"Injection Molding of Elastomers -Process Model with Statistic Experimental Design"

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1. Introduction

The advantage of injection molding is the gentle and even processing of a compound. The vulcanization of molded parts in the mold can start at a higher temperature level than is possible with other transformation processes. The result of this is shorter cycle times (1,2) (Figure 1).

In the last few years new attempts have been made to achieve a better understanding of the relations between processing parameters and the characteristics of molded parts (3,4,5,6,7,8, 9, 10, 11). Process regulations have been employed to control the injection molding process in tight tolerances on the basis of parameter characteristics (12, 13).

There hardly exist any models except for some approaches in the processing of thermoplastics, that make a correlation between processing parameters and product properties(14). Of course, the properties of a thermoset material are determined by their degree of cross link density (8). If this is true, the cross link density, which is obtained at the "so called" optimum cure time is the objective of the injection molding process. To have a successful strategy for such an investigation a measurement method of cross link density is advisable.

With the help of statistical process design (15, 16, 17), which does not see the injection molding process as "Black Box," a solution of this problem seems to be in sight. The aim now is to understand the dependence of the process parameters and then to make the outcome quantitatively predictable by a series of experiments using Statistical Design of Experiments.

2. General Considerations

Parameters important for the injection molding process seem to be so immense that an analysis leading to quantitative correlation equations may be almost impossible. There are speeds, pressures, strokes / volumes, times, temperatures, speeds and functions of many different kinds. All of these parameters have a contribution to the injection molding process (8, 9).

It is obvious, that some of the parameters contribute only to machine operation like the closing and opening speed of the mold or plastication delay time for example. Other parameters could contribute to the quality of the part strictly in the sense that they determine whether or not the part is an obvious failure, such as underfilling the cavity. Additional parameteres of this type include porosity, knit lines, blistering or part distortion.

There are also many parameters which influence directly the performance of the part. Of course the influence of the mold temperature and cure time on crosslink density is well known. The knowledge of the influence of the in-mold pressure on the spring constant is probably less well understood (24). Many other open questions exist, such as the effect of the plastification temperature on crosslink density and its statistical significance for example (8).

Before one starts to investigate and model the injection molding process, the stability and repeatability of each parameter during the experiment have to be verified and controlled. Each machine has not only to follow exactly the parameter settings, but more important is to get the

same result for a response even after a machine setting change. It has been shown that the injection molding machine has a similar precision as a "small scale" laboratory machine (6, 18). Based on this work it is concluded that the parameters are repeatable ehough to make an attempt at statistical model development.

2.1 Injection Molding Machine Considerations

Dosage accuracy of the machine is a parameters often selected as critical. The dosage depends on the exact measure of the stroke of the plunger and should be precise in the case of an ultrasonic measurement. It should be mentioned here, that the plunger should not be emptied, if after pressure is applied. Otherwise the after pressure can not have any effect. After pressure has to work on a rubber cushion to be effective. In case of sponge injection molding dosage accuracy is one of the most important factors because the plunger is always emptied. After pressure would hint proper cell formation.

Plunger speed has to be maintained constant during injection, meaning it should follow the setting precisely and if machine control allows several injection phases with different speed settings, it should accurate between switch overs (Figure 2). The machine has to be installed with enough hydraulic power otherwise will not be able to follow the setting. The higher the viscosity of the compound, the more important is the observation of this fact. One of the benefits of a constant plunger speed is the leverage of viscosity deviations of the compound because of material variations. In the given time interval, when the compound is injected at constant speed, the hydraulic pressure will vary in the same sense as the viscosity. If the compound is lower in viscosity than targeted, the injection work input will decrease in a linearly proportional manner, but the hydraulic pressure drop is less than expected. The result is less heat rise in the compound meaning less decrease of viscosity. The compound will reach the cavity at lower temperature. Considering the opposite extrem a higher viscosity compound will have a higher heat rise and yield less hydraulic pressure change than expected. The compound will reach the cavity at higher temperature. This will be discussed further with in-mold pressure variation.

If we consider the compound temperature as a result of the plastification prior work has shown this to be of little variation (Figure 3). Within one batch it is possible to consider the material influence as excluded. After a certain number of cycles the machine will reach its steady state. It is very important of course to start measurements after steady state is achieved. In most experiments stability is reached after approximately more than five cycles. The response "Plastification Temperature" (TM) shows a value inside the measurement error. When the compound is ejected out of the nozzle in the open without a mold, one observes a steep temperature rise and later reaches a plateau for the remaining ejection time. This temperature value is considered also inside the measurement error. The Effect of this behavior on hydraulic pressure and factor selection for the statistical model will be dicussed later

The sensitivity of the hydraulic pressure is shown when affected by temperature variations of the compound developed during residence time of the compound in the filled plunger before injection (Figure 4) (19). In the case of a cold plunger temperature, setting the layer in contact to the steel surface of the plunger will cause the material to cool down. Because of low

temperature conductivity the inner layers of the compound will keep most of the temperature. During injection we observe the instability in the heat rise curve in conjunction with pressure peaks in the hydraulic. If we take the temperature setting close to the compound temperature developed during heat rise from plastication, the instability in the heat rise curve during ejection disappears and the hydraulic pressure shows sufficient stability in this process phase. The reason that one can not consider the plunger temperature as an independent variable will be discussed later in this paper.

Mold temperature has most of the influence on vulcanization of the compound. The influence is twofold. During injection the compound will not only heat up by "injection work input" but also by conductivity. After injection of course heat conductivity is the only reason for heat rise neglecting the contribution of vulcanization heat. In this experiment the heating up phase of a mold is shown and the temperature measurement after the setting is changed (9). We have taken in to account that this temperature behavior should be the same for all cavities of a mold and the temperature differences should be negligible, otherwise part performance deviations will be the result.

Finally no measurement is published of in-mold pressure variation in Rubber Molding to our knowledge. But experiments has been done with thermoplastic material (Figure 5)(20). The dependencies of the in-mold pressure at least are the same for both materials because both are processed as a polymer melt. The example shows some variation and data are not "normal" distributed. In the case of injection molding of rubber a process regulation for the after pressure may help to decrease the variation of the in-mold pressure.

3. Experimental Considerations

One way to investigate the injection molding process is to list all influencing factors and categorize them estimating the amount of influence on the part performance or similar criteria. To solve this problem the possibility of dividing the injection molding process into steps without losing track of the whole process has to be investigated (Figure 6).

The injection molding process can be divided into three different steps, the plastification, injection and vulcanization. The unloading and loading should be ignored in this analysis because its irrelevance on part performance. This statement can be made under the assumption, that this process step has no effect on the factors or the process. The relation of the factors to the responses can be investigated now with regression analysis. Still the number of factors is too high and the results of the experimental design will be poor. Therefore the precondition to solve this problem is the classification and the separation of the process factors into clear independent and dependent parameters. For the design purpose only the exclusively independent factors can be chosen and a link to properties of the molded parts can be created. To minimize the amount of experiments to be performed for the design, one has to take over a response as an independent factor into the next step.

Starting the process with the feeding of a strip of rubber in the plastification unit one considers the following factors: screw cylinder temperature [TSC], back pressure [PSTAU] and screw

speed [VSC]. These factors can be set different and independent from each other. The responses are the output, the power consumption of the screw motor and the compound temperature [TM]. This is a three-factor design. The correlation with the responses will be discussed in detail.

For the injection phase we have the injection speed [VS], and the mold temperature as independent factors. It is known, that the viscosity is a determining factor for the work input or the yield hydraulic pressure during injection, but one has to consider the viscosity as a response. The viscosity is a result of the temperature the compound already reached after plastification [TM] and the further temperature rise due to the conversion of the injection energy to heat. This means the missing independent factor for the injection phase is the compound temperature after plastification [TM] but before injection. The correlation of TM with the factors of the plastification are known from the investigation of the first process step, which enables one to choose a parameter set for the plastification phase that yields a target TM value needed for an Experimental Design of the injection phase. This TM has to be taken as the missing independent variable to analyze the injection phase. This is now the second three-factor design.

The third process phase is the vulcanization. The vulcanization is said to be dependent on temperature and time. It is also known, that — for the optimum vulcanization — either the time is dependent on temperature or the opposite. If one takes the temperature like in the previous process step as the independent factor, one has to consider the time as a response. This is opposite to most attempts, where the time is taken as a factor. As pointed out, the actual pressure in the cavity during vulcanization may also have some influence on the vulcanization properties, meaning part performance. The actual cavity pressure is a result of the after pressure, which can be taken as independent. Searching for a third factor one finds the vulcanization speed, but this means the compound is not seen as constant. If one chooses the compound as a factor one gets again a three-factor design for the injection phase. In case of a constant compound — neglecting any compound variation — the result is a two-factor design for the vulcanization phase.

The temperature development of the compound in the cavity during vulcanization is dependent of course on the temperature conductivity, the heat capacity and the reaction heat of vulcanization. These strictly compound-related factors should be considered as constant. They contribute mostly to the in-mold pressure after the gate of the cavity is sealed because the vulcanization progress and the after pressure have no effect anymore on the in-mold pressure

As one can see there exist almost three independent factors for each phase. Using the methods of the statistical experimental design the injection molding process can be worked out in three Designs of Experiments with three variables, with a defined number of experiments. Now description and quantification of the injection molding process and the effects on the molded parts performance is possible (1, 8, 12). Also the costs for these experiments and the benefits for production can be estimated in terms of a cost-benefit analysis before starting such an investigation. With the help of these three designs it should be possible to find the best settings for the injection molding which makes it possible to achieve an operating window for zero defect production within the shortest cycle time possible.

An extension of this approach is, that other compound factors can be considered like polymer, fillers and oil, with the resulting measurable responses like compound viscosity, heat capacity, and many others.

3.1 Special Remarks on the Plastification Phase

The choice of the plastification parameter - screw speed, back pressure and the wall temperature of the screw cylinder - determines the compound's mass temperature extruded in the plunger cylinder. Temperature homogeneity is seen as a result of screw speed and back pressure and determines the plastification time. Again the aim of plastification is to reach a certain mass temperature. With the help of a statistical process model the connection between the regulation parameters and the mass temperature is made calculable. Looking at the factors and responses one will notice that the parameter plunger cylinder temperature belongs to the dependent factors and is not considered. An explanation for that can be derived from the hydraulics' reaction while injecting the compound.

3.1.1. Correlation between screw- and plunger cylinder temperature

The energy, which is consumed by the compound during plastification, depends on the viscosity and the heat capacity of the compound. The viscosity is determined by the polymer, the filling level and the oil. The effect of the screw cylinder's temperature, the plunger temperature, the backpressure and the screw speed will be determined in the first experiment with an EPDM compound (1).

With this EPDM compound one receives after plastification a compound temperature of 86*C. The machine parameters for the screw cylinder is at 70*C, for the plunger cylinder at 90*C, the screw speed is 55 rpm and the back pressure is 10 bar. The compound temperature shows very little dependence on screw speed and backpressure.

While injecting the compound the temperature development and the hydraulic pressure were observed. The hydraulic pressure curve showed pressure peaks, while the temperature curves showed deflection, which disappeared at a higher plunger cylinder temperature setting. The relation of the shape of the temperature curve correlates with the hydraulic pressure peaks and could be confirmed in extended trials.

The plunger temperature has an effect on the outer layers of the compound that is extruded into the plunger cylinder. At a plunger temperature of more than 10*C below mass temperature pressure peaks in the hydraulics can be observed during injection. These pressure peaks will vanish if the plunger temperature is adjusted to the mass temperature by 5*C. A steady temperature rise on the temperature probe of the machine nozzle can be observed, while at higher temperature differences there is a steady increase (first derivative of curve). The plunger temperature should be adjusted in that way so that the mixture will not cool down in the outer layers while it stays in the plunger room. The temperature of the plunger cylinder

shall not deviate by more than 10*C from the compound temperature [TM]. For compounds, that reach much higher temperatures than 110*C, it is indicated for safety reason to set the plunger cylinder temperature at a lower degree as recommended to avoid scorch (19). This rule prevents the cooling of the compound in the outer layers and extensively also the danger of scorch in the plunger room. At a lower temperature of the plunger wall the induced hydraulics variations will have a negative effect on the stability and repeatability of injection as well as on the after pressure regulation (underfilling, overfilling).

To summarize, the temperature of the plunger cylinder can not regulate freely according to these findings, but depends on the factors: back pressure, screw speed and screw cylinder temperature.

4. Experimental Part

The plastification phase is investigated now according to above results with compunds based on polymers of different provenience. The aim was to achieve regression equations for the plastification. The tests have been carried out with an EPDM and NR compound (1).

4.1 Plastification of an EPDM compound

At a screw speed of 55 rpm up to 80 rpm, a back pressure of 6 to 16 bar and a screw cylinder temperature of 65*C up to 80*C, a compound temperature of 77*C up to 84*C and an output of 1,52 g/sec - 3.9g/sec are observed (Figure 7). The influence of the factors on the compound temperature is relatively small while the influence on the output is relatively big. Considering the fact that the quadratic analysis of a statistical experimental design allows only a limited choice of area (15) the results for the compound temperature and the output pinpoint to wall slippage. At a 70,5*C temperature of the screw cylinder and low back-pressure, the mass temperature grows slowly with regard to the screw speed. At an increased back-pressure a linear increase of the compound temperature with rising screw speed can be observed. There exists a linear dependence between output and screw speed over the entire back pressure area. At low back-pressure and increased screw speed, less energy will be absorbed and then being transformed into heat. An increase of the screw cylinder temperature will shift these effects to a higher screw speed. This observation is in accordance with corresponding testing results of the wall slippage (21).

4.2 Plastification of a NR compound

The third example deals with a compound on a NR basis (Figure 8). A far-reaching linear dependency of compound temperature and power on the screw speed can be observed. The mass temperature difference between maximum and minimum value within the framework of the testing conditions is relatively unimportant (10*C). Even the screw cylinder temperature has only a minor influence on the mass temperature. As expected, back pressure and cylinder temperature has a negative effect on the output. The back pressure's negative influence on the

mass temperature does not vary too far from the tests' errors of measurements and therefore shall not be overvalued.

4.3 Examination of results from plastification process

The result of these tests is a family of regression equations, which describe the influence of the factors (independent variables) on the responses (yield parameters) of the plastification phase. Considering these equations the compound temperature and the output for the compounds investigated can be calculated. For reasons of simplification and clarification effects of secondary order have been neglected and only coefficients of first order have been listed in the table. The coefficients have the same magnitude and direction for all tested compounds except for the Intercept [A0] (Figure 9). This coefficient shows the same order as the viscosity of the compounds independent of the type of rubber. We are able to have such a simplified look at these tests if it can be supposed that the heat capacity or similar material values assume similar magnitudes for all compounds or if they depend on the filler to oil ratio and loading. Though it is right that the viscosity of a mixture determines the yield of the compound temperature at plastification (as far as no other effects like the wall slippage effect for example appear) a machine adjustment algorithm can be achieved with the help of information about a few model compounds. In practice it can be used to start up a machine and it only needs little correction for the production period.

5. Injection Phase

The Injection Phase means that the compound is transferred through runner and gates into the cavity. The compound flows through a runner with different diameters and has some heat exchange with the surface of the runner. While injected the compound's temperature rise from the temperature [TM] achieved during plastification by work input conversion to heat to a higher temperature [TMinj]. The integral of pressure difference time differential injection time is the injection energy, which is dissipated into temperature.

$$(1) E = \int \Delta p \bullet dt$$

The injection phase is investigated in a four-factor design (Figure 10). A peroxide accelerated EPDM compound is used for these trials (10, 11). The after-pressure is added as a fourth factor in this experiment, because the after-pressure regulation of the injection machine used was set inactive. At least this allows to observe the influence of this factor on the in-mold pressure. First the results are discussed at constant after-pressure. The runner / mold temperature is varied between $\pm 10^{\circ}$ C.

The hydraulic pressure needed to inject the compound and to fill the cavity is almost dependent linearly on the temperature of the compound TM and the injection speed. Same hydraulic pressure consumption during injection can be obtained with either a TM of 125*C and a speed of 30% or a TM of 140*C and a speed of 50%. This knowledge is important because it allows for keeping the machine operating in a safe mode. Excessive consumption of hydraulic pressure gives inconsistent injection times (22).

Surprisingly, the temperature [TMinj], which is the temperature of the compound when reaching the cavity, shows very little variation. They are two explanations possible. At low TM the compound needs higher pressure and takes more energy than at higher TM. The result is the same Tminj. The other explanation is a measurement error.

While the TMinj varies two degrees, the pressure measured in the cavity near the runner [PW] shows a difference of 20 bar. The values were taken as a maximum pressure obtained at the end of the holding time. At this time one should consider an influence of the thermal expansion on the pressure while the vulcanization shrinkage at this process stage will not be observed.

6. The vulcanization

In the above-described experiments the vulcanization is done at a constant vulcanization time. Under these condition a significant influence of the TM and the injection speed on elongation is observed. But as pointed out there should be little but some influence of the TMinj because the small variation of this response, resp. factor. The modulus of the vulcanizates, taken as a synonym for crosslink density, had very little variation, but the elongation at break had some. With this unacceptable measurement error one needs a more precise measurement in the cavity. which excludes all influences through handling. The impedance could be the sufficient tool (23). If one varies the injections speed, leaving all other factors constant, one can see, that vulcanizations speed is obtained different (Figure 11). The point, where the curves reach a plateau, can be taken as optimum cure determining the vulcanization time. Also the variation of mold temperature, which is of much bigger influence on cure speed than TMinj of course, allows to identify the optimum cure time (Figure 12). It can be concluded, that impedance measurement allows the in-line measurement of the cure time, which is a necessary if we want to consider the time as a response. It gives us also the possibility of a closed loop control of the crosslink density in production. With the help of this technique the concept developed in this paper can be proved.

7. Summary and conclusion.

Statistical Experimental Design can be used to describe the Injection molding process quantitatively. Only a few experiments are needed because the process steps can be separately investigated and most responses show linear dependence from the factors identified as clearly independent. A careful test of the capability of the injection molding machine is a precondition otherwise the statistical model becomes insufficient

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9. Abbreviations

*C Degree Celsius

EPDM Ethylene Propylene Diene Elastomer

NBR Nitrile Butadiene Rubber

NR Natural Rubber

PSTAU Back Pressure

PW Cavity Pressure close to Gate

TM Mass Temperature after Plastification

TMinj Mass Temperature after Injection at Gate

TSC Temperature of Screw Cylinder

T Temperature

VSC Screw Speed

VS Injection Speed

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Fig.1: Comparison of Compression versus Injection Molding

- a -unvulcanized
- b -undervulcanized
- c vulcanized
- d over cured
- 1,2 Plastification
- 2,3 Time in Plunger before Injection
- 3,4 Injection
- 4,5 Conduction Heat in Mold

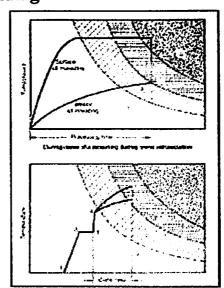


Fig.2: Accuracy of Plunger Speed during Injection

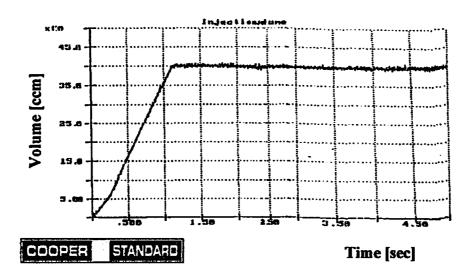


Fig.3: Variation of Compound Temperature after Plastification [TM]

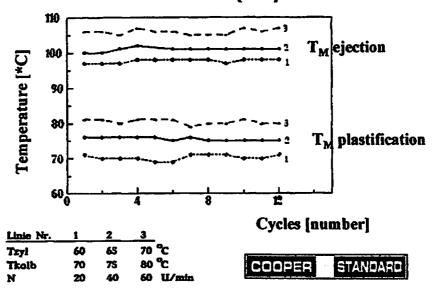


Fig.4: Temperature during Ejection in the Open and Corresponding Hydraulic Pressure

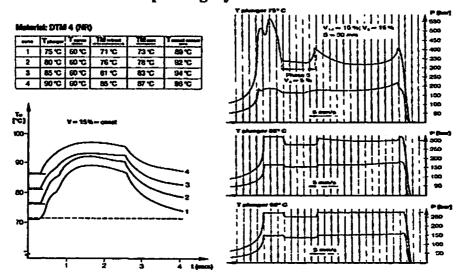


Fig.5: Pressure Distribution inside the Cavity after Injection

Injection of Thermoplastic Material

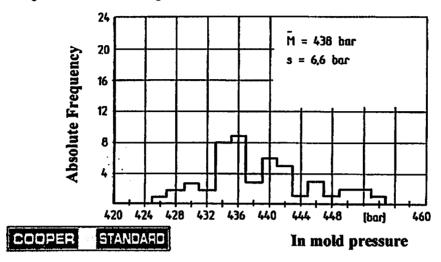


Fig.6: Statistical Process Model Building

- Model should allow:
 - Precess setup for
 - shortest cycle time
 - but 6Sigma capability at the same time
 - Process Varition Control with
 - Injection Work Input Observation
 - After Pressure Regulation
 - Heating Time Control / Regulation?

Fig.7: Plastification of an EPDM Compound

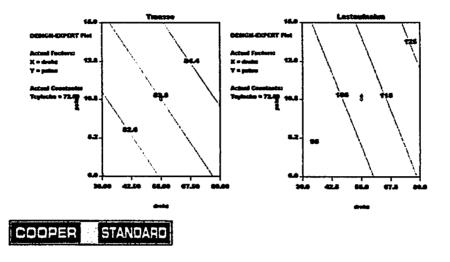
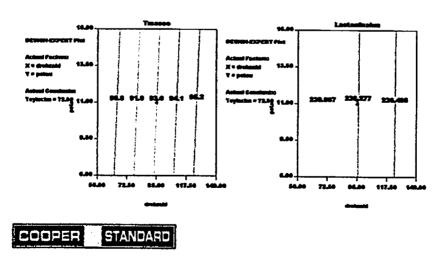


Fig.8: Plastification of an NR Compound



4

Fig.9: Regression Analysis of Plastification

	Response	Ao	A _I (T _{cyl})	A ₂ (P _{Stau})	A ₃ (S _{Speed})
EPDM	T _{Masse}	61	0.25	0.2	0.04
	Work Input	138	-1	1.5	0.52
	Output	-0.74	0.02	-0.06	0.05
NR	TMasse	55.8	0.42	-0.03	0.07
	Work Input	486	-3.3	0	0.06
	Output	4.85	-0.03	-0.15	0.03

Fig.10: Dependence of TMinj on Injection Speed

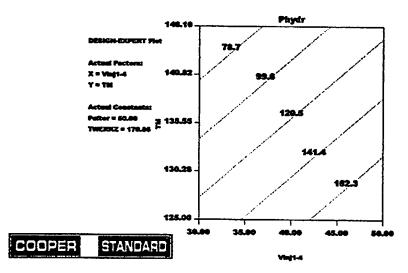


Fig.11: Dependence of Impedance on Time at different Injection Speeds

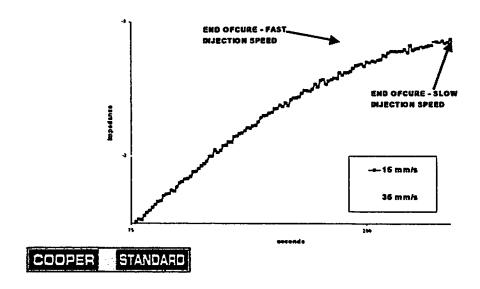


Fig.12: Dependance of Impedance on Time at different Mold Temperatures

