Fabrication of low optical losses Al_2O_3 layer used for Er^{3+}-doped integrated optical amplifiers

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***Abstract**: Al_2O_3 is commonly used as host material for Er^{3+}-doped integrated optical amplifiers. In this paper, a Graeco-Latin square is used in DC reactive magnetron sputtering deposition experiment in order to get low optical losses Al_2O_3 layer. By reasonable selection and careful arrangement of experimental parameters, an optimal combination of deposition parameters is obtained via statistic analysis with the fewest experimental runs. The result forms the base for the further fabrication of Er^{3+}-doped Al_2O_3 layer. The Graeco-Latin square experiment can also be used to investigate the influence of each parameter on the deposition rate of Al_2O_3 layer.*

1. Introduction

Al_2O_3 is one of the most promising host material used for integrated optical amplifier due to its excellent optical properties. It can be obtained by various techniques: co-reactive magnetron sputtering deposition, radio-frequency magnetron sputtering, molecular beam epitaxy (MBE) and various CVD.

This paper will focus on the optimization of the fabrication parameters used for the deposition of Al_2O_3 by co-reactive sputtering in order to get low optical losses Er-doped Al_2O_3 film. A series of experiments are conducted according to Graeco-Latin Square, in which several significant parameters each with different value (levels) are selected and specially arranged. A statistic analysis follows after the experiment and then an optimum combination of the parameters is expected to present. The setup used for co-reactive sputtering is Balzers KSS 400V which contains three independent guns. The substrate holder keeps rotating during the fabrication in order to make the layer uniform. The optical losses are measured by prism coupling method [1,4].

2. Experimental Design [6,7]

2.1 Selecting the experimental parameters (factors)

The objective of our experiment is to investigate how the fabrication parameters influence the optical properties of Al_2O_3 obtained by co-reactive sputtering and then gives an optimum combination of them. The dependent variable to be measured is optical loss. The independent variables (factors) that exert significant influence on the optical loss are:

1) *Ar flow rate for Al target.* Ar acts as the working gas for sputtering. A certain amount of Ar flow rate is necessary to ignite and maintain plasma. Since plasma current and voltage have been found to change with the variation of the Ar flow rate, it is estimated that the deposition rate, the topography and the density of Al_2O_3 film may also change with the Ar flow rate. The influence of this factor will be investigated in our experiment. Considering that high Ar flow rate will increase the working pressure

inside the chamber and may cause Ar in-cooperation into Al_2O_3 film, which will affect the optical properties of the layer, a relatively low Ar flow rate is used.

2) *O_2 flow rate.* O_2 is the reactive gas in co-reactive sputtering. High O_2 flow rate is required in order to form stoichiometric Al_2O_3 film and effectively prevent oxygen deficiency that will greatly increase the optical loss of the layer [3]. However, high O_2 flow rate can also cause full oxidation of Al target and reduce the deposition rate. The influence of this factor will also be investigated in order to find a suitable O_2 flow rate.

3) *DC sputtering power.* High DC sputtering power means high deposition rate and high dynamic energy of sputtered Al molecules which makes the layer more compact, but it also gives rise to sputtering a cluster of Al molecules which forms the scattering center of the layer. There is a check of this factor in our experiment.

4) *Substrate temperature.* The temperature of substrate significantly affects morphology of sputtered film. Low temperature will cause large amount of voids in the deposited film [5] which greatly increase the optical loss of the layer and hence, should avoid. However, this does not mean the higher the temperature, the better the layer. Too high temperature may cause high cost and complexity of the equipment and other unexpected problem. Finding an acceptable substrate temperature is also included in our experiment.

Beside these factors, the distance between target and substrate, working pressure in the chamber, target thickness and its oxidation extent and the concentration of the Er doped also influence the optical loss of the layer. These factors will be investigated future.

2.2 Determining the number of factor levels

Now we have decided 4 factors. Since the relationship between the optical loss and the factors is not clear, we gives 4 levels to each factor, which also extend the range of the factor to be investigated. Based on our previous experience [2,3], the factors and their variation range list as following:

Ar flow rate for Al target (sccm): 25, 30, 35, 40
O_2 flow rate (sccm): 45, 50, 55, 60
DC power (W): 200, 225, 250, 275
Temperature of substrate (ºC): 350, 400, 450, 500
The Er concentration will be 0.2 atm.% in this experiment.

Table1 A Graeco-Latin square

	I	II	III	IV
1	Aα	Bγ	Cδ	Dβ
2	Bβ	Aδ	Dγ	Cα
3	Cγ	Dα	Aβ	Bδ
4	Dδ	Cβ	Bα	Aγ

2.3 Determining the orthogonal array

If a factorial experiment is conducted with the factors and their levels decided above, all levels of a factor should be “crossed” with all levels of the other factors, which means $4 \times 4 \times 4 \times 4 = 4^4 = 256$ experimental runs is required. This is unacceptable for both the cost and the time consumed. To minimize the experimental runs while the influence of each factor on the optical loss can still be investigated, a Graeco-Latin square (Table 1) is adopted. With this arrangement, the main effect of Ar, O2, DC power and substrate temperature on the optical loss of the layer can be determined from only 16-run experiments, but the information about interaction effects between these factors can not be calculated due to limited experiment runs.

2.4 Randomizing the experimental runs

Table2 Experimental sequence and results

No.	Exp. Sequ.	Parameter				Loss (dB/cm)	Matrix
		Ar (sccm)	O2 (sccm)	P(W)	T (°C)		
1	4	25	45	200	350	5.086	0000
2	9	25	50	225	450	0.853	0112
3	2	25	55	250	500	2.052	0223
4	12	25	60	275	400	1.597	0331
5	8	30	45	225	400	1.002	1011
6	10	30	50	200	500	1.543	1103
7	11	30	55	275	450	0.541	1232
8	3	30	60	250	350	3.133	1320
9	13	35	45	250	450	0.854	2022
10	1	35	50	275	350	1.814	2130
11	5	35	55	200	400	2.435	2201
12	6	35	60	225	500	1.296	2313
13	14	40	45	275	500	1.091	3033
14	7	40	50	250	400	2.908	3121
15	16	40	55	225	350	0.825	3210
16	15	40	60	200	450	1.299	3302

To control bias error that will inevitably appear in the experiment, a complete randomization of the experimental sequence is necessary. A number of methods of randomization are available. In this experiment, 16 pieces of paper written the matrix in Table2 were well shuffled and put into a bag. The random test sequence was established by taking a piece of paper out of the bag once a time. The experiment sequence is listed in the 2nd column of table2.

3. Data analysis [7,8]

16-run experiment was conducted according to the randomized experiment sequence. Each run takes exactly the same 30 minutes. Table2 listed the optical losses measured (λ=1580 nm). These data were analyzed for variance. A summary of this analysis, which indicated significant effects at the 5% level or less for optical loss, is listed in Table3, where *df* is degree of freedom, *SS* stands for sum of squares, *MS* is mean square. From these data the F number is calculated and compared with critical F value (F_c). The calculation indicates that none of factors has significant effects on optical loss.

Table3 Significant test for factors

Source	*df*	*SS*	*MS*	*F*	F_c	*Significant?*
Ar	3	9.527	3.176	0.864	9.28	NO
O_2	3	12.67	4.223	1.149	9.28	NO
Power	3	30.764	10.255	2.791	9.28	NO
Temp.	3	4.683	1.561	0.425	9.28	NO
Error	3	11.022	3.674			
Total	15	68.666	4.578			

The determination of specific levels of each factor that significantly affect optical loss is made by the Duncan multiple range test. The general steps are:

a) Calculate the means of the factor levels and rank them from low to high (Table4).

Table4

Ar	1.5548	1.5998	2.3971	3.4558
O_2	1.4635	1.7797	2.0080	3.7564
Power	0.9941	1.2608	2.2366	4.5160
Temp.	1.4956	1.9854	2.7145	2.8121

b) Calculate the standard error ($\hat{s}_{\bar{y}_j}$) of the mean for each factor level. ($\hat{s}_{\bar{y}_j}$ =0.9584)

c) Calculate Least Significant Ranges (LSR): n (df of error)=3, k =2,3,4 (Table5).

Table5

	k=4	k=3	k=2
α=0.05	4.516	4.516	4.501
LSR	4.328	4.328	4.314

d) Take the differences between the factor means in order (Table6):

e) Comparison. This is shown in Table 7 where H_0 is the hypothesis that if the listed 2 level is significantly different. The "decision" shows "accept", which means that no significant difference between levels.

Table6 differences in order

Factor	*differencrs*					
	3-0	3-1	3-2	2-0	2-1	1-0
Ar	1.901	1.856	1.0587	0.8423	0.7973	0.0451
O_2	2.2923	1.9766	1.7484	0.5445	0.2282	0.3163
Power	3.5219	3.2553	2.2794	1.2425	0.9759	0.2667
Temp.	1.3165	0.8267	0.0976	1.2189	0.7291	0.4898

4. Conclusion

The calculation above indicates that in the given parameter range none of factors has significant effects on optical loss. The difference between parameter levels is also not significant. The optical loss for combination of "1-2-3-2" seems lowest from the measurement. From Table 3 it can also be found that MS for error is quite high due to the low degrees of freedom of the error. This means that the experimental error, including error caused by different thickness of the layers, plays an important role to the experimental result. Taking as many factors as possible under control (for instance, fabrication of the layers with the same thickness) and the replication of the experiment may improve this situation. We'll continue our work in this aspect. The result also implies that there may exist some really significant factors that are not included in this experiment.

Table7 Comparison

Factor	H_0	Diff.	LSR 0.05	Decision
Ar	3=1?	1.9010	4.3281	Accept
	3=2?	1.8560	4.3281	Accept
	3=0?	1.0587	4.3137	Accept
	0=2?	0.8423	4.3281	Accept
	0=1?	0.7973	4.3137	Accept
	2=1?	0.0451	4.3137	Accept
O_2	3=2?	2.2929	4.3281	Accept
	3=1?	1.9766	4.3281	Accept
	3=0?	1.7484	4.3137	Accept
	0=2?	0.5445	4.3281	Accept
	0=1?	0.2282	4.3137	Accept
	1=2?	0.3163	4.3137	Accept
Power	0=1?	3.5219	4.3281	Accept
	0=3?	3.2553	4.3281	Accept
	0=2?	2.2794	4.3137	Accept
	2=1?	1.2425	4.3281	Accept
	2=3?	0.9759	4.3137	Accept
	3=1?	0.2667	4.3137	Accept
Temp.	2=3?	1.3165	4.3281	Accept
	2=1?	0.8267	4.3281	Accept
	2=0?	0.0976	4.3137	Accept
	0=3?	1.2189	4.3281	Accept
	0=1?	0.7291	4.3137	Accept
	1=3?	0.4898	4.3137	Accept

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