# METHODS TO REDUCE OPTICAL COATING AND OTHER PROCESS DEVELOPMENT TIME AND COST WHILE INCREASING PROCESS STABILITY

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**Abstract**

The development of optical coating processes to meet a set of performance requirements can involve dozens of process parameters which need to be controlled and optimized. The Design Of Experiments (DOE) methodology will be described along with its application to the development of optical coating process parameters. DOE can be used beneficially not only in optical coatings but also in most processes such as for a product, a service, a task, and others.

## 1. Introduction

Optical coating processes require the control of a great many process parameters. At many times and places in the past, coating development has been far less than efficient and not always successful. Some of the World's industries, such as automobile manufacturing, have used and evolved process development and refinement tools which provide a maximum amount of process understanding and control with a minimum number of tests or experiments. "User friendly" software tools and texts like that of Schmidt and Launsby[1] are now available which make process optimization and stabilization more certain and less time consuming. The DOE methodology offers a systematic approach to process development and optimization. A key characteristic is that it gives a maximum amount of information and insight in process development for a minimum number of test runs. As a result, the time and cost of process development are reduced and the probability of success is increased.

In any process development, it is advisable to have the process flow diagram in mind and preferable on paper. We should then consider all of the parameters which can cause variations and effect the results. Some of these can be controlled; some cannot be controlled and are therefore noise; and some are the subject of further experimentation. There may be a great number of parameters that could be the subject of experiments. In such case, there are screening procedures in DOE methodology to determine, with a minimum number of experiments, which are the vital few that have the major influence on the results. These few parameters are then the subject of detailed experimentation and optimization.

## 2. The Problem and Solution

There has been a tendency in process development in the optical coating industry and others to vary *one variable parameter of a process at a time* to find the desired maximum or minimum result for that variable and then to do the same with the next variable, etc., etc. One problem with this approach is that it requires many test runs if there are many parameters to be optimized. The second problem is that *this* approach *may not find the optimum* result for the combined parameters which have been varied. Figure 1 illustrates this effect. If variable B were first optimized with respect to the desired result Y and then the optimization of A were started from that point, it would appear that the optimum point in A and B was at point X. However, the real optimum is at point Z. With the proper statistical sampling techniques of DOE, it is possible to much more closely locate the true optimum at

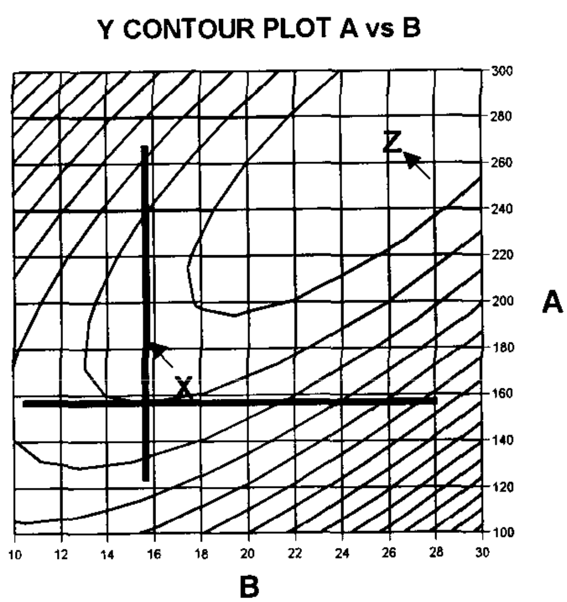


**Figure 1. Contour plot of result Y as a function of variables A an B.**

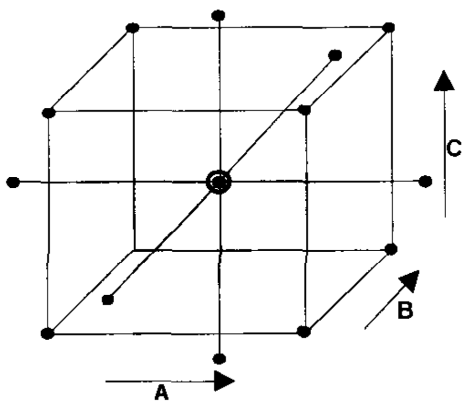


**Figure 2. CCD sampling scheme for 3 variables. Dots are sample points.**

point Z by only 5 sample points. For example, these might be data at points in the four corners of Fig. 1 and at the center point.

The case of Fig. 1 is for only two variables. Typically there are three or more critical parameters to be optimized. When there are three variables, it is still possible to show the distribution of sampling points graphically. Figure 3 shows the positioning of sampling points for one of the preferred DOE configurations known as the Box-Wilson or Central Composite Design (CCD)[1]. This is very efficient and flexible for second order modeling. Such a design also has rotatability so that the predicted response can be estimated with equal variance regardless of the direction from the center of the design.

Another frequently used design is the Box-Behnken Design shown in Fig. 3. These are potentially more efficient than CCD's for three factors (variables) and three levels. They allow estimation of linear and quadratic effects and all 2-way interactions. When the number of factors is greater than 4, the CCD would be more efficient. In both of these designs, the central or axial point is sampled 3 or more times to measure the repeatability of the data and allow the estimation of the standard deviation.

Experiments are then conducted at the conditions of each of the sample points and the results are recorded. The results are then processed in the DOE software[2] to fit (least squares) the data to a model for linear and quadratic effects and 2-way interactions. The model of the results can then be displayed in 2 and 3-dimensional graphics to aid visualizations of the process behavior. With the aid of these graphics, it is usually possible to find the values of each variable which will give the optimum process results.

## 3. An Example Case

We will illustrate the use of DOE with a fictitious example. Please note that this example is NOT TAKEN FROM REAL DATA, it is only for illustration purposes. We will imagine that we want to optimize the deposition of titania ($TiO_2$) using ion assisted deposition (IAD) with oxygen ($O_2$) and argon (Ar). The two major results desired are to have absorption (k) less than .001 and the spectral shift with humidity of less than 2 nm. There may be other results of secondary interest such as index of refraction and hardness, but adjusting for these could only be considered if the range of variables which satisfy the first two requirements leave some latitude to choose the best index and/or hardness results within that range.

We will further imagine that we have a conventional optical batch coater with heaters, electron beam gun (e-gun), ion gun, etc. What are the most important variables that could effect the desired results? From experience, we may know that some of these are: temperature, pressure, deposition rate, $O_2$/Ar mixture, ion current, ion voltage, etc. Our experience may allow us to bypass the screening experiments to determine which variables are important. The controls at our disposal are: deposition rate by e-gun power, ion gun current, temperature by heaters, and pressure and $O_2$/Ar mixture by mass flow controllers of the gasses to the ion gun. The flow is measured in Standard Cubic Centimeters per Minute (SCCM). Let us say that we know by experience that the temperature which would give the best results is the highest value that the equipment can provide. Therefore we will run the experiments at that temperature, eliminating it as a variable. Similarly, we know that the process runs most rapidly (and therefore economically) at the highest ion current that the source can provide. Again, we

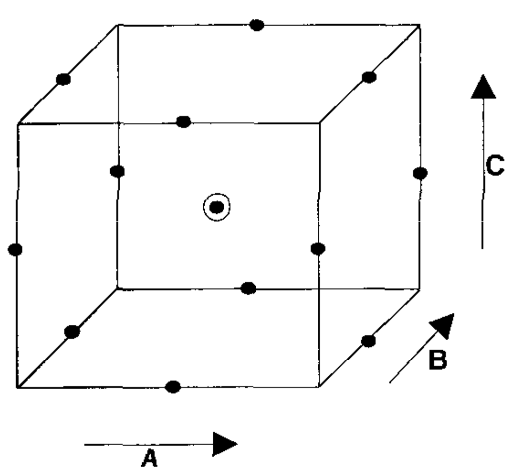


**Figure 3. Box-Behnken Design sample point scheme.**

| Column # | 1 | 2 | 3 | | RESULTS |
|---|---|---|---|---|---|
| Row # | RATE-A/S | Ar-SCCM | O2-SCCM | | k x 1000 |
| 1 | 2 | 20 | 40 | | 1.25 |
| 2 | 2 | 60 | 40 | | 0.75 |
| 3 | 10 | 20 | 40 | | 2.5 |
| 4 | 10 | 60 | 40 | | 2 |
| 5 | 2 | 40 | 20 | | 4 |
| 6 | 2 | 40 | 60 | | 0 |
| 7 | 10 | 40 | 20 | | 9 |
| 8 | 10 | 40 | 60 | | 0 |
| 9 | 6 | 20 | 20 | | 7.5 |
| 10 | 6 | 20 | 60 | | 0 |
| 11 | 6 | 60 | 20 | | 5.5 |
| 12 | 6 | 60 | 60 | | 0 |
| 13 | 6 | 40 | 40 | | 1.5 |
| 14 | 6 | 40 | 40 | | 1.3 |
| 15 | 6 | 40 | 40 | | 1.6 |

**Figure 4. Design sheet for experimental points in Box-Behnken Design and k x 1000 results of those experiments.**

will run at that current and eliminate another variable. The process pressure and $O_2$/Ar mixture will be affected by the flow rates of both gasses, but the pressure will also be influenced by the deposition rate due to the gettering of the reactive $TiO_2$. As a result of all of the above considerations, we conclude that the three independent variables that we will optimize are: Rate of deposition (measured in Angstoms per second (A/S) by a crystal monitor), Ar-SCCM, and $O_2$-SCCM.

We need to next decide what would be a reasonable range over which to experiment with each of these variables. Due to our experience with the coating chamber and equipment to be used, we choose 20 to 60 SCCM for both gasses and 2 to 10 A/S for the range of the rate.

| FACTOR | COEF | P(2 TAIL) | TOL | LOW | HIGH | EXPER | ACTIVE |
|---|---|---|---|---|---|---|---|
| Constant | 1.466667 | 0.002116 | | | | | |
| RATE-A/S | 0.9375 | 0.001747 | | 2 | 10 | 6 | X |
| Ar-SCCM | -0.375 | 0.05941 | | 20 | 60 | 40 | X |
| O2-SCCM | -3.25 | 4.48E-06 | | 20 | 60 | 40 | X |
| AB | 0 | 1 | | | | | X |
| AC | -1.25 | 0.002272 | | | | | X |
| BC | 0.5 | 0.070593 | | | | | X |
| AA | 0.079167 | 0.74169 | | | | | X |
| BB | 0.079167 | 0.74169 | | | | | X |
| CC | 1.704167 | 0.000666 | | | | | X |
| | | | | | | | |
| R Sq | 0.991461 | | | | | | |
| Adj R Sq | 0.976092 | | | | | | |
| Std Error | 0.436559 | | | | | | |
| F | 64.50843 | | PRED Y | | | 1.4667 | |
| Sig F | 0.000123 | | | | | | |

**Figure 5. Results of the analysis of the data in Fig. 4.**

We can now enter these choices in an appropriate DOE software program such as DOEKISS[2] to calculate the points to be sampled which satisfy the Box-Behnken Design (in this case). *Figure 4* shows such a design sheet. We then need to choose which interactions of the variables will be included in the model to which the results will be fit. In this case, we will include all linear and quadratic interactions (as seen in Fig. 5). When the 15 experiments have been run, we enter the results into the RESULTS column of Fig. 4 and then analyze the matrix with the software.

Figure 5 shows coefficients of the model (derived from the experiments) and all of the statistical detail such as the standard error of the least squares data fit. In the EXPER column, we can enter specific values for the three independent variables and use the software to compute the predicted value (Y) based on the model. We can see here that this point at the center of the parameter space sampled (6,40,40) does not meet our need for a k-value (x 1000) less than 1. There are many more details which can be gleaned from Fig. 5, but they are beyond the scope of this paper.

The DOEKISS software facilitates the display of the results from the fit of the data to the model in a variety of graphs. Our first choice in a case such as this is usually to use a three dimensional plot of each type of result (such as "Humidity Shift" and "k x 1000") with respect to each of the independent variables taken two at a time. There would be three such plots per result if we were to view the "cube" from each of the three axes. Our general choice is to examine each of these at the plane containing the center point of the design. . We see such a plot in Fig. 6 for the k-value as a function of $O_2$ flow rate and deposition rate. In Fig. 7 for the Humidity Shift as a function of both gas flow rates, it can be seen that the lowest shifts occur at the lowest gas flows

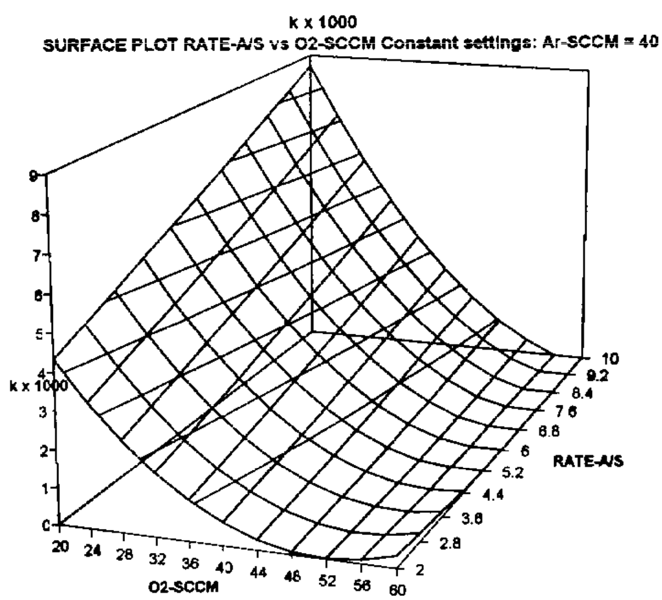


**Figure 6. Surface plot of k-value versus $O_2$ flow and deposition rate.**

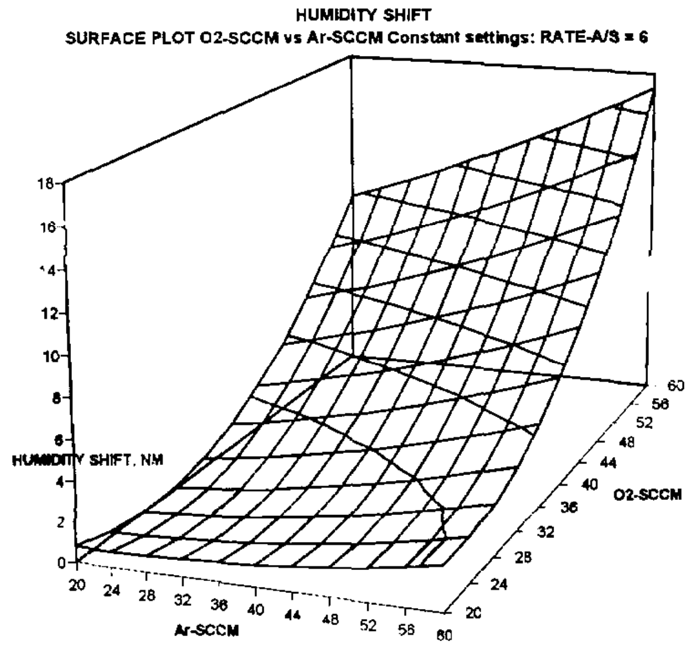


Figure 7. Humidity shift versus both gas flows.

(which result in the lowest chamber pressures). In Fig. 8 we see a similar plot for the k-value versus both gas flows. In this case, the lowest k-values are found at the highest gas flows. It can immediately be seen from Figs. 7 and 8 that the two results desired (of low humidity shift and low k-value) are in conflict, in that they "pull in opposite directions." The question to be resolved is whether there is a set of values within the range of variables where both results can be achieved.

We approach this question by using another graphic presentation, the Contour Plot. This is just a view from directly overhead of any of these plots, like a topographic map. In this case we choose to look down from the deposition rate axis to see the effects of the two gas flows on the k-value and the humidity shift. If there is an overlap between the regions of gas flow within some range of deposition rates that satisfy both of our objectives for these two results, we can solve the problem. Figure 9 is the contour plot of the humidity shift with gas flows at a Rate of 2 A/S. The lighter region meets the requirement of less than 2 nm shift. Figure 10 is the contour plot of the

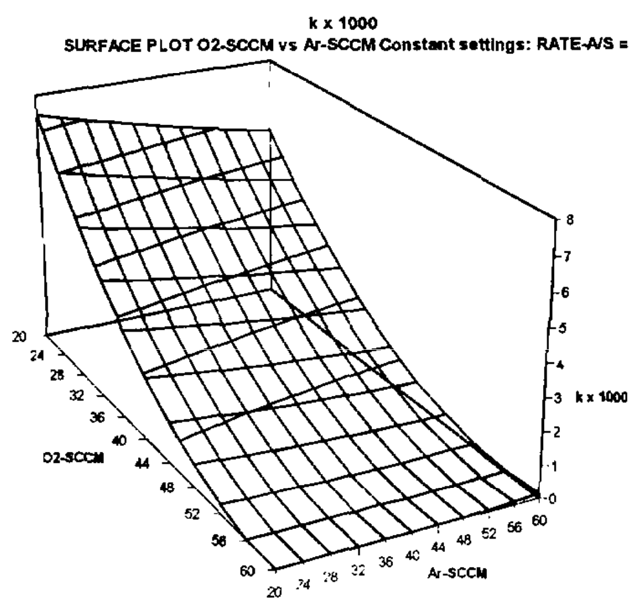


Figure 8. K-value versus both gas flows.

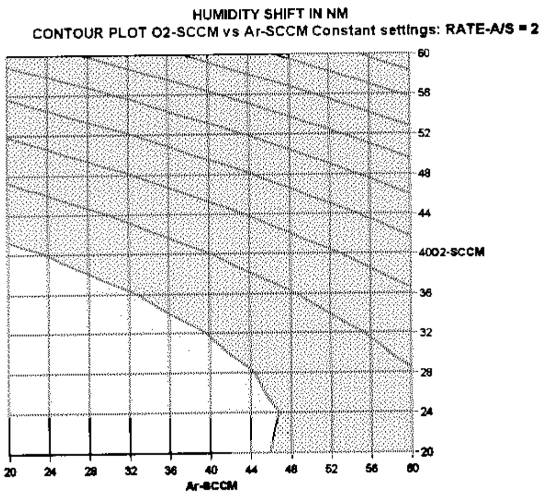


**Figure 9. Contour plot of the humidity shift with both gas flows showing acceptable region in white.**

k-value with gas flows at a Rate of 2 A/S. The light region is where k meets the requirement to be less than .001. It can be seen that the two white areas of Figs. 9 and 10 only overlap at a very small area in the vicinity of where the Ar flow is 20 and the $O_2$ flow is 41 SCCM. It was found from similar contour plots (not shown) of $O_2$ flow versus Rate at an Ar flow of 20 SCCM that there is no region of overlap at deposition rates greater than 2.5 A/S. We can conclude at this point that our result goals are only likely to be met in the variable region below 2.5 A/S in deposition rate, less than 24 SCCM of Ar flow, and greater then 41 SCCM of $O_2$ flow.

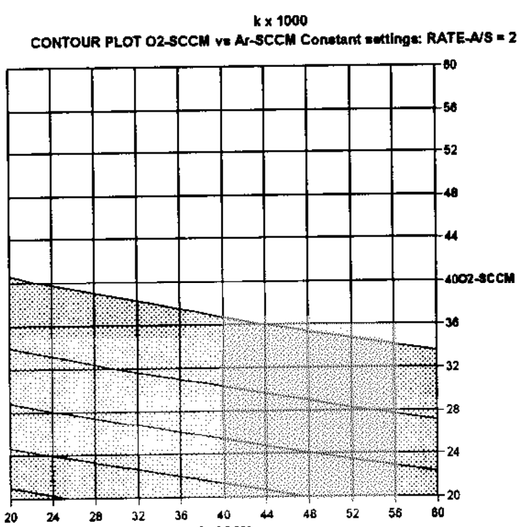


**Figure 10. Contour plot of the k-value with gas flows at a Rate of 2 A/S showing acceptable region in white.**

We might confirm these conclusions but an experimental test at these predicted values of the variables. If the results were satisfactory, we could consider that we are done with the DOE. If the results were not entirely satisfactory, we could use the additional set of data in the DOEKISS program as Historical Data to compute a further refined model which could even more nearly coincide with the experiments in the region of interest. However, in this particular case, we can see in Fig. 4 that the first experiment was essentially very near this variable point (2,20,40) and gave a result of k x 1000 = 1.25. Therefore, this particular would not be expected to add significantly to the data base. It appears that a test run at a rate of 2 A/S with 16 SCCM of Ar and 43 SCCM of $O_2$ would be more likely to satisfy the requirements while confirming the predictions. The data from such a test run could also be reprocessed as described above.

## 4. Summary

We have demonstrated the usefulness of the DOE methodology in finding the characteristics of a process and the optimum parameters to achieve desired results. The methodology is systematic and based on solid statistical concepts. The practical use of this tool or system is not dependent on deep understanding of the details of statistical mathematics. This like the fact that one can drive an automobile without being an experienced mechanic. The tools are "user friendly". We have shown some of the available graphics which aid in process visualization and how they might be used to gain insight and make process decisions. This methodology gleans to most information practical from a minimum number of tests. This has proved to be a great aid to efficient and successful process development.

## 5. References

1. S. R. Schmidt and R. G. Launsby, ***Understanding Industrial Designed Experiments***, Air Academy Press, Colorado Springs, CO, USA (1994).
2. ***DOEKISS*** Software, Digital Computations, Inc. and Air Academy Associates, LLC, Colorado Springs, CO, USA (1997).