

# INJECTION MOULDING OF MICROFEATURED PARTS

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**Abstract** – The injection molding of a microfeatured component, a diffractive optical element, was studied. The component has a surface grating with amplitude 0.5 micrometers and wavelength 3 micrometer. This grating is successfully replicated using four different polymers; one polymethylmethacrylate (PMMA), one ethylene-norbornene copolymer (cyclic olefin copolymer, COC) and two polycarbonates (PC). The replication quality is determined by atomic force microscopy (AFM) and white light interferometry (WLI). For all four polymers it is possible to achieve almost perfect replication at optimal settings. The replication quality is observed to increase with increasing injection velocity and mould temperature for all polymers. Increasing the holding pressure also has a positive effect on replication, and the planarity of the produced plastic parts is observed to improve with increasing holding pressure.

## Introduction

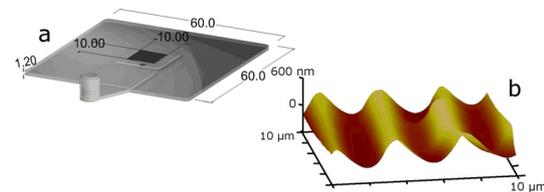
Polymer materials have been seen as ideal candidates for replicating microfeatures since at least the middle of the 20th century. In his talk at Caltech in 1959, Richard Feynman suggested how the entire Encyclopædia Britannica could be printed on the tip of a needle, letter by letter, for then to be replicated in a polymeric material [1]. A pure downscaling of the font would require letters written with a linewidth of 8 nm. It has been shown that fibrillar structures with details down to 10 nm [2] can be replicated using injection moulding, as well as characteristic wave patterns from deep reactive ion etching (DRIE) with features down to 5 nm [3]. At least in some special cases, replicating this famous encyclopaedia at the tip of a plastic needle could be possible using today's industrial polymer processing techniques.

This study is based on moulding trials with a diffractive optical element (DOE). The DOE design was described by Løvhaugen et al. [4]. It is an essential part of a low-cost infrared spectrometer, which e.g. can be used to identify different types of polymers. The functionality of the component comes from a diffractive grating on the surface of the part. This grating has a wavelength of 3  $\mu\text{m}$ , amplitude 500 nm and is sputtered with a 30 nm thin gold layer after moulding to obtain the required reflectance. The injection moulded part is shown in Figure 1.

The most common microfeatured polymer components on the consumer market are optical storage media such as the CD, the DVD and the Blu-ray Disc. A music CD bought in a record shop is injection moulded with music in the shape of small pits in polycarbonate which are read out using a LASER. These pits are roughly 0.5  $\mu\text{m}$  in diameter and 125 nm deep, similar in size to the microfeatures in this work.

A change in the topography of a surface on this size scale can also completely alter the physical properties. By introducing a microscale pattern, the effective surface area of a substrate can be increased

enormously. This again, inspired by the structure of the Lotus leaf, can be used to produce superhydrophobic surfaces [5].



**Figure 1** a) Drawing of the injection moulded part with dimensions [mm]. The microfeatures are located in the central square area. A pressure/ temperature sensor located in the cavity opposite to the microfeatures is indicated with a dark grey circle. b) The topography of the microfeatures measured on an injection moulded DOE using AFM.

Several authors have reported that increasing the mould temperature and the injection velocity improves the replication of microfeatures [6-14]. However, only limited quantitative data exists on how the mould temperature and the injection velocity influence replication. On the other hand, too high mould temperatures can damage microfeatures [15], high mould temperatures will increase the cycle time [16], high injection velocity may lead to poor surface quality [17], and the injection velocity is limited by the injection moulding machine. This makes it important to obtain quantitative data, linking process parameters with replication quality, in order to optimize the injection moulding of parts with microfeatures.

It has been demonstrated that higher melt temperature [8, 17, 18] and higher holding pressure [8, 18] can have a positive effect on replication. There is also a coupling between process and geometry including microfeatures. The effect of holding pressure increases with increasing cavity thickness [8]. Grooves parallel to the flow direction are easier to replicate than those perpendicular to the flow [8, 18, 19]. Features near the gate may [8, 20] or may not [17] be easier to replicate,

e.g. depending on the aspect ratio of the microfeatures. Flow instabilities that reduce the replication quality of certain microfeatures occur at certain processing conditions [7, 21].

To improve the replication of injection-moulded microfeatures, several groups have reported on variotherm processes. In these processes the cavity surface is heated, prior to injection, and then cooled in order for the polymer to reach the ejection temperature. For amorphous polymers the cavity surface is typically heated to temperatures above the glass transition temperature. The cavity surface can be heated from the interior of the mould (by circulating liquids or electrical heating) or from the surface. A smaller mass is heated with the latter techniques, giving shorter cycle times. Implementations of surface heating include induction heating [22], proximity effect heating [23] and infrared heating. Positive effects on product quality have also been reported with cavity evacuation [8, 10, 19] and injection-compression moulding [24]. In the present study, conventional injection moulding equipment was used.

## Experimental

### Part geometry and mould insert

The macro and microstructure of the injection moulded DOE is shown in Figure 1. The area with microfeatures is 10 mm x 10 mm and the mould master, a nickel shim is clamped in a modular base mould. The mould insert was a 0.3 mm thick Ni shim made by electroplating a resist structured by electron beam lithography [4]. The surface topography of the shim was an irregular wave pattern with wave height ~500 nm and wavelength 3  $\mu$ m.

**Table 1** Materials and processing parameters.

Grade	Polymer type	$T_g^a$ [°C]	Mould temp. <sup>b</sup> [°C]	Injection velocity <sup>c</sup> [mm/s]	Melt temp. <sup>d</sup> [°C]	Holding pressure [MPa]
Plexiglas POQ62	PMMA	99.6	40-80	130-1200	240	60
Lexan OQ 1026	PC	138.1	65-120	130-1300	340	10-100
Lexan LS2	PC	138.0	110-120	1300	300	80-120
Topas 5013S-04	COC	130.0	60-125	130-1300	270	50

a) Glass transition temperature measured using DSC at heating rate 10 °C/min. Sample first heated to above  $T_g$ , cooled down and then heated again.

b) Recommended mould temperatures: 60-90 °C (POQ62), 75-95 °C (OQ 1026), 80-100 °C (LS2) and 95-125 °C (5013S-04).

c) This is the melt front speed through the cross-section with microfeatures, i.e. volume flow divided by cross sectional area.

d) The temperature set for the last cylinder element and the nozzle, confirmed by measurements in the barrel end cap (using a Kistler 4083A sensor)

### Polymers

Four amorphous materials were used in the injection moulding trials. The materials were a polymethylmethacrylate (PMMA) (Plexiglas POQ62 from Evonik Röhm), an ethylene-norbornene copolymer, often referred to as a cyclic olefin copolymer (COC) (Topas 5013S-04 from Topas Advanced Polymers) and two polycarbonates (Lexan OQ 1026 and Lexan LS2 both from SABIC Innovative Plastics). The three first have a good ability to replicate microfeatures and flow easily according to the manufacturers. The LS2 is an optical grade polycarbonate with medium viscosity. The glass transition temperatures and processing settings are given in Table 1.

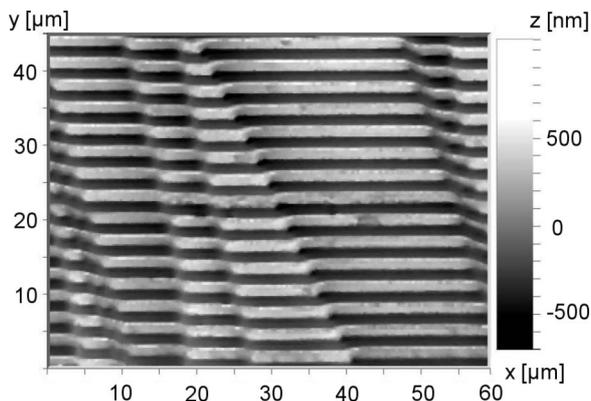
### Injection moulding

The injection moulding was performed on a servoelectric Battenfeld EM 50/120 machine with maximum clamping force 500 kN and screw diameter 25 mm. The injection velocity, the mould temperature and the holding pressure were varied. An overview of the parameters used is shown in Table 1. Note that in this paper *injection velocity* is defined as the *flow front velocity* given as the volume flow divided by the cross sectional area where the microfeatures are located. It has been shown that at high injection rates, the relationship between volume flow [cm<sup>3</sup>/s] and flow front velocity [mm/s] is not completely linear [10], probably due to compression effects. This possible effect is neglected when specifying the flow front velocities in this paper.

The mould temperature was controlled using circulating water at a constant temperature, and the mould temperatures reported below refer to the set water temperature. A combined temperature and pressure sensor (Kistler 6190BA) located in the mould surface opposite to the microfeatures confirmed that this was also close ( $\pm 2$  °C) to the surface temperature when the melt was injected. Some temperatures that were significantly outside the recommended settings were tested, in order to check how this influenced the replication of the microfeatures. When changing the water temperature, the mould temperature was allowed to stabilize so that the maximum and minimum temperature of the mould within a cycle did not change from cycle to cycle.

#### Characterisation using white light interferometer

WLI was used to record topographical images, using a WYKO NT-2000 from Veeco Instruments. This was used to determine the replication quality and the curvature of the moulded parts. To determine the degree of replication, topographical images were taken from the same location near the middle of the microfeathered area seen in Figure 1 for the nickel shim and the moulded DOEs. An example of such an image can be seen in Figure 2. Instead of using just a single peak to determine the replication we used a statistical method called the Power Spectral Density (PSD) which takes out information in the frequency domain. This method has been shown to correlate well with AFM measurements of single peaks, and the method is described in more detail elsewhere [25]. The method gives a scalar variable describing the replication and is a lot faster than AFM measurements.



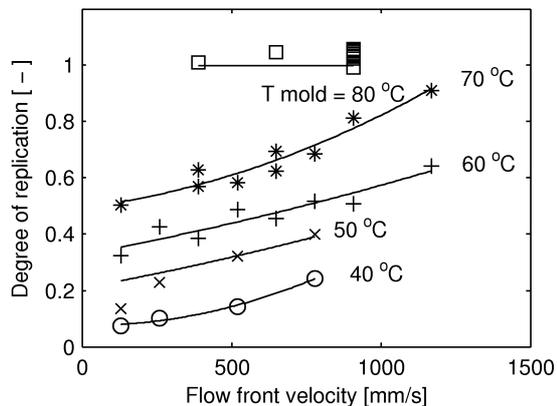
**Figure 2** Topography by WLI for an injection-moulded PMMA DOE.

## Results

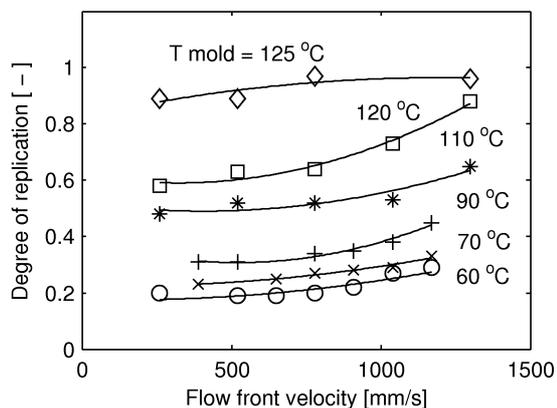
#### Degree of replication vs. processing conditions

The degree of replication found with the PMMA as a function of processing settings is seen Figure 3. It can be seen that the replication clearly improves with increasing injection velocity and mould temperature. At 80 °C, the replication is complete at all velocity

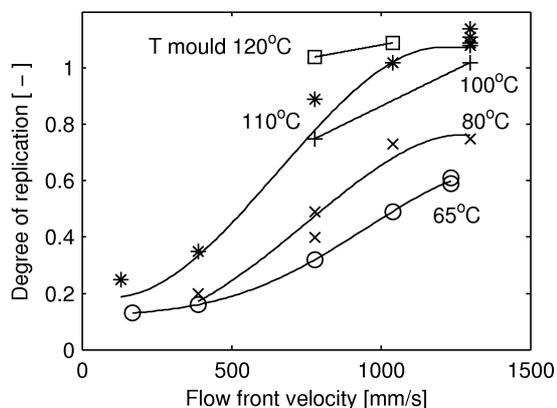
settings. It is also seen that if the temperature is lowered by only 10 °C, the replication quality decreases significantly.



**Figure 3** The degree of replication as measured using WLI/PSD for PMMA DOEs as function of mould temperature and flow front velocity. The lines are guides for the eyes.



**Figure 4** The degree of replication for COC DOEs as a function of mould temperature and flow front velocity. The lines are guides for the eyes.



**Figure 5** The degree of replication for the PC (OQ 1026) DOEs as a function of mould temperature and flow front velocity. The lines are guides for the eyes.

A high degree of replication was also observed for the COC as can be seen in Figure 4. However, the mould temperature had to be set to the maximum value within the recommended range from the manufacturer.

The degree of replication for the PC (OQ 1026) is shown in Figure 5. For this polymer it is possible to achieve complete replication at 110 °C and above. We did also observe a positive effect of increasing the holding pressure.

For the other PC (LS2), it was interesting to see if also medium viscosity polymers could replicate the microfeatures. We did not perform experiments using as many processing settings as for the other polymers, but we observed an increase in the degree of replication with both mould temperature and injection velocity. Using a mould temperature of 120 °C, an injection velocity of 1300 mm/s and a holding pressure of 100 MPa the degree of replication as measured with WLI was 1.0.

When comparing the different polymers regarding the effect of mould temperature relative to  $T_g$  we observed that at high mould temperatures (20 K below  $T_g$ ) the best results are obtained for the PMMA and the PC (OQ 1026). It can also be observed that the PMMA is more sensitive to a reduction in mould temperature than the other two.

#### Curvature

The curvature was measured using WLI for three different holding pressures for each of the PC grades (constant mould temperature and injection velocity). Figure 6 shows that the curvature can be reduced by increasing the holding pressure. The low-viscosity grade OQ 1026 gives the highest curvature and increasing the holding pressure further above ~40 MPa does not lead to further improvement.

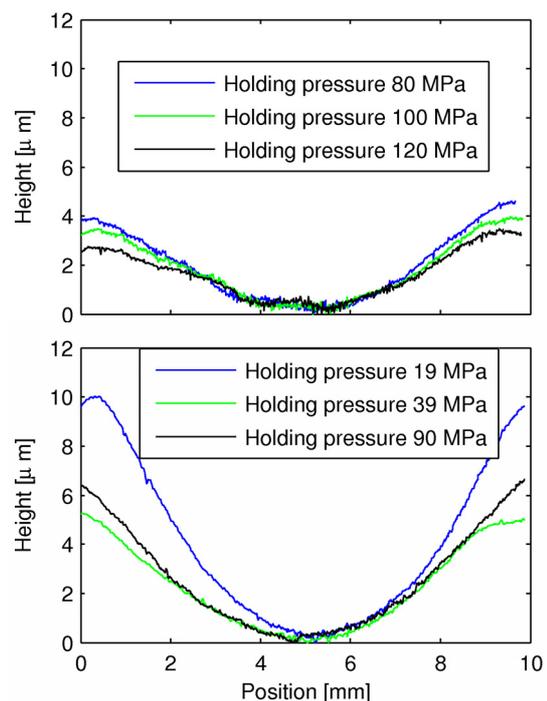
#### Discussion

##### Effect of changing the mould temperature

Increasing the mould temperature will keep the polymer molten for a longer time. As we show by numerical simulations [26], it only takes the polymer 2-3 ms to cool down from the injection temperature to below an effective no-flow temperature. Exactly how fast this temperature drop is, will depend strongly on the heat transfer coefficient between mould wall and polymer as can be seen e.g. in the simulations described in ref [12]. Still there is no consensus regarding which value to use, and experimental evidence suggests that the value changes during the injection moulding cycle [27].

The thermal properties; density, heat capacity and thermal conductivity, for the four polymers are similar. The main difference between the polymers is how the rheological properties depend on temperature and shear

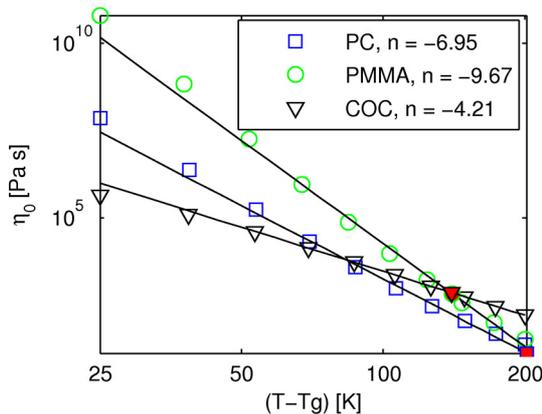
rate. In Figure 7 the zero shear rate viscosities of the COC, the PMMA and the PC (OQ 1026) are plotted as a function of temperature according to the Cross WLF coefficients from the Moldflow database [28]. The viscosity of the PMMA shows much stronger temperature dependence than the other two. In the injection moulding experiments the PMMA and the COC are injected at  $T_g + 140^\circ\text{C}$ . At this temperature the two polymers have almost the same zero shear rate viscosity. The PC, however, is injected at a higher temperature relative to its  $T_g$ , at which the viscosity value is only 1/10th of the values for the other two polymers at their respective melt temperatures at injection.



**Figure 6** The curvature over the microfeatured area for a DOE moulded LS2 (above) and OQ 1026 (below).

##### Effect of changing the injection velocity

The injection velocity may influence the replication via several different effects. a) Faster injection means less cooling of the polymer before reaching the mould wall. b) Faster injection will give more shear heating. c) Faster injection will cause a viscosity reduction due to shear thinning. d) Faster injection will cause a higher pressure and faster pressure-build up at the wall, which will enhance the flow into the microfeatures. e) The magnitude of the injection velocity may affect the heat transfer to the mould (the heat transfer coefficient). f) When switching from velocity control to pressure control, the effective switch-over volume, and associated pressure, may increase with increasing injection velocity due to the retardation time of the injection unit. Some of these effects will be discussed below.



**Figure 7** Log-log plot of zero shear rate viscosity as function of temperature. The melt temperature at injection is indicated by red symbols. The slope  $n$  is defined from  $\log \eta_0 = n \log(T - T_g) + \log b$ .

The pressure effect will be considered first. Faster injection will cause a higher pressure and faster pressure-build up at the wall. For a Newtonian fluid injected between two parallel plates the pressure field behind the flow front has been solved analytically [29]. The pressure at the wall at a given distance behind the flow front is proportional to the injection velocity. The pressure also depends linearly on the distance from the flow front, except in the fountain flow region at the flow front. The net effect of this is that the pressure as a function of time is proportional to the square of the injection velocity for a Newtonian fluid. However, a polymer melt is highly shear thinning and thus not Newtonian. Shear thinning and shear heating will cause the polymer to flow more easily when injected at a high velocity and thus counteract the effect the velocity has on the pressure building up at the wall.

The filling of the part in Figure 1 without microfeatures was simulated with Moldflow [28] in the Hele-Shaw framework (planar flow). The predicted pressure correlated well with the measured pressure at the sensor. The simulations, presented in detail elsewhere [25], indicate that the flow in the main geometry is well described using a generalized Newtonian model for the polymer melt.

A high injection velocity will cause shear thinning and shear heating. These effects will enhance the flow into the microfeatures. The maximum temperature during the injection moulding cycle, as measured with the thermocouple flush with the mould wall, was around 3 K higher when injecting the melt at 1300 mm/s than when injecting at 200 mm/s. The details of the rheology on this size scale are not completely understood and it is suggested that the flow into microfeatures can be highly viscoelastic [7]. As discussed in our simulation paper [26], the shear heating in the microfeature and the main part is not considered important in this case.

The injection velocity may also influence the heat transfer coefficient. Xu et al. [12] claimed good agreement between simulated and experimental replication of microfeatures when using a heat transfer coefficient varying via the local Nusselt number.

#### *Empirical relationship between process parameters and degree of replication*

As shown in Figure 8, a very good correlation exists between the degree of replication for the COC and the PMMA and the following expression:

$$\frac{v_{inj}^2}{\left(\frac{T_g - T_{mould}}{\Delta T}\right)^n} \quad (1)$$

Here  $v_{inj}$  [m/s] is the velocity of the flow front during injection,  $T_g$  [K] is the glass transition temperature of the polymer,  $T_{mould}$  [K] is the mould temperature,  $n$  [-] is the absolute value of the slope in Figure 7 and  $\Delta T$  is a temperature scale which was chosen to be 60 K to make the data for the two materials overlap. The expression is chosen as a ratio between the driving force that forces the polymer into the microfeatures and the resistance to flow.

The driving force is the pressure building up in the cavity after the flow front has passed the microfeatures. It will depend on the viscosity of the melt, the geometry of the cavity and the injection velocity. In this case the geometry is kept fixed, and as shown in Figure 7 the zero shear rate viscosities of the two polymers are similar at the injection temperature, meaning that even though these effects will clearly influence the replication, they will enter the expression above as a constant term. It was also observed in both experiments and the Moldflow simulations that the mould temperature only had a marginal influence on the cavity pressure. The only parameter that is varied and which influences the cavity pressure is the flow front velocity. It will influence the viscosity through the shear rate, and it will (assuming a constant viscosity) cause the pressure to increase proportional to  $v_{inj}$  squared.

The resistance to flow into the microfeatures depends on the geometry of the microfeatures and the viscosity of the polymer in the microfeatures. Once again the geometry is kept fixed and will enter as a constant term. For simplicity we have chosen to assume that the shear thinning effect in the microfeatures (easier flow), will cancel out the effect of the shear thinning in the main geometry (lower pressure).

The characteristic temperature in the microfeatures will decrease rapidly from the melt temperature down to the mould temperature. The temperature decrease will depend on the heat transfer coefficient, the thermal diffusivity and the contact temperature (thermal

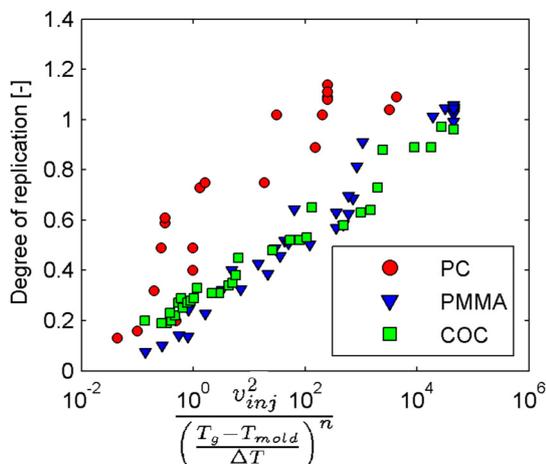
effusivity). If we neglect the differences between the polymers regarding thermal diffusivity and effusivity, and assume that the heat transfer coefficients will be similar for the polymers, we can express the characteristic temperature in the microfeatures as the difference between the mould temperature and the glass transition temperature to the power of  $n$ , similar to how the zero shear rate viscosity varies with temperature as

$$\eta_o = b(T - T_g)^n \quad (2)$$

as seen in Figure 7.

Even though a simple scalar expression can not concentrate all information from the physically complex injection moulding process, the expression given in Equation (1) can provide useful information when predicting and optimizing the injection moulding process.

For the PC grade OQ 1026, the fit is not as good as the two other polymers and it generally has better replication than the two others for the same value of the expression in equation 1. However the slope is almost the same. We believe the reason why it shows better replication is that it has a much lower viscosity at the melt temperature.



**Figure 8** The degree of replication for the PMMA, the COC and the PC (OQ 1026) at different moulding conditions plotted as function of the empirical expression from Equation 1. See main text for details.

## Conclusion

We have replicated a diffractive optical element by injection moulding using four different amorphous polymers. It is observed that all the polymers can give an almost perfect replication of the diffractive grating, even the polymer which does not have especially low viscosity.

The curvature of the part is critical for the functionality of the DOE and it is observed that this can be reduced to less than 3  $\mu\text{m}$  over 10 mm by increasing the holding pressure.

An empirical relationship is presented which describes how the replication quality increases with increasing injection velocity and increasing mould temperature.

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