

Optimization of Dielectric Sol-Gel Coating Synthesis Parameters via Genetic Algorithm

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Abstract—This study optimized the parameters of silicon dioxide coating formation using a genetic algorithm. The coatings were prepared via the sol-gel method. Experiments were conducted using a face-centered central composite design, with the following input parameters: the ratio of silicon organic compounds in the initial sol, spin-coating rotation speed, and annealing temperature. The output characteristics included coating thickness, refractive index, and leakage currents of the sol-gel coatings. The influence of coating formation parameters on these characteristics was evaluated. The coating process optimization was performed using a genetic algorithm with the objective of minimizing leakage currents for sol-gel coatings of specified thickness.

Keywords—genetic algorithm, sol-gel technology, optimal parameters volt-ampere characteristics, coatings for microelectronics

I. INTRODUCTION

The advancement of microelectronics, driven by the demand for device miniaturization and enhanced reliability, necessitates the improvement of dielectric coatings. Currently, hybrid organic-inorganic dielectric layers are widely used in microelectronics. This is due to their combination of valuable physicochemical and technological properties. Among organic-inorganic dielectrics, glass-like materials have gained the most prevalence. These materials are characterized by low electrical conductivity and dielectric losses, high strength, hardness, thermal stability, and radiation resistance, as well as the absence of aging [1].

Due to their simplicity and low cost, sol-gel materials synthesized by chemical methods from a variety of silicon-based precursors, such as methyltriethoxysilane and tetraethyl orthosilicate, are of great interest. By varying the ratio of the initial components and using different types of solvents, it is possible to tailor the material with a set of predefined properties: thermal, plastic, electrophysical, mechanical, etc. [2-3].

While the sol-gel method offers flexibility in tailoring material properties, it faces challenges in optimizing multiple

interdependent parameters. Traditional approaches based on factor screening prove insufficiently efficient. The application of genetic algorithms provides an effective solution to these problems, reducing both time and costs associated with such research [4]. The use of genetic algorithms in developing new sol-gel materials will enable efficient solutions for microelectronics.

Previous studies [5-8] have demonstrated the successful optimization of process parameters using genetic algorithms.

The present research employed the MOGA module of ANSYS DesignXplorer to optimize the parameters of sol-gel coating formation for microelectronic applications.

II. RESEARCH RESULTS

The dielectric sol-gel coatings were prepared following the synthesis scheme presented in Fig.1.

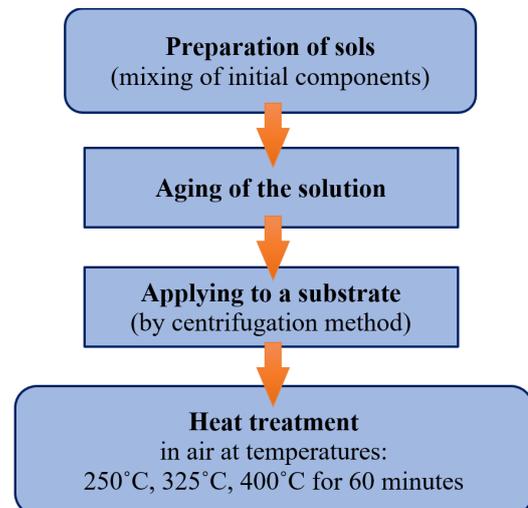


Fig. 1. Scheme of synthesis dielectric sol-gel coatings.

In order to examine how the initial concentration ratio of methyltriethoxysilane (MTES) to tetraethyl orthosilicate (TEOS) affects the properties of the resulting sol-gel coatings, a series of film-forming solutions was prepared with different MTES:TEOS mass ratios (5:3, 4:3, and 1:1). The film-forming solutions were prepared as follows: specified quantities of methyltriethoxysilane ($\text{CH}_3\text{Si}(\text{OC}_2\text{H}_5)_3$) and tetraethyl orthosilicate ($\text{Si}(\text{C}_2\text{H}_5\text{O})_4$) were mixed with a solvent and catalyst. During this process, the solution temperature increased due to hydrolysis and polycondensation reactions. The obtained solutions were subsequently aged at ambient temperature (22 ± 2) °C for 2–3 days to ensure complete maturation.

To study the effect of deposition conditions, the prepared film-forming solution was applied to KDB-4 grade silicon wafers. The spin-coating process typically consists of four stages: solution application, spreading, excess solution removal, and solvent evaporation. The deposition parameters were optimized using an Apogee Cee 200X spin coater (Cost Effective Equipment, USA), which enables liquid solution deposition on flat substrates at rotational speeds ranging from 10 rpm to 12,000 rpm. Following deposition, the wafers were transferred to a programmable SNOL 8.2/1100 muffle furnace for air annealing. Three annealing temperatures were selected for investigation based on their ability to produce homogeneous coatings: 250°C, 325°C, and 400°C. The thermal treatment protocol featured a constant heating rate of $\approx 3.5^\circ\text{C}/\text{min}$, followed by a 60-minute isothermal hold at the target temperature.

Refractive indices and thicknesses of the fabricated samples were measured with an LEF-757 laser ellipsometer (Institute of Semiconductor Physics, Russia) by taking five-point measurements across each wafer followed by data averaging, while current-voltage characteristics were evaluated using a Semishare E6 four-point probe station (Semishare, China) coupled with a GSM7-20H10 parametric analyzer (Good Will Instrument, China).

The study investigating the influence of initial composition and processing parameters on sol-gel coating properties was conducted using the Ansys DesignXplorer module. A central composite experimental design was implemented for three factors (P1–P3): where P1 represents the initial methyltriethoxysilane to tetraethyl orthosilicate ratio, P2 is the spin-coating rotational speed, and P3 denotes the annealing temperature. According to the experimental design, tests were performed for 15 input parameter combinations (see Table 1).

The following output parameters characterizing the sol-gel coatings were determined: coating thickness (h), leakage currents (I_1 , I_2 , I_3) at 50 V, 100 V, and 150 V respectively, and refractive index (n). Thus, the research object model consisted of response functions relating output parameters (h , I_1 , I_2 , I_3 , n) to input factors (K , ω , T). For response surface generation, a nonparametric regression method implemented in the DesignXplorer module of ANSYS Workbench was employed.

TABLE I. EXPERIMENTAL DESIGN AND RESULTS

N	$P1$ K	$P2$ ω, rpm	$P3$ $T, ^\circ\text{C}$	h , nm	I_1 , nA	I_2 , nA	I_3 , nA	n
1	1	3000	325	333.56	3.7	44.1	219.8	1.291
2	1.3	3000	325	328.70	2.5	27.0	147.7	1.303
3	1.67	3000	325	324.17	4.4	58.5	211.4	1.35
4	1.3	3000	250	296.42	2.6	29.1	163.8	1.354
5	1.3	3000	400	284.43	2.5	27.3	139.2	1.392
6	1.67	1500	400	782.47	3.1	49.8	144.6	1.395
7	1	4500	400	178.29	3.2	47.5	207.1	1.398
8	1	1500	400	714.28	2.7	42.8	184.7	1.402
9	1.3	1500	325	726.70	2.1	20.6	99.6	1.407
10	1.67	4500	400	235.81	4.1	55.0	198.2	1.407
11	1	4500	250	194.97	4.8	46.6	239.5	1.409
12	1.67	1500	250	764.16	3.3	52.2	171.5	1.412
13	1.3	4500	325	164.03	2.3	24.6	112.0	1.413
14	1.67	4500	250	223.81	4.0	54.2	171.2	1.413
15	1	1500	250	713.35	5.0	50.8	215.3	1.414

The response surface was generated using a nonparametric regression approach [9].

The developed models were assessed using the following criteria:

coefficient of determination

$$R^2 = 1 - \frac{\sum_{i=1}^n (d_i - y_i)^2}{\sum_{i=1}^n (d_i - \bar{d})^2},$$

Root Mean Square Error (RMSE)

$$\text{RMSE} = \sqrt{\frac{1}{n} \sum_{i=1}^n (d_i - y_i)^2}$$

where d_i represents the experimental values, and y_i denotes the values predicted by the model. The results of model evaluation are presented in Table 2.

TABLE II. MODEL EVALUATION RESULTS

<i>Criterion</i>	h	I_1	I_2	I_3	n
R^2	0.9994	0.9990	0.9992	0.9988	0.9990
$RMSE$	5.7 (nm)	0.03 (nA)	0.33 (nA)	1.32 (nA)	0.0012

The values of the determination coefficient and RMSE for the output parameters confirm that the model demonstrates satisfactory agreement with the experimental data.

The influence of input parameters on the output parameters that define sol-gel coatings was assessed (see Fig.2). The centrifuge rotation speed has the greatest impact on the film thickness and refractive index values. The extent of leakage currents is primarily determined by the initial ratio of MTES to TEOS.

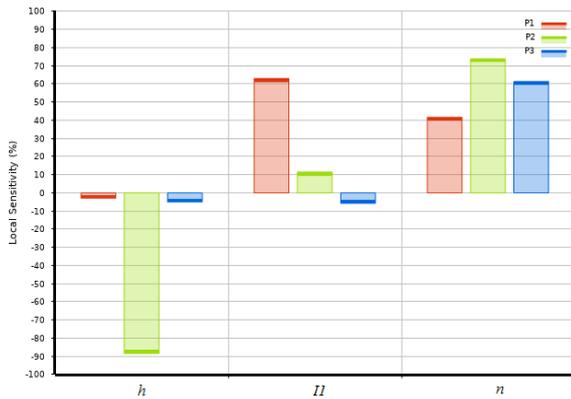


Fig. 2. Sensitivity diagram of optimized parameters.

Fig.3 shows the dependence of leakage current I_1 on the initial MTES/TEOS ratio P_1 and centrifuge rotation speed P_2 . Fig.4 presents the coating thickness h as a function of both centrifuge rotation speed P_2 and annealing temperature P_3 . Fig.5 displays the refractive index n dependence on centrifuge rotation speed P_2 and annealing temperature P_3 .

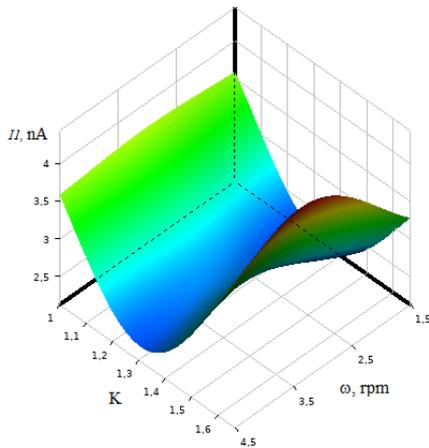


Fig. 3. Leakage current I_1 as a function of sol-gel coating formation parameters.

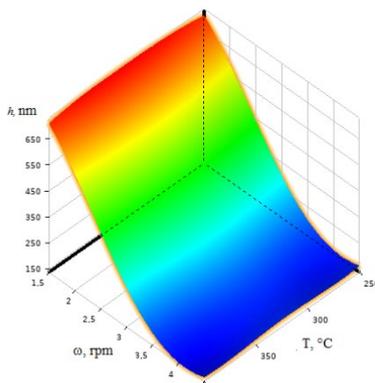


Fig. 4. Coating thickness h as a function of sol-gel coating formation parameters.

The optimization of sol-gel coating formation parameters was performed using the multi-objective genetic algorithm (MOGA) with an initial population size of 100 individuals and 100 individuals per iteration.

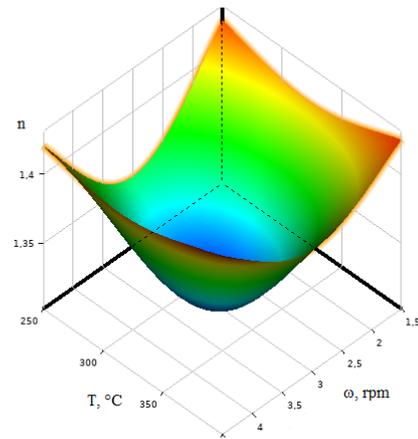


Fig. 5. Refractive index n as a function of sol-gel coating fabrication parameters.

Multi-criteria optimization was performed according to the following criteria: minimization of leakage currents, and maximization of refractive index values for coatings with specified thickness.

The optimization results are presented in Table 3, with experimentally obtained parameter values shown in parentheses.

TABLE III. OPTIMIZATION RESULTS

$P1$ K	$P2$ ω, rpm	$P3$ $T, ^\circ C$	$h,$ nm	$II,$ nA	n
1.28	2977	378	300 (309)	2.4 (2.2)	1.356 (1.331)
1.28	2282	391	500 (542)	2.1 (2.2)	1.338 (1.396)
1.28	1634	349	700 (737)	2.1 (2.1)	1.395 (1.392)

The maximum relative error of the genetic algorithm predictions for output parameters did not exceed 10%.

III. CONCLUSION

This study employed a genetic algorithm to optimize processing parameters for sol-gel dielectric coatings designed for microelectronics applications. Experimental investigations followed a central composite design with three key factors: (1) methyltriethoxysilane to tetraethyl orthosilicate ratio, (2) centrifuge rotation speed, and (3) annealing temperature. The output parameters included coating thickness, refractive index, and leakage currents at various voltages. Response surfaces were constructed from experimental data using nonparametric regression methods. Through genetic algorithm optimization, we identified parameter combinations that simultaneously minimize leakage currents and maximize refractive index values while maintaining specified coating thicknesses.

This research demonstrates that combining genetic algorithms with experimental design methodology provides an effective framework for producing sol-gel dielectric coatings with tailored properties. The approach significantly reduces both development time and material costs compared to conventional parameter screening methods.

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