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Prediction of etching rate of alumino-silicate glass by RSM and ANN

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In this study, response surface methodology (RSM) and artificial neural network (ANN) were applied to predict material removal rate in chemical etching process of alumino-silicate glass (SiO_2 57/Al_2O_3 36/CaO/MgO/BaO). 2^k Factorial design was performed to evaluate linearity condition among process parameters. Analysis of variance (ANOVA) was performed and quadratic model was found most significant for data values of process parameters. New models were able to predict etching rate of alumino-silicate glass, with a great confidence. Input parameters analyzed were temperature, etching period and type of setup with and without condensation.

Keywords: Alumino-silicate glass, ANN, Etching rate, RSM

Introduction

Alumino-silicate glass (ASG) is frequently used in semiconductor and optical industry and to control release of volatile chemicals. Due to its insensitivity to thermal effect, ASG is used as doped fiber in all-optical devices[1]. It is also widely used in micro-electro mechanical system (MEMS) due to its good durability, smoothness, high electrical property, and high optical transparency[2,3]. Saito *et al*[4] claimed that etching rate of ASG increased with increasing leaching reaction of alumina. Saito *et al*[5] also investigated effects of glass composition and etching conditions on etching rate with pH, concentration and temperature of solution.

Bezerra *et al*[6] summarized that application of Design of Experiment (DOE) in optimization was largely diffused and consolidated principally because of its advantages over experimentation approach that is based on classical one-variable-a-time optimization. Hung *et al*[7] applied response surface methodology (RSM) to investigate effect of various controlled factors on performance of silicon trench etches on Cl_2/HBr/O_2 based chemistry. Dabnun *et al*[8] applied RSM in predicting surface roughness of machinable glass ceramic. DOE was also applied in industry for modeling and optimization of chemical etching process in niobium cavities[9].

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Artificial neural network (ANN) models had consistent results compared to RSM[10,11]. Desai *et al*[12] concluded that ANN perform at better accuracy and generation capability as compared to RSM. Fredj *et al*[13] also found that ANN had consistently performed better than RSM. Ko *et al*[14] evaluated growth rate of ZnO thin film fabricated by D-optimal and ANN. Kim *et al*[15] used ANN to model characteristics of oxide film etched in CHF_3/CF_4 gas chemistry and examined etch mechanisms as a function of process factors. Shie & Yang[16] focused on optimizing photoresist coating process using ANN with only nine experimental runs, in order to identify process parameters in wafer fabrication. However, there is lack of studies on chemical etching of of ASG using RSM and ANN model.

This study presents predictive model of ASG etching rate with Central Composite Design (CCD) at different levels and ranges of variables with right design of experiment and set up types using RSM, ANN and ANOVA.

Experimental

Among chemical resistant characteristics, ASG is fair to concentrated acid and good to dilute acid and alkalis. Physico-thermal properties of ASG are as follows: apparent porosity, 45%; density, 2.65 g/cm; refractive index, 1.54; coefficient of thermal expansion, 5; upper continuous use temperature, 600°C. Etching experiments were carried out on ASG to observe etching rate under

Table 1 — Analysis of variance (ANOVA) for first-order model

Source	Sum of squares	df	Mean square	F value	p-value Prob › F	
Model	0.0600	7	0.013	36.53	0.0001	Significant
A-condensation	0.0028	1	0.0028	8.66	0.0257	
B-Temperature	0.0052	1	0.0051	15.60	0.0075	
C-Period	0.0074	1	0.0074	226.73	‹ 0.0001	
AB	0.0017	1	0.0027	8.31	0.0276	
AC	0.0013	1	0.0027	4.05	0.0608	
BC	0.0014	1	0.0013	4.23	0.0855	
ABC	0.0032	1	0.0014	6.75	0.0205	
Curvature	0.0300	2	0.0150	46.66	0.0002	Significant
Pure error	0.0016	6	0.0003			
Cor total	0.1200	15				

Table 2 — Model sum of squares

Source	Sum of squares	df	Mean square	F value	p-value Prob › F	
2FI vs Linear	0.00660	3	0.00216	1.01	0.4078	
Quadratic vs 2FI	0.02100	2	0.01000	8.36	0.0027	Suggested
Cubic vs Quadratic	0.01300	5	0.00252	3.31	0.0376	Aliased

Table 3 — Model summary statistics

Source	Std. deviation	R-Squared	Adjusted R-squared	Predicted R-squared	Press	
Linear	0.047	0.6413	0.5646	0.4433	0.077	
2FI	0.047	0.6886	0.5652	0.1880	0.110	
Quadratic	0.035	0.8386	0.7666	0.4566	0.075	Suggested

condensation, without condensation, two levels of etching temperature and etching period. Middle points were added in these experiments to predict curvature surface using quadratic model and estimation of error in experiment data. Samples were etched in 12 M HCl, then cleaned up and rinsed with continuous distilled water. After 1 h, samples were baked at 110°C for about 1 h before recording data. Etching rate was calculated by dividing reduced weight (g) by etching period (min). Obtained data was mapped using 2^k Factorial design, RSM and ANN.

Design of Experiment

This experiment design is divided into 2^k Factorial design for first-order model and RSM for second-order model. After determination of first stage data, ANOVA is performed. At this stage, checking at curvature term is extremely important. A significant curvature term indicate quadratic or higher-level process model for next CCD. Otherwise, first stage data obtained by 2^k Factorial Design have to be analyzed in case of in significant curvature term indicating linear process model. In terms of time and cost saving, 2^k Factorial design is a must procedure and it also helps to provide right ranges of process parameters subjected to design of experimentation. Total numbers of runs are 27 and all running sequences have been randomized and generated following CCD procedure. With three inputs, DE 7 generated 13 numbers of test combinations to study whole process. Design Expert 7.0 (DE 7) was used to perform task and calculations.

Results and Discussion

First-order Model (2^k Factorial Design)

ANOVA generated for 2^k Factorial Design (Table 1) shows that curvature is a significant term in etching process of ASG. This implies that a quadratic model should be considered to model this process. Curvature value (0.0002) is less than 0.05, indicating that only 0.02% of process is fitted to linear process and there is

66.668% confidence in possible fitting of this process into higher curvature form (quadratic, cubic or higher level). Therefore, RSM is needed to apply in order to determine quadratic model for the process.

Second-order Model (using RSM)

In order to obtain second order predictive model, RSM is employed using CCD, which is a design type under RSM. This is chosen due to its flexibility. In CCD, process variables' peaks occur simultaneously. ANOVA is essential to decide if process fits to selected model or not. Tables 2-4 show model sum of squares, model summary statistics and ANOVA, respectively.

At level of 95% of confidence, process was checked for its adequacy and quadratic model is selected instead of linear or cubic model. It was found (Table 4) that this quadratic model is fitted into the process. Dominant factors have been determined (Table 4) by evaluating their p-values. Once process model has been confirmed, next stage will be monitoring changes of each dominant factor by examining generated graphs. P-value shows significance of corresponding factors. Smaller the magnitude of p-value, more significant the model is. In this process, it is clearly shown that temperature is most dominant factor followed by etching period and then, set up condition. Predictive models are given as follows:

Etching rate with condensation (g/min) =

$$0.325–0.0077(t)–0.015(T)+0.00003(t)(T) + 0.0017(T^2) \quad \ldots(1)$$

Etching rate without condensation (g/min)=

$$0.363–0.0078(t) –0.016(T)+0.00003(t)(T) +0.0017(T^2) \quad \ldots(2)$$

where, t is etching period (min) and T is etching temperature (°C).

Least dominant factor found to have only 28.3% influence on the process, whose p-value is larger than

Table 4 —ANOVA for ASG etching in12 M HCl

Source	Sum of squares	df	Mean squares	F value	p-value Prob › F	
Block	0.00067	1	0.00067			
Model	0.12000	8	0.01500	11.66	‹ 0.0001	Significant
A-Etching period	0.00176	1	0.00176	1.44	0.2466	
B-Etching temperature	0.04300	1	0.04300	34.35	‹ 0.0001	
C-Set up (with and without condensation)	0.00017	1	0.00017	0.14	0.7170	
AB	0.00345	1	0.00345	2.76	0.1137	
Residual	0.022	18	0.00127			
Lack of fit	0.013	7	0.00185	2.14	0.1244	Insignificant
Pure error	0.006	11	0.00086			
Cor total	0.14	27				

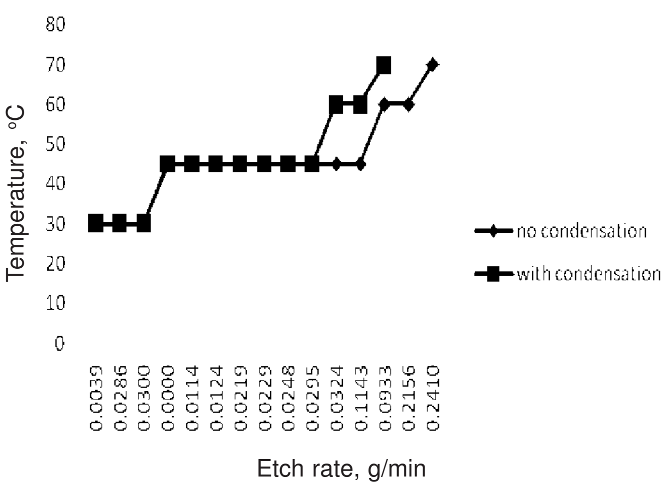

Fig. 1 — Etching rate vs temperature with different set up

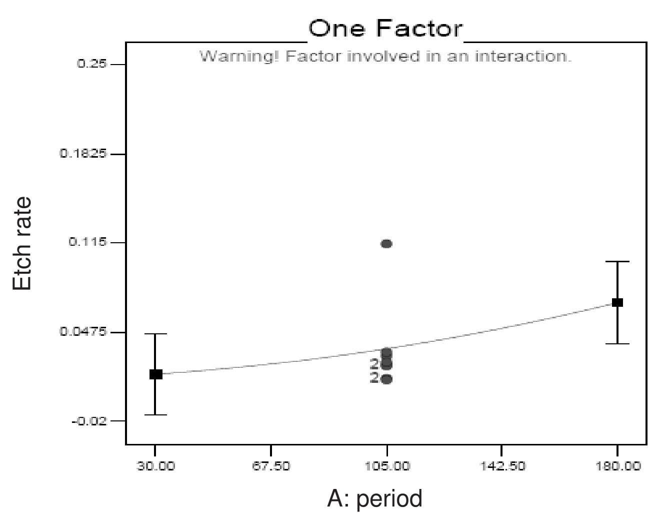

Fig. 2 — Etching period vs etching rate at 45°C

0.05 (Table 4). Thus, setup condition is an insignificant factor and decision shall be made in order to reduce testing number in further study. Higher etching rate is observed to take place at setup without refluxing condition (Fig. 1). In setup with condensation, its concentration remained same during the process. Vaporized H_2O is being condensed and return to solution during experiment. While, this phenomenon does not happen at setup without condensation. Especially at temperature near to solution boiling point, water content in solution tended to evaporate into the air. This phenomenon is reducing H_2O in HCl and increasing concentration too. Higher etching rate was observed at setup without condensation, also in agreement with reported studies[5,17]. Etching rate of ASG increases with etching period (Fig. 2). Yet, this is a quadratic reaction. Once it meets peak point, it will stop or go backward due to chemical reagent, also in agreement with reported studies[18]. Etching rate increases with increasing etching temperature (Fig. 3). Highest etching rate happens at right of the graph, which is not a peak value. Hence, further study need to be conducted in order to obtain its yield point. At higher temperatures, activation energy of solution content is higher and this will allow chloric ion to react with ceramic surface. Contour plot (Fig. 4) shows relationship between temperature, period and etching rate. Highest etching rate occurs at right corner of contour at highest temperature and period. Unfortunately, contour has not reached its peak. Thus, further study need to be conducted in order to obtain yield point for each influencing factors.

A good agreement with experimental result is observed for ANN prediction (Fig. 5). Quadratic model of RSM was able to predict etching rate. It was found that these techniques are showing great promising result with only 1.12% (CCD) and 1.05% (ANN) error. ANN had been justified as best tool to be used to predict this process.

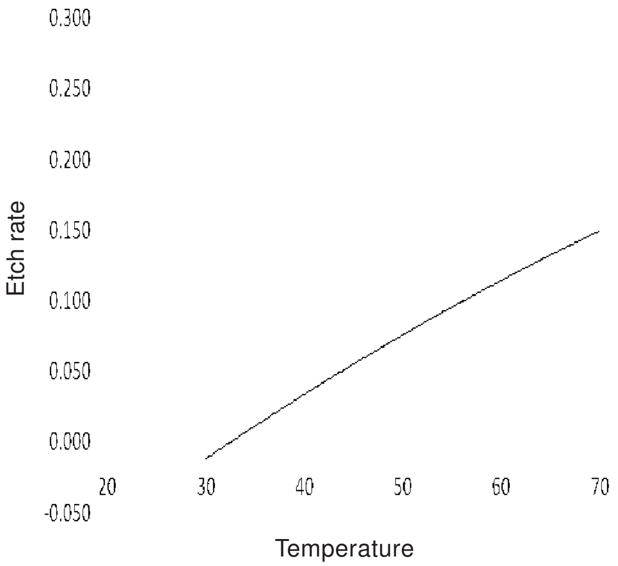

Fig. 3 — Etching temperature vs etching rate

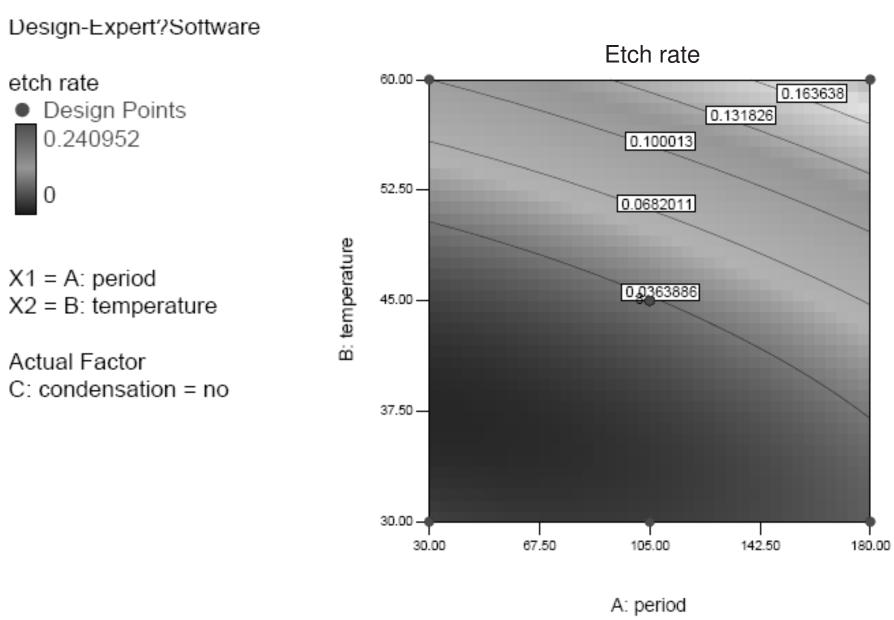

Fig. 4 — Contour plot of ASG etching rate vs temperature and period

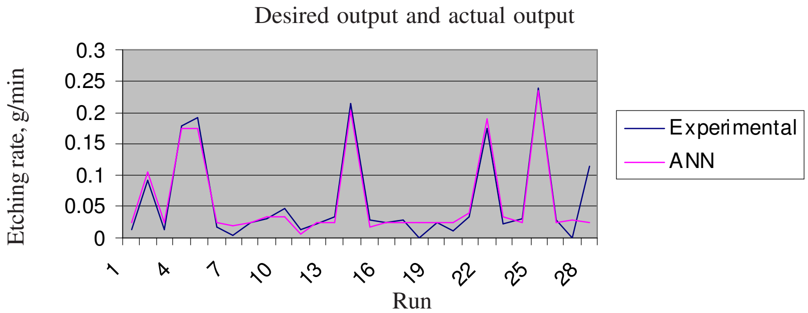

Fig. 5 — ANN prediction result

Conclusions

Chemical etching process of ASG was investigated in this work using RSM and ANN. Model produced by RSM was adequately verified at 95% of confidence interval. ANOVA showed that etching process of ASG was quadratic with etching temperature and etching period having highest dominance as input factors. Generated values by RSM model showed a good agreement with those received experimentally and by ANN. However, ANN showed more precise predictions as compared to RSM model. Non-condensation setup gave best etching rate at 60°C and 180 min. Yet, changes of solution content should be taken into consideration.

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