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Effect of Preheat on Molding Compounds

By J. W. McFarland

Published June 1980

Final Report

Prepared for the United States Department of Energy
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**Kansas City
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EFFECT OF PREHEAT ON
MOLDING COMPOUNDS

By J. W. McFarland

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Final Report
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The effects of preheating the preforms and B-staging the raw materials were investigated for a silicone and a diallyl phthalate thermosetting molding compound. Preheating the preforms caused large increases in the mechanical and electrical properties of both compounds. Heat treating the raw materials had very little effect. Compression-molded specimens were 100 percent stronger than the transfer-molded ones for the silicone and 46 percent stronger for the diallyl phthalate.

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CONTENTS

Section	Page
SUMMARY.	6
DISCUSSION	7
SCOPE AND PURPOSE.	7
PRIOR WORK	7
ACTIVITY	7
<u>Silicone Molding Compound.</u>	7
<u>Diallyl Phthalate Molding Compound</u>	12
ACCOMPLISHMENTS.	18
APPENDICES	
A. SPIRAL FLOW TEST METHOD.	28
B. MOLDING PROCEDURES	30

ILLUSTRATIONS

Figure		Page
1	Spiral Flow Versus Preheat for Silicone Compound	10
2	Flexural Strength Versus Preheat for Compression-Molded Silicone.	13
3	Tensile Strength Versus Preheat for Compression-Molded Silicone.	14
4	Dielectric Strength Versus Preheat for Compression-Molded Silicone.	15
5	Flexural Strength Versus Preheat for Transfer-Molded Silicone	16
6	Dielectric Strength Versus Preheat for Transfer-Molded Silicone.	17
7	Spiral Flow Versus Preheat for Diallyl Phthalate Molding Compound.	19
8	Flexural Strength Versus Preheat for Compression-Molded Diallyl Phthalate.	22
9	Tensile Strength Versus Preheat for Compression-Molded Diallyl Phthalate.	23
10	Dielectric Strength Versus Preheat for Compression-Molded Diallyl Phthalate.	24
11	Flexural Strength Versus Preheat for Transfer-Molded Diallyl Phthalate	25
12	Tensile Strength Versus Preheat for Transfer-Molded Diallyl Phthalate	26
13	Dielectric Strength Versus Preheat for Transfer-Molded Diallyl Phthalate	27
A-1	Mold for Spiral Flow Test	29

TABLES

Number		Page
1	Total Weight Loss and Water Content	8
2	Conditioning of Test Specimens for Compression-Molded and Transfer-Molded Silicone.	8
3	Properties of Compression-Molded Silicone Specimens	11
4	Properties of Transfer-Molded Silicone Specimens	12
5	Conditioning of Test Specimens for Compression-Molded and Transfer-Molded Diallyl Phthalate	18
6	Properties of Compression-Molded Diallyl Phthlate Specimens	20
7	Properties of Transfer-Molded Diallyl Phthalate Specimens.	21

SUMMARY

A study was made to determine what effects preheating preforms and B-staging the raw materials would have on the mechanical and electrical properties of two thermosetting molding compounds. The materials studied were a glass-filled silicone and a glass-filled diallyl phthalate. Both non-B-staged and B-staged materials were molded at 0, 25, 50, 75, and 100 percent of maximum preheat time. The B-staging cycle was arbitrarily selected as that which would reduce the spiral flow to one-half its original length.

On the silicone compound, the flexural, tensile, and dielectric strengths of compression-molded specimens increased markedly with an increase in preheat. At maximum preheat both flexural and tensile strengths were approximately double those with no preheat; the dielectric strength increased about 50 percent. This was true of both the non-B-staged and B-staged materials. Preheating the preforms had very little effect on the transfer-molded silicone specimens. However, the flexural strength and dielectric strength of transfer-molded specimens were about half and two-thirds, respectively, of those of compression-molded specimens. It was very difficult to transfer-mold the B-staged silicone compound.

The flexural and tensile strengths of the compression-molded diallyl phthalate compound increased rapidly with an increase in preheat. At maximum preheat the flexural strength of non-B-staged material was 88 percent higher than at no preheat. Tensile strength was 47 percent higher. At maximum preheat the dielectric strength of non-B-staged material was 5 percent below that obtained with no preheat.

Flexural, tensile, and dielectric strengths of transfer-molded diallyl phthalate were far below the respective values for compression-molded specimens. Flexural strength increased only slightly, while tensile strength increased substantially with an increase in preheat. At maximum preheat, the flexural strength of the non-B-staged material was 4.6 percent above its value at no preheat, and the tensile strength was 29 percent higher.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this project was to determine the effect of preheat on the mechanical and electrical properties of two thermosetting molding compounds, one a silicone material and the other a diallyl phthalate.

Included in the scope is a study of the effect of B-staging. In the B-staging operation, the material was heated in an oven at a given temperature until the spiral flow was reduced to one-half its original length.

PRIOR WORK

Both materials evaluated have been used for several years. The procedure has been to work out a molding process for each production part as the need arose. This is the first study of preheat and B-staging techniques to use standardized specimens.

ACTIVITY

A glass-filled silicone and a glass-filled diallyl phthalate molding compound were tested in the B-staged and non-B-staged conditions. In this study, the times and temperatures selected were those which would reduce the spiral flow of each material to one-half its original length. Tests included flexural strength (in accordance with ASTM D-790), tensile strength (ASTM D-638), and dielectric strength (ASTM D-149).

The silicone and diallyl phthalate compounds were tested for total weight loss and water content both before and after B-staging (Table 1). For the silicone, the total weight loss was greater than the change in water content, indicating a slight loss of low molecular weight resin. In the case of diallyl phthalate, water was the only material lost during the B-staging operation.

Silicone Molding Compound

Table 2 shows how the silicone material was B-staged and preheated for each test and the spiral flow for each condition. The original spiral, with no B-stage and no preheat, was 349 mm. A B-staging cycle of 15 minutes at 93°C reduced the spiral to 165 mm. The method used for the spiral flow determination is given in Appendix A.

Table 1. Total Weight Loss and Water Content

Property	Silicone		Diallyl Phthalate	
	No B-Stage	B-Stage 15 min at 93°C	No B-Stage	B-Stage 30 min at 121°C
Total Weight Loss (Percent)	0.81	0.63	1.49	1.30
Water Content (Percent)	0.62	0.52	0.33	0.11

Table 2. Conditioning of Test Specimens For Compression-Molded and Transfer-Molded Silicone

Condition Number	B-Stage (min at 93°C)	Preheat (s at 200 mA)	Maximum Preheat (Percent)	Spiral Flow (mm)
1	None	None	0	349
2	None	30	25	533
3	None	60	50	540
4	None	90	75	445
5	None	120	100	222
6	15	None	0	165
7	15	30	25	171
8	15	60	50	178
9	15	90	75	203
10	15	120	100	203

In the preheating operation, the molding compound was made into a pill, the pill was placed in a dielectric heater, and the heater was set at 200 mA. Maximum preheat time was determined as that time at which the pill could still be easily squeezed in the hand. For this silicone material, maximum preheat time was found to be 120 s. Arbitrarily-selected preheat times to be used for test specimens were 0, 25, 50, 75, and 100 percent of maximum.

Figure 1 shows spiral flow as a function of preheat. As the silicone was preheated, the non-B-staged material increased in spiral flow to a maximum of 540 mm at 50 percent of maximum preheat. Above 50 percent, the spiral flow decreased rapidly. The spiral flow of the B-staged material rose only slightly with an increase in preheat.

Tensile, flexural, and electrical specimens were compression-molded and transfer-molded using both B-staged and non-B-staged materials. Molding parameters are given in Appendix B. Test results are shown in Tables 3 and 4 and in Figures 2 through 6. Measurements were made on the diameter of the electrical discs. The measurements are shown in Table 3, but they were not plotted on a graph because the dimensional changes were small and were considered to be within the range of experimental error.

Figures 2 and 3, respectively, show that the flexural and tensile strengths of compression-molded specimens increased considerably with an increase in preheat. For the flexural strength (Figure 2), the non-B-staged silicone had higher values than the B-staged material at preheat times up to 50 percent of maximum. However, at maximum preheat the flexural strengths of the non-B-staged and the B-staged materials were approximately the same at 91.1 and 91.9 MPa, respectively. At maximum preheat, the flexural strength of non-B-staged, compression-molded material was 105 percent higher than at no preheat.

For the tensile strength (Figure 3), the non-B-staged material had lower values than the B-stage material at preheat times up to 50 percent of maximum. Again, at maximum preheat the tensile strengths of the non-B-staged and the B-staged compounds were very close to each other at 35.4 and 33.6 MPa respectively. The tensile strength of non-B-staged, compression-molded silicone at maximum preheat was 105 percent higher than at no preheat.

For both the B-staged and non-B-staged silicone, the dielectric strength of the compression-molded specimens (Figure 4), increased with an increase in preheat. The dielectric strengths were very close together up to 75 percent of maximum preheat. However, at maximum preheat the dielectric strength of the non-B-staged silicone was 14,567 V/mm while that of the B-staged specimens was only 12,244 V/mm. The dielectric strength of non-B-staged, compression-molded silicone molding compound was 80 percent higher at maximum preheat than at no preheat.

Figure 1. Spiral Flow Versus Preheat for Silicone Compound

Table 3. Properties of Compression-Molded Silicone Specimens

Condition Number	Flexural Strength (MPa)	Tensile Strength (MPa)	Dielectric Strength (V/mm)	Disc Diameter (mm)
1	44.5	17.3	8110	101.783
2	68.7	18.5	8386	101.838
3	88.6	25.3	12835	101.864
4	76.2	32.5	11850	101.887
5	91.1	35.4	14567	101.895
6	43.2	22.3	8543	101.788
7	59.4	24.4	9606	101.806
8	69.8	27.8	11693	101.874
9	92.7	32.3	11968	101.849
10	91.9	33.6	12244	101.869

Measurements were made of the diameter of the compression-molded electrical discs. These values, given in Table 3, show that neither B-staging nor preheat had an appreciable effect on the shrinkage of the compression-molded silicone compound.

As shown in Table 4, transfer-molded silicone specimens had poorer mechanical and electrical properties than the compression-molded specimens. In contrast to tests on the compression-molded specimens, the flexural and dielectric strengths of the transfer-molded specimens did not increase appreciably with an increase in preheat. At maximum preheat the flexural strength of non-B-staged material was 12 percent higher than it was at no preheat, and the dielectric strength was 16 percent lower.

Originally, it was thought that the electrical properties of the silicone compound were dependent on the resin component and that the dielectric strengths of compression-molded and transfer-molded specimens would be approximately the same. However, the graphs in Figures 4 and 6 show that non-B-staged, compression-molded silicone had a dielectric strength 30.7 percent higher than the transfer-molded material. It was not possible to transfer-mold a disc from B-staged material that had been given maximum preheat.

Table 4. Properties of Transfer-Molded Silicone Specimens

Condition Number	Flexural Strength (MPa)	Dielectric Strength (V/mm)	Disc Diameter (mm)
1	39.2	11142	100.940
2	39.6	10433	100.940
3	37.6	10630	100.914
4	39.2	10079	100.863
5	44.1	9331	100.990
6	37.1	9016	100.965
7	42.9	10866	100.914
8	39.2	9882	100.965
9	42.7	10118	100.889
10	40.1	*	*

*Could not make disc.

Tensile specimens could not be transfer-molded from the silicone compound. The method of test is ASTM-D-638, and the specimen is 216 mm long and 3.2 mm thick. The material would not flow well enough to make this part in the transfer mold.

The diameters of the transfer-molded electrical discs were measured and are given in the last column of Table 4. As with the compression-molded discs, B-staging and preheating the material had no effect on shrinkage. Differences seen in the diameters are believed to be within the limits of experimental error.

Diallyl Phthalate Molding Compound

Preheat and B-stage cycles for the diallyl phthalate molding compound are given in Table 5. Included is a column showing the spiral flow for each condition. Spiral flow of the as-received material was 472 mm. After a B-stage cycle of 30 minutes at 121°C, the spiral flow, with no preheat, was 236 mm.

Figure 2. Flexural Strength Versus Preheat for Compression-Molded Silicone

The diallyl phthalate material was preheated in the same manner as that described for the silicone. Maximum preheat was found to be 20 s at 200 mA. Again, preheat times were arbitrarily set at 0, 25, 50, 75, and 100 percent of maximum.

Figure 3. Tensile Strength Versus Preheat for Compression-Molded Silicone

Spiral flow as a function of preheat is shown in Figure 7. An increase in preheat caused a rapid increase in flow of the non-B-staged diallyl phthalate. The flow of the B-staged material rose only slightly with an increase in preheat. At maximum preheat the spiral flow of the non-B-staged and B-staged materials were not close together, as they were for the silicone.

Specimens for tensile, flexural, and dielectric strength were compression-molded and transfer-molded from both non-B-staged and B-staged materials. The molding parameters are shown in Appendix B.

Figure 4. Dielectric Strength Versus Preheat for Compression-Molded Silicone

Shrinkage information was obtained by measuring the diameter of the electrical discs. Test results are listed in Tables 6 and 7 and are plotted in Figures 8 through 13.

Test results on the compression-molded specimens, given in Table 6 and plotted in Figures 8 and 9, show that an increase to maximum preheat of non-B-staged material causes increases of 88 and 47 percent respectively in flexural and tensile strengths. The flexural strength of the non-B-staged diallyl phthalate was much higher than that of the B-staged material. However, the tensile strengths of the non-B-staged and B-staged compounds were very close to each other. This is believed to be caused by the way the glass fibers orient themselves in the flexural and tensile molds.

Figure 5. Flexural Strength Versus Preheat for Transfer-Molded Silicone

Dielectric strength of compression-molded specimens, shown in Table 6 and plotted in Figure 10, dropped 5 percent for the non-B-staged diallyl phthalate. However, for the B-staged material, the dielectric strength rose 24 percent from no preheat to maximum preheat. Measurements on the electrical discs show that neither B-staging nor preheating the material had any appreciable effect on shrinkage of the compression-molded material.

Figure 6. Dielectric Strength Versus Preheat for Transfer-Molded Silicone

Flexural, tensile, and dielectric strengths of the transfer-molded material are shown in Table 7. The diameter of each electrical disc is given in the last column. Graphs of the mechanical and electrical properties are given in Figures 11 through 13.

Figures 11 and 12 show that the flexural and tensile strengths of transfer-molded diallyl phthalate specimens are far below the corresponding compression-molded specimens, as shown in Figures 8 and 9. Preheating the material did cause some increase in the physical properties of transfer-molded specimens, but the increase was not as great as it was for the compression-molded specimens. The flexural and tensile strengths of non-B-staged material at maximum preheat were 4.6 and 29 percent higher than their respective values at no preheat.

Table 5. Conditioning of Test Specimens for Compression-Molded and Transfer-Molded Diallyl Phthalate

Condition Number	B-Stage (Min at 121°C)	Preheat (s at 200 mA)	Maximum Preheat (Percent)	Spiral Flow (mm)
11	None	None	0	472
12	None	5	25	467
13	None	10	50	533
14	None	15	75	602
15	None	20	100	561
16	30	None	0	236
17	30	5	25	224
18	30	10	50	251
19	30	15	75	269
20	30	20	100	244

Electrical specimens were transfer-molded from both non-B-staged and B-staged material. The results of dielectric strength testing are plotted in Figure 13. At maximum preheat the B-staged material had a dielectric strength about 1000 V/mm higher than that of the non-B-staged material. A comparison of Figures 10 and 13 shows that non-B-staged, compression-molded specimens had 46.5 percent higher dielectric strength than did the transfer-molded ones.

Measurements of the electrical discs are shown in the last column of Table 7. These results are very close together and are within the limits of experimental error. It is concluded that the B-staging and preheating operations had no effect on the shrinkage of the transfer-molded diallyl phthalate specimens.

ACCOMPLISHMENTS

A study was made to determine the effects of preheating the pre-forms and B-staging the raw materials prior to molding on two thermosetting molding compounds. The compounds investigated were a silicone and a diallyl phthalate. Both non-B-staged and B-staged materials were molded at 0, 25, 50, 75, and 100 percent of maximum

Figure 7. Spiral Flow Versus Preheat for Diallyl Phthalate Molding Compound

Table 6. Properties of Compression-Molded Diallyl Phthalate Specimens

Condition Number	Flexural Strength (MPa)	Tensile Strength (MPa)	Dielectric Strength (V/mm)	Disc Diameter (mm)
1	52.2	40.7	15630	101.930
2	48.5	52.1	15984	101.956
3	68.2	65.9	16614	101.981
4	98.4	67.8	15472	101.956
5	98.5	59.9	14803	101.981
6	51.7	46.4	14488	101.956
7	52.5	44.0	16181	101.905
8	52.1	60.9	16850	101.956
9	70.9	64.4	16654	101.956
10	79.7	70.9	18031	101.956

preheat time. Specimens were compression-molded and transfer-molded at each preheat condition.

An increase in preheat was accompanied by an increase in mechanical and electrical strengths of the compression-molded specimens for both the silicone and diallyl phthalate compounds. The compression-molded specimens had much higher mechanical and electrical strengths than did the transfer-molded specimens.

Preheating the preforms had little or no effect on transfer-molded specimens of either compound. This result may have been caused by forcing the material through a very small gate in the transfer-molding operation. The B-staging technique seemed to have very little effect on the strength of transfer-molded silicone specimens, but the B-staged material had poor flow. B-staging the diallyl phthalate material caused the dielectric strength of transfer-molded specimens to increase. The B-staged diallyl phthalate compound also had poor flow.

Table 7. Properties of Transfer-Molded Diallyl Phthalate Specimens

Condition Number	Flexural Strength (MPa)	Tensile Strength (MPa)	Dielectric Strength (V/mm)	Disc Diameter (mm)
11	64.5	33.8	11339	101.143
12	58.3	32.3	9882	100.965
13	40.2	35.0	7441	100.965
14	61.2	33.3	10748	101.016
15	67.5	43.7	10039	101.067
16	58.1	20.7	9213	101.219
17	55.8	18.1	9331	101.194
18	64.6	18.5	8307	101.067
19	65.8	41.6	12087	101.041
20	63.8	37.7	11260	101.092

Figure 8. Flexural Strength Versus Preheat for Compression-Molded Diallyl Phthalate

Figure 9. Tensile Strength Versus Preheat for Compression-Molded Diallyl Phthalate

Figure 10. Dielectric Strength Versus Preheat for Compression-Molded Diallyl Phthalate

Figure 11. Flexural Strength Versus Preheat for Transfer-Molded Diallyl Phthalate

Figure 12. Tensile Strength Versus Preheat for Transfer-Molded Diallyl Phthalate

Figure 13. Dielectric Strength Versus Preheat for Transfer-Molded Diallyl Phthalate

Appendix A

SPIRAL FLOW TEST METHOD

This test shall be run in the transfer-mold shown in Figure A-1. The procedure shall be as follows.

1. Make at least 5 runs.
2. Use the mold temperature designated for each material.

Silicone: $177 \pm 5^{\circ}\text{C}$

Diallyl phthalate: $149 \pm 5^{\circ}\text{C}$

3. Pressure on material: 22.1 MPa.
4. Cull Thickness: 3.2 ± 0.8 mm

(Start with a 25-g preform and adjust the weight until the 3.2-mm cull is obtained).

5. Do not preheat the preform.

Place the material in the preform mold; the material and preform shall be at a temperature of $24 \pm 3^{\circ}\text{C}$. Place the preform mold in a press. Temperature of the press platens shall be $24 \pm 3^{\circ}\text{C}$. Compress the material to 68.9 ± 1 MPa. Remove the preform.

Place the spiral flow mold in a transfer press and allow the press and mold to stabilize at the given temperature. Place the 24°C preform in the transfer pot, bring the ram down, and quickly bring the transfer pressure to 22.1 MPa. Hold the pressure until the material has cured.

Figure A-1. Mold for Spiral Flow Test

Appendix B

MOLDING PROCEDURES

SILICONE MOLDING COMPOUND

Compression Molding

Temperature: 163°C
Pressure: 34.5 MPa
Time: 25 minutes

Transfer Molding

Temperature: 149°C
Pressure: 43.4 MPa
Time: 20 minutes

DIALLYL PHTHALATE

Compression Molding

Temperature: 149°C
Pressure: 44.1 MPa
Time: 15 minutes

Transfer Molding

Temperature: 149°C
Pressure: 58.4 MPa
Time: 15 minutes

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SPECIAL PROJECTS: Molding Compounds

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