Using design of experiments to improve precision glass moulding

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Abstract: A design of experiments (DOE) approach was used to characterise the effect of process parameters, including heating and cooling rates, soaking times, moulding viscosities, and forces applied during the moulding and cooling stages on the repeatability of the final thickness of moulded N-BK7 and LBAL35 glasses using a precision glass moulding (PGM) machine. Analysis of the DOE showed that process parameters that lengthen the overall time of the moulding process tend toward more repeatable final thicknesses. Using the ideal parameters found by the DOE, the error in the final thickness was held within ±0.05 mm.

Keywords: precision glass moulding; PGM; design of experiments; DOE; optical fabrication.

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Kathleen Richardson is currently a Professor of Optics and Materials Science and Engineering at CREOL/the College of Optics and Photonics at the University of Central FL, and runs the Glass Processing and Characterization Laboratory (GPCL), carrying out design, synthesis and characterisation of novel glass and glass ceramics for infrared optical applications. He is a Fellow of ACerS, SPIE, OSA and the SGT, and has served as a past and current Director on the Boards of the American Ceramic Society and SPIE, respectively.

1 Introduction

Optical fabrication is an important aspect of current high performing optical systems. Traditional grinding and polishing techniques can fabricate spherical lens elements with tight tolerances, and in high volumes. However the performance of these spherical lenses elements does not match that of aspherical lens elements. To meet the demands for lighter, cheaper, or more compact systems, design has shifted toward the use of aspheric or freeform optical elements, such as those presented in Yi et al. (2006). These elements present new fabrication challenges as one or both of their surfaces deviate from a spherical shape. Non-traditional fabrication methods for these aspherical lens elements include precision glass moulding (PGM) and single point diamond turning (SPDT). However, as discussed by Zhou et al. (2006) tool wear issues with diamond turning of brittle materials such as glass, can be a limiting factor in the viability of SPDT for aspherical lens elements. Work done by Firestone et al. (2005) and Wachtel et al. (2013), on the other hand, have demonstrated two separate, custom-designed, PGM machines for fabricating spherical and aspherical lenses as well as microlens arrays. Additionally, Yi and Jain (2005) showed the ability to mould ‘large precision aspherical lenses’ with surface variations and irregularities ‘comparable to or better than lenses produced using convention methods’. Results on the importance of individual process parameters have been published several other authors: Tsai et al. (2008) investigated force-displacement relationships, Zhao et al. (2009) investigated cooling rate relations to refractive index, and Fischbach et al. (2010) investigated sticking between the glass and mould as a function of cooling times, pressing times, and pressing forces. While each of these results

are important to understanding PGM, we unaware of a larger-scale investigation on the importance of the processing parameters, particularly as they pertain to the variability of the final thickness of the moulded optics. With these recent advancements in mind, an evaluation of several PGM process parameters on resulting part uniformity has been undertaken.

In order to produce precision-moulded optical elements, the effect of process parameters used during the moulding cycle, such as moulding force, moulding temperature (or viscosity), cooling rates, etc., and their impact on the final, post-pressed form and optical quality of the work piece must be understood. For example, recent work by Zhao et al. (2009) showed that variations in cooling rates and moulding temperatures have an effect on the observed drop in refractive index after the PGM process.

However, in order to expand the analysis to incorporate additional parameters and set points, an even larger number of experiments must be conducted in order to quantify the effects of each parameter independently. Changing one of these parameters often changes the thermal expansions and/or the thermal histories of both the instrument and the workpiece and thus requires new instrument baseline and standard measurements; this results in such testing becoming time intensive in either the research laboratory or on the production floor.

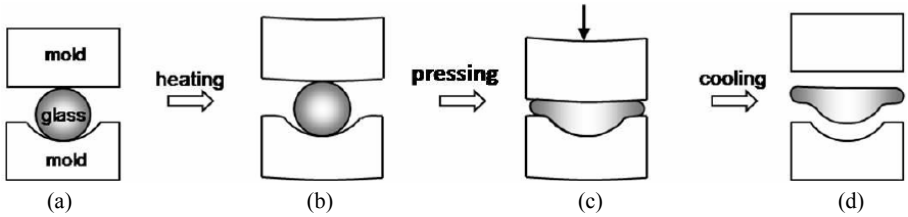
As discussed by Taguchi et al. (2004), using a design of experiments (DOE) can be an effective method for determining the effects of each process parameter on the final metric (in this case, the final thickness of a moulded optic), while simultaneously minimising the number of experiments needed to determine these results. A custom DOE incorporating six process parameters important to PGM has been employed in the present study to evaluate the impact of this multitude of tool and workpiece process variables, on final part form. The DOE was performed on two oxide glasses: Ohara's LBAL-35, a 'low T_g' glass (Tg = 527°C), and Schott's N-BK7, a 'high T_g' glass (T_g = 557°C). LBAL-35 was chosen based on previous thermal and structural characterisation performed by Gaylord et al. (2010), and predictive modelling conducted by Ananthasayanam et al. (2012). Similar work on N-BK7 was performed by Gaylord (2008) and Mosaddegh (2010). The analysis of this initial DOE allowed for a reduction of the number of parameters, and the creation of a smaller, more specific, DOE for further study. The modified DOE details the importance of the remaining process parameters, as well as their optimal values for the PGM process.

2 Precision glass moulding

PGM is a multi-step process, where each stage has set points that can be varied. The first stage, or heating stage, involves heating the glass sample to a temperature above its glass transition temperature (T_g), where it becomes less viscous with increasing temperature. Depending on the glass type, heating rates during this stage range from approximately 50°C per minute to an upwards of 150°C per minute, as demonstrated in Ananthasayanam et al. (2012). During the second stage, or soaking stage, the glass sample is held at a constant temperature for a set time, and allowed to thermally equilibrate. The so-called 'soaking time' typically ranges from a few seconds second to a few minutes, and will be affected by both the material volume being heated and by its thermal transport properties, which dictate the time required to reach 'equilibrium'. The third stage involves applying a pressing or moulding force to the glass sample, which

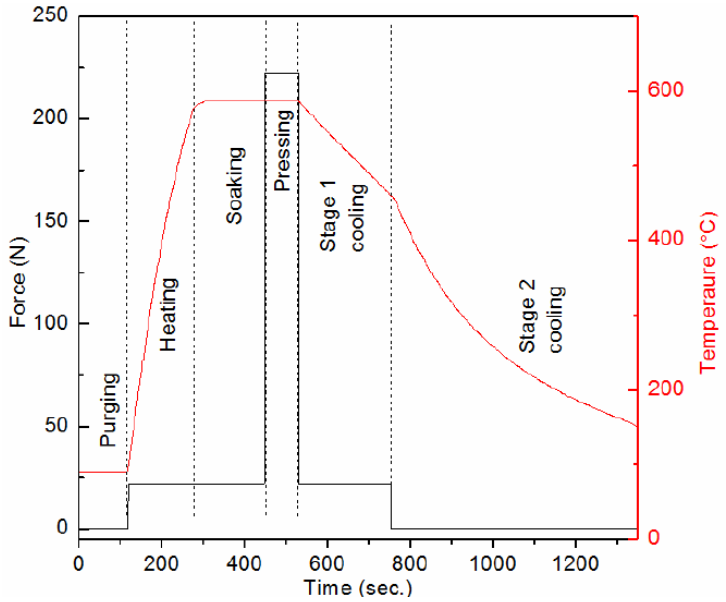
conforms the sample to the shape of the adjacent mould surfaces. Moulding forces during this stage range from approximately 300 N to 1,100 N. It is assumed at this stage that the glass is fluid enough at the equilibrating 'soak' temperature that the applied load causes the material to flow to conform to the shape of the mould cavity. The final stage is the cooling stage, which is further separated into two sub-stages. During the first cooling stage, the sample is slowly cooled below its T_g at a constant rate, while a constant 'maintenance' force is applied to keep the mould surfaces in contact with the workpiece. During the second cooling stage, the maintenance force is removed and the sample is cooled at an increased rate since the glass' thermal history below T_g is assumed to be independent of cooling rate. The cooling rate and maintenance force during the first cooling stage in this study range from 10–40°C per minute and approximately 20–450 N, respectively. The moulding process is depicted in Figure 1, and a typical temperature and force cycle is outlined in Figure 2.

Figure 1 PGM process



Source: Mosaddegh (2010)

Figure 2 Typical thermal and force cycles during the PGM process (see online version for colours)



Source: Wachtel et al. (2013)

3 Design of experiments

The goal of this investigation was to determine the effect of commonly used PGM process parameters on the repeatability of a moulding cycle outcome. The metric used for the evaluation was an assessment of the centre thickness repeatability of a post-moulded optic. Because of the broad range of values the moulding parameters can take, a DOE approach was used to minimise the number of experiments needed while maximising their significance. The orthogonal array method, or Taguchi method, was used to design the set of experiments that needed to be conducted. An $L_{18}(21 * 3^7)$ matrix allows for the characterisation of eight orthogonal (linearly independent) process parameters, and their relative impact on the chosen metric (in this case the centre thickness of the moulded optic).

The design of the $L_{18}(21 * 3^7)$ matrix requires one parameter to be tested at a high and low value, while the remaining seven parameters attested at high, medium, and low values. To accommodate this formalism, the glass type was chosen as the first parameter (to incorporate the two glass types), permitting testing of each machine parameter at a high, medium, and low value. This DOE approach required 18 separate experiments, as compared to the 1,458 unique experiments required by a full factorial analysis to characterise each of the parameters and their set points.

Table 1 Experiments defined by the DOE L_{18} matrix with the corresponding parameter set points

Experiment	*Material*	*Log_{10} viscosity (Pa • S)*	*Heating rate (°C/min)*	*Soaking time (sec)*	*Moulding force (N)*	*Cooling rate stage 1 (°C/min)*	*Cooling stage 1 moulding force (N)*
1	L BAL 35	7	50	1	330	10	22
2	L BAL 35	7	75	120	660	20	220
3	L BAL 35	7	100	240	1,100	40	440
4	L BAL 35	8	50	1	660	40	440
5	L BAL 35	8	75	120	1,100	10	22
6	L BAL 35	8	100	240	330	20	220
7	L BAL 35	9	50	120	330	20	440
8	L BAL 35	9	75	240	660	40	22
9	L BAL 35	9	100	1	1,100	10	220
10	N-BK7	7	50	240	1,100	20	22
11	N-BK7	7	75	1	330	40	220
12	N-BK7	7	100	120	660	10	440
13	N-BK7	8	50	120	1,100	40	22
14	N-BK7	8	75	240	330	10	440
15	N-BK7	8	100	1	660	20	22
16	N-BK7	9	50	240	660	10	220
17	N-BK7	9	75	1	1,100	20	440
18	N-BK7	9	100	120	330	40	22

In this study, two materials and six process parameters, each with three set points, have been used. Because of the orthogonality of the parameters in the designed matrix, eliminating one parameter from the analysis will not affect the results of the remaining parameters. This characteristic will be exploited below to reduce the size of the matrix in subsequent tests and refine the accuracy of the results. Table 1 lists the parameters and their set points that are required by the $L_{18}(21 * 3^7)$ matrix.

Viscosity (η) was chosen as a set point rather than a specific moulding temperature to normalise the effect of the chosen glass. If a single temperature was chosen rather than a viscosity, different glasses could not be directly compared, as each glass has for its own unique chemical composition, an unique η-T curve. For example if the temperature is below the glass transition temperature (T_g) of one sample, it will act as a brittle solid. Another glass type subjected to the same temperature could be well above its T_g, and act as a viscous fluid. For example if a temperature set point of 565°C was chosen, the NBK7 samples would essentially be at a log viscosity of 12 Pascal-seconds (Pa.s), while the LBAL35 would be at a log viscosity of 9 Pa.s. This difference represents a three order of magnitude shift when comparing the two glasses. For this reason, it was determined that moulding viscosity, rather than moulding temperature, was the appropriate metric.

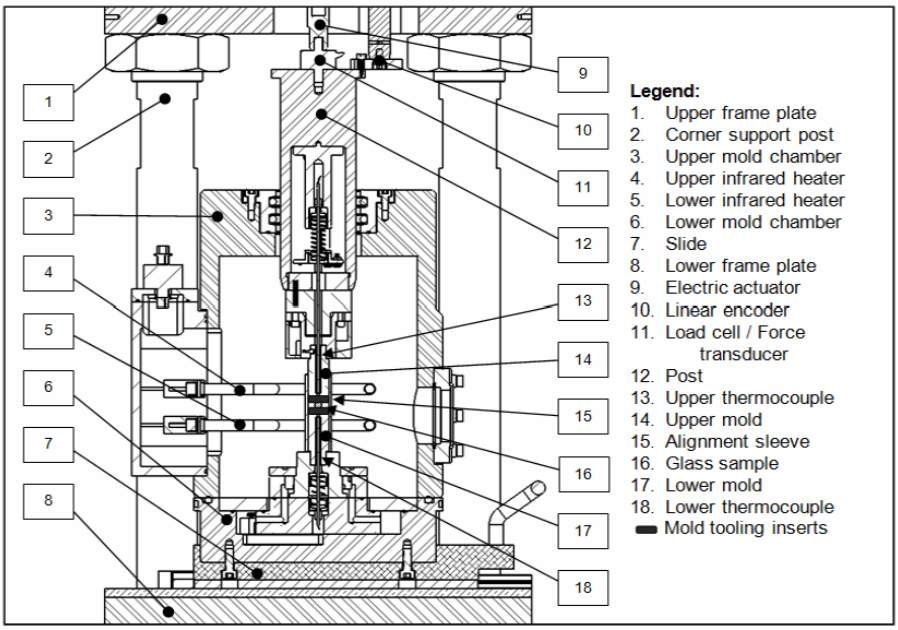
4 Experimental study

All moulding experiments were conducted using a research grade PGM machine manufactured by Dyna Technologies Inc. as described in Wachtel et al. (2013). DTI's GP-5000HT is a bench-top moulding machine with the precision of a standalone moulding machine, which also possesses the flexibility, functionality and control over process parameters needed for research and laboratory testing.

For each experiment required by the DOE, five samples were moulded into post-pressed plano flats. Each sample was initially a plane parallel plate (PPP) disk with a thickness of 2 mm and a diameter of 5 mm; the samples had an optical grade inspection polish on the two parallel circular faces. At the beginning of each set of experiments, the tungsten carbide (WC) mould surfaces and all glass samples were cleaned with acetone and ethanol. The PPPs were moulded to a final target thickness of 1 mm. Figure 3 shows a simplified version of the moulding setup including the upper and lower moulding surfaces, a thermally conducting sleeve, and the infrared heaters.

Previous experiments from Wachtel et al. (2013) have shown that the first three moulding cycles of each day constitute a 'warm-up' phase, in which the standard deviation of the final thickness of the moulded parts is larger than that obtainable in subsequent pressing cycles. After the first three moulding cycles the thicknesses of the moulded parts are much more consistent, thus warm-up measurements are not included in any subsequent evaluation. This increased range of final thicknesses is due to small-scale thermal expansion changes of the moulding machine itself due to the rapid thermal cycling experienced during the moulding process. As the number of cycles progress, the system reaches a 'steady-state' phase and the changes due to thermal expansion between the moulding cycles becomes negligible. To avoid this unwanted error, all experiments were conducted after the warm-up period had ended.

Figure 3 Cross-section of the GP-5000HT revealing its critical components



Note: The pneumatic air cylinders that lift the upper mold chamber along with the control boxes, which contain the electrical equipment for operating the GP-5000HT, have been excluded.

Source: Wachtel et al. (2013).

The post-moulded thickness of each piece was measured to determine the average thickness of the experimental set. These thicknesses were used to determine the error associated with that specific set of process parameters. The surface roughness of the samples and mould tools were measured after each set to track the part or mould surface degradation or failure to assess ultimate lifetime of the tools. It is worth noting that the lifetime of the WC mould tools used in our studies to date (estimated to be 140 cycles between room temperature and moulding temperatures above 500°C) far exceeds the number of moulding cycles required for this DOE (45 cycles).

5 Initial results

After the thicknesses of the samples were measured, individual thickness values were compared to the average thickness observed for their given set. The difference between the average thickness of the set and the individual piece thickness was taken, and normalised so that average of the thicknesses are centred about 1 mm (the target thickness). This normalisation stems from the observation in previous experiments that, given enough time and resources, the moulding process could be adjusted to achieve a desired final thickness. These normalised thickness values are labelled as P_i values in the Taguchi formalism, where i is the sequential number in which the experiment was

conducted. Each experimental dataset will have five P_i values, one for each sample moulded. The P_i values for all 18 experiments are outlined in Table 2.

Table 2 P_i values and SNR values for each experiment defined by the initial DOE

	P_1	P_2	P_3	P_4	P_5	*SNR*
1	0.9797	0.9892	1.0119	1.0139	1.0053	36.5190
2	0.9928	0.9989	0.9973	1.0031	1.0080	44.7605
3	1.0064	0.9902	1.0166	0.9875	0.9993	38.4775
4	1.0015	1.0278	0.9939	0.9858	0.9910	35.6143
5	1.0044	1.0025	1.0033	0.9969	0.9929	46.2310
6	0.9971	0.9926	0.9958	1.0163	0.9983	40.5995
7	0.9903	0.9881	0.9978	1.0494	0.9745	30.8004
8	1.0059	1.0043	0.9972	0.9995	0.9931	45.6290
9	0.9762	1.0011	1.0461	1.0001	0.9766	30.9128
10	1.0032	1.0118	1.0072	0.9979	0.9800	38.2116
11	0.9926	0.9862	0.9940	1.0092	1.0180	37.6484
12	1.0094	1.0055	0.9857	0.9910	1.0084	39.2553
13	1.0027	1.0105	0.9855	1.0042	0.9972	40.5372
14	0.9723	0.9899	0.9799	1.0405	1.0175	30.9415
15	0.9951	0.9989	1.0019	1.0047	0.9994	48.9331
16	1.0134	0.9880	0.9990	1.0030	0.9967	40.6731
17	0.9980	1.0036	0.9916	1.0096	0.9972	43.3188
18	0.9896	0.9691	0.9879	1.0289	1.0245	31.7929

From the P_i values, four additional metrics were calculated. The sum squared average and the sum of the squares are given by S_m (1) and S_t (2), respectively. The difference between these two parameters is given by S_e (3). The parameter V_e (4) is then calculated as S_e divided by one less than the number of experiments run. The signal-to-noise ratio (SNR) is given by (5). The SNR for each set of experiments is outlined in Table 2. For the analysis of this particular experimental design, a larger-the-better SNR is desired. Since this was a preliminary investigation of the effect of the process parameters, there was no specific target value for the SNR, rather just an initial analysis of which parameters would contribute to the larger-the-better SNR.

$$S_m = \frac{\left(\sum_1^i P\right)^2}{i} \tag{1}$$

$$S_t = \sum_1^i (P^2) \tag{2}$$

$$S_e = S_t - S_m \tag{3}$$

$$V_e = \frac{S_e}{i-1} \tag{4}$$

$$SNR = 10 \times \log_{10}\left[\frac{1}{i} \times \frac{S_m - V_e}{V_e}\right] \tag{5}$$

The Taguchi method analyses the SNR of the parameter set points, rather than the individual experiments. To achieve this requirement, the SNR for a parameter set point is defined as the average SNR for each experiment involving that set point.

The difference between the maximum and minimum SNR for a parameter defines the parameter's importance. The larger the difference between the maximum and minimum SNRs, the greater impact a change in that parameter will have on the target function (final thickness in this case). The SNR for each parameter set point, along with the difference between maximum and minimum SNR and importance ranking, is outlined in Table 3.

Table 3 SNR of the set points for each parameter, and the importance ranking of the parameters

Parameter set point	*Glass*	*Viscosity*	*Heating rate*	*Soak time*	*Mould force*	*Cooling rate*	*Cooling force*
1	38.8349	39.1353	37.0978	38.8343	34.7170	36.4921	41.2295
2	39.0638	40.5248	41.4217	38.9298	42.4825	41.1142	39.2273
3	-	37.1878	38.3285	39.0838	39.6485	38.3167	36.3912
Difference	0.2289	3.3370	4.3238	0.2495	7.7655	4.6220	4.8383
Importance rank	7	5	4	6	1	3	2

According to the Taguchi method analysis, which suggests more important parameters are characterised by larger differences in SNR values, the most important parameter observed during the moulding cycle in our study is the moulding force, followed by cooling force, then the cooling and heating rates. However during the moulding cycles with the viscosity at its lowest set point ($\log_{10}\eta = 7$ Pa.S), the targeted moulding forces were never reached. When the viscosity is at this minimum set point, the glass sample is too fluid and will reach its final thickness quickly after only a small amount of force is applied. Because of the increased fluidity of the glass, the moulding cycle was completed well before the final moulding force was reached. This interrupts the framework of the Taguchi method, and therefore this result cannot be trusted without additional confirmation. In order to correctly analyse the effect of the process parameters on the centre thickness repeatability, a revised DOE that takes these initial results into consideration will need to be carried out.

Despite the initial failure of the framework of the Taguchi method, several key observations can be made. First, the range of viscosities (three orders of magnitude) was too broad. The lower viscosities, nine higher temperatures, caused the glass to flow too easily and the required moulding forces were unattainable. The variation in viscosity could also be masking the effect of the other parameters. Therefore, to improve the results of this analysis the set point of the viscosity was held constant, at a $\log_{10}$ viscosity of 9 Pa.S, for further experiments. Second, because of the extremely small difference in SNR, the initial results indicate that switching the glass type may not play an important role in the error of the final thickness of the moulded optic. The temperature dependent viscosity curves differ between glasses, but by using the viscosity rather than the

temperature as the parameter of interest, the material properties are essentially normalised. The two glasses investigated are in the same oxide glass family, with only slight differences in modifier concentration, and work done by Angel et al. (1989) has shown that the fragility, or temperature-dependent viscous response, for similar glasses is closely matched. Thus, we can assume that within a glass family, the viscous response of the glasses will be very similar.

6 Revised DOE

Based on the results from the first DOE discussed above, it was assumed that the effect of the glass type (within a glass family) was negligible, and that the viscosity needs to remain constant in order to clarify the roles of the other process parameters. Therefore, these two parameters were held constant in our second DOE (i.e., only one glass type and a constant viscosity of 107 Pa.s). The effect of the viscosity is of great importance, but because of its large 'dynamic' range (several orders of magnitude), further experiments will be required to fully quantify its effect. The effect of such a large change in a single material property (the viscosity) was to mask the more subtle changes exhibited in response to other system parameters. Thus, the remainder of the present work will focus on a reduced test matrix in which the viscosity and glass type are held fixed.

This leaves a revised test that contains five possible parameters to investigate: heating rate, soaking time, moulding force, cooling rate, and the force applied during the cooling stage. Another commonly used pre-defined matrix (L_9) allowed for four parameters, having three set points each. In order to accommodate the dimensionality of this matrix, the force applied during the cooling stage was held constant at 22 N. As with the viscosity, the decision to fix the force to accommodate the matrix size was taken because the force had the largest potential of masking the remaining parameters' effects. And just as with the viscosity, the force applied during the cooling stage will require additional experiments to quantify its effects. Based on the results on the initial DOE matrix discussed above, the ranges of the soaking time and moulding force set points were adjusted in order to keep a tighter control on these variables. The experiments specified by the L_9 matrix, along with the revised parameter set points are outlined in Table 4. Identical experimental procedures were used for the revised DOE as employed in the original DOE.

Table 4 Experiments defined by the revised DOE, and the corresponding parameter set points

Experiment	*Heating rate (ºC/min)*	*Soaking time (sec)*	*Moulding force (N)*	*Cooling rate stage1 (ºC/min)*
1	50	120	360	10
2	50	240	445	20
3	50	480	535	40
4	75	120	445	40
5	75	240	535	10
6	75	480	360	20
7	100	120	535	20
8	100	240	360	40
9	100	480	445	10

7 Further results

In a manner consistent with Section 4, NBK-7 samples were moulded following the revised parameter set points, as outlined in Table 4, and analysed using (1–5). The resulting P-values and signal-to-noise ratios for the nine experiments are outlined in Table 5. The effective signal-to-noise ratios for each of the parameters set points, and the importance rankings of the parameters are outlined in Table 6.

The results of the revised DOE show that varying the cooling rate has the largest effect on the repeatability of the final thickness of our moulded samples when the viscosity and cooling force are held constant. This can be seen as the cooling rate has the largest variation in SNR of the remaining four parameters. The heating rate had the second largest variation in the SNR, and thus based on the narrower dataset, it ranked as the second most influential parameter. When the moulding forces and soaking times are varied, their effects on the repeatability of the post-moulded thicknesses are minimal in comparison the effects of the heating and cooling rates. With the exception of the soak time parameter, the general trend for maximum repeatability tends toward lengthening the total time of the moulding cycle: slower heating rates, lower moulding forces (longer actual moulding times), and finally slower cooling rates. By combining the results from the original DOE, it can be determined that, for our sample sizes, that once the glass has had enough time to thermally equilibrate, additional soaking time does not necessarily correspond to higher repeatability in the post-moulded thicknesses.

Table 5 P_i values and SNR values for each experiment defined by the revised DOE

Experiment	P_1	P_2	P_3	P_4	P_5	*SNR*
1	0.999	1.008	0.998	0.999	0.996	47.130
2	1.007	1.011	.007	0.996	0.986	39.959
3	0.989	0.996	1.022	1.015	.0977	34.624
4	1.021	0.974	0.989	1.025	0.991	33.133
5	1.002	1.004	0.989	1.022	0.986	36.646
6	0.988	0.988	1.019	1.012	0.994	36.823
7	0.990	0.986	0.999	1.003	1.021	37.306
8	1.009	1.001	1.016	0.991	0.983	37.544
9	1.012	1.004	1.009	0.980	0.995	37.878

Table 6 SNR of the set points for each parameter for the revised DOE, and the importance ranking of the parameters

Parameter set point	*Heating rate*	*Soak time*	*Mould force*	*Cooling rate*
1	40.571	39.190	40.499	40.551
2	35.534	38.050	36.990	38.029
3	37.576	36.442	36.192	35.100
Difference	5.037	2.748	4.307	5.451
Importance rank	2	4	3	1

8 Conclusions

A DOE approach was used to determine the effects of process parameters during the PGM process on the repeatability of the final thickness of a moulded optic. While certain aspects of the initial DOE failed, key observations were made:

- within a glass family (i.e. oxide glasses) the effect of specific glass type is negligible
- the range of the viscosity (3 orders of magnitude) was far too large, and should be held constant to be able to determine the effects of the other parameters
- the force applied during the first cooling stage should be held constant at a minimum value to avoid further compression as the glass cools through its T_g.

Using this information a smaller, revised, DOE was created to analyse the effects of the remaining process parameters during a PGM cycle on Schott's NBK7. The revised DOE shows that the cooling rate has the largest impact on the repeatability of the final thickness of our moulded parts. The general trend to maximise repeatability is to lengthen the moulding process by using slower heating and cooling rates, longer soaking times (up to a point where the glass has equilibrated), and lower moulding forces.

Additionally, using the 'ideal parameters' outlined by the DOE analysis we were able to achieve a repeatability of ±0.05 mm on the final thickness of the moulded pieces. Further studies will investigate different glass families, such as chalcogenides, to illustrate how the differences in the inherent material properties can change how the parameters affect the final moulded sample.

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