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MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF AUSTEMPERED DUCTILE IRON PRODUCED BY INTERRUPTED COOLING IN WARM WATER

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ABSTACT: Ductile iron were quenching in warm water (80^0C) for 20 seconds after austenitising at 850^0C. Austempering was then carried out at 180^0C, 210^0C, 240^0C, 270^0C and 300^0C respectively. Samples were held in the furnace for 30 minutes, 60 minutes, 90 minutes, 120 minutes and 150 minutes respectively. After the isothermal treatment, the samples were cooled in air. Some selected samples were austempered in a salt bath ($NaNO_3$ +KNO_3) at 270^0C for the same period stated above. All the test samples were then subjected to mechanical tests and microscopic examination. Optimum strengths were found at austempering temperatures of 270^0C and 300^0C and holding times of 90 minutes for the interrupted cooling methods. Optimum hardness values were obtained at austempering temperatures of 180 and 210^0C. These properties were found to be better than that obtained from the salt bath method. The salt bath method however, produced alloys with better ductility. Interrupted quenching process, using warm water, was found to be suitable for the production of fine structures, such as lath martensite, ausferrite, bainite and fine pearlite, in ductile irons.
Keywords: warm water, austenitisation, austempering temperature, austempering time

1. INTRODUCTION

The properties of cast iron components are controlled by the microstructure which is determined by the chemistry and processing method (Mark, 2000). Matrix control, obtained in conventional ductile iron either "as-cast" through a combination of composition and process control, or through heat treatment, gives the designer the option of selecting the grade of ductile iron which provides the most suitable combination of properties (Keough, 1998). The mechanical properties of ductile iron and austempered ductile iron (ADI) are primarily determined by the alloy matrix. The matrix in conventional ductile iron is a controlled mixture of pearlite and ferrite. Tempered martensitic matrices may be developed for wear resistance, but lack the ductility of either as-cast ductile iron or ADI. The properties of ADI are due to its unique matrix of acicular ferrite and carbon stabilized austenite called ausferrite. The austempering process is neither new nor novel and has been utilized since the 1930's on cast and wrought steels.

Austempered ductile iron (ADI) offers superior combination of properties because it can be cast like any other member of the ductile iron family, thus offering all the production advantages of a conventional ductile iron casting. Subsequently it is subjected to the austempering process to produce mechanical properties that are superior to conventional ductile iron, cast and forged aluminum and many cast and forged steels (Keough, 1998).Isothermal heat treatment of ductile iron to convert it to austempered ductile iron is normally done in nitrate/nitrite and cyanide salt baths. Attention is however shifting to the use of vegetable oil, mineral oil and polymer solution (Larry, 2004). This shift is primarily due to the disadvantage of using molten salt such as high

energy consumption as a result of high temperature of the salt bath. Disposal of such salt and hazards associated with some of the salts is also of concern (Hassan and Issa 2009). Thus attention is shifting to the use of eco-friendly methods (Oyetunji and Barnabas, 2012; Hassan and Issa, 2009).The aim of this research work is to explore the viability of producing austempered ductile iron using eco-friendly method. The use of cyanides and other harmful salts as quenching medium exposes the heat treather to hazards as earlier stated. Moreover, the disposal of some of these salts constitute environmental problem. It is also noteworthy that the salts have higher heat capacity than other quenching medium and therefore lead to higher energy consumption. However, water, the quenching medium used in this research is cheap, environmentally friendly and readily available. This research will provide information on the effectiveness of super cooling with water (at between 70°C and 80°C) and austempering as an alternative to the salt bath process of making ADI. This will extend the frontier of knowledge in the development of high quality austempered ductile iron.

2. METHODOLOGY

All ductile iron test specimens, used for this researh work, were cast in Madison, USA. The procured ductile irons were machined to standard tensile test samples. Some samples were sectioned for hardness test. All test samples were subjected to traditional process of normalizing. This was done to effect homogenization of the as–cast structure. Some selected samples were austenitised and super-cooled in warm water held at 80°C.The samples were held in the warm water for 20 seconds. The samples were then transferred quickly to a furnace held between 180°C and 300°C. Samples were held for 30 minutes, 60 minutes, 90 minutes, 120 minutes and 150 minutes respectively. After the isothermal treatment, the samples were cooled in air. Also, some selected samples were austempered in a salt bath ($NaNO_3$ +KNO_3) at 270°C for the same time period stated above. All samples which have been subjected to super cooling and austempering were subjected to mechanical tests. Microscopic analysis of specimen was carried out using optical microscope.

Table 1: Chemical composition of the ductile iron as cast

% C	%Si	%Mn	%Mg	%S	%P	%Fe
3.6	2.0	0.3	0.05	0.01	0.07	Bal.

This gives a carbon equivalent of 4.29%, which is eutectic and is considered to be an ideal composition for unalloyed ductile cast iron. The ultimate tensile strength, yield strength, percentage elongation and hardness of the as-cast sample of ductile iron are 564.70 N/m², 440 N/m², 15% and 21.80Rc and that of the as normalized test sample are 761.5 N/m², 565.2 N/m², 20.2 and 22.8Rc respectively.

3. RESULTS

Results obtained after interrupted quenching in warm water (80°C) and secondary holding in the heat treatment furnace showed variation in the ultimate tensile strength (UTS), yield strength, percentage elongation and hardness which largely depend on the different austempering temperatures (180°C, 210°C, 240°C, 270°C, 300°C) and the holding time (30 minutes, 60 minutes, 90 minutes, 120 minutes, 150 minutes). The variation in the ultimate tensile strength (UTS), yield strength, percentage elongation and hardness obtained at different austempering temperature and time are shown in Figures 1, 2, 3 and 4 respectively. These data were also compared with the conventional austempering process in the salt bath.

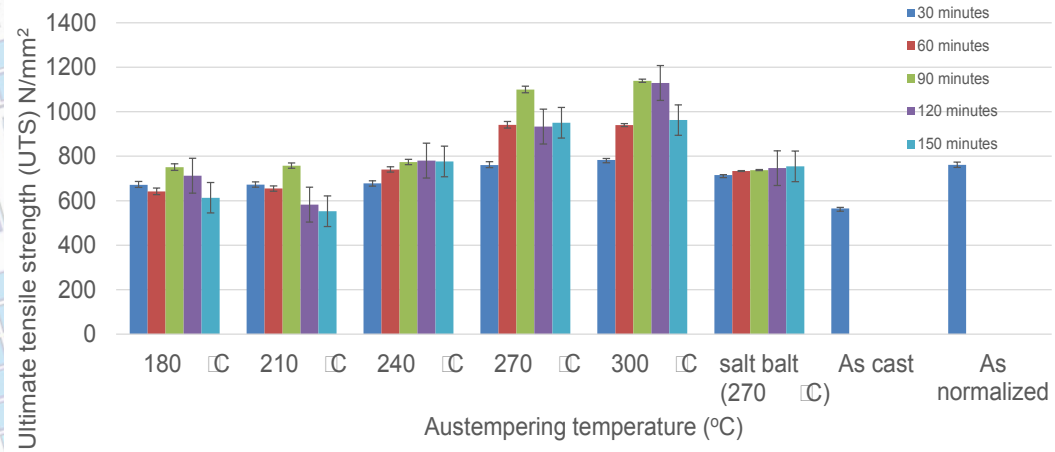

Figure 1. Ultimate tensile strength at different austempering temperature and time.

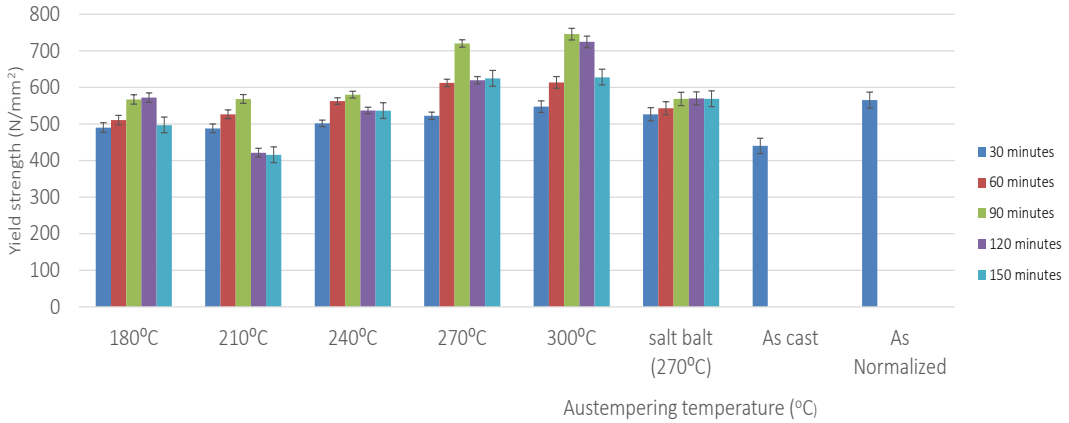

Figure 2. Yield strength at different austempering temperature and time

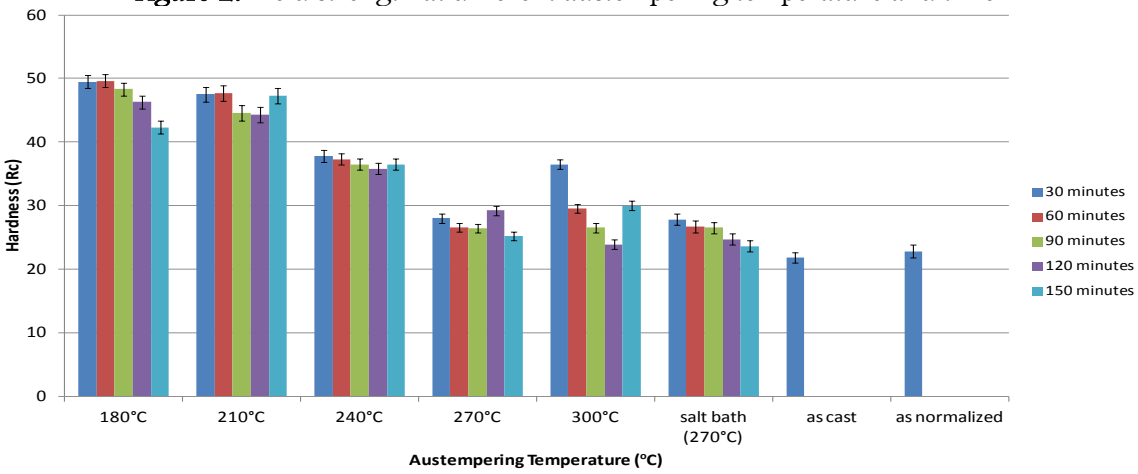

Figure 3. Hardness at different austempering temperature and time

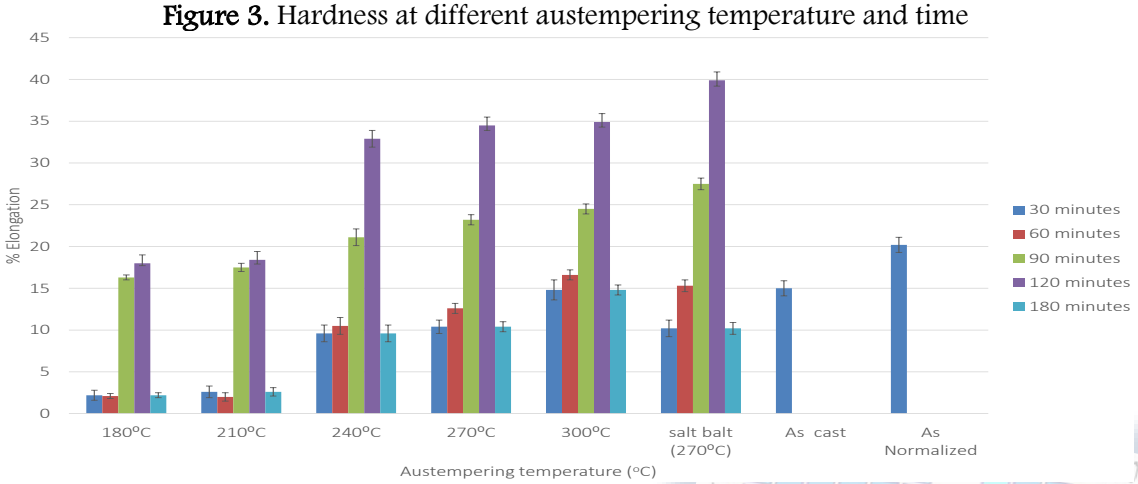

Figure 4. Percentage elongation at different austempering temperature and time

A B

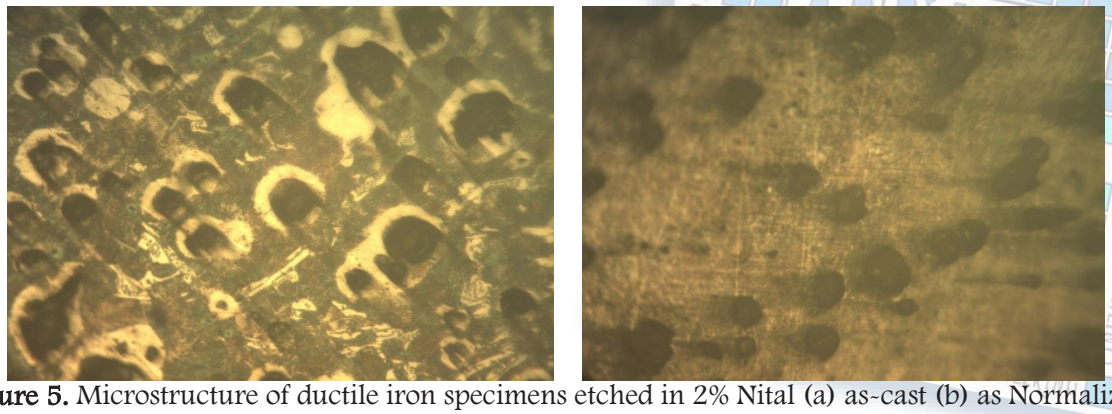

Figure 5. Microstructure of ductile iron specimens etched in 2% Nital (a) as-cast (b) as Normalized

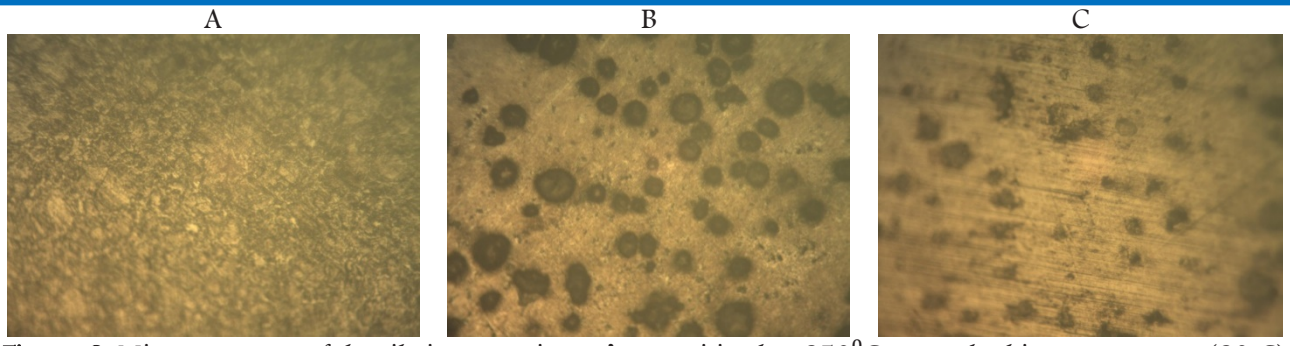

Figure 6. Microstructure of ductile iron specimens' austenitized at 850^{0}C, quenched in warm water (80°C) austempered at 180^{0}C, etched in 2% Nital (a) 30 minutes; (b) 90 minutes; (c) 150 minutes. The structure consists of nodules of graphite (dark balls) and retained austenite (bright constituent) in ausferrite (x200).

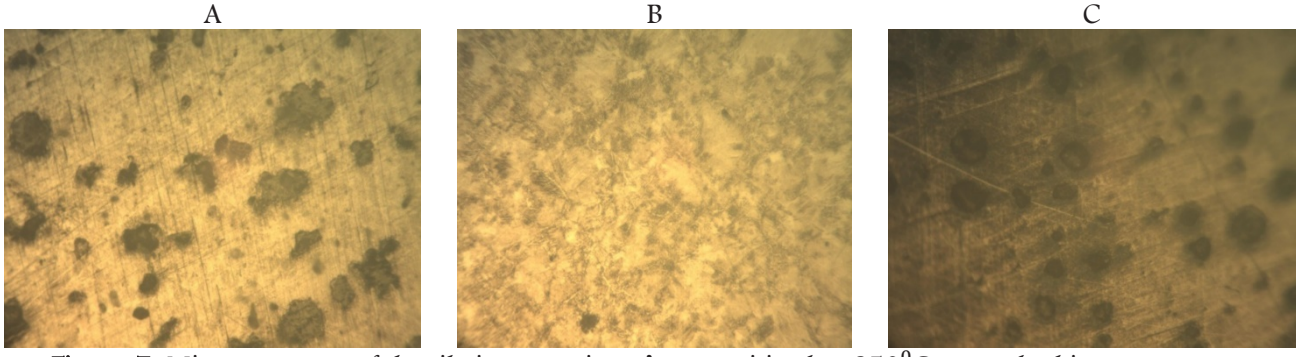

Figure 7. Microstructure of ductile iron specimen's austenitized at 850^{0}C, quenched in warm water (80^{0}C), austempered at 270^{0}C, etched in 2% Nital (a) 30 minutes; (b) 90 minutes; (c) 150 minutes. The structure consists of nodules of graphite (dark balls) and retained austenite (bright constituent) in ausferrite (x200)

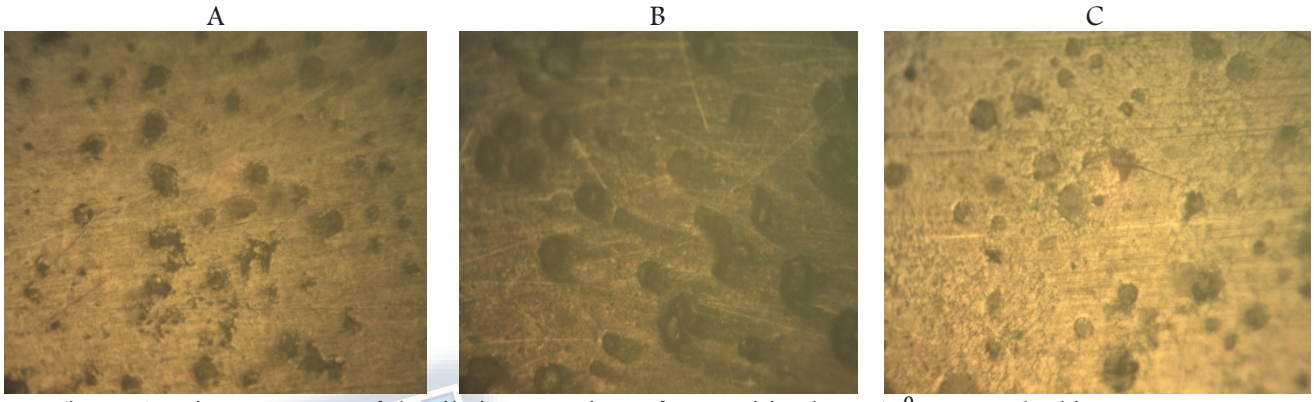

Figure 8. Microstructure of ductile iron specimens' austenitized at 850^{0}C, quenched in warm water (80^{0}C), austempered at 300^{0}C, etched in 2% Nital (a) 30 minutes; (b) 90minutes; (c) 150 minutes. The structure consists of nodules of graphite (dark balls) and retained austenite (bright constituent) in ausferrite (x200)

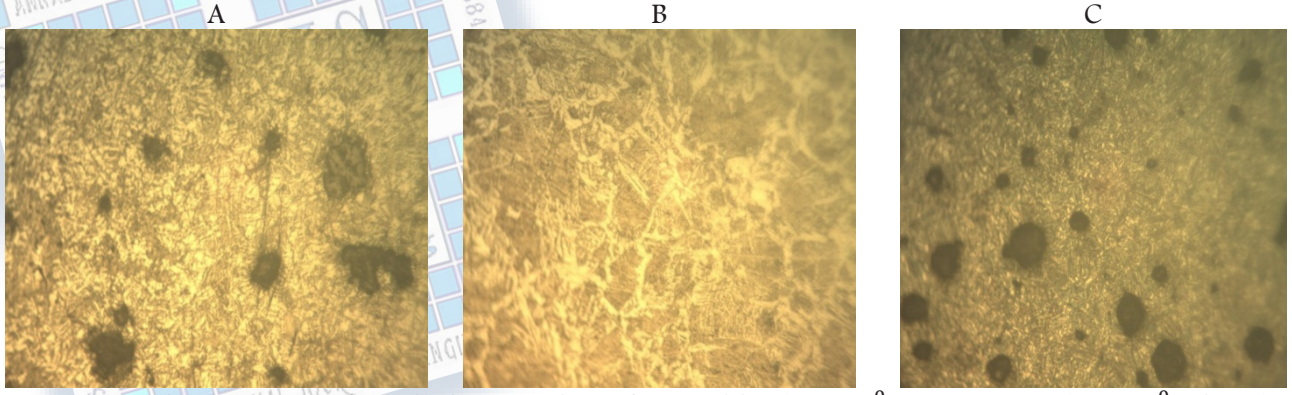

Figure 9. Microstructure of ductile iron specimens' austenitized at 850^{0}C, austempered at 270^{0}C, in salt bath, etched in 2% Nital (a) 30 minutes; (b) 90 minutes; (c) 150 minutes. The structure consists of nodules of graphite (dark balls) and retained austenite (bright constituent) in ausferrite (x200)

4. DISCUSSION

The as-cast structure of ductile iron revealed nodules of graphite, pearlite and ferrite when etched in 2% Nital which is shown in Figure 5. The microstructure is in conformity with the conventional structure of a ductile iron. It is well known that the important microstructural features of austempered ductile iron are the morphology of the ferrite, the volume fraction of retained austenite, the carbon content in retained austenite, and the presence or absence of carbide in austenite or ferrite. A mixture of bainitic ferrite and retained austenite, along with graphite nodules, is the most desirable combination of phases in these cast irons. Undesirable phases, such as martensite and iron carbides, may also be present in smaller quantities, but it is understood that the high volume fraction of retained austenite is very important towards the achievement of optimum combination of mechanical properties (Kovacs, 1987). Thus, the mechanical properties of austempered ductile iron may be related to three microstructural variables: bainite morphology, austenite volume fraction, formation of martensite and the graphite nodule (Blackmore and Hardings, 1984; Putatunda and Gadicherla, 2000). During austempering ductile iron undergoes two-stage transformation process. In stage I, the austenite (γ) decomposes into ferrite (α) and high carbon austenite (γHc)

$$\gamma \rightarrow \alpha + \gamma(Hc) \text{ (stage I)}$$

If the sample is held at the austempering temperature for too long, then a next reaction (stage II) takes place where high carbon austenite further decomposes into ferrite and carbide:

$$\gamma(Hc) \rightarrow \alpha + \text{carbide (stage II)}$$

The product of this second reaction is undesirable because it embrittles the material and degrades the mechanical properties. (Eric et al., 2004).

The effect of austempering temperature and time on the microstructure of austempered ductile iron is shown in Figures 5, 6, 7, 8, 9. When austempered at 180^{0}C for 30 minutes to 2 hours 30 minute. At 30 minutes high hardness, moderate ultimate tensile strength and yield strength were obtained. There was little increase in the hardness and yield strength but decrease in the ultimate tensile strength when austempered at 60 minutes. As the austempering time increases to 90 minutes there is reduction in the hardness obtained but increase in the ultimate tensile strength and yield strength. At 120 minutes there is reduction in the ultimate tensile strength and hardness but increased yield strength when compared with the result obtained at 90 minutes. Austempering for 150 minutes shows reduction in ultimate tensile strength, yield strength and hardness. Austempering at 210^{0}C for 30 minutes to 150 minutes, the result obtained shows good ultimate tensile strength, yield strength and hardness. At 60 minutes of austempering there is little increase in hardness value obtained and yield strength but decrease in the value of ultimate tensile strength. At 90 minutes higher values of ultimate tensile strength and yield strength was obtained but there is reduction in the hardness value. For 120 minutes there is drastic reduction in hardness, ultimate tensile strength and yield strength.

Austempering at 150 minutes brings reduction in the ultimate tensile strength, and yield strength but increased hardness. Austempering temperature at 240^{0}C, the result obtained shows improved ultimate tensile strength and yield strength but reduction in hardness value. At austempering time between 30 minutes to 90 minutes the ultimate tensile strength and yield strength continues to increase as hardness value continue to decrease. At 120 minutes of holding time there is increase in the ultimate tensile strength but decrease in the yield strength and hardness. At 270^{0}C of austempering temperature, higher values of ultimate tensile strength and yield strength was obtained but reduction in hardness value. From 30 minutes to 90 minutes, increase in ultimate tensile strength and yield strength continues at the same time reduction in the hardness values. At 120 minutes there is decrease in ultimate tensile strength and the yield strength but increased hardness. At 150 minutes, shows a little increase in ultimate tensile strength and yield strength and reduction in hardness.

Austempering at 300^{0}C, the highest value of ultimate tensile strength and yield strength was obtained and moderate values of hardness. The ultimate tensile strength and yield continues to increase until 90 minutes where the highest values of ultimate tensile strength and yield strength was obtained but reduction in the hardness value. At 120 minutes reduction in ultimate tensile strength, yield strength and hardness are observed while at 150 minutes, reduction in ultimate tensile strength and yield strength but increased hardness. This trend is in conformity with the report of (Gur et al, 2008; Rao and Putatunda, 1997) Austempering for short time (30 minute)

produced long continuous martensite paths in the eutectic cell boundaries Figures 5, 6, 7, 8, 9. As the austempering time increases up to 60 minutes, the amount of martensite derived from low carbon austenite decreases. In all the series, increment in the austempering time cause bainitic ferrite and high carbon austenite to displace martensite. After approximately 120 minutes austempering time, martensite almost disappeared. The results show that an austempering time between 60 minutes to 90 minutes at 300^{0}C is well within the process window. The relatively low ultimate tensile strength, yield strength and high hardness values at short austempering durations are attributed with martensite which forms in low carbon unreacted austenite areas during cooling from austempering temperature to room temperature. Increasing austempering time causes more ductile ausferritic structure to displaced hard martensite (Gur et al, 2008).

The variation in austempering temperature shows variation in the values of the results obtained. As the austempering temperature increases from 180^{0}C to 300^{0}C there is increase in the ultimate tensile strength and yield strength but decrease in hardness. This is as a result of percentage of volume fraction of high carbon retained austenite and volume fraction of the martensite plate in the structure. At 180^{0}C there is high volume fraction of martensite plate and low volume of high carbon retained austenite, this result in high hardness and low ultimate tensile strength and yield strength. As the temperature increases the volume fraction of high carbon retained austenite increases, this led to increasing ultimate tensile strength, yield strength and decreasing hardness value.

Results obtained show that at austempering temperature of 270^{0}C the results of the interrupted quenching in warm water as compared with the results obtained from conventional salt bath shows better hardness, ultimate tensile strength and yield strength. The result in Figure 4 shows that the ductility of the test samples increases with austempering temperature and holding time (Rao and Putatunda, 1997). The highest percentage of elongation was obtained at austempering temperature of 300^{0}C when held for 150 minutes. The higher the austempering temperature and holding time the higher the ductility of the material.

5. CONCLUSION

Interrupted quenching process, using warm water, was found to be very suitable for the production of fine structures, such as lath martensite, bainite, ausferrite and fine pearlite, in ductile irons. The process is simple, safe, convenient and cost effective. Other merits of the process include its capability for application, in place of more expensive molten salt, in small scale ductile iron production set up, for production of fine structures in large ductile iron components

REFERENCES

[1.] Blackmore P.A and Harding R.A.:The effects of metallurgical process variables on the Properties of Austempered Ductile Irons,Journal of Heat Treating, Volume 3, Issue 4, 310-325, 1984.

[2.] Eric .O, Jovanovic M.T; Slidjanin L. and Ragnovic D.: Microstructure and Mechanical Properties of Cu-Ni-Mo Austempered Ductile Iron, Journal of Mining and Metallurgy, 40B (1), 11-19, 2004.

[3.] Gur Hakan C, Kilicli Vulcan and Erdongan Mehmet: Investigating the Austempered Parameters of Ductile Iron by Magnetic Barkhausen Noise Technique, 17th World Conference on Non-destructive Testing Shanghai China, 2008.

[4.] Isah, L.A. and S.B. Hassan: Suitability of Khaya Senegalensis Seed Oil as a Quenchant in Austempering Heat Treatment of Ductile Iron, Journal of Metallurgy and Materials Engineering, 4(2), 38-43, 2009.

[5.] Keough, J.R.: Austempered Ductile Iron, http://w.w.w.ductile.org/didata/section/4intro.1998.

[6.] Kovacs B.V.: Austempered Ductile Iron: Fact and Fiction" Modern Casting,pp.38- 41, 1990.

[7.] Larry, O.: Proper Quenching Options Yields, Modern Application News, 2000.

[8.] Mark Ihm: TRW Automotive, Introduction to Gray Cast Iron (brake rotor) Metallurgy Pp49-53, 2000.

[9.] Oyetunji A. and Barnabas A.A: Development of martempered Ductile Iron by Step-Quenching Method in Warm Water: Journal of Emerging Trends in Engineering and Applied Sciences 3 (3), 470-474, 2012.

[10.] Putatunda S. K and Gadicherla P. V.: Effect of Austempering Time on Mechanical Properties of a Low Manganese Austempered Ductile Iron, Department of Chemical Engineering and Materials Science, College of Engineering, Wayne State University, Detroit, Journal of Materials Engineering and Performance, vol. 9, 193-203, 2000.

[11.] Rao P. P and Putatunda S. K.: Influence of Microstructure on Fracture Toughness of Austempered Ductile Iron, Department of Chemical Engineering and Materials Science, College of Engineering, Wayne State University, Detroit, Metallurgical and Materials Transactions A, vol. 28A, 1457-147,1997.