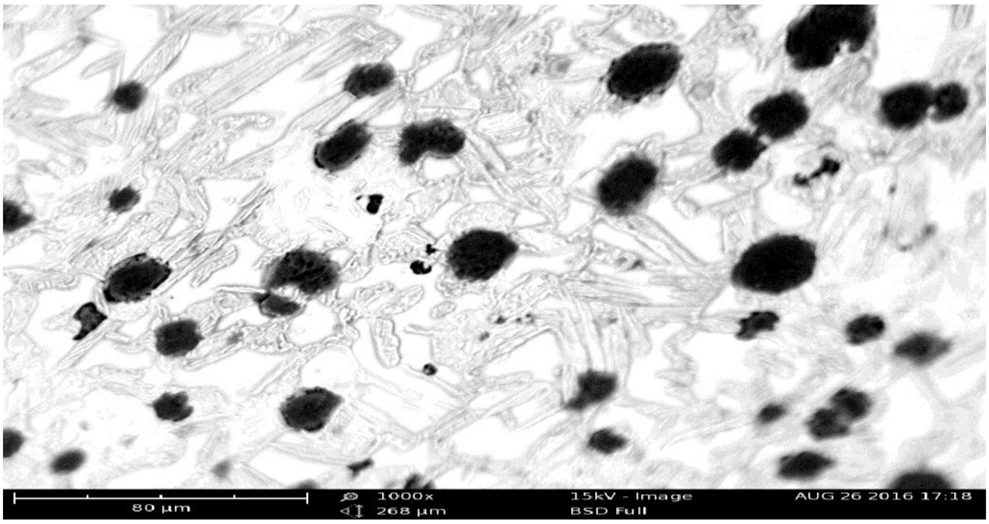

Plate VII: SEM image of sample austenitized at 910°C and austempered in hot Sesame oil bath at 300°C for 2hours;

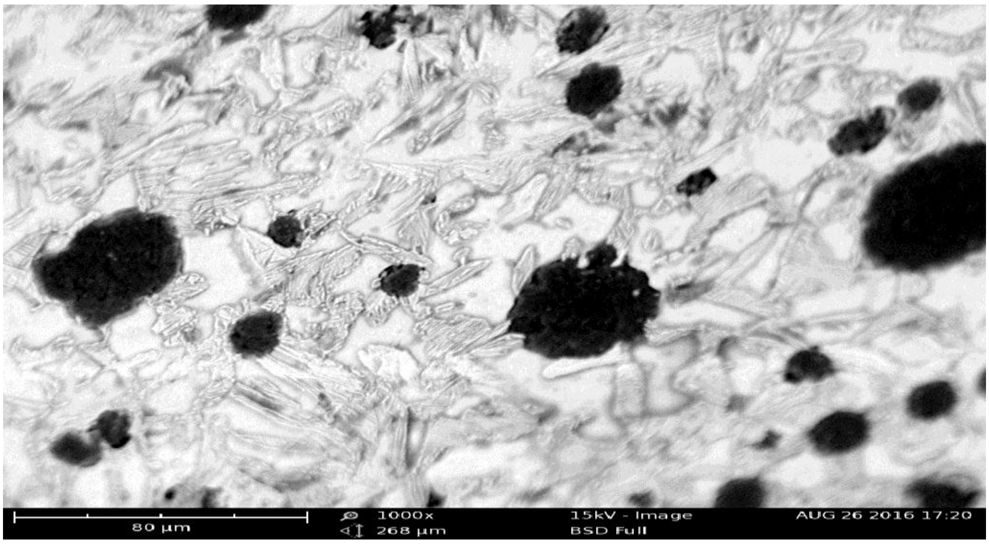

Plate VIII: SEM image of sample austenitized at 910°C and austempered in hot Sesame oil bath at 300°C for 3hours

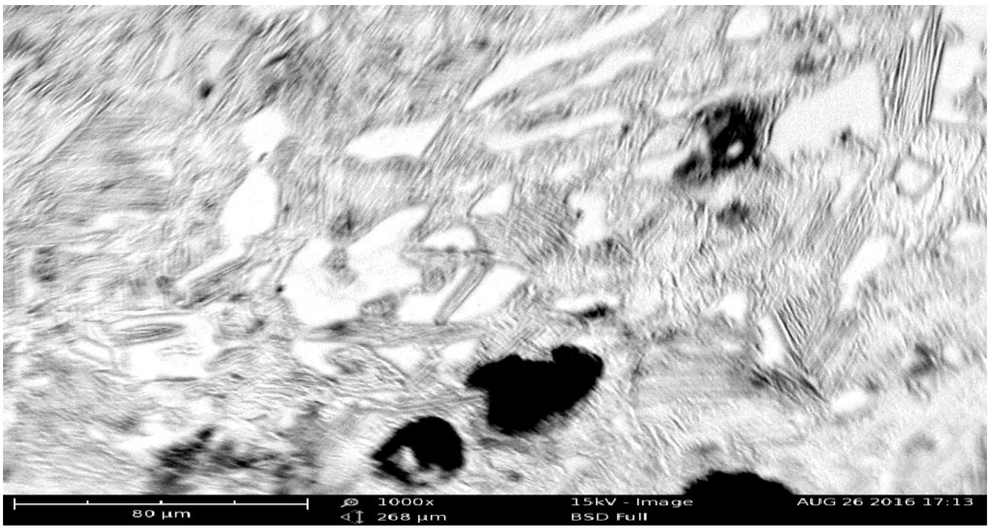

Plate IX: SEM image of sample austenitized at 910°C and austempered in hot Sesame oil bath at 300°C for 4hours.

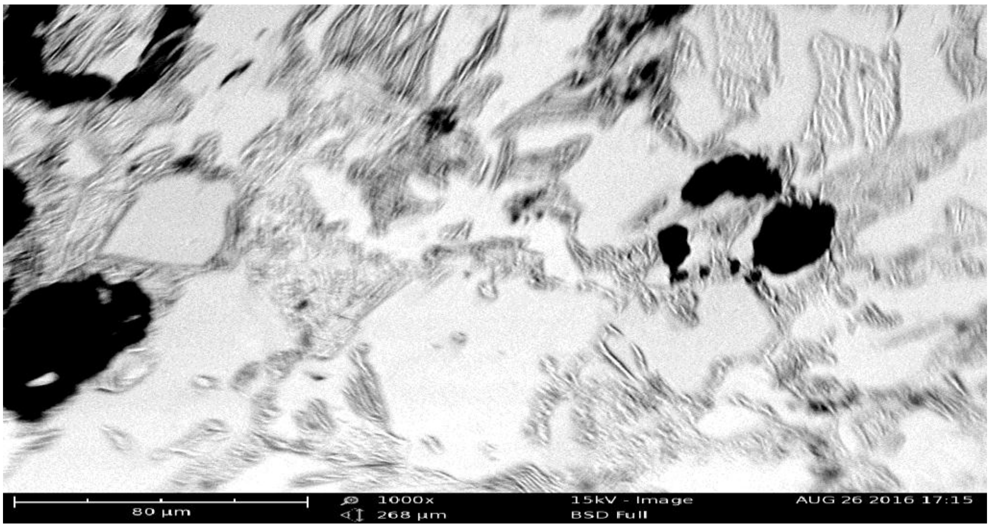

Plate X: SEM image of sample austenitized at 910°C and austempered in hot Sesame oil bath at 300°C for 5hours

However, samples austempered at 4 and 5 hours proved to show relatively coarser ausferrite lath with fine precipitates of brittle carbides. The brittle carbide formed as a result of second stage austempering reaction. This caused the inferior mechanical properties in samples austempered at 4 and 5 hours. These SEM images are similar to those obtained from sample austempered in molten salt bath by Tun and Lwin [15] and Mohamed, *et al.* 25].

4 Conclusions

Based on the results of this study, the following conclusions can be drawn:

Austempering in hot sesame oil bath gave microstructure that consist of ausferrite structure and retained austenite in the samples similar to those in literature, thus could be used as an austempering quenching medium. The hardness of ductile cast iron samples increased progressively as the austenitizing temperature increase from 850°C to 910°C. Since the sesame oil investigated has been able to produce ausferrite structure, and considering the fact that it is cheap and environmentally friendly compared to molten salt and lead bath, therefore should be added to the list of existing austempering quenching media

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