Mixture Design of Experiments (DOE) for Optimal Plasma Etch

By Mark J. Anderson, Stat-Ease, Inc. (mark@statease.com)

Executive summary

Design of experiments (DOE) provides statistical tools for fab engineers to improve their operations. But they needn't restrict their studies only to process factors: Adjustments in formulations may prove to be beneficial as well. This article demonstrates how to uncover "sweet spots" where multiple fab-process specifications can be met in a most desirable way. It offers a real-life, semiconductor manufacturing case study that illustrates how to apply powerful response surface methods (RSM) for mixture design and statistical analysis. The resulting predictive models pinpointed a reformulation of plasma that produced more precise etch specifications (smaller offsets in critical dimensions) at greater throughput (selectivity).

Designing a mixture experiment that covers all the bases

To illustrate how to apply mixture design, we present a relatively simple study that involves three gases used in a single-wafer plasma etching process.[1] The experimenters first performed a screening design on five process factors – power, pressure, overetch, hard bake and the SF_6/He gas-mixture ratio. This was done via a 16-run half-fraction of a two-level factorial design (2^{5-1}) with 4 center points added for estimation of pure error. (For amusing, but informative detailings of this multifactor process screening template, see how the author applied it to reliably start a small engine for a vital piece of yard equipment.[2,3])

As a result of this initial process study, the fab engineers knew where to best set the first four factors listed above. However, they decided to follow up by doing an in-depth investigation of the three components of the gas mixture within ranges of partial pressures shown below (in microtorr – symbolized "μm"):

A. SF_6, 100 to 160 μm
B. He, 100 to 160 μm
C. N_2, the remainder as ballast to bring total pressure up to the fixed total of 650 μm.

They entered these mixture design specifications into a personal computer software package developed for this purpose.[4] The first thing it did was some simple arithmetic on the slack variable C to determine that it must range from 330 to 450 microtorr to satisfy the total constraint – the fixed total pressure, 650 μm, of all three gaseous ingredients in the mixture. Then, using a distance-based criterion, the program selected a variety of blends from within the constrained region.

Table 1 shows the experimental design in a convenient layout that identifies the blends by type. The actual run order for experiments like this should always be randomized to counteract any time-related effects due to aging of material, etc. Also, we recommend that you always replicate at least four blends to get a measure of error. In this case, the experimenters re-ran each of the blends at the vertices of the feasible mixture region at different times at random intervals throughout the experiment (never one right after the other). The experimenters expected the mixture to exhibit “strong nonlinear behavior” so they made sure the design included many levels (5) of each of the two active ingredients (components A and B).

Table 1. Design matrix and data for gas-plasma mixture experiment

ID	Location	A: SF_6 µm	B: He µm	C: N_2 µm	Off spec microns	Selectivity ratio
1	Vertex	100	100	450	0.26	0.91
2	“	100	100	450	0.30	0.88
3	Vertex	100	160	390	0.23	0.77
4	“	100	160	390	0.23	0.76
5	Vertex	160	100	390	0.62	0.84
6	“	160	100	390	0.68	0.87
7	Vertex	160	160	330	0.33	0.99
8	“	160	160	330	0.36	1.02
9	Center Edge	100	130	420	0.27	0.91
10	Center Edge	130	160	360	0.31	0.91
11	Center Edge	130	100	420	0.39	0.87
12	Center Edge	160	130	360	0.30	0.99
13	Check Blend	115	115	420	0.23	0.91
14	Check Blend	145	145	360	0.26	0.91
15	Centroid	130	130	390	0.34	0.92

The geometry of this mixture experiment region can be seen in Figure 1.

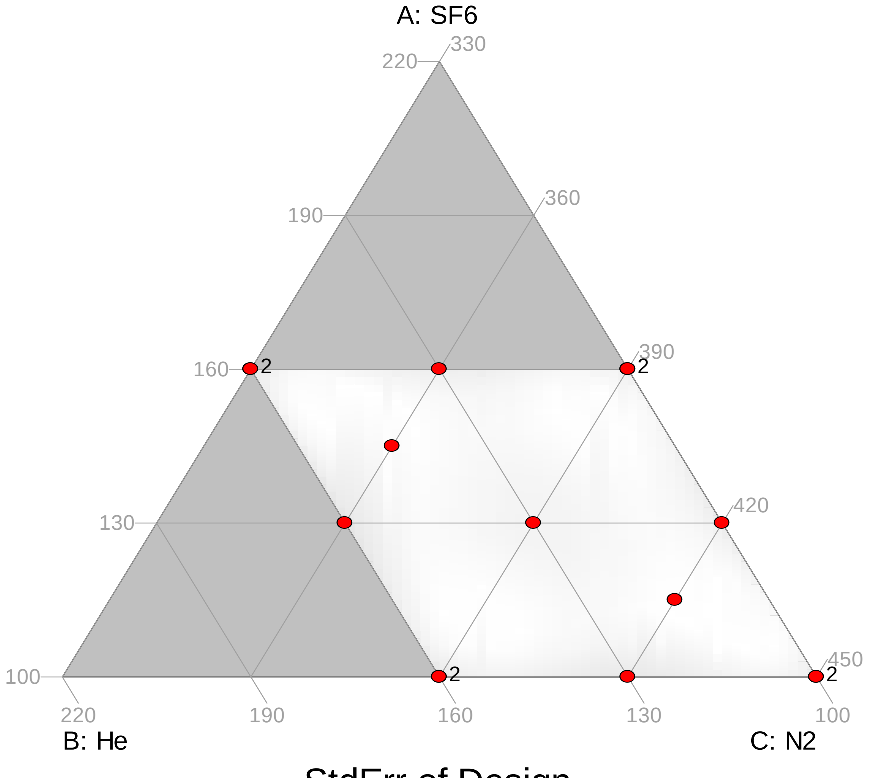


Figure 1. Location of experiment blends within feasible mixture space

The points labeled "2" are the vertices of the constrained region, each of which was replicated. The gas levels can be tracked by following the gridlines. For example, the replicated vertex at the lower right is blend number ("ID") 1 with the minimum levels of components A (SF_6) and B (He) of 100 microtorrs each, which causes the ballast gas C (N_2) to achieve its maximum amount of 450 microtorr. (For further practice with trilinear graphs, see how the author applied mixture design to develop an optimal formulation for homemade play putty.[5,6])

Fitting a predictive model

As shown below, the two responses (designated mathematically as "Y") were fitted via least squares regression to a special form of polynomial equation developed for mixtures (detailed in a textbook by Cornell[7]):

$$\hat{Y} = f(A, B, C, AB, AC, BC)$$

We call this simply a "mixture model." This particular one contains second order terms (AB, AC and BC) that fit nonlinear blending behavior. Note that the function, unlike ones used to graph responses from a process, contains no intercept term, thus accounting for the overall constraint that all mixture components must sum to one. The "Ŷ" (referred to by statisticians as "Y-hat") represents the predicted response. It's the dependent variable. The independent variables (A, B, C), sometimes represented mathematically by X's, are typically converted from their original metric, such as 0 to 100 percent, to a coded format going from 0 to 1, thus facilitating interpretation of the resulting coefficients. For example, the first blend in Table 1 (ID#1), a vertex, is coded as (0,0,1). The fitted coded-equations, both of which exhibited outstanding model-statistics, are:

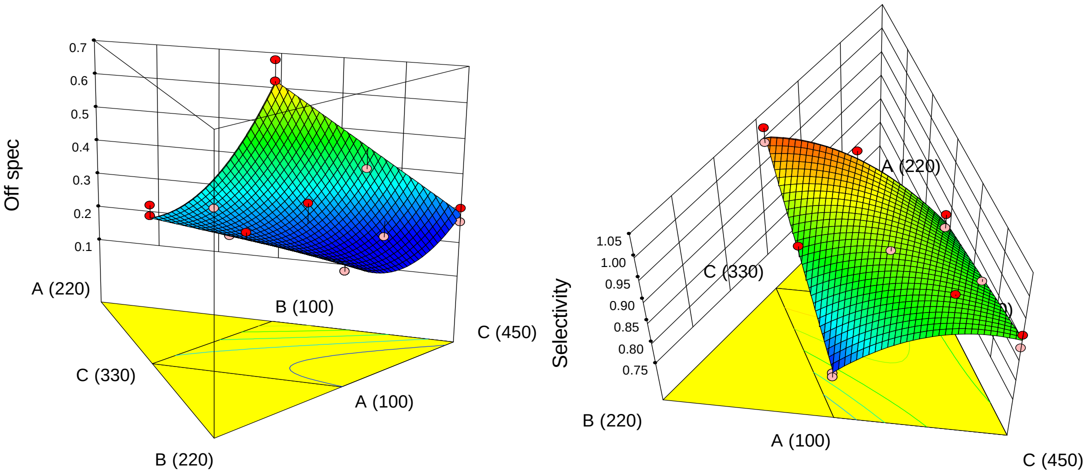
$$\text{Off spec} = 0.95A + 0.93B + 0.28C - 2.45AB - 1.39BC \quad (p=0.002,\ R^2_{adj} = 0.82)$$

$$\text{Selectivity} = 0.81A + 0.27B + 0.90C + 1.86AB + 0.76BC \quad (p<0.0001,\ R^2_{adj}=0.89)$$

Neither model contains the nonlinear blending term AC, because in both cases their probability values (“p”) at levels of 0.66 and 0.54; respectively, far exceeded the generally-acceptable significance threshold, which typically must fall below 0.1 to be considered worthy of publication. However, whether an insignificant individual term like AC is kept in the model, or not, makes little difference in the end.

Response surface graphs tell the story

The mixture models become the basis for response surface graphs, which can be generated from the mixture DOE software – no need to be bogged down in the mathematics: The pictures tell the story! The graphs provide valuable insights about the formulation. Figures 2a and b show 3D representations of the two responses, with 2D contours projected below it, as a function of the three gases used in the plasma-etching process on single wafers. They are rotated to provide a better view of the curvatures.



Figures 2a,b. 3D response surfaces for the two responses

Experiments like this are designed to locate the ‘sweet spot’ where quality requirements are met at greatest productivity. In this case the fab engineers were tasked with reducing “off-spec” below 0.25, while maintaining selectivity at 0.85 or higher. Figure 3 shows the region where these response criteria are achieved. It includes an actual run, ID#9, run at a center edge point. The flagged point is another possibility – one of many that are predicted to meet the requirements of the single-wafer etching process. However, anything outside the operating window exceeds 0.25 in off-spec and/or fails to achieve the desired 0.85 selectivity.

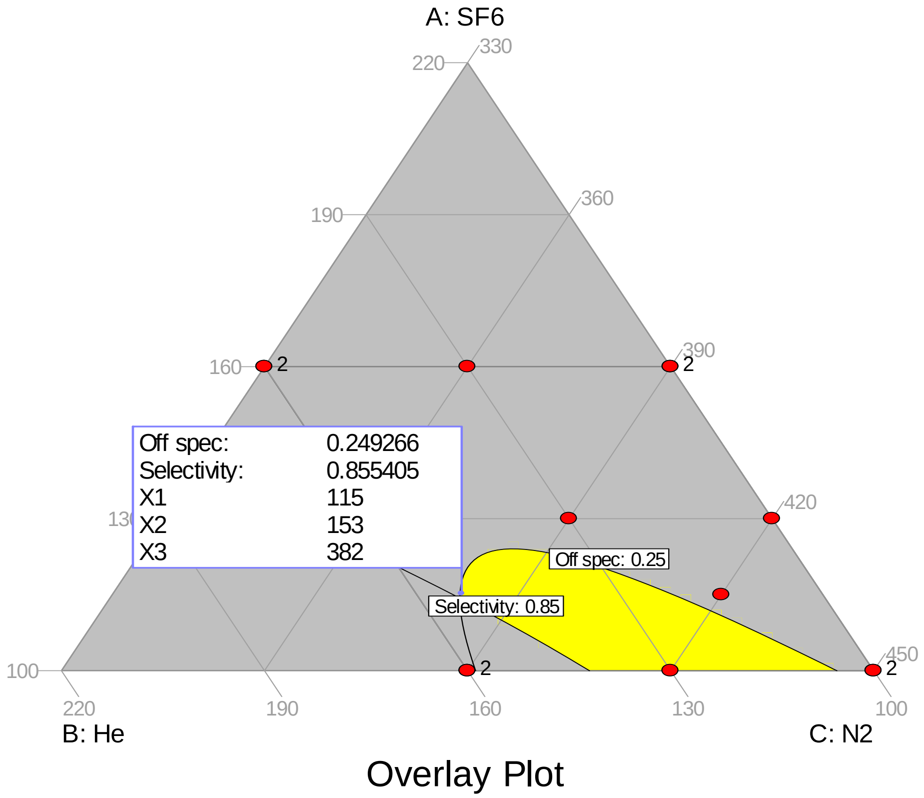


Figure 3. The sweet spot for achieving low Off-spec with high Selectivity

The most desirable gas mixture, pinpointed via a computer search, is pictured in Figure 4 – 100 microtorr of SF_6, 118 microtorr of He with 432 microtorr of N_2 as ballast to bring the total pressure up to its fixed total of 650 microtorr.

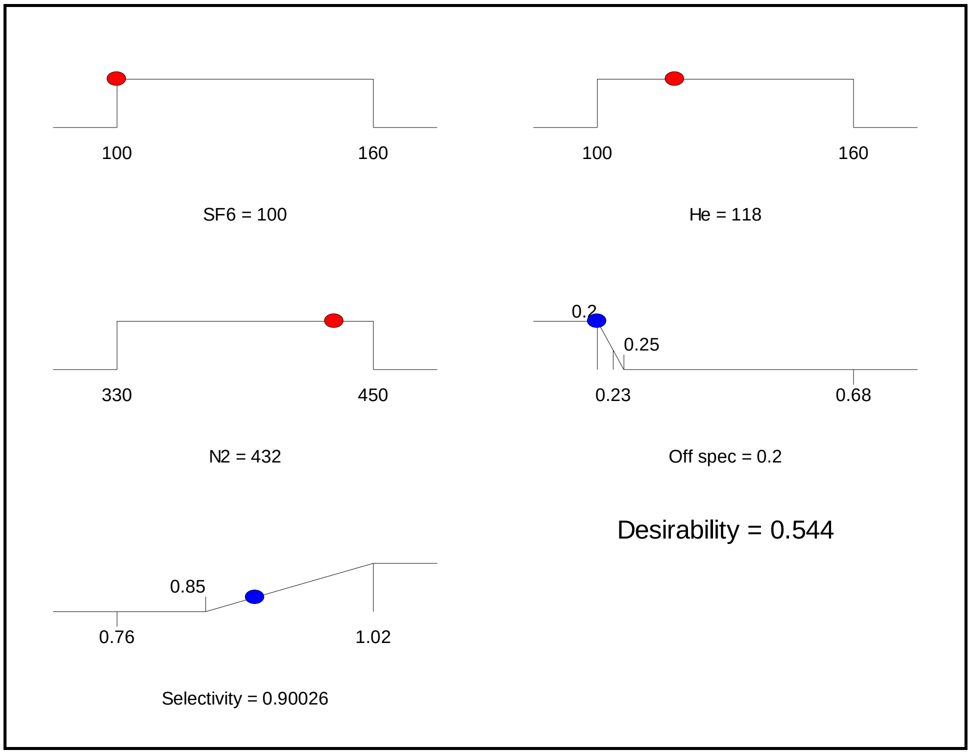


Figure 4. Most desirable mixture of the three gases

The ramps translate the predicted responses of off-spec and selectivity, 0.2 and 0.9; respectively, to their relative desirabilities. In this case the quality of achieving a highly desirable (low) level of off-spec comes at price – the productivity in terms of selectivity does not come out as high as the fab engineers might have hoped. By placing more importance on one response versus another, the optimal blend can be biased, but this must be done judiciously as dictated by the needs of manufacturing and the customer.

In this case, the fab engineers found that their reformulated plasma gas significantly improved the performance of the single-wafer etching process, thus validating the results of their mixture experiment.

Conclusion

By using design of experiment (DOE) methods tailored for mixture design, fab formulators can greatly enhance their exploration of alternative blends. Then with the aid of response surface methods (RSM), they can discover the most desirable combination of components within the feasible mixture space.

The Authors

Mark is a principal of Stat-Ease, Inc. He is a chemical engineers by profession. Mark co-authored the two-part series of engineering primers *DOE Simplified*[2] and *RSM Simplified*.[8] He's written numerous articles on design of experiments (DOE), many of which can be seen or ordered as reprints from www.statease.com.

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