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Investigation of the pressure generated in the mould cavity during polyurethane integral skin foam moulding

P. Grőb[*], *J. Marosfalvi*

Department of Machine and Product Design, Budapest University of Technology and Economics, Műegyetem rkp. 3., H-1111 Budapest, Hungary

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Abstract. An industrial scale measuring system was set up to investigate the pressure arising in the mould cavity during polyurethane integral skin foaming. The system is able to measure the pressure arising in the mould cavity and the pressure distribution using a piezoresistive pressure sensor. The pressure distribution was measured at 18 points along the mould surface at constant production parameters. Then six production parameters, which affect the pressure, were investigated in detail with the Taguchi method of experimental design. The results of the design were processed by ANOVA (analysis of variance). Three major influencing parameters were estimated by regression analysis. Finally an equation was developed to give a good estimation to the pressure arising in the mould cavity.

Keywords: *processing technologies, industrial applications, design of experiments, pressure measurement, polyurethane*

1. Introduction

Polyurethane foaming as an empirical technology has predominantly been based on experience up to date. There is little information available about the real foaming process of products, the reaction pressure generated, and its distribution; therefore the design of foaming moulds and their optimization from various aspects – deformation, costs etc. – primarily rely on experience and estimates.

A few people dealt with the pressure generated at foaming previously. Campbell [1] described in detail the way how the pressure develops: in the beginning the blowing agent is in liquid form and dissolved in the mixture. After the chemical reaction starts the temperature increases and when the temperature reaches the boiling point of the blowing agent it starts to evaporate. Due to this the foam starts to expand, it fills the mould cavity. Having finished the mould filling the inner pressure in the foam increases.

Gupta and Khakhar [2] divided the generation of the pressure into three stages: in the first stage there is no foaming, the mixture flows into the mould, the pressure is equal to the atmospheric pressure; in the second stage the foam starts to expand, the density decreases, the pressure is still equal to the atmospheric pressure; the third phase starts when the expanding foam fills the mould cavity completely. In the last phase the density becomes constant and the pressure increases. After the foam reaches the gel-point (the gel-point is the point at which an infinite polymer network first appears), there is no more change in the density. He tried to describe the changes of the pressure in time with the changes of the amount of the blowing agent and the density. Similarly, as Campbell demonstrated, the pressure arises when the foam completely fills the cavity and which coincides with the changes of the density and the amount of the blowing agent.

[*]Corresponding author, e-mail: grob.peter@gt3.bme.hu

Beruto *et al.* [3] studied the connection between the blowing agent and the pressure arising in the bubbles of the foam. He calculated a so-called 'foaming power', which was a mechanical work what the foaming system was doing against the enviroment. The foaming power can be calculated with Equation (1):

$$W_e = \Omega \int P_e(t) \mathrm{d}h(t) \tag{1}$$

where W_e is the foaming power [W]; Ω is the volume of the foam [dm^3], $P_e(t)$ is the pressure [N/m^2], $h(t)$ the displacement of the foaming system [m]. He found that, if the amount of the blowing agent decreases, the foaming power will decrease too. If the total amount evaporates, the power becomes zero.

The literature discusses measurements of the pressure generated during foaming at several instances, but none of them have been measured the pressure in the mould cavity directly. Clarke [4] made attempts to determine cycle time from pressure data. He assumed that the changes in the closing pressure of the hydraulic cylinder correspond to the pressure generated in the mould. Vespoli *et al.* [5] built a Kistler pressure transmitter into the mould; however, it was not placed into the mould cavity but at the beginning of the feed bush. He intended to determine the viscosity changes from the pressure changes. He used the value of pressure rise to validate his viscosity function estimate.

Ryan *et al.* [6] built an in-line rheometer with two pressure transducers. The rheometer was placed between the mixing head and the mould to investigate whether the behaviour of the mixture behind the mixing head is Newtonian or not. He found out that it is a good approximation to consider the mixture as a Newtonian fluid and from the pressure-difference the apparent viscosity can be calculated. Kim *et al.* [7] also built a special rheometer to measure the pressure-growth to assess the viscosity. He set up the pressure transducer at the inlet point of the mould. Likewise Vespoli, he used the value of pressure rise to validate his viscosity function estimate. The viscosity calculations are important, because when the mould filling time is longer than the gel-time, a pre-mature gelation occurs, which leads to defective products. From the changes of viscosity the gel-time can be calculated.

Yokono *et al.* [8] used his pressure measurement data for validate his simulation of the arising pressure. The simulation is based on the principle of adiabatic compression. Kodama *et al.* [9] built his pressure transmitter into the lateral wall of a large-size mould. He attempted to make inferences from the pressure figure on the expansion of the foam after removal from of the mould.

From the pressure measurements, only Kodama's measurement [9] was performed directly in the mould cavity, but he also performed measurements only at one location. It is important to mention that the primary aim of these works was else than to determine the value and distribution of pressure.

There are only a few publications which contain useful information related to the pressure arising in the mould cavity during polyurethane foaming. The main reason for this can be that the foaming technology is still based on some empirical experience, and the companies do not publish their information. However, in the absence of this experience and information the proper design and the optimizing of the foaming moulds can not be made in advance.

The aim of our work was to set up a measuring system of industrial scale to gain real *in situ* information on the foaming process. The measuring system was made suitable for measuring the reaction pressure and its distribution. We tried to obtain more accurate information on the foaming process.

2. The applied mathematical methods

2.1. The Taguchi method

The Taguchi method, developed by Genichi Taguchi [10], is one of the experimental design methods, based on a fractional factorial design. He simplified and standardized the fractional factorial design method and made it easy to use for everyone. It is intended to select the appropriate, previously specified orthogonal array matrix and then to assign the factors to the appropriate columns according to the specified rules.

In addition to its easy application the greatest advantage of the method is that the results are displayed not only numerically but also illustrated in graphs. In case of examining each factor, e.g. the steeper a curve is, the more significant its impact on the target value. In the same way, interactions can also be examined graphically: by depicting the

impact of the two factors in the same graph. It can be observed that there is interaction between them if the curves intersect each other; and if they do not intersect each other, there is no correlation between them in the given range.

2.2. Analysis of variance (ANOVA)

Variance analysis is a statistical method suitable for comparing the expected values of groups with identical standard deviation and Gaussian distribution, also known as ANOVA – generated from the initial letters of its English name: ANalysis Of Variance. So the ANOVA is an extended two sample t-test.
For variance analysis, the H_0 null hypothesis is that the factor does not affect the process. So it can be demonstrated not only what degree of impact the factor has, but also which of the factors examined affect the target function examined and which of them not – as regards the reliability level concerned.
First the sum of squares (S) have to be calculated, then the mean square (or variance) can be considered. Next step is to obtain the variance ratio (F). This F value is compared to the value of the F-test table at the desired confidence level. If the $F<F_{table}$ than the null hypothesis is accepted, so that factor does not affect the process. Finally the percentage contribution (P) is calculated, which is shown the percentage contribution of each factor to the process.

3. Experimental

3.1. The technology of polyurethane foaming

The foaming technology of polyurethanes differs from traditional injection moulding. In general, it is termed RIM according to the English abbreviation (Reaction Injection Moulding). By RIM there are two liquid reactive components, stored separately, which are mixed with high pressure in the mixing head, and then the mixture is poured immediately into the mould. In the mould a chemical reaction starts, the liquid mixture becomes solid foam, simultaneously the foam expands, and then the curing begins in the course of which the product solidifies and takes the shape required [11, 12].

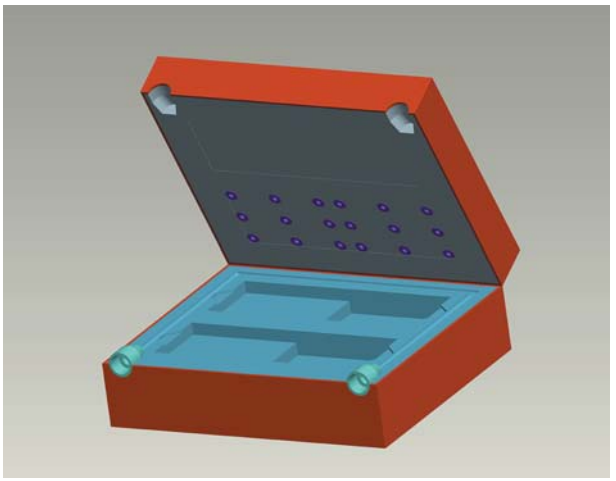

Figure 1. The 3D model of the test mould with staggered mould cavity and the measurement points (purple colour)

3.2. The test-mould

A test-mould for pressure measurements was designed and manufactured in cooperation with Ratipur Ltd., Komló, Hungary. The following requirements were specified for the test mould: the mould should be manufactured according to the industrial technology; the mould should be suitable for testing both flexible, rigid and integral skin foam systems; the specimen should be of 'industrial' size; the mould should be suitable for fixing both thermal and pressure sensors; the mould cavity should be suitable to admit inserts to produce different specimens; the mould should be possible to inject from several places and directions; each mould cavity should be sealed separately.
The 3D model of the test mould is shown in Figure 1.

3.3. Pressure measurement

There are 18 measurement points set up uniformly in the mould to measure pressure. These points cover the surface of the entire mould cavity. They were placed with particular care ensuring that measurement points that critical points, e.g. at the step (sudden change in cross-section), or at the edge of the product, should be detected as well.
Measurement points were set up only in the upper part of the mould and only above one of the mould cavities. Figure 2 shows the mould arrangement with the measurement spots.

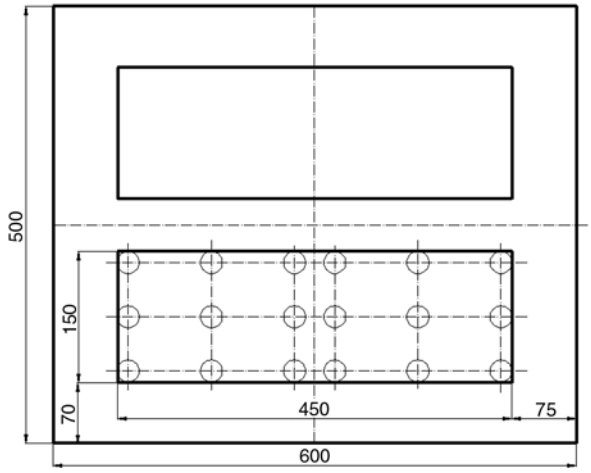

Figure 2. Outline of the arrangement of the test mould and the 18 measurement spots

An insert for sensor was installed to each measurement point. The sensor – type number 4079A by the Kistler Company, it is a piezoresistive type combined heat and pressure sensor developed for the RIM technology – can be placed into this insert. The detected pressure data are transmitted through the cable to the amplifier. The signal boosted by the amplifier gets into an A/D converter; this is required for computerized processing. Finally, the received signal was recorded by the Windaq software of DataQ Instruments Inc.

3.4. The examined material

The examined material is the foam nominated by the code ELASTOFOAM I4703/100/schw, produced by Elastogran Kemipur Ltd. (Hungary). This foam system is suitable for producing flexible integral skin foam products of 400–800 g/dm^3 density and 50–80 Shore A rigidity. The system contains two components: component 'A' is a mixture of polyols, catalyst and other additives, component 'B' is the izocyanate, in this case it is methylene diphenyl diisocyanate (MDI). The cream time, this is the time, when the material volume begins to increase, for this system is 30 sec, the rise time, it is the end of foaming, is 90 sec, the tack-free time, when the foam surface becomes tack-free, equal to the rise time. The density of the freely rising foam is 130 g/dm^3. The blowing agent is n-pentane. The formulation for this foam system is the following: 100 wt% polyol, 53.5 wt% izocyanate, 5.0 wt% n-pentane.

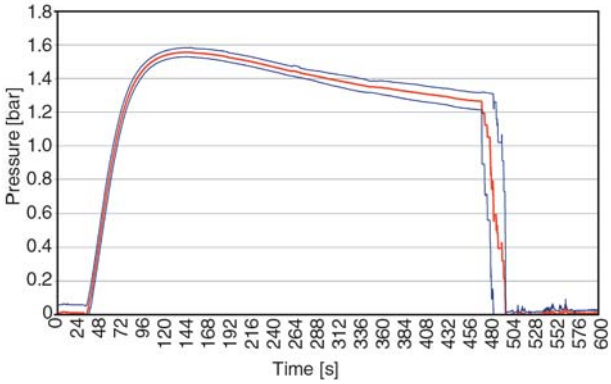

Figure 3. The time vs. pressure curve investigating the pressure distribution

4. Results and discussion

4.1. Pressure distribution

Taking into account that no information was available on the pressure distribution in the mould cavity, first it was examined whether there is a significant difference in the pressure data measured at the 18 measurement points at constant production parameters. The time vs. pressure graph is presented in Figure 3.

The red curve represents the average of 18 measurements; the blue curves represent the standard deviation of the measured data. As it can be seen in the diagram the standard deviation of the data is quite small, it can be concluded that there is no significant difference among the 18 measurement points in terms of the maximum and the runoff of the pressure generated. This also means that pressure distribution is uniform along the surface of the product; it does not depend on geometry of the cavity and the location.

4.2. Estimation of pressure based on empirical data

In the polyurethane foam industry the pressure and the average density are important parameters, so the connection between them is not negligible. The pressure is essential for the mould design; the density is a defined requirement from the customer. The connection between density and pressure is found in the industrial practice as an empirical estimate. An empirical formula for the scale of the pressure generated was recommended by the manufacturer of the foam system. According them the

estimated pressure can be calculated with Equation (2):

$$\text{estimated pressure} = \frac{\text{density of the product}}{\text{density of the freely rising foam}} \text{ [bar]} \quad (2)$$

The density of the product [g/dm^3] is the ratio of the quantity of material injected and the volume of the mould; and the density of the freely rising foam [g/dm^3] is a technological parameter. The ratio of these two data will yield the estimate of the inner pressure value to affect the mould.

The pressure data were measured during the manufacturing of foam products with different average densities. This information was compared with the estimated ones, this contrast is shown in Figure 4. The red line represent the estimated ones, the black line represent the measured data.

It can be observed in Figure 4 that the lines of estimated and measured pressure data are diverging. The pressure, which is calculated with the empirical formula, is higher than the real pressure value. This means that the moulds designed with this method are overestimated.

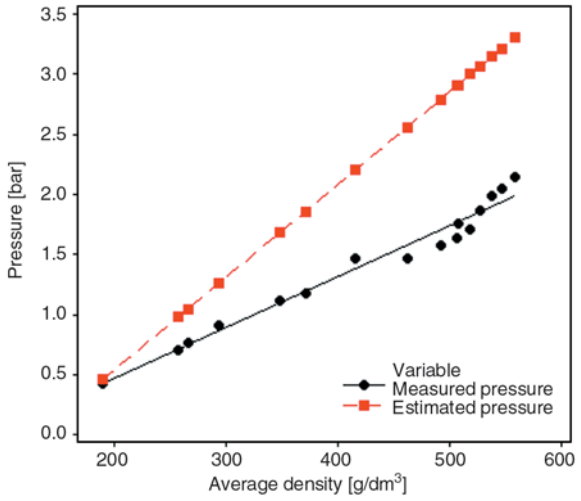

Figure 4. Measured and estimated pressure vs. average density

4.3. Selecting substantial factors

Six technological factors affecting the pressure were investigated. These factors were as follows: mould temperature (MT), temperature of the components (CT), injected mass flow rate (MF), injection time (IT), volume (VO), and the mixing ratio by weight of the polyol and isocyanate (MR). These are the most important factors in the industrial practice.

Our aim is to select the significant factors. These factors were investigated at two levels. Table 1 shows the set of the levels. The engineers of the company provided us with assistance in selecting levels. The design was a 2^{6-1} design with 6 factors and 32 runs.

Figure 5 shows a graphical representation of the results. The steeper the line; the larger the impact of the given factor on the target function – on the maximum value of pressure in the present case. Table 2 contains a numerical presentation of results and the order of the factors.

As it can be seen from Figure 5 and Table 2, the injected mass flow rate (MF) has the greatest impact on the generated pressure; it is followed by the volume of the mould cavity (VO) and the injection time (IT). It can be clearly observed that the impact of these three factors on the target function is much stronger than that of the others. As expected, by increasing the injection time, the injected mass will be increased, and consequently the pressure will also be increased. The same can be observed in case of changing the geometry: higher pressure will be result at lower volume with

Table 1. Levels of the factors to the examination for selecting the substantial factors

	Level 1	Level 2
Mould temperature (MT)	35°C	45°C
Temperature of the components (CT)	23°C	29°C
Injection time (IT)	5 s	7 s
Injected mass flow rate (MF)	150 g/s	200 g/s
Volume (VO)	2.53 dm^3	1.69 dm^3
Mixing ratio (MR)	100:51	100:54

Table 2. Numerical presentation of of the six factor's impact on the pressure

	Mould temperature (MT)	Temperature of the components (CT)	Injection time (IT)	Injected mass flow rate (MF)	Volume (VO)	Mixing ratio (MR)
Level 1	1.92	1.95	1.67	1.63	1.63	1.99
Level 2	2.09	2.06	2.34	2.38	2.38	2.01
Difference	0.17	0.12	0.67	0.76	0.75	0.02
Order	4	5	3	1	2	6

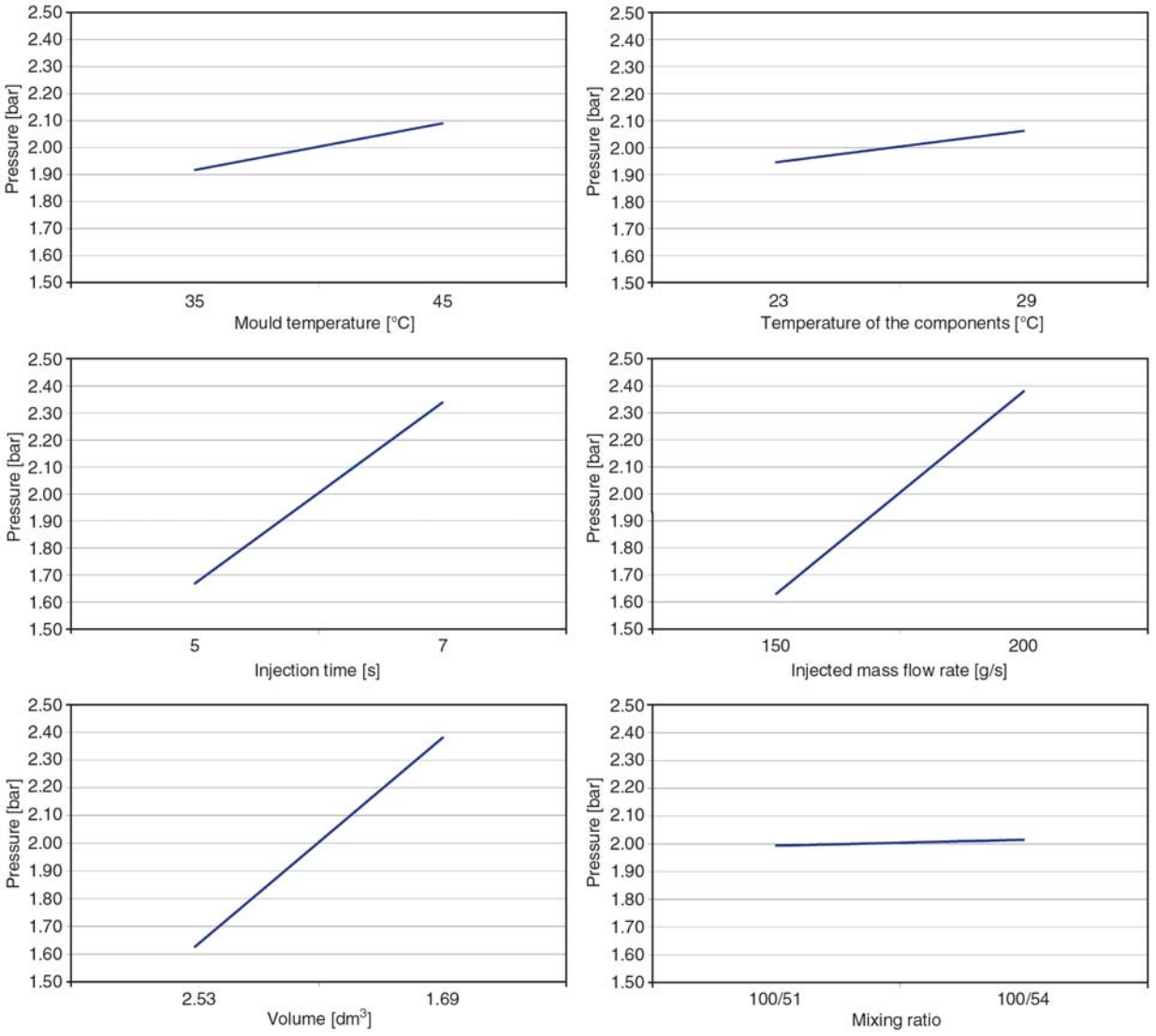

Figure 5. Graphic presentation of the six factor's impact on the pressure

the same mould charge. At the same time, it was found that the pressure decreases by reducing the injected mass flow.

Processing results by variance analysis (ANOVA)

Table 3 contains the ANOVA evaluation of results. Degrees of freedom (f) are a measure of the amount of information that can be uniquely determined from a given set of data. It equals one less that the number of levels. Here the number of levels was two. The total degrees of freedom are equal to the total trial numbers minus one. The number of trials was 32. Sum of squares (S) is a measure of the

Table 3. The ANOVA data table for pressure

	Degree of freedom (f)	Sum of squares (S)	Variance (V)	Variance ratio (F)	Percentage contribution (P)
MT	1	0.24	0.24	1.53	1.41
CT	1	0.11	0.11	0.69	0.64
IT	1	3.59	3.59	23.08	21.28
MF	1	4.58	4.58	29.41	27.11
VO	1	4.55	4.55	29.21	26.93
MR	1	0.0036	0.0036	0.023	0.02
Error	25	3.27	0.16	1	22.605
Total	31	16.88	–	–	100

deviation of the experimental data from the mean value of the data. Variance (V) is a quotient of the square sum and the degrees of freedom. Variance measures the distribution of the data about the mean of the data. Variance ratio (F) is used to measure the significance of the factors. It is equal to the quotient of variance of each factor and the variance of the error.

The last column in Table 3 indicates the percentage contribution (P), which was obtained by dividing the sum of squares by the total sum of squares and multiplying the result by 100, of each factor in terms of the entire process. The three major factors (injection time (IT) 21.28%; injected mass flow rate (MF) 27.11%; volume (VO) 26.93%) have totally a 75% impact on pressure. Let us mention that the impact of factors are not taken into consideration is nearly 22%. This means that there are other factors, besides the factors involved in the investigation, which affect the pressure. Thus, it may be considered that further factors should be involved in the investigation to get a more accurate approximation.

By comparing the variance ratios (F) with the value pertaining to 95% reliability ($F_{95}(1.25) = 4.2417$), it can be concluded that only the injection time (IT), injected mass flow rate (MF) and the volume (VO) affect the process.

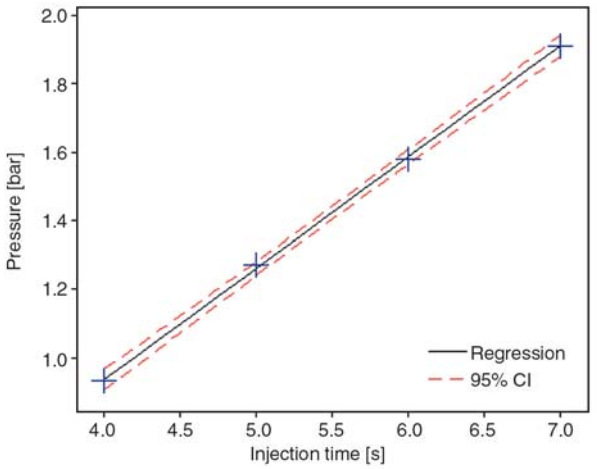

Figure 6. The regression line and the confidence interval for injection time

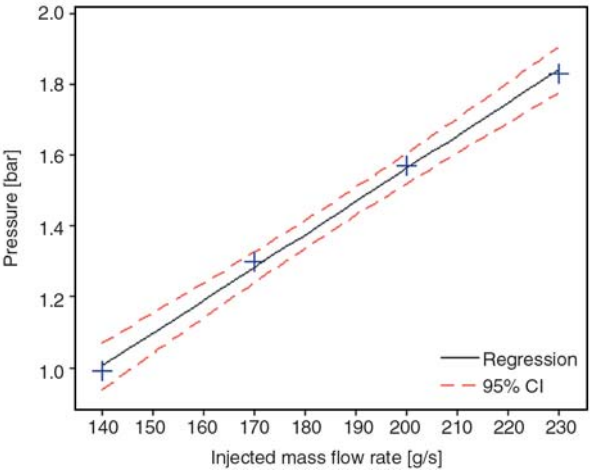

Figure 7. The regression line and the confidence interval for injected mass flow rate

4.4. Estimation of the pressure based on the three main factors

The three main factors (injection time (IT), injected mass flow rate (MF), volume (VO)) were further investigated. Second time a four-level measurement was designed. Table 4 shows the set of the levels.

To the measured points, supposing a linear relation, a regression line was fitted, using the least square method. The regression lines are shown in Figures 6–8. The blue points represent the measured points, the black continuous line is the regression line, and the red dashed line is the line of the confidence interval for 95%.

Table 5 shows the equations of the regression lines and the coefficient of determination. The coeffi-

Table 4. Levels of the factors to the examination for estimation of the pressure

	Level 1	Level 2	Level 3	Level 4
Injection time (IT)	4 s	5 s	6 s	7 s
Injected mass flow rate (MF)	140 g/s	170 g/s	200 g/s	230 g/s
Volume (VO)	2.53 dm^3	2.24 dm^3	1.96 dm^3	1.69 dm^3

Table 5. The equations of the regression lines and the coefficient of determinations

	Equation of the regression line	Coefficient of determination (R^2)
Injection time (IT)	$p = 0.325(IT) - 0.3650$	0.999
Injected mass flow rate (MF)	$p = 0.0093(MF) - 0.298$	0.998
Volume (VO)	$p = 0.4216(MF) - 0.298$	0.997

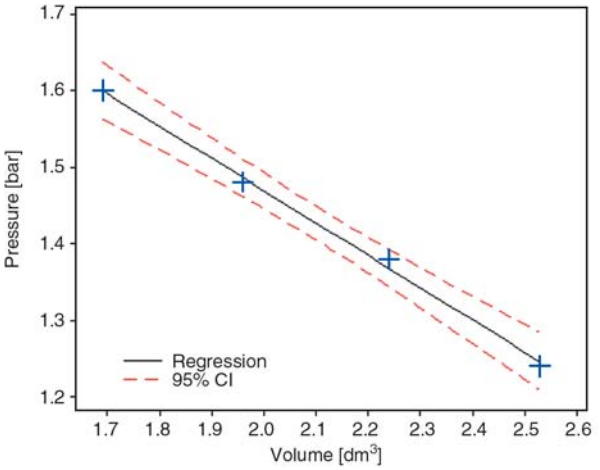

Figure 8. The regression line and the confidence interval for volume

cient of determination indicates the strength of a linear relationship between the line and the points. If the coefficient is close to 1, that means excellent regression estimation.

The value of the pressure (p) is obtained in [bar], if the injection time (IT) is in [s], the injected mass flow rate (MF) is in [g/dm^3] and the volume is in [dm^3]. The equations shown in Table 5. are good approximations of the measured data, because of the values of coefficient of determination are near 1.

The equations in Table 5 are adequate if the values of the other factors are constant. E.g. the pressure can be predicted from the injection time, when the injected mass flow and the volume is invariable. It is hard to achieve in the industry, so a multiple linear regression test was made. This test took into consideration the effects of all factors. Equation (3) shows the result of the multiple linear regression:

$$p = -1.21 + 0.324 \cdot (\mathrm{IT}) + 0.00938 \cdot (\mathrm{MF}) - 0.422 \cdot (\mathrm{VO}) \quad (3)$$

Coefficient of determination: $R^2 = 0{,}987$.

If the injection time, the injected mass flow rate and the volume of the mould are known, a good estimation can be given with this equation.

5. Conclusions

The pressure generated in the mould cavity during polyurethane integral skin foam moulding was investigated. A test mould was designed and built to measure the maximal pressure arising in the mould cavity and the distribution over the mould surface. It was found that the value of the pressure is the same along the surface of the product; it does not depend on the geometry of the mould cavity. The measurements proved that the empirical correlation used in the polyurethane foam industry for mould design considerably overestimates the moulds in ranges of higher average density. It was established that the three major manufacturing parameters, which have effects on the value of the pressure, are the injected mass flow rate, the injection time and the geometry of the mould cavity. These three production parameters impact on pressure are nearly 75%, the rest 25% go to the other, approximately 15, production parameters. Finally a multiple regression analysis was made, which included the three major factors, to give a good estimation to the pressure arising in the mould. This equation can be used in the mould design instead the empirical correlation, which was demonstrated in our work overestimates the moulds in ranges of higher average density, leads to a better designed mould.

Acknowledgements

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