Applied Mechanics and Materials
ISSN: 1662-7482, Vol. 808, pp 125-130
doi:10.4028/www.scientific.net/AMM.808.125
© 2015 Trans Tech Publications, Switzerland

Submitted: 2015-06-12
Revised: 2015-07-27
Accepted: 2015-07-27
Online: 2015-11-23

Experimental and Theoretic Research Regarding Extrusion Optimization for Reinforced Polyamide (PA 6.6 – 10 % GF)

POPESCU Adrian[1, a *], HANCU Liana[2,b], BERE Paul[3,c], and MIRON BORZAN Cristina[4,d]

[1,2,3,4]Technical University of Cluj-Napoca, Faculty of Machine Building B-dul Muncii 103-105, Cluj-Napoca, Romania

[a]Adrian.POPESCU@tcm.utcluj.ro, [b]Liana.HANCU@tcm.utcluj.ro, [c]Bere.PAUL@tcm.utcluj.ro, [d]borzan_cristina@ymail.com

Keywords: reinforced polymers, extrusion, polyamide, glass fiber.

Abstract: This paper presents the possibility of extrusion optimization using Design Expert software for statistical analyze and also for the recommendation of some manufacturing data. Mechanical characteristics are determined on samples from extruded tubes. On these samples, ANOVA (Analysis of variance) software is used, in order to determine the optimal values of the temperatures in different zones. The material is polyamide 6.6, reinforced with 10 % glass fiber (PA 6.6 – 10 % GF). The authors want to present the influence of the reinforcement degree upon some mechanical characteristics of the products made by extrusion. Considering the homogeneity of the polymeric melt the product's quality will be analyzed. During the extrusion process a series of parameters (extrusion speed, the pellets feed, processing temperature etc.) are involved and they must be taken into account to achieve calibrated and qualitative products.

Introduction

The materials analyzed in the paper are from the reinforced polymers class, namely polyamide. It was choose this type of polymer due the wide range of currently available on the world marked and for the reason of the necessity of increasing the mechanical characteristics of the industrial products. To choose a properly equipment in order to process a peculiar product, the followings should be considered: the material, the geometry and the dimensions as well as the number of parts. In this paper the main task is to improve the properties and the mechanical characteristics for extruded products made of polyamide 6.6 reinforced with 10% glass fiber (*PA 6.6 - 10 % GF*). One of the improving solutions is temperature's optimization for a typical extrusion process. It is known that the processing temperature is a parameter of major importance for the extrusion process.

Many applications from current researches are based on the optimization of manufacturing parameters. Right choice of input parameters is very important and with influence on the mechanical characteristics and the final product [1,2,3,4,5,8]. For example Butora P., et al concluded it is necessary to choose optimal parameters of the polymer filler and the size of the particles, their dispersion and density [6].

Considering the manufacturer's indications for *PA 6.6 – 10 % GF*, a temperature's interval is recommended and not a certain value. For the metering zone this interval is between *270 ÷ 290°C*, and for the forming zone *260 – 280 °C*. It is important to establish the temperatures for the metering and for the forming zone because the quality of the product is highly achieved in these areas.

With the help of the *ANOVA* software (*Analysis of variance*) it will be possible to determine optimal temperatures both for the metering and the forming zone. They will be established considering the maximum values for the tensile strength of the material. ANOVA method of analyze is a part of mathematical and statistical techniques, used for improving technological processes, for optimization and technology development. This method belongs to *Response Surface Methodology - RSM* and has the ability to verify if a certain value belongs or not, to the model [7].

Most *RSM* applications are particular situations in which is admitted that a lot of input data can influence the performances or the characteristics of the process. Some of them are: response or dependent variables. Input variables are sometimes independent variable.

Experimental methodology

In this study, a software application Design Expert was used. In this research, tensile strength of extruded products will be called a dependent variable and the two process parameters: metering zone temperature and die zone temperature will be called independent variables.

By using the response surface method, the first phase of establishing the conditions for optimization occurs, and then the response surface is created between process variables and objective function.

Since it is used a simple function, called the response surface, the procedure is very fast.

The accuracy of the optimal solution depends on the approximation accuracy of response surface.

In the response surface method, the selection of parameters is made, generally, by designing experiments and the numerical analyze is repeated so many times how more experiments are provided in design.

Then, based on these results, response surface is constructed. Experiment's planning purpose is to establish values of independent variables so their variation in space can be exploiting.

To obtain experimental data representative for the extrusion process, optimizing PA 6.6 - 10% GF material, a central planning-type second-order compositional 2^k *where k = 2 (k-number of independent variables*) was adopted.

After inserting the minimum and the maximum values of independent variables, a number of 13 experiments (virtual experiments, numerical simulation) were obtained. Performing the numerical analysis, the values of the dependent variable (tensile strength) are obtained.

For each experiment we need to carry out a minimum of 5 tests (according to SR EN ISO 527 - 2000).The samples will be tested at tensile strength. The data for the response variable were manually introduced in table 1.

Table 1. Experiments planning strategy (CCD)

No.	Code for the variables		Real values for independent variable		Response variable
					Tensile strength R_m [MPa]
	A	B	T_3 [° C]	T_4 [° C]	PA 6.6 – 10 % GF
1	-1	+1	**270**	**280**	**55.3**
2	0	0	**280**	**270**	**62.8**
3	+1	-1	**290**	**260**	**50.2**
4	-1	-1	**270**	**260**	**62.5**
5	+α	0	**294**	**270**	**53.4**
6	0	-α	**280**	**256**	**55.6**
7	-α	0	**266**	**270**	**57.4**
8	0	0	**280**	**270**	**64.8**
9	0	0	**280**	**270**	**63.3**
10	0	0	**280**	**270**	**64.1**
11	0	0	**280**	**270**	**63.5**
12	+1	+1	**290**	**280**	**58.7**
13	0	+α	**280**	**284**	**61.3**

Obtained input data for *"Design Expert"* software

For the experiments was used an extruder *Cincinnati Monos +45 mode type* (figure 1). This equipment has a single screw with a diameter of 45 mm. The geometry of the extruder is a special one, from the last generation, with a bimetallic cylinder realized from special alloys, resistant at corrosion and abrasion.

This type of equipment provides a very good homogeneity of the melted material because of the complexity of the worm gear's geometry and the very precise control of the temperatures for all four specific areas of the extrusion process (feeding area, compressing area, metering zone and shaping area).

With this extruder, tubes with diameters between 20 – 2000 mm and with different wall thickness can be obtained. For this research a tube with exterior diameter of 30 mm and a wall thickness of 4 mm was realized. The processing temperature of the extruder can be set up until reach out the maximum value of 400°C for each area separately.

The samples for the tensile test were cut, with a special mill, from each tube corresponding to each type of material.

Processing temperatures were set according to experimental plan established with ANOVA test analysis.

Values of independent variables can be set using thermocouples $\boldsymbol{T_3}$, $\boldsymbol{T_4}$ respectively.

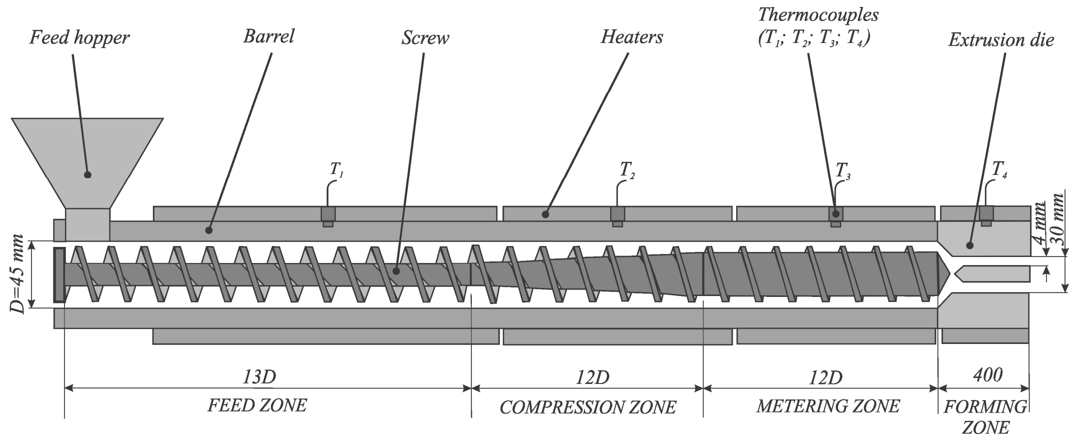

Fig. 1, Schematic Cincinnati Monos + 45 type single screw extruder

The samples were cut, according to *SR EN ISO 527 – 2000* from the tubes and the dimensions are: L_3= 150 mm, L_1= 60 mm, R= 60 mm, b_1= 10 mm, b_2= 20 mm, h= 4 mm, L_0=50 mm, L= 115mm (figure 2). The samples were cut using a specially designed dies, mounted on a hydraulic press. Tensile tests of samples were carried out on tensile test machine *Instron type 3366* shown in figure 3.

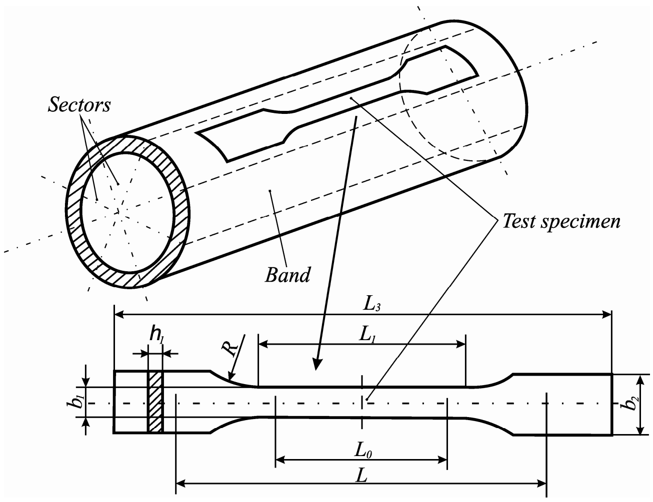

Fig. 2, Tensile test specimens according to SR EN ISO 527-2000

Fig. 3, Instron type 3366 tensile testing machine

After determining the arithmetic average values of the tensile stress for each experiment, they were introduced in the Design Expert program, in order to determine the mathematical model of the tensile strength $\boldsymbol{R_m}$ according to the temperatures $\boldsymbol{T_3}$ and $\boldsymbol{T_4}$ for the chosen material.

Numerical Results

To determine which model best describes the mathematical relationship between dependent variable and independent variables, the following were used: Fischer statistical method, determination coefficient R^2, and adjusted factor of determination R^2_{adj}.

ANOVA test is presented in table 1, in which mathematical models are compared for PA 6.6 - 10% GF.

For each source it was examined the probability ("Prob> F") in order to see if it is below 0.05, a value that represents maximum permissible level of statistical significance.

It can be seen from table 2 that besides Quadratic model vs. 2FI, all other models are above 0.05 which is the significant statistical level. This means that only Quadratic vs. 2FI model is significant, the others models are excluded.

Table 2. ANOVA results

Mathematical model	*Sum of Squares*	*df*	*Mean Square*	*F*	*p-value Prob > F*
Mean vs Total	45951.88	1	45951.88		
Linear vs Mean	37.42	2	18.71	0.82	0.4662
2FI vs Linear	61.62	1	61.62	3.35	0.1003
Quadratic vs 2FI	**155.90**	**2**	**77.95**	**57.71**	**< 0.0001**
Cubic vs Quadratic	7.05	2	3.52	7.33	0.0326
Residual	2.40	5	0.48		
Total	46216.27	13	3555.10		

To ensure the effectiveness of selected model we will consider coefficient of determination R^2 and predicted residual error sum of squares (PRESS - Predicted Residual Error Sum of Squares) (see table 3).

PRESS is a measure for the ability of the model to approximate each point in the space of independent variables.

R^2 is a measure of dispersion from the average value. As "*R Squared*" value is closer to the unit value, the better model approximates the response function. On the basis, the Quadratic model was selected for *PA 6.6 – 10% GF*.

After establishing the type of the mathematical model for approximating the dependent variables, it has to be checked whether a term should or should not be part in the model.

The most common criteria in order to add or remove a variable is based on partial *F test*.

Table 4 presents the ANOVA panel for Quadratic model of *PA 6.6 - 10% GF* material.

ANOVA test method confirms adequacy quadratic model, the term "sample F" is smaller than 0.0001 (see table 4).

Table 3 The determination coefficient $\boldsymbol{R^2}$

Mathematical model	***Standard Deviation***	R^2	R^2_{adj}	R^2- calc.	***PRESS***
Linear	4.76	0.1415	-0.0302	-0.5368	406.32
2FI	4.29	0.3746	0.1661	-0.4644	387.19
Quadratic	**1.16**	**0.9642**	**0.9387**	**0.7956**	**54.04**
Cubic	0.69	0.9909	0.9782	0.9800	5.28

Fischer *37.75* Value of Statistics shows that the model is significant. There is only a 0.01% probability that average model lies outside the confidence interval.

The value of "*Prob> F*" is less than 0.05 and indicates that model terms are significant.

In our case all the terms *A, B, AB,* A^2 *and* B^2 are significant terms of the model."*Prob> F*" values are greater than 0.1000 and indicates that insignificant terms can be excluded in the regression equation (see table 4).

Table 4 ANOVA test table for response surface

Source	***Sum of Squares***	***df***	***Mean Square***	***F***	***p-value Prob > F***
Model	**254.94**	**5**	**50.99**	**37.75**	**< 0.0001**
A-T3	26.55	1	26.55	19.66	0.0030
B-T4	10.87	1	10.87	8.05	0.0251
AB	61.62	1	61.62	45.63	0.0003
A^2	122.87	1	122.87	90.97	< 0.0001
B^2	49.55	1	49.55	36.68	0.0005

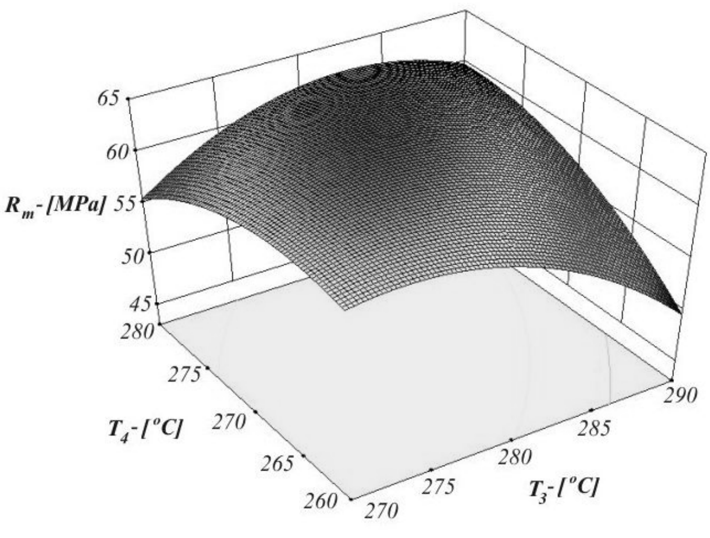

Fig. 4, Variation of tensile strength according to temperature

According to the above data, the regression equation for the tensile strength mathematical model of *PA 6.6-10% GF* is obtained:

$$\boldsymbol{R_m(T_3, T_4)} = -2265.85152 + 13.09582\, T_3 + 3.74778\, T_4 + 0.039250\, T_3 T_4 - 0.042636\, T_3^2 - 0.027075\, T_4^2. \quad (1)$$

where:

R_m – tensile stress [MPa];
T_3 – temperature in the metering zone [°C];
T_4 – temperature in the forming zone [°C].

In figure 4, a response surface is graphically presented and corresponds to regression equation for tensile stress mathematical model (1).

Conclusions

In order to achieve *PA 6.6 – 10 % GF* optimum melt homogeneity and maximum tensile resistance, process temperature must be properly set.

We may see that the tensile strength minimal value is only obtained when the difference between the temperature from the metering and the forming areas is maximum (like: $T_3 = 290$ °C şi $T_4 = 260$ °C). Those results from forming zone are bigger than metering's zone extrusion installation danger can appear and extruded profile calibration becomes hard to handle. Maximum tensile strength value is obtained when metering's zone temperature is 280 °C and forming's zone temperature is 270 °C. In this case, besides the very good mixing of the material due to the low temperature, in the profile zone is formed an optimal extrusion pressure and also a good extruded profile calibration. In conclusion, it can be said that the metering's zone temperature must be 280 °C and forming's zone temperature must be 270 °C. In order to obtain a higher tensile strength for a reinforced fiber glass extruded polymer (PA 6.6 – 10 % GF), the forming's zone temperature must be lower than the metering's zone temperature. This difference should not be of a great value because there is a danger of damaging of the extrusion equipment and also the technological process will be compromised. A very important factor in manufacturing products from reinforced polyamide is the temperature, so knowing the optimum values of it makes possible to manufacture products with a high quality. Researches can be extended for other degrees of reinforcement for PA 6.6 polymers.

References

[1] V. A. Ceclan, P. Bere, M. Borzan, S. Grozav, C. Borzan, Development of Environmental Technology for Carbon Fibre Reinforced Materials Recycling - Revista Materiale Plastice Bucuresti, (2013), pp. 79-83.

[2] A. Crai, H. Iancau, S. Grozav, C. Popescu, G. Onetiu, The Study Of Parameters Process Of The Injected Parts By Thermoplastic Material, The 20th International Conference DAAAM 2009-Danube Adria Association for Automation and Manufacturing, November, ISSN 1726-9679, (2009).

[3] N. Panc, L. Hancu, N. Bâlc, Research regarding the improvement of the performance of rubber dies, Proceedings of the World Congress on Engineering, Vol I, Londra, (2011) pp. 782-786.

[4] A. Popan, N. Balc, A., Carean, A. Luca, New industrial applications of the abrasive water jet processing, The 3rd International conference on Computing and Solutions in Manufacturing Engineering, COSME'12, 25th-26th October, Brasov, Romania. Published in Academic Journal of Manufacturing Engineering, AJME-2012, Vol. X, Issue 3, ISSN 1583-7904, (2012), pp. 97-102.

[5] D. Popescu, F. Popișter, R. Curta, C. Neamţu and A. Eles, Redesign Of "Safety Ring" Workpiece From Protection Assembly Of An Office Chair, Acta Technica Napocensis - Series: Applied Mathematics And Mechanics UT. Vol 57, No 4 (2014). pp 497-504.

[6] P. Bútora, A. Náplava, M. Ridzoň, J. Bílik, V. Tittel, Particle filled polyethylene composites used in the technology of rotational moulding. In Vedecké práce MtF STU v Bratislave so sídlom v Trnave. Research papers Faculty of Materials Science and Technology Slovak University of Technology in Trnava. Vol. 19, No. 31, ISSN 1336-1589, (2011), pp. 9-18.

[7] A. Luca, N. Balc, I. Drstvensek, A. Popan, Analysis of aluminum parts for accuracy improvement in vacuum casting process, Academic Journal of Manufacturing Engineering, AJME-, Vol. IX, Issue 3, ISSN 1583-7904, (2011), pp. 75-80.

[8] D. Leordean, C. Dudescu, T. Marcu, P. Berce, N. Bâlc, Customized implants with specific properties, made by selective laser melting, RAPID PROTOTYPING JOURNAL, Volume: 21, Issue: 1, 2015, ISSN: 1355-2546, pp. 98-104, FRI: 2,031;

Modern Technologies in Manufacturing
10.4028/www.scientific.net/AMM.808

Experimental and Theoretic Research Regarding Extrusion Optimization for Reinforced Polyamide (PA 6.6 – 10 % GF)
10.4028/www.scientific.net/AMM.808.125

DOI References
[6] P. Bútora, A. Náplava, M. Ridzoň, J. Bílik, V. Tittel, Particle filled polyethylene composites used in the technology of rotational moulding. In Vedecké práce MtF STU v Bratislave so sídlom v Trnave. Research papers Faculty of Materials Science and Technology Slovak University of Technology in Trnava. Vol. 19, No. 31, ISSN 1336-1589, (2011).
10.2478/v10186-011-0051-5
[8] D. Leordean, C. Dudescu, T. Marcu, P. Berce, N. Bâlc, Customized implants with specific properties, made by selective laser melting, RAPID PROTOTYPING JOURNAL, Volume: 21, Issue: 1, 2015, ISSN: 1355-2546, pp.98-104, FRI: 2, 031.
10.1108/rpj-11-2012-0107