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Design Of Experiments Approach Halves Development Time

Optimizing the formulation of a new metalworking fluid required assessing multiple additives

By David Slinkman and Yixing (Philip) Zhao, Houghton International

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The metalworking fluids market is $9-billion-plus segment of the specialty chemicals sector that includes cutting, forming, protecting and quenching fluids. One of the most important trends in cutting fluids is for products that work with multiple metals to reduce the number of fluids needed by a factory. Developing fluids suitable for multiple materials adds another layer of complexity to a design process that already involves selecting as many as 25 different additives and determining their optimal proportions. Houghton International used design of experiments (DoE) to develop — in half the time required by the company's previous methods — a line of multi-metal cutting fluids that handle steel, cast iron, aluminum and cast aluminum.

The first stage in the development process for a new cutting fluid is to investigate individual lubricity additives that reduce friction between the cutting tool and workpiece. In this case, Houghton used one factor at a time (OFAT) studies to screen potential additives. We determined the performance of these additives by measuring the coefficient of friction between a test specimen and a pin with the additive applied. Comparing the measured coefficients of friction to a control value gave relative lubrication efficiency. Our chemists tested each selected lubricity additive on different metals and at different application conditions (Table 1). From the test results, we selected lubricity additives for the new formulations based on their ability to provide good lubricity on steel or aluminum or both.

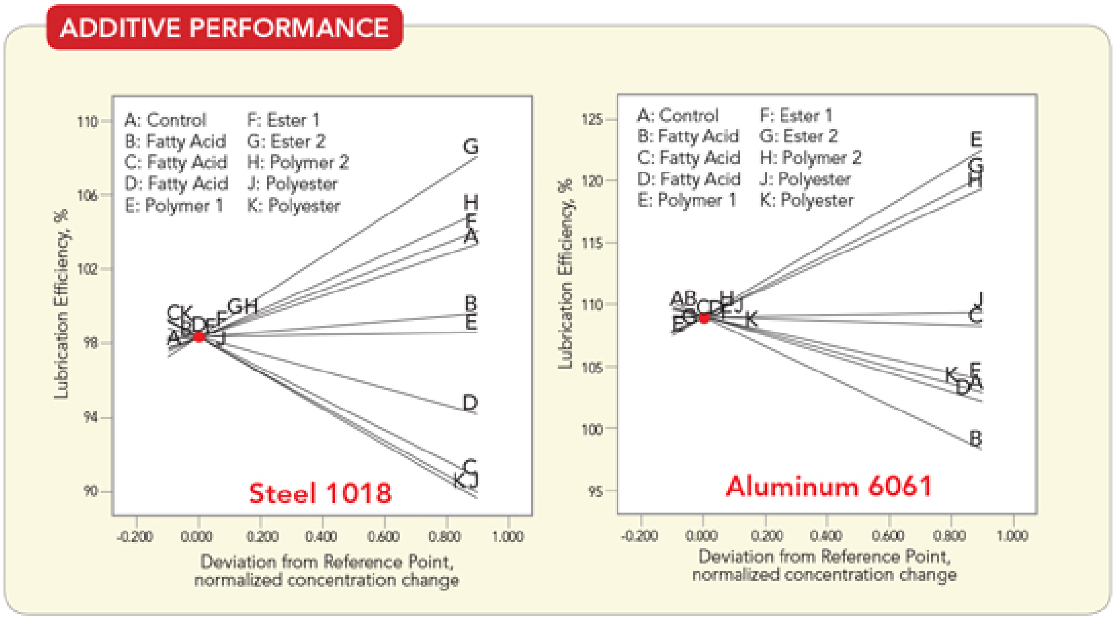
LUBRICATION EFFICIENCY

Additive	Steel 1018		Aluminum 6061	
	Boundary/ Extreme Pressure, %	Mixed/Elasto Hydraulic Lubrication, %	Boundary/ Extreme Pressure, %	Mixed/Elasto Hydraulic Lubrication, %
Control	100	100	100	100
Fatty Acid A	115	130	141	90
Fatty Acid B	83	125	108	126
Ester A	104	154	144	112
Ester B	104	113	112	124
Polyester A	115	129	151	129
Polyester B	120	133	154	101
Polymer A	79	124	-37	102
Polymer B	89	117	62	115
Cl-Additive	88	131	106	47
S-Additive	117	142	151	97
P-Additive	120	147	155	123

Lubrication Efficiency
Table 1. Comparison versus a control highlighted the dramatic differences in additives' performance for steel and aluminum.

Typically, the lubricity package of a cutting fluid includes a number of additives. With many different possible additives to consider, Houghton chemists in the past relied on OFAT methods to identify the right formulation for a new product. This approach provides a quick way to get a rough comparative measurement of additive performance. The problem with OFAT at this stage is that it requires three or four repeats of tests or measurements to evaluate the effects of each additive. However, even after performing these tests, chemists are unsure if the difference in performance between various additives is statistically significant. They also have no way to understand possible interactions between the additives without running many more samples. As a result, this approach is time-consuming and leaves chemists uncertain about whether they have achieved the best formulation.

Houghton International has improved its abilities to develop cutting fluids and other new products over the past five years by applying DoE to its development process. In moving to DoE-based development, managers wanted to ensure that chemists would continue to spend nearly all their time on chemistry and not become bogged down or distracted dealing with statistical software. Houghton accomplished this goal by selecting Design-Expert from Stat-Ease, Inc. This statistical software is designed for people without any statistical background — yet still provides all the analytical power needed for designing specialty chemical products. We found it easy to train our chemists in its use and to convince them to use it. People in many of our global development centers now take advantage of the software. Stat-Ease provides all the different experimental designs, analytical methods and statistical reports that we need to develop successful products.



Additive Performance
Figure 1. The lubrication efficiency of a component differs markedly depending upon the metal.

Choosing The Best Additives

After using the OFAT method to identify promising additives, we turned to mixture design and analysis tools to select the best four or five for more-comprehensive evaluation.

In developing the new multi-metal cutting fluid, Houghton chemists started by calling up the results of previous OFAT studies of ≈40 different additives. They selected the ten most promising ones and set up a mixture experiment in Design-Expert [1]. This DoE required 25 runs, far fewer than the OFAT method would need to evaluate the impact of each additive individually over a range of concentrations. The chemists performed lubricity tests of these mixtures on 1018 steel, 6061 aluminum and 319 cast aluminum. Multicomponent mixture design reduces the number of experiments required to develop formulations. All variables are included and simultaneously varied in the 25 runs. The analysis sorts out the individual effects of each variable as well as its statistical significance. The evaluation showed that some additives are good only on one type of metal while others are good on both (Figure 1).

Based on the mixture experiment, the chemists selected for further study four lubricity additives that provide the best overall performance. Determining this would have required at least twice as many samples using the OFAT method.