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Predicted hardness of Austempered Vermicular Graphite Iron

A. Jakubus [a, *], **M.S. Soiński** [a], **P. Mierzwa** [b]
[a] Jakub from Paradyz Academy in Gorzow Wielkopolski,
52 Fryderyk Chopin Street, 66-400 Gorzów Wielkopolski, Poland
[b] Czestochowa University of Technology
* Correspondence contact: e-mail: jakubusaneta@wp.pl

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Abstract

The aim of this study was to determine the hardness of vermicular cast iron subjected to austempering, depending on the parameters of the heat treatment process. The heat treatment was conducted based on orthogonal experimental design, with a total of 27 experiments performed. The samples underwent austenitization at temperatures of 890°C, 925°C, and 960°C, followed by austempering at 290°C, 340°C, and 390°C. The austenitization and austempering times were set to 90 min, 120 min, and 150 min. To analyse the influence of these parameters, a full polynomial regression model was developed. The proposed model, which describes the hardness of the cast iron after heat treatment, showed a predicted coefficient of determination (R^2) of approximately 78%. For optimization purposes, the Response Surface Methodology (RSM) was employed. The results of the ANOVA analysis indicated that the austempering temperature (Tpi), the square of the austenitization time ($\tau\gamma^2$), the interaction between austenitization temperature and time ($T\gamma$ $\tau\gamma$), as well as the interaction between austenitization and austempering temperatures ($T\gamma$ Tpi) had the most significant impact on the examined parameter. Following variance analysis, the model was refined once more to eliminate insignificant predictors. The simplified model improved the predicted coefficient of determination to 93%. The optimal conditions for the analyzed parameters, assuming a maximum hardness of approximately 440 HB, were obtained under the following heat treatment conditions: $T\gamma$ = 930°C, Tpi = 290°C, $\tau\gamma$ = 150 min, and τpi = 150 min.

Keywords: Compacted graphite iron, Austempering, Austenitization, Vermicular cast iron, AVGI, RSM, Regression models, Hardness

1. Introduction

The production of various components made of vermicular cast iron, which operate under demanding conditions (e.g., engine camshaft sleeves, transmission gears), and undergo heat treatment, requires a thorough understanding of the structural transformations accompanying this process. Heat treatment significantly influences the mechanical properties of cast iron.

Typically, the industrial implementation of a new material, such as austempered vermicular cast iron (AVGI), needs extensive experimental testing. The results of these studies offer essential insight into the material's properties, the influence of heat treatment on microstructure, and the mechanical and plastic performance of the alloy, as well as its behaviour under operational conditions. Since such research is time-consuming and costly, the use of mathematical models to predict microstructure and material properties based on technological parameters is of particular importance.

In study [1], an analysis was conducted to investigate the effects of heat treatment parameters on the mechanical properties of vermicular cast iron, such as tensile strength, yield strength, and elongation. Polynomial regression, analysis of variance (ANOVA), and Response Surface Methodology (RSM) were used. The regression models enabled the prediction of tensile strength, elongation after stretching, and yield strength with accuracies of 82%, 80%, and 49%, respectively. It was determined that achieving the maximum tensile strength (940 MPa), yield strength (880 MPa), and elongation (0.7%) in the studied vermicular cast iron requires the following heat treatment parameters: T_{γ} = 890 °C; T_{pi} = 290 °C; τ_{γ} = 120 min; τ_{pi} = 150 min.

In study [2], the authors also employed statistical methods to predict the hardness of austempered ductile iron (ADI). The spheroidal graphite cast iron was austenitized at 950°C for 120 minutes, followed by austempering in a salt bath at 290°C, 320°C, 350°C, and 380°C for 30, 60, 90, and 120 minutes, respectively. The proposed mathematical model for predicting the hardness of austempered ductile iron achieved a 95.20% validation accuracy compared to experimental results.

Regression models are widely used for approximating values based on a dataset, allowing for a reduction in the number of required experiments. Many researchers have applied similar approaches to analyse various manufacturing processes [3–6]. For instance, in study [7], the authors used the Taguchi method to optimize melding parameters, including compaction, compaction time, and air pressure, and to examine the effects of these factors on material flowability.

In reference to the presented studies, it was deemed appropriate to conduct an analysis of the influence of selected heat treatment parameters on the hardness of vermicular cast iron, in a manner analogous to study [1]

2. Experimental Procedure

A detailed description of the investigated cast iron is provided in studies [8–10]. The ferritic-pearlitic vermicular cast iron (see Fig. 1) was subjected to heat treatment to obtain an ausferritic matrix. The process was conducted based on an orthogonal experimental design.

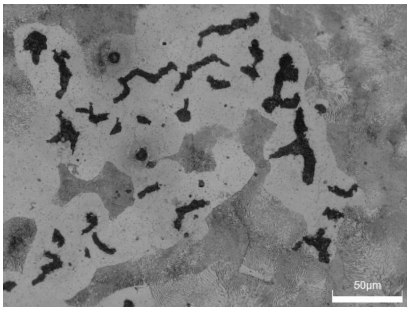


Fig. 1. Microstructure of vermicular cast iron, metallographic specimen etched with Nital

For the statistical analysis of the experimental data, Minitab 21.1 software (Pennsylvania State University, Pennsylvania, PA, USA) was used. Four independent variables were selected for the study, including austenitization and austempering temperatures, as well as austenitization and austempering times, within the following ranges:

— X_1 - austenitization temperature (Tγ) from 890°C to 960°C;
— X_2 - austempering temperature (Tpi) from 290°C to 390°C;
— X_3- austenitization time [min] (τγ) from 90 min to 150 min;
— X_4 - austempering time (τpi) from 90 min to 150 min.

A total of 27 experiments were conducted, as presented in Table 1. The Brinell hardness of the material was measured using a 5 mm diameter steel ball under a load of 7350 N. The hardness test results are presented in Figure 2, while Figure 3 illustrates representative microstructures of the investigated alloys, corresponding to the experimental conditions listed in Table 1.

Table 1.
Experimental design used to analyze the effect of heat treatment parameters on alloy hardness [1]

No	Heat treatment parameters			
	Austenitization temperature (Tγ) [°C]	**Austempering temperature (Tpi) [°C]**	**Austenitization time (τγ) [min]**	**Austempering time (τpi) [min]**
	X_1	**X_2**	**X_3**	**X_4**
1.	890	290	90	90
2.	960	290	90	90
3.	890	390	90	90
4.	960	390	90	90
5.	890	290	150	90
6.	960	290	150	90
7.	890	390	150	90
8.	960	390	150	90
9.	890	290	90	150
10.	960	290	90	150
11.	890	390	90	150
12.	960	390	90	150
13.	890	290	150	150
14.	960	290	150	150
15.	890	390	150	150
16.	960	390	150	150
17.	890	340	120	120
18.	960	340	120	120
19.	925	290	120	120
20.	925	390	120	120
21.	925	340	90	120
22.	925	340	150	120
23.	925	340	120	90
24.	925	340	120	150
25.	925	340	120	120
26.	925	340	120	120
27.	925	340	120	120

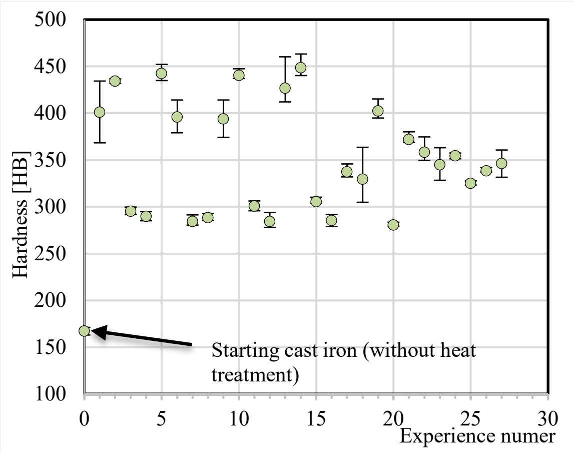
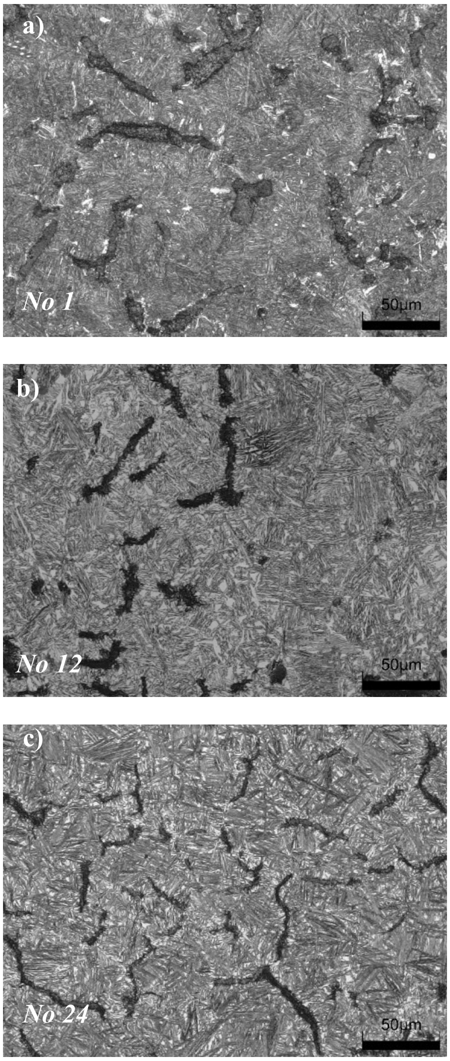


Fig. 2. Hardness of heat-treated vermicular cast iron classified according to the order of planned experiments



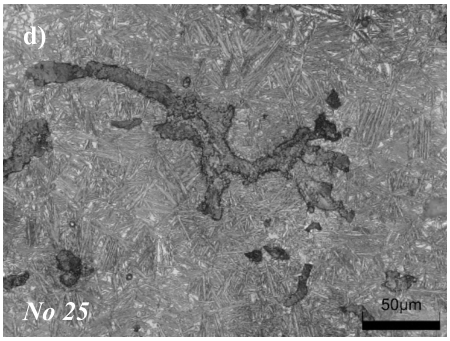


Fig. 3. Microstructures of vermicular graphite cast iron after heat treatment. Heat treatment parameters: a) Tγ=890ºC, Tpi=290ºC, τγ=90min ,τpi=90min, b) Tγ=960ºC, Tpi=390ºC, τγ=90min, τpi=150min; c) Tγ=925ºC, Tpi=340ºC, τγ=120min ,τpi=150min; d) Tγ=925ºC, Tpi=340ºC, τγ=120min ,τpi=120min. Etched with Nital

3. Influence of Heat Treatment Parameters on Hardness (HB)

The general ANOVA results for the main effects, squared main effects, and two-way interaction effects are presented in Table 2. The significant parameters at $\alpha = 0.2$ ($p < 0.2$) are the four interaction models. According to the established model, the most significant main effect is the austempering temperature (Tpi). For the squared main effects, the most significant parameter is the austenitization time ($\tau\gamma^2$), with a p-value of 0.028. Regarding the two-way interactions, the significance condition is satisfied in two cases (with p-values less than 0.2). Therefore, the significant interactions include the austenitization temperature and time (Tγ τγ) as well as the austenitization and austempering temperature (Tγ Tpi). The proposed overall model, which captures the relationship between the hardness of cast iron after heat treatment and the considered parameters (see Equation 1), exhibited a high coefficient of determination (R^2) of 97.09%, closely approaching the adjusted R^2 value of 93.7%, indicating the linearity of the regression model. The ability of the model to predict new observations is confirmed by the high value of the predicted coefficient of determination of 78.79%.

$$\begin{aligned} HB = &-7108 + 16{,}2\,X_1 + 2{,}66\,X_2 - 0{,}14\,X_3 - 4{,}58\,X_4 \\ &- 0{,}00798\,X_1{*}X_1 - 0{,}00111\,X_2{*}X_2 + 0{,}02469\,X_3{*}X_3 \\ &+ 0{,}00747\,X_4{*}X_4 - 0{,}00307\,X_1{*}X_2 - 0{,}00595\,X_1{*}X_3 \\ &+ 0{,}00298\,X_1{*}X_4 - 0{,}00208\,X_2{*}X_3 - 0{,}00100\,X_2{*}X_4 \\ &+ 0{,}00403\,X_3{*}X_4 \end{aligned} \tag{1}$$

For the multi-criteria optimization of heat treatment parameters and their effect on cast iron hardness, the Response Surface Methodology (RSM) was applied. Figure 4 presents the influence of austenitization and austempering temperatures and times on the hardness of cast iron. Each of the six plots in Figure 4 illustrates the interaction between two heat treatment parameters and their impact on the hardness of vermicular cast iron.

The influence of the most critical parameter in the discussed model, namely the austempering temperature, requires particular attention. The course of the curves presented in the accompanying graphs shows that as the austempering temperature (X_2) increases, hardness decreases. For instance, Figure 4a shows that at an austempering temperature (X_2) between 320 and 340°C, the resulting hardness is approximately 330–360 MPa.

Table 2.
Analysis of variations in the influence of thermal parameters on the hardness (HB) of vermicular cast iron

Source	***DF***	***Adj SS***	***Adj MS***	***F-Value***	***P-Value***
Model	*14*	*81092,9*	*5792,3*	*28,63*	*0,000*
Linear	*4*	*77087,8*	*19272,0*	*95,26*	*0,000*
X_1 - Austenitization temperature [°C] T_γ	*1*	*5,6*	*5,6*	*0,03*	*0,871*
X_2 - Austempering temperature [°C] T_{pi}	***1***	***76832,0***	***76832,0***	***379,76***	***0,000***
X_3 - Austenitization time [min] τ_γ	*1*	*43,6*	*43,6*	*0,22*	*0,651*
X_4 - Austempering time [min] τ_{pi}	*1*	*206,7*	*206,7*	*1,02*	*0,332*
Square	*4*	*2359,1*	*589,8*	*2,92*	*0,067*
*X_1^2 - Austenitization temperature [°C] * Austenitization temperature [°C] T_γ^2*	*1*	*245,8*	*245,8*	*1,22*	*0,292*
*X_2^2 - Austempering temperature [°C] * Austempering temperature [°C] T_{pi}^2*	*1*	*19,8*	*19,8*	*0,10*	*0,760*
X_3^2 - Austenitization time [min]* Austenitization time [min] τ_γ^2	*1*	*1269,8*	*1269,8*	*6,28*	*0,028*
*X_4^2 - Austempering time [min] * Austempering time [min] τ_{pi}^2*	*1*	*116,2*	*116,2*	*0,57*	*0,463*
2-Way Interaction	*6*	*1646,0*	*274,3*	*1,36*	*0,307*
X1*X2 - Austenitization temperature [°C] * Austempering temperature [°C] T_γ T_{pi}	***1***	***462,2***	***462,2***	***2,28***	***0,157***
X1*X3 - Austenitization temperature [°C] * Austenitization time [min] T_γ τ_γ	***1***	***625,0***	***625,0***	***3,09***	***0,104***
*X1*X4 - Austenitization temperature [°C] * Austempering time [min] T_γ τ_{pi}*	*1*	*156,3*	*156,3*	*0,77*	*0,397*
*X2*X3 - Austempering temperature [°C] * Austenitization time [min] T_{pi} τ_γ*	*1*	*156,2*	*156,2*	*0,77*	*0,397*
*X2*X4 - Austempering temperature [°C] * Austempering time [min] T_{pi} τ_{pi}*	*1*	*36,0*	*36,0*	*0,18*	*0,681*
*X3*X4 - Austenitization time [min] * Austempering time [min] τ_γ τ_{pi}*	*1*	*210,3*	*210,3*	*1,04*	*0,328*
Error	*12*	*2427,8*	*202,3*		
Lack-of-Fit	*10*	*2203,1*	*220,3*	*1,96*	*0,385*
Pure Error	*2*	*224,7*	*112,3*		
Total	*26*	*83520,7*			

DF = degree of Freedom; Adj SS = adjusted sums of squares; Adj MS = adjusted mean squares; F value is a value on the F distribution; p-value—p-value or test probability; VIF—variance inflation factor.

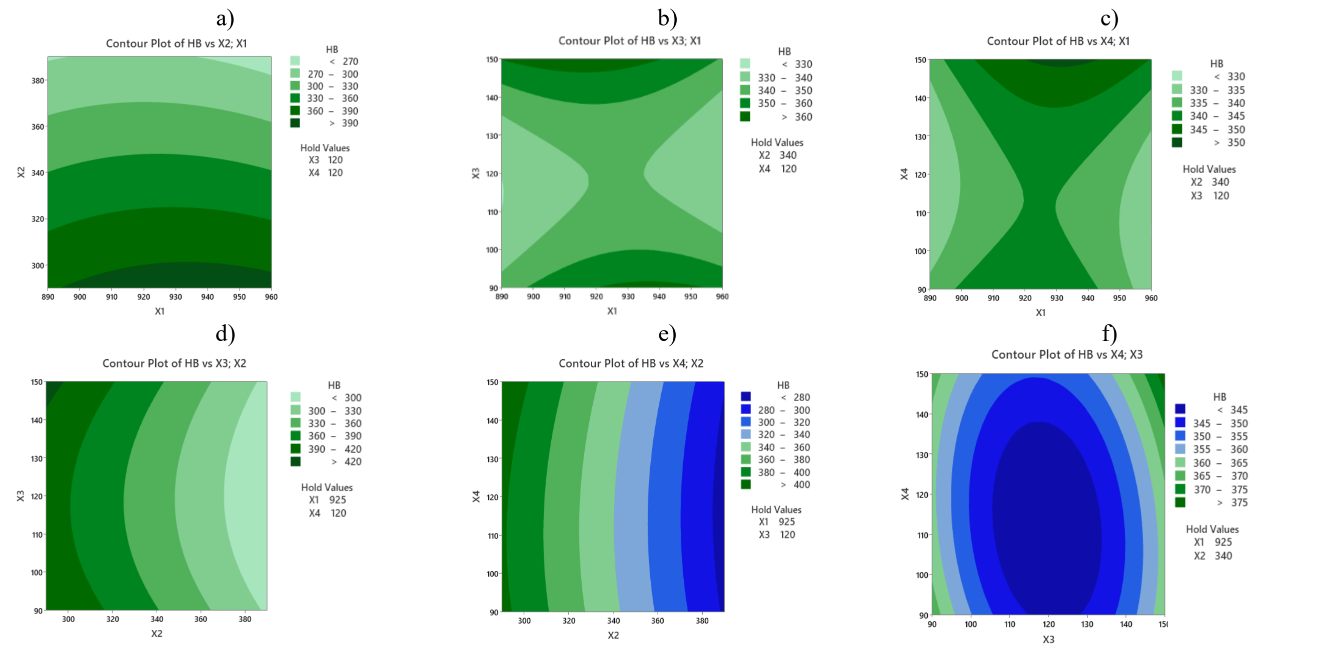


Fig. 4. The effect of heat treatment parameters on hardness is presented. The figure presents the relationships between the following heat treatment parameters: a) Tγ and Tpi; (b) Tγ and τγ; (c) Tγ and τpi; (d) Tpi and τγ; (e) Tγ and τpi; (f) τγ and τpi

The regression equation is considered significant if at least one of the model coefficients b1, b2,…bn is significantly different from zero and/or if the coefficient of determination (R-sq) is significantly different from zero. In the overall model, four variables are identified as significant predictors, indicating that the model can be simplified without a substantial loss in approximation quality. After analysing the significant variables, predictor selection was performed by removing non-significant predictors from the full model based on the F-test. The regression equation was determined using the backward elimination method. The proposed model, which is not overloaded with unnecessary predictors, is as follows:

$$HB = 339{,}33 - 65{,}33\,X_2 + 18{,}33\,X_3{*}X_3 - 5{,}37\,X_1{*}X_2 - 6{,}25\,X_1{*}X_3 \qquad (2)$$

The hardness of cast iron after heat treatment is influenced by parameters such as $\boldsymbol{T_{pi}}$, $\boldsymbol{\tau_{\gamma}^2}$, and the interactions $\boldsymbol{T_{\gamma}\,T_{pi}}$ and $\boldsymbol{T_{\gamma}\,\tau_{\gamma}}$. The squared correlation coefficient of the proposed simplified equation is 95.71%. The predicted coefficient of determination increased by approximately 1% to a value of 94.93%. Both the coefficient of determination and its adjusted value indicate the linearity of the proposed model. The developed function accounts for approximately 93% of the variability in the explanatory variable (HB), with only about 7% of the variability remaining unexplained. The proposed model exhibits a strong ability to predict new observations, achieving an accuracy of 93.13%, which represents an improvement of approximately 14% compared to the full model.

The boundary variables that reach the maximum of each response are indicated as "Cur." (see Fig. 5). The optimization objective was to maximize the hardness (HB). In the case of the material hardness data, the determined desirability is at a satisfactory level of 98%.

Analysing the influence of $\boldsymbol{T_{pi}}$, it can be observed that an increase in austempering temperature leads to a decrease in material hardness. In the case of the austenitizing temperature (Tγ), an increase results in a slight hardness increase up to approximately 930°C, after which hardness decreases with further temperature rise. The optimal austenitizing time (τ_{γ}), is approximately 150 minutes. A reduction in τ_{γ} leads to a decrease in HB. The maximum alloy hardness can be achieved with an austenitizing and austempering time of 150 minutes.

In conclusion, to obtain a cast iron alloy hardness of approx. 440 MPa, the heat treatment should be conducted with the following parameters: T_{γ} = 930°C; T_{pi} = 290°C; τ_{γ} = 150 min; and τ_{pi} = 150 min.

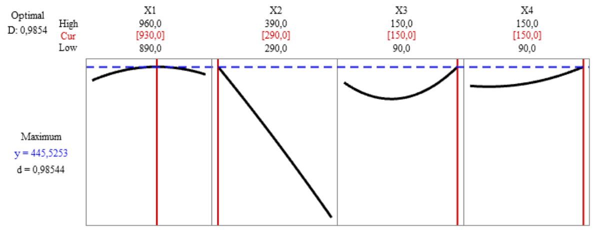


Fig. 5. Desirability function applied in multiple responses (Minitab 21.4.3.0 Statistical Software)

The graph (see Fig. 6) presents a comparison between the hardness of vermicular cast iron (HB) determined using the regression models and the corresponding experimental data.

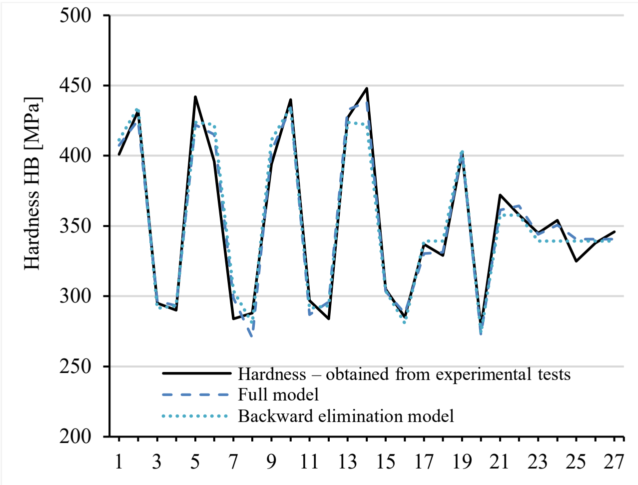


Fig. 6. Comparison of the obtained material hardness results with the proposed mathematical models

4. Summary

The objective of this study was to assess the potential for estimating the hardness of vermicular cast iron, based on the parameters of the heat treatment process, including austenitizing and austempering. The research was conducted using a factorial design approach. A central composite design was employed, and a total of 27 experiments (i.e., heat treatments with varying parameters, including 3 repetitions at the zero point) were performed. The derived mathematical model demonstrates that the primary factors influencing the hardness of vermicular cast iron after heat treatment include the austempering temperature, the square of the austenitizing time, the interaction between austenitizing and austempering temperatures, and the interaction between austenitizing temperature and time. The simplified regression model predicts the experimental results with an accuracy of approximately 93%. To achieve a vermicular cast iron hardness of approximately 440 MPa, the heat treatment should be conducted using the following parameters: Tγ = 930°C, Tpi = 290°C, τγ = 150 min, and τpi = 150 min.

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