Experimental Strategy—
Application of Taguchi's Quality Engineering Method To Zinc Phosphate Coating Uniformity

By Shih-Min Lin and Ten-Chin Wen

Plating process quality can be improved at no additional cost or even at a saving by Taguchi's quality engineering approach. The experimental strategy described in this paper is different from traditional experimental design, in that it is concerned with reducing variance in the manufactured product by determining the best settings for the manufacturing process parameters without tightening tolerances on them. By employing dynamic characteristic statistics, a process for uniform zinc phosphate coating can be determined from the linear relationship between coating substrate geometric area (signal factor) and coating weight (output characteristic).

Traditional statistical experiment design explores the cause-and-effect relationship between input and response parameters. The theory is aimed at deriving a mathematical equation relating mean response to model factors. Fractional-factorial and response surface designs, developed by Box et *al.,*[1-4] are commonly used in traditional experiment design. Fractional-factorial design is based on assumptions derived from the fact that higher order interaction effects, as well as the interaction effects between minor variables, are often negligible and can, as a result, be ignored. Accordingly, the trial runs can be reduced and main/interaction effects can be identified through the defining relation.[1,2,4] Response surface methodology[3,4] in conjunction with central composite design, estimates the factor curvature effects, with their optimum condition being determined by means of a mathematical regression equation and a response surface contour plot.

For extensive reviews of experimental strategy relating to plating studies, the reader is referred to Dini and references therein.[5] Recently, Traut et *al.*[6] and Mandich[7] employed response surface and fractional factorial design in the optimization of gold plating baths and electroless plating catalyst baths, respectively. Lin and Wen applied orthogonal array and central composite design in the optimization of additive concentrations in Watts nickel plating baths.[8] These statistical methods enable examination of system variable effects over their entire operating range and determine an optimum operational parameter.

Because statistical experimental design generally assumes that response variance remains constant for all model factor levels, reductions in variability critical for quality improvement are, as a consequence, ignored, The Taguchi Method, by contrast, is concerned with minimizing variance in the manufacturing process, This is achieved through a transformation of repetitive data to other values which represent a measure of the variation within a trial when noise factors are present. The Taguchi Method strives to reduce cost and promote quality by determining the best settings for the parameters. These settings represent lowest product variance and manufacturing cost.

Over the past two years, several papers in this journal[9-11] have employed the Taguchi Method in plating studies. The influence of various parameters on dendrite formation in an acid copper system was studied by Wan and McCaskie, who used an orthogonal array to plan the experiment and analysis of variance (ANOVA) to determine the significant factors.[9] An orthogonal array was also employed in the study of the main/interaction effects of process variables on current efficiency in hard chromium pulse plating.'" Actually, all of the above approaches are similar in form to the fractional factorial design method, because the purpose of each was to reveal the effects of process variables. A successful application by Wen and Lin of Taguchi's quality engineering method minimized variability in the aluminum coloring process through the use of two kinds of S/N ratios (*LB* and NB) in evaluating the process parameters

Table 1
Orthogonal Array Classification

Type of system	Standard O.A.*	Mixed level**
2-level series	L_4, L_8, L_{16}, L_{32}	L_{12}
3-level series	L_9, L_{27}	L_{18}, L_{36}

*Interactions can be assigned by using linear graphs or interaction tables.
**Effects of interactions cannot be assigned because the interaction between any two columns is partially confused with the remaining columns. Columns give information on main effects only.

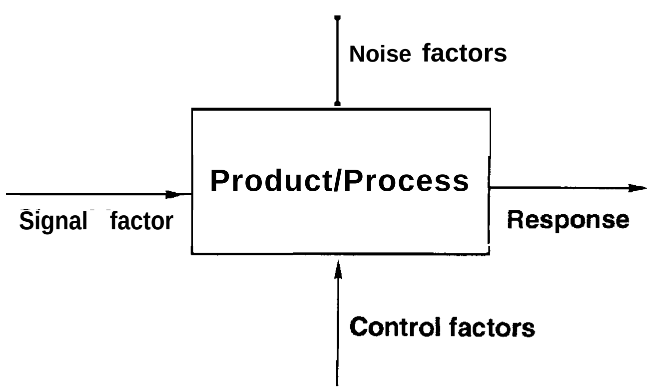

Fig. 1—Taguchi P-diagram of product/process.

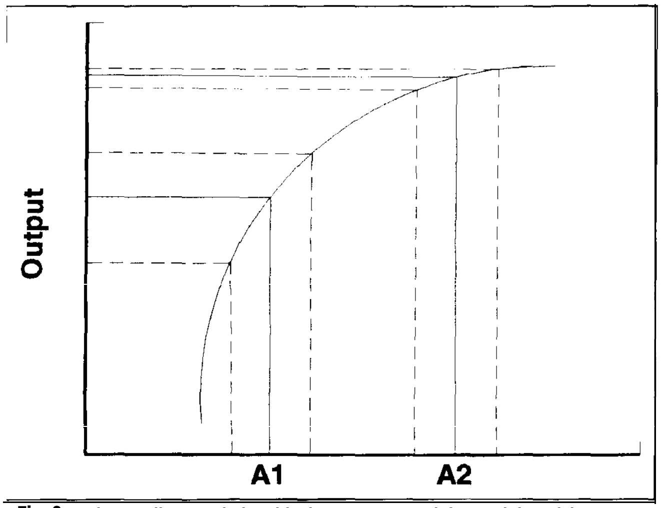

Fig. 2— The nonlinear relationship between control factor (A) and its output characteristic.

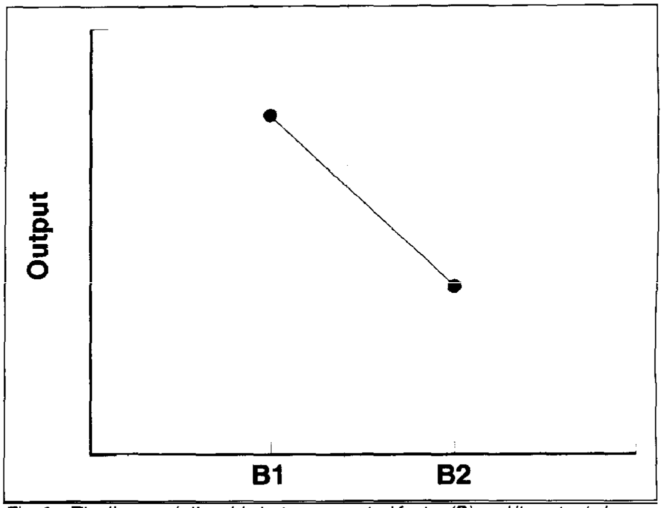

Fig. 3—The linear relationship between control factor (B) and its output characteristic.

influencing variability and mean response.[11] This paper introduces and attempts to clarify the concept and methodology behind Taguchi's quality engineering method by applying the methodology to the study of zinc phosphate coating uniformity.

Engineering Quality by Design

Taguchi views the design of a product or a process as consisting of three steps.[12-14] The first, system design, represents the phase where new concepts, ideas, methods, etc., are generated for the purpose of providing new or improved products to customers. This highly creative step, in which the experience and skill of the designer play an important role, usually takes place without regard to quality or cost restrictions. The second step, parameter design, is crucial for minimizing product variation and can be done at no extra cost or even at a saving, and represents, by far, the most important approach in quality improvement. In this step, Taguchi explored the orthogonal array in planning experiments, as well as investigating the limiting of experimental runs and S/N ratios for measurements of product quality. The last step, tolerance design, improves quality by tightening product or process parameter tolerances in order to reduce performance variation. This step is implemented only after parameter design, inasmuch as a compensation mechanism[12] should also be considered as a tolerance factor to be optimized along with the component tolerances.

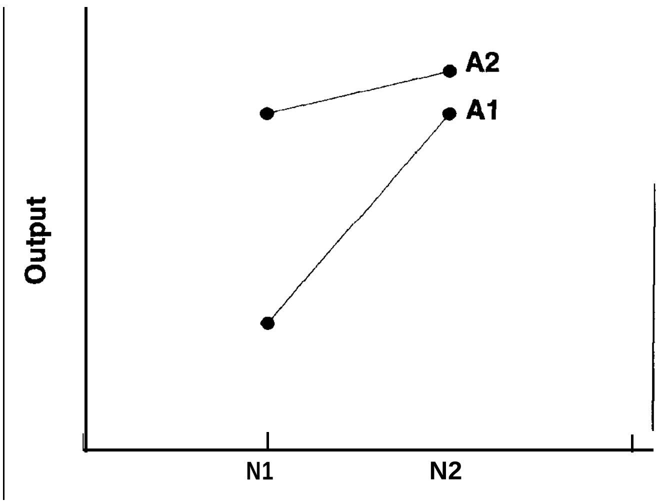

Fig 4—Interaction effect of noise factor (N) and control factor (A) on output characteristic.

In contrast with traditional experiment design, parameter design ignores interaction effects between controllable variables, because investigation of these effects requires a considerable expenditure of time and materials.[4,12] In traditional experiment design, to study the interaction effects of two controllable factors, a resolution V fractional factorial design is usually adopted.[4] With such a method, for example, sixteen experiments, denoted as 2_V^{5-1}, are required in studying the effects of five process variables (main and two factor interaction effects). The defining relation, I = ABCDE, suggested by Box et al.,[4] was established to identify those relationships that exist between the effects. In such design, all main effects are muddled with four-factor interactions, and those for two-factor interactions are confused with three-factor interactions. It should be noted that if the basic assumption of Box et *al.*[4] is employed, where higher-order interaction effects (three- and four-factor interaction in this case) are often negligible and can be ignored, the main and two-factor interaction effects can be obtained without confusion with the higher-order interaction effects. Actually, 16 experiments can provide information on 15 process variables if their interaction effects can be ignored (i.e., the process variable effects are linearly additive.) This is a fundamental assumption of the Taguchi Method.

This additive nature is strongly influenced by the choice of quality characteristic. Taguchi views the quality characteristic of a product or process as consisting of four levels. The first level, customer quality, is expressed by the customer, for example, the appearance or uniformity of a deposited film in the nickel/chromium plating process. To meet the customer's needs, engineers often try to convert the customer quality aspect into the second level—a more specific quality characteristic, termed the specific quality. Morphology and structure, or the thickness of a deposited film, are examples of specific quality. Specific quality represents a tangible engineering factor on which research can be conducted and which entails investigation of the numerous interactions between the controllable variables. The large extent of these interactions is the reason why, in traditional experiment design, a great deal of attention is paid to the study of interaction effects. These interactions, if present and not fully accounted for, can invalidate laboratory results, leading to product nonreproducibility during manufacturing.

The third level of quality, robust quality, is distinguished from the previous two levels by inclusion of information on variability. In contrast, such specific qualities as thickness or number of

Table 2
Phosphating and Stripping Process Solution Compositions

Solution No.	Type of solution	Composition	Operating temperature	Cycle time (min)
1	Alkaline cleaning	NaOH 4 g/L	80 °C	2
2	Hot rinse	Distilled water	75 °C	1
3	Cold rinse	Distilled water	Room	1
4	Acidic cleaning	HCl 25%	Room	10, 15 or 20
5	Phosphating	ZnO H_3PO_4 $NaNO_2$ $Ni(NO_3)_2 \cdot 6H_2O$ NaH_2PO_4	70,80 or 90 °C	5, 10 or 15
6	Stripping	CrO_3 50 g/L	75 °C	15

defects only yield information on the mean values and fail to provide information on variability. Because it is impossible to reduce variability using such quality characteristics for data analysis, Taguchi proposed a transformation of the specific quality to robust quality (i.e., the signal-to-noise [S/N] ratio), in order to measure average response and variation simultaneously. The statistical meaning of the S/N ratio will be discussed in more detail in the next section.

The fourth level, functional quality, achieves a level of quality in the technical function of an engineered product by treating the manufacturing process not as static, but as one that can be adjusted, with the ultimate goal of obtaining an “ideal” product. For example, a product possessing ideal resistance would mean that its output voltage is proportional to the input current, obeying Ohm’s law, while the ideal function of an injection molding machine is to yield molded parts with dimensions proportional to that of the mold. Accordingly, these characteristics are often called dynamic characteristics.[12]

In general, the ideal function of a product or a process can be found from the linear relationship between one input variable (signal factor) and its output characteristic under situations where noise factors exist. This concept can be easily extended to the fields of electroplating and surface treatment. For example, an ideal electroplating process is achieved when the thickness of the plated film is proportional to the plating time. Accordingly, an ideal plating process parameter setting can be determined by studying the linear relationship between the plating time (signal factor) and the thickness of the plated film (output characteristic). Acting on the basis that, in a uniform coating process, the weight of phosphate coating is proportional to the coating area, this paper employs Taguchi’s method in the study of zinc phosphate coating uniformity, as mentioned earlier.

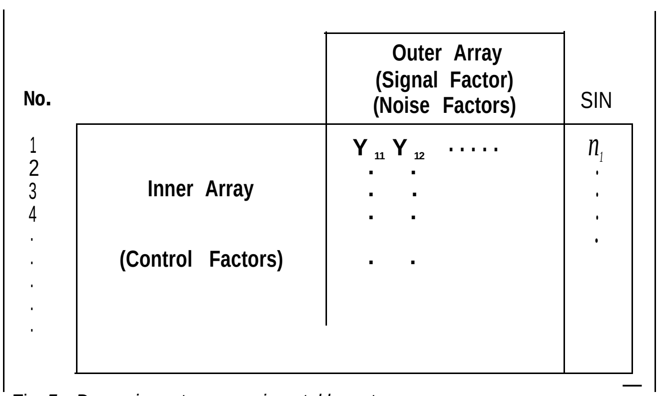

Fig. 5—*Dynamic system experimental layout.*

S/N Statistics

S/N ratios have been used in evaluating and improving the quality of communication systems since the beginning of this century. In the 1970s, Dr. Taguchi began using the S/N ratio concept in evaluations and improvements of measuring systems.[14,15] Recently, S/N ratios have become an important index with which to evaluate the quality of a product or a process by addressing the factors that affect variability.[12,16]

In our previous paper,[11] we employed three kinds of S/N ratios (defined by Taguchi), depending on the type of characteristic involved: the larger-the-better (LB), the nominal-best *(NB),* and the smaller-the-better (SB). The ideal output quality characteristics in these three cases are, respectively, equal to infinity, a specific target, and zero. This index was constructed by Taguchi so that quality is better when the S/N ratio is larger.

The S/N ratio is essentially the inverse of the coefficient of variation (CV),[16] defined as:

$$CV = \frac{SD}{\bar{y}}, \tag{1}$$

where SD and bar y are the standard deviation and mean, respectively. Note that as variation around the average gets smaller, CV decreases. Accordingly, Taguchi constructed SN ratio formulas as signal (mean) divided by noise (SD) expressed as follows:

$$SN = 20 \log \frac{\bar{y}}{SD}. \tag{2}$$

In the case of the smaller-the-better, because the mean of the ideal output is equal to zero, the above equation can be expressed as

$$SN_{SB} = -20 \log(SD) \tag{3}$$

where

$$SD = \left(\frac{1}{n}\sum_{i=1}^{n} y_i^2\right)^{1/2}. \tag{4}$$

By considering the reciprocal of the quality characteristic, a larger-the-better type problem can be transformed into one of smaller-the-better. Accordingly, the objective function to be maximized in this case is given by:

$$SN_{LB} = -10log\,(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}). \quad (5)$$

In the case of a nominal-best type problem, the ideal output is equal to a specific target. Inasmuch as the square of the expected value of the mean $([E(\bar{y})]^2)$, is equal to $\frac{1}{n}$ (Sm-Ve), SN_{NB} is given as

$$SN_{NB} = 10log\,(\frac{1}{n}\frac{(Sm - Ve)}{Ve}), \quad (6)$$

where

$$Sm = \frac{(\Sigma yi)^2}{n} \quad (7)$$

is the variation of the mean; and

$$Ve = \sum_{i=1}^{n}\frac{(y_i - \bar{y})^2}{n-1} \quad (8)$$

is the sample variance.

These three cases are often called static or non-dynamic characteristics, because the target is a fixed value. In the case of dynamic characteristics, on the other hand, the ideal output is $y = \beta M$, where M, y, and β represent, respectively, the input signal factor, the output characteristic, and the resulting coefficient. With dynamic characteristics, there are three objectives to accomplish: improve linearity, increase sensitivity and reduce variability. These objectives can be evaluated together in one equation:

$$SN_{\beta} = \frac{sensitivity}{variability} = \frac{\beta^2}{\sigma^2}. \quad (9)$$

In this case, the square of the expected value of β, $([E(\beta)]^2)$, is equal to $\frac{1}{r}$ $(S_B - Ve)$, accordingly, SN_B is given as

$$SN_{\beta} = 10log\,(\frac{1}{r}\frac{(S_{\beta} - Ve)}{Ve}), \quad (Io)$$

where

$$S_{\beta} = \frac{\Sigma_{i=1}^{n}(M_i y_i)^2}{\Sigma_{i=1}^{n} M_i^2} \quad (11)$$

is the sum of the square of β and r= $\Sigma_{i=1}^{n} M_i^2$.

Classification of Parameters

Factors (or parameters) influencing the quality characteristic or response of a process or a product can be grouped into the following classifications: control, signal, and noise. The relationships between parameters and process/product were represented by the Taguchi P-diagram, as shown in Fig. 1. Factors affecting variation are classified as control factors and are used to improve stability. Because control factors can be used to exploit nonlinearity to find a combination of product parameter values that yields the smallest quality characteristic variation around the desired target value, control factor identification represents the most important task in parameter design. The nonlinear relationship between control factor and output characteristic is shown in Fig. 2. From this figure, it is clear that the control factor at a high level value (A2) possesses smaller variation than that at a low level value *(A 1).* As a result of their nonlinear relationships, these different product parameter combinations can yield quite different quality characteristic variations.

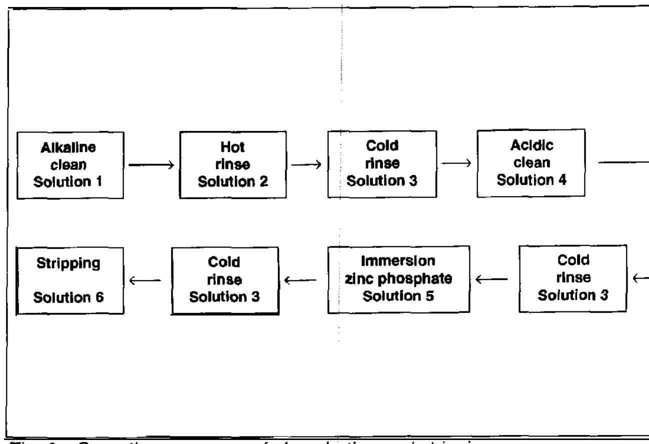

Fig. 6—Operating sequence of phosphating and stripping process.

Factors not affecting variation are classified as signal factors, and are used in adjusting the mean to the desired value. The relationship between signal factor and its output characteristic is linear, shown for an ideal dynamic case in Fig. 3. A typical signal factor in plating process is plating time, with the thickness of the plated film acting as output characteristic. In our previous paper[11] we showed that the anodizing time in an aluminum coloring process represented a signal factor with which the thickness of aluminum oxide film could be controlled.

Factors for which settings (or levels) are difficult to control in the field or for which settings are expensive to control, are considered to be noise factors. Incorporation of noise factors into parameter design is crucial for evaluations of control factors possessing the lowest variability over variations in noise factor levels. Actually, the S/N ratio, a measure of a function's stability, is an estimate of the interaction between controllable and noise factors. This situation is shown in Fig. 4, where the control factor *A* at a high level value (A2) is less sensitive than a low level value *(A 1)* to the noise variation, and consequently possesses a higher S/N ratio. Typical noise factors in the plating process are the shape of the plated film and locations where differences in coating uniformity are present. Traditionally, the ability of a particular plating bath to yield a uniform coating has been known as its throwing power, with a value of 100 percent being perfect. For a review of throwing power in determining plating processes, see Ref. 17. Alternatively, an

Table 3
Factors and Levels for Array $L_{18}(2^1 \times 3^7)$

Factors		Level 1	Level 2	Level 3
A $Ni(NO_3)_2 \cdot 6H_2O$	(g/L)	2	3	
B Acid-clean time	(min)	10	15	20
C Phosphating temp.	(°c)	70	80	90
D Phosphating time	(min)	5	10	15
E ZnO	(g/L)	0.5	1.0	1.5
F H_3PO_4	(g/L)	3	5	7
G $NaN0_2$	(g/L)	0.15	0.3	0.5
H NaH_2PO_d	(g/L)	20	25	30

Signal Factor	Level 1	Level 2	Level 3	Level 4
M Geometric area of low-carbon steel (cm^2)	20	50	100	200

ideal plating process can be determined by planning experiments where plating time, plating film thickness, and plated film locations (where differences in coating uniformity are present), are represented as signal, output, and noise factors, respectively.

In these instances, a dynamic S/N ratio is used in determining the best settings for the controllable factors, A typical layout of this kind of experimental design is shown in Fig. 5.

Orthogonal Arrays

The Taguchi Method contributes to experimental design of orthogonal arrays (OA), linear graphs and interaction tables, [12-15] all of which provide an efficient way of studying a number of parameters with a minimum number of tests. Orthogonal arrays can be classified on the basis of the number of assigned factors and levels, as well as to whether they check for interactions. Orthogonal array classifications are summarized in Table 1. Selection of the OA to be used is based on the quantity of factor, level, and interaction information the engineer possesses or chooses to study (i.e., the number of main effects and interactions),

For the purpose of reducing trial runs and achieving a robust result, $L_{12}(2^{11})$, $L_{18}(2^1 \times 3^7)$, and $L_{36}(2^{11} \times 3^{12})$ arrays are recommended by Taguchi, inasmuch as the interaction between any two columns is partially confused with the remaining columns.[12,13] The layout of these OAs can be easily obtained from several references. [12-15] Note that the number of assigned levels is fixed in a particular array.

Application

Zinc phosphate coating of low-carbon steel plates **was** performed according to the well-known operation sequence shown in Fig. 6, with typical solution compositions given in Table 2.[1]* The effects of the following parameters on the uniformity of the coating were investigated: (A) $Ni(NO)_2 \cdot 6H_20$ concentration; (B) cleaning time in acidic solution; (C) phosphating temperature; (D) phosphating time; (E) ZnO concentration; (F) H_3PO_4 concentration; (G) $NaNO_2$ concentration; and (H) NaH_2P0_4

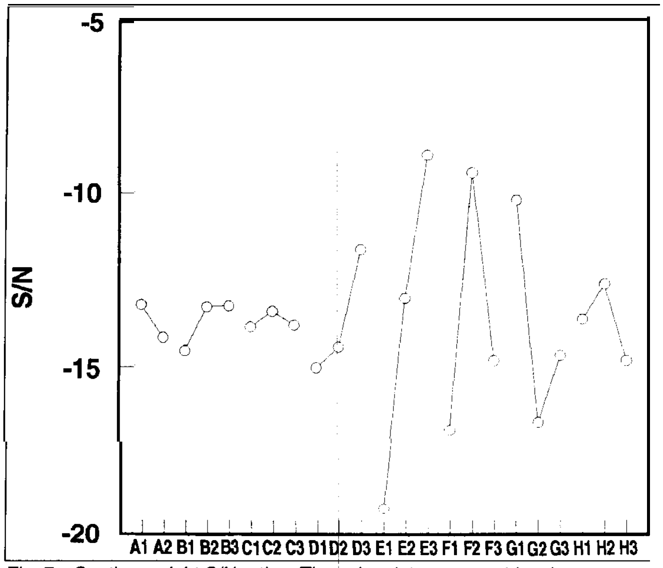

Fig. 7—Coating weight S/N ratios. The subscripts represent levels.

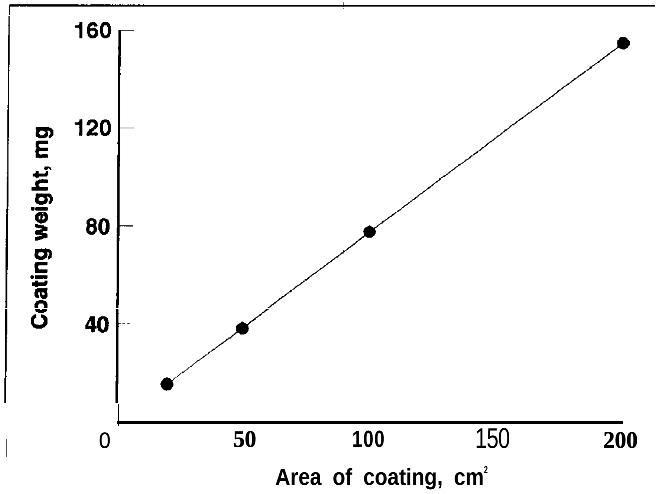

Fig 8—Zinc phosphate coating weight as a function of coating plate area.

Table 4
Experimental Coating Weight Data (mg) and Calculated Ratios (dB)
For Array $L_{18}(2^1 \times 3^7)$; M1, M2, M3, and M4 represent signal factor levels

	A	B	C	D	E	F	G	H	M1	M2	M3	M4	S/N
1	1	1	1	1	1	1	1	1	7.6	20.1	35.1	62.2	-19.88
2	1	1	2	2	2	2	2	2	13.6	34.0	76.9	150.4	-10.24
3	1	1	3	3	3	3	3	3	12.1	27.5	58.6	110.9	-9.80
4	1	2	1	1	2	2	3	3	10.0	25.6	53.9	101.2	-10.47
5	1	2	2	2	3	3	1	1	18.0	47.9	100.2	195.2	-5.06
6	1	2	3	3	1	1	2	2	19.9	52.9	91.8	224.7	-19.91
7	1	3	1	2	1	3	2	3	1.5	4.1	8.7	25.0	-25.83
8	1	3	2	3	2	1	3	1	14.9	36.6	89.7	183.5	-15.74
9	1	3	3	1	3	2	1	2	15.5	38.8	79.4	161.3	-2.48
10	2	1	1	3	3	2	2	1	14.8	37.2	81.1	158.9	-7.34
11	2	1	2	1	1	3	3	2	3.7	11.1	22.6	59.2	-23.45
12	2	1	3	2	2	1	1	3	17.8	41.4	84.0	145.8	-18.01
13	2	2	1	2	3	1	3	2	14.1	36.3	66.7	126.0	-14.03
14	2	2	2	3	1	2	1	3	17.5	40.8	90.9	186.3	-11.29
15	2	2	3	1	2	3	2	1	10.3	26.5	60.1	136.8	-19.20
16	2	3	1	3	2	3	1	2	25.6	66.9	129.3	252.5	-5.91
17	2	3	2	1	3	1	2	3	6.5	12.6	28.6	52.3	-14.98
18	2	3	3	2	1	2	3	1	13.3	33.2	75.5	136.6	-14.87

concentration. Fixed levels for these eight parameters are given in Table 3. A signal factor (geometric area of low-carbon steel plate), also shown in Table 3, was adopted to study the uniformity of the phosphating process. A standard $L_{18}(2^1 \times 3^7)$ array, shown in Table 4, was deemed suitable for studying these seven 3-level factors and one 2-level factor.

Low-carbon steel plates, according to the sequence in Fig. 6, were first washed with 4 g/L NaOH(alkaline-cleaned) at 70 °C, rinsed with hot distilled water (-70 °C), then rinsed with room-temperature distilled water. The treated samples were washed with 25 percent HCl (acid-cleaned) for 10, 15 or 20 rein, depending on the experiment in question (see Table 3). The treated plates were again rinsed with distilled water, then phosphate, in coating baths containing different concentrations of phosphating chemicals to meet design level requirements, at phosphating times of 5, 10 or 15 rein, and at temperatures of 70, 80 or 90 ‘C, depending on the experiment in question.

The weight of the phosphate coating was determined by subtracting the weight of the coated plate before and after stripping.[19] The stripping procedure consisted of immersion for 15 min in a 75 g/L chromic acid solution at 75 °C.

Coating weight data and their calculated S/N values are listed in Table 4. To evaluate the uniformity of the phosphating process, the S/N values were calculated, using equation (1 O). The average effects of parameters on S/N values are shown in Fig. 7. An examination of Fig. 7 reveals that phosphating time (D), ZnO(E), H_3P0_4(F), and $NaNO_2(G)$ concentrations are the key factors influencing the uniformity of phosphate coatings. This result is ascribable to the fact that ZnO and H_3PO_4 are the sources of Zn^{+2} and PO_4 ions which react together, generating the coatings,[20] while $NaN0_2$ acts as an accelerator, governing the reaction rate.[21]

The optimum setting was found to *be A1B2C2D3E3F2G1H2.* This setting possesses control factor levels that result in the highest ratio and yield the best coating performance. The result of the confirmation run employing this optimum setting is shown in Fig. 8, and shows a linear relationship between coating weight and geometric area of coating, indicating that a uniform coating process has been achieved.

Conclusions

Quality engineering approaches, developed by Dr. Genichi Taguchi and known as the Taguchi Method, are universally applicable to all engineering fields. Although the design matrices (orthogonal arrays) used in planning the experiments are similar to the method of traditional experiment design, the thought process behind the concept/computations is different. Signal-to-Noise ratio is an important index with which to evaluate the quality of a product or a process under conditions where noise factors exist. To avoid interaction effects between controllable variables, L_{12}, L_{18}, and L_{36} orthogonal arrays are often employed in planning the experiments. Process parameters are classified as control factors that reduce variance and signal factors that adjust the mean. The two-stage goal of the Taguchi Method for process optimization is to improve stability first, after which the mean is adjusted to the target value.

Acknowledgment

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