

Application of Design of Experiment for Modelling of Etching of Ceramics

Ting H.T., K.A. Abou-El-Hossein, and Chua H.B.

Abstract—Design of Experiment is a useful approach in experimentation. DOE is a planned technique for determining the effect of the input parameters of a process on its outputs. It is a quality improvement method that can be employed to dramatically improve industrial products and processes. Through its use, it is possible to isolate the cause and effect linkages between product or process variables and the resulting output measures of function, quality, cost and performance. DOE is divided into simple comparison factor, two level factorial design, general factorial design and response surface method RSM. RSM is an essential tool for process optimization. Response surface methodology models allow one to determine optimal settings for the process and identify best conditions that enable the process to be robust to uncontrollable and unpredictable variables. DOE benefits experimenters by providing a simple comparison method and establishing a strategy to decrease experimental trial periods. Therefore, DOE techniques are less time consuming compared to traditional methods of experimentation. They also need shorter analysis periods. In this review, we describe few types of DOE: two-level full and response surface methodology. This review covers the applications of DOE in the industry and research field.

Keywords: Design of Experiment, Process Optimization

1. INTRODUCTION

In manufacturing processes, engineers always face two practical problems, which are process' parameters that will yield the desired product quality, and maximize manufacturing system performance. Their decision making mostly based on their experience and on-going process monitoring. Many of these phenomena are complex and interact with a large number of factors. To attain high process performance, researchers have proposed models that try to simulate the conditions during machining and establish the relationships between various factors and desired product characteristics. Efficient experimental design method is economical for characterizing a complicated process. It requires fewer experiments in order to study all levels of all input parameters, and filters out some effects due to statistical variations [1].

An efficient experimental design method has been developed for experiment planning, collection and data acquisition. Design of Experiment (DOE) has been widely used in the industry and research field to optimize processes. DOE has been known as it efficiency in characterizing a complicated process. It requires fewer experiments in order to study all levels of all input parameters, and filters out some effects [2]. In etching processes, DOE is used to estimate the relationship between etching parameters and process performance. Many researchers have developed their experiment through DOE to avoid studying secondary factors.

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In order to establish an adequate functional relationship between the etching rate, and the etching parameters (solution, solution concentration, temperature, period), a large number of tests are required, requiring a separate set of tests for each and every combination of etching condition and workpiece material. This increases the whole number of tests and as a result the experimentation cost also increase. The aim of this paper is to present various types of DOE and their application, and discuss the preliminary results that are obtained from the experiments conducted according to response surface methodology (RSM).

2. DESIGN OF EXPERIMENT (DOE)

DOE is a combination of mathematical and statistical techniques. Mathematical models can be used to predict and better analyze result behavior in different condition with a limited number of experimental tests [3]. DOE is being divided into a few types, such as two-level factorial design, fractional design, response surface methodology and others. Basically, their concepts are similar to each other. The differences are in their statistical model and characteristics.

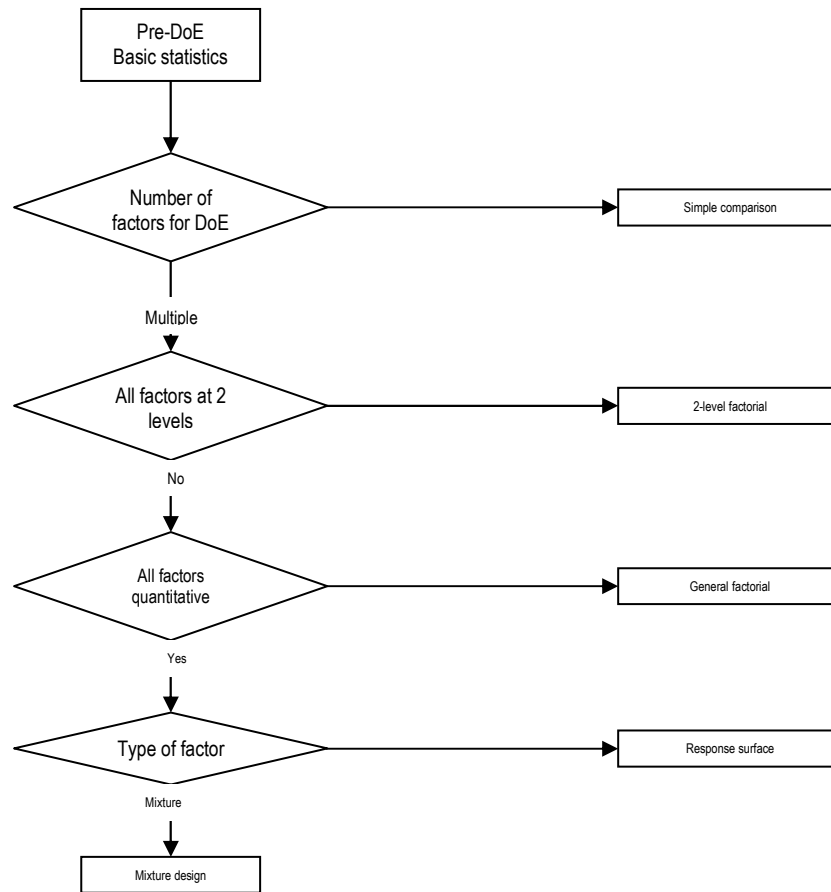


Figure 1 shows the flowchart guide to DOE [4].

DOE is used in various industrial applications to optimize a process. In the industry, DOE is used to enhance the productivity and product quality. As automation systems become more complex, they can only be analyzed using DOE method. DOE also reduce process cost and reduce time of trial-and-error. According to Baldassari et al. [5], DOE approach was able to reduce the effect of mixing time and enhance the quality of their specimens. Perlot et al. [6] concluded that DOE was able to express the influence of process parameters on the response and statistical method to determine the significance of the coefficients of the regression equation. In certain circumstances, DOE is used in simulation to enhance data accuracy, and filter out the errors and unnecessary factors. Montevechi et al. [7] found that DOE was able to improve the simulation process by avoiding the trial-and-error techniques to seek solutions. In their process, they

also found that the significance effects of the interactions were confirmed, aiding the managerial decision making process.

DOE is being divided into three categories: comparative objective, screening objective and response surface objective [8]. Comparative objective is used when experiment primarily goal is to make a conclusion about one a-priori factor, and the question of interest is whether the factors is “significant”. Screening objective is chosen when primary purpose of the experiment is to select the few important main effects from the many less important ones. While, response surface objective is used to find optimize the process, troubleshoot process problems and to make a process more robust against external and non-controllable influences. Table 1 shows the design selection guideline.

Number of factors	Comparative Objective	Screening Objective	Response surface objective
1	1 factor completely randomized design	-	-
2-4	Randomized block design	Full/ fractional factorial	Central composite/ Box-Behnken
5 or more	Randomized block design	Fractional factorial/ Plackett-Burman	Screen first to reduce number of factors

Table 1 shows the design selection guideline [8].

2.1 TWO-LEVEL FACTORIAL DESIGN

In statistics, factorial experiment is an experiment whose design consists of two or more factors, each with discrete possible values, and whose experimental units take on all possible combinations of these levels across all such factors. Factorial design is a tool that allows experimenters to experiment on many factors simultaneously. The simplest factorial design is two-level factorial which involves a few factors at two levels or values. In some cases, they involve different types of levels, such as quantitative (temperature, pressure or time); or qualitative (two machines, or two operators). A complete replicate of such a design requires 2^k observations and is called a two-level (2^k) factorial design, where k is the number of factors in design.

Compared to one-factor-at-a-time (OFAT), 2^k design provides wider inductive basis, which covers a broader area or volume to draw inferences about the process, and it reveals interactions of factors [2]. One of the major disadvantages is 2^k factorial design is that it limits all factors at two levels, thus the response is approximately linear over the range of the factor level chosen.

2.1.1 PLANNING STAGE FOR 2^k FACTORIAL DESIGN [4]

1. Determine the factors to be investigated.
2. Design and run a 2^k factorial experiment in a localized region of the response surface.
3. Compute the estimates of the effects and thereby calculate the coefficient of the linear model:

$$y = b_0x_0 + b_1x_1 + b_2x_2 + b_3x_3 + \varepsilon \quad (1)$$

4. Select a reference factor to be used as a guide in determining the appropriate steps along the direction of each factor in order to continue moving along the path of steepest ascent.
5. Select a few experimental conditions along the path response of steepest ascent and run trials to determine if the response continues to increase. If the response ceases to increase, a new path should be generated.
6. If a new path is needed, design and run a new 2^k factorial experiment. All previous steps are repeated until no substantial improvement in the response is obtained.

2.2 RESPONSE SURFACE METHODOLOGY

Response surface methodology (RSM) is useful for modeling and analysis of problems in which a response of interest is influenced by several factors [2]. There are a few types of RSM: Central composite design (CCD), Box-Behnken and 3^3 design. In this paper, only CCD is discussed. CCD is intended for sequential experimentation, thus making it flexible for industrial process development.

CCD is classified into central composite circumscribed (CCC), central composite inscribed (CCI) and central composite face centered (CCF). CCC designs are the original form of CCD. It axial points are at some distance α (distance from the center of the design space) from the center based on the properties desired for the design and the number of factors in the design. CCI design uses the factor settings as the axial points and creates a factorial or fractional factorial design within those limits. It is a scaled down CCC

design with each factor level of the CCC design divided by α to generate the CCI design. In CCF design, start points are at the center of each face of the factorial space. This variety requires three levels of each factor.

In RSM, the experimental responses to DOE are fitted to quadratic function. Factors that are considered as most important are used to build a polynomial model in which the independent variable is the experiment's response [2]. Mead and Pike [9], and Hill and Hunter [10] reviewed the earlier work on response surface methodology (RSM). They used RSM for tool life modeling, surface roughness modeling, and in other machining processes [11]. Danun et al. [12] concluded that RSM was useful techniques for surface roughness tests. Relatively, a small number of designed experiments are required to generate much useful information that is used to develop the predicting equations for surface roughness. Hung et al. [13] found that RSM design has been used to investigate the effect of various control factors on the performance of silicon trench etch on $Cl_2/HBr/O_2$ -based chemistry. As the result of analysis, the TCP source power, bias power, and Cl_2 content of Cl_2/HBr mixture all influenced the etch rate positively.

Overall, CCD is the most popular of the many classes of RSM designs due to its ability to be partitioned naturally into two subsets of points; the first subset estimates linear and two-factor interaction effects while the second subset estimates curvature effects. CCDs are efficient, in providing much information in a minimum number of required runs, and flexible due to its variety of choices that enables choices, enables them to be use under different operability. Desai et al. [14] summarized the application of RSM in fermentation production and found that RSM is useful in getting insight information of a system directly, and interactions between different components. Though RSM is a highly recommended method, it still showing data inaccuracy, less generalization capability and it performance was not consistent compared to other methods. Benardos et al. [15] stated that experimenter was able to optimize the process after running RSM and knowing whether or not transformations on the responses or any of the process variables are required [16].

2.3.2 PLANNING STAGE FOR RSM

1. Design and run a three-level factorial experiment in the region where the path of the steepest scent yields no substantial improvement in response.
2. Compute the coefficients of the model:

$$y = b_0x_0 + b_1x_1 + b_2x_2 + b_3x_3 + \varepsilon \quad (2)$$

3. using the above model, determine the nature of the stationery point of the response surface. The stationary point is one where the gradient vanishes.

3. EXPERIMENTAL PROCEDURE

10mm X 10mm X 10mm zirconia specimens are used as the substrates and hydrochloric acid is being chosen as the solution in this etching process. Zirconia is being cleaned with distilled water for about 10mins, and then it is being dried in an oven under 115°C for about 2hours. Lastly, weight is being taken in a close-up balancer before and after etching process. The parameters investigated in this study are (a) temperature (30°C, 65°C and 100°C), (b) concentration (7Molar, 10Molar and 12Molar), (c) etching period (30mins, 195mins and 360mins), and (d) stirring process (0, 2 and 4levels). RSM experiment utilized a CCD including 20runs (with three center points) are conducted for this study. 20runs are randomly organized to make sure the observation is independently distributed.

4. RESULT & DISCUSSION

Run order	temperature	concentration	period	stirring	Etch rate (gram)	Run order	temperature	concentration	period	stirring	Etch rate (gram)
1	30	7	360	0	0.0025	16	100	7	360	4	0.0013
2	100	7	360	0	0.0010	17	65	10	195	2	0.0014
3	100	7	30	0	0.0010	18	30	12	30	4	0.0014
4	30	7	30	0	0.0018	19	100	7	30	4	0.0015
5	100	12	360	0	0.0015	20	30	12	360	0	0.0032
6	65	10	195	2	0.0010	21	65	10	195	2	0.0010
7	30	12	30	0	0.0012	22	65	10	30	2	0.0015
8	30	12	360	4	0.0020	23	30	10	195	2	0.0012
9	30	7	360	4	0.0010	24	65	10	195	4	0.0013
10	65	10	195	2	0.0010	25	100	10	195	2	0.0010

11	65	10	195	2	0.0011	26	65	10	195	0	0.0014
12	100	12	360	4	0.0010	27	65	7	195	2	0.0010
13	30	7	30	4	0.0010	28	65	12	195	2	0.0013
14	100	12	30	4	0.0026	29	65	10	195	2	0.0012
15	100	12	30	0	0.0019	30	65	10	360	2	0.0012

Table 2 Experiment results from CCDesign

The results of the experiments are summarized in table 2. After analyzing, the etch rate of zirconia can be specified by the following equations:

$$ER = 10^{-3} (2.3 - 0.03T - 2.2C + 0.02t - 5.1S + 0.005TC - 0.0006Tt + 0.04TS + 0.00003Ct + 0.09CS - 0.006tS - 0.0001T^2 + 0.001C^2 + 0.000087t^2 + 0.06S^2) \quad (3)$$

where ER is the etching rate in gram, T is temperature, C is solution concentration, t is etching period, and S is stirring process. In CCD, optimization is used to obtain the maximum etching rate in lowest temperatures and period. An adjusted of R^2 value of 0.6736 is obtained from the ANOVA, indicating that this model as fitted explains of 67.36% of the variability of the etch rate. From equation (3), there exists a 99.8% positive effect etching rate of increasing HCl concentration. While etching period has shown the least related parameters in this process. Figure 2 shows the excellent fit of the observed values.

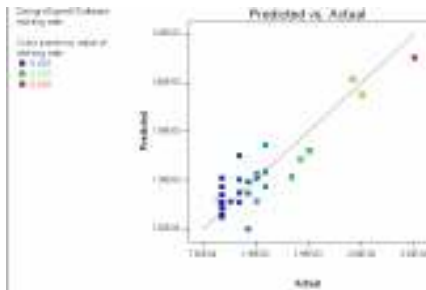


Figure 2. Comparison the observed and predicted etch rate.

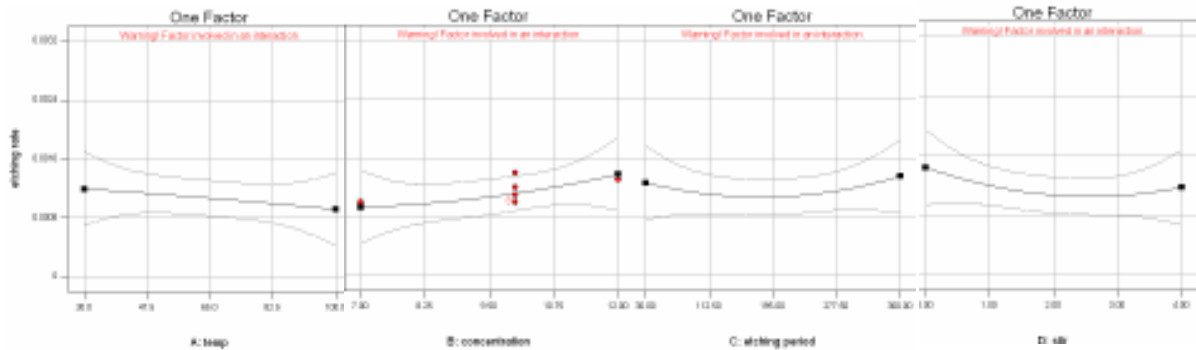


Figure 3. Main effects plot of zirconia etching rate in terms of temperature, HCl concentration, etching period and stirring.

Figure 3 shows that etching rate increases with the increasing of HCl concentration but decrease significantly with temperature. While, etching period and stirring process show least changes in the etching rates. Furthermore, HCl concentration has the broadest experimental response range than other parameters. It indicates that this parameter is the most influencing factor on etch rate in this study. It should be noted that the determination of influential factors and their relative strength is based on the levels chosen for those factors. Regardless of the analytical method, any factor would tend to look less important if the levels close to each other and any factor would tend to look more important if the levels were farther apart. Figure 4 shows represent the etching rate in 3D response surface as a function of two main influencing parameters.

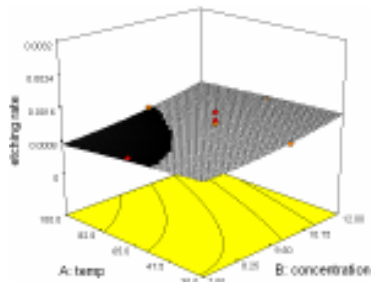


Fig. 4. Etching rate due to temperature and HCl concentration

5. CONCLUSION

Design of experiments is an efficient model to be used in the industry. Current work presented a review of the different approaches that are used for optimizing process and filter out 'error' factors. As evident from the referenced papers, in recent years there has been a great deal of research activity being carried out with DOE and the results that have been produced are good. As a conclusion, DOE is an efficient approach to minimize workload, and it is able to enhance process quality.

In this study, RSM was used to optimize the chemical etching of zirconia. Among the main process parameters, concentration, relationship between temperatures with etch period and temperature with stir process were selected. Based on the experiment data, it was found that (1) response contour and graphs provide useful information about the maximum attainable etching rate, and that (2) equation given in RSM can be used to predict and analyze process's behavior in different conditions with a limited number of experimental tests. Further studies should be done for finding the suitable process parameters.

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