THE EFFECT OF HEAT TREATMENT ON THE IMPACT STRENGTH OF ADI

GIĘTKATomasz[1], CIECHACKI Krzysztof[1], GUMIENNY Grzegorz[2]

[1]*University of Science and Technology in Bydgoszcz, Mechanical Engineering Faculty, Department of Materials Science and Engineering, Bydgoszcz, Poland, EU, tgietka@utp.edu.pl*
[2]*Technical University of Łódź, Department of Materials Engineering and Production Systems, Łódź, Poland, EU*

Abstract

This publication attempts to investigate the effect of heat treatment parameters on the impact strength of ADI. Ductile iron was tempered with isothermal transformation according to two variants. Both variants of heat treatment consisted of two-stage austenitizing. Iron was cured at t_A = 950⁰C, then gradually cooled to a temperature of the supercritical $t_{A'}$= 850, or 800⁰C (in the latter variant). Isothermal transformation was carried out under the same conditions. Isothermal hardening was carried out at 300 or 400⁰C and resisting the time of from 7.5 to 240 min. During these studies, the impact energy of the sample was notched. The results were compared with values given in the standard PN EN 1564 : 2012.

Keywords: ADI, impact test, isothermal transformation, austempered parameters

1. INTRODUCTION

ADI (Austempered Ductile Iron) is a type of ductile iron with a ferritic-austenitic matrix structure, characterized by a favourable combination of high strength, satisfactory ductility and fracture toughness. The wide ranging mechanical properties of ADI are achieved by a properly selected cycle of heat treatment leading to the formation of the final microstructure, which is a mixture of stable austenite and lamellar ferrite in the form of "needles" (ausferrite). The type of microstructure obtained is to a great extent controlled by the chemical composition of the base ductile iron and the parameters of the heat treatment process. This process consists of austenitizing in the temperature range of 800 - 950⁰C (pearlite - austenite transformation) and austempering in the temperature range of 230-400⁰C (austenite - ferrite transformation) [1-5, 7, 8, 12].

In the cast iron hardening operation, the austenitizing treatment that consists in heating at the temperature higher than Ac_1 should enrich austenite with carbon up to the limit determined by the E'S' line and uniform the metal matrix. During the heating, the cast iron with the initial ferritic microstructure only carbon atoms from graphite segregations are diffused to the austenite. The metal matrix austenitizing process and the role of graphite in its carbonisation are described in study [2].

The effect of cast iron austenitizing depends on the chemical composition, initial structure, granular graphite dispersion, heating temperature and time, as well as the uniformity of elements distribution in the eutectic grains and the size of matrix grains. A classical method of austenitizing before the isothermal transformation is single-stage austenitizing [1, 2, 12].

The analysis of the impact of austenite content on the impact resistance or impact energy of the ADI cast iron, including the austenitizing temperature, ausferritizing temperature and time in the upper and lower ausferrite range, was the subject of research in the study [2-7].

The chemical composition of ductile non-alloy cast iron is mainly restricted to the C, Si, Mn, S and P content. From the point of view of austenitizing at isothermal hardening, carbon, silicon and manganese are of particular importance.

To increase the treatment time range and thus the austenite content in the ADI cast iron matrix, the following elements were introduced: Mn, Mo and Cu [2, 4, 5], as well as Cu and Ni [8].

The objective of this study is to examine the impact of the variable conditions of two-stage austenitizing at two isothermal hardening temperatures in the upper and lower ausferrite range. The impact resistance of the samples was tested at the ambient temperature for one grade of isothermally hardened non-alloy ductile cast iron.

The research issues are directly related to the quality of the cast material in the production process.

2. MATERIAL, PROGRAMME AND TEST METHOD

One heat of ductile cast iron was assumed for the research. The cast iron was melted in a medium frequency induction furnace with the capacity of 3.5 t. The cast iron was spheroidised with manganese mortar VL53M (bell method, in a slim ladle) and modified with ferro-silicon FeSi75. The cast iron was cast into “wet” forms, reflecting YII samples in accordance with PN-EN 1563:2012 [11]. The chemical composition of the cast iron heat obtained was determined using a SPECTROLAB M5 spark spectrometer. The shares of the particular elements are given in **Table 1**.

Table 1 Composition and mechanical properties after casting ductile cast iron

Chemical composition, (wt. %)						**Coefficients**	
C	Si	Mn	P	S	Mg	S_c	C_e
3.65	2.59	0.182	0.027	0.014	0.043	1.06	4.47
Mechanical properties							
R_m, (MPa)		A_5, (%)		H, (HV10)		KCG, (J/cm^2)	
507		12.1		156		106	

The cast iron as-cast had a ferritic-perlite (10%) matrix structure and a correct granular graphite form (volume share 11.5%, 112 segregations/mm^2 microsection surface). The ductile cast iron microstructure is shown in **Figure 1**.

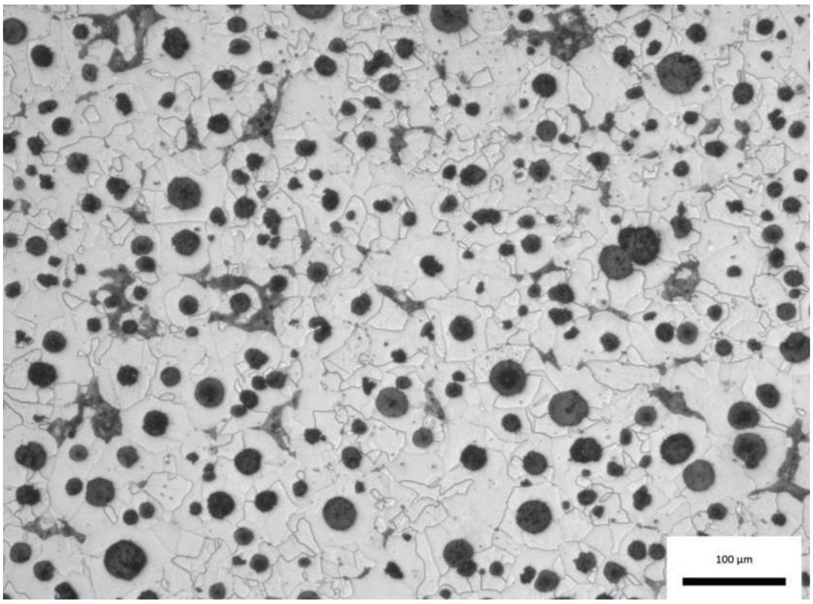

Figure 1 Structure of the cast iron as-casted, microscopic magnification 250x, etched with nital

YII billets were cut according to **Figure 2** to prepare impact resistance samples with the dimensions of 10 x 10 x 55 mm without a notch and the results obtained were compared with the minimum values specified in PN EN 1564:2012.

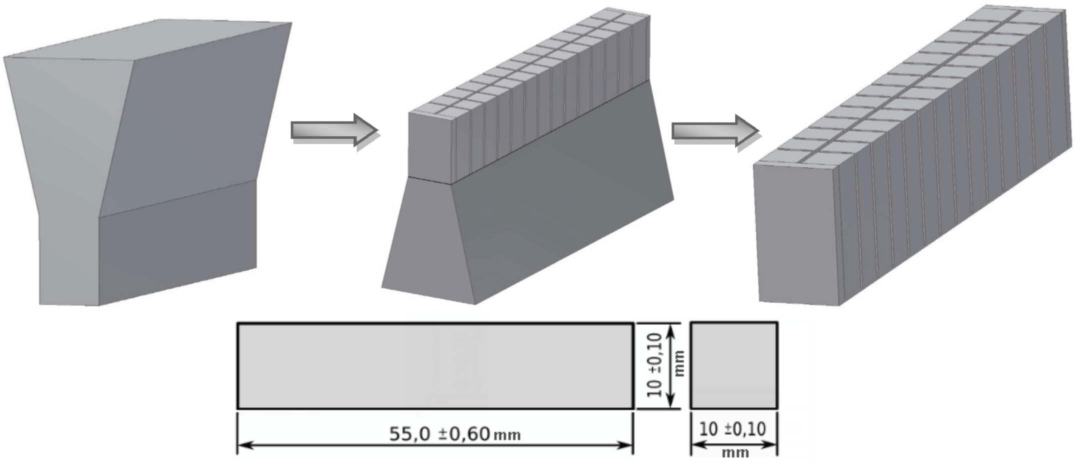

Figure 2 The method of taking impact resistance samples without a notch from YII billets

Impact resistance samples were hardened with an isothermal transformation, in accordance with the diagram shown in **Figure 3**. The heat treatment variants tested had the same nominal austenitizing temperature (T_A) of 950^{0}C, after 60 minutes samples were cooled down to two different temperatures ($T_{A1'}$ = 850^{0}C and $T_{A2'}$ = 800^{0}C), the austenitizing time after cooling down was 30 minutes. At the next stage, the samples were moved from silicone carbide chamber furnace to a bath furnace with SO140 salt bath for ausferritizing. The parameters that characterised ausferritizing were two ausferritizing temperatures (T_{AF1} = 400^{0}C and T_{AF2} = 300^{0}C), as well as six ausferritizing times (t_{AF} = 7.5; 15; 30; 60; 120; 240 minutes). For each treatment, impact resistance measurements were made on three samples from one YII billet.

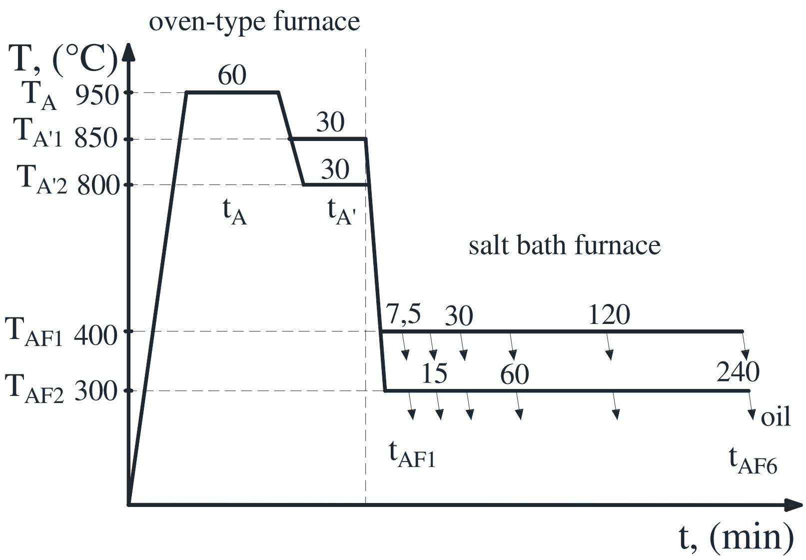

Figure 3 Hardening diagram with the isothermal transformation of impact resistance samples

The impact resistance test was made with a PSW 300 Charpy V-notch with the maximum impact energy of 300 J. The selected impact resistance sample breakthroughs were observed using a scanning electron microscope (SEM).

3. TEST RESULTS AND THEIR ANALYSIS

The impact resistance in the function of ausferritizing time and temperature for two different austenitizing output temperatures are shown in **Figures 4 and 5**.

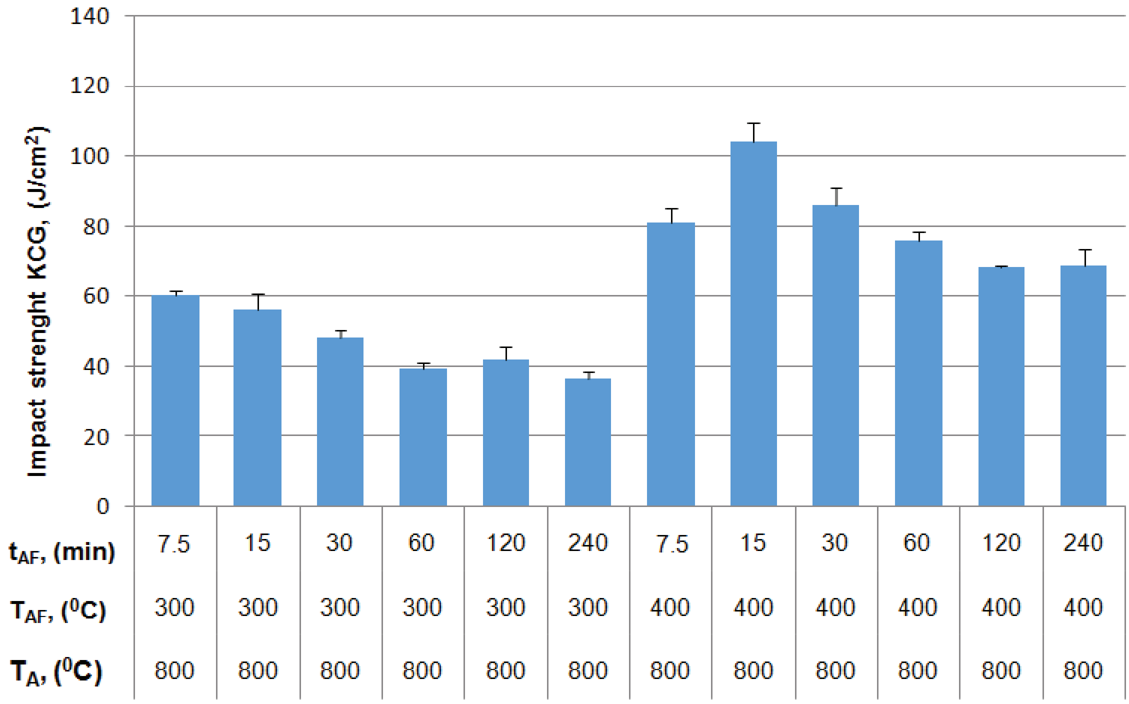

Figure 4 Impact strength in the function of ausferritizing time (t_{AF}) and ausferritizing temperature (T_{AF}) for $T_{A'}$ = 800^{0}C

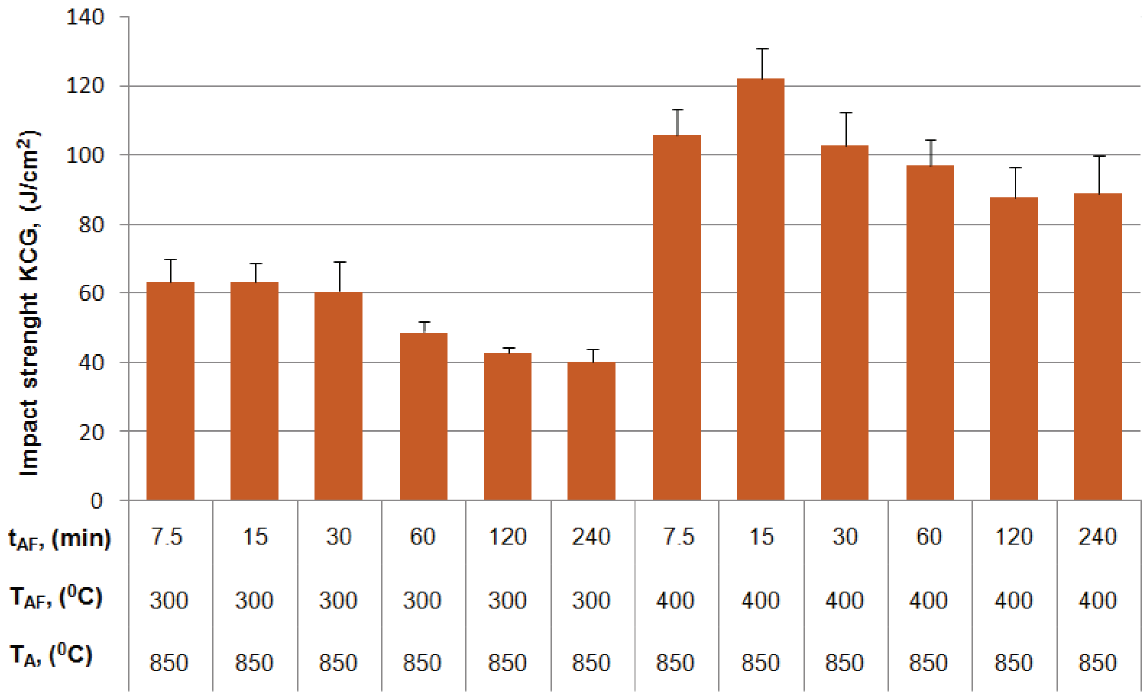

Figure 5 Impact strength in the function of ausferritizing time (t_{AF}) and ausferritizing temperature (T_{AF}) for $T_{A'}$ = 850^{0}C

To evaluate the impact of three independent variables treated as numerical variables ($T_{A'}$, T_{AF}; t_{AF}) for one dependent variable (KCG), it was decided to specify the following regression equation (1).

$$y_n = f(X1_n, X2_n, X3_n) \quad (1)$$

where:

y_n - corresponds to the KCG impact resistance value, J/cm^2;

$X1_n$ - corresponds to the austenitizing temperature after cooling down $T_{A1'}$ = 850^{0}C or $T_{A2'}$ = 800^{0}C;

$X2_n$ - corresponds to the ausferritizing temperature T_{AF1} = 400^{0}C or T_{AF2} = 300^{0}C;

$X3_n$ - corresponds to the ausferritizing time t_{AF} = 7.5; 15; 30; 60; 120; 240 minutes.

The research plan included one full experiment, meaning **24** experiments (the so-called SUBCLASSES) (because 2 · 2 · 6 = 24). Each experiment consisted of **three *KCG*** impact resistance measurements. Thus, overall: 3 · 24 = **72** impact resistance measurements were made. For statistical evaluation, the statistical significance level (type I error) was assumed: p (α) < 0.05 [10]. Statistical calculations were made in licensed statistical suites: Statistica v. 7.1 PL by StatSoft and MedCalc Statistical Software v.14.10.2 (MedCalc Software bvba, Ostend, Belgium).

A condition to use variance analysis for factorial systems is the homogeneity of the variance of the tested attribute in the particular groups. It was tested with two Levene's and Bartlett's tests (**Table 2**). In the former and in the latter case, the value was p > 0.05, therefore was no reason to reject the hypothesis of the homogeneity (equality) of variance.

Table 2 Checking the hypothesis of the homogeneity of variance of the given attribute in particular groups

Homogeneity of variance, Levene's test	MS Effects	MS Error	F	p
KCG	13.218	8.122	1.627	**0.0773**
Bartlett's test of homogeneity of variance	**Bartlett Chi-kw.**	**df**	**p**	
KCG	27.6	23.00	**0.2304**	

The next stage included the variance analysis of factorial systems (**Table 3**).

Table 3 The results of the variance analysis of factorial systems

Effects	SS	Degrees of Freedom	MS	F	p
Free term	350240.6	1	350240.6	10047.04	0.0000
$T_{A'}$	3040.4	1	3040.4	87.22	**0.0000**
T_{AF}	29010.3	1	29010.3	832.19	**0.0000**
t_{AF}	6915.2	5	1383.0	39.67	**0.0000**
$T_{A'} \cdot T_{AF}$	849.2	1	849.2	24.36	**0.0000**
$T_{A'} \cdot t_{AF}$	53.2	5	10.6	0.30	**0.9076**
$T_{AF} \cdot t_{AF}$	826.6	5	165.3	4.74	**0.0013**
$T_{A'} \cdot T_{AF} \cdot t_{AF}$	152.3	5	30.5	0.87	**0.5056**
Error	1673.3	48	34.9		

SS -is for Sum of Squares Within groups; MS - is for Mean Square Within groups

Additionally, a Tukey's post-hoc test was performed, the results of which are shown in **Table 4**.

Table 4 Results of inter-class comparisons according to the Tukey's post-hoc test

No. subclass	Factors			No. Subclass											
	$T_{A'}$	T_{AF}	t_{AF}	1	2	3	4	5	6	7	8	9	10	11	12
1	800°C	300°C	7,5	p =	1,0000	0,6590	0,0146	0,0615	0,0111	0,0154	0,0002	0,0006	0,1966	0,9893	0,9944
2	800°C	300°C	15	1,0000	p =	0,9893	0,1349	0,3801	0,0877	0,0012	0,0002	0,0002	0,0238	0,6590	0,7736
3	800°C	300°C	30	0,6590	0,9893	p =	0,9771	0,9998	0,8773	0,0002	0,0002	0,0002	0,0003	0,0204	0,0532
4	800°C	300°C	60	0,0146	0,1349	0,9771	p =	1,0000	1,0000	0,0002	0,0002	0,0002	0,0002	0,0002	0,0006
5	800°C	300°C	120	0,0615	0,3801	0,9998	1,0000	p =	1,0000	0,0002	0,0002	0,0002	0,0002	0,0005	0,0021
6	800°C	300°C	240	0,0111	0,0877	0,8773	1,0000	1,0000	p =	0,0002	0,0002	0,0002	0,0002	0,0003	0,0005
7	800°C	400°C	7,5	0,0154	0,0012	0,0002	0,0002	0,0002	0,0002	p =	0,0029	1,0000	1,0000	0,5914	0,8263
8	800°C	400°C	15	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,0029	p =	0,0671	0,0003	0,0002	0,0002
9	800°C	400°C	30	0,0006	0,0002	0,0002	0,0002	0,0002	0,0002	1,0000	0,0671	p =	0,8945	0,0720	0,2161
10	800°C	400°C	60	0,1966	0,0238	0,0003	0,0002	0,0002	0,0002	1,0000	0,0003	0,8945	p =	0,9928	0,9993
11	800°C	400°C	120	0,9893	0,6590	0,0204	0,0002	0,0005	0,0003	0,5914	0,0002	0,0720	0,9928	p =	1,0000
12	800°C	400°C	240	0,9944	0,7736	0,0532	0,0006	0,0021	0,0005	0,8263	0,0002	0,2161	0,9993	1,0000	p =
13	850°C	300°C	7,5	1,0000	0,9967	0,2392	0,0020	0,0097	0,0019	0,0914	0,0002	0,0042	0,5914	1,0000	1,0000
14	850°C	300°C	15	1,0000	0,9971	0,2458	0,0021	0,0101	0,0019	0,0884	0,0002	0,0041	0,5817	1,0000	1,0000
15	850°C	300°C	30	1,0000	1,0000	0,5914	0,0110	0,0478	0,0086	0,0204	0,0002	0,0008	0,2392	0,9948	0,9973
16	850°C	300°C	60	0,7758	0,9974	1,0000	0,9409	0,9984	0,7954	0,0002	0,0002	0,0002	0,0004	0,0336	0,0800
17	850°C	300°C	120	0,0929	0,4898	1,0000	1,0000	1,0000	0,9999	0,0002	0,0002	0,0002	0,0002	0,0008	0,0032
18	850°C	300°C	240	0,0110	0,1235	0,9858	1,0000	1,0000	1,0000	0,0002	0,0002	0,0002	0,0002	0,0002	0,0004
19	850°C	400°C	7,5	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,0011	1,0000	0,0273	0,0002	0,0002	0,0002
20	850°C	400°C	15	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,0695	0,0002	0,0002	0,0002	0,0002
21	850°C	400°C	30	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,0068	1,0000	0,1328	0,0004	0,0002	0,0002
22	850°C	400°C	60	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,1672	0,9974	0,8230	0,0124	0,0002	0,0009
23	850°C	400°C	120	0,0003	0,0002	0,0002	0,0002	0,0002	0,0002	0,9985	0,1481	1,0000	0,7149	0,0300	0,1128
24	850°C	400°C	240	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,9717	0,1670	1,0000	0,4008	0,0058	0,0364

No. Subclass	Factors			No. Subclass											
	$T_{A'}$	T_{AF}	t_{AF}	13	14	15	16	17	18	19	20	21	22	23	24
1	800°C	300°C	7,5	1,0000	1,0000	1,0000	0,7758	0,0929	0,0110	0,0002	0,0002	0,0002	0,0002	0,0003	0,0002
2	800°C	300°C	15	0,9967	0,9971	1,0000	0,9974	0,4898	0,1235	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002
3	800°C	300°C	30	0,2392	0,2458	0,5914	1,0000	1,0000	0,9858	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002
4	800°C	300°C	60	0,0020	0,0021	0,0110	0,9409	1,0000	1,0000	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002
5	800°C	300°C	120	0,0097	0,0101	0,0478	0,9984	1,0000	1,0000	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002
6	800°C	300°C	240	0,0019	0,0019	0,0086	0,7954	0,9999	1,0000	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002
7	800°C	400°C	7,5	0,0914	0,0884	0,0204	0,0002	0,0002	0,0002	0,0011	0,0002	0,0068	0,1672	0,9985	0,9717
8	800°C	400°C	15	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	1,0000	0,0695	1,0000	0,9974	0,1481	0,1670
9	800°C	400°C	30	0,0042	0,0041	0,0008	0,0002	0,0002	0,0002	0,0273	0,0002	0,1328	0,8230	1,0000	1,0000
10	800°C	400°C	60	0,5914	0,5817	0,2392	0,0004	0,0002	0,0002	0,0002	0,0002	0,0004	0,0124	0,7149	0,4008
11	800°C	400°C	120	1,0000	1,0000	0,9948	0,0336	0,0008	0,0002	0,0002	0,0002	0,0002	0,0002	0,0300	0,0058
12	800°C	400°C	240	1,0000	1,0000	0,9973	0,0800	0,0032	0,0004	0,0002	0,0002	0,0002	0,0009	0,1128	0,0364
13	850°C	300°C	7,5	p =	1,0000	1,0000	0,3342	0,0158	0,0013	0,0002	0,0002	0,0002	0,0002	0,0016	0,0003
14	850°C	300°C	15	1,0000	p =	1,0000	0,3423	0,0164	0,0014	0,0002	0,0002	0,0002	0,0002	0,0015	0,0003
15	850°C	300°C	30	1,0000	1,0000	p =	0,7149	0,0732	0,0081	0,0002	0,0002	0,0002	0,0002	0,0004	0,0002
16	850°C	300°C	60	0,3342	0,3423	0,7149	p =	0,9997	0,9567	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002
17	850°C	300°C	120	0,0158	0,0164	0,0732	0,9997	p =	1,0000	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002
18	850°C	300°C	240	0,0013	0,0014	0,0081	0,9567	1,0000	p =	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002
19	850°C	400°C	7,5	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	p =	0,1551	1,0000	0,9709	0,0660	0,0714
20	850°C	400°C	15	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,1551	p =	0,0330	0,0010	0,0002	0,0002
21	850°C	400°C	30	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	1,0000	0,0330	p =	0,9999	0,2662	0,3056
22	850°C	400°C	60	0,0002	0,0002	0,0002	0,0002	0,0002	0,0002	0,9709	0,0010	0,9999	p =	0,9515	0,9796
23	850°C	400°C	120	0,0016	0,0015	0,0004	0,0002	0,0002	0,0002	0,0660	0,0002	0,2662	0,9515	p =	1,0000
24	850°C	400°C	240	0,0003	0,0003	0,0002	0,0002	0,0002	0,0002	0,0714	0,0002	0,3056	0,9796	1,0000	p =

The tests of the impact of heat treatment on ADI cast iron impact resistance and the statistical analysis of the results enabled the description of the mechanical property with the following equation (2):

$$KCG = T_{A'} \cdot 0.2721 + T_{AF} \cdot 0.4102 - t_{AF} \cdot 0.0915 - 290, \frac{J}{cm^2} \quad (2)$$

For the specified equation, a correlation coefficient was determined, the value of which was R=0.9298, when the determination coefficient was R^2=0.8646. This proves a strict correlation between the dependencies examined. A correct statistical evaluation with this method requires a rest distribution similar to normal distribution. For confirmation, a rest normality plot was plotted for a multi-coefficient regression function, which is shown in **Figure 6**.

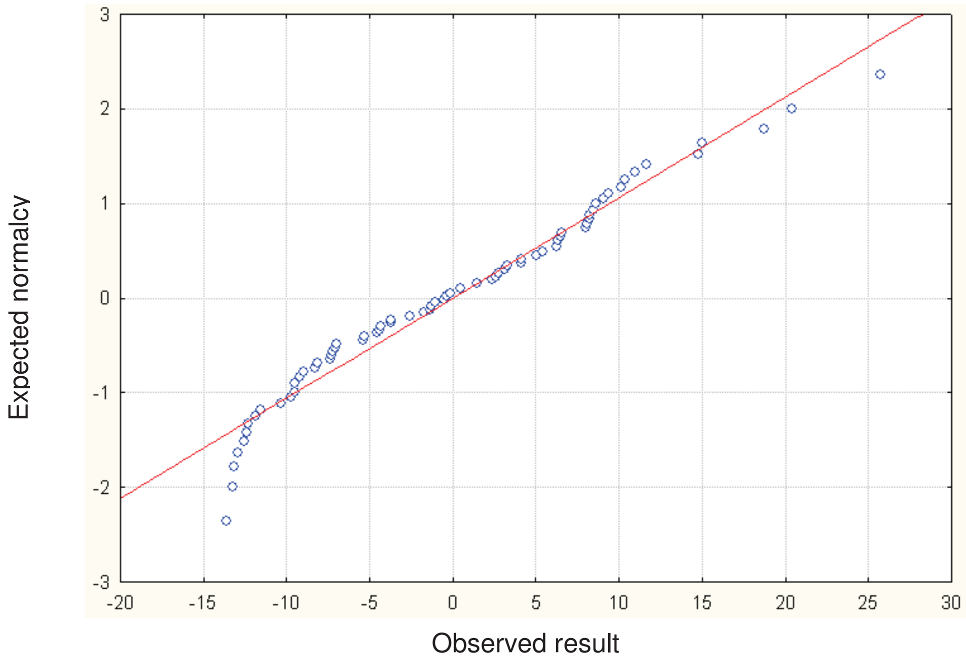

Figure 6 Rest normality plot for a multi-coefficient regression function

Very good equation alignment (2) is also confirmed by the predicted values vs observed values plot (**Figure 7**).

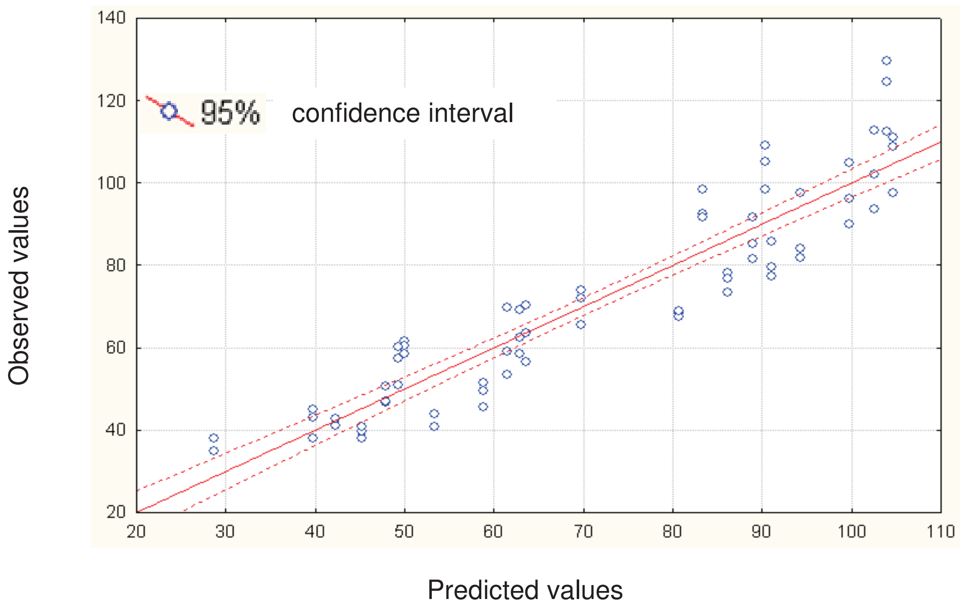

Figure 7 Predicted values vs observed values plot

4. CONCLUSION

By analysing only linear factors (without interaction), it is possible to ascertain that the first parameter, i.e. the ausferritizing temperature increase (T_{AF}), and the second parameter, i.e. austenitizing temperature after cooling down ($T_{A'}$), has a statistically significant impact on the increase of the impact resistance. The impact of the second parameter on the impact resistance is almost three times lower than of the first parameter. Extending the ausferritizing time (t_{AF}) causes the impact resistance to lower. Isothermal transformation hardening from a lower austenitizing temperature still ensures a relatively high strength, but also an exceptionally high impact resistance, above the minimum values specified in PN-EN 1564:2012 [11]. For example, the minimum value required for the EN-GJS 1400-1 grade is 35 J/cm^2. Very favourable heat treatment effects result from the formation of an ausferritic matrix in the entire eutectic grains area.

ACKNOWLEDGEMENTS

I would like to sincerely thank Jan Szymszal, Professor of the Silesian University of Technology, PhD. Eng., for his help in the statistical analysis of the results.

REFERENCE LIST

[1] PODRZUCKI C., Żeliwo. Struktura, własności, zastosowanie, t.I i II, Wyd. ZG STOP, Kraków, 1991.

[2] DYMSKI S., Analysis of the influence of austempering parameters on selected mechanical properties of ADI, Archives of Foundry Engineering, 2004, Vol. 4, Book 12, PAN - Katowice, PL ISSN 1642-5308, pp. 155 - 166.

[3] DYMSKI S., ŁAWRYNOWICZ Z., GIĘTKA T., Impact strength of ADI, Archives of Foundry Engineering, 2006, Vol. 6, No. 21 (1/2), PAN - Katowice, PL ISSN 1642-5308, pp. 369 - 376.

[4] GIĘTKA T., CIECHACKI K., SZYKOWNY T., The influence of temperature breaking on impact strength of ADI, Archives of Foundry Engineering, Vol. 15, Special Issue 2/2015 ISSN (1897-3310), pp. 87 - 92.

[5] BATRA U., RAY S., PRABHAKAR S.R., Impact properties of copper-alloyed and nickel-copper alloyed ADI. Journal of Materials Engineering and Performance, Vol. 16, Issue 4, 2007, pp. 485 - 489.

[6] DELLA, M., ALAALAM, M., GRECH, M., Effect of Austenitizing Conditions on the Impact Properties of an Alloyed Austempered Ductile Iron of Initially Ferric Matrix Structure, Journal of Materials Engineering and Perfomance, Vol. 7, 1998, pp. 265 - 272.

[7] SOKOLNICKI M., GUZIK E., Austempered Ductile Iron to Work in the Conditions of Dynamic Load, Archives of Foundry Engineering, Vol.14, Special Issue 4/2014 ISSN (1897-3310), pp. 115 - 118.

[8] OLEJARCZYK-WOŻEŃSKA I., ADRIAN A., ADRIAN H., MRZYGŁÓD B., Parametric representation of TTT diagrams of ADI cast iron, Archives of Metallurgy and Materials, Vol. 57, No. 2, 2012, pp. 613-617.

[9] OLEJARCZYK-WOŻEŃSKA I., ADRIAN A., ADRIAN H., MRZYGŁÓD B., Algorithm for controllingthe quench hardening process of constructional steels, Archives of Metallurgy and Materials, Vol. 55, No. 1, 2010, pp. 171-179.

[10] MALIŃSKI M., SZYMSZAL J., Współczesna statystyka matematyczna w medycynie w arkuszach kalkulacyjnych. Wyd. Śląskiej Akademii Medycznej. Katowice 1999.

[11] PN-EN 1564:2012, Founding - Ausferritic spheroidal graphite cast Irons.

[12] KRZYŃSKA A., KOCHAŃSKI A., Austenitization of ferritic ductile iron, Archives of Foundry Engineering, Vol. 14, Issue 4/2014 ISSN (2299-2944), pp. 49 - 54.