

Optimizing Processes with Design of Experiments

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TECHNICAL PRIMER

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Introduction

Do you need an efficient way to determine which process changes will yield the greatest gains?

Has your organization outgrown one-factor-at-a-time experimentation?

In the rush to get answers, are you forced to cut corners, limiting your understanding of the real drivers of process robustness, effectiveness and efficiency?

Is the time devoted to attempts at problem-solving curtailing opportunities for innovation and process improvement?

If you answered yes to any of these questions and you have insufficient data to address your questions, then read on to see how world-class design of experiment (DOE) capabilities in JMP are helping users gain insights to fix such issues quickly and permanently, giving their organization a competitive edge now, and the bandwidth to innovate for future growth. If you already have sufficient existing data, you might also want to read the companion technical primer, *Improving Processes With Statistical Models*.

DOE is helpful when you may not have the appropriate data to solve a problem – you may have collected data for a different purpose so it is not directly relevant, or key variables may have been varied in the same way at the same time. Sometimes the processes you are investigating are so new that there is limited prior data available. In these situations, JMP for DOE helps users define the most cost-effective plan to collect new, relevant data and analyze it simply and quickly.

Through real-world case studies, you can discover best practices for defining new, cost-effective data collection plans using recent advances in DOE, statistically model the important patterns of variation in the resulting data, and visually interact with these models to identify optimum and robust operating conditions.

How can design of experiments help my business?

Companies gain value from JMP for DOE in a number of ways. One company couldn't predictably scale up and transfer into production processes for making new products. This resulted in delayed product launches and poor predictability of supply once in production. The organization's engineering practice was to vary one factor at a time (OFAT) to try and fix problems. Employees lacked DOE knowledge and considered the learning overhead too high.

To reduce the learning threshold and perceived complexity of getting started with DOE, an application customized to the company's own terminology and engineering language was developed. This enabled their engineering community to adopt DOE easily, and they are now scaling up and transferring process right the first time. Process scale-up and transfer is now predictable and efficient, and engineers at many production plants worldwide have adopted DOE.

Compared with the old approach of varying one factor at a time, the company is now optimizing and scaling up production processes with fewer individual experimental runs, saving an estimated US\$3 million per production site per year in reduced experimental effort alone. There is huge upside from getting to market faster and more predictably.

Another company needed to double the capacity of a product line to meet growing demand. They had limited understanding of the key process steps, a large number of potentially important variables that could be influencing throughput, and limited budget for experimentation. They used a definitive screening design – an innovative new design of experiments – to minimize the experimental outlay. Statistically modeling the resulting data delivered the know-how to help double the production rate with no capital investment. That's right – twice the product, no additional capital outlay! The use of a definitive screening design saved hundreds of thousands of dollars from the development budget, and the development group gained internal credibility.

Background to design of experiments

DOE was pioneered in the 1920s by Sir Ronald A. Fisher at England's Rothamsted Experimental Station. Fisher first applied DOE to increase crop yields in agriculture; ever since, DOE has played a major role in increasing agricultural production (see Figure 1).

US corn yields

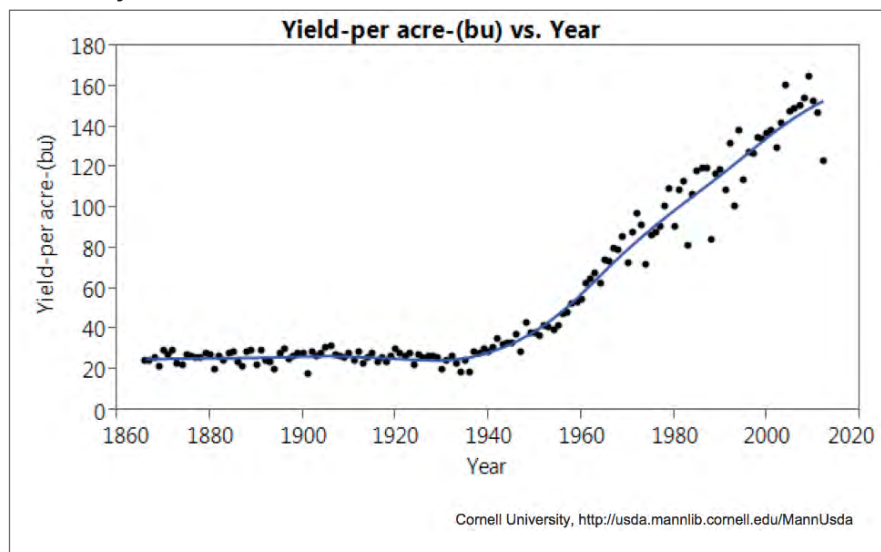


Figure 1: Increasing agricultural yields since advent of DOE

Fisher introduced four DOE principles:

1. **Factorial concept** – varying all the factors together using a factorial grid rather than varying one factor at a time.
2. **Randomization** – randomizing the order of the individual experimental runs in the factorial grid to avoid bias from lurking (unidentified) variables.
3. **Blocking** – to reduce the noise from nuisance variables.
4. **Replication** – to reduce the potential masking of experimental factors due to noise (unpredictable) variation.

The DOE method has been improved and enhanced in the ensuing decades:

- Later in the 1930s, Frank Yates simplified the analysis of DOE data by introducing the Yates algorithm, which is the reason why many designs are still coded on a -1 to +1 scale.
- In the 1940s, DJ Finney introduced the fractional factorial design, which allowed many factors to be investigated at half or even a quarter the cost of Fisher's factorial design.
- In the 1950s, George Box and others deployed DOE in chemistry and industrialized its application.
- Each decade since has seen improvements in the DOE method. Within the past few years, the definitive screening design was invented by Brad Jones from SAS and Chris Nachtsheim from the University of Minnesota.

DOE methods are now applied widely in design, development, scale-up, manufacturing and “quality by design” (QbD) areas to ensure that key questions are answered predictably and economically.

When to apply DOE

DOE can potentially help any time you have technical or business problems and insufficient data to answer your questions – particularly if:

- You have more problems than time available.
- The time to solve problems is unpredictable or you find yourself repeating or performing a large number of cycles of learning.
- You feel you are forced to cut corners and make decisions on incomplete information.
- Products and processes are defined or transferred with incomplete understanding of how they work.
- You sometimes get stuck in a reactive, vicious cycle where fixing problems with existing products and processes limits your time for innovation and new process or product development.

Then, providing you have the ability to collect new data through active intervention, DOE will help define the data you need to collect and, by analyzing the resulting data, answer your questions efficiently and effectively.

Why apply DOE?

The DOE method will help you optimize products and processes predictably and quickly. It will enable you to make better, more informed decisions, bringing predictability to decision making, thereby reducing risk to your business.

You will be able to transfer products and processes with a better knowledge of how they work. Because of this, there will be less need to improve or “fire fight” products or processes when in production, which makes room for more R&D time to be spent in innovation, new product and process development.

DOE technicalities

The properties of products and processes are usually affected by many factors or inputs. For example, Figure 2 illustrates a chemical process where the chemist believes there are five inputs that may be responsible for causing variation in the two responses or outputs. When determining the set of inputs, statistical analysis of prior data can help identify the factors to investigate. Otherwise, it is better to err on the side of caution and include as many factors as you think might have an effect on one or more of your responses, provided you have sufficient budget.

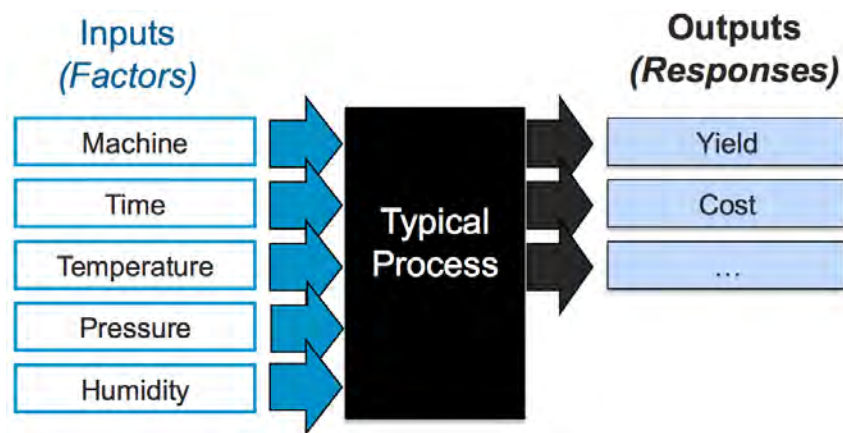


Figure 2: A chemical process

The traditional experimental approach

A common scientific and engineering approach is to experiment by varying one factor at a time (OFAT). The OFAT approach is illustrated using just two of the factors in Figure 2 (temperature and time) to investigate their effect on one of the responses (yield). Prior knowledge indicates that it is best to investigate temperature over the range 500 to 550 degrees C (900 to 1,000 degrees F) and to vary time over the range 500 to 1,300 minutes.

The first sequence of experiments were performed by keeping temperature fixed at 520 degrees C and varying time within the range of 500 to 1,300 minutes in increments of 100 minutes. This resulted in the yield curve in Figure 3; a suggested time of 1,100 to 1,200 minutes is required to maximize yield.

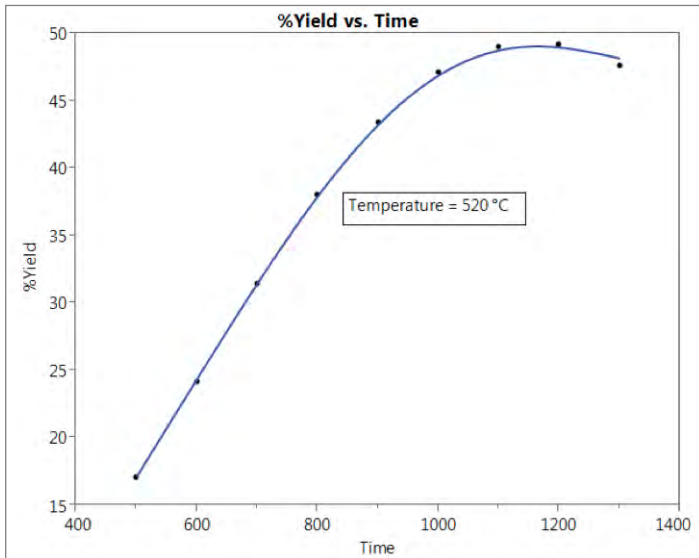


Figure 3: Relationship between yield and time at 520 degrees Celsius

The next set of experiments were performed by varying temperature from 500 to 550 degrees C in increments of 10 C (50 degrees F) while keeping time fixed at 1,100 minutes. (1,100 minutes was favored to a slightly higher value in order to keep the cost of running the process lower).

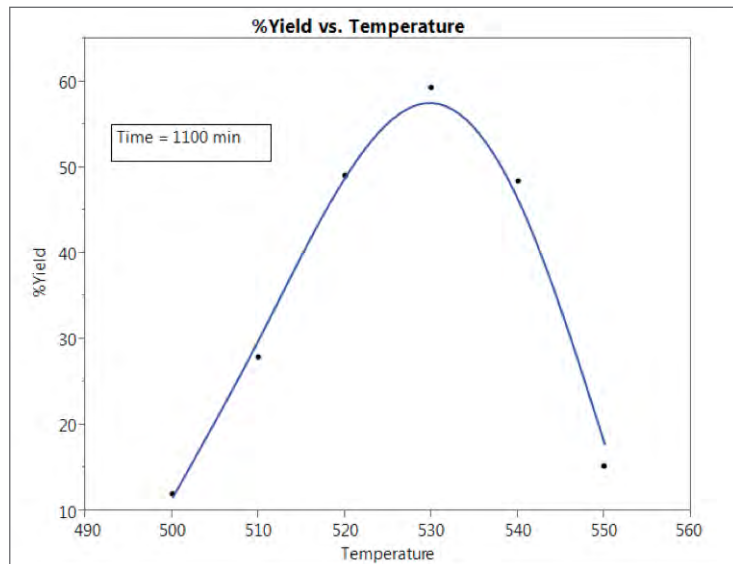


Figure 4: Relationship between yield and temperature at 1,100 minutes

The best setting of temperature at 1,100 minutes is 530 degrees C, which resulted in a yield of just under 60 percent. Figure 5 is a plot of temperature against time using a coloring gradient for the value of yield at each experimental point. It shows highest yield of 59.3 percent at 1,100 minutes and 530 C. It also indicates a lot of white space, which is a concern. This white space indicates we know very little about what yield to expect when:

- Time is less than 1,000 minutes and temperature is greater than 530 degrees C.
- Time is less than 1,000 minutes and temperature is less than 510 degrees C.
- Time is greater than 1,200 minutes and temperature is greater than 530 degrees C.
- Time is greater than 1,200 minutes and temperature is less than 510 degrees C.

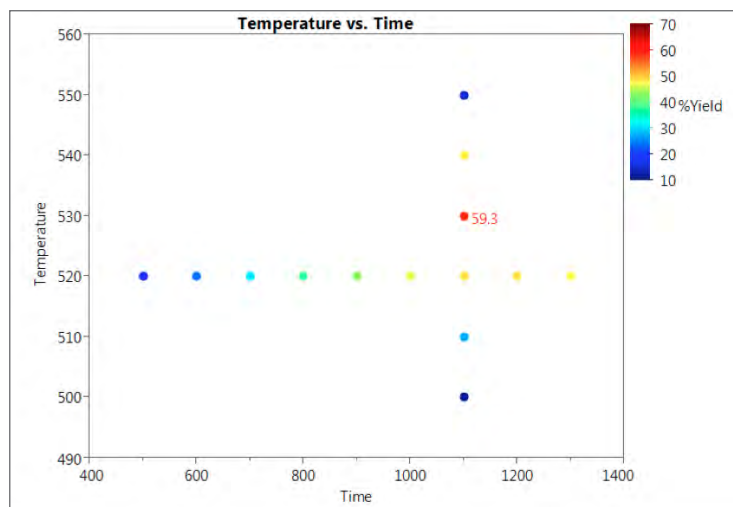


Figure 5: Time vs temperature with experimental points colored by yield

When we learn that a temperature of 550 degrees C and a time of 500 minutes produces a higher yield of 66.5 percent – at significantly lower cost – the risk of experimenting in a series of one-dimensional portions is self-evident.

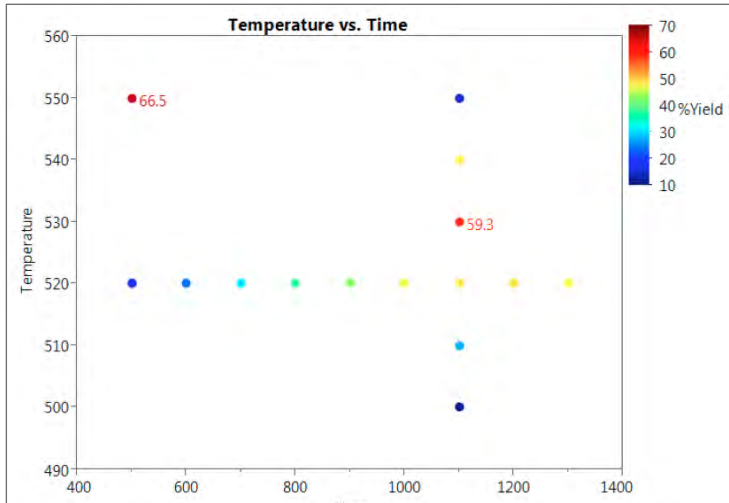


Figure 6: A better yield is obtained at 550 degrees C and 500 minutes

OFAT approaches frequently lead to suboptimal solutions. This is because OFAT assumes the effect of one factor is the same at each level of the other factors, i.e., that factors do not interact. Unfortunately, in practice, factors frequently interact, and the chances of two or more factors interacting increases the greater the number of factors investigated and the wider the range over which factors are varied.

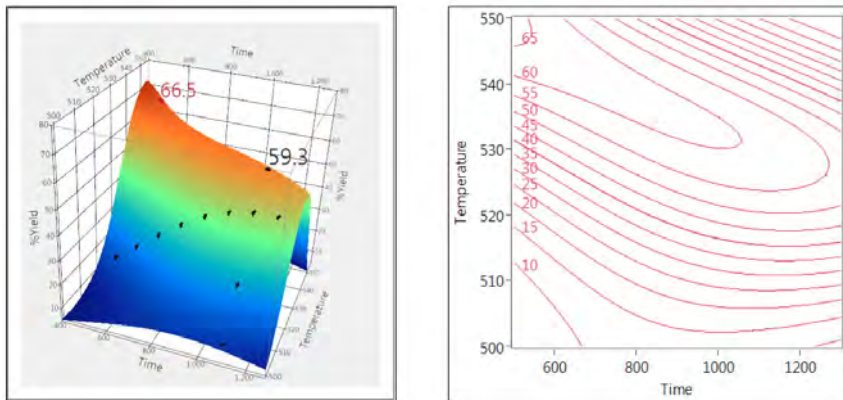


Figure 7: Relationship among yield, time and temperature

The 3-D plot and contour diagram in Figure 7 illustrate the interactive effect of time and temperature on yield with the ridge effect that runs from time and temperature combinations of 1,200 and 528 with a yield of 56 percent, to 900 and 536 with a yield of 61 percent, to 500 and 550 with a yield of over 65 percent. The effect of this interaction (or ridge) is that the best setting for temperature chosen by OFAT depends upon the value of time.

For example, if time is fixed at 1,000, the best setting of temperature is 534, resulting in a yield of slightly more than 60 percent. Whereas, as we have already seen, if time is fixed at 500 the best setting of temperature is 550, resulting in a yield of slightly more than 65 percent. In the presence of interactive effect of factors on the response, the solution identified by OFAT depends on your starting point, and it is only by chance that the global optima is determined.

DOE provides the most efficient and effective way of investigating relationships between factors and responses. The efficiency and effectiveness of DOE compared with OFAT increases the greater the number of interactive effects between factors.

The DOE needed to investigate the effects of time and temperature on yield, and to find the global optima, is given in Figure 8. This is a 3x3 grid of points in time and temperature, with three additional replicates of the center point to give a baseline for noise or uncontrolled variation. These 12 rows in the resulting data table indicated in Figure 8 give a balanced distribution of combinations of time and temperature throughout the experimental space indicated in Figure 5. By statistically analyzing the resulting data and dependence of yield on time and temperature, we have no gaps in our knowledge. Analyzing the resulting data with multiple regression yields the 3-D surface and contour plot depicted in Figure 7, which show that the best place to operate is time of 500 minutes and temperature of 550 degrees C. Further improvement is implied if we go to a lower time and a higher temperature, but an additional cycle of experimentation would be needed to achieve this gain in a reliable manner.

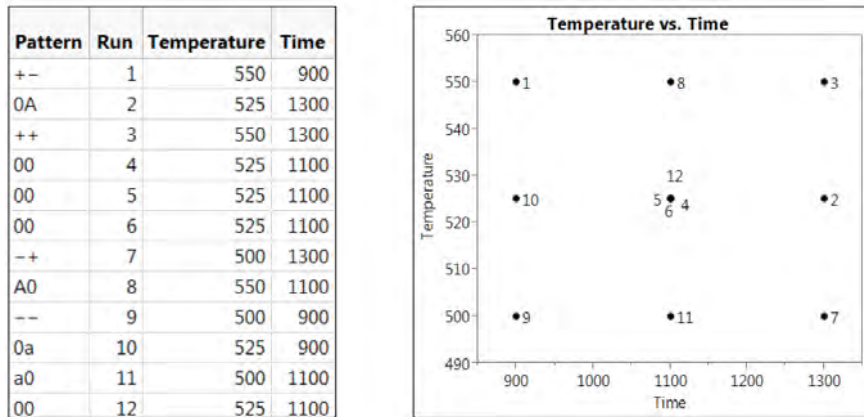


Figure 8: DOE in time and temperature

Learning is incremental

Figure 9 illustrates that we typically start with data or a theory that is analyzed to help assess our situation or theory, which typically leads to more questions that require collection of new data via DOE to provide answers.

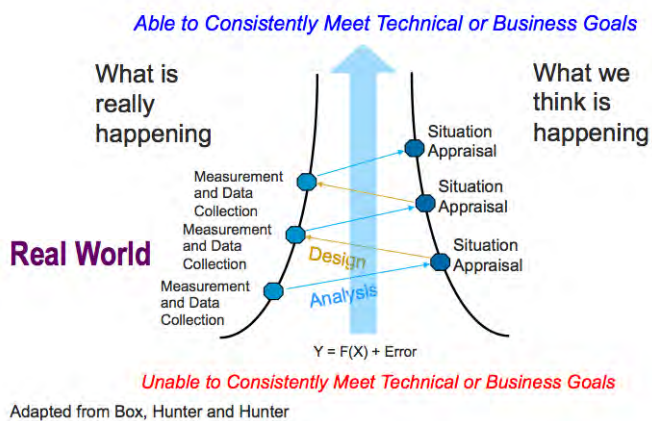


Figure 9: Learning is incremental

If we have a large number of factors and utilize OFAT approaches, we may perform many iterations or cycles of learning, sometimes a product or process may come back to us from production for additional cycles of learning to fix a product or process issue that would not occur if we were able to develop and transfer products and processes with a better understanding. Classical DOE methods – as developed by Fisher, refined by Finney and industrialized by Box – will reduce the total number of learning cycles relative to OFAT, and increase the predictability of R&D. Modern advances in DOE such as definitive screening by Jones and Nachtsheim may further reduce the number of cycles of learning.

By extracting the required information from fewer cycles of learning and doing so predictably, JMP for DOE delivers the information needed to make the correct decisions and increases the predictability of getting to those decisions with a limited budget and time.

Data-driven DOE: Integrating observational data

Figure 9 illustrated that learning is incremental. At the start of the DOE learning process, you may have existing data, in which case effective statistical modeling of that data may aid the design of the next DOE. In particular, analysis of prior data may help decide which factors to include in your DOE and the range over which to vary them.

Existing data may be messy, which makes it difficult to correctly extract information on the factors and factor ranges. Messy data issues may include the factors being related (e.g., an increase in one X results in increase or decrease of another X); some of the data cells may be recorded incorrectly; other cells may be empty or missing. JMP statistical discovery ensures that you can extract meaning from your messy data to identify the potential factors and factor ranges. Integrated with modern DOE methods, we reduce total learning time, effort and cost.

Case Study 1: Driving new product introduction by developing robust processes for hard-to-manufacture products

A specialty chemicals supplier of pigments to liquid crystal display (LCD) manufacturers is struggling to manufacture enough pigment to required specifications to meet customer demand. To achieve sharp displays, the pigment particles must be milled down to less than 200 nanometers, and the time taken to do this is extremely variable. The milling stage is energy-intensive and a bottleneck, and the long mill time is incurring excessive energy cost and affecting throughput. The company needs a faster process or additional milling equipment to run in parallel with existing equipment.

Figure 10 shows an upward trend in the time to mill to less than 200 nanometers for recent production batches of pigment. To avoid the capital cost of adding additional milling equipment, we need to get mill time below five hours, which appears to be a challenge; none of the prior batches has achieved this goal.

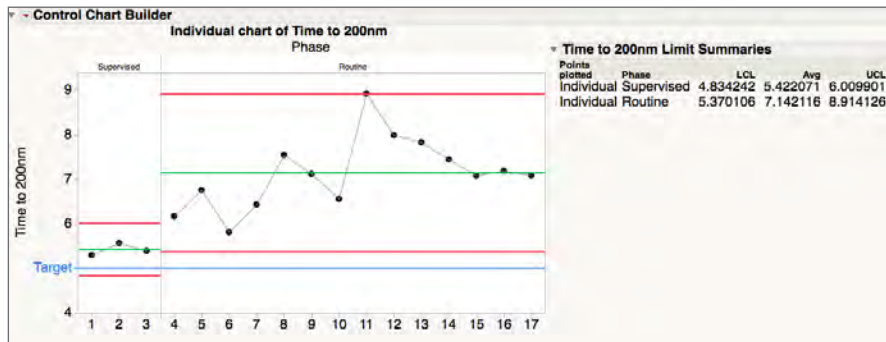


Figure 10: Time to mill of first 17 batches

OFAT was used to develop the process initially, but because the process is taking too long, the company needs to repeat earlier development work to try and learn how to speed the process. In addition to the cost of repeating prior cycles of learning, the current process incurs a high energy cost due to long milling time and does not provide enough material to meet the demands of LCD manufacturers.

If the milling process cannot be operated to the required speed of five hours or less to mill down to 200 nanometers, the company will need to purchase a second milling machine, and still tolerate the ongoing cost of an energy-inefficient process. This will reduce profitability, the availability of capital to invest in other projects, and dividends returned to investors.

Milling is carried out in a horizontal bead mill. This is a chamber filled with beads, through which the dispersion of pigment is passed. The beads in the chamber are agitated at high speeds to grind down the pigment particles.

Analysis of data from the prior 17 production runs using a bootstrap forest identified the top factors to investigate with DOE.

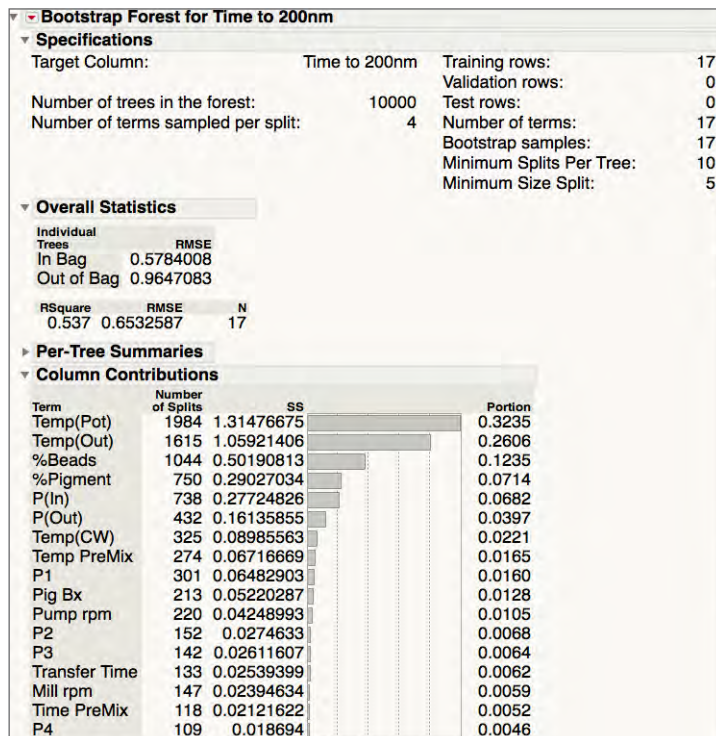


Figure 11: Input variable selection

Engineering judgment indicated that output temperature [Temp(Out)] is correlated with Temp(Pot) and could not be the driver of variation in mill time. Temp(Out) was therefore left out of the DOE. P1 was also excluded, and a cut point at Temp PreMix was used based on the need to limit total cost of experimentation. Inputs from Temp PreMix and above in Figure 11, with the exception of Temp(Out) and P1, were included in the DOE.

A definitive screening design in seven factors was designed, which resulted in 17 DOE combinations. This was deemed the most efficient way of progressing, as it allows screening for the critical factors, and - provided no more than three factors are important - then interactive and nonlinear effects can be modeled to optimize the process without the need for additional experimentation.

Figure 12 contains the completed worksheet for the 17-run definitive screening design in seven factors. Figure 13 shows the profiler and 3-D plot of the statistical model resulting from the DOE. Just three of the seven factors had an important effect on mill time to 200 nm – Temp(Pot), %Beads and %Pigment.

The settings of these three factors needed to minimize mill time to 200 nm are also indicated on Figure 13: Temp(Pot) of 50 C, %Beads of 68.8 and %Pigment of 13.5 with a predicted mill time of 2.6 hours (95 percent confidence interval of 2.2 to 3.0 hours). Three confirmation runs at these settings verified the time to mill to 200 nm was now well below the upper limit of five hours (see Figure 14).

8/0 Coils								
17/0								
	Temp(Pot)	Temp CW	%Beads	%Pigment	P(In)	P(Out)	Temp PreMix	Time to 200nm
1	50	11.25	70	15	0.9	1.3	25	6.2
2	35	7.5	70	15	1.15	0.7	25	11.3
3	50	7.5	70	13.5	0.9	1	50	2.5
4	35	7.5	65	15	0.9	1.3	50	10.3
5	35	15	70	14.25	0.9	0.7	50	9.4
6	50	7.5	65	14.25	1.4	1.3	25	3.6
7	35	15	67.5	13.5	0.9	1.3	25	7.9
8	50	7.5	67.5	15	1.4	0.7	50	4.9
9	35	15	65	15	1.4	1	25	10.8
10	50	15	65	13.5	1.15	1.3	50	4.1
11	35	7.5	70	13.5	1.4	1.3	37.5	8.0
12	35	11.25	65	13.5	1.4	0.7	50	9.2
13	42.5	11.25	67.5	14.25	1.15	1	37.5	6.1
14	42.5	7.5	65	13.5	0.9	0.7	25	6.5
15	50	15	65	15	0.9	0.7	37.5	5.1
16	50	15	70	13.5	1.4	0.7	25	2.5
17	42.5	15	70	15	1.4	1.3	50	8.8

Figure 12: Completed worksheet for definitive screening design in seven factors

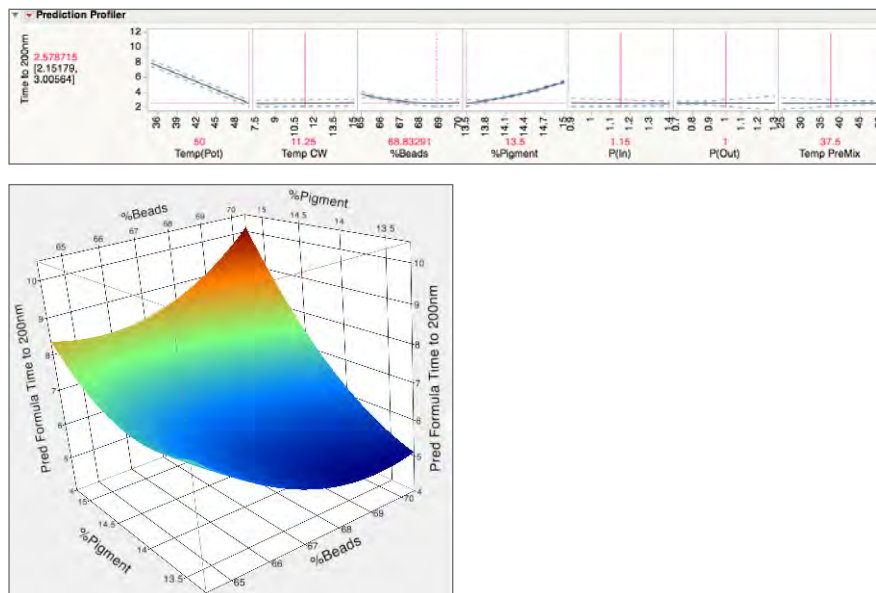


Figure 13: Profiler and 3-D plot of statistical model

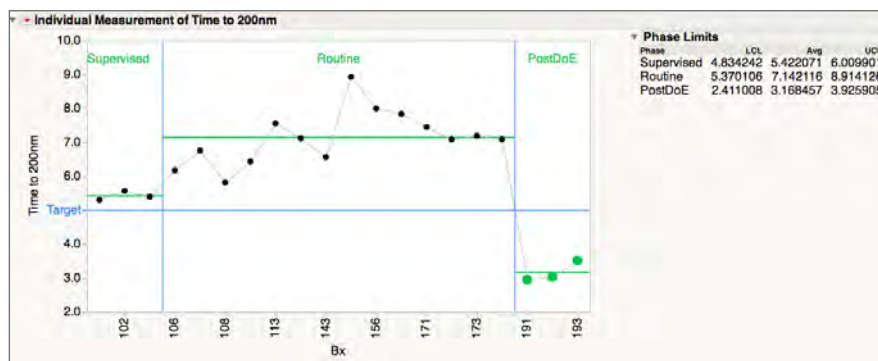


Figure 14: Three confirmation runs

Summary

Data mining of prior data provided the variables to input to a definitive screening design, which provided an efficient experimental plan. Statistically modeling the resulting data helped identify a permanent solution. The problem was solved quickly, saving hundreds of thousands in the development budget and enhancing the credibility of the site as a location for cost-effective, high-value manufacturing.

Case Study 2: Optimizing a manufacturing process to increase yield and product quality

A semiconductor manufacturer has been experiencing problems in operating a low-pressure chemical vapor deposition (LPCVD) process in a furnace tube to deliver a new product specification. It processes four lots of wafers, each of 24 wafers, each run of the equipment.

During the process, a new layer of silicon nitride is deposited on each wafer, with the goal of each wafer receiving silicon nitride of uniform target thickness and refractive index. The process operates by heating the furnace tube and having gases flow from one end of the tube to the other, passing over and between the wafers. It is run according to a recipe that controls things like the rate of heating, temperature, gas flows and pressures.

The process is monitored with four test wafers, one per lot for each run of the process. Each test wafer has 49 measurement locations within the wafer. Figure 15 shows a plot of film thickness and refractive index for each test wafer. The x-axis identifies the run ID and test wafer ID within run. The y-axis plots the value of film thickness or refractive index for each of the 49 measurement locations within each test wafer. Each graph also shows the specification limits and target.

The primary issue with film thickness is that it is not centered within the specification window, resulting in every wafer being out of specification. There is little wafer-to-wafer variation and moderate (relative to specification range) within-wafer variation. Refractive index is also not centered within the specification window, has wafer-to-wafer variation and low within-wafer variation (relative to specification range). OFAT approaches to improving the situation have failed to deliver any significant improvement.

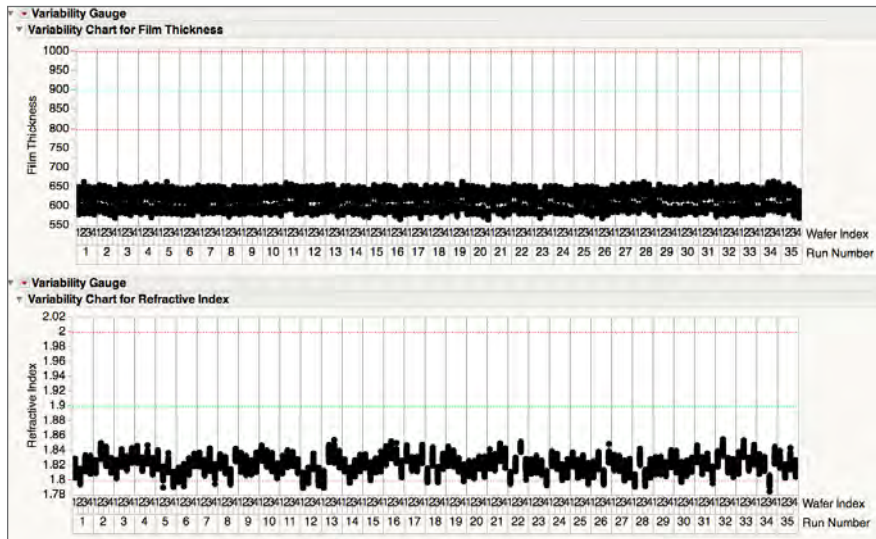


Figure 15: Performance relative to specification

Continuing with OFAT is resulting in ineffective solutions and repeat work. Inability to solve the furnace tube issue is causing low yield of desired functional devices end of line. The company was unable to produce enough devices of required function with existing equipment. Unless the process could be made to consistently deliver in-specification product, they will need to buy additional equipment. This approach also means we can expect to continue producing many lower-functioning devices that command a lower price. The business impact is reduced profitability and availability of capital to invest in other projects.

Prior to designing an experiment to understand the drivers of silicon nitride thickness and refractive index, the process engineer wanted to understand the pattern of variation in silicon nitride and refractive index within wafer, between wafers within a lot and between lots. To enable this, he produced a wafermap trellis as illustrated in Figure 16. Each column within the graph trellis represents a test wafer within a production run, and a row within the trellis represents a production run. The intersection of a row and column within the trellis is a wafer, and the value of the test measurement at each of the 49 test locations within a wafer is represented by a colored circle.

In the case of silicon nitride thickness, there is little between-wafer and between-run variation. The key component of variation is within-wafer, and a radial pattern is illustrated. Thickness measurements are lowest at the outer edge (outer zone) of the wafer, with the highest thickness measurements as we move inward (zone 2). Thickness reduces as we move inward to zone 1, and further reduces at the center. Thickness is being deposited with a radial pattern (similar to a wave pattern emanating from the center of a wafer).

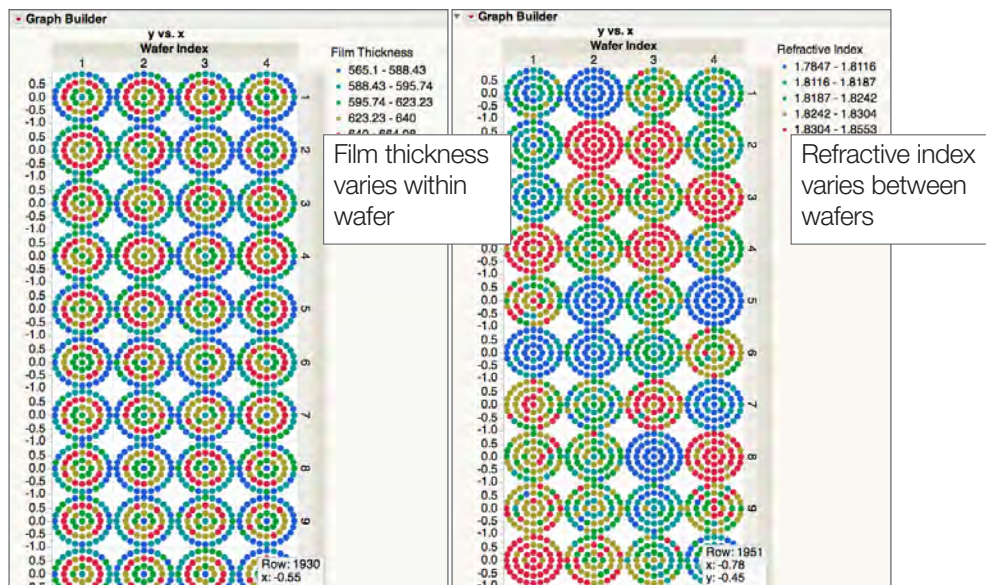


Figure 16: Wafermap trellis

The wafermap trellis of refractive index indicates some between-wafer variation within run and between-run variation, but very consistent measurements within a wafer.

Based on the patterns of within-wafer, between-wafer and between-run variation, the process engineer defined the responses (outputs) to be measured and optimized as mean thickness at the center, zone 1, zone 2, and outer zone along with mean refractive index.

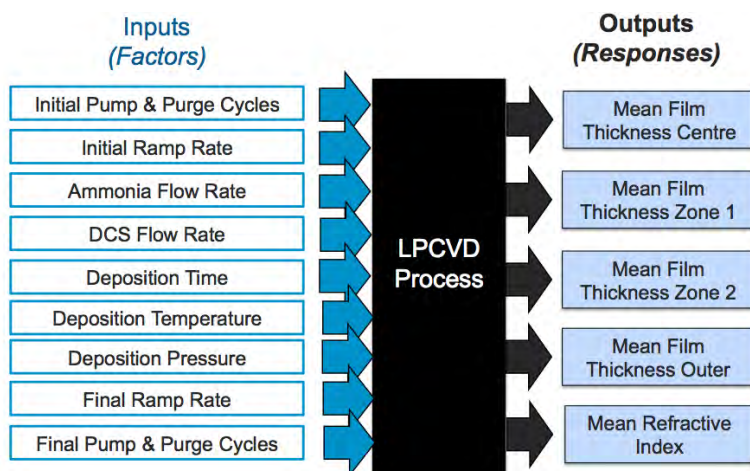


Figure 17: Inputs and outputs

These are summarized in Figure 17 along with the factors (inputs) to be varied in the DOE.

A definitive screening design in nine factors was designed, which resulted in 21 DOE combinations. This was deemed the most efficient way of progressing, as it allows screening for most important factors. If three or fewer factors are important, then interactive and nonlinear effects can be modeled to optimize the process without the need for additional experimentation.

Figure 18 contains the completed worksheet for the 21-run definitive screening design in nine factors. Figure 19 shows the profiler of the statistical model resulting from the DOE. Just three of the nine factors had an important effect on the five responses – deposition time, temperature and pressure.

Definitive Screening Completed Worksheet												
140- ±210	Initial Pump and Purge Cycles	Initial Ramp Rate	Amonia Flow Rate	DCS Flow Rate	Deposition Time	Deposition Temperature	Deposition Pressure	Final Ramp Rate	Final Pump and Purge Cycles	Ave Refractive Index	Ave Film Thickness Centre	
1	5	5	350	60	20	200	700	320	500	5	2.058	695.9
2	5	5	500	60	40	300	700	260	350	5	1.803	702.1
3	5	5	500	80	20	300	700	320	200	0	2.039	682.7
4	5	5	200	80	40	200	770	320	200	5	2.058	602.8
5	2.5	350	70	30	250	770	290	350	2.5	1.834	594.1	
6	0	500	60	20	300	770	260	500	0	1.599	615.8	
7	2.5	200	60	20	200	700	260	200	0	1.622	506.9	
8	2.5	500	80	40	300	840	320	500	5	2.043	1116.0	
9	5	200	80	40	250	700	260	500	0	1.597	623.4	
10	5	200	70	20	300	840	260	200	5	1.595	932.5	
11	0	200	80	20	300	700	290	500	5	1.818	766.3	
12	0	350	80	40	300	840	260	200	0	1.596	609.6	
13	5	200	60	30	300	840	320	500	0	2.065	1126.4	
14	0	500	80	30	200	700	260	200	5	1.604	498.2	
15	0	200	80	20	200	840	320	350	0	2.057	916.5	
16	0	500	70	40	200	700	320	500	0	2.064	689.6	
17	0	500	60	20	250	840	320	200	5	2.048	1004.0	
18	0	200	60	40	200	840	260	500	5	1.607	710.8	
19	5	500	80	20	200	840	260	500	2.5	1.592	698.9	
20	0	200	60	40	300	700	320	200	2.5	2.037	863.1	
21	5	500	60	40	200	840	290	200	0	1.838	782.4	

Figure 18

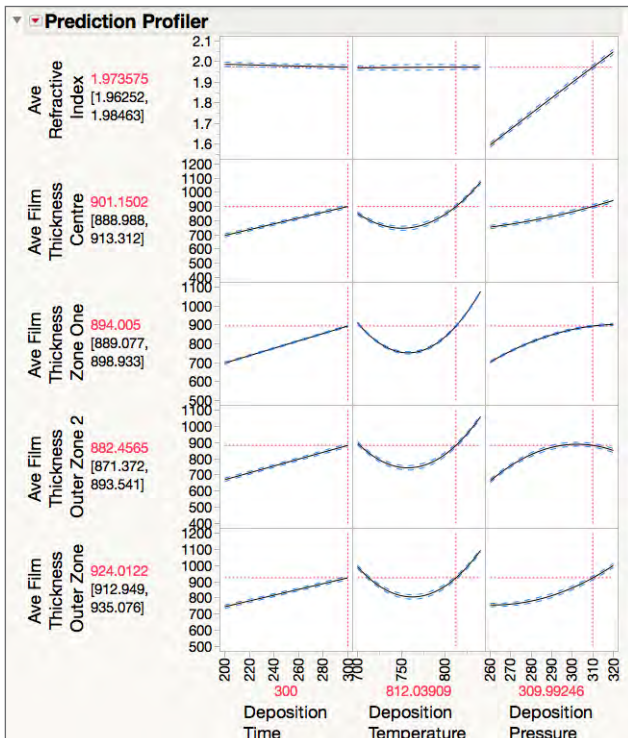


Figure 19

The settings of these three factors needed to center the five responses within their specification range are also indicated in Figure 19:

- Deposition time of 300.
- Deposition temperature of 812.
- Deposition pressure of 310.

These settings produce the following predicted responses:

- Predicted average refractive index of 1.97.
- Average thickness at center of 895.
- Average thickness at zone 1 of 900.
- Average thickness at zone 2 of 880.
- Average thickness at outer zone of 926.

The new process recipe for deposition time, temperature and pressure was implemented cautiously at first and validated after observing that three production runs delivered wafers all within specification. Figure 20 confirms the improvement for the first 20 production runs with the new recipe. Film thickness is centered very close to target, and none of the measurements within a wafer are close to the specification limits.

The process is therefore highly capable of consistently depositing silicon nitride to the required thickness. Refraction index is not quite centered, and wafer-to-wafer variation is evident. However, all refractive index measurements are well within the specification limits, meaning the process is highly capable with regard to refractive index. Although it is possible to improve refractive index further, there is little benefit in doing so.

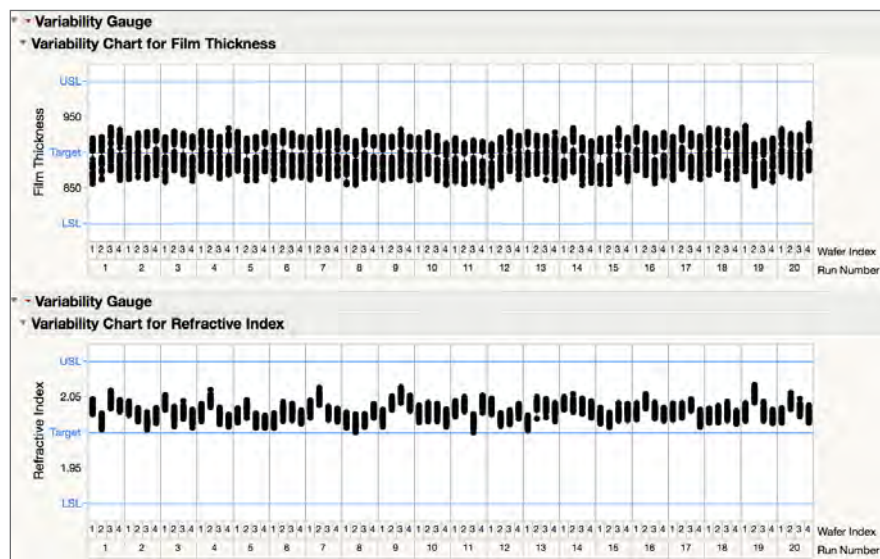


Figure 20: Performance post-improvement

Summary

Exploring prior data helped define the output variables to optimize with DOE. A definitive screening design provided an efficient and effective experimental plan with which to provide the data needed to understand the furnace tube process. Statistically modeling the resulting data provided a solution that ensured future wafers have the desired properties for silicon nitride thickness and refractive index. Further improvements are possible but not necessary.

The problem was solved quickly and permanently, saving hundreds of thousands in development budget by removing the need for now-redundant cycles of learning that are typically required with OFAT. The company assured product supply, enabling sales projections, and saved millions of dollars in additional processing equipment that is no longer needed.

Conclusion

In the interest of brevity, just two examples were presented. If you are able to experiment or actively intervene in your process, you can similarly use DOE to create the data you need to statistically model your process and increase real understanding. This paper has attempted to explain:

- What DOE is.
- The types of problems that benefit from DOE.
- Simple and effective ways to apply DOE.
- How to extract knowledge from DOE.
- How to present and communicate models and resultant knowledge from DOE to other stakeholders.
- How to optimize products and processes better, faster.

DOE might help you or your company to:

- Do more effective work in less time.
- Increase understanding of your products and processes.
- Optimize products and processes more efficiently.
- Increase the predictability of design, development and engineering projects.
- Deliver a competitive edge.
- Increase the availability of money for further innovation and improvement.

Questions? Please contact the JMP office nearest you: jmp.com/contact

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