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MODELLING THE HARDNESS PROPERTY OF PRODUCED MARTEMPERED DUCTILE IRON THROUGH INTERRUPTED QUENCHING METHOD

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Abstract: In this research, modelling the hardness property of produced Martempered Ductile Iron (MDI) through interrupted quenching method was studied. Martempered ductile iron was produced by the adoption of interrupted quenching in warm water (cheap, convenient and safe to operate – compared to the conventional method in salt bath furnace). The product (MDI) was found to possess unique high hardness values of 52.9Rc at a low tempering temperature and holding time having a predominantly martensite, graphite nodules enveloped with ferrite shells in its microstructures. A computer model was developed to predict the hardness values of the developed MDI and a model equation, $Y = 62.504 - 0.079T$ (^oC) $- 0.054t$ (minutes) was established, with a good degree of correlation and reliability for the hardness characterization. The model was tested using statistical tools and manual, it was found to compare favourably well with the experimental results.
Keywords: Modeling, Martempered Ductile Iron, hardness, tempering temperature, statistical tools

1. INTRODUCTION

Ductile irons, also known as spheriodised or Nodular cast irons, has the structure of graphite nodules in a matrix which may be ferritic, pearlitic or ferritic-pearlitic (ductile grades), bainitic (strong and tough grades) or martensitic (strong and hard grades). The variations in the matrix structure have caused the mechanical properties of ductile cast irons to vary widely and have made them applicable in a wide range of industrial applications, especially in the areas requiring high strength, high toughness and high ductility. Ductile iron has replaced malleable cast iron in area of applications because of its superior mechanical properties, ease of production and good economy.

The conventionally used ADI has 3.6%C, 2.5%Si, 0.3%Mn, and 0.05% Mg chemistry with other additives in lean amount. Branka et al, (2004) and Hassan et al, (2000) reported of the primary purpose of adding Cu, Ni, or Mo to ADI is to increase the hardenability of the matrix so as to ensure the prevention of pearlite formation during the austempering process. It's versatile use will continue to expand as the design engineers continue to improve on these properties. An understanding of the properties of materials is essential in both the design and applications in any engineering project if it is to prove satisfactory for its intended purpose (Neil, and Ravindra, 1996). The major users of ductile iron components include the automobile and machine tools industry. (Hemanth , 2001).

Unit cell model (UCM) calculations are widely used to predict macro-scale constitutive behavior of composites (Gurson, 1977), which was later improved upon by Tvergaard and Needleman (1984) and it is usually referenced as GTN model. A number of authors, Dong et al, (1997); Berdin et al, (2001) proposed modeling the mechanical behavior of cast irons using GTN model. The model accounts for the nucleation, growth and coalescence of microvoids by means of the void volume fraction, which is the average measure of a void matrix aggregate. Recent contributions in the field are due to Pirondi et al, (2006), Bonora et al, (2006), Horstemeyer (2007) and Zairi et al, (2008) who proposed two different micro mechanical models based on porosity evolution. However, not much work has been carried out on modeling of mechanical properties of ductile irons heat-treated by interrupted cooling over a wide range of temperatures.

This study, therefore, focuses on modeling the hardness property of produced martempered ductile iron through interrupted quenching method for theoretical determination of this property. This will rapidly increase the determination of this property using the experimental way which consumes time, laborious and money consuming. This will also lead to reduction in production cost of the MDI since testing is an integral part of production cost.

2. MATERIALS, EQUIPMENT AND METHODS

All ductile iron test specimens, used for this research work, were cast in Madison, USA. The test specimens were cut into 20 mm length by 20 mm breadth. Normal materials for hardness test specimen preparation, such as abrasive papers, polishing cloth, and tissue paper and desiccators, were adopted.

Table 1: Chemical Compositions of the Ductile Iron

C	Si	Mn	S	P	Fe
3.6	2.0	0.3	0.01	0.04	Rest

A mass spectrometric analyzer was used to determine the chemical composition of the samples which is as shown in Table 1. Two heat treatment furnaces (Vecstar and Carbolite) were used, one for the austenitization of specimens at 850^0C for 30 minutes to annul the mechanical history and to promote homogeneity; and the other for austenite transformation at selected lower temperatures (between 250^oC – 450^oC) over a holding period of 30 – 180 minutes. An electric oven was used for austenite transformation at very low temperatures (as low as 170^oC) while the quenchant used was maintained at 80^0C. The hardness value of the treated materials was conducted and evaluated using a digital Rockwell hardness testing machine with an indentation load of 150KN and a daheng software driven optical microscope was used to analyze the microstructure of the developed martempered ductile iron (Oyetunji and Barnabas, 2012). Model was then developed for the hardness property of the developed MDI using SPSS (Statistical Package for Social Science) package. The equation developed is as shown in equation 1.

$$Y=62.504-0.079T(^oC)-0.054t \text{ (minutes)} \quad (1)$$

where T is Temperature () and t is the time in minutes.

3. DATA GENERATION AND VALIDATION OF DEVELOPED MODELS

The hardness property observed from experimental result and chemical compositions with holding times and temperatures were used to generate data. This was used to develop the model using ANOVA and the mathematical equation shown in equation 2 was established as model equation.

Physical relation and statistical tests were used to validate the model developed. Physical relation was carried out on the experimental and numerical data of hardness property and their differences were taken. The experimental and numerical data of hardness property were subjected to the following statistical tests: Paired t-test; Correlation Coefficient; and Standard Error of Prediction in accordance with Oyetunji and Adebayo, 2009.

i. Pair t-tests

The pair t- test technique uses the following nomenclatures:

d = Pair difference between the experimental and numerical data on each column; $\bar{d}$ = Mean Pair difference; Var (d) = Variance of Pair difference; Var($\bar{d}$) = Variance of mean difference; t_v = Theoretical number of degrees of freedom available for the estimation of the variance; that is;. (n-1); n = Number of paired test samples; α = Confidence interval (%); Sd = Standard deviation; The paired t-test will be evaluated by using the following expressions (Oyetunji, 2007) and (Oyetunji, 2010).

(a) Mean Pair difference ($\bar{d}$)

$$(\bar{d}) = \sum \frac{(Numerical\,data - Experimental\,data)}{No.of\ paired\ test\ samples} \tag{1}$$

(b) Variance of Pair difference

$$Var(d) = \frac{\sum d^2 - \frac{(Numerical\ data - Experiment\,al\ data)^2}{No.of\ Paired\ test\ samples}}{\text{No of paired test samples - 1}} \tag{2}$$

(c) Variance of mean of pair difference Var ($\bar{d}$)

$$Var(\bar{d}) = \frac{Var(d)}{No\,of\ paired\ test\ samples} \tag{3}$$

(d) Standard deviation of mean of pair difference (sd)

$$Sd = \sqrt{Var(\bar{d})} \tag{4}$$

(e) Acceptable tolerance interval for a given confidence interval and numerical data

$$= \bar{d} + t_{v(1-\alpha)}\,Sd \tag{5}$$

The result of the test was presented in Table 4.

ii. Correlation Analysis and Coefficient of Correlation

The following relations from Karl Pearson's coefficient of correlation (equations 6 and 7) were used to determine simple correlation:

(i) Karl Pearson's coefficient of correlation

$$P_{x,y} = \frac{C_{OV}(X,Y)}{\partial x\,\partial y} \tag{6}$$

where: $-1 \leq Pxy \leq 1$ and

$$Cov(x,y) = \frac{1}{n}\sum_{j=1}^{n}(x-\bar{x})(y-\bar{y}) \tag{7}$$

where: Cov (x,y) = Covariance of X and Y; ∂x = Variance of X; ∂y = Variance of Y; Pxy = Correlation Coefficient; $\bar{x}$ = Mean of X; $\bar{Y}$ = Mean of Y; X = Experimental value; Y = Numerical value; N = No. of pair test samples

(ii) Pearson's rank correlation coefficient

$$R = 1 - \frac{6\sum d^2}{n(n^2-1)} \tag{8}$$

(iii) Coefficient correlation

$$R = \sqrt{1 - \frac{\sum(Y_a - Y_c)^2}{\sum(Y_a - \bar{Y})^2}} \tag{9}$$

This is a statistical technique that is used to test for the relationship between two variables. The variables are said to be correlated when an increase or decrease in one variable is accompanied by an increase or decrease in the other. It is commonly used along with the regression analysis to measure how well the regression lines explain the variations of the dependent variable. If an increase in one variable corresponds to an increase in the other, the correlation is said to be positive. But if increase in one corresponds to decrease in the other, the correlation is referred to as negative. If there is no relationship between the two variables, they are said to be independent (Oyetunji, 2010).

iii. Standard Error of Prediction

This is the deviation of the predicted value from the observed value. It is given by:

$$E_{YX} = \sqrt{\frac{\sum(Y - Yr)^2}{n}} \tag{10}$$

where: Y = the actual value (Observed) and Yr = the predicted value (Oyetunji, 2010).

The Karl Pearson's equation for calculating the standard error of the predicted numerical data (Y) is given as:

$$SEyx = \sqrt{\left[\frac{1}{n(n-2)}\right] n\sum y^2 \left(\sum Y\right)^2 - \frac{[nExy - (Ex)(Ey)]^2}{nEx^2 - (Ex)^2}} \tag{11}$$

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where Y = Numerical data; X = Experimental data; and n = No. of pair test samples.

4. RESULTS

The validation results were shown in Tables 2, 3, and 4.

Table 2: Pair-t Test Analysis that Determine the Acceptable Confidence Interval of the model

Time (Min).	Pair difference (d)	Square of pair difference (d^2)	Mean of pair difference (đ)	Variance of pair difference Var (d)	Mean of variance of pair difference Var (đ)	Standard deviation (Sd)	Confidence Interval (α)	Degree of freedom t_v	Acceptable Interval (đ + $t_{v(1-\alpha)}$ Sd)
30	-4.341	56.089	-0.3617	4.95631	0.41304	0.64267	94.5%	2.20	1.05 -1.78
60	3.278	57.717	0.2732	5.1656	0.4305	0.6561	94.5%	2.20	1.71 - 1.2
90	7.028	75.06648	0.585667	6.4500	0.5375	0.73314	94.5%	2.20	2.2 - 0.9
120	2.088	67.50206	0.174	6.10352	0.5086	0.7132	94.5%	2.20	1.74 - 1.4
150	3.098	95.663	0.25816	8.6239	0.7186	0.8477	94.5%	2.20	2.14 - 1.6

The results for the Correlation Coefficient and Standard Error tests are contained in Table 5 from which the:

i) Correlation coefficient ranged from 0.9595 to 0.9925, which has been found to be in the very high correlation range of 0.9 to 1.0 (Oyetunji and Adebayo, 2010).
ii) Standard error test was found to be between 2.16 and 2.82, which is very low and which signifies that the modeling equation had adequately and accurate predict the hardness value of MDI.

Table 3: Correlation Coefficient and Standard Error Analysis Tests for the Model

Holding Periods (Minutes)	Correlation coefficient of the Model	Standard error of the Model
30	0.987809	2.162
60	0.9925	2.193
90	0.9595	2.501
120	0.98296	2.823
150	0.98296	2.823

Hardness values of ductile cast irons subjected to interrupted quenching in warm water prior to holding at various tempering temperatures and times as shown in Table 4.

Table 4: Hardness value (Experimental Value of Ductile iron subjected to interrupted quenching in warm water)

Transformation temperatures (°C)	Hardness values (R_c) for various holding times (minutes) transformation temperatures					
	30	60	90	120	150	180
175	52.9	51	48.8	47.2	45.5	42.9
200	46.0	42.9	39.8	40.7	37.3	33.6
225	43.8	40.1	39.0	36.7	34.5	32.9
250	42.2	38.7	35.9	33.6	30.7	28.9
275	35.9	35.4	33.0	32.1	29.8	27.3
300	38.7	36.5	31.7	30.8	28.6	26.9
325	32.7	31.0	30.1	29.9	28.0	27.2
350	33.6	29.7	28.6	27.4	26.7	26.2
375	31.8	31.0	29.7	28.2	27.6	27.7
400	37.8	27.2	26.1	26.0	25.9	25.2
425	26.6	23.6	23.1	21.2	20.5	19.6

Table 5: Calculated Data From The Model Developed

Transformation temperatures (°C)	Hardness values (R_c) for various holding times (minutes) transformation temperatures					
	30	60	90	120	150	180
175	52.9	51	48.8	47.2	45.5	42.9
200	46.0	42.9	39.8	40.7	37.3	33.6
225	43.8	40.1	39.0	36.7	34.5	32.9
250	42.2	38.7	35.9	33.6	30.7	28.9
275	35.9	35.4	33.0	32.1	29.8	27.3
300	38.7	36.5	31.7	30.8	28.6	26.9
325	32.7	31.0	30.1	29.9	28.0	27.2
350	33.6	29.7	28.6	27.4	26.7	26.2
375	31.8	31.0	29.7	28.2	27.6	27.7
400	37.8	27.2	26.1	26.0	25.9	25.2
425	26.6	23.6	23.1	21.2	20.5	19.6

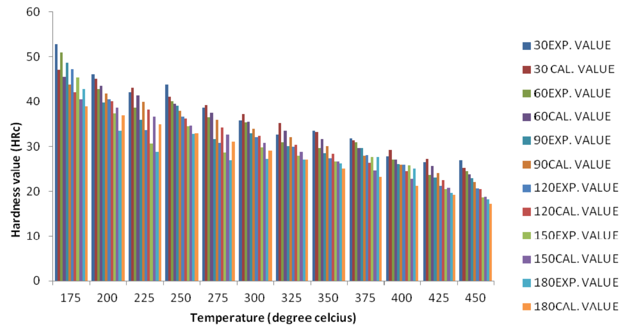


Fig. 1: Variation of Hardness Values with Immersion Temperature of Ductile Iron

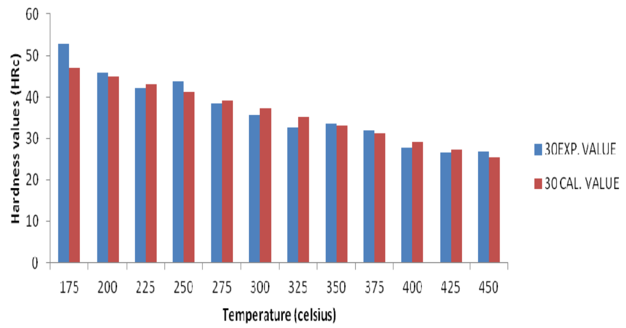


Fig. 2 : Variation of Hardness Values with Temperature for both Experimental (Exp.) and Calculated (CAL.) values

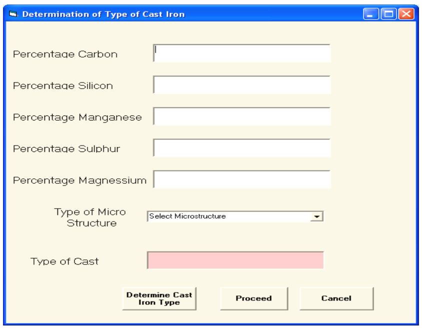
5. COMPUTER AID SOFTWARE DEVELOPMENT

The computer software developed for predicting hardness values of heat treated ductile irons was designed according to the following phases:

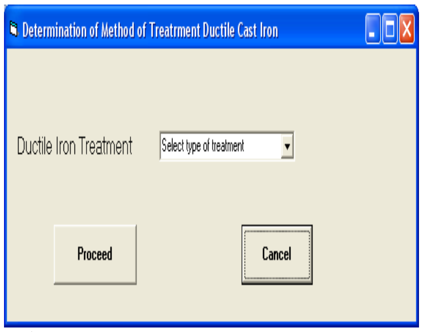
Phase 1: Title Phase Menu

This phase welcomes the user to the system as shown Phase 1 Menu. It contains information about the name of the software, the developer, the supervisor, the essence of the study and the department in which the work was carried.

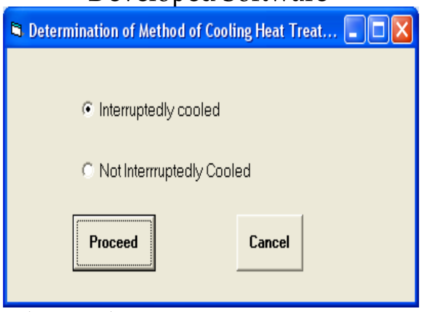
Phase 1: Title Phase Menu for the Developed Software



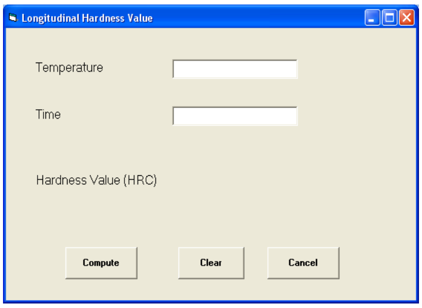
Phase 2: Material Identification Phase



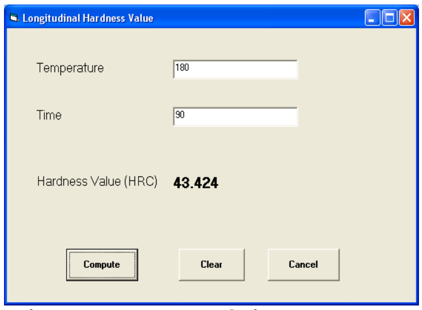
Phase 3(a): Heat Treatment Menu – I



Phase 3(b): Heat Treatment Menu – II



Phase 4: Entering the input Variables of Holding Temperature (°C) and Time (minutes)



Phase 5: Property Selection Menu

Phase 2: Data Entry Menu

In this phase, data necessary for computation or determination of an output is entered. Such data include material specification and chemical composition as in menu. This also includes material identification such as gray cast iron, or steel. However this software is only applicable to ductile irons and will run only for this. In addition the chemical composition input must match that for the material specified otherwise the programme will not proceed. There are some other intermediate phases to be run before reaching this menu.

Phase 3: Heat Treatment Menu

After this, the property to be evaluated is selected by entering the variable inputs for the determination of the hardness values of ductile iron of known composition after being subjected to a given heat treatment.

Phase 5: Property Selection Menu

These concerns the output property desired to be computed using the model, which is hardness value in this exercise. Once selected, the output or report phase is reached.

Phase 6: Computation/Report Menu.

This menu gives the final answer, which is the required hardness value for a ductile iron subjected to a holding temperature of 180°C for 90 minutes after being quenched in warm water for 40 seconds. In this computation the hardness value is 43.424Rc and the structure will be martensite. This is contained in Property selection men shown in Phase 5

Computer Requirements

The computer hardware that can used for the access and execution of the software should be of the following specifications:

i. Type: Personal Computer
ii. Pentium 2 and above or Laptop with at least 64 MB RAM,
iii. Microsoft Windows 98/NT/2000/XP and
iv. 64 MB and 512 MB hard disk free space.
v. The program has been tested and validated runs on computer systems of higher grade like Pentium-3 and Pentium-4

6. CONCLUSIONS

Investigation of the suitability of warm water for interrupted quenching of ductile cast irons has been found to be successful. From the results, the following results were established:

a) Martempered ductile iron was produced by the adoption of interrupted quenching in warm water (cheap, convenient and safe to operate – compared to the conventional method in salt bath furnace). The product (MDI) was found to possess unique high hardness values of 52.9Rc at a low tempering temperature and holding time.

b) The microstructure developed is predominantly martensite, graphite nodules enveloped with ferrite shells.

c) A computer model was developed to predict hardness values of ductile irons subjected to heat treatment by this process. This resulted in:
   (i) establishment of model equation, $Y = 62.504 - 0.079T(^oC) - 0.054t(\text{minutes})$, with a good degree of correlation and reliability.
   (ii) development of a computer software for theoretical evaluation of hardness values.

References

[1.] Aderoba, A. (1995): Tools of Engineering Management: Engineering Management Project Besade Publishers Nigeria Limited, Akure- Nigeria. Pp. 65-67.
[2.] Berdin C, Dong, M.J and Prioul C (2001): Local approach of Damage and Fracture Toughness for nodular cast iron. Engineering Fracture Mechanics, 68, Pp 1107 – 1117
[3.] Bonora N and Ruggiero N (2005): Micromechanical Modeling of Ductile cast iron incorporating damage part I Ferritic Ductile cast iron. International Journal of Solid and Structures, 42, 5-6, 1401 -1424
[4.] Branka Bosnjak, Branko Radulovic (2004), Effect of austenitising temperature on austempering kinetics of Ni-Mo alloyed Ductile iron, Material in Technology. Vol.38 No6, Pp 307-312.
[5.] Donelan, P. (2000): Modelling Microstructural and Mechanical Properties of feritic ductile cast iron. Materials Science and Technology, vol .16, No.3,Pp261-269.
[6.] Gurson Tvergaard N (1997): Model for rate and temperature dependent Materials with Isotropic and Kinematic Hardening, Springer-Verlag, Pp 34-40
[7.] Hassan Hasirci,Mehmet Erdogan (2000): Effects of Alloying Elements (Cu and Ni) and tempering time on microstructure and mechanical properties in austempered spheroidal graphite cast irons. Journal of Science. Vol. 13, No.4, Ankara.
[8.] J. Hemanth. Effect of sub-zero (cryogenic) and water-cooling chilling on solidification and mechanical behavior of cast iron. Materials Science and Engineering. A. Vol 333A. 2001, Pp 60-66.
[9.] Neil, J. and Ravindra, K. D. (1996). Civil Engineering Materials: Macmillan Press Ltd, Hong Kong. P 187.
[10.] Mulholland M., Khraishi T., Shen. Y.l and Horstemeyer M (2006): Void Growth and Interraction experiments, Implications to the optimal Straining rate in Super- plastic Forming. International Journal of Plasticity 22, Pp 1728-1744
[11.] Oyetunji, A. and Adebayo Abdullah Olawale (2011): Development of Models for the Prediction of Mechanical Properties of Rolled Ribbed Medium Carbon Steel. Innovative Systems Design and Engineering. Vol 2, No 3. Pp. 208-236, www.iiste.org, ISSN 2222-1727 (Paper) ISSN 2222-2871.
[12.] Oyetunji, A. (2010). "Modelling the Impact Toughness of grey cast iron produced from Steel and CO_2 moulds". Journal of Applied Sciences Research, ISInet Publication 6(5), 393-400.
[13.] Oyetunji,A (2007): Modelling Mechanical properties of grey cast iron. Ph.D Thesis submitted to the Department of metallurgical and Materials Engineering, Federal University of Technology, Akure-Nigeria.
[14.] Ravishankar K.S,; Udupa, K. R and Prasad, R. P. (2008): Development of Austempered Ductile Iron for High Tensile Strength and Fracture Toughness by Two step Austempering Process, 68th World Foundry Congress, 7th-10th, Pp35-40.
[15.] Zairi F., Nait- Abdelaziz M, Gloaguen J.M., and Lefebvre J.M (2008): Modelling of the elasto-viscoplastic damage behavior of glassy polymers International Journal of Plasticity, 24, Pp 945-965.