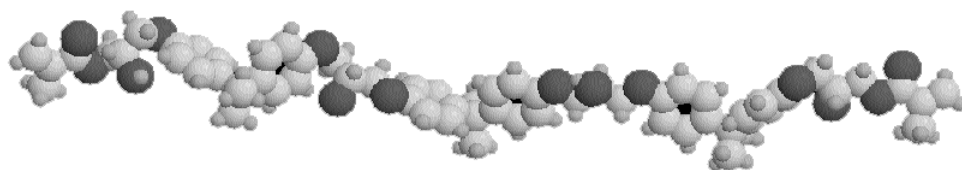

Technical Research



CoREZYN Premium Vinyl Ester Molecule

Proper Cure of Vinyl Ester Resins

INTERPLASTIC CORPORATION
Thermoset Resins Division

ABSTRACT

Complete cure of a resin is critical to achieving the optimum corrosion resistance and physical properties of a laminate. Typical composites are made in thicknesses ranging from 0.005 in. (0.13 mm) to 0.400 in. (10.2 mm). Previous work has shown that higher catalyst levels yield a higher degree of cure for thin panels made at 77°F (25°C). However, fiberglass reinforced plastic/glass reinforced plastic (FRP/GRP) products are made under a variety of temperatures and cure conditions. This study was done to help FRP/GRP manufacturers address parameters that they can control to make quality parts under a variety of conditions.

Three parameters were evaluated in a designed experiment. These parameters are critical to making a quality part in hand lay-up, spray-up and other processes. Two of the controllable parameters are catalyst and promoter levels. The third parameter, temperature, is independent of the other two. This study shows how the FRP/GRP manufacturer can design a promoter/catalyst package to yield quality parts even under adverse temperatures and ambient conditions.

INTRODUCTION

Most FRP/GRP fabricators using vinyl ester resin in their construction process manufacture laminates less than 0.10 in. (2.5mm) thick. Applications for these thin laminates range from corrosion barriers in tanks, pipe, and ducting, to skin coats used in swimming pools, boats, spas, and in sandwich composites. The performance of the composite is dependent upon the cure of the resin. Resin manufacturers offer many of these resins promoted, however many fiberglass shops prefer to design their own catalyst promoter systems. Fabricators commonly increase the level of catalyst to improve the cure. Understanding these systems can help fabricators select a combination of catalyst and promoters that are right for their manufacturing conditions.

A significant amount of fabrication is done at temperatures below 77°F (25°C). This is especially true in the spring, fall and winter seasons. Low temperatures are detrimental to the final cure of resins and final cure is especially critical in thin laminates. Air, resin and mold temperature influence resin cure. Mold temperature is the most important factor, since a cool surface can quickly lower the temperature of the resin and lengthens

the gel time.

While growth in thin laminate manufacturing continues to increase, there have not been any recent studies exploring how to optimize the cure development. Thin cross sections do not generate exotherms of more than 104°F (40°C). Previous work with thick laminates showed exotherms ranging from 99°F (37°C) to 334°F (168°C). High exotherms have been shown to yield higher Tg's.¹ Since thin laminates do not reach the same temperatures as their thick counterparts, they require an optimized catalyst promoter package to generate thoroughly cured composites. Catalyst/promoter packages are combinations of additives that have a synergistic relationship in the crosslinking reaction. The optimum levels of these components can help FRP/GRP manufacturers bring the best possible products to market.

Another method used to increase the resin's crosslink density is to post-cure the parts. Typically fiberglass shops don't post-cure parts unless they are specifically requested or required to because it creates an additional step and cost. While today's competitive market has driven companies to be cost conscious, they still need a means to generate a thoroughly cured composite.

There are instances where composites are transferred to storage areas at temperatures below 50°F (10°C) or put in service before they are three days old. Shifting composites to cooler environments can slow and even stop the reaction from reaching the optimum level of cure. Post-curing the parts at this point may not increase the hardness, degree of cure and glass transition.

Our study explored the catalyst and promoter selection and their levels at various temperatures. Typical levels of the following common catalyst promoter systems will be discussed: methyl ethyl ketone peroxide (MEKP)/cobalt octoate/N, N-dimethylaniline (DMA) and benzoyl peroxide (BPO)/DMA. Temperatures of 59°F (15°C), 68°F (20°C) and 77°F (25°C) were studied.²

Designed experiments, optimizing a few parameters, were set up similar to previous work.³ These experiments were used to investigate the effects of the catalyst/promoter packages over a range of normal operating temperatures. Models of data were used to predict the levels of the catalyst and promoters required to yield a highly cured composite at various temperatures.

Three techniques were used to evaluate the resin cure:

1. Glass transition temperature determined by dynamic mechanical analysis,^{4,5}

2. degree of cure by differential scanning calorimetry (DSC)⁶ and,
3. laminate hardness by Barber-Coleman Barcol impressor gauge.⁴

Static physical properties of the laminates were also checked to find if the catalyst/promoter package and the temperature affected them.

EXPERIMENTAL

The resin was a non-promoted, bisphenol-A, epichlorohydrin-based, vinyl ester with a viscosity of 450 centipoise at 45% styrene. Appropriate levels of the promoters were added to the resin for each experiment. The promoters used were: 12% cobalt octoate and N,N-Dimethylaniline (DMA). The methyl ethyl ketone peroxide (MEKP) catalyst used was L-50 (Akzo Chemie.) and the benzoyl peroxide (BPO) was ANS-55 (Arkema, Inc.). L-50 was chosen because it is an MEKP catalyst specifically designed to work well with vinyl ester resins. ANS-55 was selected because it does not contain high levels of water or plasticizers as diluents that can influence the physical and corrosion resistance properties of the composite.

The experiments were a custom central composite design. One design included eleven experiments, the eight corners of the cube and three replications of the center point. The other design was used for the physical properties in which seven experiments were used and incorporated four corners and three center points. The models of the data were computed without any transformation of the values. Quadratic and cubic equations were used for the models because they provided the best fit for the data. The experiments were set up and analyzed with a program entitled Design Expert by Stat-Ease.[®]

A flat, rectangular, aluminum mold 1 in. (2.5 cm) deep by approximately 47 in.² (305 cm²) was used to construct the laminates. Thirty minutes before laminating, the mold was equilibrated in a Lindbergh/Blue M model 1120 water bath and then suspended 0.5 in. (1.25 cm) deep in the water bath. It was removed from the bath immediately before the laminate was rolled out. A one-mil-thick cellophane film was placed on the mold's surface before each test and before the laminate was rolled out.

The laminates were constructed of three plies of 1.5-oz/ft² (460 grams/m²) Owens Corning M-723 fiberglass mat. A ratio of one part fiberglass to two parts resin was used. The resin was promoted,

adjusted to the designated temperature, catalyzed and then the laminate was rolled out. After that, the mold was immediately returned to the water bath. A duplicate laminate for each experiment was constructed - one for the physical properties (T_g and degree of cure), the other for monitoring the hardness development.

The laminates were removed from the mold four hours after the resin was catalyzed. They were aged for an additional 44 hours at an ambient temperature of 77°F ±4°F (25°C ±1.6°C). After the initial period, placing the laminates in a freezer at 14°F (-10°C) quenched the reaction. The laminates were stored in the freezer for a minimum of seven days and then removed from the freezer and conditioned at 77°F (25°C) for 24 hours before testing. During the 24-hour conditioning period, the laminates were cut and routed into test specimens.

The laminates used for the hardness measurements were removed from the water bath for approximately one minute for each reading. Hardnesses were measured with Barber-Coleman GYZJ 934-1 and GYZJ 935 Barcol Impressor gauges, according to ASTM D-2583, starting 1.5 hours after the catalyst was added and monitored for the next 2.5 hours. After each reading was taken, the mold was immediately suspended in the water bath again.

Static flexural and tensile physical properties of the laminates were tested according to ASTM D-790 and ASTM D-638 on an Instron Model 4505 Universal Tester.

Differential scanning calorimetry was run on a Mettler TA20 unit. The reference cell contained a specimen of the laminate that was in the same weight range as the samples. Using the reference specimen made it possible to negate the absorption of energy by the fiberglass in the sample cell. Multiple scans of 77°F (25°C) to 437°F (225°C) were run on the reference sample to ensure that it did not show an exothermic peak before using it as the reference cell.

Samples were taken from the center of the laminate, their weights were each 70 mg ±3 mg. They were scanned from 77°F (25°C) to 437°F (225°C) at a rate of 18°F (10°C) per minute. The maximum energy released by the liquid resin during curing was determined and then used to calculate the degree of cure. A sample of the liquid resin was catalyzed with 1% tertiary-butyl perbenzoate (TBPB). Three samples of the catalyzed resin were weighed out at approximately 5 mg into aluminum crucibles. They were hermetically sealed. These samples were scanned with the same temperature program used to find the maximum energy released by the liquid resin.

The dynamic mechanical analysis was run on Rheometric's RASII solids analyzer to find the glass transition temperature of the cured resin. Samples cut from the center of the laminate and were 2.05 in.±0.02 in. long x 0.50 in. ±0.02 in. wide x 0.10 in. ±0.016 in. thick (52 mm ±0.5 mm long x 12.5 mm ±0.5 mm wide x 2.5 mm ±0.4 mm thick). The air sides of the laminate were sanded flat with 240-grit emery paper. The mold side surface required no surface preparation. The specimens were scanned from 86°F (30°C) to 392°F (200°C), at 3.6°F (2°C) intervals, with a one-minute soak at each temperature. The pre-stress load was 300g and the strain was 2.7×10^{-4} .

RESULTS

Data generated on the MEKP system is compiled in Tables 1 and 3. It generated excellent models when analyzed with the design experimentation program (Table 2). Models of Tg, degree of cure and hardness development for the MEKP/cobalt system had regression coefficients over 0.97. Analysis of the static physical properties, listed in Table 4, yielded models with respectable regression coefficients. Several interesting trends were seen and should provide a better insight into the catalyst/promoter system.

A typical dynamic mechanical analysis scan is shown in Figure 1. They all had a major peak, which is the Tg reported, and a minor peak at a lower temperature. The peak at the lower temperature merges with the major peak if the sample is post-cured. Tg was found dependent on the level of DMA, but not on the temperature or catalyst level (Figure 2). Tg increased linearly at 0.9°F (0.5°C) per additional 0.015% DMA. Analysis of the data showed 0.12% DMA yielded a minimum Tg of 244°F (118°C).

All three variables affected the ultimate hardness of the laminate (Figure 3). As the MEKP was increased from 0.75% to 1.6%, the hardness increased six points. It reached a maximum at 1.6% MEKP. The hardness decreased linearly one point per additional 0.015% DMA.

Temperature caused the hardness to drop one point as it was increased from 59°F to 77°F (15°C to 25°C). Figures 4, 5, and 6 show the model at 77°F (25°C), 68°F (20°C), and 59°F (15°C) respectively. Composites made with this resin having a Barber-Coleman 934 gauge Barcol hardness of 30 are considered by the resin manufacturers to be satisfactorily cured. A minimum Barcol hardness of 30 can be achieved over the range of temperatures

studied. The levels required for MEKP are 0.95% to 1.75% and DMA are 0.05% to 0.12%.

A combination of low levels of MEKP and DMA resulted in incomplete polymerization of the vinyl ester resin and yielded low hardnesses. This could be due to a low level of free radicals formed during the induction period and curing process.

High levels of the catalyst and DMA also had lower hardnesses. This phenomenon can also be caused by the over catalyzation and promotion of the resin. The combination of these high levels in the resin can accelerate the formation of free radicals. A high concentration of free radicals can terminate the cross-linking reaction as free radicals from the MEKP, polymer and styrene react with one another instead of the unsaturation in the styrene and the polymer. These reactions can cause the formation of short polymer chains, compared to the preferred polymerization reaction, which forms long intertwined chains. Hardness development for several experiments is shown in Table 3.

The degree of cure determined by DSC was based on 116.5 BTU/lb (271 J/g). Testing was done on the liquid resin to ascertain this value. A glass content of approximately 33% in the laminate was considered in these calculations. Using a reference cell containing a fully cured portion of the laminate automatically subtracts the contribution of the fiberglass and mass to the heat flow. This isolates the residual energy released from the crosslinking reaction. It also creates a more stable baseline by eliminating disturbances such as glass transitions and other shifts. A typical scan is shown in Figure 7.

DMA and temperature have almost no affect on the degree of cure. MEKP did affect the degree of cure (Figure 8). The degree of cure increased 2.5 points from 0.75% MEKP to 1.4% MEKP and dropped one point as the level of catalyst was raised to 1.75%. Based on this data, the optimum range of MEKP is 1% to 1.5%.

The static physical properties of the laminates (Table 4) generated moderately good models (Table 2). Catalyst levels between 1.2% and 1.6% yielded maximum flexural modulus, tensile strength, and tensile modulus. Flexural strength decreased at a rate of 145 ksi (1 kPa) per 0.25% MEKP. DMA and temperature had little affect on the flexural strength. DMA and temperature did affect the physical properties, but not to the extent as catalyst did. There was not an optimum range of DMA and temperature to yield maximum physical properties.

The data (Table 5) produced excellent models (Table 6) for the Tg, degree of cure and hardness develop-

ment. The static physical properties (Table 8) provide models that had some poor regression coefficients (Table 6).

A typical dynamic mechanical analysis scan is the same as the one shown for the MEKP system in Figure 1. The model of the T_g data in Figure 9 was a perfect fit as seen in the correlation coefficient of 1.000. When the DMA level and temperature were raised, the T_g increased. T_g increased by 1.8°F (1°C) for every 0.28% increase in DMA and 4.9°F (2.7°C) in temperature. BPO had little affect on the T_g from 1.0 to 1.2%. Levels above 1.2% caused the T_g to decrease approximately 1.8°F (1°C) per 0.10% of catalyst. The model (Figures 10, 11, 12) shows the synergistic effect of BPO and DMA on T_g. The levels of catalyst and promoter required to maintain a T_g above 246°F (119°C) can become small as the temperature is decreased.

The degree of cure for the BPO/DMA system was also based on the 271 J/g reference point and a glass content of approximately 33%. DMA and temperature caused the degree of cure to increase in a linear fashion. The cure increased 1% for each 0.05% increase in DMA and 18°F (10°C). BPO had the greatest affect on the extent of cure between 1% and 1.5%. The cure reached a plateau at 1.5% and actually dropped slightly (Figure 13) at higher levels.

Figures 14, 15 and 16 illustrate the relationship between the degree of cure, BPO level and DMA concentration at three different temperatures. These models reveal that attaining a high degree of cure is dependent on the catalyst level at low temperatures. As the temperature increased, the degree of cure becomes more dependent on the DMA and less on the catalyst.

Analysis of the final hardnesses (Table 5) generated an excellent model (Figure 17). All three variables increased the hardness linearly. DMA had the most affect by increasing the Barber-Coleman 934 gauge hardness one point for every 0.0225% increase. Increasing the temperature 3.8°F (2°C) raised the Barber-Coleman 934 gauge hardness one point. BPO raised the hardness one point for every 0.4% increase in the catalyst. Figures 18, 19 and 20 show the dependence of hardness on the BPO and DMA at three different temperatures. The hardness was totally dependent on the DMA level at 77°F (25°C) and required a certain ratio of BPO and DMA to achieve Barcol hardnesses over 22. Hardness development of several laminates is shown in Table 8.

The static physical properties of the laminates (Table 8) generated moderately good models

(Table 6). Catalyst levels between 1.4% and 1.8% yielded maximum flexural modulus, tensile strength, and tensile modulus. Flexural strength increased at a rate of 145 ksi (1 kPa) per 0.25% BPO. DMA and temperature caused the flexural strength to drop 145 ksi (1 kPa) as they were increased at 0.10% and 3.8°F (2°C) respectively. DMA and temperature did affect the physical properties, but not to the extent as the catalyst. There was not an optimum range of DMA and temperature to yield maximum physical properties.

The MEKP/cobalt system was more forgiving to variation in the levels of catalyst and promoter to get the same cure measurements. A T_g of 244°F (118°C), degree of cure of 85%, and Barcol hardness of 20 were chosen as minimums for optimization. These show the BPO/DMA system had a much smaller working window (Figure 21) than the MEKP/cobalt system (Figure 22) to achieve the minimum values at 59°F (15°C). Areas where these minimal values were all reached increased in size as the temperature was raised for BPO. The MEKP optimization maintained similar levels over the three temperatures tested.

CONCLUSIONS

This work is a study of a single moment in the life of an FRP/GRP laminate. We tried to pick an appropriate point that relates to actual occurrences in fiberglass shops.

A few conclusions have been drawn from the data.

1. MEKP/cobalt systems that had a high T_g, degree of cure and hardness, and desired physical properties can be achieved with an MEKP level ranging from 1.25% to 1.60% and a DMA level of 0.085% to 0.15%. This appeared to be true over the whole range of temperatures evaluated.
2. Optimization of the BPO/DMA levels to yield high T_g, degree of cure and hardness, as well as good physical properties are BPO 1.0 to 1.6% and DMA 0.15 to 0.30%. Lower temperatures require the maximum levels of BPO and DMA.
3. BPO/DMA yielded lower T_g's then the MEKP/cobalt system.
4. BPO/DMA reached a higher overall degree of cure then the MEKP/cobalt system.
5. MEKP/cobalt systems had higher ultimate Barcol hardness then the BPO/DMA systems.
6. BPO/DMA reached a higher degree of cure at

lower temperatures than the MEKP/cobalt system however, it also reached lower Tg's and hardnesses.

7. The physical properties of the laminates were slightly affected by the catalyst/promoter system, but the highest properties were seen at the higher degree of cures.
8. Barcol hardness can only be used as a crude tool to monitor the extent of cure of a laminate. Low Barcol hardness does not necessarily mean poor cure of the resin.
9. BPO/DMA systems had a faster initial hardness development with lower ultimate hardnesses. Faster initial hardness development has lead some fabricators to use BPO/DMA.
10. The working levels of active BPO for laminates 0.10 in. (2.5 mm) thick and temperatures from 59°F to 77°F (15°C to 25°C) range from 1% to 1.5%. Higher levels do not appear to provide any benefit to the properties of the composite.
11. Hardness development and gel time data of the different systems are in Tables 7 and 8. The BPO/DMA system had faster initial hardness development with comparable cup gel times and peak exotherms.

BIBLIOGRAPHY

1. Grentzer, T.H., D.A. Rust, S.K. Lo, C.J. Spencer, and G.W. Hackworth. *Proceedings of the 46th Annual Technical Conference for the Composites Institute*. SPI, Section 1-B, 1990.
2. Cassoni, J.P., G.A. Harpell, P.C. Wang, and A.H. Zupa. *Proceedings of the 32nd Annual Technical Conference for the Composites Institute*. SPI, Section 3-E, 1977.
3. Herzog, D., P. Burrell, and M. Teigen. *Proceedings of the 44th Annual Technical Conference for the Composites Institute*. SPI, Section 16-C, 1989.
4. Grentzer, T.H., K.E. Kitchen, S.K. Lo, C.J. Spencer, and D.A. Rust. *Proceedings of the 46th Annual Technical Conference for the Composites Institute*. SPI, Section 2-B, 1990.
5. Gillham, J.K. *Polymer Engineering and Science*. 15:371, 1979.
6. Tollens, F.R., R. Hill, and L.J. Lee. *Proceedings of the 47th Annual Technical Conference for the Composites Institute*. SPI, Section 17-B, 1992.

DATA

Table 1: Measurement of Cure - MEKP

| MEKP LEVEL | N,N-DMA LEVEL | TEMP °F/°C | Tg °F/°C | DEGREE OF CURE | HARDNESS 934-1 |
|------------|---------------|------------|----------|----------------|----------------|
| 0.75 | 0.05 | 59/15 | 239/115 | 85.4 | 30 |
| 1.75 | 0.05 | 59/15 | 239/115 | 83.9 | 37.5 |
| 0.75 | 0.15 | 59/15 | 254/123 | 80.8 | 22.5 |
| 1.75 | 0.15 | 59/15 | 254/123 | 86.5 | 34 |
| 0.75 | 0.05 | 77/25 | 239/115 | 82.9 | 22.5 |
| 1.75 | 0.05 | 77/25 | 243/117 | 85.3 | 40.5 |
| 0.75 | 0.15 | 77/25 | 254/123 | 85.3 | 34 |
| 1.75 | 0.15 | 77/25 | 250/121 | 84.9 | 22.5 |
| 1.25 | 0.1 | 68/20 | 247/120 | 85.9 | 32.5 |
| 1.25 | 0.1 | 68/20 | 246/119 | 86.0 | 32.5 |
| 1.25 | 0.1 | 68/20 | 246/119 | 86.3 | 32.5 |

Table 2: Design Experimentation Analysis Models - MEKP

| VARIABLE | TYPE OF TRANSFORMATION | REGRESSION COEFFICIENT | COEFFICIENT OF VARIANCE, % |
|------------------------------|------------------------|------------------------|----------------------------|
| GLASS TRANSITION | NONE | 0.9782 | 0.74 |
| DEGREE OF CURE | NONE | 0.9970 | 0.25 |
| HARDNESS, ASTM D2548 | NONE | 1.0000 | 0.00 |
| FLEXURAL STRENGTH, ASTM D790 | NONE | 0.7291 | 4.51 |
| FLEXURAL MODULUS, ASTM D790 | NONE | 0.9178 | 3.56 |
| TENSILE STRENGTH, ASTM D638 | NONE | 0.9116 | 1.73 |
| TENSILE MODULUS, ASTM D638 | NONE | 0.9467 | 2.94 |
| ELONGATION, ASTM D638 | NONE | 0.8509 | 15.21 |

Table 3: Gel, Cure & Hardness Measurements - MEKP

| | | | | | | | |
|---|---------|---------|---------|---------|---------|---------|---------|
| TEMPERATURE °F/°C | 59/15 | 59/15 | 77/25 | 77/25 | 68/20 | 68/20 | 68/20 |
| MEKP, WT % | 0.75 | 1.75 | 0.75 | 1.75 | 1.25 | 1.25 | 1.25 |
| N,N-DMA, WT % | 0.15 | 0.05 | 0.05 | 0.15 | 0.10 | 0.10 | 0.10 |
| GEL TIME, MINUTES¹ | 21:00 | 23:30 | 12:30 | 9:30 | 12:15 | 12:20 | 12:15 |
| CURE TIME, MINUTES¹ | 35:00 | 38:30 | 25:30 | 18:30 | 23:35 | 23:40 | 23:20 |
| PEAK EXOTHERM, °F/°C¹ | 318/159 | 333/167 | 335/168 | 358/181 | 334/168 | 334/168 | 345/174 |
| 1.5 HOURS² | 35-45 | -- | 35-45 | 50-65 | 20-40 | 30-45 | 25-40 |
| 1.75 HOURS² | 35-45 | -- | 40-60 | 50-65 | 30-40 | 40-50 | 35-45 |
| 2 HOURS² | 40-50 | 1-10 | 50-60 | 50-70 | 40-55 | 60-70 | 45-60 |
| 2.25 HOURS² | 50-60 | 10-25 | 50-65 | 65-75 | 40-55 | 60-70 | 50-65 |
| 4 HOURS² | 50-60 | 55-65 | 70-80 | 15-85 | 70-85 | 75-90 | 75-85 |

1. Run on a 100-gram mass.

2. Hardness measured with a Barber-Colman GYZJ 935 Barcol Impressor gauge.

Table 4: Static Physical Properties of Laminates - MEKP

| | | | | | | | |
|------------------------------|-------|-------|-------|-------|-------|-------|-------|
| MEKP LEVEL | 1.75 | 0.75 | 0.75 | 1.75 | 1.25 | 1.25 | 1.25 |
| N,N-DMA LEVEL | 0.05 | 0.15 | 0.05 | 0.15 | 0.10 | 0.10 | 0.10 |
| TEMP. °F/°C | 59/15 | 59/15 | 77/25 | 77/25 | 68/20 | 68/20 | 68/20 |
| FLEXURAL STRENGTH KPa | 174.5 | 154.5 | 154.5 | 164.8 | 166.9 | 160.7 | 152.7 |
| FLEXURAL MODULUS KPa | 6814 | 5903 | 6414 | 6897 | 7241 | 7310 | 6862 |
| TENSILE STRENGTH KPa | 82.76 | 85.52 | 77.24 | 82.07 | 85.52 | 82.76 | 83.45 |
| TENSILE MODULUS KPa | 7931 | 7862 | 6621 | 6897 | 8069 | 7655 | 7724 |
| ELONGATION % | 1.2 | 1.3 | 1.4 | 1.3 | 1.3 | 1.3 | 1.2 |
| HARDNESS 934-1 | 37.5 | 22.5 | 22.5 | 22.5 | 32.5 | 32.5 | 32.5 |
| GLASS % | 34.6 | 33.5 | 32.5 | 34.9 | 33.7 | 33.1 | 34.2 |

Table 5: Measurement of Cure - BPO

| BPO LEVEL | N,N-DMA LEVEL | TEMP. °F/°C | Tg °F/°C | DEGREE OF CURE | HARDNESS 934-1 |
|------------------|----------------------|--------------------|-----------------|-----------------------|-----------------------|
| 1.0 | 0.075 | 59/15 | 243/117 | 86.1 | 26 |
| 2.0 | 0.075 | 59/15 | 203/95 | 92.2 | 10 |
| 1.0 | 0.30 | 59/15 | 246/119 | 80.1 | 10 |
| 2.0 | 0.30 | 59/15 | 239/115 | 96.3 | 37.5 |
| 1.0 | 0.075 | 77/25 | 239/115 | 87.1 | 17.5 |
| 2.0 | 0.075 | 77/25 | 232/111 | 82.3 | 22 |
| 1.0 | 0.30 | 77/25 | 253/123 | 96.3 | 36 |
| 2.0 | 0.30 | 77/25 | 239/115 | 93.6 | 32.5 |
| 1.5 | 0.15 | 68/20 | 239/115 | 91.1 | 22.5 |
| 1.5 | 0.15 | 68/20 | 239/115 | 89.5 | 22.5 |
| 1.5 | 0.15 | 68/20 | 239/115 | 90.5 | 22.5 |

Table 6: Design Experimentation Analysis Models - BPO

| VARIABLE | TYPE OF TRANSFORMATION | REGRESSION COEFFICIENT | COEFFICIENT OF VARIANCE, % |
|--------------------------------|------------------------|------------------------|----------------------------|
| GLASS TRANSITION | NONE | 1.0000 | 0.06 |
| DEGREE OF CURE | NONE | 0.9953 | 0.90 |
| HARDNESS ASTM D2548 | NONE | 0.9613 | 14.18 |
| FLEXURAL STRENGTH ASTM D790 | NONE | 0.6582 | 3.82 |
| FLEXURAL MODULUS ASTM D790 | NONE | 0.8616 | 5.58 |
| TENSILE STRENGTH ASTM D638 | NONE | 0.4206 | 6.07 |
| TENSILE MODULUS ASTM D638 | NONE | 0.5746 | 10.34 |
| ELONGATION ASTM D638 | NONE | 0.7667 | 4.39 |

Table 7: Gel, Cure & Hardness Measurements - BPO

| | | | | | | | |
|--|---------|---------|---------|---------|---------|---------|---------|
| TEMP. °F/°C | 59/15 | 59/15 | 77/25 | 77/25 | 68/20 | 68/20 | 68/20 |
| BPO, WT % | 1.0 | 2.0 | 1.0 | 2.0 | 1.5 | 1.5 | 1.5 |
| N,N-DMA, WT % | 0.30 | 0.075 | 0.075 | 0.30 | 0.15 | 0.15 | 0.15 |
| GEL TIME MINUTES¹ | 20:00 | 32:00 | 32:00 | 7:20 | 17:25 | 17:20 | 17:10 |
| CURE TIME MINUTES¹ | 33:00 | 54:40 | 50:15 | 15:20 | 30:35 | 29:50 | 30:30 |
| PEAK EXOTHERM °F/°C¹ | 311/155 | 313/156 | 340/171 | 342/172 | 343/173 | 336/169 | 338/170 |
| 1.5 HOURS² | 50-60 | -- | 30-50 | 70-80 | 50-70 | 50-70 | 45-60 |
| 1.75 HOURS² | 50-60 | -- | 50-60 | 70-85 | 65-75 | 60-75 | 55-75 |
| 2 HOURS² | 50-60 | 15-25 | 50-60 | 75-85 | 70-80 | 65-80 | 70-80 |
| 2.25 HOURS² | 50-60 | 30-40 | 50-65 | 75-85 | 70-80 | 65-80 | 70-80 |
| 4 HOURS² | 55-65 | 40-50 | 50-65 | 75-85 | 75-85 | 75-90 | 75-85 |

1. Run on a 100-gram mass.

2. Hardness measured with a Barber-Colman GYZJ 935 Barcol Impressor gauge.

Table 8: Static Physical Properties of Laminates - BPO

| | | | | | | | |
|------------------------------|-------|-------|-------|-------|-------|-------|--------|
| BPO LEVEL | 2.0 | 1.0 | 1.0 | 2.0 | 1.5 | 1.5 | 1.5 |
| N,N-DMA LEVEL | 0.075 | 0.30 | 0.075 | 0.30 | 0.15 | 0.15 | 0.15 |
| TEMP. °F/°C | 59/15 | 59/15 | 77/25 | 77/25 | 68/20 | 68/20 | 68/20 |
| FLEXURAL STRENGTH KPa | 143.4 | 149.7 | 159.3 | 160.7 | 146.2 | 160.0 | 155.6 |
| FLEXURAL MODULUS KPa | 5841 | 6435 | 6517 | 7172 | 6966 | 7634 | 7655 |
| TENSILE STRENGTH KPa | 86.90 | 87.14 | 79.31 | 81.38 | 82.76 | 80.69 | 110.34 |
| TENSILE MODULUS KPa | 7724 | 7586 | 6897 | 8000 | 7931 | 7862 | 9310 |
| ELONGATION % | 1.4 | 1.3 | 1.3 | 1.2 | 1.3 | 1.3 | 1.4 |
| HARDNESS 934-1 | 10.0 | 10.0 | 17.5 | 32.5 | 22.5 | 22.5 | 27.5 |
| GLASS % | 34.6 | 33.5 | 32.5 | 34.9 | 33.7 | 33.1 | 34.2 |

Figure 1: Typical Dynamic Mechanical Analysis Scan

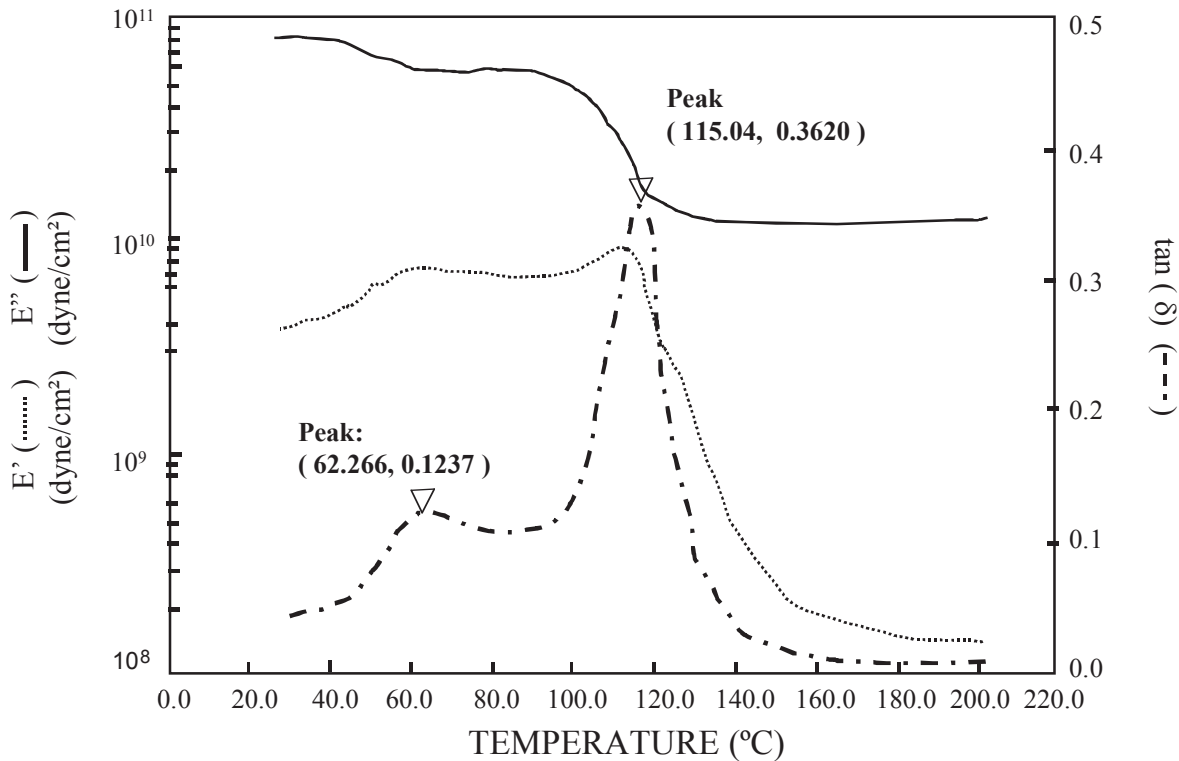


FIGURE 2
RESPONSE Tg

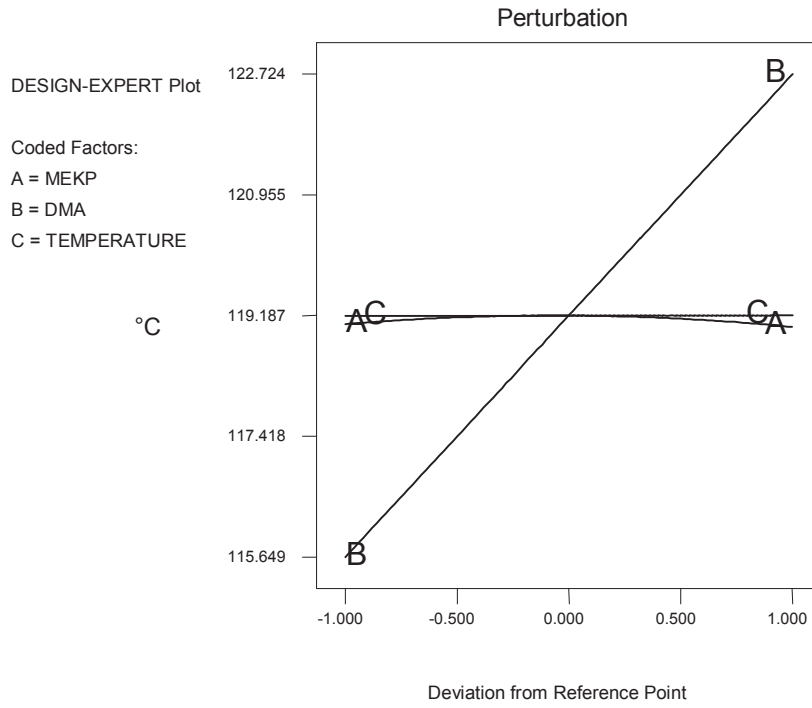


FIGURE 3
Response: Hardness

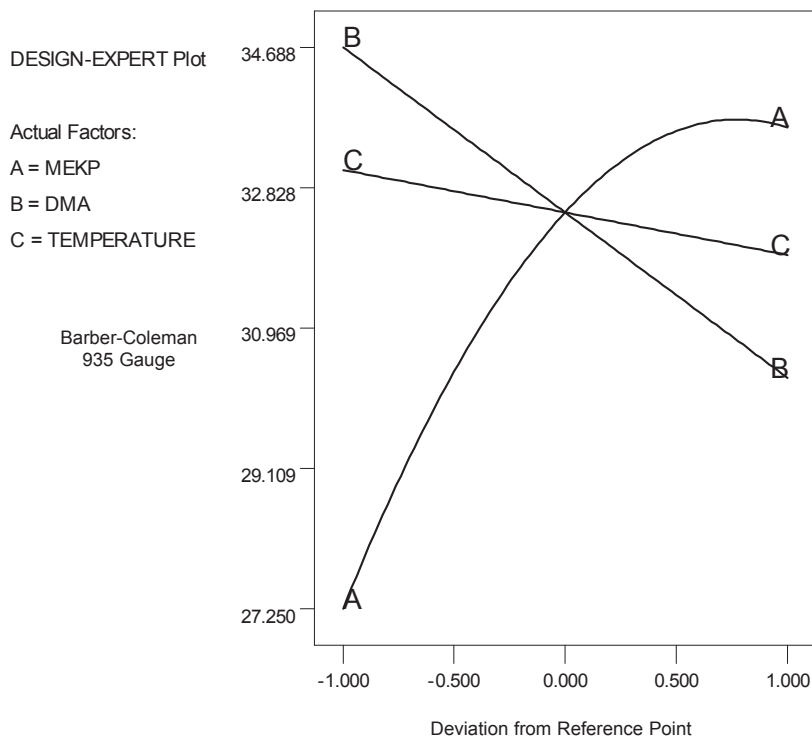


FIGURE 4
Response: Hardness

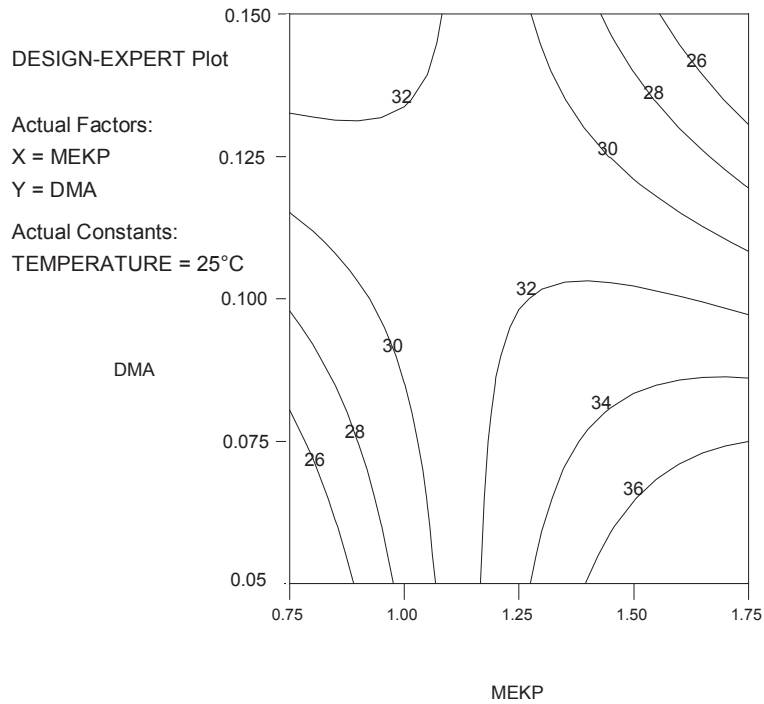


FIGURE 5
Response: Hardness

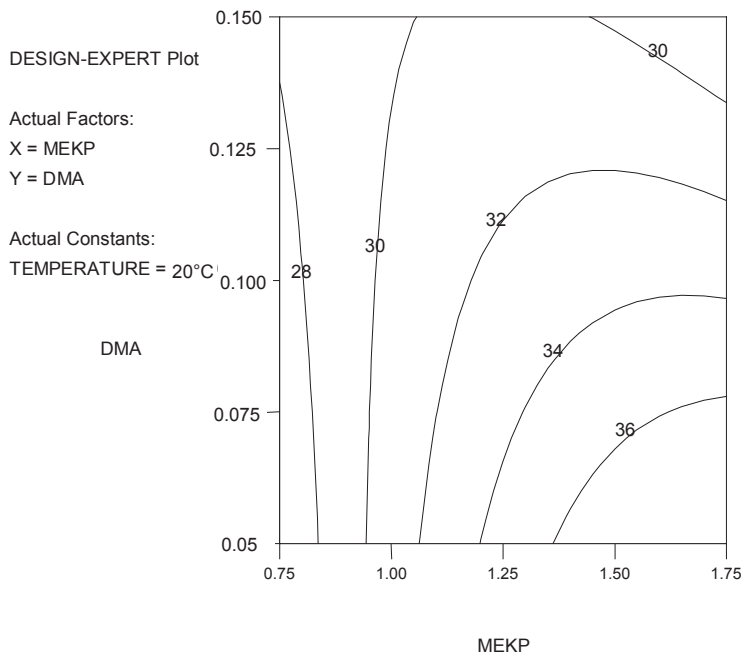


FIGURE 6
Response: Hardness

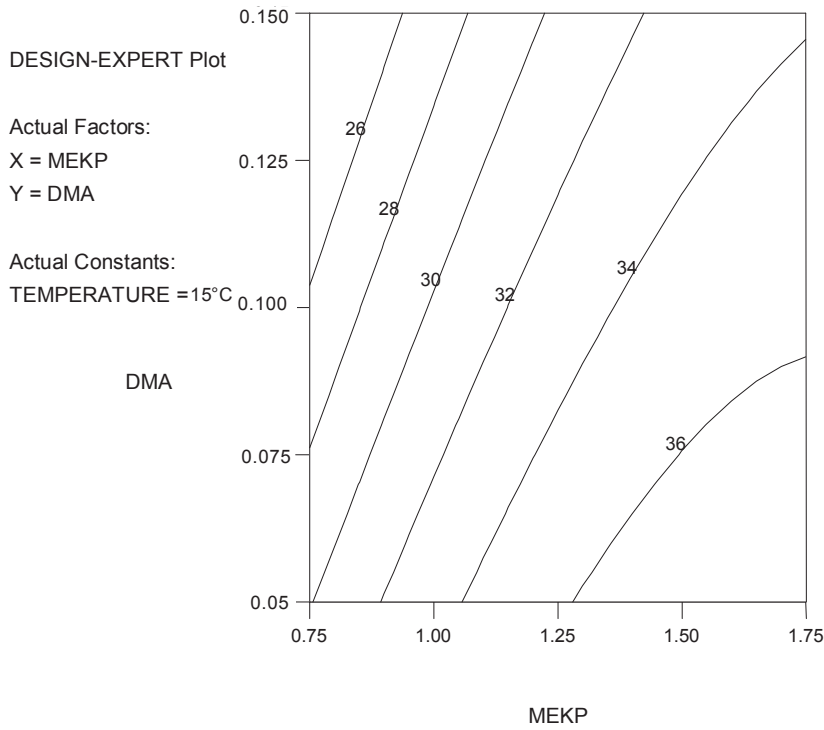


FIGURE 7: Typical Differential Calorimetry

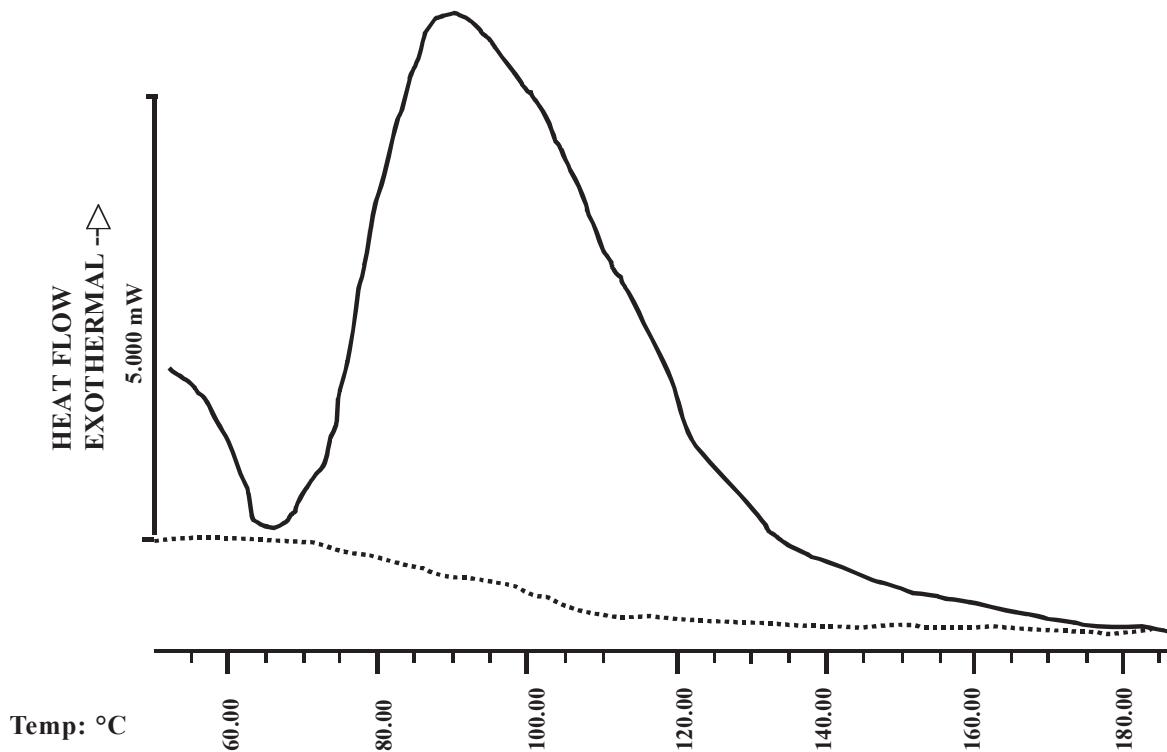


FIGURE 8
Response: Degree of Cure

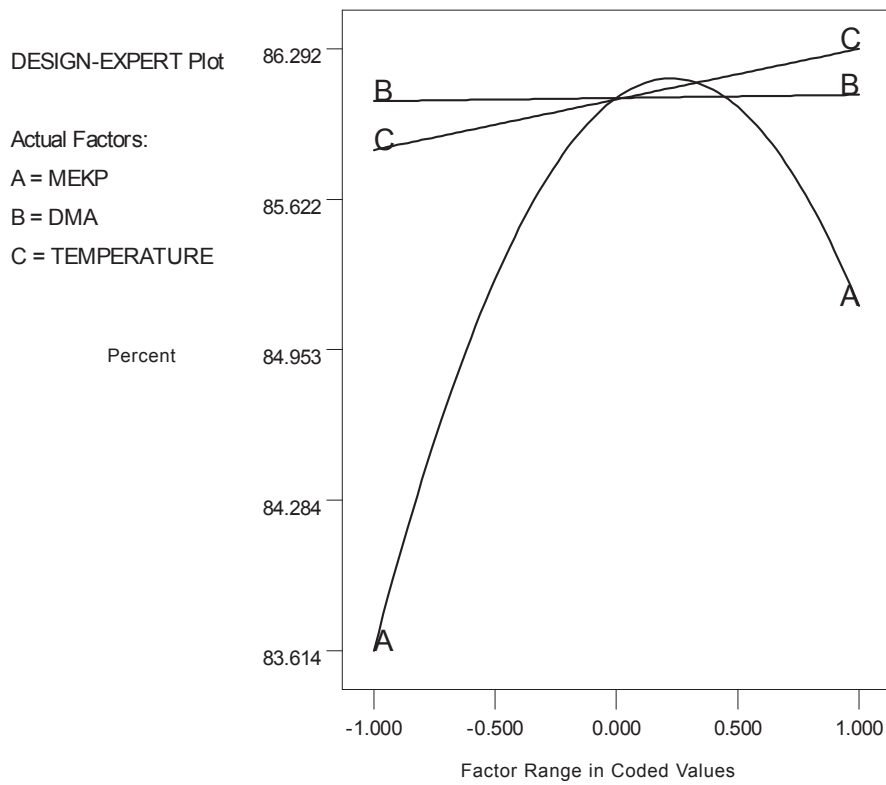


FIGURE 9
Response: Tg

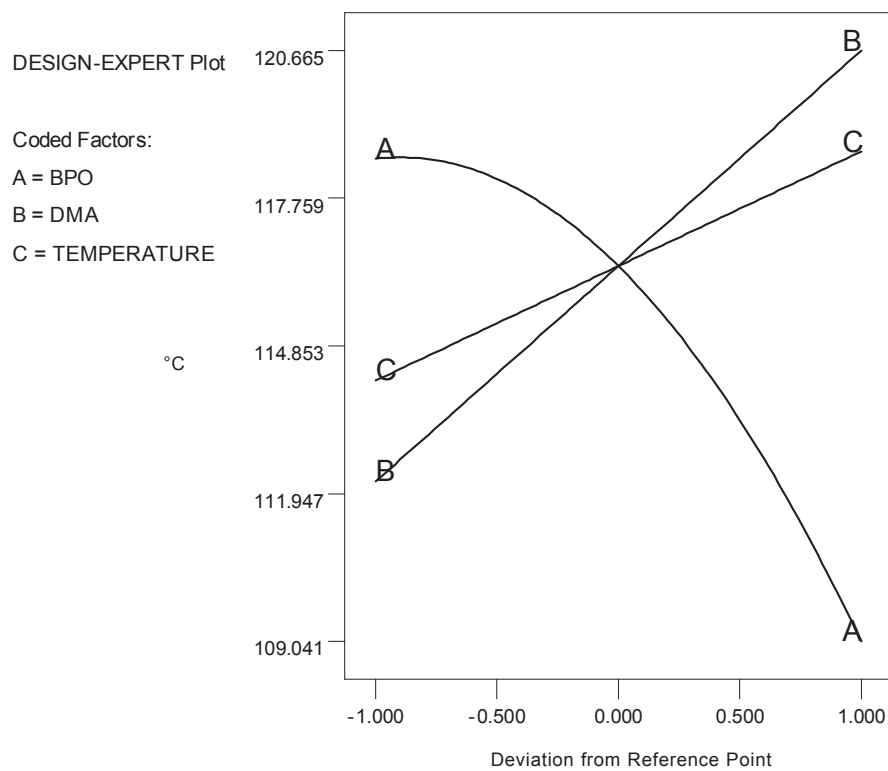


FIGURE 10
Response: T_g

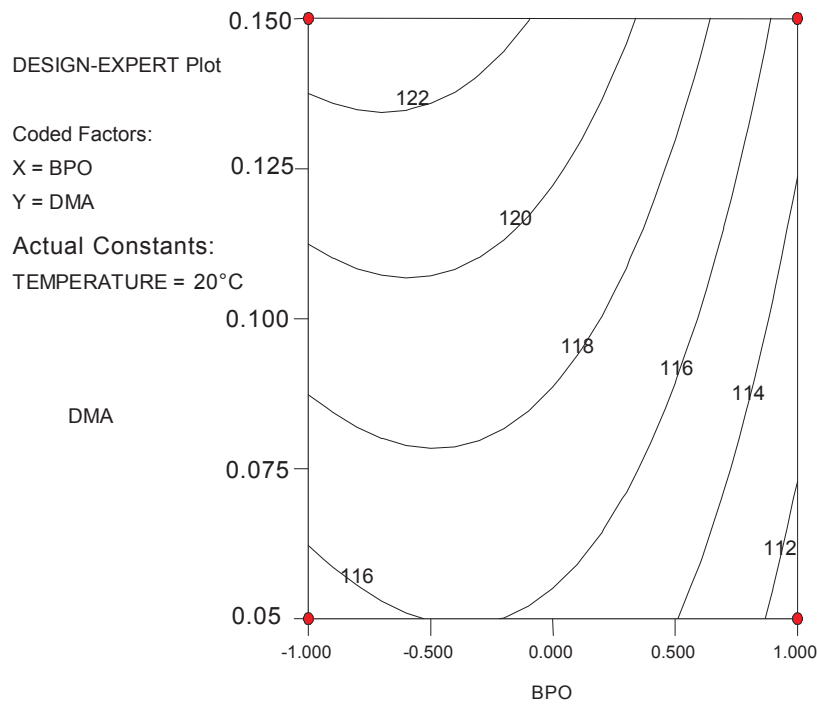


FIGURE 11
Response: T_g

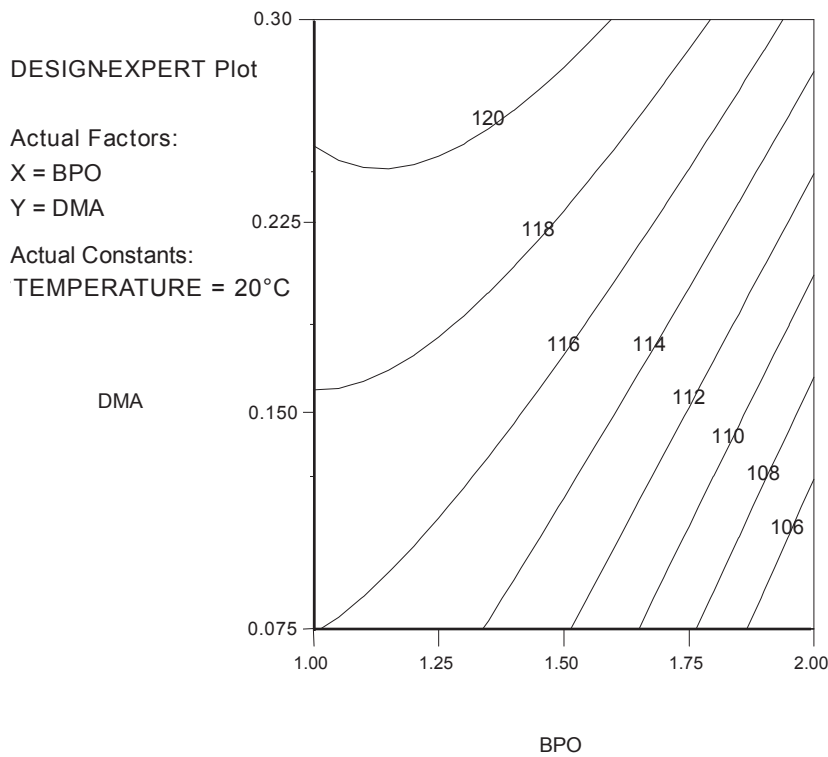


FIGURE 12
Response: Tg

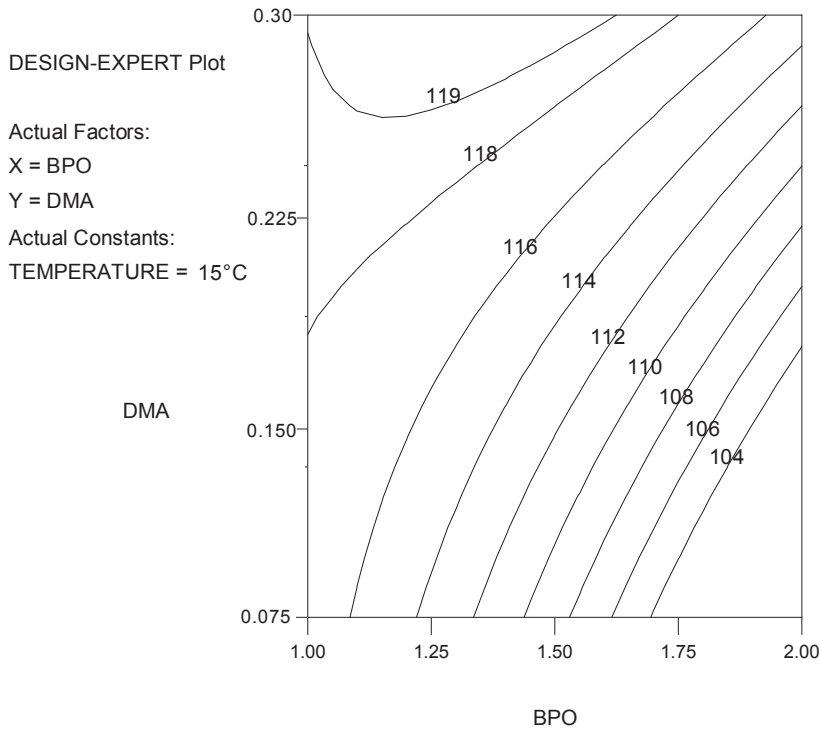


FIGURE 13
Response: Degree of Cure

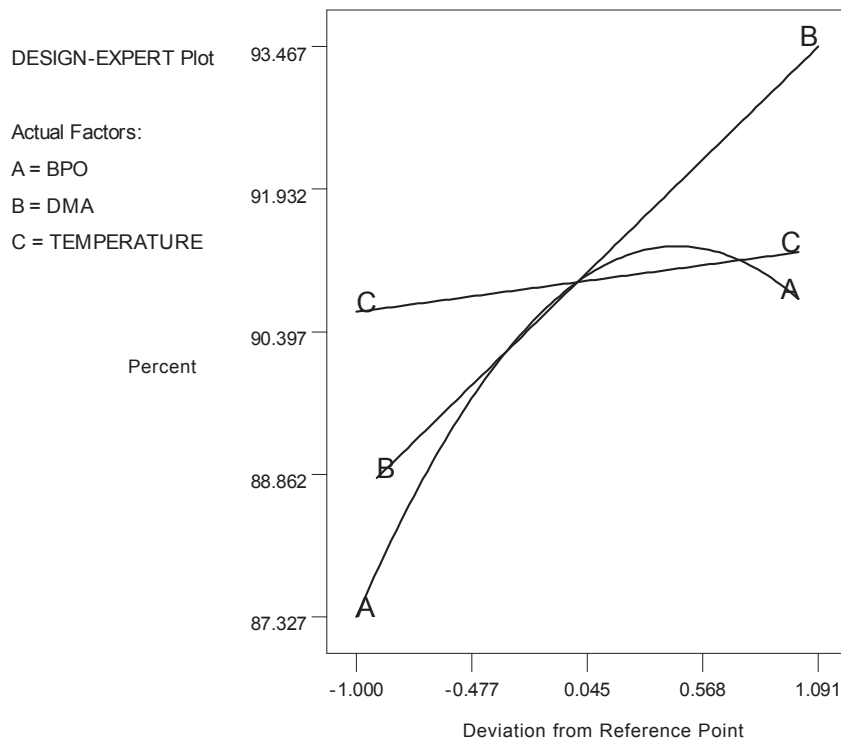


FIGURE 14
Response: Degree of Cure

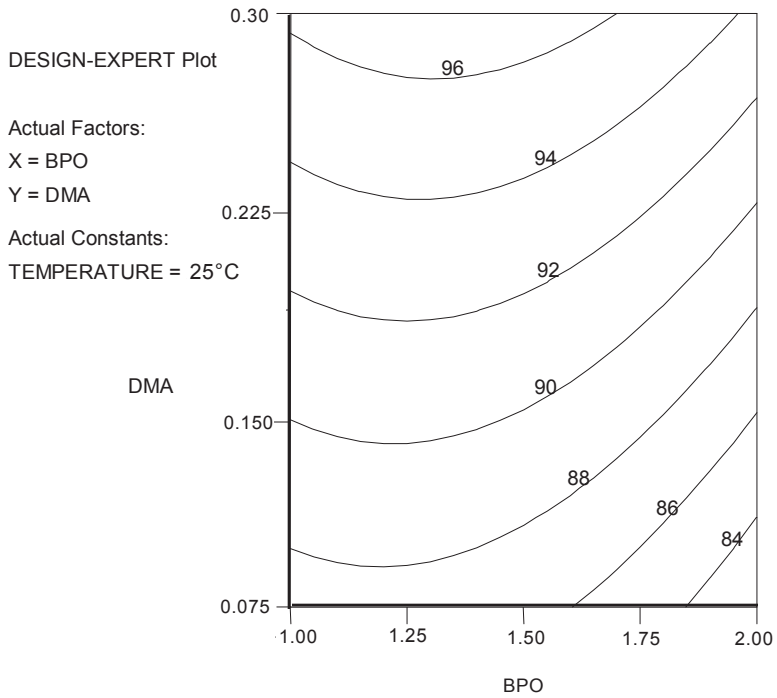


FIGURE 15
Response: Degree of Cure

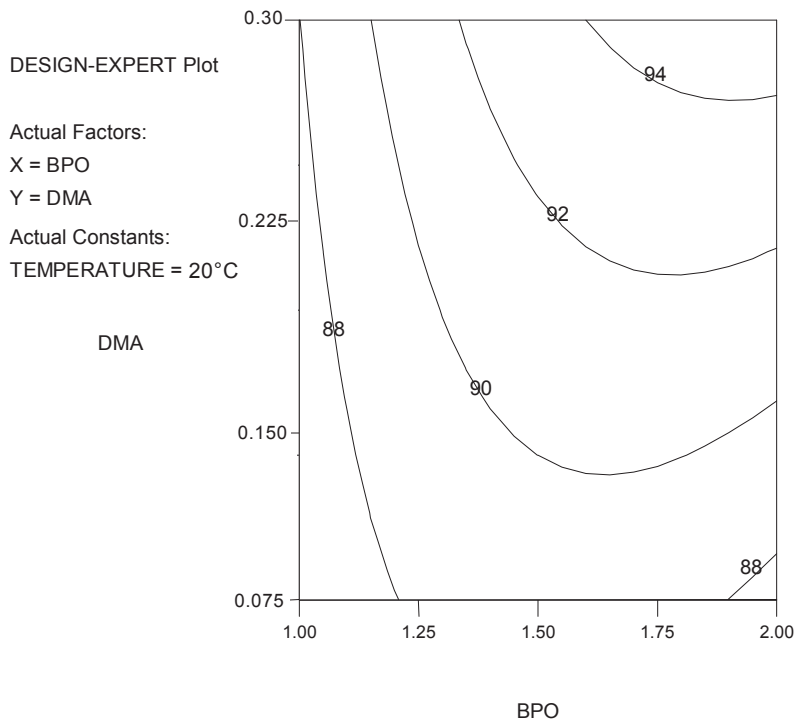


FIGURE 16
Response: Degree of Cure

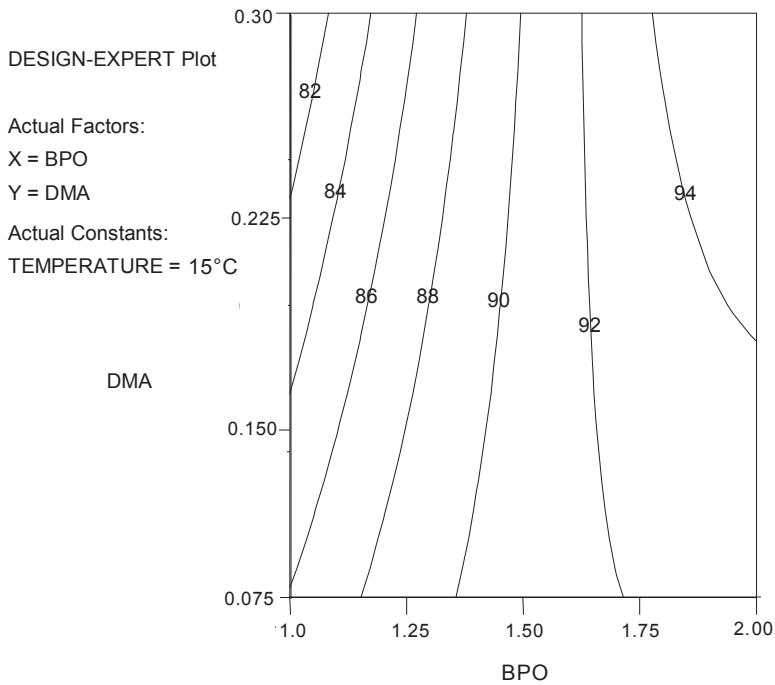


FIGURE 17
Response: Hardness

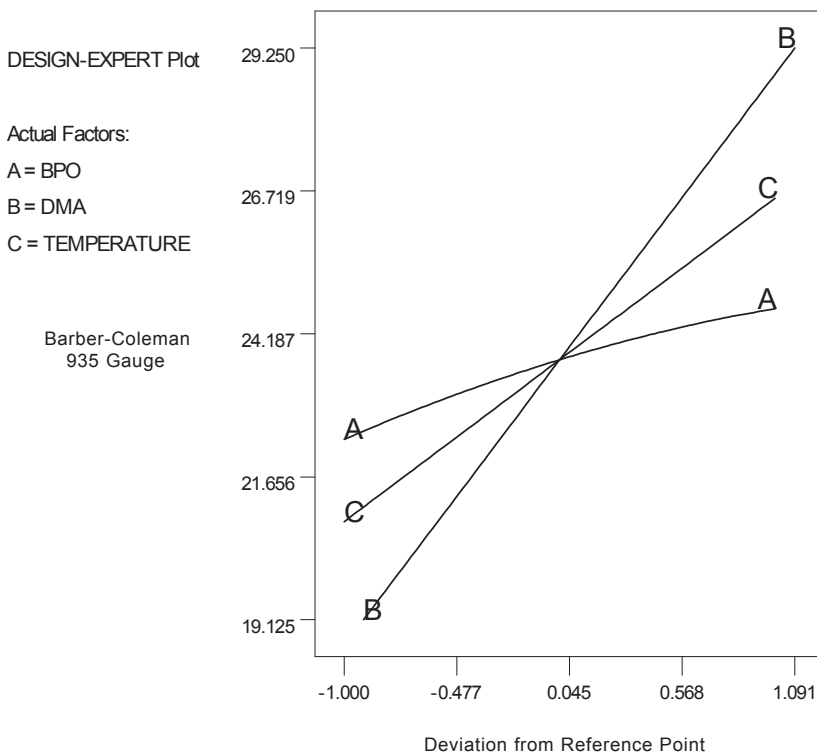


FIGURE 18
Response: Hardness

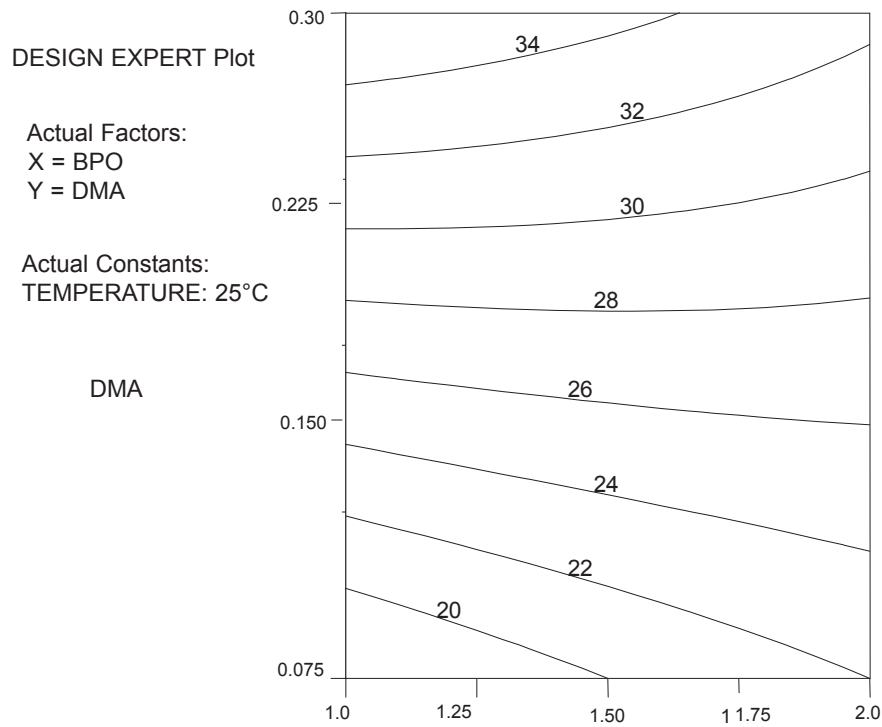


FIGURE 19
Response: Hardness

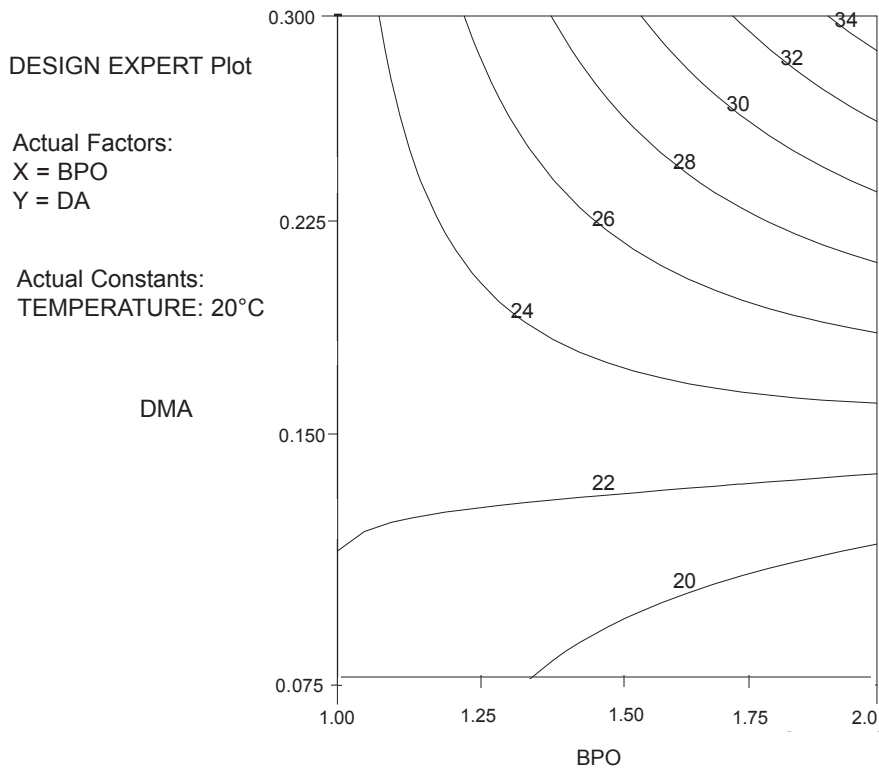


FIGURE 20
Response: Hardness

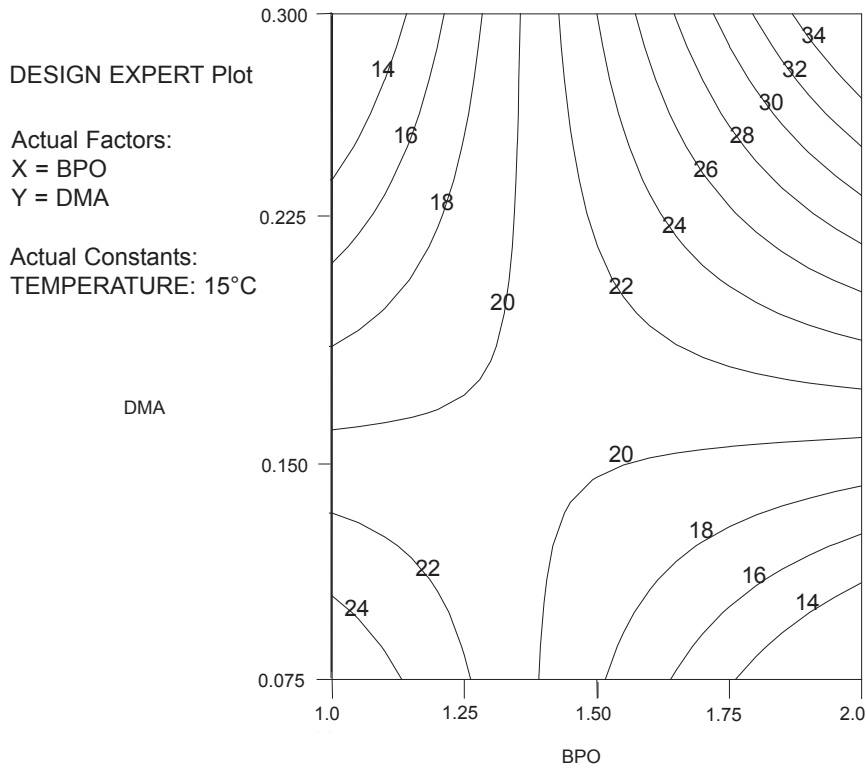


FIGURE 21
Design Expert Graphical Optimization

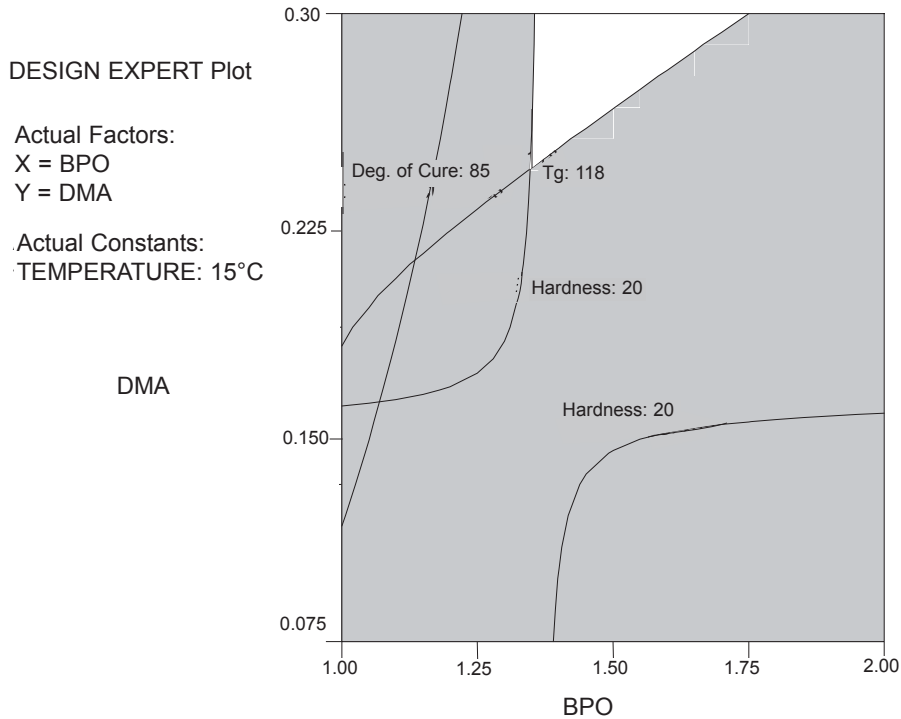
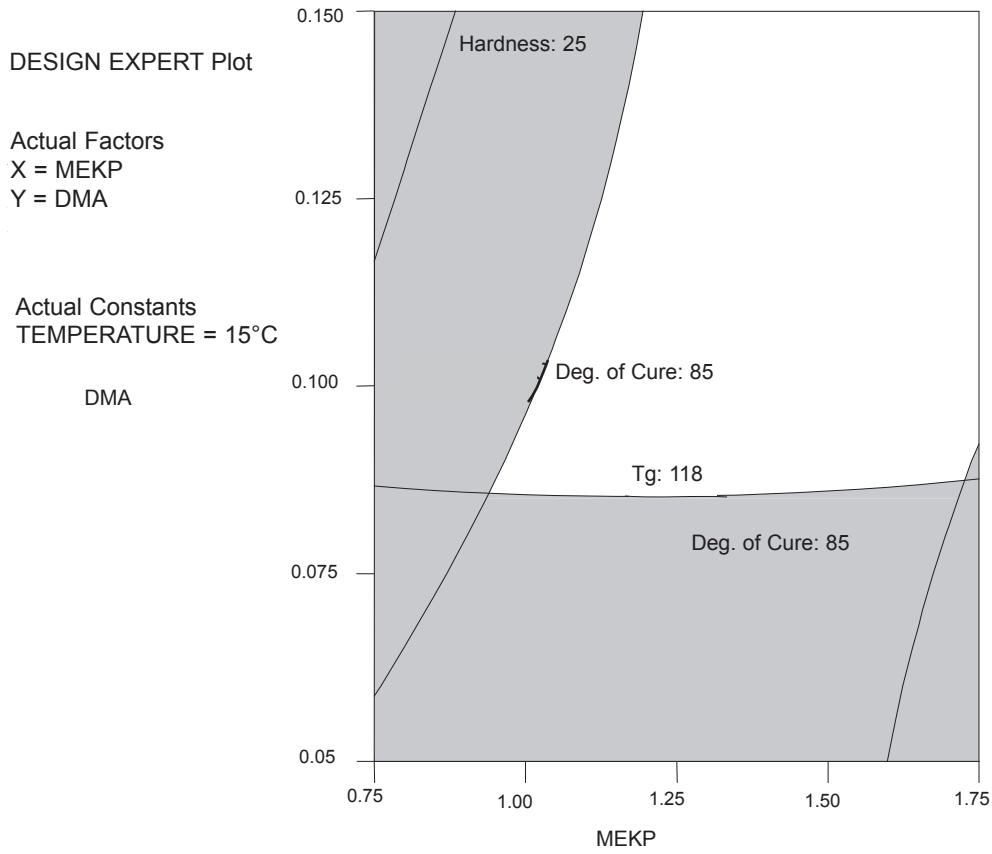
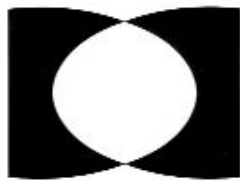


FIGURE 22
Design Expert Graphical Optimization





INTERPLASTIC CORPORATION
Thermoset Resins Division

1225 Willow Lake Blvd., St. Paul MN 55110-5145
800.736.5497 Fax: 651.481.9836
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