

SHAPING THE FRACTURE RESISTANCE OF A PA6 COMPOSITE WITH 25 % GLASS FIBER CONTENT BY SELECTING INJECTION PARAMETERS

Dariusz Kwiatkowski, Jacek Nabialek*

*Department of Technology and Automation, Faculty of Mechanical Engineering
Czestochowa University of Technology
Częstochowa, Poland
dariusz.kwiatkowski@pcz.pl, jacek.nabialek@pcz.pl*

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Abstract. This article presents a statistical analysis of the effect of selected injection molding conditions on fracture toughness, measured using the stress intensity factor (WIN). A composite based on polyamide 6 with 25 % short E-glass fiber content was tested. Samples for SENB (Single Edge Notch Bend) tests were made using a double-cavity injection mold. To determine the relationship between the selected injection molding conditions and fracture toughness, SENB test samples were manufactured based on a developed test plan. The plan was prepared using literature on the theory of experimental design and the Design of Experiment (DoE) module of the statistical data analysis software STATISTICA by StatSoft. The design used is a central composite plan with a rotation coefficient of $\alpha = 1.6818$. Each input quantity can occur at five levels, represented by the quantities $-\alpha$, -1 , 0 , $+1$, and $+\alpha$. The extreme values of the design ($-\alpha$ and $+\alpha$) are the star points, $(-1, +1)$ are the principal points, and point (0) is the center point of the test plan. The design consists of 18 different configurations, according to which test samples were manufactured. A Pareto analysis was performed to estimate the importance of individual injection conditions in the model equation (regression). The results, including the effect of selected injection parameters on the stress intensity factor in PA6 composites with glass fiber, are presented graphically. The test results were analyzed and conclusions were drawn.

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1. Introduction

In contemporary product quality assurance methods, there is a clear tendency to integrate quality-related activities into a wide range of technological and organizational processes. Quality is understood as the degree to which expectations regarding

* Corresponding author

the value of a given characteristic are met, such as dimensional and geometric accuracy, surface finish, or crack resistance. Quality-related activities should ensure the stability of these characteristics in the presence of potential disturbances. Classical Statistical Process Control (SPC) methods are limited to detecting process irregularities that lead to a deterioration in product quality. These methods are characterized by their lack of responsiveness to disturbances that destabilize the manufacturing process. Another important aspect of quality-related activities is the pursuit of maximum product durability and reliability [1-3].

Actions aimed at improving the quality of the technological process may include [4-6]:

- ✓ determining the technological process conditions under which the quality characteristics attain optimal values,
- ✓ identifying process conditions that effectively reduce the impact of disturbances while maintaining the established or permissible values of the quality characteristics,
- ✓ establishing manufacturing conditions that ensure the stability of quality characteristics in the presence of potential disturbances.

The above-mentioned classification is not complete. It is possible to combine many of the above-mentioned criteria to determine optimal quality characteristics and reduce their dispersion.

The above classification is not exhaustive. It is possible to combine several of the listed criteria to determine optimal quality characteristics and reduce their dispersion. Any procedure for the optimal selection of technological parameters based on an adopted quality criterion requires formulating a mathematical model that quantitatively describes the quality characteristics of the technological process. By determining the optimal conditions for conducting the technological process according to the selected quality criterion Q , the objective is to obtain a static model of the relationship $Q(x, p, z)$ and, on this basis, determine the coordinates and values of the desired extremum [7]:

$$Q = Q(x, p, z) \quad (1)$$

where:

- x – a set of independent variables influencing the quality characteristics and the course of the technological process,
- p – constant values characterizing the technological process,
- z – disturbances.

To simplify relation (1), we assume that the disturbances z are stationary. Under this assumption, the quality characteristic Q can be expressed as follows [7-9]:

$$Q = Q(x, p) \quad (2)$$

or if the influence of the constant values on the technological process is ignored:

$$Q = Q(x) \quad (3)$$

Depending on the method used to determine the value of the objective function, the number of independent and response variables, and the adopted mathematical model of the process, appropriately selected analytical, numerical, or experimental methods – or their combinations – are applied to define the operating conditions of the process. The problem of optimal selection of technological parameters based on the adopted quality criterion can be addressed using two different mathematical modeling approaches [10].

The first method relies on an in-depth analysis of the process under study, describing the theoretical foundations of the physical phenomena and formulating algebraic, differential, and integral equations, or their combinations. Solving these equations typically leads to an analytical form of the desired functional relationship. The fundamental drawback of this approach is the complex mathematical structure of the analyzed equations, which directly affects the efficiency of the research. Moreover, the resulting solution describes the internal state of the process rather than the desired relationship itself. In such cases, additional steps are required to establish a relationship between the control variables x and the values of the quality characteristics $Q(x)$.

The second method for modeling quality characteristics involves determining the most accurate approximation of the $Q(x)$ relationship based on a finite number of experimental studies (Response Surface Method). The function $Q(x)$ is often referred to as the response function, and its graphical representation is known as the response surface. This method is based on regression analysis. The main drawback of this approach is the lack of information about the analytical form of the $Q(x)$ relationship. It is assumed that the function under study is continuous, differentiable, and expresses a cause-and-effect relationship between the control variables x and the quality characteristic. Optimization of the injection molding process can also be performed using computational techniques, including neural networks, genetic algorithms, and fuzzy systems [11-13].

To better understand the effect of injection molding conditions on fracture toughness, experiments were conducted based on the theory of experimental design. Experimental design theory is a broad and continuously evolving field of knowledge and an indispensable tool in scientific research. It is particularly useful when the research involves a large number of variables. Using standard experimental designs, it is possible to locally approximate the desired function with a regression model, assuming that injection molding parameters and fracture toughness are causally related. The validity of this assumption is formally verified using statistical tests, with fracture toughness typically approximated by first- or second-degree polynomials [14].

To simplify the problem, it is assumed that the desired relationships are static. This condition is satisfied because the injection molding process of thermoplastics is, in most cases, a stable technological process. The injection molding process is characterized by a large number of quality characteristics, and the influence of design factors and processing conditions on their values is often contradictory. It is

also important to note the complex nature of the physical phenomena occurring during the injection molding process [15-17].

The purpose of multivariate regression analysis is to quantify the relationship between fracture toughness and the independent variables. Selecting the appropriate form of the model equations is a crucial task. This function may represent a simple linear relationship or take a more complex form by incorporating power terms and interaction effects between factors. The general form of such a model, containing linear, quadratic, and interaction terms, is expressed by the following equation [3]:

$$Q = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{44} x_4^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{14} x_1 x_4 + \beta_{23} x_2 x_3 + \beta_{24} x_2 x_4 + \beta_{34} x_3 x_4 \quad (4)$$

where:

β_0 – displacement coefficient (intercept),

β_i – regression coefficient of the quality characteristic Q with respect to x_i .

In the analyzed injection molding process, the individual input variables are designated as follows:

- injection temperature: $T_w = x_1$,
- mold temperature: $T_f = x_2$,
- packing pressure: $p_d = x_3$,
- injection speed: $v_w = x_4$.

Therefore, equation (4) takes the following final form:

$$Q = \beta_0 + \beta_1 T_w + \beta_2 T_f + \beta_3 p_d + \beta_4 v_w + \beta_{11} T_w^2 + \beta_{22} T_f^2 + \beta_{33} p_d^2 + \beta_{44} v_w^2 + \beta_{12} T_w T_f + \beta_{13} T_w p_d + \beta_{14} T_w v_w + \beta_{23} T_f p_d + \beta_{24} T_f v_w + \beta_{34} p_d v_w \quad (5)$$

Based on the estimation results, further steps may be taken to reduce the number of variables describing the studied response, which leads to model simplification. However, the value of the coefficient of determination R^2 should be verified each time. If its value decreases significantly as a result of model simplification, further reductions should be discontinued. Based on the fracture toughness measurements, the regression coefficients of the model equation were determined for each tested polymer composite. The obtained solutions were then evaluated to verify the correctness of the regression model. If the distribution of residuals deviated from normality, an additional term expressing the curvature effect was introduced into the regression equation, after which the calculation procedure was repeated.

To optimize the injection process with respect to increasing crack resistance, four independent variables were selected from among several injection molding parameters: injection temperature T_w [°C], mold temperature T_f [°C], injection speed v_w [mm/s], and packing pressure p_d [MPa]. When determining the ranges of variation for the input variables, in addition to tests performed on an injection molding machine using a properly prepared mold, data, and recommendations provided by the composite manufacturer were also taken into account [18] (Table 1).

To determine the relationship between the selected injection conditions and fracture toughness, fracture toughness tests were conducted according to a developed experimental plan. The applied design was a central composite design with a rotation coefficient $\alpha = 1.6818$. The design consisted of 18 experimental runs. A “grid” of input variables (T_w , T_f , p_d , v_w) was created by dividing the range of each input parameter into 10 intervals, and the output variable K_{Ic} was calculated using the obtained regression equations. The output variable was then assigned an appropriate utility value U (from $U = 0$ – useless, to $U = 1$ – useful). During optimization, the value $U = 1$ was assigned to the maximum value of K_{Ic} , and $U = 0$ to the minimum value. Within the interval between these extremes, utility changed linearly.

The procedure for optimal selection of input variables was carried out using the Design of Experiment (DoE) module of the STATISTICA statistical data analysis software.

2. Materials, apparatus and research methodology

A polyamide 6 composite with 25 % glass fiber content, trade named RESTRAMID B27 25GF, manufactured by Polimarky, was used for fracture toughness testing. Samples for fracture toughness testing were injection molded using a Krauss Maffei KM 65-160 injection molding machine. The injection molding machine is equipped with a high-end C4 control system. To produce the fracture toughness test samples, a mold insert was manufactured for the injection mold. The mold insert allows for the injection of SENB (Single Edge Notch Bend) samples in the shape of a cuboid beam measuring $80 \text{ mm} \times 10 \text{ mm} \times 4 \text{ mm}$ with a rectangular notch in the center. The insert consists of a matrix molding plate with cooling channels, ejector pins, and a handle for efficient insertion into the housing. A plate called the molding insert was also manufactured, in which the elements forming the sample notch are pressed into place. The mold temperature was set using a Wittmann Temprow Plus 140 thermostat.

3. Analysis and discussion of the results

Residual analysis plays an important role in assessing the adequacy of the fitted model. Residuals represent the differences between the experimentally determined values of the stress intensity factor and the corresponding values calculated from the model equation. In the first stage of the analysis, an attempt was made to keep the model equation as simple as possible; however, this approach did not always yield satisfactory results. For the PA6 composite with 25 % glass fiber content, the model equation must take a more complex form that includes linear, quadratic, and interaction terms.

Figure 1 shows the dependence of the expected normal value on the residuals of the fracture toughness model for PA6 with 25 % glass fiber content. The vast

majority of points lie along a straight line, indicating a good fit of the model equation to the experimental fracture toughness data for this material. The model is characterized by a very high Pearson's correlation coefficient ($R = 0.99788$) and a coefficient of determination ($R^2 = 0.99748$), confirming its excellent fit to the experimental data. In addition, the experimental plan consisting of 18 runs is statistically sufficient, as it results from a reduced three-level factorial design. This fractional design ensures model identifiability, enables reliable estimation of interaction effects, and provides an adequate number of degrees of freedom for a robust assessment of the model's predictive quality.

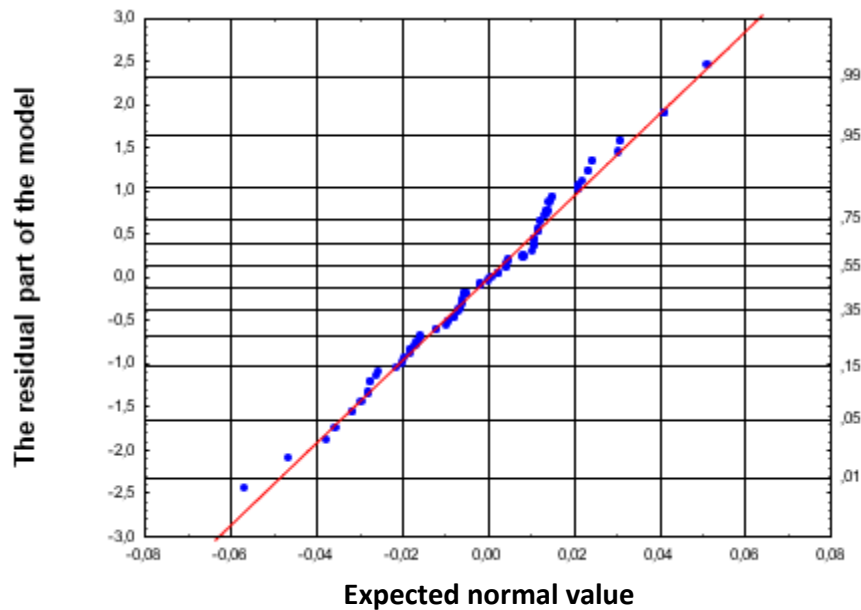


Fig. 1. Expected normal value vs. residuals of fracture toughness model for PA6 with 25 % glass fiber content

Example values of the calculated regression coefficients β_{ij} of the model equation for PA6 with 25 % glass fiber content are presented in Table 1. After substituting these coefficients into the model equation, the fracture toughness of the PA6 composite with 25 % glass fiber content can be determined at any point within the space defined by the input variables: injection temperature, mold temperature, injection speed, and packing pressure.

Example results of the analysis of variance for the fracture toughness model of PA6 with 25 % glass fiber content are shown in Table 2. The table includes the key ANOVA values used to verify the significance of the regression model: the sums of squares (SS) and mean squares (MS) corresponding to the individual components of the model equation, the Fisher-Snedecor F statistic, the degrees of freedom (df), and the critical probability value. Analysis of variance makes it possible to determine which input variables have a dominant influence on the response and

which variables can be disregarded. However, the nature of the influence cannot be inferred from this analysis alone.

Table 1. Coefficient values of the model equation for PA6 with 25 % glass fiber content

The terms of the equation		Equation coefficients	Value	Error σ_i
		$\beta_0 \left[\text{MPa} \cdot \sqrt{\text{m}} \right]$	−3.535393	0.533901
Linear	T_w	$\beta_1 \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{^\circ\text{C}} \right]$	0.07893	0.004073
	T_f	$\beta_2 \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{^\circ\text{C}} \right]$	−0.01876	0.003590
	p_d	$\beta_3 \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{\text{MPa}} \right]$	0.02575	0.001660
	v_w	$\beta_4 \left[\frac{\text{MPa} \cdot \sqrt{\text{m}} \cdot \text{s}}{\text{mm}} \right]$	−0.02870	0.001350
Quadratic	T_w^2	$\beta_{11} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{(^\circ\text{C})^2} \right]$	−0.00015	0.000008
	T_f^2	$\beta_{22} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{(^\circ\text{C})^2} \right]$	0.00007	0.000012
	p_d^2	$\beta_{33} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{\text{MPa}^2} \right]$	−0.00019	0.000008
	v_w^2	$\beta_{44} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}} \cdot \text{s}^2}{\text{mm}^2} \right]$	0.00001	0.000002
Interfactor	$T_w T_f$	$\beta_{12} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{(^\circ\text{C})^2} \right]$	0.00011	0.000012
	$T_w p_d$	$\beta_{13} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{^\circ\text{C} \cdot \text{MPa}} \right]$	0.00001	0.000004
	$T_w v_w$	$\beta_{14} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}} \cdot \text{s}}{^\circ\text{C} \cdot \text{mm}} \right]$	0.00007	0.000005
	$T_f p_d$	$\beta_{23} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}}}{^\circ\text{C} \cdot \text{MPa}} \right]$	0.00002	0.000005
	$T_f v_w$	$\beta_{24} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}} \cdot \text{s}}{^\circ\text{C} \cdot \text{mm}} \right]$	0.00007	0.000006
	$p_d v_w$	$\beta_{34} \left[\frac{\text{MPa} \cdot \sqrt{\text{m}} \cdot \text{s}}{\text{mm} \cdot \text{MPa}} \right]$	−0.00001	0.000002

Based on the ANOVA results, it was also determined that all terms of the model equation (linear, quadratic, and interaction terms) are statistically significant at the significance level $\alpha = 0.05$. Three interaction terms lie at the threshold of significance: $T_f p_d$, $T_w p_d$, and $p_d v_w$.

The most illustrative way to present the impact of individual components of the model equation is by using a Pareto chart of standardized effects. The boundary between statistically significant and insignificant effects is indicated by a solid line corresponding to the 5 % significance level.

Table 2. Analysis of variance results of the toughness model for PA6 with 25 % glass fiber content

Components of the sum	SS	df	MS	F	p
T_w	1.35424	1	1.354240	4576.830	0.000000
T_w^2	0.11439	1	0.114385	386.579	0.000000
T_f	2.50000	1	2.500000	8449.074	0.000000
T_f^2	0.01046	1	0.010461	35.355	0.000000
p_d	0.06055	1	0.060552	204.643	0.000000
p_d^2	0.17336	1	0.173356	585.878	0.000000
v_w	0.39601	1	0.396010	1338.367	0.000000
v_w^2	0.01275	1	0.012755	43.107	0.000000
$T_w T_f$	0.02464	1	0.024642	83.281	0.000000
$T_w p_d$	0.00225	1	0.002250	7.604	0.007353
$T_w v_w$	0.06845	1	0.068450	231.336	0.000000
$T_f p_d$	0.00225	1	0.002250	7.604	0.007353
$T_f v_w$	0.03432	1	0.034322	115.996	0.000000
$p_d v_w$	0.00169	1	0.001690	5.712	0.019436
Fit	0.01616	2	0.008080	27.308	0.000000
Pure error	0.02160	73	0.000296		
Grand total SS	17.77623	89			

Figure 2 presents a Pareto analysis of standardized effects for PA6 with 25 % glass fiber content. The symbols “L” and “Q” denote the linear and quadratic components of the model equation, respectively. The notation “1L mod. 4L” refers to the interaction between components 1 and 4. The “+” or “−” sign next to the absolute value of the effect estimate indicates whether the analyzed variable increases or decreases as a result of the corresponding factor.

Based on the Pareto analysis, it can be concluded that the greatest influence on the fracture toughness of PA6 with 25 % glass fiber content is exerted by mold temperature, injection temperature, and injection speed. Increasing mold temperature

and injection temperature leads to an increase in the stress intensity factor. A different effect is observed for injection speed, where an increase in this processing parameter results in a decrease in fracture toughness.

Using the model equation, it is also possible to extrapolate the experimental results to generate three-dimensional surface plots illustrating changes in the critical stress intensity factor as a function of two selected variables, while keeping the remaining two constant. Typically, variables with the strongest influence on the response are selected for such visualizations. Based on the regression analysis, it was found that the fracture toughness of PA6 is primarily determined by injection conditions that most strongly affect matrix crystallization and, consequently, the resulting microstructure. These key parameters are mold temperature and injection temperature. A higher injection temperature reduces the viscosity of the polymer melt, improving macromolecular packing and replication of the mold cavity geometry. A higher mold temperature facilitates cavity filling, while a reduced temperature gradient between the melt and the mold wall helps minimize residual stresses and macromolecular orientation. These effects translate directly into improved mechanical properties of the molded parts.

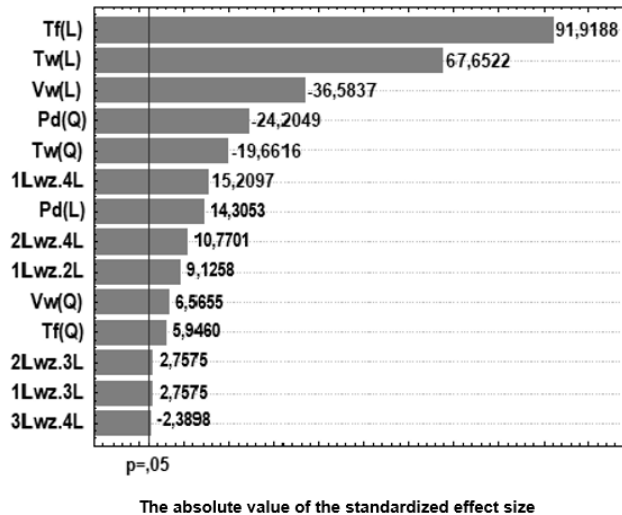


Fig. 2. Pareto effects analysis of the standardized fracture toughness model for PA6 with 25 % glass fiber content

The highest fracture toughness was achieved for SENB specimens made of PA6 composite with 25 % glass fiber content, injected into a mold heated to 90 °C and at an injection temperature of 280 °C (Fig. 3a). The maximum values of the critical stress intensity factor were obtained for samples molded at low injection speeds (Fig. 3b, 3c, and 3d). Higher injection speeds shorten the mold cavity filling time and reduce heat losses along the flow path; however, excessively high speeds may lead to the formation of air bubbles in the molded parts, which in turn compromises their mechanical properties.

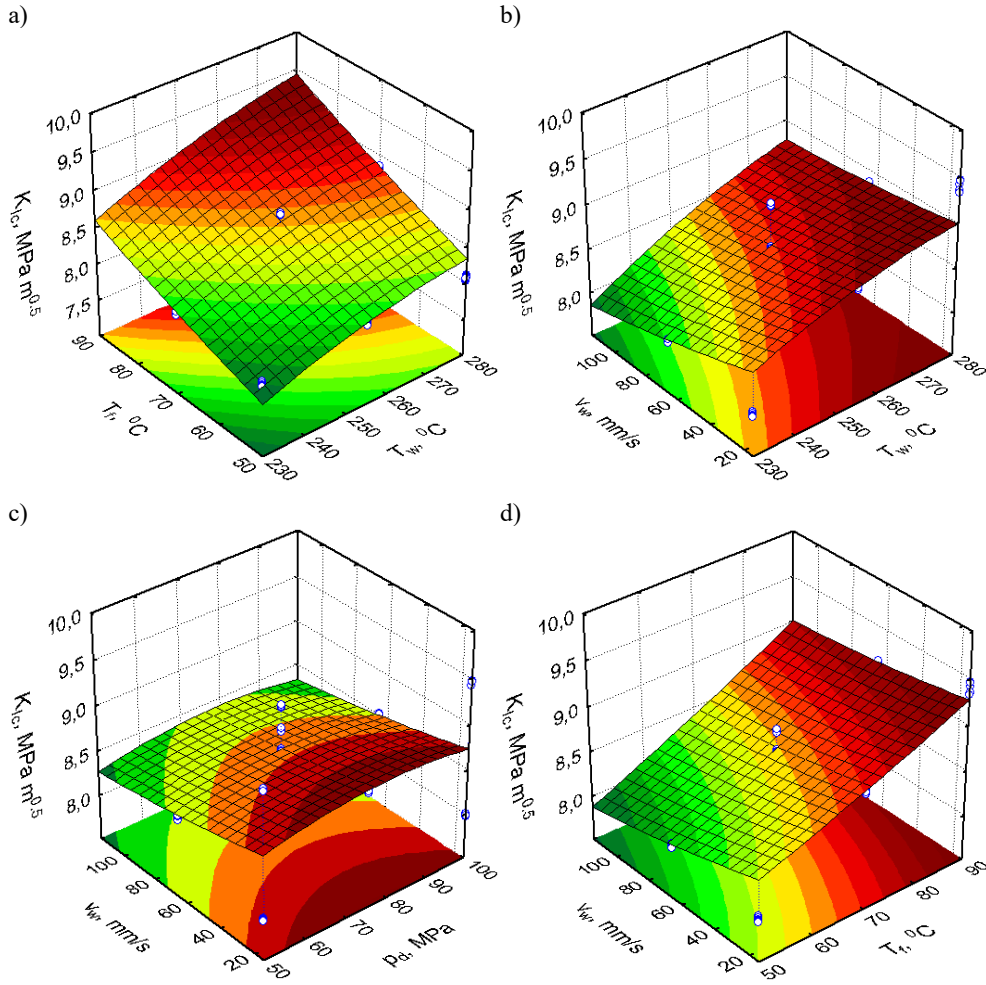


Fig. 3. Stress intensity factor value for PA6 with 25 % glass fiber content as a function of: a) mold temperature and injection temperature ($p_d = 75$ MPa, $v_w = 65$ mm/s), b) injection speed and injection temperature ($T_f = 70^\circ\text{C}$, $p_d = 75$ MPa), c) injection speed and packing pressure ($T_f = 70^\circ\text{C}$, $T_w = 255^\circ\text{C}$), d) injection speed and mold temperature ($T_w = 255^\circ\text{C}$, $p_d = 75$ MPa)

Figure 4 presents the optimization of the injection molding process aimed at increasing the fracture toughness of a PA6 composite with 25 % glass fiber content. The figure illustrates the dependence of the stress intensity factor K_{Ic} on four injection molding parameters: T_w , T_f , p_d , and v_w .

For example, the upper left panel of Figure 4 shows the dependence of the stress intensity factor K_{Ic} on injection temperature at optimal values of mold temperature, packing pressure, and injection speed, i.e.:

$$K_{Ic} = K_{Ic}(T_w) \big|_{T_f = T_{f\text{-optym}}, p_d = p_{d\text{-optym}}, v_w = v_{w\text{-optym}} \quad (6)$$

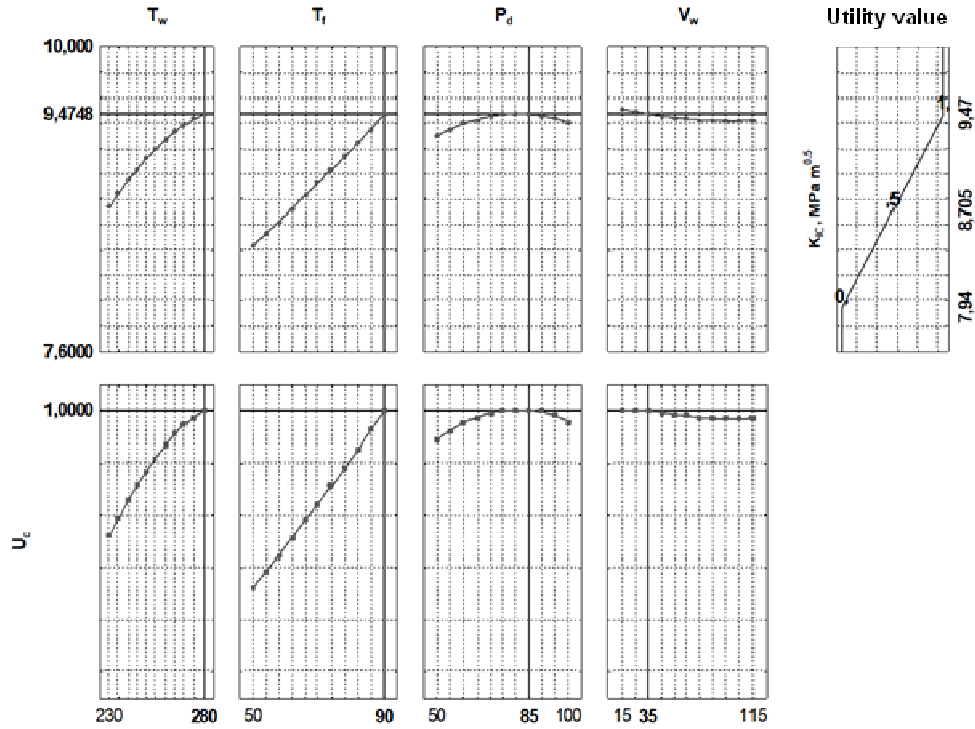


Fig. 4. Graphical interpretation of the injection process optimization for increasing the fracture resistance of a PA6 composite with 25 % glass fiber content

In the upper right corner of Figure 4, the output utility value U (corresponding to K_{Ic}) can be read for the optimal injection parameters ($T_{w-optym}$, $T_{f-optym}$, $p_{d-optym}$, $v_{w-optym}$). The lower part of the figure presents four dependencies of the total utility U_c on each of the injection molding parameters. For example, the graph in the lower right corner shows the dependence of the total utility U_c on injection speed v_w at optimal values of injection temperature, mold temperature, and packing pressure, i.e.:

$$U_c = U_c(v_w) | T_w = T_{w-optym}, T_f = T_{f-optym}, p_d = p_{d-optym} \quad (7)$$

Four input parameters (injection molding process parameters) were optimized with respect to the fracture toughness of the PA6 composite containing 25 % glass fiber. As a result of the optimization procedure, the following set of optimal input parameters was obtained: $T_w = 280$ °C, $T_f = 90$ °C, $p_d = 85$ MPa, $v_w = 35$ mm/s.

4. Conclusions

The highest fracture resistance was observed for SENB specimens made of the PA6 composite with 25 % glass fiber content, manufactured by injection molding

at an injection temperature of 280 °C, a mold temperature of 90 °C, a holding pressure of 85 MPa, and an injection speed of 35 mm/s. For this composite, higher injection speeds lead to a reduction in fracture resistance. Excessively high injection speeds may cause processing anomalies in the molded parts, such as the formation of air bubbles, which significantly degrade mechanical properties. At high injection speeds, delamination may also occur if shear stresses between the boundary layer and the core become sufficiently high to disrupt macromolecular chains.

Based on the regression analysis, it was found that the fracture resistance of the PA6-glass fiber composite is primarily determined by the injection conditions that exert the strongest influence on matrix crystallization and, consequently, on the resulting microstructure. These key parameters are mold temperature and injection temperature. The high values of the R² coefficients indicate that the model equations obtained through statistical analysis accurately reflect the experimental results. Mold temperature and injection temperature contribute most significantly to explaining the variability of the studied response, while the remaining input variables and their interactions also contribute, but to a lesser extent.

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