

THE INFLUENCE OF PROCESSING CONDITIONS ON THE DIMENSIONAL STABILITY OF PIGMENTED MOULDINGS

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EXECUTIVE SUMMARY

Certain organic pigments such as the phthalocyanines have a reputation within the moulding industry for causing shrinkage and warpage problems in components manufactured from polyolefins. The popularity of these particular pigments stems from their stability at high temperatures and resistance to light, acids and alkalis. This study assesses whether changes in the processing conditions used to injection mould a simple plate moulding are sufficient to control the dimensional instability of differently coloured feedstocks. Holding pressure, holding time, melt and mould temperatures and injection time are all considered as variables. The results obtained using phthalocyanine blue added to high density polyethylene either as a masterbatch or by direct blending are compared with those obtained using virgin material and feedstocks coloured with irgalite yellow.

The conclusions of this investigation are as follows:

- Phthalocyanine blue either added directly to HDPE or introduced as a masterbatch causes less shrinkage perpendicular to the flow direction than either virgin or yellow but more shrinkage parallel to it. This alters the anisotropic shrinkage ratio from approximately 1.2 to 0.55.
- High holding pressure improves dimensional stability.
- Longer holding times limit the shrinkage perpendicular to flow and are associated with heavier plates.
- Changing the processing conditions will not significantly affect the ratio of shrinkage measured parallel and perpendicular to the direction of flow.
- Out of plane distortion is reduced by increasing the speed at which the cavity fills - faster flow rates during filling (i.e. shorter injection times), increasing mould or melt temperature and by raising the hold pressure.
- Longer holding times do not appear to affect out-of-plane distortion.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,
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1 INTRODUCTION

Certain organic pigments such as the phthalocyanines have a reputation within the moulding industry for causing shrinkage and warpage problems in components manufactured from polyolefins¹⁻⁴. This behaviour may or may not be acceptable depending on the function or application of the moulding. In general, dimensionally unstable mouldings cannot be used in applications that require tight geometrical tolerances^{5,6}, typical examples include the manufacture of snap-fit caps, precision dispensers and crates.

Recent studies have shown that phthalocyanine blue has a pronounced effect on the morphology of both polyethylene and polypropylene^{7,8}. There is evidence to suggest that phthalocyanine blue behaves as a nucleating agent for both polypropylene, seeding the α -crystalline form⁸, and high density polyethylene⁹. As a consequence, melts containing pigment crystallize at higher temperatures than their virgin counterparts. The effect is more pronounced for polypropylene than for high density polyethylene (HDPE)⁹. The increase in crystallization temperature has the potential to increase the dimensional instability of mouldings produced from melts containing phthalocyanine blue by generating greater levels of internal stress. Microscopical studies of pigmented samples show that the spherulites formed during melt crystallization are substantially smaller than those found in virgin material⁷. Morphological differences of this type have been shown to affect the mechanical performance of mouldings^{1,10,11}.

The purpose of this investigation is to assess whether or not changing the processing conditions can be used to limit the influence of these 'active' pigments on the dimensional stability of mouldings. In particular this study focuses on the influence that processing conditions have on the shrinkage and warpage behaviour of mouldings manufactured from HDPE.

2 A DESIGN STRATEGY FOR MOULDING TRIALS

We have already established a correlation between certain pigments and dimensional instability for the grade of high density polyethylene used in this study¹². In our previous study we compared the shrinkage and warpage behaviour of square plates injection moulded from four differently coloured feedstocks with those manufactured from virgin material. The coloured feedstocks were obtained by adding small quantities of masterbatch. These masterbatches contained a number of components including a wetting agent (zinc stearate) and a carrier polymer (linear low density polyethylene (LLDPE)). Although LLDPE is fully compatible with high density polyethylene (HDPE) in both molten and crystalline states¹³ it is possible that some of the other masterbatch components could affect the dispersion of the pigment particles. To test the thesis that some components of the masterbatch could affect the shrinkage and/or warpage behaviour of mouldings in this study we prepared coloured feedstocks *without* using a masterbatch. These were manufactured by tumble mixing pellets of HDPE directly with pigment powder. This mixture was then extruded to form a coloured feedstock.

For this investigation the following pigments were chosen: irgalite yellow (yellow 62), phthalocyanine blue - masterbatch, phthalocyanine blue - direct mix. Virgin HDPE was used as a control.

Previously published studies have shown that changes in holding pressure, holding time, melt and mould temperatures or injection speed have the most influence on the dimensional stability of injection moulded items¹⁴⁻²⁰. These examinations have in general been limited to measurements of shrinkage and none of them considered the influence of processing on the dimensional stability of mouldings manufactured from pigmented feedstocks. However, the results of these earlier investigations have helped us to identify the most important variables that affect shrinkage and warpage. In an earlier study²¹ we assessed the influence that processing conditions have on the dimensional instability of plates moulded from HDPE by using the injection moulding *simulation* software package, Moldflow²². The variables of melt and mould temperature, holding pressure, holding time, cooling time and injection time were considered. Different values were chosen for each of these processing variables, i.e. 3 different melt temperatures, 4 different injection times. A 'design of experiments' (DOE) screening experiment was used^{23,24} (fractional factorial array) to reduce the full factorial array of 288 potential combinations to a more manageable fractional factorial array with 22 cases. The results of this investigation showed that cooling time was not a statistically significant variable and thus it has been ignored in the present study.

In summary, in this paper we consider the influence that colour, injection time, holding pressure, holding time and melt and mould temperatures have on dimensional stability. In particular we have chosen to explore the influence of;

- 4 colours
- 3 injection times
- 2 holding pressures
- 3 melt temperatures
- 2 holding times
- 2 mould temperatures

If we were to consider the influence of all the different combinations of these factors then we would need to manufacture 288 plates each requiring different moulding conditions. Assuming that 5 replicas will be required for each run then we would need to measure 1440 plates. This is clearly impractical. Using the DOE approach this number can be reduced to a more acceptable 41 runs as shown in Table 1 or 205 plates, accounting for 5 replicas per run. This fractional factorial array was generated using the DOE package²⁵ *Doewin2*. The design matrix considers different combinations of factors e.g. colour with holding pressure, melt temperature with mould temperature. Amalgamating factors into interaction group terms can reveal dependencies that may be invisible if the investigation focuses only on identifying causal relationships between dimensional instability and single factors.

The matrix shown in Table 1 has the following characteristics (provided by the DOEWIN2 package) ; average variance coefficient (AVC) of the estimated parameters = 0.34, D-efficiency* = 0.40 and a minimum detectable effect of 1.08.

Colour	Injection time (s)	Holding Press. (bar)	Melt temp. (°C)	Holding time (s)	Mould temp. (± 1.5 °C)
Virgin (0000)	4.4	20	188.5 $\pm$ 3.5	20	23
Virgin (0001)	4.4	20	203.0 $\pm$ 3.0	20	23
Virgin (0002)	4.4	20	214.5 $\pm$ 1.5	20	23
Virgin (0010)	4.4	40	188.5 $\pm$ 3.5	20	23
Virgin (0012)	4.4	40	214.5 $\pm$ 1.5	20	23
Virgin (0100)	3.1	20	188.5 $\pm$ 3.5	20	23
Virgin (0102)	2.9	20	214.5 $\pm$ 1.5	20	23
Virgin (0111)	2.9	40	203.0 $\pm$ 3.0	20	23
Virgin (0200)	1.4	20	190.0 $\pm$ 2.0	20	40
Virgin (0211)	1.5	40	191.5 $\pm$ 3.5	20	40
Blue (1000)	4.4	20	188.5 $\pm$ 3.5	40	23
Blue (1001)	4.4	20	203.0 $\pm$ 3.0	40	23
Blue (1012)	4.3	40	210.0 $\pm$ 3.5	40	23
Blue (1100)	3.1	20	188.5 $\pm$ 3.5	40	23
Blue (1101)	3.1	20	203.0 $\pm$ 3.0	40	23
Blue (1110)	3.1	40	188.5 $\pm$ 3.5	40	23
Blue (1112)	2.9	40	210.0 $\pm$ 3.5	40	23
Blue (1200)	3.0	20	190.0 $\pm$ 2.	40	40
Blue (1201)	1.5	20	191.5 $\pm$ 3.5	40	40
Blue (1202)	1.5	20	210.0 $\pm$ 3.5	20	40
Blue (1211)	1.5	40	191.5 $\pm$ 3.5	40	40
Blue (1212)	1.5	40	210.0 $\pm$ 3.5	40	40
Yellow (2000)	4.4	20	190.0 $\pm$ 2.0	40	40
Yellow (2002)	4.4	20	214.5 $\pm$ 1.5	40	40
Yellow (2011)	4.4	40	191.5 $\pm$ 3.5	40	40
Yellow (2012)	4.4	40	214.5 $\pm$ 1.5	40	40
Yellow (2101)	2.9	20	191.5 $\pm$ 3.5	40	40
Yellow (2102)	2.9	20	214.5 $\pm$ 1.5	40	40
Yellow (2111)	2.9	40	191.5 $\pm$ 3.5	40	40
Yellow (2112)	2.9	40	214.5 $\pm$ 1.5	40	40
Yellow (2201)	1.5	20	203.0 $\pm$ 3.0	40	23
Yellow (2210)	1.5	40	190.5 $\pm$ 2.5	40	23
Yellow (2212)	1.5	40	214.5 $\pm$ 1.5	40	23
MB blue (3001)	4.4	20	191.5 $\pm$ 3.5	20	40
MB blue (3002)	4.4	20	209.5 $\pm$ 3.5	20	40
MB blue (3010)	2.9	40	190.0 $\pm$ 2.0	20	40
MB blue (3011)	4.4	40	191.5 $\pm$ 3.5	20	40
MB blue (3102)	2.9	20	209.5 $\pm$ 3.5	20	40
MB blue (3110)	1.5	40	190.0 $\pm$ 2.0	20	40
MB blue (3202)	1.5	20	210.0 $\pm$ 3.5	20	23
MB blue (3211)	1.5	40	203.0 $\pm$ 3.0	20	23

Table 1: The design matrix used for the moulding trials. An explanation of the colours and reasons for the choice of parameters can be found in section 3.3. The codes in brackets are used to identify mouldings in each batch. The holding pressures and times are machine set values, all other quantities are measured.

Limiting the number of runs does limit the number of interactive terms that can be considered, although some are taken into account to constrain the D-efficiency and AVC values to within acceptable limits.

D-efficiency (determinant efficiency) is a scaled value of the determinant of the information matrix. It is used as a measure of D-optimality. A D-efficiency fraction of score is best. D-efficiency compares the design to a theoretical (but not always possible) orthogonally balanced design.

Orthogonality is a characteristic of an experimental design that ensures independence of estimates. If the underlying linear model is correct, an orthogonal design is associated with balanced data. More generally, orthogonality and balance are considered as separate design parameters. The degree of orthogonality is measured by the D-efficiency score. A balanced design is such that each column of a design array has the same number of values of that parameter. The data is balanced when it is distributed over the experimental region so that each point makes an equal contribution to the parameter estimates. $D\text{-efficiency} = \det(IM) / k$ i.e the determinant of the Information Matrix normalised by the number of rows.

3 EXPERIMENTAL

3.1 MOULDING GEOMETRY

The simple edge-gated square plate moulding shown in Figure 1 was chosen for this study. The unusual domed region between the sprue and the gate ensures that the cavity fills uniformly during mould filling by equalizing the flow paths of the melt through the spreader. This design was developed by the NPL to enhance the anisotropic properties of the moulding as measured by differences in Young's modulus²⁶. The performance of the cavity and its cooling circuit have been described elsewhere²⁶.

The mould cavity and cooling circuit are contained within a block of P20 steel of dimensions 296 mm x 496 mm x 168 mm. The dimensions of the square plate cavity are 150 mm x 150 mm x 4 mm.

3.2 MATERIALS AND SAMPLE PREPARATION

Pelletised masterbatches containing the components listed in Table 2 (supplied by Addcolour Plastics Ltd) were tumble mixed with pellets of HDPE (DSM Stamylan). These feedstocks were then used to injection mould 4 mm thick square plates. The concentration of pigment in the *direct* blend of phthalocyanine blue and pellet was 0.2 %.

Masterbatch composition					
Pigment	% Pigment	% Zinc Stearate	% Polyethylene wax	% LLDPE	% Pigment in feedstock
Irgalite Yellow (yellow 62)	6.66	1%	3	89.34	0.13
Phth. Blue	6.66	1%	3	89.34	0.13

Table 2: The composition of the masterbatch from which square plates of HDPE were moulded. The concentration of pigment in the feedstock used to manufacture mouldings is also given.

3.3 PROCESSING CONDITIONS

Values for each of the processing variables shown in Table 1 need to be chosen with care as some combinations are impractical i.e. those that result in short shots. The challenge here is to find processing windows for all the combinations of factors. Initial estimates of the melt temperature window were obtained from the manufacturer's guidelines for processing Stamylan. Estimates for the range of mould temperature, holding pressure, holding time and injection time were based on generic data for HDPE as supplied by Moldflow²². These values were subsequently modified during a set of initial runs to establish a range of variables that, when combined, would produce acceptable mouldings. These are listed in Table 3.

Processing variable	Target values
Injection time (s)	1.5, 3, 4.5
Holding pressure (bar)	20, 40
Holding time (s)	20, 40
Melt temperature (°C)	190, 200, 215
Mould temperature (°C)	23, 40

Table 3: Target values for the processing variables used

Unfortunately the practicalities of the injection moulding process mean that it is very difficult to ensure that these target values are actually met in the cavity, for example, shear heating of the melt as it flows through the gate becomes increasingly significant with decreasing injection time. This will raise the melt temperature beyond that measured at the nozzle tip. Lowering the holding time, reduces the cycle time and causes the cavity wall temperature to rise as the cooling circuit attempts to dissipate a higher heat flux. Interactions of this type i.e. between different different factors has been accounted for in the choice of fractional array (Table 1).

A pressure transducer mounted behind one of the ejection pins was used to monitor cavity pressure. This transducer is centrally located within the cavity at a point near to the gate. Figure 2 shows the pressure histories of two mouldings. The only difference between the processing conditions used to manufacture these mouldings is in the holding pressure, one of which is 40 bar and the other 20 bar. It is apparent that the time over which the holding pressure is applied to the cavity is substantially shorter for the lower holding pressure, despite constant holding time settings being used for the machine. This shortfall is due to the gate freezing.

A further complication arises when short injection times are used. Short injection times are associated with the rapid flow of melt into the cavity. This results in melt compression. Decompression of the melt after the cavity is filled can cause backflow if insufficient holding pressure is applied. Alternatively, if the gate freezes before all the material within the cavity has solidified then the melt decompression can compensate, at least in part, for lack of hold pressure. What happens in practice depends on how long it takes for material within the mould

cavity to freeze and that in turn depends on both the effective melt and mould temperatures.

For each run shown in Table 1 the injection moulding machine was allowed to run through at least six moulding cycles before conditions were deemed to be stable. Virgin HDPE was used to clean the barrel between successive colour changes. This procedure ensured that all traces of the previous colour were purged.

The temperature of the melt was measured by inserting a platinum resistance thermocouple into molten material after it had been extruded from the barrel (the barrel being positioned some distance from the mould). Such measurements were made after several injection cycles had been completed after changing the temperature of the barrel heaters. Machine stability was essentially monitored by the pressure traces provided by the transducer mounted within the cavity as shown in Figure 2. When the processing conditions were stable six plates were 'collected' for each set of conditions listed in Table 1. These plates were removed from the machine immediately after ejection and placed on to the surface of a wooden table where they cooled to room temperature (17 °C - 18 °C). Five plates from each batch of six were used to generate shrinkage and warpage data.

3.4 MEASURING SHRINKAGE AND WARPAGE

Calculations of plate shrinkages and in-plane warpages are based on measurements of the lengths and breadths of each plate in 2-dimensions. Computation of the out-of-plane warpage requires information in the third dimension. This information can be obtained from the measured 3-D coordinates of each plate. The experimental arrangement is shown schematically in Figure 3. These were determined using shadow Moiré interferometry. In this technique the shadow from a grating (with a 2 mm pitch) is projected on to the surface of a plate. Interference between the reflected shadow and the grid itself produces a pattern that can be captured using a camera and stored on computer. The coordinates of each plate are obtained by unwrapping the phase information contained within the pattern of interference fringes. This operation was performed by the software package²⁷ Fringe Application V3.20. A thin layer of highly reflective paint was applied to each plate before measurements were made to compensate for the translucency of the polyethylene. Measurements of the plate coordinates were made at least six weeks after they were moulded. During this time the plates were stored in vertically at room temperature (~ 20 °C).

The phase unwrapping programme produces coordinates in dimensionless optical units that have to be converted into S.I. units to facilitate comparison with other measurements. This requires calibrations in all three dimensions to be made. In the xy plane (Figure 5) this was achieved by measuring the dimensions of a plate at five equally spaced points using a vernier caliper. A specially manufactured gauge was used to calibrate the out-of-plane data in the z direction. This gauge consisted of two planes at different heights connected by a smooth slope. Successful calibration relies on the relationship between the difference in height between the two planes being known in both optical and S.I. units.

Mounting the plates in the sample holder does lead to some variation in its orientation with respect to the camera. This small error was corrected by mounting the z calibration gauge on the same backing board as that used to mount the plates.

3.4.1 Determination of shrinkage

Shrinkage is defined as the ratio between the measured distance between any two points on opposite edges of a moulding and that measured at the same point inside the mould cavity. Obviously this procedure cannot be followed in plates that are subject to significant out-of-plane deformation. To overcome this limitation we determine the arc length between points located on opposite edges of the plate. This procedure has been described in detail elsewhere^{12,21} and hence will only be briefly covered here.

A three-dimensional surface is used to fit the coordinates obtained from analysing the optical interference pattern as shown in Figure 4. Using this surface the arc length, a can be calculated between any two points on opposite edges of a plate according to:

$$a_{\parallel}(x_k, y, z) = \int_{\tilde{e}_2}^{\tilde{e}_4} \sqrt{1 + \left(\frac{\partial f(x_k, y, z)}{\partial y}\right)^2} dy \quad (1)$$

$$a_{\perp}(x, y_k, z) = \int_{\tilde{e}_1}^{\tilde{e}_3} \sqrt{1 + \left(\frac{\partial f(x, y_k, z)}{\partial x}\right)^2} dx. \quad (2)$$

$f(x, y, z)$ denotes the polynomial or spline function used to fit the z (out-of-plane) co-ordinates. The subscripts $\parallel$ and $\perp$ refer to directions parallel and perpendicular to that of the flow. This is shown schematically in Figure 5 where the edges e_1 to e_4 are defined. The shrinkage at any location in x or y is then generated by substituting the arc length, for example $a_{\perp}$ for $|\tilde{e}_1(x, y_k, z) - \tilde{e}_3(x, y_k, z)|$, into the following equation:

$$s_{\perp, k} = \frac{|e_1(x, y_k, z) - e_3(x, y_k, z)| - |\tilde{e}_1(x, y_k, z) - \tilde{e}_3(x, y_k, z)|}{|e_1(x, y_k, z) - e_3(x, y_k, z)|} \quad (3)$$

$e_1(x, y_k, z)$ is the k^{th} point on edge 1 of an undeformed plate and $\tilde{e}_1(x, y_k, z)$ is the corresponding point on the equivalent deformed edge. Unfortunately this method of evaluating shrinkage has its limitations. It is not possible to compute the true shrinkage between the physical edges of each plate for every pair of corresponding edge points because of discontinuities that can arise in the path of the integrals in equations 2 and 3 if the outermost edges bow in towards the centre. This is shown schematically for equation 2 in Figure 6. In all cases the same coordinate frame

was used for deformed plates as that used to describe the mould cavity and the flow direction is parallel to 'y'.

3.4.2 Shrinkage scores

The shrinkage across the plate in either direction depends on where it is measured, for example, shrinkages perpendicular to the flow direction tends to increase with increasing distance from the gate. Because of this variation along an axis it is difficult to compare shrinkages measured in different plates unless it is done on a point by point basis. This cumbersome approach can be avoided by defining a shrinkage score. Here the mean and range of shrinkages are calculated in directions both parallel and perpendicular to that of the flow i.e.

$$\text{Mean } S_{\perp} = \text{mean}_k(s_{\perp,k}), R_{\perp} = \text{range}_k(s_{\perp,k}) \quad (4)$$

and

$$\text{Mean } S_{\parallel} = \text{mean}_k(s_{\parallel,k}), R_{\parallel} = \text{range}_k(s_{\parallel,k}), \quad (5)$$

The shrinkage range, R is defined as the difference between the maximum and minimum shrinkage values.

An overall measure of plate shrinkage can be obtained by combining the mean and range of shrinkage following:

$$S_{\parallel} = (\text{mean}S_{\parallel} + R_{\parallel}) / 2 \quad (6)$$

and

$$S_{\perp} = (\text{mean}S_{\perp} + R_{\perp}) / 2 \quad (7)$$

3.4.3 Determination of warpage

Warpage can occur both in-plane and out-of-plane. In-plane warpage occurs when parallelism between two opposite edges is lost despite the distance between them remaining constant. In-plane warpage can occur in directions both parallel to and perpendicular to that of the flow. The in-plane warpage for edges 1 and 2 are given by;

$$W_{\perp}(e_1) = \text{mean}_k \left(\frac{\tilde{e}_1(x, y_k, z)}{|e_1|} \right), R(W_{\perp}(e_1)) = \text{range}_k \left(\frac{\tilde{e}_1(x, y_k, z)}{|e_1|} \right) \quad (8)$$

and

$$W_{\parallel}(e_2) = \text{mean}_k \left(\frac{\tilde{e}_2(x_k, y, z)}{|e_2|} \right), R(W_{\parallel}(e_2)) = \text{range}_k \left(\frac{\tilde{e}_2(x_k, y, z)}{|e_2|} \right) \quad (9)$$

where ‘ $\sim$ ’ denotes the deformed edge and $|e_i|$ the length of the undeformed edge e_i . Functions for edges 3 and 4 are obtained by substituting e_3 for e_1 and e_4 for e_2 respectively.

The range of out-of-plane warpage is given by;

$$R(W_z) = \text{range}_k(z_k)$$

where $z_k = f(x_k, y_k, z_k)$ is the fitted algebraic surface at points x_k and y_k . Out-of-plane warpages, $R(W_{ei})$ for each of the plate edges were defined in a similar manner restricting f to the relevant edge data.

As significant amounts of information are lost by calculating the mean out-of-plane warpage we have chosen only to consider the range of deformation. This method is equivalent to measuring the vertical distance between any two opposite edges of a warped plate.

3.4.4 Warpage scores

The in-plane warpage at each point along each edge can be combined to give single measures of the mean and range of in-plane warpage e.g..

$$W_{\perp} = \left[\left| W_{\perp}(e_1) - W_{\perp}(e_3) \right| \right]$$

and

$$R(W_{\perp}) = \left[R(W_{\perp}(e_1)) + R(W_{\perp}(e_3)) \right] / 2$$

This procedure can be applied to warpage data obtained in a direction parallel to that of the flow.

An overall measure of in-plane warpage can be obtained by combining the mean and range following;

$$\text{Warpage } W_{\perp} = (W_{\perp} + R(W_{\perp})) / 2$$

and

$$\text{Warpage } W_{\parallel} = (W_{\parallel} + R(W_{\parallel})) / 2.$$

3.4.5 Angle scores

Changes in the angle formed at the intersection of the diagonals of the square plate moulding can be used as a measure of distortion. In an undeformed square plate this angle is given by $\theta(L_{\perp}, L_{\parallel})$. Since $L_{\perp} = L_{\parallel}$ then $\theta = \pi/2$ and so $\cos(\theta) = 0$. The mean x and y values for the four edges of the plate are given by $M(W_{\parallel})$ and $M(W_{\perp})$. Taking the dot product of the vectors formed by these vertices and normalizing yields the cosine of the angle θ of the deformed plate, as illustrated in Figure 5. This cosine is then taken as the angle score.

4 DATA INTERPRETATION

We have used two approaches to interpret the shrinkage and warpage data. The first is based on the analysis of means (section 4.1). This qualitative method can be used to rank different pigments or processing conditions according to the effect they have on dimensional instability. The second approach relies on a statistical analysis of the variance within the data (section 4.2). The analysis of variance consists of a decomposition of the variance in a sample into components due to a number of factors, that are assessed for significance with respect to a hypothesis. The more quantitative variance analysis allows us to assess the statistical significance of trends that we observe in the analysis of means.

4. ANALYSIS OF MEANS (ANOM)

The principle of this analysis is to take the average value of, for example, the mean and range of the out-of-plane warpage that are obtained for each value of a processing factor. Taking melt temperature as an example, the shrinkage scores parallel to the flow direction are divided into three groups representing melt temperatures of 190, 200 and 215 °C each containing a number of data sets. These data sets are then analysed to yield a 'mean' for each group of the data sets within that group as shown in Figure 7. From this figure it is apparent that whilst the mean shrinkage score, $S_{||}$ is very sensitive to both colour and holding pressure it is relatively *insensitive* to variations in mould temperature.

This form of ranking of the ranges of factor means (loosely termed the analysis of means approach) provides qualitative information relating processing variables to changes in dimensional stability. Quantitative information relating different processing factors to dimensional stability can be obtained by analysing the data using more comprehensive statistical models.

As an aside plots of this nature can be used to define a set of processing conditions that can be used to control the 'quality' of mouldings. For example, the lowest shrinkage scores, $S_{||}$ can be obtained by manufacturing mouldings using the conditions that correspond to the lowest values of each factor indicated in Figure 7. However this does assume that interactions between the various factors *do not* influence shrinkage.

4.2 ANALYSIS OF VARIANCE (ANOVA)

Each of the different processing variables considered in this study will contribute to the variance within the dimensional instability data. Some of the processing variables will have no more influence on the variance than would be expected through chance variability. In the analysis of variance these influences will be seen as statistically insignificant. The opposite is true for variables that are statistically significant. A factor is considered to be significant, for example, if the hypothesis that the variance due to colour is attributable to chance can be rejected with at least 95% confidence.

5 RESULTS AND DISCUSSION

The shrinkage and warpage scores for each of the batches listed in Table 1 can be found in Annex 1.

5. THE INFLUENCE OF COLOUR ON DIMENSIONAL STABILITY

Table 4 shows a qualitative ranking based on the analysis of means approach for different pigments for each of the measures used to assess dimensional instability.

Shrink $S_{\perp}$	Shrink $S_{\parallel}$	Warp $W_{\perp}$	Warp $W_{\parallel}$	Whole plate, RW_z	Angle score	edge 1 RWe_1	edge 2 RWe_2	edge 3 RWe_3	
DB blue	yellow	DB blue	yellow	yellow	virgin	MB blue	MB blue	MB blue	less distortion ↑
MB blue	virgin	MB blue	virgin	virgin	yellow	yellow	DB blue	yellow	
yellow	MB blue	yellow	MB blue	MB blue	MB blue	virgin	yellow	virgin	↑
virgin	DB blue	virgin	DB blue	DB blue	DB blue	DB blue	virgin	DB blue	more distortion

Table 4 : The influence of pigment type on dimensional stability. The pigments are ranked with those causing the most distortion at the bottom. Cells that contain two pigments have an equal ranking. MB and DB blue refer to the method used to add the pigment i.e. masterbatch and direct mixing respectively.

It is apparent that phthalocyanine blue causes more shrinkage perpendicular to the direction of flow than virgin or yellow. However the opposite is true for shrinkage parallel to the flow direction. This is graphically shown in Figure 8 where the shrinkage measured perpendicular to the flow direction is plotted against that measured parallel to it. Figure 9 shows how the shrinkage anisotropy ratio of $S_{\perp}$ to $S_{\parallel}$ depends on colour. Clearly there is a significant difference in the value of this ratio between the blue plates (irrespective of whether the pigment is added as a masterbatch or mixed directly) and those of both the virgin and yellow.

	$S_{\perp} / S_{\parallel}$	Weight
high ↑ ↑ low	Yellow	MB blue
	Virgin	virgin
	MB blue	DB blue
	DB blue	yellow

Table 5: The influence of pigments on the anisotropic shrinkage ratio (Figure 8) and weight of the mouldings.

Unbalanced anisotropic shrinkage is a driving force for warpage, particularly that which occurs out-of-plane. The fact that the shrinkage ratios for the masterbatch and directly mixed blue are significantly less than those for virgin and yellow could explain why mouldings containing phthalocyanin blue warp. This thesis is supported by the analysis of means data shown in Table 5, although a plot of the out-of-plane warpage as a function of all colours and combinations of processing conditions (Figure 10) indicates that holding pressure is a more influential variable. Clearly there are two distinct populations in this figure corresponding to low and high holding pressures respectively where the latter causes less distortion.

5.2 THE INFLUENCE OF PROCESSING CONDITIONS ON DIMENSIONAL STABILITY

Figure 11 shows how the weight of mouldings measured together with the runner and sprue varies with colour and all combinations of the different processing conditions. These results fall into two distinct groups that reflect the applied holding pressure. High holding pressures are, as expected, associated with heavier mouldings. The quantitative analysis (analysis of variance) of the data shown in Table 6 shows that all the processing variables including colour are statistically significant although Figure 11 shows that holding pressure is the most significant variable.

	Colour	Injection time	Hold pressure	Melt temperature	Holding time	Mould temperature
Shrinkage, $S_{\perp}$	***		***	***		*
Shrinkage, $S_{\parallel}$	***		***	*		
Warpage, $W_{\perp}$	***		***			
Warpage, $W_{\parallel}$	***		***	***		
Whole plate, RW_z	***	***	***	***		***
Angle score	***		***	*		
$S_{\perp} / S_{\parallel}$	***					
Range $S_{\perp} / S_{\parallel}$			***			
Weight	***	***	***	***	***	**
edge 1, RWe_1	**	*		***		
edge 2, RWe_2	*	**	***			
edge 3, RWe_3	***		***	*		
edge 4, RWe_4			***			**

Table 6 : This table shows which factors are statistically significant in the analysis of variance for each of independent variable of interest. The symbols (explained in the key) relate to probabilities. Probabilities of less than 0.1% are highly significant.

Key: blank : $p \geq 5\%$
 * : $1\% \leq p < 5\%$
 ** : $0.1\% \leq p < 1\%$
 *** : $p < 0.1\%$

Plots of shrinkage scores obtained for both parallel and perpendicular to the direction of flow and the angle scores as a function of batch number didn't show any obvious correlations with any of the processing variables. However the statistical analysis (Table 6) shows that there are some significant links between different processing variables and measures of dimensional instability. Colour is statistically significant for all measures of instability with the exception of the range of the shrinkage ratio.

Factor combinations that are statistically significant for at least one measure of dimensional instability are shown in Table 8. The links identified in this table reflect those shown in table 6, with colour and holding pressure having the most influence on shrinkage and warpage. We note (from Tables 6 and 7) that holding times do not appear to have any effect on out-of-plane distortion. This is particularly interesting as moulders extend holding times to anneal out internal stresses that if present in the moulding could cause warpage.

	Colour : Holding pressure	Colour : Melt temp.	Inj. time : hold pressure	Inj. time : melt temp.	Hold Pressure : melt temp.	Hold pressure : mould temp.	Melt temp : mould temp.
Shrinkage, $S_{\perp}$		***			*		
Shrinkage, $S_{\parallel}$	***						
Warpage, $W_{\perp}$	**				***		
Warpage, $W_{\parallel}$	***						
Whole plate,	**	***	***	***			
Angle score	***				*		
$S_{\perp}/S_{\parallel}$	*			*			
Range $S_{\perp} / S_{\parallel}$							**
Weight	***	***	***	***	***		
edge 1, RWe_1	**		***	**	*		
edge 2, RWe_2	***		***			*	
edge 3, RWe_3	***	*			*		
edge 4, RWe_4		*	*				

Table 8 : This table shows the interactions between different factors that are statistically significant in the analysis of variance for each independent variable. The symbols (explained in the key to Table 6) relate to probabilities.

Table 9 identifies trends in the processing conditions that are statistically significant that could be used to control shrinkage and warpage of mouldings. From Table 9 it is clear that increasing the holding pressures is the easiest way to control dimensional stability. The question of whether or not changes in holding pressure will be sufficient to compensate for the effects of phthalocyanine blue is more difficult to answer. We have shown that it is possible to limit shrinkage and warpage through increasing the hold pressure. However since phthalocyanin blue alters the shrinkage ratio it may mean that it is practically impossible to produce

	Injection Time	Hold Pressure	Melt Temp.	Hold time	Mould temp
Shrinkage, $S_{\perp}$		high > low	high = med. > low		(low > med.)
Shrinkage, $S_{\parallel}$		high > low	(high > low = med.)		
Warpage, $W_{\perp}$		high > low			
Warpage, $W_{\parallel}$		high > low	high > low = med.		
Whole plate, RW_z	low > med. > high	high > low	high > med. > low		high > low
Angle score		high > low	(high = low = med.)		
$S_{\perp} / S_{\parallel}$					
Weight	high > low = med.	high > low	high = med. > low	high > low	low > high
range1e	(low > med. > high)		high > med. > low		
range2e	med. > low > high	high > low			
range3e		high > low	(high > med. > low)		
range4e		high > low			high > low

Table 9: The influence of different processing conditions on the dimensional stability of moulded plates. These data represent statistically significant correlations between different processing conditions and measures of dimensional instability (see Table 7). Values for each of the processing variables can be found in Table 1.

Yang and Nunn ¹⁶	Girard et al ²⁰	Salloum et al ¹⁴	Yang and liou ²⁸	Mammat et al ¹⁸	Jou ¹⁵	Jones ²⁹	ANOVA shrinkage ¹²
Melt temp.	Holding pressure	Holding pressure	Melt temp.	Holding pressure	Mould Temp.	Melt Temp.	Holding pressure
Holding pressure	Cooling time	Cooling time	Shot size	Holding time	Melt temp.	Mould temp.	Holding Time *
Mould temp.	Injection Pressure	Injection Pressure				Cooling time	Melt temp. *
							Inj. time *
							Mould Temp. *

Table 10 : A comparison of the influence of most significant processing factors (ranked in order of decreasing significance) on the amalgamated shrinkage that occurs in directions parallel and perpendicular to that of the flow direction. The asterisks shown for the ANOVA results indicate that there were no differences between these quantities.

identical mouldings from virgin and blue feedstocks in the same cavity. In practice any small differences in shrinkage behaviour that might arise could be accommodated within the part tolerance providing that out-of-plane warpage is controlled.

The findings of this investigation are broadly in agreement with those previously reported as shown in Table 10. However we note that the relationship between dimensional instability and different processing conditions will to some extent depend on the measures used to characterise shrinkage.

6 CONCLUSIONS

- Phthalocyanine blue either added directly to HDPE or introduced as a masterbatch causes less shrinkage perpendicular to the flow direction than either virgin or yellow but more shrinkage parallel to it. This alters the anisotropic shrinkage ratio from approximately 1.2 to 0.55.
- High holding pressure improves dimensional stability.
- Longer holding times limit the shrinkage perpendicular to flow and are associated with heavier plates.
- Changing the processing conditions will not significantly affect the ratio of shrinkage measured parallel and perpendicular to the direction of flow.
- Out of plane distortion is reduced by increasing the speed at which the cavity fills - faster flow rates during filling (i.e. shorter injection times), increasing mould or melt temperature and by raising the hold pressure.
- Longer holding times do not appear to affect out-of-plane distortion.

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Annex 1

Shrinkage, warpage and angle scores for each of the runs listed in Table 1 together with the mean weight of each set of plates. These data represent averages for batches of five plates. The range for each score is given in brackets.

	$S_{\perp}$	$S_{\parallel}$	$W_{\perp}$	$W_{\parallel}$	RW_z	Angle score	Weight (g)
Virgin (0000)	0.019 (0.001)	0.014 (0.005)	0.492 (0.004)	0.492 (0.003)	0.017 (0.005)	0.004 (0.006)	114.6 (0.0)
Virgin (0001)	0.019 (0.005)	0.015 (0.004)	0.490 (0.005)	0.492 (0.001)	0.016 (0.006)	0.005 (0.006)	114.9 (0.1)
Virgin (0002)	0.019 (0.002)	0.014 (0.003)	0.491 (0.003)	0.492 (0.002)	0.017 (0.004)	0.004 (0.004)	115.0 (0.1)
Virgin (0010)	0.016 (0.006)	0.015 (0.004)	0.492 (0.004)	0.493 (0.004)	0.011 (0.005)	0.001 (0.003)	118.3 (0.0)
Virgin (0012)	0.014 (0.004)	0.013 (0.009)	0.493 (0.004)	0.494 (0.007)	0.011 (0.006)	0.003 (0.007)	119.8 (0.3)
Virgin (0100)	0.021 (0.003)	0.015 (0.003)	0.490 (0.003)	0.492 (0.003)	0.017 (0.001)	0.006 (0.007)	114.8 (0.0)
Virgin (0102)	0.021 (0.004)	0.015 (0.002)	0.490 (0.003)	0.492 (0.001)	0.017 (0.004)	0.007 (0.005)	115.2 (0.4)
Virgin (0111)	0.014 (0.003)	0.014 (0.007)	0.492 (0.002)	0.493 (0.005)	0.011 (0.007)	0.002 (0.002)	118.7 (0.1)
Virgin (0200)	0.021 (0.004)	0.014 (0.005)	0.491 (0.002)	0.492 (0.004)	0.018 (0.004)	0.006 (0.006)	114.3 (0.1)
Virgin (0211)	0.015 (0.002)	0.014 (0.005)	0.492 (0.004)	0.493 (0.005)	0.010 (0.002)	0.004 (0.005)	118.8 (0.1)
Blue (1000)	0.015 (0.002)	0.022 (0.003)	0.496 (0.003)	0.485 (0.001)	0.021 (0.002)	0.020 (0.003)	115.1 (0.0)
Blue (1001)	0.014 (0.002)	0.021 (0.003)	0.495 (0.002)	0.485 (0.003)	0.018 (0.004)	0.020 (0.005)	115.3 (0.0)
Blue (1012)	0.008 (0.001)	0.013 (0.004)	0.498 (0.001)	0.494 (0.002)	0.014 (0.003)	0.009 (0.004)	119.7 (0.3)
Blue (1100)	0.014 (0.001)	0.021 (0.007)	0.497 (0.003)	0.486 (0.001)	0.028 (0.012)	0.020 (0.004)	115.3 (0.1)
Blue (1101)	0.013 (0.001)	0.021 (0.003)	0.496 (0.001)	0.485 (0.003)	0.020 (0.005)	0.021 (0.004)	115.1 (0.1)
Blue (1110)	0.011 (0.002)	0.015 (0.003)	0.498 (0.001)	0.493 (0.003)	0.016 (0.005)	0.010 (0.006)	119.1 (0.2)
Blue (1112)	0.008 (0.002)	0.014 (0.004)	0.500 (0.002)	0.493 (0.004)	0.013 (0.004)	0.011 (0.006)	119.6 (0.1)
Blue (1200)	0.015 (0.003)	0.023 (0.008)	0.496 (0.002)	0.485 (0.003)	0.027 (0.005)	0.023 (0.005)	115.0 (0.0)
Blue (1201)	0.013 (0.001)	0.021 (0.004)	0.496 (0.002)	0.485 (0.002)	0.027 (0.003)	0.022 (0.003)	115.3 (0.1)
Blue (1202)	0.013 (0.001)	0.021 (0.003)	0.495 (0.001)	0.486 (0.001)	0.019 (0.004)	0.020 (0.004)	115.4 (0.3)
Blue (1211)	0.010 (0.001)	0.017 (0.007)	0.498 (0.003)	0.492 (0.003)	0.015 (0.002)	0.013 (0.007)	119.4 (0.3)
Blue (1212)	0.008 (0.006)	0.013 (0.006)	0.499 (0.002)	0.494 (0.006)	0.011 (0.001)	0.009 (0.012)	120.0 (0.6)
Yellow (2000)	0.020 (0.003)	0.014 (0.003)	0.491 (0.005)	0.493 (0.002)	0.015 (0.006)	0.006 (0.004)	114.7 (0.1)
Yellow (2002)	0.020 (0.004)	0.015 (0.002)	0.491 (0.002)	0.492 (0.003)	0.015 (0.004)	0.005 (0.005)	115.2 (0.1)
Yellow (2011)	0.016 (0.002)	0.015 (0.004)	0.492 (0.002)	0.494 (0.006)	0.009 (0.003)	0.003 (0.010)	119.3 (0.1)
Yellow (2012)	0.013 (0.002)	0.014 (0.008)	0.494 (0.003)	0.497 (0.003)	0.011 (0.009)	0.004 (0.009)	119.9 (0.2)
Yellow (2101)	0.021 (0.002)	0.013 (0.003)	0.491 (0.003)	0.492 (0.001)	0.015 (0.003)	0.007 (0.003)	115.1 (0.0)
Yellow (2102)	0.020 (0.003)	0.016 (0.006)	0.490 (0.002)	0.492 (0.003)	0.017 (0.002)	0.007 (0.005)	115.2 (0.1)
Yellow (2111)	0.015 (0.004)	0.012 (0.011)	0.492 (0.003)	0.495 (0.007)	0.010 (0.002)	0.007 (0.011)	119.5 (0.1)
Yellow (2112)	0.014 (0.004)	0.012 (0.004)	0.493 (0.003)	0.494 (0.006)	0.009 (0.003)	0.004 (0.007)	120.0 (0.1)
Yellow (2201)	0.019 (0.003)	0.015 (0.007)	0.492 (0.001)	0.491 (0.004)	0.022 (0.002)	0.004 (0.005)	115.2 (0.1)
Yellow (2210)	0.015 (0.004)	0.012 (0.009)	0.492 (0.004)	0.493 (0.004)	0.012 (0.003)	0.003 (0.008)	118.6 (0.1)
Yellow (2212)	0.014 (0.001)	0.012 (0.010)	0.493 (0.003)	0.495 (0.008)	0.010 (0.004)	0.007 (0.013)	120.1 (0.3)
MB blue (3001)	0.017 (0.003)	0.021 (0.003)	0.493 (0.001)	0.486 (0.005)	0.019 (0.002)	0.014 (0.005)	114.6 (0.1)
MB blue (3002)	0.017 (0.002)	0.021 (0.007)	0.493 (0.001)	0.486 (0.006)	0.016 (0.002)	0.012 (0.013)	114.8 (0.1)
MB blue (3010)	0.012 (0.002)	0.014 (0.006)	0.496 (0.001)	0.493 (0.003)	0.013 (0.004)	0.005 (0.005)	118.2 (0.1)
MB blue (3011)	0.012 (0.003)	0.014 (0.003)	0.496 (0.002)	0.493 (0.004)	0.012 (0.002)	0.006 (0.005)	118.6 (0.0)
MB blue (3102)	0.016 (0.002)	0.020 (0.005)	0.493 (0.001)	0.486 (0.006)	0.018 (0.002)	0.013 (0.013)	114.5 (0.1)
MB blue (3110)	0.012 (0.002)	0.014 (0.003)	0.496 (0.002)	0.493 (0.003)	0.014 (0.006)	0.005 (0.007)	118.0 (0.1)
MB blue (3202)	0.015 (0.003)	0.018 (0.007)	0.494 (0.002)	0.488 (0.007)	0.018 (0.007)	0.010 (0.015)	114.8 (0.1)
MB blue (3211)	0.012 (0.002)	0.014 (0.004)	0.496 (0.001)	0.492 (0.002)	0.017 (0.004)	0.005 (0.004)	118.3 (0.1)

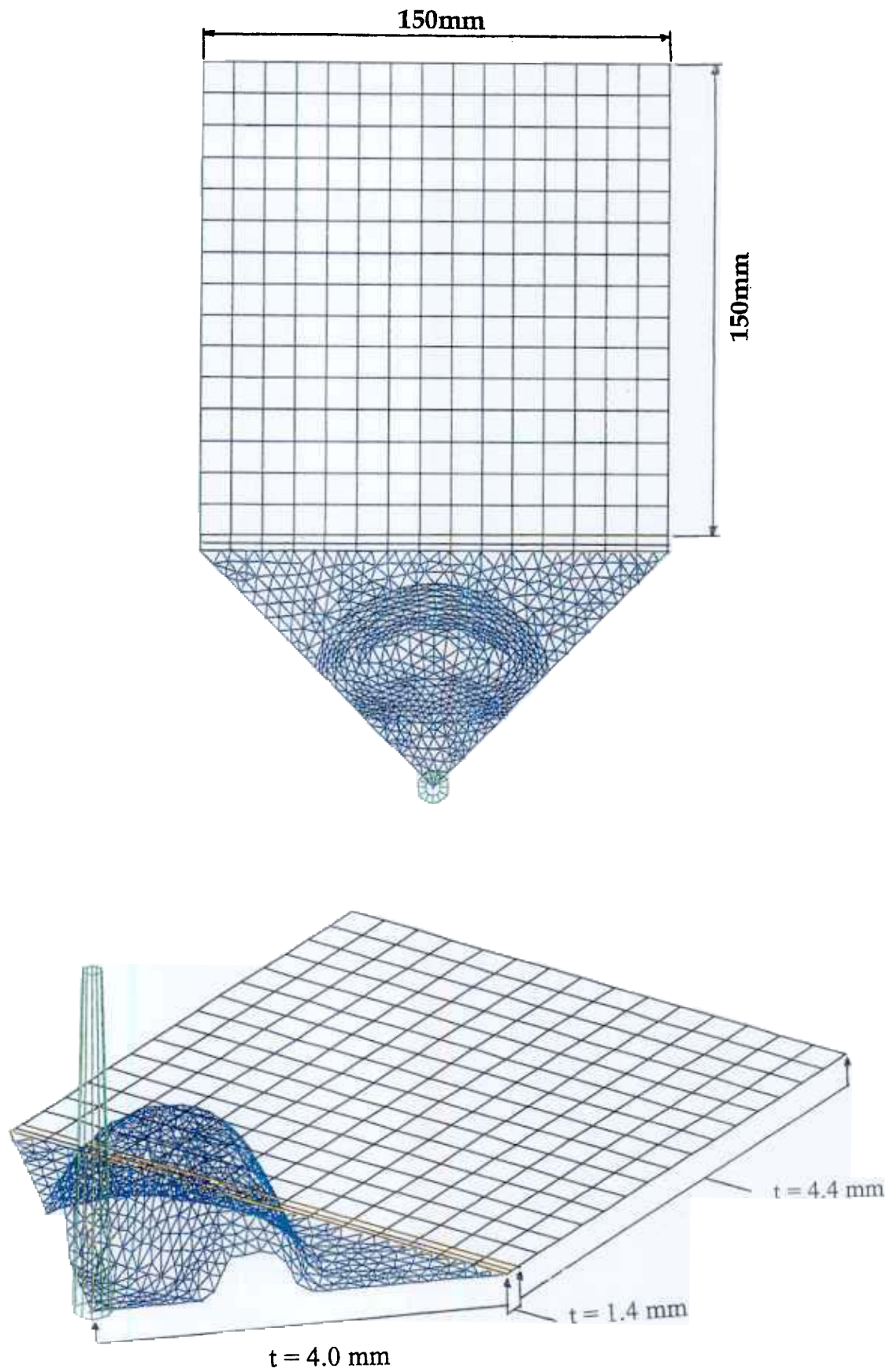


Figure 1: Cavity dimensions, t denotes thickness.

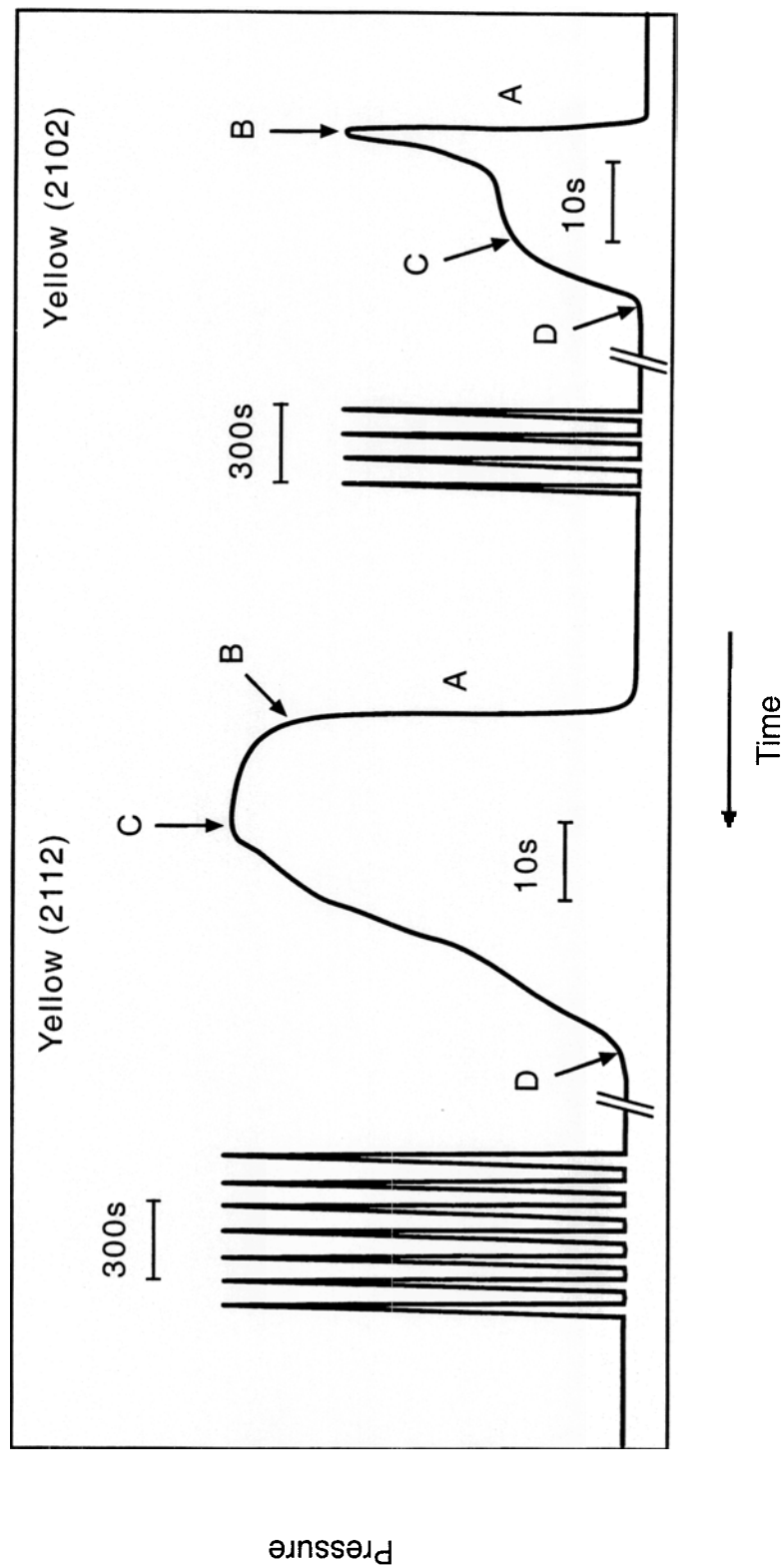


Figure 2. Identical injection times, melt and mould temperatures and holding times were used to manufacture the yellow mouldings corresponding to these pressure traces. (The conditions used can be traced through the sample codes from table 1). The difference between the trace on the left and that on the right is in the machine set holding pressure (40 bar and 20 bar respectively). A = star of injection, B = start of hold pressure, C = gate freeze off, D = part ejection. Note that for yellow 2102 that the residual in ejection pressure is greater than the hold pressure. The pressure decay between C and D is due to shrinkage of the moulding after the gate has frozen.

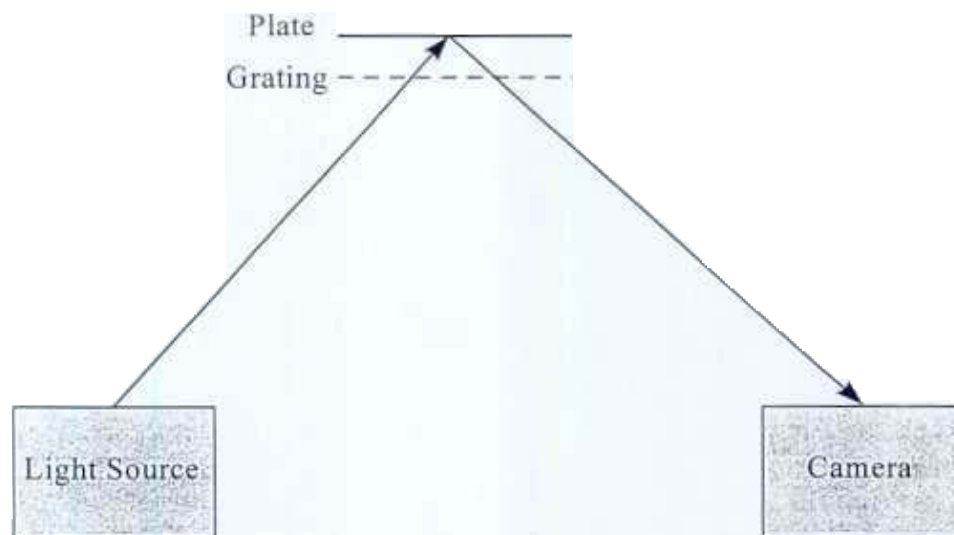
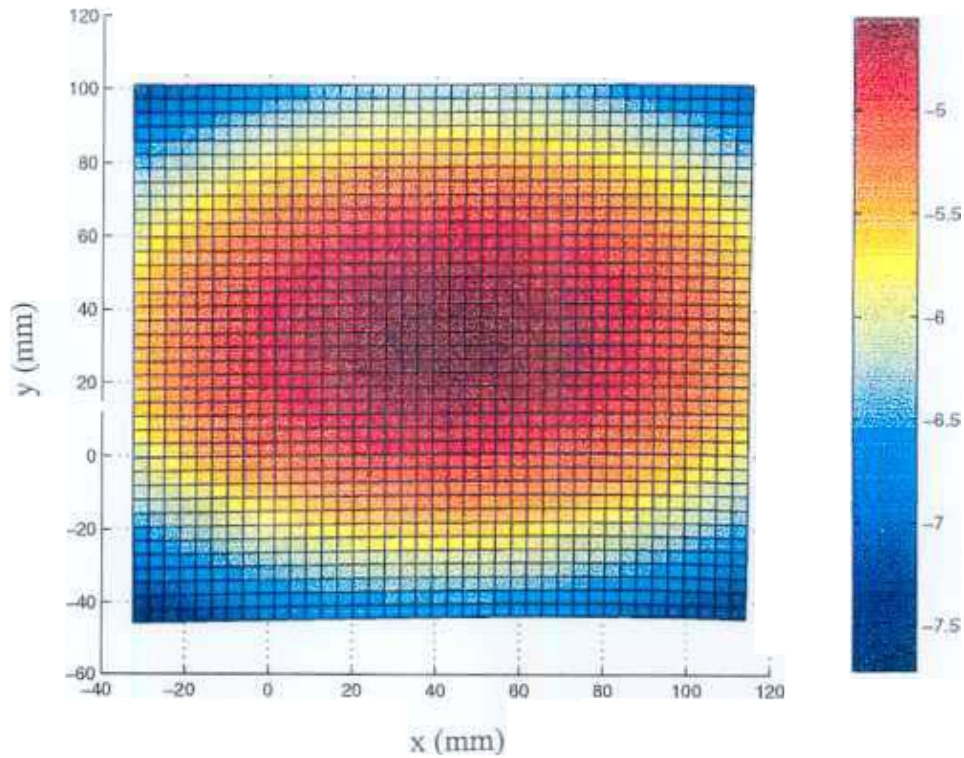


Figure 3: A schematic representation of the interferometer.

A



B

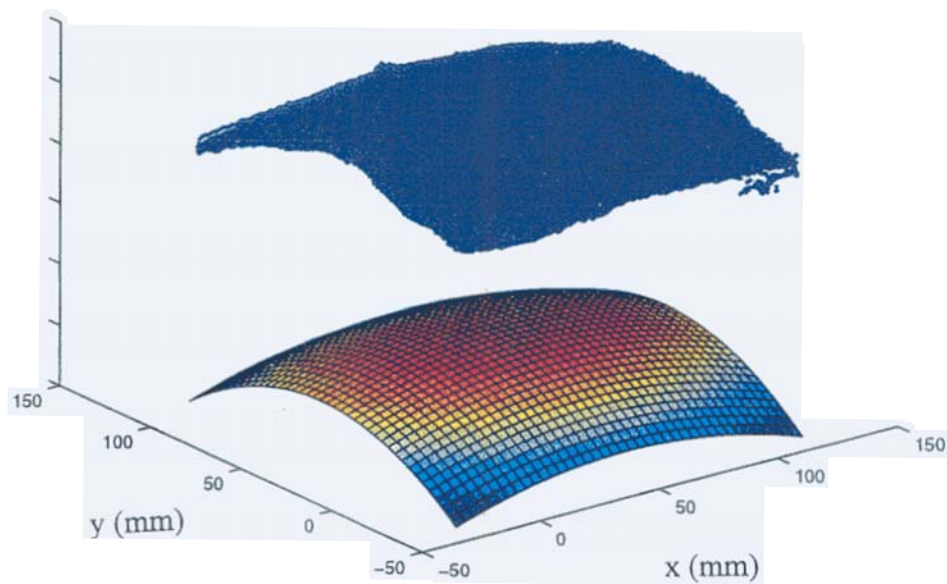


Figure 4: a) A contour plot showing out-of-plane warpage for a plate containing MB blue. The melt enters the cavity along the top edge of the plot corresponding to $y=100$.
 b) Fitting a three dimensional surface to the point cloud derived from analysis of the optical interference pattern gives a contour plot representing out-of-plane warpage of the plate.

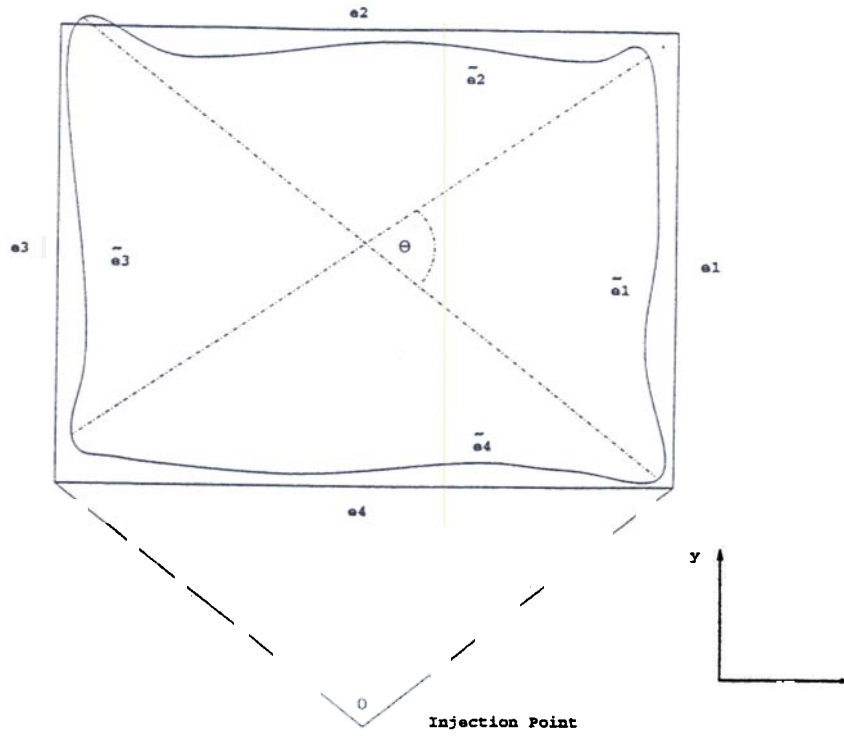
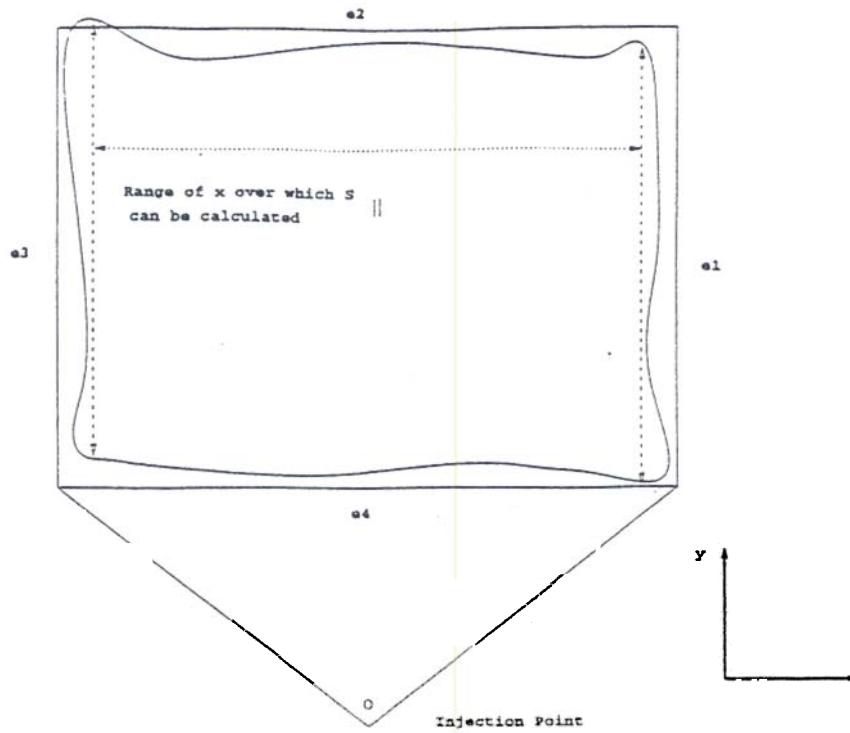


Figure 5: A comparison of the projections of the undeformed plate with edges e_1 , e_2 , e_3 and e_4 and a deformed plate with edges $\tilde{e}_1$, $\tilde{e}_2$, $\tilde{e}_3$ and $\tilde{e}_4$ on to the xy plane. The angle, θ formed by the intersections of the diagonals of the deformed plate is also shown.

A



B

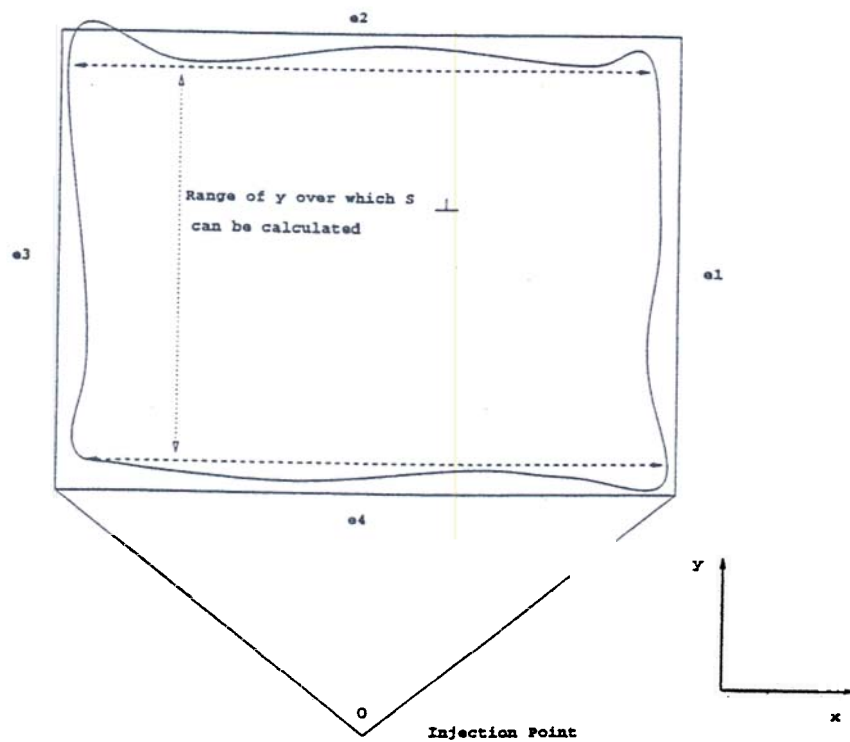


Figure 6:A: Equation 1 can be used to compute the shrinkage parallel to the direction of flow between the dashed line boundaries.

B: Equation 2 can be used to compute the shrinkage perpendicular to the direction of flow between the dashed line boundaries.

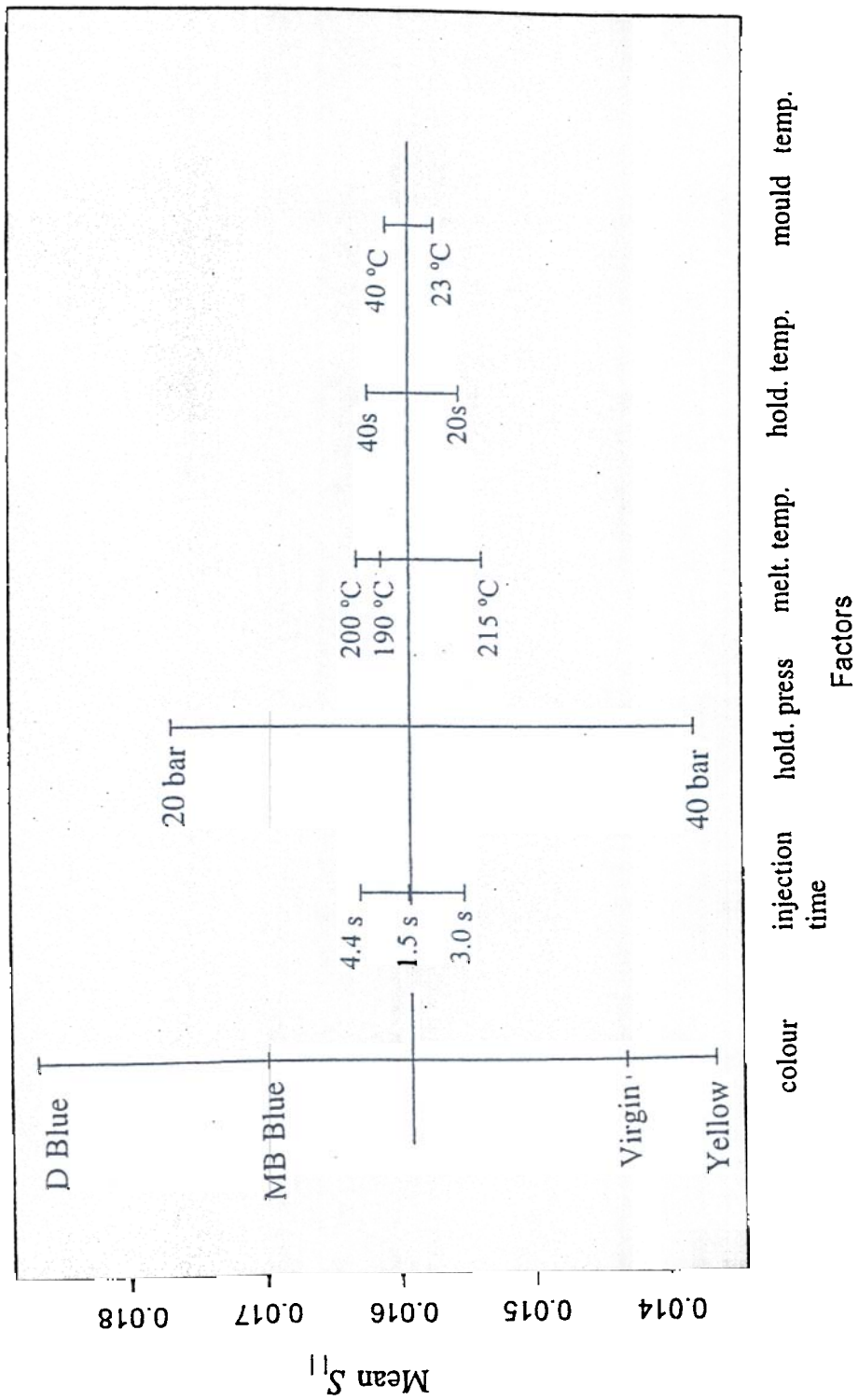


Figure 7: The analysis of means for shrinkage parallel to the direction of flow.

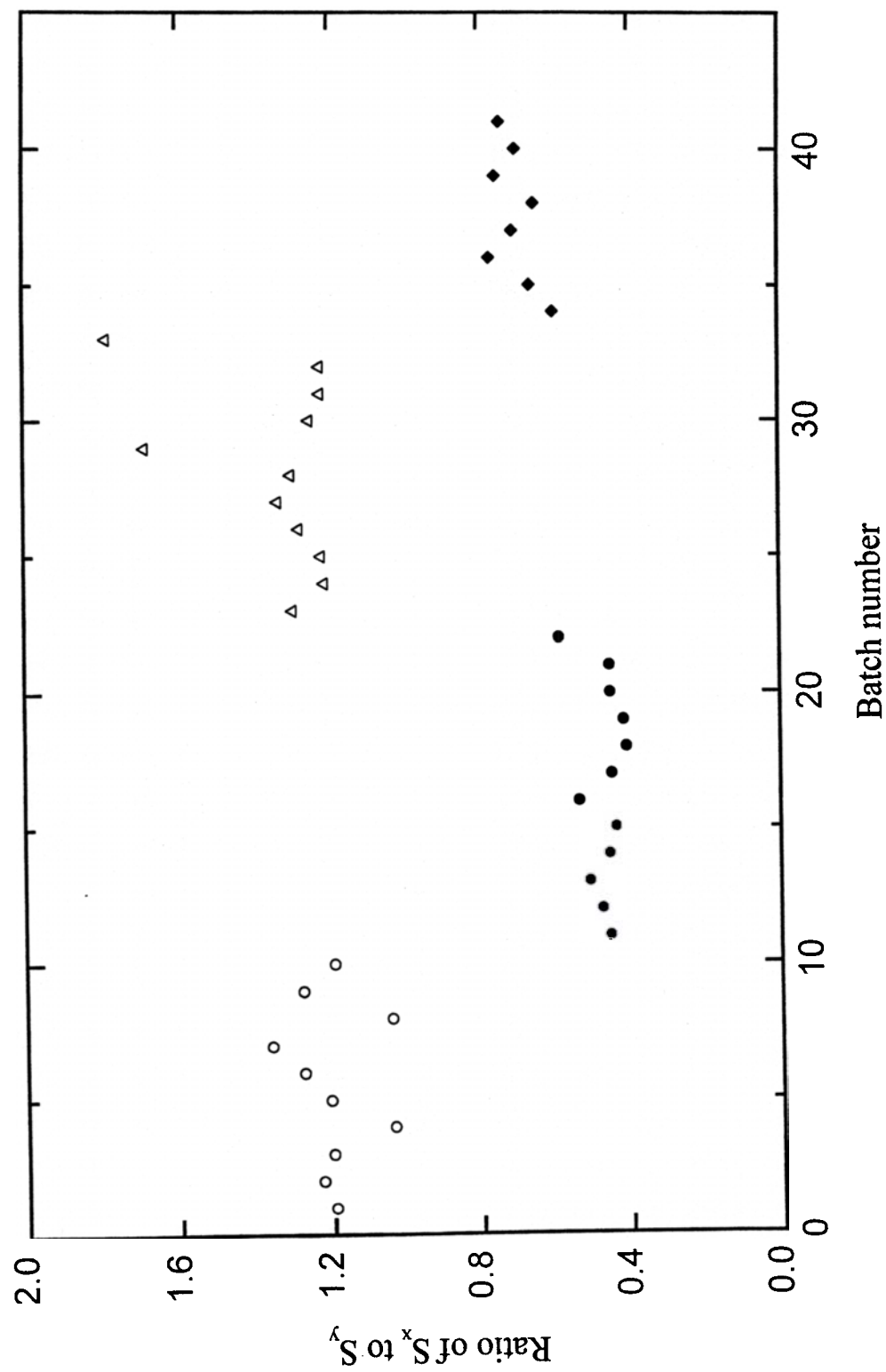


Figure 9: The shrinkage ratio is strongly dependent on the type of pigment present (O = virgin, Δ = yellow, ● = direct mix blue, ◆ = masterbatch blue). These data represent the average of five different plates within a batch. The processing conditions relating to the batch numbers can be traced from table 1.

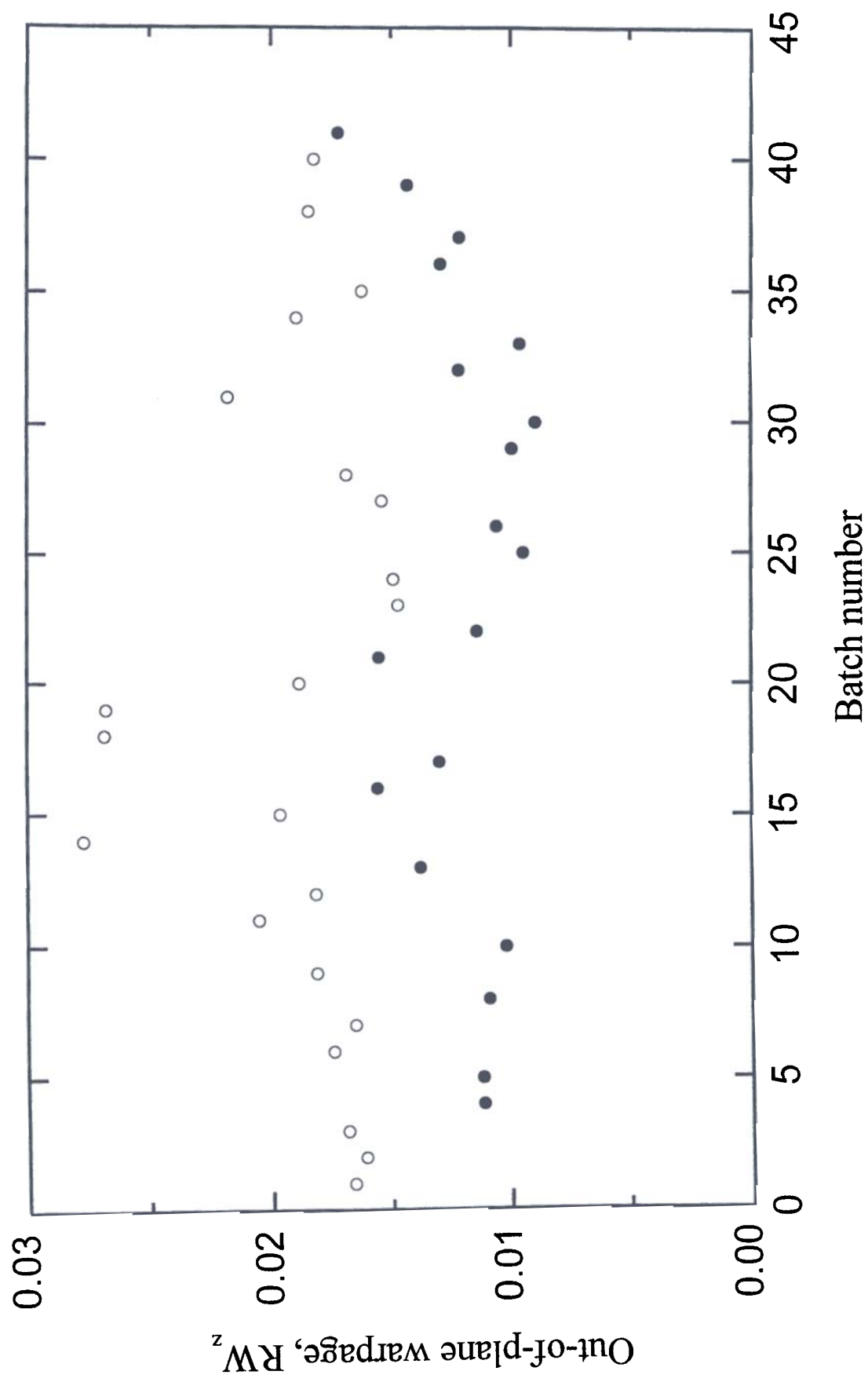


Figure 10: Out-of-plane warpage is most affected by changes in holding pressure, $\circ$ = low, $\bullet$ = high. These data represent the averages of five different plates within a batch.

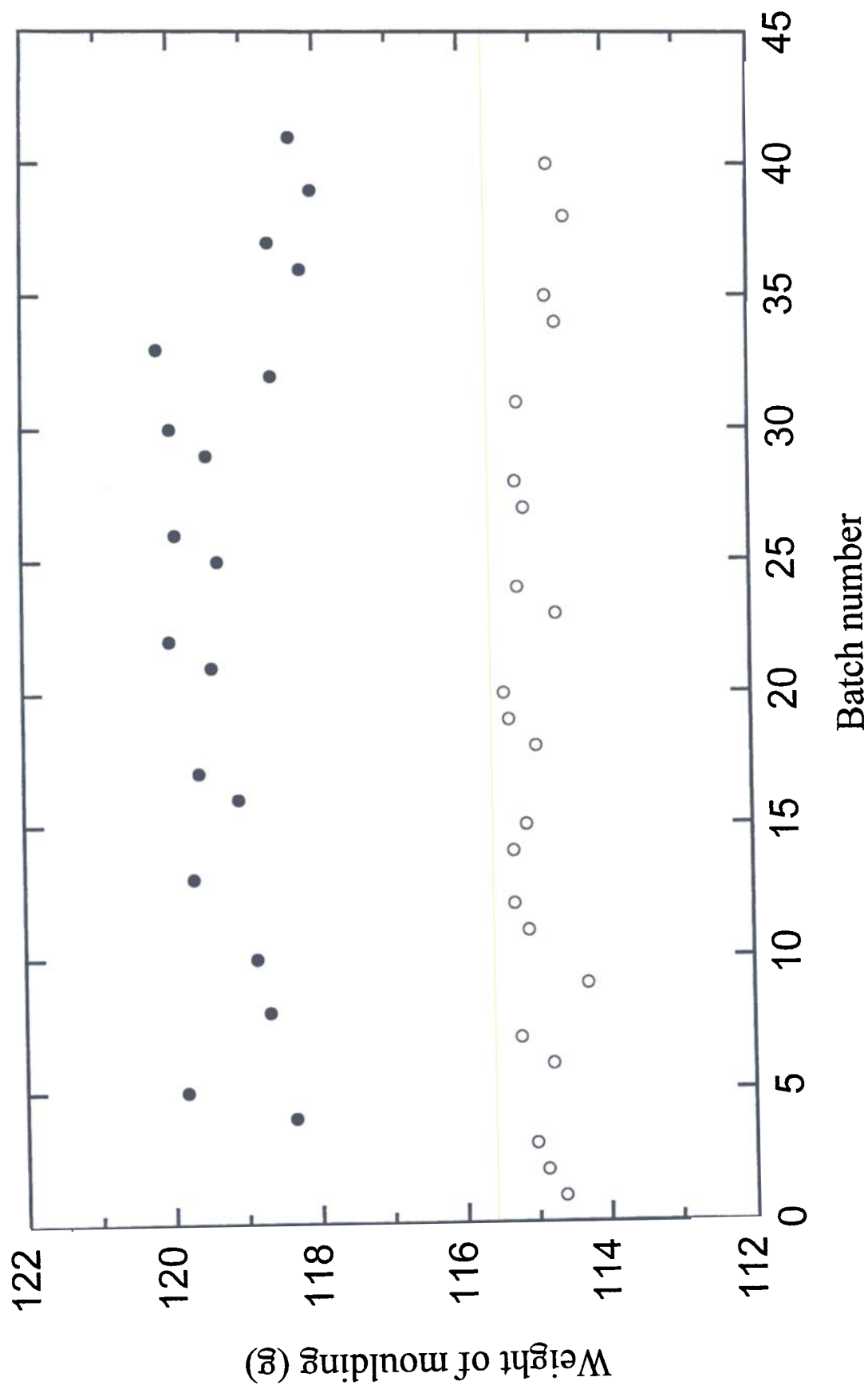


Figure 11: The variation in the mean weight of each batch of five plates together runners and sprues as a function of colour and different processing conditions. (O = low holding pressure, ● = high holding pressure)