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Prediction of Mechanical Properties of AlSi13Cu2Fe Alloy Using the ATND Methods

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The present paper describes an experimental testing method connected with use of the thermal-voltage-derivative analysis (the ATND method in short) to crystallization recording of the AlSi13Cu2Fe eutectic alloy in aspects of possible prediction of its mechanical properties, basing on dependencies obtained from regression analysis, performed on the basis of temperature and voltage values of characteristic points seen on curves from the ATND method. Results of the experiment have demonstrated a significant relation between values of the characteristic points from the ATND method and the mechanical properties (yield strength $R_{0,2}$ and hardness HB 10/1000/30). Moreover, obtained dependencies have enabled prediction of the yield strength $R_{0,2}$ and the hardness HB 10/1000/30 as early as at preparation stage (melting) of the investigated alloy, what allows quick correction of performed modification process.

Keywords: *Alloy, mechanical properties, ATND, regression analysis*

1. Introduction

Aluminum–silicon (Al-Si) alloys belong to important group of casting alloys. Such alloys are characterized by good castability, relatively low melting temperature, corrosion resistance, high strength to specific weight ratio (greater than steel), amongst other things. Therefore, these alloys find their application in the automotive industry, machinery and household industries, as well as in military and aviation applications. Their mechanical properties are directly affected by shape, size and distribution of silicon crystals, aluminum grain size, as well as shape and parameters of dendrite, which are considered as the main factors influencing application field of the Al-Si alloys in production of the castings [1-7].

The coarse structure of the alloy, disadvantageous from technical point of view, having adverse effect on the mechanical properties of the castings, can be effectively eliminated by use of modification processes. The process of chemical modification comprises adding small quantities of chemical elements (the most often - Na, Sr, Sb) to the melt, what changes coarse and large needles of silicon into a fine and well-rounded form [8-14], advantageously influencing the mechanical properties, mainly the plasticity.

Therefore, conscious control of the technological parameters to optimize obtained structure of the material, and hence to adaptation of material properties to a specific requirements, emerges as an important factor leading to quality improvement of the castings. Thermal analysis consisting in recording of temperature during crystallization of the alloy belongs to one from the most common methods used for assessment and estimation of the grain size, extent of the alloy's modification, and temperature of phase transformations of the alloy[15-18].

In many cases, however, a simple thermal analysis is not sensitive enough to describe the thermal processes being in progress, and in such cases a simultaneously plotted curve is used, which is considered more helpful. The curve illustrates the derivative dT/dt, what enhances less visible changes present on the cooling curve T = f(t) [19] (method of the thermal-derivative analysis – ATD method). Except such methods, other methods based on thermal effects (DTA, ATDG method) [20-22], thermo-electric effects (ATD-AED method) [23], and thermo-voltage effects (ATND method) are also used [24-29].

The ATND (thermal- voltage- derivative analysis) method is the author's own method of analysis of the crystallization process. This method was developed as a combination of the ATD method and measurement of the voltage present during crystallization and phase transformations of the casting alloys, complementing information about structural transformations of the alloy. The testing probes measure differences of electric potential within volume of the test piece. The voltage arisen during crystallization of the test piece is small but well measurable (included within range of ± 5 mV). The phenomenon of the electric voltage present during the crystallization process belongs to the cumulative electric effect connected with changes of the state of aggregation. Combination of the above mentioned methods gives a distinct picture of the changes occurring during crystallization and solidification of the alloy. The generated voltage and temperature are recorded during the process. The course of the crystallization process is presented in form of diagrams, created quickly and accurately during solidification of the alloy, reflecting structural changes of the alloy during the solidification (formation of phases and eutectic mixtures).

At the current stage of the research, such method is used under laboratory conditions to record the crystallization processes in aluminum casting alloys, to identify phase transformations [24,27-28], and to predict the mechanical properties [25,26,29].

The main topic of the present investigation is prediction of the mechanical properties (yield strength $R_{0,2}$ and hardness HB 10/1000/30) of the AlSi13Cu2 alloy with the use of the ATND method, providing information on properties of the alloy as early as at the stage of its preparation, what is especially important in case of performed modification, aimed at improvement of the mechanical properties of the alloy.

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2. Experimental procedure

The AlSi13Cu2Fe alloy is classified as a multi-component alloy used in the foundry industry. This alloy is graded as a near eutectic alloy (silicon, as the alloying element is present in a quantity near to eutectic). The chemical composition of the alloy is presented in Table 1.

The analysis of the chemical composition was performed with use of emission spectroscopy method (spectrometer of the ARL 3440 type).

To prepare test pieces for the testing, the alloys were melted in an electric resistance furnace at temperatures of about 760 - 780 °C. The next treatment consisted in refining of the melt by the Rafal 1 flux. This operation entails the treatment of aluminum alloys (or magnesium alloys) with the degassing flux at 730°C for 5 min. The flux is based on hexachloroethane. The flux is in the form of 50-g pellets submerged on the bottom of the metal bath and is used in quantity corresponding to 0.4% of the weight of the melt [30]. Refined alloys, after removal of oxides and slag from the surface of the liquid metal, were modified with the AlSr10 master alloy in quantity of 0.6% mass of charge (0.06% Sr) at temperature of 780 ^{o}C.

The modified alloy was poured into a metal mould intended for production of standardized castings of the test pieces used to determination of the mechanical properties. The metal mould was adapted to controlling of the crystallization processes with use of the ATND method (Fig. 1). The metal mould was heated to temperature of 250 ^{o}C.

After removal of the sink head and the gating system from cylindrical part of the casting having diameter of 15 mm and length of 180 mm, it has been produced the test pieces with threaded heads, having diameter of 6 mm (Fig. 2).

The static tensile test was performed on the Schenck PSB 100 testing machine according to the PN-EN ISO 6892-1:2010 standard [31]. Recording of strain of the test piece under load was performed with use of the Schenck-DSA 25/10M extensometer with measuring base of 24,9 mm and measuring range of ±2,5 mm. The test was performed with constant displacement speed of the cross-beam of the tester equal to $1,6x10^{-5}s^{-1}$. The Brinell hardness measurement according to the PN-EN ISO 6506-1:2008 standard [32], using the Brinell hardness tester of the PRL 82 type, was carried out using a steel ball with a diameter of 10 mm, under load of 9800 N, sustained for 30 seconds.

The crystallization process of the investigated alloy was recorded with use of the ATND method, with marked characteristic points on the thermal (t_1 - t_3) and voltage (U_1 - U_3) curves, what is shown in the Fig. 3.

Characteristic points on the thermal and voltage curves taken from the ATND method are connected with crystallization of the alloy and phase transformations in solid state, which has direct effect on the structure, and thus on the mechanical properties of the material. During recording of the crystallization process and cooling of the investigated alloy, these points are present in form of a distinct "peaks" on curves of the ATND method.

The ATND method consists in continuous measurement of temperature and electric voltage generated on the probes during crystallization and phase transformations of the solidified alloy. The generated voltage and temperature of the investigated test pieces were recorded in course of the measurement. The course of the crystallization is presented in form of diagrams created during solidification of the alloy.

Based on obtained values of the characteristic points from the ATND method (independent variables) and values of the mechanical properties (dependent variables), computer files were created with input data to regression analysis, performed with use of the Statistica, version 10, packet developed by the StatSoft Company.

The first order polynomial was assumed as a function of the tested object (1).

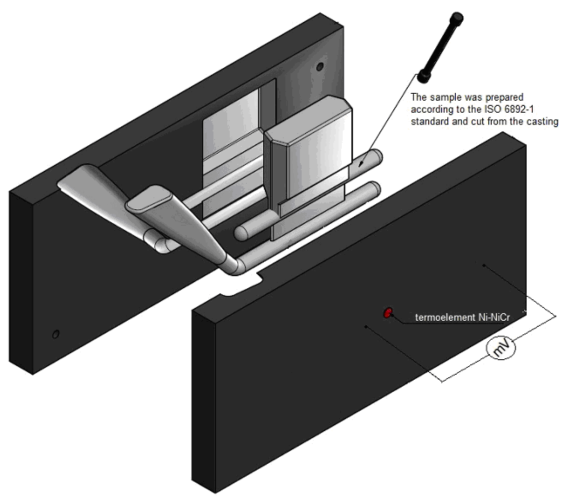

Fig. 1. Metal mould to pouring of the test pieces and controlling of the crystallization process with use of the ATND method

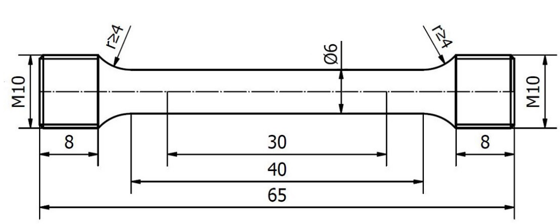

Fig. 2. Test piece

Table 1. Chemical properties of the AlSi13Cu2Fe alloy

Chemical composition (% weight)											
Si	**Fe**	**Cu**	**Zn**	**Ti**	**Mn**	**Ni**	**Sr**	**Pb**	**Cr**	**Mg**	**Al**
11.89	0.805	2.22	0.32	0.018	0.15	0.036	0.045	0.036	0.036	0.163	Balance

$$z = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + ... + b_nx_n \pm \varepsilon \quad (1)$$

where:

z - dependent variable ($R_{0,2}$, HB),

x_1, x_2, ..., x_n - independent variables ($t_1 \div t_3$, $U_1 \div U_3$),

b_0, b_1, ..., b_n - estimator of regression,

ε - standard error of estimation.

3. Description of the obtained results

As result of the regression analysis, the dependencies (2, 4-5) were obtained, which describe effect of the characteristic points values on change of the mechanical properties of the AlSi13Cu2Fe alloy modified with strontium.

3.1. Yield strength $R_{0,2}$

The $R_{0,2}$ value for the modified alloy was included within limits from 125 to 148 MPa. The complete model of the $R_{0,2}$ value is presented by the equation (2).

$$R_{02} = 379.1 - 0.08t_1 - 0.41t_2 + 0.06t_3 - 10.8U_1 + 10.3U_2 + 5.2U_3 \pm 2.29 \text{ [MPa]} \quad (2)$$

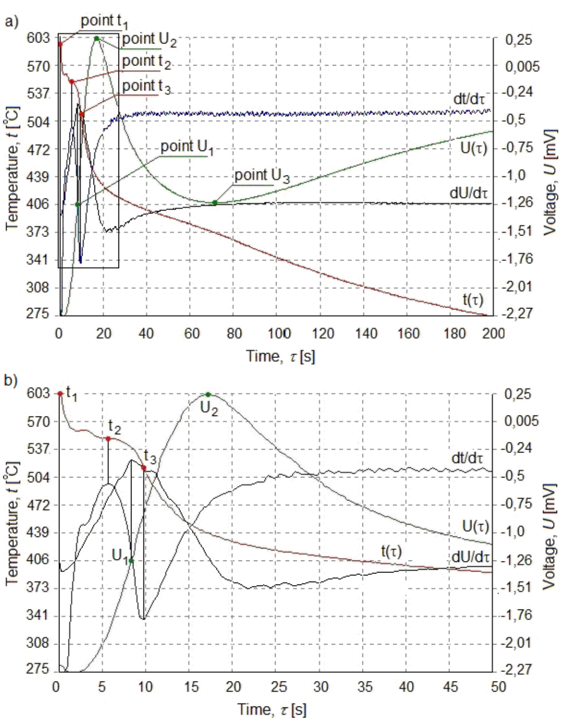

Fig. 3. Curves from the ATND method with marked characteristic points for the investigated alloy: a) complete run of the crystallization process, b) magnification of the marked area [26]

A high determination coefficient R^2=0.92, denoting 92% of general variability of the yield strength $R_{0,2,}$ was explained by this model. The determination coefficient R^2 is understood as ratio of the explained variability to the total varialility. The R^2 is a number from interval <0, 1> ; and the situation is optimal when this number is high. Presenting this ratio in percents we answer the question of what percentage of the variability Y is explained by the variability X. Computer calculated coefficient of linear determination is also expressed by the equation (3) below, as a measure corrected by the number of degrees of freedom, namely [33]:

$$R^2_{corr.} = 1 - \frac{n-1}{n-k-1}\left(1 - R^2\right) \quad (3)$$

where: n- size of the group,

k- number of variables, including the free term

The determination coefficient corr. R^2 amounts to 0.88. The complete model presented here comprises three variables plus the free term only, having significance level of $p < \alpha = 0.05$. Other members are variables having insignificant influence, which did not fulfill the significance condition ($p<\alpha=0.05$). The significance level α is equal to predetermined by us allowable probalibity error, consisting in the fact that we accept obtained result as the true one. In many areas of the research work the value of α=0,05 is accepted as typical limiting value of the significance level. When value p obtained from the test is below this value, the result is assessed as statistically significant. The analysis of variance confirms that from the general sum of squares of the variable $R_{0,2}$ only 8% of variability can not be explained by regression, whereas statistical significance of the independent variables having effect on the dependent variable $R_{0,2}$ is fulfilled at the level of $p = 0.00002$ ($F_{obl} > F_{(\alpha;df1;df2)}$ ($F_{obl} = 21.83 > F_{(0,05;6;11)} = 3.09$).

Test of significance of the F_{obl} model according to scheme of the proceeding presented in the Table 2 constructed as quotient of mean WSK/RSK, serves to assessment of an effect of the independent variable on the dependent variable $F_{obl}>F_{kr}$ where $F_{kr}=F_{(\alpha,df1,df2)}$. It's theoretical distribution has form of the Fisher-Snedecor's distribution [34].

In the complete model, there are present also variables with insignificant influence, which undergo elimination. The analysis was terminated after elimination of three variables: temperature variables - t_1, t_3 and voltage variable - U_3.

The model reduced to value of the yield strength $R_{0,2}$ and represented by the equation (4) comprises five variables (inclusive of the free term), complying with condition of the significance ($p < \alpha = 0.05$).

$$R_{0,2} = 330.2 - 0.36t_2 - 7.08U_1 + 7.58U_2 \pm 2.35 \text{ [MPa]} \quad (4)$$

The determination coefficient R^2 was reduced with respect to the complete model (difference in value of -0.03) and it amounts to 0.89. The corrected coefficient R^2 amounts to

Table 2. ANOVA table

Source of variability	Sums of square deviations	Number of degrees of freedom	Mean square deviations	F test of significance
Explained by regression	WSK	df1 = k-1	WSK/(k-1)	$\frac{WSK/(k-1)}{RSK/(n-k)}$
Remainders	RSK	df2 = n-k	RSK/(n-k)	

where: k- number of variables, including the free term. n- size of the group

0.87 (change of the value is equal to -0.01) and it constitutes a comparative measure for the models with different number of variables. It means, that the obtained model is correct, and four variables were introduced to the model (together with the free term), reducing its structure.

The analysis of variance confirms statistical significance of the aggregate effect of the influence of the independent variables on the dependent variable at residual critical level of $p = 0.00002 < \propto = 0.05$ - $F_{obl} > F_{(\alpha;df1;df2)}$ ($F_{obl} = 29.42 > F_{(0,05;4;13)} = 3.34$). The total influence is not only significant, but also very strong.

The Fig. 4 presents how the values of the yield strength $R_{0,2}$ are shaped, both the predicted and observed (from the test) ones.

To compare the effect of changes of the characteristic points values on the yield strength $R_{0,2,}$ these values were plotted on spatial diagrams representing character of their influence on the dependent variable (Fig. 5). Average values obtained from the measurement (assumed values of constant points) of the characteristic points of the refined and modified AlSi13Cu2Fe alloy were used to plot the diagrams.

Increase of the yield strength $R_{0,2}$ is connected mainly with change of the voltage in the points U_1 and U_2, and to a smaller extent, with decrease of the temperature t_2.

3.2. Hardness HB 10/1000/30

Hardness of the alloy after the modification ranged from 73.5 to 89 HB 10/1000/30. The complete model for the value HB 10/1000/30 is represented by the equation (5).

$$HB = -34 + 0.04t_1 + 0.66t_2 - 0.51t_3 + 0.61U_1 + 1.8U_2 + 2.58U_3 \pm 1.61 \quad (5)$$

In spite of presence of only two variables with a satisfactory level of significance in the equation (5), a high determination coefficient $R^2 = 0.95$ was obtained, which means that as many as 95% of total variability of the HB was explained by the model. The corrected determination coefficient, corr. R^2 amounts to 0.92. Statistic significance of the effect of influence of the independent variables on the dependent variable HB is fulfilled at $p = 0.000001 < \alpha = 0.05$ ($F_{obl} > F_{(\alpha;df1;df2)}$) ($F_{obl} = 36.23 > F_{(0,05;6;11)} = 3.09$).

Elimination of the variables with insignificant influence was terminated after removal of two variables. Reduced model for the hardness HB 10/1000/30 was represented by the equation (6).

$$HB = -38.6 + 0.78t_2 - 0.58t_3 + 2.85U_2 + 3.62U_3 \pm 1.63 \quad (6)$$

Performed elimination of the variables with insignificant influence did not have any considerable effect on changes

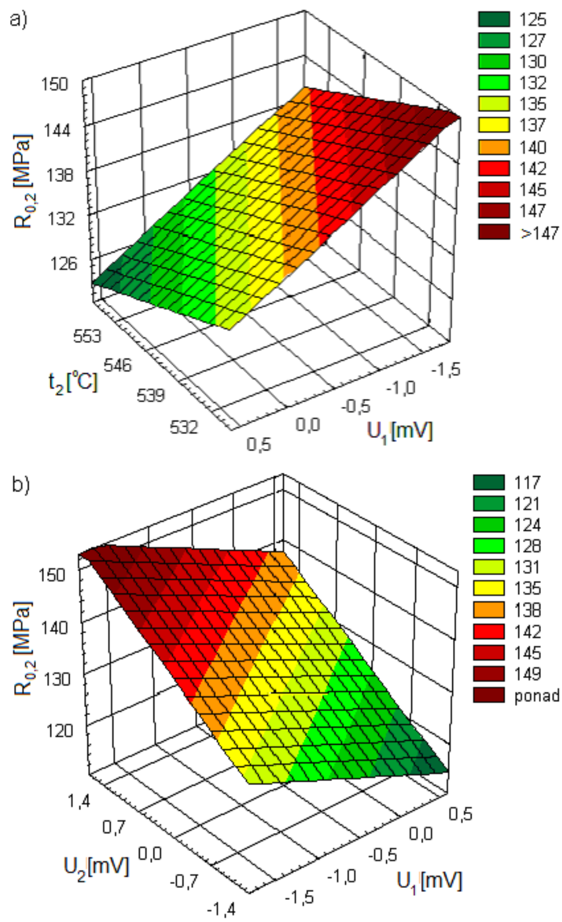

Fig. 5. Effect of change of values of characteristic points: a) t_2, U_2, and b) U_2, U_1 on the yield strength $R_{0,2}$

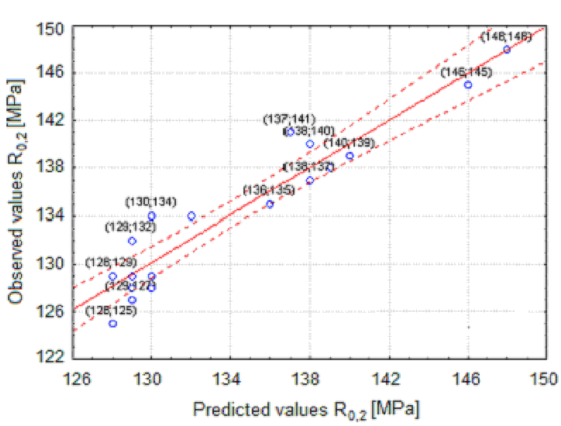

Fig. 4. Diagram of predicted and observed values for the variable $R_{0,2}$

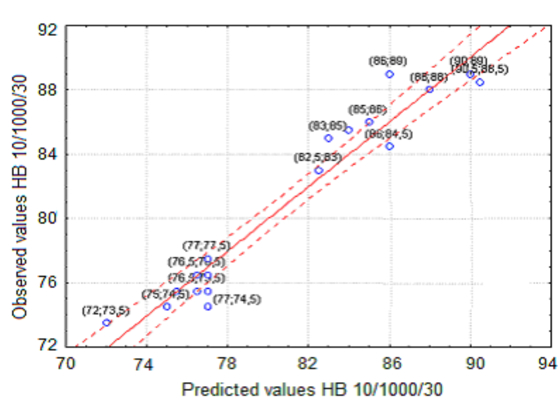

Fig. 6. Diagram of predicted and observed values for the variable HB 10/1000/30

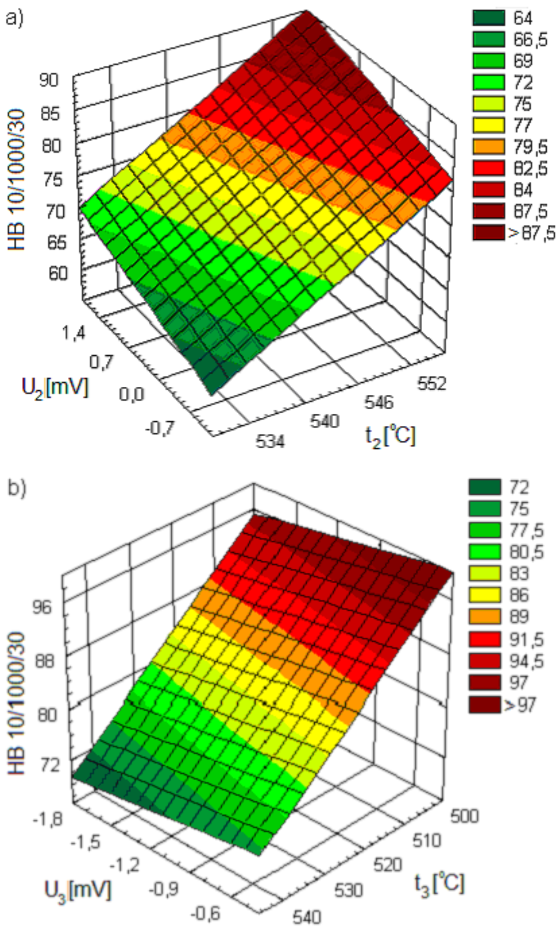

Fig. 7. Effect of change of values of characteristic points: a) U_2, t_2 and b) U_3, t_3 on the hardness HB 10/1000/30

of the determination coefficient R^2 (change with - 0.01). The coefficient corr. R^2 remained unchanged. In the equation (6) there are present five variables (together with the free term), from which only the free term exceeded critical level of significance $\propto = 0.05$. The analysis of variance confirms that only 6% of the variability cannot be explained by regression. Moreover, it has been confirmed significant influence of the independent variables on the dependent variable HB at residual critical level p = 0.000001 < α = 0.05 ($F_{obl} > F_{(\alpha;df1;df2)}$ ($F_{obl} = 52.5 > F_{(0,05;4;13)} = 3.17$).

The Fig. 6 presents a system of predicted and observed values of the dependent variable HB 10/1000/30.

To ilustrate how individual variables influence on change of the hardness HB 10/1000/30, in the Fig. 7 in form of spatial diagram are shown direction and 'intensity' of influence of the temperature and the voltage variables.

Increase of the tempearture t_2 and decrease of the temperature t_3 result in the increased hardness HB 10/1000/30.

4. Conclusions

Obtained results of the research have confirmed correlation between values of temperature and voltage of the characteristic points seen on the curves from the ATND method and the mechanical properties (yield strength $R_{0,2}$ and HB 10/100/30 hardness) of the AlSi13Cu2Fe alloy modified with strontium.

The obtained dependencies (4 and 6) enable prediction of the mechanical properties of the investigated alloy under laboratory conditions at significance level of α = 0.05, which allows quick quality control of the alloy as early as at its preparation stage.

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