

Design of Experiments Helps Optimize Injection Molding of Conductive Compounds

Conductive compounds such as plastic resins with carbon nanotubes as an additive make it possible to combine static dissipation and electrostatic discharge (ESD) protection with the design freedom of injection molding. A significant challenge of implementing carbon nanotubes is that the injection molding conditions have a large effect on the resistivity of the finished product. RTP Company, a leading compounder of custom engineered thermoplastics, recently used design of experiments (DOE) to determine which injection molding process conditions optimize conductive properties for a particular material. DOE made it possible to explore the complete processing space and provide users with a formula to calculate the conditions that would deliver the required resistivity levels. “The predictive capabilities of DOE make it possible to fully explore the processing window in only 32 runs,” said Ned Bryant of RTP Company.

A carbon nanotube loading of only 1% to 4% provides a thermoplastic compound with a uniform surface resistivity of 10^4 to 10^9 ohms/sq. This level of loading maintains the resin’s key physical properties including ease of processing, shrink rate, impact strength and surface finish. Carbon nanotube compounds provide a more uniform conductive surface that reduces the hot spots found with carbon fiber-filled compounds. These structures also enable thin-wall molds to fill at lower temperatures. Nanotube compounds are ideally suited for wafer processing, disk-drive components and cleanroom applications. They are beneficial in automotive applications needing static discharge protection, such as fuel system components. Other automotive uses are body attachments such as mirror housings, door handles, fuel filler doors, wheel covers, bumpers, fenders, and interior parts.

The surface conductivity of finished carbon nanotube components is highly dependent on the processing conditions. Yet molders often do not have the time to run the many experiments normally needed to optimize surface conductivity. So they look to materials suppliers for guidance. RTP Company previously performed a series of experiments for customers that typically involved varying one factor at a time in order to evaluate its effect

on conductivity. These experiments provided answers to very limited questions such as: “What is the effect of mold temperature on surface conductivity with all other conditions held constant?” The assumption was made that mold temperature did not interact with any other variables but these experiments did not determine whether or not this was actually true. These experiments were useful at solving such limited questions in isolation. But they did not address other important issues such as: “What are the best material processing conditions that will deliver the desired level of surface conductivity in the finished product?”

More recently RTP Company has begun using DOE to map out the complete processing space in order to enable processors to optimize molding conditions while taking into account second order effects. DOE provides a solid estimate of the value of independent variables for every possible combination of the factors by varying the values of all factors in parallel. This approach determines not just the main effects of each factor but also the interactions between the factors. DOE also requires far fewer experimental runs than the traditional one-factor-at-a-time approach and makes it possible to identify the optimal values for all factors in combination.

“The statistical knowledge required to perform DOE from scratch can be an obstacle to its use,” said Ned Bryant, Senior Product Development Engineer for RTP Company. “I save a considerable amount of time and aggravation by using Design-Expert® software (from Stat-Ease, Inc., Minneapolis, Minnesota) to design and analyze the results of experiments. Design-Expert is easy to use yet can perform many different types of experiments. Matching the right experimental method to the problem at hand helps increase accuracy while reducing the number of required experimental runs.”

In a recent typical experiment, Bryant used DOE to determine the surface conductivity of an RTP Company electronics grade polycarbonate/carbon nanotube compound over an extensive range of processing conditions. The goal was to help processors achieve optimal results with the material. The advantages of the new material include the ability to use thinner wall thicknesses and low specific gravity, both of which combine to reduce material costs by about 5+%. The new material also makes it possible to use lower process temperatures, eliminates isotropic effects, allows use of regrind to lower material costs and provides excellent surface appearance.

Bryant selected a fractional factorial experimental design that examines first and second order effects while requiring only half as many runs as a full-factorial experiment. With six factors to be evaluated a full-factorial experiment would require $2^6=64$ runs while a fractional factorial requires only $2^6-1=32$ runs. The factors evaluated in this experiment were:

- A. Mold Temperature (170°F to 220°F)
- B. Extruder Barrel Temperature (500°F to 580°F)
- C. % Backpressure (1% to 10%)
- D. Injection Speed (4 to 9)
- E. Hold Pressure (10% to 40%)
- F. Hold Time (6 to 15 seconds)

Design-Expert software generated the experimental design by specifying the conditions for each of the 32 runs based on the inputs provided by Bryant. RTP Company personnel then molded 3 inch by 5 inch plaques at each of the 32 run conditions and measured the corresponding surface resistivity with a Voyager test meter. Bryant entered the responses from the experiment into Design-Expert and the software performed statistical analysis.

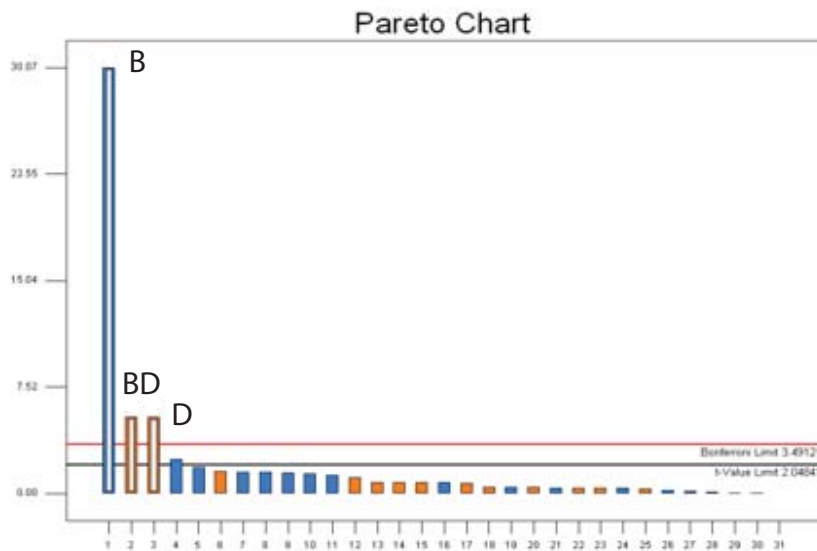


Figure 1: Pareto chart shows the impact of each factor

The Pareto chart shown in Figure 1 shows how much of the variability in the response can be attributed to each of the factors. The results show that the vast majority of the variability results from varying the extruder barrel temperature (B) and a lesser amount is due to the injection speed (D) and the

interaction between extruder barrel temperature and injection speed (BD). The other four variables have minimal impact on the surface resistivity. “This information in and of itself was extremely important because it tells processors which variables they need to hold very precisely and which ones they can safely ignore,” Bryant said.

Final Equation in Terms of:				
Actual Factors				
Log SR =				
53.75031				
-0.0837	* Extruder Temps			
-2.056	* Injection Speed			
0.00411	* Extruder Temp * Injection Speed			

Figure 2: Final equation expresses surface resistivity as a function of two factors and one multiple factor interaction

Design-Expert software also fit the data to an equation that predicts the value of surface resistivity for any values of the factors. This equation, shown in Figure 2, can be used by processors to adjust processing conditions in order to obtain a wide range of surface resistivities.

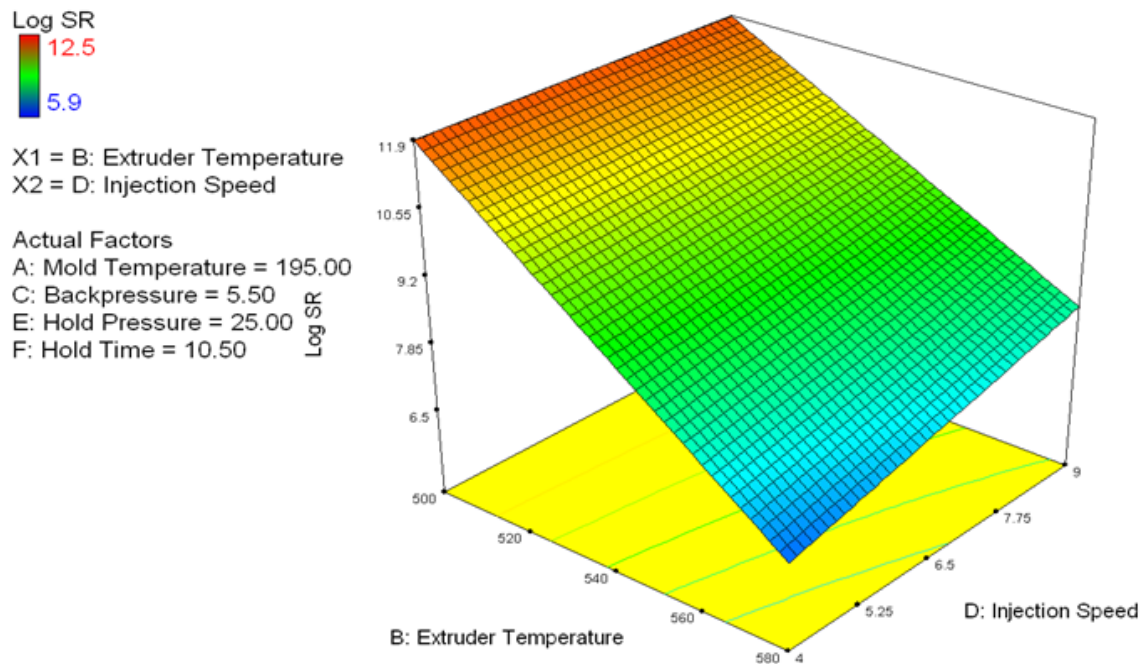


Figure 3: Response surface map

Figure 3 is a response surface map that shows how extruder temperature and injection speed affect surface resistivity. The interaction of the two variables is reflected in the curvature (twisted plane) of the graph. This type of graph provides an intuitive understanding of the effect of the factors on the response. It shows how a small change in extruder temperature has a big effect on surface resistivity, underlining the importance of holding this variable within tight limits. It also shows that as surface temperature is increased past 560°F and injection speed reduced below 5 inches per second, surface resistivity drops sharply as a result of a multiple factor interaction.

“DOE helps our customers set their processing conditions to achieve the required levels of surface resistivity with our materials,” Bryant said. “It shows them which process conditions they need to focus on holding precisely and which ones they don’t need to worry about. DOE also provides significant statistical information that helps demonstrate its validity. We have been getting very positive feedback from our customers concerning our ability to provide these results. They appreciate getting the information and immediately understand its significance. They achieve better results faster and are more likely to use our materials.”

For more information:

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