Design of Experiments Helps Improve the Robustness of Off-Highway Vehicle Coating

Determining the optimum conditions for applying coatings suitable for tough applications such as agricultural vehicles is a difficult challenge. The conventional method is to perform a series of experiments while varying one factor a time. This approach is usually capable of finding a single set of conditions that will provide the desired result. However there is rarely enough time to test all combinations. Thus there is no way to ensure that quality will be maintained despite inevitable processing variations. Henkel Corporation overcomes this challenge by using designed experiments that utilize a small number of runs to evaluate all factors simultaneously. “Statistical analysis of these experiments determines all of the combinations of operating conditions that will produce the desired results,” said Bill Fristad, Technical Director, Automotive & Industrial R&D for Henkel Corporation. “Then we can design the application to ensure high levels of quality despite variations in process conditions.”

Henkel adhesive, sealant and surface treatment technologies are used by agricultural and construction machinery manufacturers to improve product performance, reduce production costs and create efficient manufacturing processes. Henkel markets products under its own name and some of the brands include Granocoat®, Bonderite®, Alodine®, and Terophon®. Henkel provides adhesives and sealants for assembly operations and pretreatments, autodeposition coatings, lubricants, and cleaners for painting and fabrication processes. Henkel provides custom-formulated products to fit each customer’s requirements and backs them up with technical support. Henkel scientists and engineers follow the manufacturing progression from conception through production.

Tough coating application

Recently, an agricultural equipment manufacturer asked Henkel to help optimize a tough coating application. The applied coating had to withstand a number of tests including crosshatch adhesion, reverse impact, pencil hardness, methyl ethyl ketone (MEK) double-rubs to substrate and 504-hour neutral-salt-spray (NSS) testing as shown in Figure 1. The last test is the most challenging in this application. The paint is applied to the panel and then a line is scribed all the way through to the bare metal. The panel is exposed to a saltwater fog for 21 days and the total amount of coating which has been lost on both sides of the scribe, a value known as creep, is measured. In this case, the customer required that creep be no more than 3 mm.

Figure 1: Panel shown after exposure to 504-hr NSS testing.

Henkel engineers wanted to evaluate two different coatings for this application, A and B. Normally, these coatings are cured at 325^{o}F for 25 minutes. In this case, Henkel engineers wanted to evaluate temperatures within +/- 25^{o}F and times within +/- 15 minutes of the standard values. The conventional approach would have been to run a series of experiments, changing one variable at a time to determine its effect on creep. For example, engineers would pick a time and then vary the temperature for a series of batches. Or they would pick a temperature and vary the time. It takes approximately one month to coat a batch of samples and run the 504-hour NSS test.

The problem with conventional one-factor-at-a-time (OFAT) experiments is that they do not capture interactions between different factors. For example, raising curing time may have a much greater effect on creep performance when the temperature is higher. Using this method, it is necessary to test every possible combination of factors in order to understand the entire range of process conditions. For their application, Henkel engineers estimate this iterative series of experiments would have taken 6 to 12 months, much more time than they had. “The nature of the OFAT method plus time limitations on virtually every project make it necessary to take guesses about the best process conditions,” said Chris Weller, Research Chemist for Henkel. “It’s usually not that hard to find acceptable operating conditions but it’s very possible that a slight change in these conditions might cause quality problems.”

DOE helps increase process robustness

To overcome these limitations, Henkel several years ago switched to the design of experiments (DOE) method. By varying the values of all factors in parallel, DOE drastically reduces the number of runs required to determine the optimal value of each factor. This approach determines not just the main effects of each factor but also the interactions between the factors. “DOE gives us an understanding of how any combination of factors within the selected range will affect our responses,” Fristad said. “This makes it possible to provide our customers with values of each factor that will not only work but will maximize the robustness of the application.” Henkel uses Design-Expert® software from Stat-Ease, Inc., Minneapolis, Minnesota, because it is designed for use by subject-matter experts, such as scientists and engineers, who are not necessarily experts in statistical methods. The software walks users through the process of designing experiments and evaluating the results for significant outcomes.

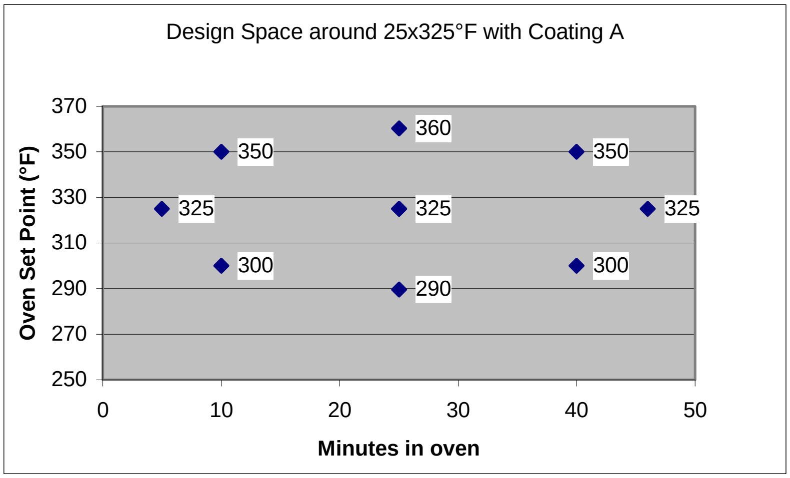

Figure 2: Designed experiment to evaluate cure window. Vertical axis is temperature in Fahrenheit and horizontal axis is minutes in oven.

In this application, Henkel engineers selected a response surface method (RSM) that provides a considerable amount of information on experimental variable effects for the optimization of processes. Weller entered the range of factors that he wanted to evaluate and the software laid on a "central composite" design of nine unique combinations as shown in Figure 2. Henkel technicians performed each experimental run specified by the software for each coating and then ran the NSS test and measured the creep of each sample. They entered the results of the NSS test into Design-Expert and the software generated response surface maps that provided visual depictions of the entire application space. These maps provide a very intuitive understanding of how coating quality responds to the various factors.

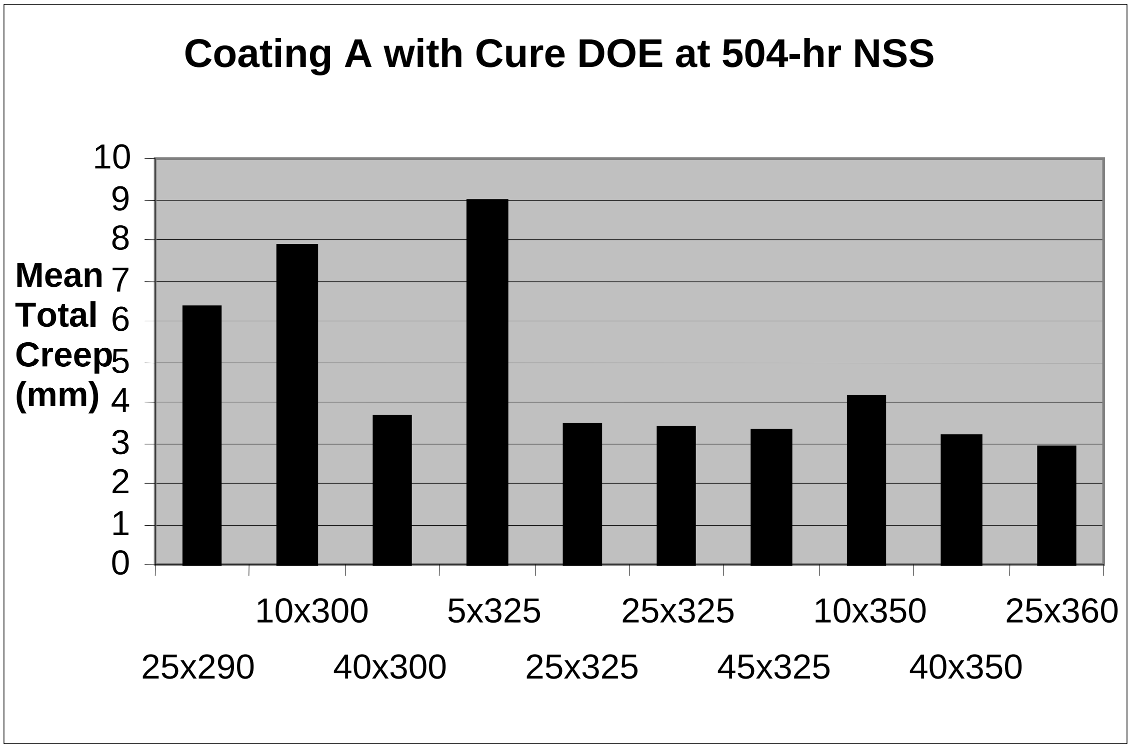

Figure 3: Bar chart showing results for coating A. Bottom axis units are minutes in oven by temperature in degrees Fahrenheit.

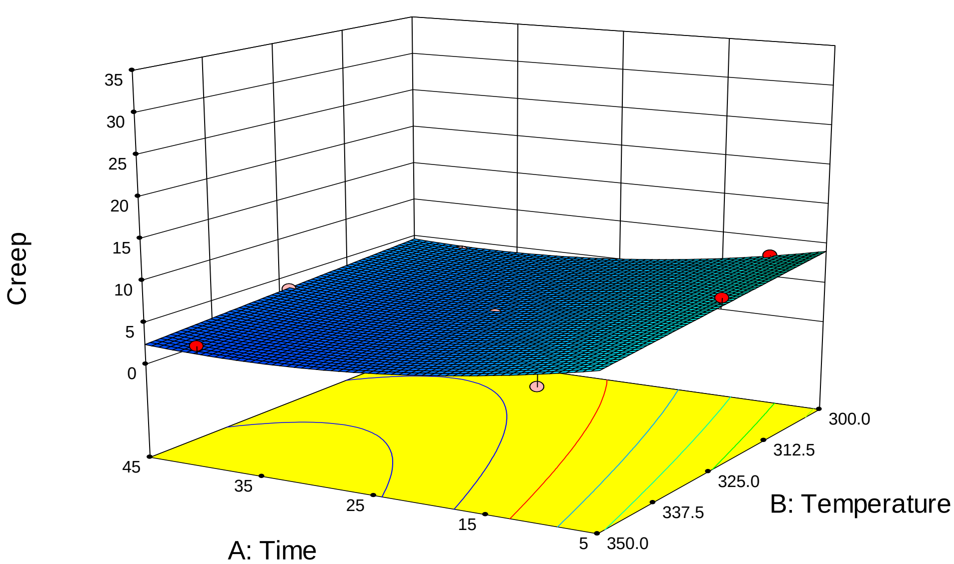

Figure 4: Response surface for coating A

Figure 3 shows the bar chart that represents the NSS test results for coating A. Note that the bar chart results do not clearly indicate how the two factors affect the response. Figure 4 on the other hand shows the same results in the form of a response surface map. This figure provides a clear indication of the portion of the design space that provides acceptable results. The curvature of the response surface map shows that there is a considerable amount of interaction between the two factors. If the factors were independent, then the map would be a plane. Response surface maps help Henkel application engineers understand the complete application space.

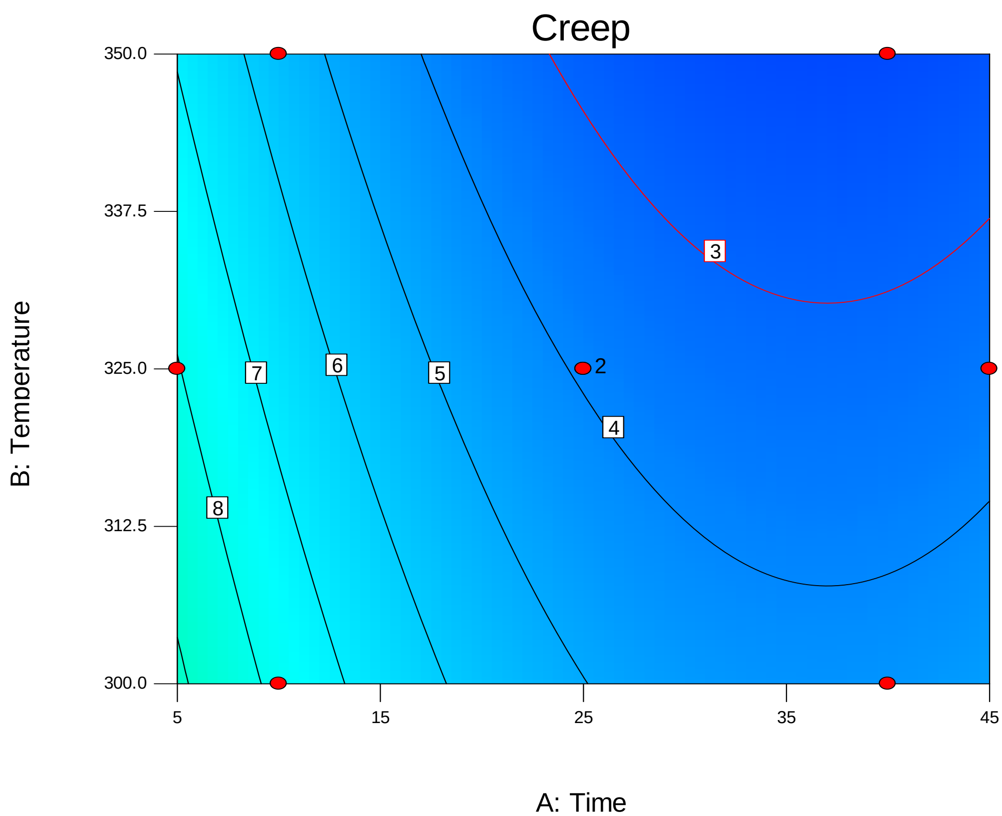

Figure 5: Contour map of creep as a function of cure time and temperature for coating A.

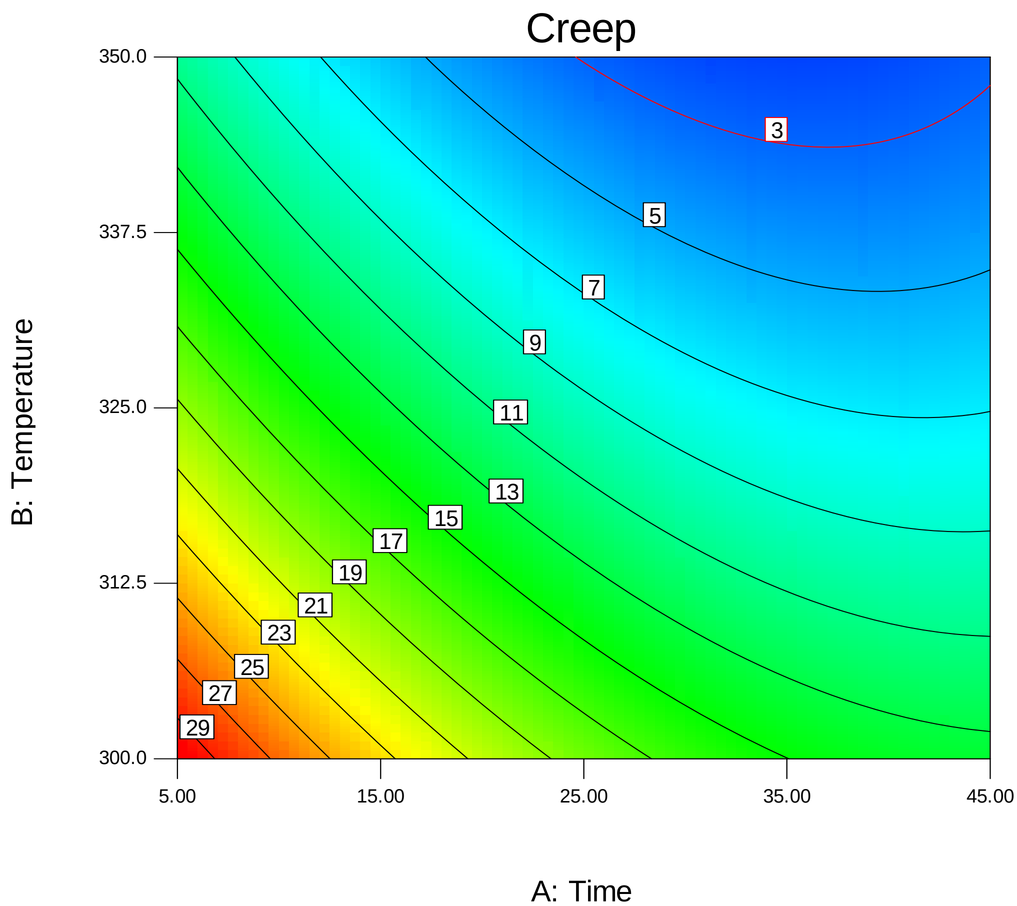

Figure 6: Contour map of creep as a function of cure time and temperature for coating B.

Figures 5 and 6 show creep as a function of cure time and temperature for coatings A and B. These plots show that the region where creep is less than 3 mm is much larger with coating A than with coating B. In particular, the plot shows that coating B is very sensitive to a reduction in cure time or temperature. Process conditions can be developed for either coating that will meet the customer's quality requirements. But when coating A is used the process conditions can be centered within a large region where any point within the region will meet the requirements. "We recommended to our customer that they use coating A with process conditions centered within the low creep region," Fristad concluded. "The result is a very robust process."

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