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ESTIMATION OF THE EFFECT OF PRODUCTION PARAMETERS ON MECHANICAL PROPERTIES OF SINTERED STEELS USING ANOVA

The object of the study was to assess the influence of selected production parameters of sintered Fe-Mn-Cr-Mo-C steels i.e. chemical composition, sintering temperature, sintering atmosphere and heat treatment on the following mechanical properties: impact toughness, hardness of the surface, tensile strength, bend strength after static tensile tests.

In the investigations, the general linear model (GLM) of the multivariate analysis of variance ANOVA was used. All assumptions of ANOVA, i.e. randomization of the experiment, the normality of the residuals, equality of variance at different levels have been fulfilled and verified. The predictive strength of the constructed models expressed by the adjusted determination coefficient (R^2_{adj}) is at medium or large level – R^2_{adj} is in the range from 41.46% to 76.97%. This work is focused mainly on the ANOVA methodology. A wide physical interpretation of the results will be possible after the optimization of the ANOVA models used.

Keywords: powder metallurgy, analysis of variance (ANOVA)

1. Introduction

Powder metallurgy is a method of obtaining metallic products from powders without the necessity of changing the material's state into a liquid. It is a competitive technique to conventional methods of producing and processing metallic materials, such as casting, plastic working or subtractive manufacturing [1]. Elements made of sintered materials are widely applied in many industrial branches; the most extensive use of the powder technology is in the automotive industry, in which there is a huge need for reliable – with very good properties – parts of complicated shapes [2].

The powder metallurgy technology mainly consists of 4 stages: (1) manufacturing and powder preparation, (2) formation, (3) sintering and (4) finishing, post-sintering treatment [1,2]. The most important stage of this technology is sintering. In this process, similarly to any other process, broadly understood as processing of 'inputs' into 'outputs' [3]. Beside the input parameters which can be controlled, such as temperature, time, etc., there is a series of factors which cannot be controlled, as their character is random, i.e. they are impossible to regulate (the so-called 'noise'). The variability generated by the uncontrollable factors (noise) causes each outcome parameter of the sintering process to constitute a realization of the random variable. As a consequence of the unbreakable variability of each process is a static, i.e. ambiguous, character of the relation between the controllable 'inputs' and the broadly understood outcome of the process. A description of the behaviour of the process, due to variability, is the subject of statistical process control (SPC), whereas the tool for evaluating the effect of the selected controllable factor(s) on the specific process is the analysis of variance (ANOVA).

2. Analysis of variance (ANOVA)

The subject of the analysis of variance (ANOVA) is the assessment of the selected factor(s) on the outcome of the process, i.e. on the behaviour of the specific outcome parameter in the presence of the influence of a series of other factors, of a random and non-random character. In other words, ANOVA answers the question whether, in view of all the other factors determining the behaviour of the outcome parameter, the selected factor(s) has/have a statistically significant effect. The selected factor(s) correspond(s) to the so-called levels. For example, if the selected factor is the sintering atmosphere, the levels for this factor are all the atmosphere variants used in the tests.

In general, we can distinguish between the univariate and multivariate ANOVA. In the univariate ANOVA, we are interested in the effect of one specific factor at the selected levels on the behaviour of the outcome parameter. In the case of the multivariate ANOVA, we are interested in the effect of the specific number of factors, with a declared number of levels for each of them, on the behaviour of the outcome parameter. Additionally, in the multivariate ANOVA, we are also interested in the interactions between the selected factors. The presence of interaction means that one of the selected factors changes the character of operation of (an)other selected factor(s). For example, let us

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consider the case of bi- and trivariate ANOVA. In the bivariate analysis of variance, we evaluate the effect of two selected factors and one bivariate reaction on the selected outcome parameter. In the trivariate analysis of variance, we evaluate the effect of three double interactions (factor I * factor II, factor I * factor III, factor II * factor III) and one triple interaction (factor I * factor II * factor III) on the behaviour of the outcome parameter [4,5].

The manner of inference in ANOVA depends on the considered model. If the assumed levels of the analyzed factor(s) correspond to all the practically possible ones, we have the case of the so-called constant model. In such a case, if the ANOVA result points to the effect of the levels of the analyzed factor(s) on the behaviour of the outcome parameter, one should answer the question, by means of the so-called post-hoc tests: between which levels can we observe a statistically significant difference, in respect of the outcome parameter? The Tukey test and the Fisher test are most frequently used among the post-hoc tests [10].

In the case when the levels of the considered factor(s) constitute a subset of many practically possible ones, we have the case of the so-called random model. In such a case, ANOVA answers only the question whether the analyzed factor has any effect on the behaviour of the considered outcome parameter [10]. In practice, we often have the case of mixed models, in which some of the factors are constant in character, whereas others are random.

3. Experimental

3.1. Test objective, experimental material

The aim of the research was to assess the effect of the production parameters of sintered Fe-Mn-Cr-Mo-C steels on the following mechanical properties (outcome parameters): (1) KC – impact toughness, (2) HV – hardness on the surface, (3) R_m – tensile strength, (4) R_g – bend strength. Data for analysis are presented in Table 1. The manner of sample preparation was described in [6-9].

TABLE 1

Data for analysis

Sample description	Chemical composition	Sintering temperature	Sintering atmosphere	Heat treatment	KC, J/cm^2	HV 30	R_g, MPa	R_m, MPa
A	Astaloy CrL + 3Mn + 0.3C	1120°C	5%H_2-95%N_2	200°C/1h/air	—	—	1054	593
B		1250°C			—	—	1255	644
C	Astaloy CrM + 3Mn + 0.3C	1120°C			—	—	896	478
D		1250°C			—	—	1320	601
1L1-5	Astaloy CrL + 3Mn + 0.15C	1250°C		NT*	6.19	231	1105	593
1L6-10				200°C/1h/air	5.89	158	1114	606
1L11-15			air + 52g FeMn	NT	5.85	243	1172	595
1L16-20				200°C/1h/air	7.10	224	1158	520
1L21-25		1120°C	5%H_2-95%N_2	NT	4.66	218	970	525
1L26-30				200°C/1h/air	5.08	170	979	523
1L31-35			air + 52g FeMn	NT	4.39	185	1088	530
1L36-40				200°C/1h/air	5.26	202	1138	616
1M1-5	Astaloy CrM + 3Mn + 0.15C	1250°C	5%H_2-95%N_2	NT	4.92	247	1083	694
1M6-10				200°C/1h/air	5.66	256	1178	748
1M11-15			air + 52g FeMn	NT	6.12	273	1191	636
1M16-20				200°C/1h/air	5.35	300	1203	660
1M21-25		1120°C	5%H_2-95%N_2	NT	4.45	246	897	446
1M26-30				200°C/1h/air	3.32	191	976	564
1M31-35			air + 52g FeMn	NT	3.55	184	906	526
1M36-40				200°C/1h/air	3.61	300	809	551
3L1-5	Astaloy CrL + 3Mn + 0.7C	1250°C	5%H_2-95%N_2	NT	3.05	—	666	386
3L6-10				200°C/1h/air	4.93	—	1003	658
3L11-15			air + 52g FeMn	NT	3.38	—	721	428
3L16-20				200°C/1h/air	4.52	—	1163	530
3L21-25		1120°C	5H_2-95N_2	NT	2.37	—	438	308
3L26-30				200°C/1h/air	4.11	—	836	499
3L31-35			air + 52g FeMn	NT	2.80	—	606	341
3L36-40				200°C/1h/air	3.17	—	726	460
3M1-5	Astaloy CrM + 3Mn + 0.7C	1250°C	5%H_2-95%N_2	NT	3.67	—	638	386
3M6-10				200°C/1h/air	7.14	—	1112	579
3M11-15			air + 52g FeMn	NT	3.61	—	577	392
3M16-20				200°C/1h/air	7.44	—	973	492
3M21-25		1120°C	5%H_2-95%N_2	NT	2.47	—	397	311
3M26-30				200°C/1h/air	5.09	—	676	417
3M31-35			air + 52g FeMn	NT	2.92	—	469	272
3M36-40				200°C/1h/air	4.90	—	675	414

* NT-not tempered; Astaloy CrL – Fe-1.5%Cr-0.2%Mo; Astaloy CrM – Fe-3%Cr-0.5%Mo

In the investigations, the method of multivariate analysis of variance ANOVA was used. In reference to the mentioned outcome parameters, the following factors (production parameters) were considered: (1) chemical composition, (2) sintering temperature, (3) sintering atmosphere, (4) heat treatment. Full characteristics of the production parameters are presented in Table 2.

TABLE 2

Parameters of sintered Fe-Mn-Cr-Mo-C steels production

Production parameter (factor)	Considered levels – level denotation	Description
Chemical composition	A1 A2 A3 A4 A5 A6	Astaloy CrL + 3 Mn + 0.3 C Astaloy CrM + 3 Mn + 0.3 C Astaloy CrL + 3 Mn + 0.15 C Astaloy CrM + 3 Mn + 0.15 C Astaloy CrL + 3 Mn + 0.7 C Astaloy CrM + 3 Mn + 0.7 C
Sintering temperature	1120°C 1250°C	
Sintering atmosphere	AT1 AT2	$5\%H_2 – 95\% N_2$ air
Heat treatment	200°C NT	tempering, 200°C/1h/air not tempered

4. Implementation of ANOVA

4.1. ANOVA variants and the procedure of calculation

All the variants of the performed ANOVA analyses are presented in Table 3. For example, in variant 1, the factors affecting the impact toughness outcome parameter are: the chemical composition of the powder (levels: A3-A6), the sintering temperature (levels: 1120°C, 1250°C), the sintering temperature (levels: AT1, AT2), heat treatment (levels: 200°C, NT). The ANOVA analysis was performed according to the procedure of the general linear model (GLM) [4,5].

The course of the procedure was as follows:

- Verification of the ANOVA assumptions; (1) for the assessment of the normality of residuals, the graphic normality test was used, (2) the hypothesis of variance equality at the levels of the analyzed factors was verified using the Bartlett and Levene tests [5].
- The assumption referring to randomization, i.e. random assigning of samples to levels of the analyzed factors, was satisfied by the manner of performing the experiment.
- Performing the main ANOVA analysis with the use of the GLM model; in all the cases, beside the assessment of the effect of the analyzed factors, the model also included assessment of the effect of all the interactions.

The calculations were carried out in the Minitab 17 environment.

TABLE 3

ANOVA variants

Variant	Outcome parameter	Factor	Factor levels
1	Impact toughness	Powder chemical composition	A3, A4, A5, A6
		Sintering temperature	1120°C, 1250°C
		Sintering atmosphere	AT1, AT2
		Heat treatment	200°C, NT
2	Hardness	Powder chemical composition	A3, A4
		Sintering temperature	1120°C, 1250°C
		Sintering atmosphere	AT1, AT2
		Heat treatment	200°C, NT
3	R_m (Samples A,B,C,D, Tab. 1)	Powder chemical composition	A1, A2
		Sintering temperature	1120°C, 1250°C
4	R_m (Samples 1L1-3M40, Tab. 1)	Powder chemical composition	A3, A4, A5, A6
		Sintering temperature	1120°C, 1250°C
		Sintering atmosphere	AT1, AT2
		Heat treatment	200°C, NT
5	R_g (Samples A,B,C,D Tab. 1)	Powder chemical composition	A1, A2
		Sintering temperature	1120°C, 1250°C
6	R_g (Samples 1L1-3M40, Tab. 1)	Powder chemical composition	A3, A4, A5, A6
		Sintering temperature	1120°C, 1250°C
		Sintering atmosphere	AT1, AT2
		Heat treatment	200°C, NT

4.2. Detailed results of ANOVA

Detailed results of ANOVA for one of the outcome parameters, i.e. hardness, are presented below. Factor and levels, presented in Tables 2 and 3, are as follows:

- Chemical composition – levels: A3, A4,
- Sintering temperature – levels: 1120ºC; 1250ºC,
- Sintering atmosphere – levels: AT1, AT2,
- Heat treatment – levels: 200°C, NT.

Verification of the ANOVA assumptions - variance equality at the levels of the analyzed factors is presented in Fig. 1. Assessment of the normality of residuals - the graphic normality test is presented in Fig. 2. Detailed results of the ANOVA are shown in Table 4 and Figs. 3-4. In particular Fig. 4 shows all possible bivariate interactions; parallel lines means no interaction. Crossed lines means very strong interaction.

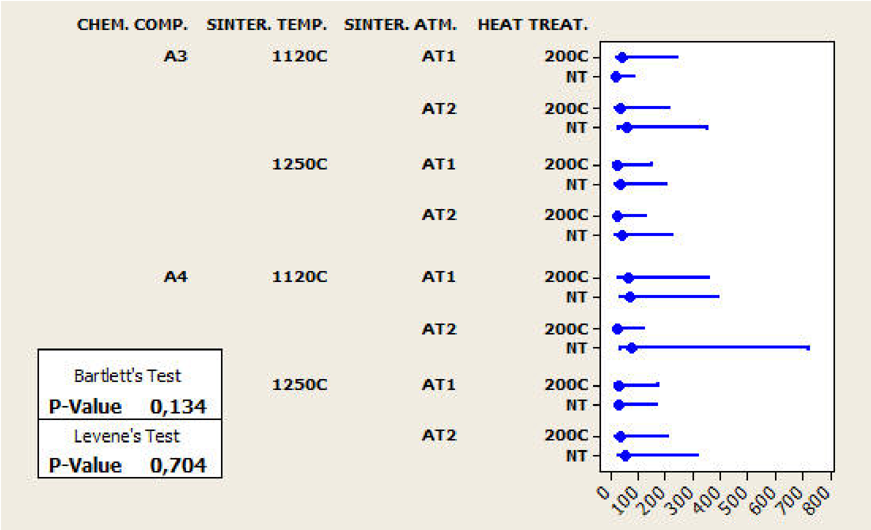

Fig. 1. Test for Equal Variances for hardness (95% Bouferroni Confidence Intervals for standard deviation)

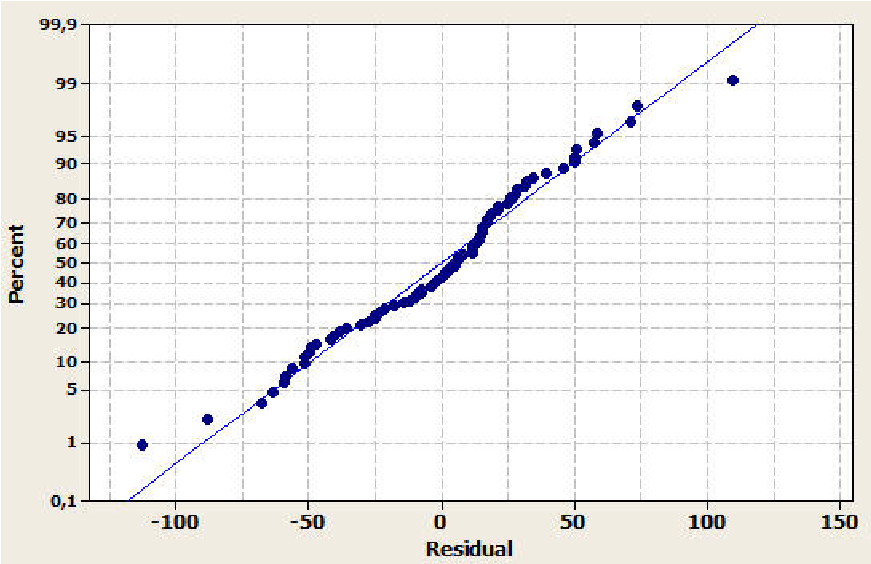

Fig. 2. Normal probability plot – response is hardness

TABLE 4

Analysis of variance for hardness, using adjusted SS for tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
CHEM. COMP.	1	46246	43481	43481	23,89	0,000
SINTER. TEMP.	1	14234	15340	15340	8,43	0,005
SINTER. ATM.	1	14517	13013	13013	7,15	0,010
HEAT TREAT.	1	746	427	427	0,23	0,630
CHEM. COMP.*SINTER. TEMP.	1	748	1128	1128	0,62	0,434
CHEM. COMP.*SINTER. ATM.	1	1322	875	875	0,48	0,491
CHEM. COMP.*HEAT TREAT.	1	12302	13468	13468	7,40	0,008
SINTER. TEMP.*SINTER. ATM.	1	2017	2449	2449	1,35	0,250
SINTER. TEMP.*HEAT TREAT.	1	1385	1704	1704	0,94	0,337
SINTER. ATM.*HEAT TREAT.	1	26220	26768	26768	14,71	0,000
CHEM. COMP.*SINTER. TEMP.* SINTER. ATM.	1	1634	1447	1447	0,80	0,376
CHEM. COMP.*SINTER. TEMP.* HEAT TREAT.	1	899	738	738	0,41	0,526
CHEM. COMP.*SINTER. ATM.*HEAT TREAT.	1	892	1038	1038	0,57	0,453
SINTER. TEMP.*SINTER. ATM.* HEAT TREAT.	1	6898	7079	7079	3,89	0,053
CHEM. COMP.*SINTER. TEMP.* SINTER. ATM.*HEAT TREAT.	1	5074	5074	5074	2,79	0,100
Error	63	114641	114641	1820		
Total	78	249776				

R-Sq(adj) = 43,17%

where: DF – degrees of freedom; Seq SS – sequential sums of squares; Adj SS – adjusted sums of squares; Adj MS – adjusted mean squares; F – F-statistic; P – p-value; R-Sq(adj) – adjusted R^2,%

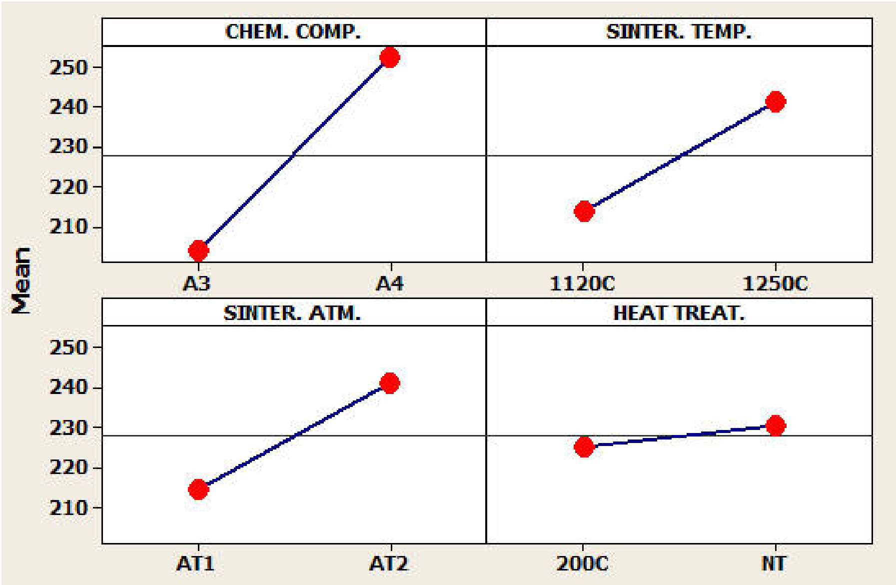

Fig. 3. Main effects plot for hardness (data means)

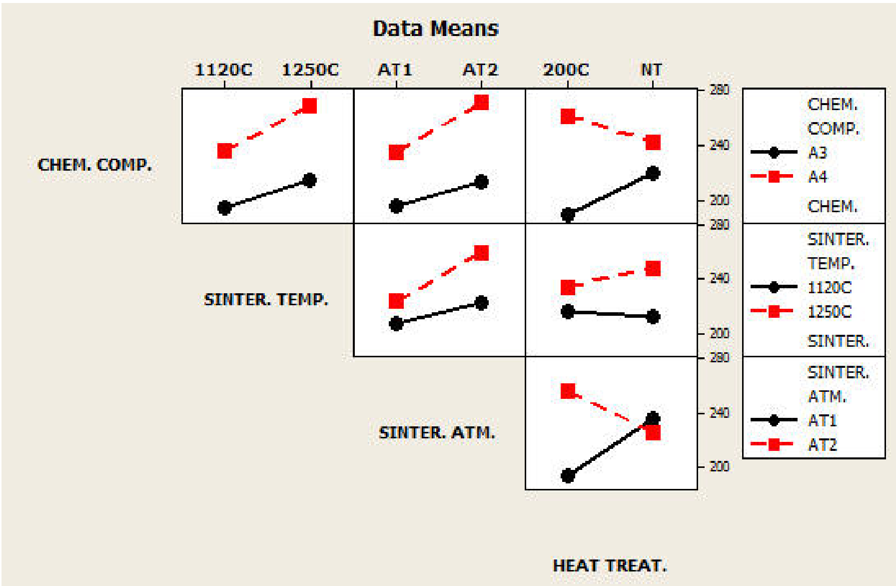

Fig. 4. Interaction plot for hardness (data means)

4.3. Compilation of ANOVA results

The compilation of all the ANOVA results is given in Table 5. The manner of result presentation is as follows. Table 5 gives the results of testing the hypothesis of the significance of the effect on the analyzed outcome parameter with the reference to each factor and the bivariate interactions. The result of hypothesis testing is the value of the so-called post-test probability, p-value. Assuming the level of significance $\alpha = 0.05$, the inference principle is as follows. If the obtained post-test probability value is lower than or equal to the assumed level of significance, i.e. hardness 0.05, the hypothesis of the lack of effect of the analyzed factor or the double interaction on the outcome parameter is rejected. Otherwise, post-test probability assumes a value higher than 0.05; there is no basis for rejecting the hypothesis of the lack of effect of the analyzed factor or double interaction on the outcome parameter. From the practical point of view, the fact of no basis for rejecting the hypothesis equals its acceptance.

In an analogous way, Table 5 presents the results of testing the hypothesis of variance equality at each level of the analyzed factor by means of Bartlett and Levene tests; in all the variants, the assumption of variance equality is satisfied.

Additionally, Table 5 shows the values of adjusted coefficient of determination R^2, which describes (in percent) the degree of the effect of the analyzed factors on the behaviour of the outcome parameter.

In all the analyzed variants, the ANOVA assumptions can be regarded as satisfied.

TABLE 5

Compilation of ANOVA results

	FACTORS				BIVARIATE INTERACTIONS							
Outcome parameter	**Chem. comp.**	**Sinter. temp.**	**Sinter. atm.**	**Heat treat.**	**Chem. comp. * Sinter. temp.**	**Chem. comp. * Sinter. atm.**	**Chem. comp.* Heat treat.**	**Sinter. temp.* Sinter. atm.**	**Sinter. temp.* Heat treat.**	**Sinter. atm.* Heat treat.**	**Adjusted R^2,%**	**p-value Bartlett; Levene**
Impact toughness	0.000	0.000	0.983	0.000	0.095	0.747	0.000	0.371	0.113	0.397	70.41	0.018 0.866
Hardness	0.000	0.005	0.010	0.630	0.434	0.491	0.008	0.250	0.337	0.000	43.17	0.134 0.704
R_m (A,B,C,D)	0.001	0.000	—	—	0.022	—	—	—	—	—	41.46	0.966 0.862
R_m (1L1-3M40)	0.000	0.000	0.030	0.000	0.002	0.063	0.000	0.003	0.277	0.062	75.63	0.786 0.953
R_g (A,B,C,D)	0.324	0.000	—	—	0.020	—	—	—	—	—	45.26	0.321 0.582
R_g (1L1-3M40)	0.000	0.000	0.148	0.000	0.009	0.103	0.000	0.991	0.032	0.103	76.97	0.025 0.507

5. Discussion of the results and conclusions

The obtained results are preliminary, which creates the further necessity of outcome optimization, e.g. by way of eliminating the factors which have no statistically significant effect from the model, analyzing the goodness-of-fit (lack-of-fit), or applying the mentioned post-hoc tests, etc. This preliminary character of the results does not allow for their thorough interpretation from the physical point of view, as yet.

To summarize the preliminary analysis results, the following conclusions can be drawn:

1. In the all analyzed variants, all the ANOVA assumptions can be regarded as satisfactorily fulfilled.
2. The obtained values of corrected coefficient of determination R^2 prove a medium, or higher than medium, degree of the effect of the analyzed factors on the outcome parameters.
3. In each analyzed variant, at least one factor has a statistically significant effect.
4. One can observe very interesting, statistically significant, bivariate interactions, e.g. for the chemical composition*temperature interaction in the case of parameters R_m and R_g, which require a very thorough and careful interpretation.

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