Response Surface Methods for Peak Process Performance

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Executive summary

This is the third article of a series on design of experiments (DOE). The first publication provided tools for process breakthroughs via two-level factorial designs.[1] The second article illustrated how to re-formulate rubbers or plastics using powerful statistical methods for mixture design and analysis.[2] The authors now bring their focus back to process improvement and show how to hit the sweet spot of high yield of in-specification products made at lowest possible cost. The key is in-depth DOE aimed at producing statistically-validated predictive models. Response maps made from these models point the way to pinnacles of process performance.

Response surface methods (RSM) are powerful optimization tools in the arsenal of statistical design of experiments (DOE). Before employing RSM, process engineers should take full advantage of a far simpler tool for DOE -- two-level factorials, which can be very effective for screening the vital few factors (including interactions) from the trivial many that have no significant impact. See our first article for a case study on factorial design and, for more details, the book we wrote for non-statisticians.[3] Assuming the potential for further financial gain, follow up the screening studies by doing an in-depth investigation of the surviving factors via RSM. Then generate a "response surface" map and move the process to the optimum location.

This article provides a brief on RSM with applications to plastics and rubber. For a complete primer, see our second book on DOE that details the more advanced tools for process optimization.[4]

RSM at its most elementary level – one process factor

To illustrate the elements of response surface methods, we present a very simple study that involves only one factor – cure temperature – and its effect on the ultimate shear strength of a rubber. The data are loosely derived from a problem presented in a standard textbook on RSM.[5] Table 1 shows the experimental design in a convenient layout that sorts the "X" variable (input) by level. The actual run order for experiments like this should always be randomized to counteract any time-related effects due to ambient conditions, etc.

This RSM design on one factor, generated with the aid of statistical software developed for this purpose,[6] provides seven levels of temperature, with three of them replicated – the two extremes (#'s 1-2 and 11-12) – twice each, and the center point (5-8) – four times over. This provides a total of 5 measures, or "degrees of freedom," for "pure" error. Note that repeated measures or resampling from a given run will provide more stable averaged results, but only a complete re-run, for example – recharging a reactor, bringing it up to temperature and so forth, will suit for measuring overall process/sample/test variation. In general, the minimum requirement for an RSM design is that each factor be tested at three levels over a continuous scale. Additional levels provide for a statistical test on lack of fit measured against the pure error obtained via replications of one or more design points.

Table 1: One-factor RSM design on rubber-curing process

#	A:Cure Temp (Deg F)	Ultimate Shear (PSI)
1	280.0	711.2
2	280.0	739.9
3	286.0	847.9
4	292.0	849.0
5	297.5	806.9
6	297.5	828.9
7	297.5	776.0
8	297.5	844.0
9	303.0	663.5
10	309.0	513.0
11	315.0	218.9
12	315.0	243.0

There is no significant lack of fit in this case as one can infer by inspection of Figure 1 – the response surface for ultimate shear strength of rubber cured at varying temperatures. Imagine fitting a straight edge to this surface – it should be no surprise that statistics then show a significant lack of fit.

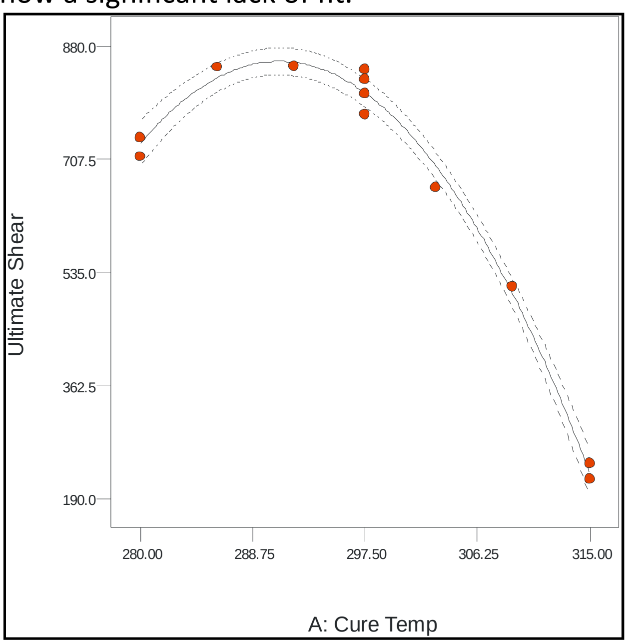


Figure 1. Response surface of ultimate shear versus cure temperature

This curve was created from the following second-order polynomial model, called a "quadratic," via least squares regression:

$$\hat{Y} = 808.77 - 250.45\ X - 328.58\ X^2$$

This experiment design provides sufficient input levels to fit a third-order (cubic) term – X^3. However, statistics show that it contributes insignificantly to the fitting of response data, thus there will be no advantage – only complication. When modeling data it is best to keep things as simple as possible by the principle of "parsimony."

The 'hat' over the response (output) variable "Y" indicates that this is a predicted value. The coefficients are based on coded values of X (the input variable) scaled from –1 to +1 over the range tested – 280 to 315 degrees F. Coded models, a standard practice for RSM, facilitate comparison of coefficients, which becomes more useful with multiple factors, as will be seen in the next example. It pays immediate dividends for predicting the ultimate shear strength at the center point value for cure temperature of 297.5 degrees F: Simply plug in zero for X, which leaves the model intercept of 808.77 as the expected outcome.

Of much greater interest for predictive purposes is the location of the maximum shear strength. For a single response measure the polynomial model lends itself to simple calculus. However, numerical search algorithms, such as simplex hill-climbing, work better in general and they can be done quickly with the aid of computers. In this case, the cure temperature is found at 290.8 degrees F (-0.381 coded) at which an ultimate shear strength of 856.5 psi is predicted with a 95% interval of 799.05 to 913.94 psi – individual results will vary within this range.

This simple example provides the basics of response surface methodology, but the big payoff comes with multiple factors tested on processes with multiple responses that all must meet predetermined specifications. The next case provides illustration.

Discovering the sweet spot for multiple responses

Success in production of polymeric high-aspect-ratio microstructures (HARMs) depends greatly on the adhesion force between the master mold and the silicone rubber during the demolding process. To study this, process engineers[7] performed a 17-run, "Box-Behnken" design (BBD) on three critical process factors known to affect their results. The BBD is a popular template for RSM because it requires only three-levels of each process factor and only a fraction of all the possible combinations. Details on the BBD can be found in references 4 and 5. Figure 2 shows the BBD structure for three factors, in this case:

A. Coil power, 100-300 watts
B. Passivating time, 10-600 seconds
C. Passivation gas flow, 10-120 standard cubic centimeters per minute (sccm).

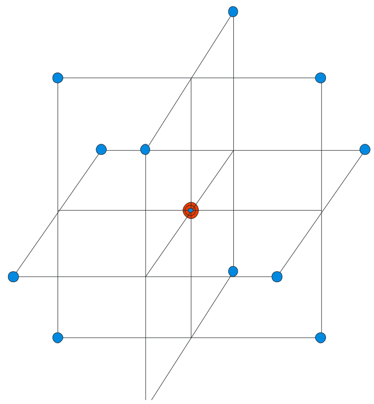


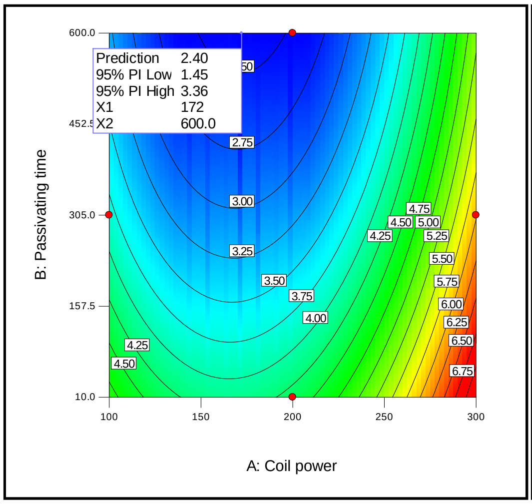
Figure 2. Box-Behnken design on three factors

The experimental matrix and results on adhesion and relative cost (discussed below) are shown in Table 2. As symbolized in Figure 2, the BBD template calls for replication of the center point a number of times, ideally five as shown for this case – the last ones in standard order ("Std") listed in the table. The actual run order was done at random – an essential element of experimentation done to establish cause-and-effect relationships.

Table 2: Design matrix for RSM on silicone rubber molding process

Std	Run	A: Coil power (W)	B: Pass. time (Sec)	C: Gas flow (Sccm)	Peel force (N)	Cost (Rel.)
1	9	100	10	65	4.95	7.5
2	1	300	10	65	6.82	9.5
3	7	100	600	65	3.58	66.5
4	3	300	600	65	4.95	186.5
5	16	100	305	10	3.98	31.5
6	6	300	305	10	6.02	92.5
7	17	100	305	120	3.98	42.5
8	10	300	305	120	4.77	103.5
9	2	200	10	10	4.07	3
10	5	200	600	10	2.62	121
11	13	200	10	120	3.27	14
12	4	200	600	120	2.92	132
13	8	200	305	65	2.98	67.5
14	11	200	305	65	3.42	67.5
15	12	200	305	65	2.82	67.5
16	14	200	305	65	3.41	67.5
17	15	200	305	65	3.02	67.5

Model-fitting done by RSM software revealed that the quadratic contour and 3D response surfaces in Figures 4 a and b; respectively, provide an adequate picture of the predicted peel force as a function of power and time (gas flow set low for minimization of this response). The flag locates the optimal setting of 172 watts coil power at 600 seconds of passivation at 10 sccm gas flow, which produces a predicted peel force of 2.4 newtons (N).



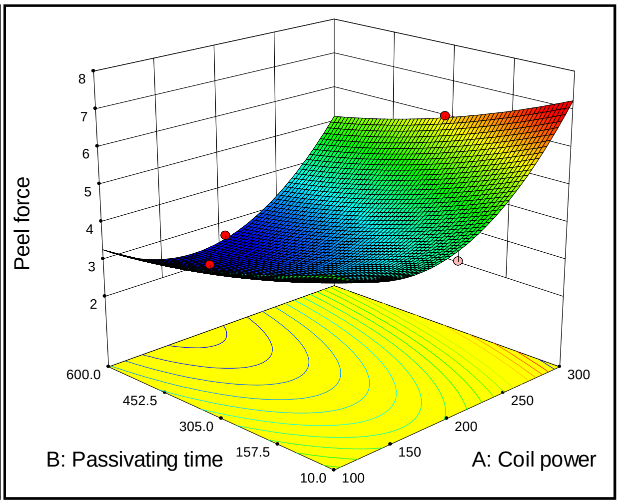


Figure 4a,b. Contour and 3D response surface plots of peel force (gas flow set low)

However, what if a higher peel force, say as high as 3.5 N, would be acceptable? Perhaps another set of conditions might then be more economical in terms of power in watt-seconds and at a lower rate of gas consumption. Programming this in as a second response (relative cost) paves the way to seeing a 'sweet spot' (Figure 5) enabled by increasing the gas flow to maximum level, thus allowing an acceptably low peel force of 3.3 at minimal passivation time (10 seconds) and only a slight increase in coil power (182.7 watts).

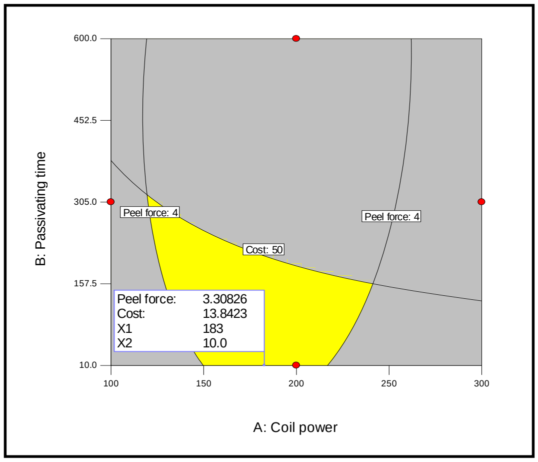


Figure 5. Sweet spot where both peel force and cost are minimized

Conclusion

Response surface methods (RSM) have been shown to be effective for achieving peak performance in processing of products made from rubber and plastics. By making use of this powerful statistical tool for design of experiments (DOE), you will likely discover a winning factor combination – one that achieves the greatest profits for your enterprise.

The author

Mark is a principal of Stat-Ease, Inc.* He is a chemical engineer by profession (State of Minnesota) and certified as a quality engineer (ASQ). Mark co-authored "DOE Simplified" and "RSM Simplified" with his colleague Patrick Whitcomb. They've also collaborated on numerous articles on design of experiments (DOE), many of which can be seen or ordered as reprints from http://www.statease.com.

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References

1. Anderson, M. J., Whitcomb, P.J., Breakthrough Improvements with Experiment Design, *Rubber and Plastics News*, June 16, 1997, pp. 14-15.
2. Anderson, M. J., Whitcomb, P.J., Mixture DOE Uncovers Formulations Quicker, *Rubber and Plastics News*, October, 2002, pp. 16-18.
3. Anderson, M. J., Whitcomb, P.J., DOE Simplified – Practical Tools for Effective Experimentation, 2nd Edition, Productivity, Inc., New York NY, 2007.
4. Anderson, M. J., Whitcomb, P.J., RSM Simplified – Optimizing Processes Using Response Surface Methods for Design of Experiments, Productivity, Inc., New York NY, 2005.
5. Myers, R.H., Montgomery, D.C., Response Surface Methodology, John Wiley and Sons, Inc., New York, NY, 2002, problem 2.21.
6. Helseth, T. J., et al, Design-Expert® Version 7.1 Software for Windows, Stat-Ease, Inc, Minneapolis ($995).
7. Yeo, L. P., Poh, S. L., Lam, Y. C., Chan-Park, Mary B., Plasma polymerization of C4F8 thin film on high aspect ratio silicon molds, manuscript posted November 9, 2004 by MIT Libraries at http://dspace.mit.edu/.