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EFFECTS OF MOISTURE AND TRANSFER
PRESSURE ON SURFACE FINISH AND
SHRINKAGE OF 52-20-30 DIALLYL PHTHALATE

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Topical Report

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EFFECTS OF MOISTURE AND TRANSFER PRESSURE ON SURFACE FINISH AND SHRINKAGE OF 52-20-30 DIALLYL PHTHALATE

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Prepared by B. A. Hefner, D/861, under EP 041236-11 and EP 041182-17

Preform pills of 52-20-30 long glass-filled diallyl phthalate (DAP) were conditioned to achieve desired moisture levels. The pills were then transfer-molded at various pressures to determine the effects of moisture content and transfer pressures on material shrinkage and surface roughness.

Analyses disclosed that moisture acted as a propellant, causing the material to pack to a greater density. The shrinkage decreased and warp was improved with increased transfer pressure or moisture content. The best surface finish was achieved adjacent to the gate. The interaction term of moisture and transfer pressure was not as significant as expected, appearing only in shrinkage, lack of compaction, and end curl.

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CONTENTS

Section	Page
SUMMARY.	5
DISCUSSION	6
SCOPE AND PURPOSE.	6
PRIOR WORK	6
ACTIVITY	6
<u>Testing Procedures</u>	6
<u>Test Results</u>	7
ACCOMPLISHMENTS.	17
FUTURE WORK.	18
DISTRIBUTION	19

ILLUSTRATIONS

Figure		Page
1	Shrinkage Versus Moisture Content of 52-20-30 DAP for Various Transfer Pressures.	10
2	Shrinkage Versus Transfer Pressure for Various Moisture Contents of 52-20-30 DAP. .	11
3	Shrinkage Versus Transfer Pressure and Moisture Content of 52-20-30 DAP	14

TABLES

Number		Page
1	Processing Criteria for Study of Diallyl Phthalate.	8
2	Matrix of Study.	8
3	Effects of Moisture and Transfer Pressure on Shrinkage, Surface Finish, and Runout for Diallyl Phthalate 52-20-30	9

SUMMARY

The purpose of this study was to evaluate the effects of moisture content and transfer pressure on material shrinkage and surface roughness on a specific lot of long glass-filled diallyl phthalate. This activity was a continuation of related work conducted under process development order 6984548. The previous work revealed that molding pressure, mold temperature, cure time, moisture content, gate size and position, and pill size could affect the material moldability, dimensional characteristics, and physical properties of transfer-molded parts.

During this study preform pills from one lot of 52-20-30 diallyl phthalate (DAP) were conditioned to achieve desired moisture levels. The pills were transfer-molded at selected transfer pressures, according to a designed study, and the results were statistically analyzed. The eight responses analyzed were shrinkage, runout or warp, surface finish opposite to gate, surface finish adjacent to gate, cull thickness, difference in shrinkage, compaction opposite the gate, and end curl.

The following major conclusions were derived from this investigation.

- Moisture acted like a propellant, or lubricant, causing the material to flow more easily.
- Shrinkage decreased as density increased with increased transfer pressure or moisture content.
- Warp improved with increased moisture and transfer pressure.
- The best surface finish was achieved adjacent to the gate with an overall mean finish of 31.8 rms microinch (0.81 micron) compared to 46.0 rms microinch (1.17 micron) opposite the gate.
- The interaction term of moisture and transfer pressure was not as significant as expected.

Further activity will include a study to determine the effects of preheating on the physical and electrical properties. Also, an additional video program will be completed, depicting various plastic materials flowing into different mold cavity and gate configurations, using the special flow study mold which contains a quartz lens window.

DISCUSSION

SCOPE AND PURPOSE

Previous studies have shown that excess moisture can reduce tensile strength and improve material moldability. The intent of this project was to evaluate the effects of moisture and transfer pressure on the shrinkage, surface finish, and warp characteristics of a specific material.

A study was conducted of a long glass-filled green diallyl phthalate molding compound (52-20-30) which meets the requirements of Material Specification 2082437. A total of 21 lines were molded with five levels each of transfer, pressure and moisture content.

PRIOR WORK

The initial transfer-molding development activity at Bendix Kansas City was started in 1965 under PDO 6984548. The significant studies during that project consisted of statistical evaluations of the effects of molding parameters, cup test and spiral test evaluation, an evaluation to determine the effects of moisture on material moldability, and a photo-mold study. The study of 52-20-30 diallyl phthalate, as well as additional photo-mold films, were started under PDO 6984548 and will be completed under EP 041236-11 and EP 041182-17.

ACTIVITY

Testing Procedures

A study was designed to evaluate the effects and interactions of moisture and transfer pressure on long glass-filled diallyl phthalate. The levels of processing are listed in Table 1.

The samples were dried in a vacuum chamber and then were conditioned until the sample being monitored had reached the desired moisture content. The samples were sealed in vapor barrier bags until needed and were warmed up to room temperature overnight while still in the bags. The percentage of final moisture content, when measured by CEC analysis, and the corrected levels of coding were as follows:

0.13 (-2.2);

0.215 (-1.35);

0.29 (-0.6);

0.35 (0.0);

0.45 (1.0); and

0.51 (1.6).

During the study, a special test mold was used to mold end-gated samples which were 0.25 by 1.00 by 3.00 inch (6.35 by 25.4 by 76.2 mm). Two 25-gram preform pills were cut in such a way that a total of 37 grams were molded. The mold had a 0.084-inch-diameter (2.13 mm) half-round gate. The following processing variables remained constant:

Mold Temperature 290 $\pm$ 2°F (161 $\pm$ 1.1°C)

Cure Time 15 minutes

Pill Weight 37 grams

Cull Thickness (Target) 0.125 inch (3.2 mm)

Clamp (Line) Pressure 1600 psi (11.03 MPa)

The molding pressure within the cavity was 3.5 times the transfer line pressure. Table 2 is a matrix of the study with the coded values that were attained.

Line 1, which did not fill [400 psi pressure (2.8 MPa) and 0.29-percent moisture], was omitted from the statistical evaluation.

Test Results

After the molding was completed, the samples were inspected for total runout, surface finish, and overall length, in accordance with the following instructions. Total runout was measured along two imaginary lines, 0.25 inch (6.35 mm), from each edge on the bottom surface (cavity side) for the full length of the sample. Surface finish was also determined on the bottom surface at each end and across the sample width. The overall length was measured at three places, and the shrinkage was then determined. Table 3 shows the mean values for the three samples of each line. Figures 1 and 2 show the effects of moisture and transfer pressure on shrinkage.

Generally speaking, the shrinkage decreases as the moisture and transfer pressure increase. However, it is difficult to determine the interactions of moisture and transfer pressure with this type of analysis. To achieve these results, the data were statistically analyzed and fitted to the following full quadratic model, with two independent variables by stepwise regression.

Table 1. Processing Criteria for Study of 52-20-30
Diallyl Phthalate

Coded Level	Transfer Pressure Setting		Moisture Content (Percent)
	psi	MPa	
-2	400	2.8	0.15
-1	700	4.8	0.25
0	1000	6.9	0.35
1	1300	9.0	0.45
2	1600	11.0	0.55

Table 2. Matrix of Study

Lines	Transfer Pressure, X_1	Percent Moisture Content, X_2
1	-2	-0.6
2-3	-1	-1.35
4-5	-1	0
6	0	-2.2
7	0	1.6
8-15	0	-0.6
16-17	1	-1.35
18-19	1	0
20	2	-0.6
21	1	1.0

Table 3. Effects of Moisture and Transfer Pressure on Shrinkage, Surface Finish, and Runout for 52-20-30 Dialllyl Phthalate

Molding Parameters				Measurements of Test Blocks				
Line	Moisture Content (Weight Percent)	Transfer Pressure psi	MPa	Shrinkage (in./in.) (mm/mm)	RMS Surface Finish=O,G* μin. μm		Total Runout for 3.0-in. (76 mm) Length in. mm	
1	0.29	400	2.76	NA	NA	NA	NA	NA
2	0.22	700	4.82	0.0056	55,30	1.40,0.76	0.0043	0.109
3	0.22	700	4.82	0.0055	48,28	1.22,0.71	0.0033	0.084
4	0.35	700	4.82	0.0048	47,45	1.19,1.14	0.0036	0.091
5	0.35	700	4.82	0.0051	42,48	1.07,1.22	0.0034	0.086
6	0.13	1000	6.90	0.0065	45,32	1.14,0.81	0.0035	0.089
7	0.51	1000	6.90	0.0030	50,28	1.27,0.71	0.0033	0.084
8	0.29	1000	6.90	0.0049	37,40	0.94,1.02	0.0028	0.071
9	0.29	1000	6.90	0.0043	30,32	0.76,0.81	0.0027	0.069
10	0.29	1000	6.90	0.0044	35,37	0.89,0.94	0.0026	0.067
11	0.29	1000	6.90	0.0043	52,32	1.32,0.81	0.0032	0.081
12	0.29	1000	6.90	0.0042	42,33	1.07,0.84	0.0033	0.084
13	0.29	1000	6.90	0.0046	40,28	1.02,0.71	0.0033	0.084
14	0.29	1000	6.90	0.0049	50,35	1.27,0.89	0.0037	0.094
15	0.29	1000	6.90	0.0041	47,37	1.19,0.94	0.0041	0.104
16	0.22	1300	8.97	0.0042	47,27	1.19,0.69	0.0036	0.091
17	0.22	1300	8.97	0.0042	50,30	1.27,0.76	0.0034	0.086
18	0.35	1300	8.97	0.0025	37,27	0.94,0.69	0.0031	0.079
19	0.35	1300	8.97	0.0026	27,35	0.69,0.89	0.0037	0.094
20	0.29	1600	11.0	0.0027	47,42	1.19,1.07	0.0029	0.074
21	0.45	1300	8.97	0.0022	45,38	1.14,0.97	0.0029	0.074

*O is end opposite from gate.

G is adjacent to gate.

Figure 1. Shrinkage Versus Moisture Content of 52-20-30 DAP for Various Transfer Pressures

Figure 2. Shrinkage Versus Transfer Pressure for Various Moisture Contents of 52-20-30 DAP

$$Y = a_0 + a_1X_1 + a_2X_2 + a_{12}X_1X_2 + a_{11}X_1^2 + a_{22}X_2^2 + C,$$

where

X_1 = Transfer line pressure (psi) (MPa),

X_2 = Percent, and

C = Constant.

Eight different responses (Y) were analyzed for each line:

- Y_1 Mean shrinkage in length, inch/inch (mm/mm);
- Y_2 Mean runout for 3-inch (76.2 mm) length (warp), total inches (mm);
- Y_3 Mean surface finish--end opposite gate, RMS-microinch (micron);
- Y_4 Mean surface finish--adjacent to gate, RMS-microinch (micron);
- Y_5 Mean cull thickness, inch (mm);
- Y_6 Difference in shrinkage = $\frac{\text{maximum} - \text{minimum length}}{2.9984}$ inch/inch (mm/mm);
- Y_7 Compaction opposite gate--a visual evaluation of the mean length in inches (mm) of dull surface or starved glass fiber at the surface opposite the gate; and
- Y_8 End curl, or width of sink, at surface opposite the gate end of part, in inches (mm).

The following are the best fitted equations to each response. Prediction variable terms are underlined.

$$\text{Shrinkage, inch per inch} = Y_1 \times 10^{-3}.$$

$$\bar{Y}_1 = 4.23 \times 10^{-3} \text{ inch (all 60 samples).}$$

$$s_{Y_1} = 1.13 \times 10^{-3} \text{ inch (all 60 samples).}$$

$$Y_1 = 3.81 - \underline{1.06X_1} - 0.71X_2 - 0.28X_1X_2 + 0.16X_2^2.$$

$$\sigma_{Y_1} = 0.30 \times 10^{-3} \text{ inch (about fitted curve).}$$

The shrinkage per inch is decreased approximately 0.001 inch (0.0254 mm) for each 300 psi (2.07 MPa) increase in transfer

pressure setting (X_1). There is about 0.0007 inch (0.0178 mm) reduction in shrinkage for each 0.1-percent increase in moisture content, or the equivalent of 200 psi (1.38 MPa) increase in transfer pressure setting.

The interaction term ($-X_1X_2$) gives further decrease in shrinkage when both transfer pressure and moisture content are at higher levels.

The moisture content appears to act as a propellant, increasing cavity pressure with resultant decrease in shrinkage per inch length.

The quadratic term ($+X_2^2$) gives a minimum at $X_2 = +2$ coded level, or 0.55-percent moisture. This is probably the upper limit of moisture content to reduce shrinkage at 290°F (161°C) mold temperature.

The optional line, 1300 psi (8.96 MPa) transfer pressure setting and 0.45-percent moisture had a shrinkage of 0.002 inch/inch (mm/mm), which is very low for DAP. It may also be noted that lines with 0.35-percent moisture or more and transfer pressure settings of 1300 psi (8.96 MPa) or higher had shrinkage of 0.003 inch/inch (mm/mm) or less, which is also low for DAP material.

A graphical representation of the data, showing the interaction of transfer pressure and moisture, is given in Figure 3.

Mean runout for 3-inch (76.2 mm) length (warp), total inches
 $= \bar{Y}_2 \times 10^{-3}$ inch.

Mean $\bar{Y}_2 = 3.32 \times 10^{-3}$ inch (all 60 samples).

$s_{Y_2} = 0.44 \times 10^{-3}$ (all 60 samples).

$Y_2 = 3.26 - 0.15X_1 - 0.16X_2$.

$\sigma_{Y_2} = 0.43 \times 10^{-3}$ inch (about fitted curve).

The fit was poor. One repeat line at 0, -0.6 was large compared to the other seven lines at the same levels. If this line were removed, the fit to the data would improve. The warp, or runout, was reduced with increased transfer pressure or moisture content.

Mean surface finish, end opposite gate = Y_3 rms (microinch).

Figure 3. Shrinkage Versus Transfer Pressure and Moisture Contents of 52-20-30 DAP

Mean $\bar{Y}_3 = 46.0$ (all 60 samples).

$s_{Y_3} = 5.89$ (all 60 samples).

$$Y_3 = 43.13 - 3.40X_1 + 3.28X_1^2 + 1.31X_2^2.$$

$\sigma_{Y_3} = 5.35$ (about fitted curve).

The fit was not good. The transfer pressure was the most significant variable with predicted minimum or best finish at $X_1 = +0.5$, or 1150 psi (7.93 MPa) transfer pressure setting.

The moisture content had a minimum or best finish at $X_2 = 0$ or 0.35-percent moisture content.

Mean surface finish, adjacent to gate = Y_4 rms (microinch).

Mean $\bar{Y}_4 = 31.80$ (all 60 samples).

$s_{Y_4} = 4.40$ (all 60 samples).

$$Y_4 = 30.81 - 1.75X_1 + 1.93X_1^2.$$

$\sigma_{Y_4} = 4.30$ (about fitted curve).

The fit was poor. The transfer pressure setting should be $X_1 = +0.45$, or 1135 psi (7.83 MPa) for best finish. The finish was better on the gate end of all parts.

The surface had a large pebble-finish pattern that was present on all test specimens. This could be a problem if a smooth mirror finish were required.

Cull thickness in transfer cylinder = Y_5 inches.

Mean $\bar{Y} = 0.1095$ inch (all 60 samples).

$s_{Y_5} = 0.23$ inch (all 60 samples).

$$Y_5 = 0.115 - \underline{0.017X_1} - 0.007X_2 - 0.010X_1^2.$$

$\sigma_{Y_5} = 0.0091$.

Using a constant charge weight pill (37 grams), the cull thickness should vary inversely with the test specimen size. The transfer pressure was a predictor variable with maximum cull thickness at $X_1 = -0.85$, or 745 psi (5.14 MPa).

The moisture content also reduces the cull thickness. The effect is approximately the equivalent of 200 psi (1.38 MPa) increase in transfer pressure setting for each 0.1-percent increase in moisture content.

$$\text{Difference in shrinkage} = \frac{\text{maximum} - \text{minimum length}}{2.9984},$$

$$= Y_6 \times 10^{-3} \text{ inch/inch (mm/mm)}.$$

$$\text{Mean } \bar{Y}_6 = 1.46 \times 10^{-3} \text{ (all 60 samples)}.$$

$$s_{Y_6} = 0.55 \times 10^{-3} \text{ (all 60 samples)}.$$

$$Y_6 = 1.23 - 0.29X_1 - 0.31X_2 + 0.185X_1^2.$$

$$\sigma_{Y_6} = 0.47 \times 10^{-3} \text{ (about fitted curve)}.$$

There is no predictor variable. The difference in shrink is reduced with increased transfer pressure or percent moisture content. The effect of transfer pressure is reduced at a higher pressure setting, with a minimum at $X_1 = +0.8$ (1240 psi) (8.55 MPa), while the shrink difference is reduced with increased percent moisture content over the range used in the study. The reduction in the shrink difference is the elimination of the sink or low region about the gate. The lowest value reported was 0.78 inch $\times 10^{-3}$ (19.8 mm) for the optional line, 1300 psi (8.96 MPa), 0.45-percent moisture content.

$$\text{Compaction opposite gate (lack of compaction)} = Y_7 \text{ inch (mm)}.$$

$$\text{Mean } \bar{Y}_7 = 0.17 \text{ inch (all 60 samples)}.$$

$$s_{Y_7} = 0.34 \text{ inch (all 60 samples)}.$$

$$Y_7 = 0.04 - \underline{0.35X_1} - 0.06X_2 + 0.14X_1X_2 + \underline{0.24X_1^2}.$$

$$\sigma_{Y_7} = 0.119 \text{ (about fitted curve)}.$$

The transfer pressure setting is a predictor variable for the lack of compaction, particularly at low transfer pressure. All lines

of the 700 psi (4.82 MPa) setting had a lack of compaction. The one line at 400 psi (2.76 MPa) setting failed to fill. One line at 1000 psi (6.89 MPa) had a fringe of poorly compacted material opposite the gate. This one line of eight at 0, -0.6 level was consistently different than the other seven lines, having a response indicating a lower transfer pressure or moisture content.

There was an increased compaction with increased moisture content over the range of values used.

The interaction term ($+X_1X_2$) implies X_1 and X_2 may be of opposite signed coded levels to reduce the lack of compaction. A completely compacted part may be obtained with high transfer pressure and low moisture content, or vice versa.

End curl or sink width at end, opposite to gate = Y_8 inch.

Mean $\bar{Y}_8 = 0.055$ inch (all 60 samples).

$s_{Y_8} = 0.120$ (all 60 samples).

$$Y_8 = 0.01 + \underline{0.12X_1} + 0.03X_2 + \underline{0.10X_1X_2} + 0.05X_1^2.$$

$\sigma_{Y_8} = 0.04$ (about fitted curve).

This is a visible reduction in thickness from the end of the block opposite the gate. It appears only on those lines of high transfer pressure and particularly with high transfer pressure and moisture content.

The transfer pressure and interaction of transfer pressure with moisture content are predictor variables. The interaction term explains the increased response when both variables are at high levels.

ACCOMPLISHMENTS

The major conclusions derived from this investigation were the following:

- Moisture seems to act as a propellant or lubricant, aiding the transfer pressure, and making the material more flowable, packing it to a greater density.
- The interaction term of moisture and transfer pressure did not appear as significant as expected. The term appeared only in shrinkage per inch, lack of compaction, and end curl or sink at the end opposite the gate.

- The sign for the terms X_1 and X_2 is always alike, and the effect is similar. The effect of the moisture content was more apparent, since the mold or cure temperature was held constant at 290°F (161°C).
- Shrinkage decreased as the material became more tightly packed. This occurred when the transfer pressure and moisture content increased. The effects of transfer pressure and moisture content could be easily confused if not carefully controlled and recorded. The extra line at 1300 psi (9.86 MPa) transfer pressure setting and 0.45-percent moisture content had a low shrinkage per inch, 2.2×10^{-3} in./in. (mm/mm), and least difference in shrinkage [0.78×10^{-3} in./in. (mm/mm)].
- The effect of moisture content as a propellant should increase with higher mold or cure temperatures.
- Runout, or warp, improved with increased moisture and transfer pressure.
- Surface finish had inflection points with the predicted best finish at 1150 psi (7.93 MPa) transfer pressure and 0.35-percent moisture for the end opposite the gate, and a transfer pressure of 1135 psi (7.83 MPa) for the moisture levels tested at the end adjacent to the gate. The best finish was achieved adjacent to the gate, with an overall mean finish of 31.8 rms microinch (0.81 micron) compared to 46.0 rms microinch (1.17 micron) opposite the gate.

FUTURE WORK

A study will be conducted of three molding compounds (glass-filled diallyl phthalate, glass-filled silicone, and Kevlar-filled epoxy) to determine the effects of preheat and moisture content on the physical and electrical properties.

A new version of the photo-mold film is being produced. This film will consist of more shots, additional cavity and gate configurations, and new materials. As reported on PDO 6984548, this work is being accomplished with the use of a mold with a glass side to facilitate photographing the fill.

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