PRINCIPAL FACTORS DETERMINATION IN THE GROWTH OF THIN FILMS USING TAGUCHI'S TECHNIQUE

E. R. Vázquez-Cerón, V. R. Barrales-Guadarrama, E. M. Rodríguez-Rodríguez,
R. Barrales-Guadarrama.

UAM Azcapotzalco. Av. San Pablo # 180, Col. Reynosa
Azcapotzalco, 02200. México, D.F., México

Email: vrbg@correo.azc.uam.mx

ABSTRACT

A methodology to improve the production and quality of deposited thin films has been developed for the PLD technique. The main purpose is the optimization of electrical and transport properties of the films through the characterization of growth parameters concerning some important characteristics of the material such as roughness, resistance, homogeneity, etc. It improves the production process of thin films, and it also decreases costs, conserving the quality at the same time. The use of interpolations and orthogonal arrangements allows to evaluate the growth parameters and to predict results eliminating the need to repeat experiments.

1. INTRODUCTION

The difference between Taguchi´s [1] and classical off-line quality control is that, while classical statistics place emphasis on the producer's risk, Taguchi's statistics weight the total cost in the life cycle of the product, and it evaluates the test results including both, the producer´s and the consumer´s points of view. That means that the product will have a minimum cost and, as a consequence, a better quality with intended function as well as less defects [2]. Taguchi's philosophy is based on two fundamental concepts: 1) The product cost goes beyond the price of the article; it has to include its performances, and 2) Improving the manufacturing process to achieve better products. Both of these concepts are summarized in the following points:

I) The loss function states that quality must be defined in monetary terms; in other words, the bigger a variation of one specification with respect to the nominal value, the bigger the money loss which is paid by the consumer. See Figure 1. At this point, it is necessary to mention that there is tolerance involving the product nominal value, where ll and hl shows the lowest and highest limits, respectively.

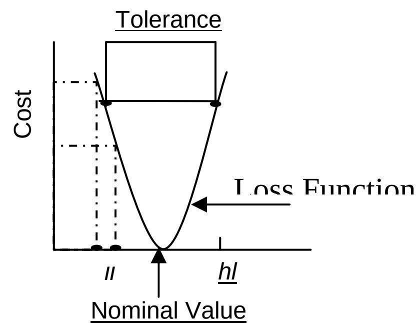

Figure 1.- Taguchi's loss function, quantities that present variability of the process,

II) The improvement and the variability, have to do with the production process and the reduction of variability in relation with the objective value.

III) In the product design process, the product quality determines the final cost.

IV) With respect to the optimization of the product design, technology is not enough. Constant innovation is necessary to improve the product design.

V) Also, regarding the optimization of the process design, it is necessary to verify the product process constantly in order to prevent the consumer from detecting the failure. If the design process is verified constantly, it will be possible to detect any failure in the process.

In this paper, Taguchi's technique is used with orthogonal arrangements, [3] which allows to determine the interaction among several variables to produce thin-film superconductors with good properties. This technique optimizes the process to get the best magnetic and electric properties of films and to reduce the production costs.

2. PROBLEM DESCRIPTION

Thin film is obtained through an ablation process [4]. During this process, a pulsed high-energy laser beams on a target causing sublimation. Vapor diffuses and becomes deposited on the substrate creating a thin film superconductor; this process is known as Pulsed Laser Deposition (PLD) [5]. Thin film deposition time varies by few seconds. This time depends on the thickness of the required film. The requirement is to obtain a thickness of around 500 nanometers. After the deposition, it is sometimes necessary to anneal to get the optimum oxygen absorption and, as a result, the highest critic temperature (T_c).

One of the most common tests used to determine the thin film quality is measuring the magnetic susceptibility. This quality depends on different parameters like substrate temperature, vacuum pressure, substrate-source distance, ablation time, and oxygen pressure, among others. On the other hand, a loss function can be built to determine the cost of poor thin film quality using a cost as a reference value. As an example, oxygen pressure can be analyzed when it goes out of the allowed ranges. Taguchi's technique uses the following mathematical relation:

$$L = k(y - m)^2 \qquad (1)$$

Where L is the loss associated with the particular oxygen pressure y, m is the nominal value and k is a constant; k depends on the cost of the highest limit *(hl)* or the lowest limit (*ll*) of the specification.

The loss associated with any part of the thin film that depends on the oxygen pressure value can be computed; therefore, the objective is lowering the pressure limits getting cheaper production costs.

3. STUDY METODOLOGY, ORGANIZATION AND WORK DISTRIBUTION

As an initial step, a null hypothesis, which will be rejected or accepted at the end of the analysis, is established. An orthogonal arrangement is used. This arrangement is based on Taguchi's matrix with seven factors, with two levels each (lower (1) and highest (2)), and eight experiments are done. This particular case is known as L8.

Using a factorial design involves performing 2^7 experiments in order to evaluate all the factors on two levels. On the other hand, employing Taguchi's matrix involves using an orthogonal arrangement, and the number of experiments is reduced to 8. In this case, it is possible to get the effect on one factor with no effect on any other. Along with Taguchi's technique, an ANalysis On VAriance (ANOVA) must be conducted. Such an analysis consists of fragmenting the information variation into the total number of variations classified by cause, which would not be possible if the arrangement is not orthogonal. Table 1 shows the orthogonal arrangement proposed by Taghuchi for an L8.

	FACTORS						
EXPERIMENT	1	2	3	4	5	6	7
1	1	1	1	1	1	1	1
2	1	1	1	2	2	2	2
3	1	2	2	1	1	2	2
4	1	2	2	2	2	1	1
5	2	1	2	1	2	1	2
6	2	1	2	2	1	2	1
7	2	2	1	1	2	2	1
8	2	2	1	2	1	1	2

Table 1. Orthogonal Arrangement for L8

Numbers 1 to 8 refer to the number of experiments. Each column is an orthogonal arrangement that contains two categories represented by 1 and 2, which indicates two levels for each factor in the growing process. It generates a series of combinations that involves categories and factors for each experiment. In Table 1, it can be observed that in the two first columns, two pair of two possible combinations are repeated: (1,1), (1,2), (2,1), (2,2). If each of these four combinations appears the same number of times in a pair of columns, it means that they are balanced and orthogonal. Table 2 shows how factors related with levels (which are represented by number 1 to refer to a low level, and number 2 to refer to a high level) allow to make the combination in the orthogonal arrangement. With these factors established, roughness (nm) was selected as the qualification parameter to evaluate the quality of the superconductor film. Table 3 shows the experimental roughness obtained in each limit as well as the necessary values to start the statistical analysis.

FACTORS	Low LEVEL (1)	High LEVEL (2)
Source-Substrate Distance	2 cm	6 cm
Substrate Temperature	400°C	650°C
Oxygen Pressure	200 mT	270 mT
Deposition Time	20 seg	2 mín
Kind of anneal	*in situ*	*ex situ*
Beam	No focus	Focus
Vacuum Pressure	7.5×10^{-2} mT	.075 mT

Table 2. We consider seven principal factors in the growth of thin film with

EXPERIMENT	ROUGHNESS THIN FILM 1	ROUGHNESS THIN FILM 1	SUM	MEAN	VARIANCE	STANDARD DEVIATION
1	100	98	198	99	2	1.4142136
2	150	160	310	155	50	7.0710678
3	50	55	105	52.5	12.5	3.5355339
4	300	305	605	302.5	12.5	3.5355339
5	130	135	265	132.5	12.5	3.5355339
6	140	145	285	142.5	12.5	3.5355339
7	120	122	242	121	2	1.4142136
8	150	154	304	152	8	2.8284271

Table 3. With the experimental data of roughness, the statistical analysis starts and solves the orthogonal arrangements

To conduct the null hypothesis test, an ANOVA is performed. The objective of the analysis is to compare the mean of two or more samples of average roughness. The comparison procedure consists of the analysis of data variations. In order to perform the ANOVA, we assume: (1) Independent samples (samples taken from many populations are independent from each other). (2) Populations have normal distribution. And, (3) Same standard deviation.

α=0.05, indicates that the thin film presents the same average roughness. Table 4 summarizes the partial results obtained.

With the data arrangement on table 4, the relation of Means Square (MS) defined as the quotient of the Sum of Squares (SS) and the Free Degree (FD), the F distribution can be obtained and becomes decisive to accept or reject the null hypothesis.

$$MS = \frac{SS}{FD} \quad \ldots(2)$$

ANOVA				
SUMMARY	SPECIAL CHARACTERISTICS			
GROUPS	NUMBER OF OBSERVATIONS	SUMMATORY	MEAN	VARIANCE
EXPERIMENT 1	2	198	99	2
EXPERIMENT 2	2	310	155	50
EXPERIMENT 3	2	105	52.5	12.5
EXPERIMENT 4	2	605	302.5	12.5
EXPERIMENT 5	2	265	132.5	12.5
EXPERIMENT 6	2	285	142.5	12.5
EXPERIMENT 7	2	242	121	2
EXPERIMENT 8	2	304	152	8

Table 4. Partial results to apply ANOVA

The ANOVA procedure uses a continuous distribution of probability called F distribution. In this experiment the null hypothesis formulates that the measurements are equal (H_0: $\mu 1 = \mu 2 = \mu 3 = \mu 4 = \mu 5 = \mu 6 = \mu 7 = \mu 8$), while the alternate hypothesis, with a significant level of

$$f = \frac{MS_{experiments}}{MS_{film}} \ldots(3)$$

With the ANOVA results, the F distribution can be computed. See Table 5.

ANOVA					
SOURCE OF VARIATION	SUM OF SQUARE	FREE GRADE	SQUARE MEAN	VALUE OF f	DISTRIBUTION F $f_{0.05}(1,7)$
EXPERIMENTS	71861.4375	(8-1)=7	10265.91964	134.085481	5.59
FILM	76.5625	(2-1)=1	76.5625		
ERROR	35.4375	(8-1)(2-1)=7	5.0625		
TOTAL	71973.4375	(8*2)-1=15			

Table 5. Results of the F distribution According to Table 5, the following analysis can be done:

1. $H_0: \mu_1=\mu_2=\mu_3=\mu_4=\mu_5=\mu_6=\mu_7=\mu_8=0.$
2.- H_1 = at least one of the μ_i's is not zero.
3. $\alpha = 0.05$.
4.- Critical regions: $f_{0.05}$ (1,7) > 5.59.
5.- Decision: As f = 134.085481 and $f_{0.05}$ is 5.59, then the null hypothesis is favorable to the alternate hypothesis.

4. RESULTS

The Taguchi´s technique analyzes the appropriate combination of several factors accord with the levels implicate in the production of process to rejected on reduce the roughness of thin film. This combination is represented in the Table 6, and is illustrated in the Figure 2.

FACTOR	ROUGHNESS MEAN AT LEVEL 1	ROGUHNESS MEAN AT LEVEL 2
(1)	609	548
(2)	529	628
(3)	527	630
(4)	405	752
(5)	446	711
(6)	686	471
(7)	665	492

Table 6

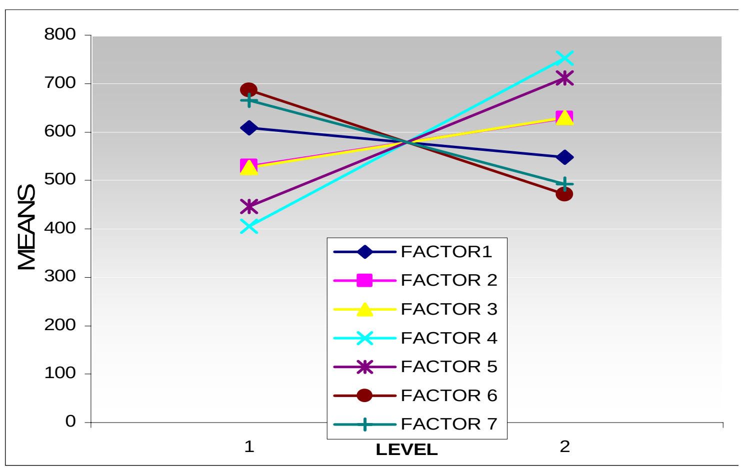

Figure2

Table 6 and Figure 2 show the comparison between level 1 and level 2 respecting each factor

The graph in the Figure 2 shows that when passing from level 1 to level 2, the factors that make the rough to reduce are factors 1, 6 and 7. On the other hand, the factors that make the rough to increase are the factors 2, 3, 4 and 5. These results show that in order to produce a good thin film, the right combination must to be like on the Table 7.

FACTOR		LEVEL	
1	Source-Substrate Distance	2	6 cm
2	Temperature Substrate	1	400°C
3	Oxygen Pressure	1	200 mtorrs
4	Deposition Time	1	20 seg
5	Anhela	1	*in situ*
6	Beam	2	Focus
7	Vacuum Pressure	2	$7.5\text{x}10^{-2}$mtorrs

Table 7. Indicates the level that each factor must reach to reduce roughness

5. DISCUSSION

The necessary parameters to obtain thin films with less roughness are determined using Taguchi's Technique according to an F distribution. This is shown in Table 7 with 95% certainty. Based on the different factors from growth influencing the roughness properties, it is possible to relate it to its magnetic characteristics or transport. Taguchi's technique demonstrated that it can be applied to any industrial process to improve the control and the quality of a product or a process no matter the number of variables involved in it. If a deeper analysis is required, a greater amount of variables involved will have to be taken into account. It will be managed to reduce the trust interval, that is to say, the prediction becomes more accurate. This method turns out to be relatively easy to apply, with reliable results, in comparison with other classic statistical methods in which, to obtain reliable results, it is necessary to conduct a great number of tests.

6. REFERENCES

[1] Phillip J. Ross, Taguchi Techniques for Quality Engineering, (Ed. McGraw-Hill, México),(1987).
[2] K.Ishikawa, Guide to Quality Control,(Asian Productivity Organization, Tokyo, Japan, 1983)
[3] R. E. Walpole y R. H. Myers, Probabilidad y Estadística, (Ed. McGraw-Hill, México), 624, (1992).
[4] P. A. Redhead, The Physical Basis of Ultrahigh Vacuum, (Ed. Springer-Verlag New York), 2000
[5] Chrisey B., Douglas, Pulsed Laser Deposition of Thin Films, (ED. Wiley Interscience) 613, 1994

Authors Biography

Ernesto Rodrigo Vázquez Cerón

He received a BE. in physics engineering and a master's degree in biomedical enginering from Universidad Autonoma Metropolitana-Izt., Mexico in 2001. His main field of interest is centered on digital signal procesing from acoustic system. Mr. Vázquez has collaborated with the Sensors and Signal Processing team as an Associate Professor in the Electronic Department at UAM-Azc., Mexico since 2000 .

Víctor Rogelio Barrales Guadarrama

He received a BE in physics enginering , a master's degree in electrical engineering and a Ph.D. degree from CINVESTAV, Mexico. His doctoral work was on laser ablation. His main field of interest is centered on thin film growth by pulse laser deposition and sputtering. Dr. Barrales worked in the Superconductivity Laboratory from 1991 to . At present, he is joining the Sensors and Signal Processing team as a Full Professor in the Electronic Department at UAM-Azc., México.

Ezequiel Rodríguez Rodríguez

He received a BE in physics engineering and a master's degree in biomedical enginering from Universidad Autonoma Metropolitana-Izt., Mexico in 2000. His main field of interest is centered on the development and application of several sensors and digital signal processing. Mr. Rodriguez has been part of the Sensors and Signal Processing team since 1999. At present, he is a Full Professor in the Electronic Department at UAM-Azc., Mexico.

Raymundo Barrales Guadarrama

He received a BE. in electronics engineering- specialization in digital systems and the master's degree in biomedical enginering from Universidad Autonoma Metropolitana-Izt., Mexico in 2003. There, he developed processing techniques for heart ultrasounds. Mr. Barrales was a founding partner of Specialized Electronics and held the position of Sales Director from 1987 to 1992 at the same company. He also collaborated with other companies in the field of electronics where he acquired work experience. His main field of interest is centered on the development and application of several sensors and digital signal processing He has been part of the Sensors and Signal Processing team since 1999. At present, Mr. Barrales is a Full Professor in the Electronic Department at UAM-Azc., Mexico. He has also been a member of IM009 and 004-3351 societies of the IEEE.