The Heat Treatment Parameters Analysis Using Two-Way ANOVA Method

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Abstract
Analysis of variance (ANOVA) is the method used to compare continuous measurements to determine if the measurements are sampled from the same or different distributions. It is an analytical tool used to determine the significance of factors on measurements by looking at the relationship between a quantitative "response variable" and a proposed explanatory "factor". This method is similar to the process of comparing the statistical difference between two samples, in that it invokes the concept of hypothesis testing. The two-way analysis of variance (Two-way ANOVA) is an extension to the one-way analysis of variance. The two-way ANOVA compares the mean differences between groups that have been split on two independent variables (called factors). A major importance represents the studies about the studies that analyze important technological parameters of heat treatment processes with Two-way ANOVA method. The paper presents an example of calculation for two-way analysis of variance repeated measures applied for the results of harness properties of an austempered ductile iron. The major objective of this research was to analyze (with Two-way ANOVA method) the influence of two independent variables on the dependent variable in the case of two alternatives of heat treatment. The independent variables are: the temperature at isothermal level, T_{iz} (in our case that has 2levels: T_{iz}= 300 and 400°C) and the maintained time at the isothermal level, t_{iz} (that has four levels: t_{iz} = 30; 60; 90 and 120 min). All calculations were made for two alternatives of austenizing temperatures of heat treatment: T_A = 830 and 900°C. By studying all the data presented in this paper following remarkable conclusions: that the austenizing temperature, T_A= 900 °C is an important variable for our heat treatment while the austenizing temperature, T_A=830 °C is not an important factor for the values of hardness properties of the studied ductile cast iron.

Keywords
analysis of variance, two-way ANOVA, heat treatment, phase transformation, hardness

1. The Two-Way Analysis of Variance

The two-way analysis of variance (Two-way ANOVA) is an extension to the one-way analysis of variance. There are two independent variables (hence the name two-way). The two-way ANOVA compares the mean differences between groups that have been split on two independent variables (called factors). The primary purpose of a two-way ANOVA is to understand if there is an interaction between the two independent variables on the dependent variable [1, 2].

The interaction term in a two-way ANOVA informs you whether the effect of one of your independent variables on the dependent variable is the same for all values of other independent variable (and vice versa).

1.1. Assumptions

For this calculations, the assumptions are:

- the populations from which the samples were obtained must be normally or approximately normally distributed.
- the samples must be independent.
- the variances of the populations must be equal.
- the groups must have the same sample size.

1.2. Factors

The two independent variables in a two-way ANOVA are called factors. The idea is that there are two variables, factors, which affect the dependent variable. Each factor will have two or more levels within it, and the degrees of freedom for each factor is one less than the number of levels.

The interaction effect is the effect that one factor has on the other factor [3, 4]. The degree of freedom here is the product of the two degrees of freedom for each factor.

1.3. Source of variation

Two-way ANOVA divides the total variability among values into four components. Prism tabulates the percentage of the variability due to interaction between the row and column factor, the percentage due to the row factor, and the percentage due to the column factor. The remainder of the variation is among replicates (also called residual variation).

1.4. Within variation

The Within variation is the sum of squares within each analyzed group. The total number of analyzed groups is the product of the number of levels for each factor. The within variance is the within variation divided by its degrees of freedom.

2. The Steps of Two-Way Analysis of Variance

In this case, it use two factors [1, 2]. Factor A has 1, 2, ..., a levels. Factor B has 1, 2, ..., b levels. There are ab treatment combinations (or cells) in a complete factorial layout [4-8]. Assume that each treatment cell has r independent observations (known as replications).

Let A_i be the sum of all observations of level i of factor A, i=1,…,a. The A_i are the row sums.

Let B_j be the sum of all observations of level j of factor B, j=1,…,b. The B_j are the column sums.

Let $(AB)_{ij}$ be the sum of all observations of level i of A and level j of B. These are cell sums.

Let *r* be the number of replicates in the experiment; that is: the number of times each factorial treatment combination appears in the experiment.

Then the total number of observations for each level of factor A is *rb* and the total number of observations for each level of factor B is *ra* and the total number of observations for each interaction is *r*. Finally, the total number of observations *n* in the experiment is *abr*.

Solving Two-Way Analysis of Variance is done in the following steps [1, 2]:

1) Calculation of the Sum of squares for A factor, SS(A), (i.e. sum of the squared deviations from the mean) is done with the relationship (1):

$$SS(A) = r * b \sum_{i=1}^{a} (\bar{y}i - \bar{y})^2 \quad (1)$$

2) Calculation of the Sum of squares for B factor, SS(B), (i.e. sum of the squared deviations from the mean) is done with the relationship (2):

$$SS(B) = r * a \sum_{j=1}^{b} (\bar{y}i - \bar{y})^2 \quad (2)$$

3) Calculation of the Sum of squares for AB factors, SS(AB) is done with the relationship (3):

$$SS(AB) = r * \sum_{i=1}^{a} * \sum_{j=1}^{b} (\bar{y}ij - \bar{y}i - \bar{y}j + \bar{y})^2 \quad (3)$$

4) Calculation of the captures variability within each group SS(Within) is done with the relationship (4):

$$SS(Within) = \sum_{i=1}^{a} * \sum_{j=1}^{b} * \sum_{k=1}^{r} (\bar{y}ijk - \bar{y}ij)^2 \quad (4)$$

5) Calculation of the Total sum of squares, SS(Total), is done with the relationship (5):

$$SS(Total) = \sum_{i=1}^{a} * \sum_{j=1}^{b} * \sum_{k=1}^{r} (\bar{y}ijk - \bar{y})^2 \quad (5)$$

where:

SS(A) = Sum of squares for A factor (i.e. sum of the squared deviations from the mean);
SS(B) = Sum of squares for B factor (i.e. sum of the squared deviations from the mean);
SS (AB) = Sum of squares for AB factors (i.e. sum of the squared deviations from the mean);
AB = the interaction between A and B;

SS(Within) = captures variability within each group;
SS(Total) = Total sum of squares;
a = levels for A factor;
b = levels for B factor;
r = total number of observations for each interaction;
abr = *n* = the total number of observations;
Y = process performance;
$\bar{y}$ = average of process variable;
i = levels of A factor;
j = levels of B factor;
k = levels of interactions.

The ANOVA Summary Table for a Two-Way ANOVA (Source of Variation) is presented in table 1.

Table 1. The ANOVA Summary Table for a Two-Way ANOVA (Source of Variation)

ANOVA					
Source of Variation	df	SS	MS	F	P-value
Factor A Factor B AB interaction Within groups	df_A=a-1 df_B=b-1 df_{AB}=(a-1)(b-1) df_w=ab(r-1)	SS_A SS_B SS_{AB} SS_w	$MS_A=SS_A/df_A$ $MS_B=SS_B/df_B$ $MS_{AB}=SS_{AB}/df_{AB}$ $MS_w=SS_w/df_w$	$F=MS_A/MS_w$ $F=MS_B/MS_w$ $F=MS_{AB}/MS_w$	
Total	df_T=abr-1	SS_T			

where:
df = the degrees of freedom is equal to the sum of the individual degrees of freedom for each sample.
abr = n = the total number of observations;
SS = Sum of squares (i.e. sum of the squared deviations from the mean);
MS = Mean square;
F = the calculated value of the Fischer criteria;
P-value is determined from the F-ratio and the two values for degrees of freedom.

3. Materials

Over the last few years, a number of thermal processes have been developed to modify the matrix structure and thus the properties of ductile cast iron [9, 10].

Austempered Ductile Iron (A.D.I.) with a bainitic matrix, obtained by heat treatment and isothermal hardening is the material which combines a lot of superior attributes of the classical Austempered Ductile Irons or forged iron, being in a serious competition with the iron used by the moment in the automotive industry [10].

A major importance represents the studies about the bainitic S.G. cast irons obtained by heat treatment, especially the classical isothermal hardening and his variant, the Dual-Phase bainitic heat treatment, which is characteristic by a large and low A_s–A_f interval of temperatures.

Depending on heat treatment parameters, in these material components owing to changes in proportions of the major phases present in the microstructure: bainitic ferrite, high carbon austenite and graphite nodules. Martensite, ferrite, iron carbides and other alloy carbides may also be present [9].

Because the final structure obtained, influence property values, a wide range of properties can be obtained in these special material.

The studied cast iron had the following composition: 3.80 %C; 2.60 %Si; 0.45 %Mn; 0.005 %P; 0.001 %S; 0.40 %Cu; 0.18 %Cr; 0.072 %Mg.

This cast iron was elaborated in an induction furnace. Nodular changes were obtained with the "In mold" method, with the help of prealloy FeSiCuMg with 10-16%Mg, added into the reaction chamber in a proportion of 1.1% of the treated cast iron.

The structure in raw state is perlito-feritic typical for a cast iron with geometrically regular nodular form. The casted raw iron had the following mechanical properties: Rm = 645 N/mm², HB = 176, KC = 11 J/cm², A = 13 %.

4. Heat Treatment

The parameters of the heat treatment done were the following: for the lots A and B, submitted to isothermal hardening, the austenizing temperature T_A = 900 °C and for the lots: A_1 and B_1 submitted to Dual-Phase bainitic treatment, the austenizing temperature T_A = 830 °C, the maintained time at austenizing temperature, τ_A = 60 min for all the lots.

The temperature at isothermal level, for all the lots was: T_{iz}= 300 and 400 °C; the maintained time at the isothermal level, t_{iz} = 30; 60; 90 and 120 min. All these four experimental lots were performed at isothermal maintenance in salt-bath, being the cooling after the isothermal maintenance was done in air.

5. Experimental Procedure and Results

The experiment groups performed at isothermal maintenance in salt-bath (55%KNO_3 + 45 %$NaNO_3$), being the cooling after the isothermal maintenance was done in air. From this material, 48 specific hardness specimens (ϕ 20×10 mm) was done. Hardness (Brinell Hardness) was done with 10 mm standard ball and 3000 kgf (HBS), according with ASTM Standard - E 10 Test Method for Brinell Hardness of Metallic Materials [11]. For each HB determination it was done three parallel determinations (*r* = 3).

The values of the mechanical results are presented in tables 2 and 3.

Table 2. Data Analysis of HB values for T_A = 900°C

Parallel observations	Samples, k = 4							
	T_{iz} = 300 °C (lot A)				T_{iz} = 400 °C (lot B)			
r = 3	τ_{iz}=30 min	τ_{iz}=60 min	τ_{iz}=90 min	τ_{iz}=120 min	τ_{iz}=30 min	τ_{iz}=60 min	τ_{iz}=90 min	τ_{iz}=120 min
Obs 1	487	442	421	381	409	362	336	319
Obs 2	475	442	409	371	400	362	344	327
Obs 3	487	432	409	371	400	371	336	319

Table 3. Data Analysis of HB values for T_A=830 °C

Parallel observations	Samples, k = 4							
	T_{iz} = 300 °C (lot A_1)				T_{iz} = 400 °C (lot B_1)			
r = 3	τ_{iz}=30 min	τ_{iz}=60 min	τ_{iz}=90 min	τ_{iz}=120 min	τ_{iz}=30 min	τ_{iz}=60 min	τ_{iz}=90 min	τ_{iz}=120 min
Obs 1	421	390	371	344	371	327	311	294
Obs 2	409	381	362	344	371	227	319	301
Obs 3	421	381	362	353	362	336	311	294

It can be certainly observed a normal evolution of the values for HB values:

- -when maintaining time at the isothermal level for both heat treatments is growing then the values of HB are decreasing;
- -when maintaining time at the same temperature of the isothermal level for both heat treatments is increasing, than the values of HB decreasing;
- -comparing the values of the mechanical properties for both heat treatments in the case of the classical isothermal austempering heat treatment, at the same time and temperature, the isothermal level determining high values for HB comparing with the "Dual-Phase" bainitic heat treatment.

This evolution of the mechanical properties is determined by the structural changes reported to the parameters of the heat treating. This evolution of the mechanical properties is determining by the structural constituents for each heat treatment [9. 10].

In the case of lots A and A_1 structure can be constituted of inferior bainite, residual austenite and martensite. These constituents are determining high values for HB. Comparing the both lots suppose a high value of inferior bainite and martensite in lot A comparing to lot A_1 (see the values of the HB).

Together with increasing the level of the isothermal maintenance temperature inside the structure will appear the superior bainite and the martensite will disappear (lot B and B_1), this is because of the ferrite value obtained in the heat treatment.

In the same time there can be observed a general characteristic about the studied lots: less maintaining time for the isothermal variation provides higher values of HB, this can be explained by the time of the isothermal level maintenance, followed by air cooling at the room temperature, is increasing the proportion of martensite, a constituent which is determining higher values for HB.

6. Calculation of Two-Way ANOVA

Two-factor ANOVA / Two-way ANOVA: is an experiment with two independent variables, call them factor 1 (in our case, the temperature at isothermal level, T_{iz}) that has two levels: T_{iz} = 300 and 400 °C and factor 2 (in our case, the maintained time at the isothermal level, t_{iz}) that has four levels: t_{iz} = 30; 60; 90 and 120 min, then there will be 2×4=8 different treatment groups. All calculations were made for two alternatives of austenizing temperatures of heat treatment: T_A = 830 and 900 °C. The dependent variable in our study is the hardness (Brinell Hardness).

The purpose of the paper is to analyze the influence of two independent variables on the dependent variable in the case of two alternatives of heat treatment.

It was used the values of HB values, which are the "Data Analysis" for calculation (see tables 2 and 3).

It was selected the level of significance (the default is 5% or 0.05). The Summary Table of the ANOVA-Two Factor with Replication is presented in Tables 4 and 5.

Table 4. Summary Table of the ANOVA-Two Factor With Replication, for T_A = 900 °C

SUMMARY	**Sample 30**	**Sample 60**	**Sample 90**	**Sample 120**	**Total**
300 (lot A)					
Count	3	3	3	3	12
Sum	1449	1316	1239	1123	5127
Average	483	438.6667	413	374.3333	427.25
Variance	48	33.3333	48	33.3333	1731.841
SUMMARY	**Sample 30**	**Sample 60**	**Sample 90**	**Sample 120**	**Total**
400 (lot B)					
Count	1209	1095	1016	965	4285
Sum	403	365	338.6667	321.6667	357.0833
Average	27	27	21.3333	21.3333	1044.265
Variance	1209	1095	1016	965	4285
TOTAL	**Sample 30**	**Sample 60**	**Sample 90**	**Sample 120**	
Count	6	6	6	6	
Sum	2658	2411	2255	2088	
Average	443	401.8333	375.8333	348	
Variance	1950	1652.167	1685.367	854	

Table 5. Summary Table of the ANOVA-Two Factor With Replication, for T_A = 830 °C

SUMMARY	**Sample 30**	**Sample 60**	**Sample 90**	**Sample 120**	**Total**
300 (lot A_1)					
Count	3	3	3	3	12
Sum	1251	1152	1095	1041	4539
Average	417	384	365	347	378.25
Variance	48	27	27	27	756.2045

SUMMARY	Sample 30	Sample 60	Sample 90	Sample 120	Total
400 (lot B₁)					
Count	3	3	3	3	12
Sum	1104	890	941	889	3824
Average	368	296.6667	313.6667	296.3333	318.6667
Variance	27	3660.333	21.3333	16.33333	1615.879
TOTAL	**Sample 30**	**Sample 60**	**Sample 90**	**Sample 120**	
Count	6	6	6	6	
Sum	2355	2042	2036	1930	
Average	392.5	340.3333	339.3333	321.6667	
Variance	750.3	3763.067	809.8667	787.4667	

The Source of Variation of the ANOVA- Two Factor With Replication are presented in Tables 6 and 7, where:

F is the calculated value of the Fischercriteria; $F_{Sample} = MS_{Sample} / MS_{Within}$; $F_{Columns} = MS_{Columns} / MS_{Within}$; $F_{Interaction} = MS_{Interaction} / MS_{Within}$;

F-crit = is the critical value of the Fischercriteria: for the level of significance, α = 0.05 (the default is 5% or 0.05.), the critical value for F;

F-crit for Sample with df (1, 16) is 4.493998;

F-crit for Columns with df (3, 16) is 3.238872;

F-crit for Interaction with df (3, 16) is 3.238872.

Table 6. Source of variation of the ANOVA- Two Factor With Replication, T_A = 900 °C

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F-crit
Sample	39540.17	1	29540.17	911.2648	1.56E-15	4.493998
Columns	29369.67	3	9789.889	302.0017	2.74E-14	3.238872
Interaction	648.8333	3	216.2778	6.671808	0.003938	3.238872
Within	518.6667	16	32.41667			
Total	60077.33	23				

Table 7. Source of variation of the ANOVA- Two Factor With Replication, T_A = 830 °C

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F-crit
Sample	21301.04	1	21301.04	44.21597	5.59E-06	4.493998
Columns	16840.46	3	5613.486	11.65228	0.000268	3.238872
Interaction	1544.458	3	514.8194	1.068644	0.39013	3.238872
Within	7708	16	481.75			
Total	47393.96	23				

7. Discussion

The following observations can be made after analyzing the results:

(a) Examining the data revealed that at shorter austempering time for all maintaining temperature, the bainitic transformation is not enough and austenite converts to martensite with the increasing the values of hardness (HB).

(b) For the isothermal maintained temperatures (t_{iz} = 300 °C), the specific structures expended is made by inferior bainitic ferrite with martensite and an amount of retained austenite, due to the hardness evidence (Table 1);

(c) For the isothermal maintained temperatures (t_{iz} = 400 °C), the specific structures expended is made by superior bainitic ferrite with martensite and with the a higher amount of retained austenite, due to the hardness evidence (Table 1);

(d) For the al isothermal maintained temperatures (t_{iz} = 300 and 400 °C), the hardness (HB) value decrease with increasing of the isothermal maintaining time (τ_{iz}) from 30 to 120 minutes, due by the structural changes reported to the parameters of the heat treating. Together with increasing the level of the isothermal maintenance, temperature inside the structure will appear the superior bainite and the martensite will disappear [6].

(e)From table 6, the austenizing temperature, T_A= 900 °C, it was compares the two values of the F test statistic: calculate value (F) and critical value (F-crit). Note that in this case the calculated value (F) is greater than the critical value (F-crit), i.e.:

- analyzing the first independent variable, the austenizing temperatures, T_{iz} = 300 °C, is observed that the calculated value F = 911.2648 is greater than the critical value (F-crit) = 4.493998 (911.2648 > 4.493998), therefore, the effect of the first independent variable is significant;
- analyzing the second independent variable, the austenizing temperatures, T_{iz} = 400 °C, is observed that the calculated value F = 302.0017 is greater than the critical value (F-crit) = 3.238872 (302.0017 >3.238872), therefore, the effect of the second independent variable is significant;
- analyzing the effect of interaction between the two independent variables, we see that the calculated value F = 6.671808 is greater than the critical value (F-crit) = 3.238872 (6.671808 > 3.238872), therefore, so both variables are the parameters for the technological process.

(f) From table 7, for the austenizing temperature T_A= 830 °C, it was compares the two values of the F test statistic: calculated value (F) and critical value (F-crit).The following observations can be made after analyzing the results:

- analyzing the first independent variable, the austenizing temperatures, T_{iz} = 300 °C, is observed that the calculated value F = 44.21597 is greater than the critical value (F-crit) = 4.493998 (44.21597 > 4.493998), therefore, the effect of the first independent variable (T_A = 900 °C) is significant;
- analyzing the second independent variable, the austenizing temperatures, T_{iz} = 400 °C, is observed that the calculated value F = 11.65228 is greater than the critical value (F-crit) = 3.238872 (11.65228 > 3.238872), therefore, the effect of the second independent variable (T_A = 830 °C) is significant;
- analyzing the effect of interaction between the two independent variables, we see that the calculated value F = 1.068644 it is smaller than the critical value (F-crit) = 3.238872 (1.068644 < 3.238872), therefore, there is no interaction between the two independent variables, so the cumulative influence of the two variables there is not an important factor for process (not a technological factor).

7. Conclusions

By studying all the data presented in this paper following remarkable conclusions:

(a) Analysis of variance (ANOVA) is the most efficient method available for the analysis of experimental data;

(b) The two-way analysis of variance (Two-way ANOVA) is an extension to the one-way analysis of variance and compares the mean differences between groups that have been split on two independent variables;

(c) Austempered Ductile Iron (A.D.I.) with a bainitic matrix, obtained by heat treatment and isothermal hardening is the material which combines a lot of superior attributes of the classical Austempered Ductile Irons or forged iron, being in a serious competition with the iron used by the moment in the automotive industry;

(d) Analyzingthe effect of interaction between the two independent variables, we see that for the austenizing temperature, T_A = 900 °C, the both independent variable and their interaction are important for our process, so variables are technological parameters;

(e) Analyzingthe effect of interaction between the two independent variables, we see that for the austenizing temperature, T_A = 830 °C, the both independent variable are important for our process, so variables are technological parameters, but the cumulative influence of the two variables there is not an important factor for process (not a technological factor).This is explained by the fact that the austenizing temperature, T_A = 830 °C is not a good factor for obtaining superior hardness values.

References

1. Weaver, B. (2005): *Two-way Analysis of Variance*. Available at: http://www.angelfire.com/wv/bwhomedir/notes/anova2.pdf. Accessed: 2016-02-29
2. ***: https://people.richland.edu/james/lecture/m170/ch13-2wy.html. Accessed: 2016-02-29
3. Hae-Young, K. (2014): *Statistical notes for clinical researchers: Two-way analysis of variance (ANOVA)-exploring possible interaction between factors.* Restor Dent Endod., ISSN 2234-7658, vol. 39 (2), May, p. 143-147
4. Assaad, H, Hou, Y., Zhou, L., Carroll, R.J., Wu, G. (2015): *Rapid publication-ready MS-Word tables for two-way ANOVA*. Springer Plus, ISSN 2193-1801, vol. 4, p. 33-42
5. Budde, J. (2009): *Variance analysis and linear contracts in agencies with distorted performance measures*. Management Accounting Research, ISSN 1044-5005, vol. 20, no. 3 (September), p. 166-176
6. Wing, K.T. (2005): *Using enhanced cost models in variance analysis for better control and decision making*. Management Accounting Quarterly, ISSN 1528-5359, vol. 4, no. 2, p. 27-35
7. Yahya-Zadeh, M. (2011): *A new framework for capacity costing and inventory variance analysis*. Journal of Applied Management Accounting Research, ISSN 1044-5005, vol. 9, no. 2 (Summer), p. 61-81
8. Armstrong, R.A., Eperjesi, F., Gilmartin, B. (2002): *The application of analysis of variance (ANOVA) to different experimental designs in optometry*. Ophthalmic and Physiological Optics, ISSN 1475-1313, vol. 22, no. 3, p.248-256
9. Harding, R.A. (2007): *The production, properties and automotive applications of austempered ductile iron*. KovoveMaterialy, ISSN 1338-4252, vol. 45, p.1-16
10. Rimmer, A. (2004): *ADI solutions aid vehicle design*. The Foundry Trade Journal, ISSN 0015-9042, vol. 2, p. 54-56
11. ***: http://www.mdmstandard.ro/download/resurse/Tabele%20de%20conversie%20ASTM %20pentru %20duritati%20%28in%20engleza%29.pdf. Accessed: 2016-02-29

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