Surface Finish Optimization in Pulse Reverse Current Electroforming

By K.P. Wong, K.C. Chan & T.M. Yue

The application of pulse reverse current in electroforming is known to result in better surface finishing. Nevertheless, it seems impractical to obtain an optimum surface finish by trial and error methods, as the process itself involves a number of variables. In this study, a Taguchi method was successfully applied to identify the dominant factors in controlling surface roughness. Moreover, an optimum surface finish for electroforming of nickel under the conditions of pulse reverse current was determined. The results show that the positive peak current density and the anodic time were the two most critical factors. The optimum surface finish (ΔR) obtained was 0.05 m; this occurred at a positive peak current density of 870 mA/cm^2, an *off* time of 3 msec, an *on* time of 5 msec and a negative peak current density of 650 mA/cm^2.

Because of high fidelity of shape reproduction from the mandrel, as well as high repetitive forming accuracy as a result of no wear or damage to the mandrel, electroforming has been demonstrated to be a powerful means of fabrication of many micro-devices or products with dimensions in the range of micrometers and even in Ångströms, such as stampers of compact discs and masters of holograms.[1-3] Inasmuch as there is an increasing demand for better surface finishing and tighter dimensional tolerances for micro-devices, many research studies have concentrated on improving the surface finish of a deposit.[3-4] One of these developments was application of pulsed current in electroforming. Recently, it was reported that a significant reduction in internal stress could be obtained when pulse reverse current was used, compared to the use of conventional pulsed current or direct current at the same average current density.[5-6] Qu[6] has also proposed an analytical equation for the development of protrusions in pulse reverse current electroforming, and the theoretical trends were found to be consistent with the experimental findings. It was suggested that in pulse reverse current electroforming, a slight dissolution of the metal surface would take place, but with a higher dissolution rate at the high points of the surface. This would result in a smoothing effect on uneven surfaces.

Although there have been many researches reporting the improvement of surface finishes by employment of pulse reverse current, there remain few attempts to find the optimum surface finish of electroforms by establishing the best level of each controlling factor. In this study, a Taguchi method was applied to identify the dominant factors controlling the surface roughness in pulse reverse current electroforming of nickel, as well as the optimum conditions for achieving the best surface finish.

Experimental Procedure

Materials & Equipment

In the electroforming experiments, the composition of the bath solution was nickel sulfamate 330 g/L; nickel chloride 15 g/L; boric acid 30 g/L and sodium dodecyl sulfate 0.2 g/L. The electrolyte was gently agitated by means of a magnetic stirrer, and the temperature was kept at 50 °C. The initial pH of the electrolyte was 4.2, a typical value used in electroforming. The cathode mandrel was made of stainless steel with dimensions of 100 x 30 x 1 mm, and it was finished with grade 220 emery papers. The surface profile of the mandrel was measured using a Talysurf, and after electroforming under various conditions, the surface roughness and the mass of the deposited layer were measured. The change of surface roughness, ΔR, is defined as follows:

$$\Delta R = R_e - R_s$$

where R_e = the magnitude of surface roughness of the deposit, and R_s = the magnitude of surface roughness of the mandrel.

In this investigation, ΔR is used to define the surface finish of a nickel electroform. The waveform of a pulse reverse

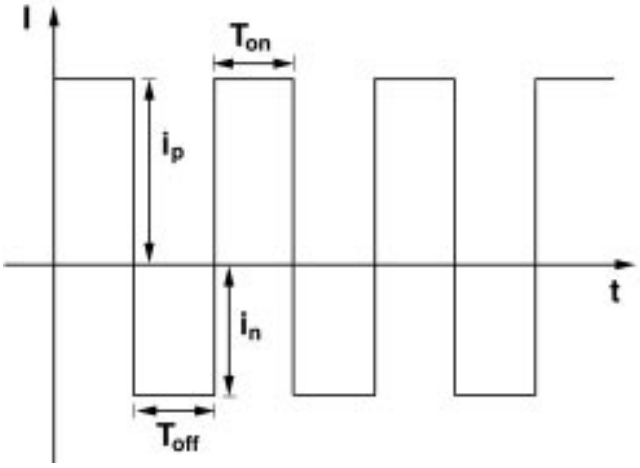

Fig. 1—Waveform of pulse reverse current.

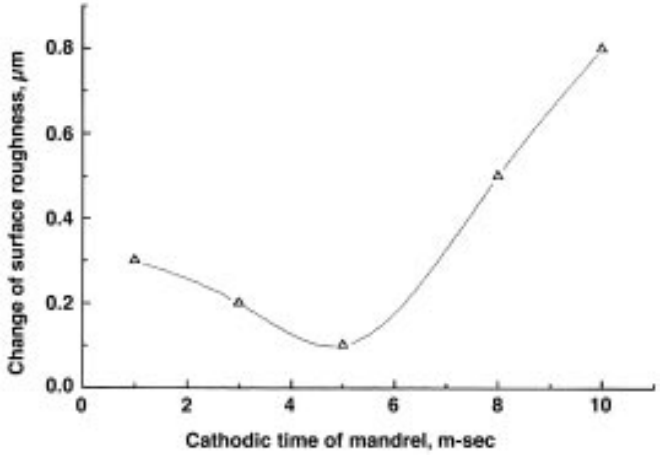

Fig. 2—Effect of cathodic on *time of mandrel (T_{on}).*

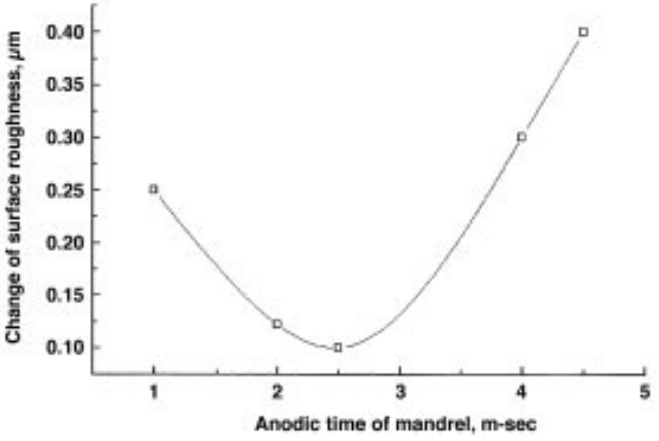

Fig. 3—Effect of anodic off *time of mandrel (T_{off}).*

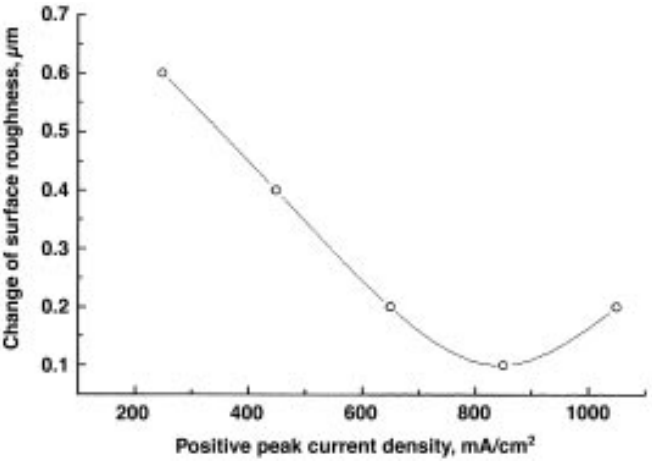

Fig. 4—Effect of positive peak current density (i_p).

current is shown in Fig. 1, which is defined by the cathodic time of the mandrel (T_{on}), the anodic time of the mandrel (T_{off}), the positive peak current density (i_p), and the negative peak current density (i_n). These parameters are considered to be the controlling factors in determining the optimum surface finish of nickel electroforms.

Methodology

According to the Taguchi method and based on the strategy, *the smaller the value of surface roughness, the better*, the following steps were performed in obtaining the optimum surface finish:

(i) a pilot experiment to determine an appropriate range for each controlling factor.
(ii) experimental design using orthogonal arrays.
(iii) matrix experiment to identify the relative merit of each controlling factor.
(iv) analysis of interaction between controlling factors .
(v) prediction of optimum level for each controlling factor.
(vi) final experiment for verification and determination of the optimum surface finish.

Results & Discussion

Results of Pilot Experiment

A pilot experiment was carried out to determine an appropriate range for the cathodic time and the anodic time of the mandrel, and the positive and negative peak current densities.

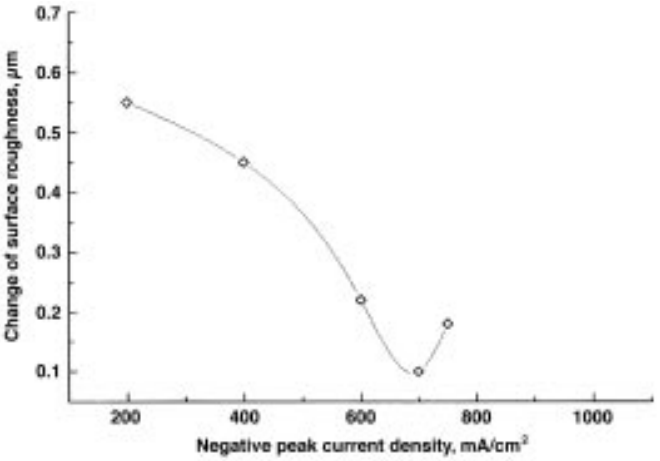

Fig. 5—Effect of negative peak current density (i_n).

(i) *Cathodic time of the mandrel (T_{on})*

The effect of the cathodic time of the mandrel on the surface roughness of the nickel deposits at a constant T_{off} of 0.5 msec, an i_p of 400 mA/cm^2 and an i_n of 400 mA/cm^2, is shown in Fig. 2. The best surface finish was obtained at a cathodic time of about 5 msec. It was considered that when T_{on} was less than 5 msec, an increase would lead to an increase in the deposition over-potential, which is known to promote fine grains.[7] When T_{on} was increased further, however, a significant portion of the applied current was consumed by the reduction of hydrogen ions, which could adversely affect the efficiency of the process, as well as the grain size.[8]

(ii) *Anodic time of the mandrel (T_{off})*

Figure 3 shows the relationship between anodic time and surface finish at a constant T_{on} of 5 msec, an i_p of 400 mA/cm^2 and an i_n of 400 mA/cm^2. A minimum surface roughness value was obtained at an anodic time of around 2.5 msec, which was equivalent to a T_{on}/T_{off} ratio of 2. This finding was in agreement with that of Ismail.[9] In addition to removing protrusions on the surface, anodic pulses can remove precursory defective solid material. The periodic regression of the solid interface removes species blocking the interface along with the solid material, thus yielding an adsorption-free crystalline interface for further grain growth.[10] In this case, R decreased with increasing anodic time if T_{off} was less than 2.5 msec. When the *off* time was increased, however, desorption of $Ni(OH)_2$ crystals and/or H_2 molecules would occur, which could result in activation of the growth centers. Finally, large grains were obtained.[11]

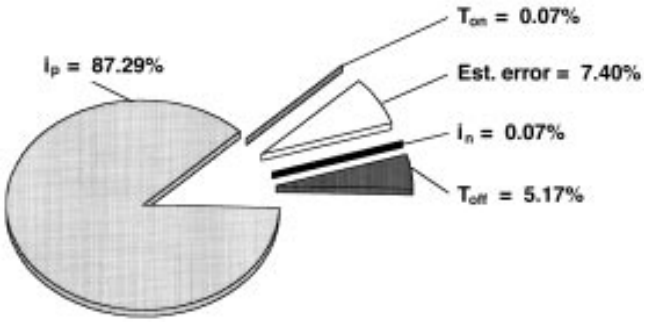

Fig. 6—Relative significance of the controlling factors.

Table 1
Settings for Each Factor in Matrix Experiments

Factor	Unit	Level I	Level II	Level II
A Cathodic Time of Mandrel (T_{on})	m-sec	4	5	6
B Anodic Time of Mandrel (T_{off})	m-sec	2	2.5	3
C Positive Peak Current Density (i_p)	mA/cm²	760	800	850
D Negative Peak Current Density (i_n)	mA/cm²	650	700	750

Table 2
Total Number of Degrees of Freedom

Factor	Degrees of Freedom
Overall mean	1
A, B, C & D	4*(3–1) = 8
Total	**9**

Table 3
Surface Roughening & S/N Ratios of Response

Exp.	A T_{on}	B T_{off}	C i_p	D i_n	Surface i.e. (μm)	(db)
1	1	1	1	1	0.4	7.95880
2	1	2	2	2	0.4	7.95880
3	1	3	3	3	0.1	20.00000
4	2	1	2	3	0.3	10.45757
5	2	2	3	1	0.1	20.00000
6	2	3	1	2	0.3	10.45757
7	3	1	3	2	0.1	20.00000
8	3	2	1	3	0.5	6.02060
9	3	3	2	1	0.2	13.97940
Overall mean						**12.98142**

S/N Ratio for S.R. = $-10 \log_{10}$(mean square S.R.)

(iii) *Positive peak current density (i_p)*
The relationship between positive peak current density and surface finish at a constant T_{on} of 5 msec, a T_{off} of 5 sec and an i_n of 100 mA/cm² is shown in Fig. 4. The best surface finish was obtained at a positive peak current density of around 850 mA/cm². A relatively short pulse of 5 msec used in the experiment resulted in formation of a thin mass-transfer boundary layer that follows the surface contour. In this case, the peaks and the recesses of the surface profile of the mandrel were equally accessible for diffusion, so asperities were not amplified.[12] Because of this, an increase of i_p would tend to produce a better surface finish when i_p was less than 850 mA/cm², above which, the effect of reduction of hydrogen ions became more dominant and led to coarser grains.

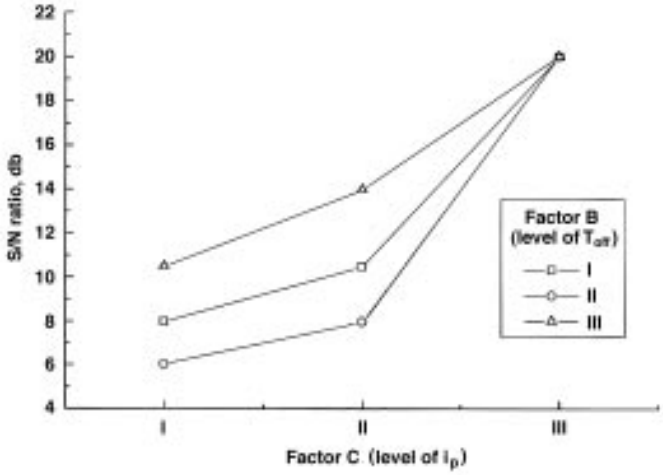

Fig. 7—Synergistic interaction between i_p and T_{off}.

(iv) *Negative peak current density (i_n)*
Figure 5 shows the effect of negative peak current density on the surface finish of nickel deposits at a constant T_{on} of 5 msec, a T_{off} of 5 msec and an i_p of 800 mA/cm². A minimum value of surface roughness was obtained at an i_n of 700 mA/cm². It is considered that when i_n is not too large, an increase in i_n favors the reduction of protrusion height. When i_n is larger than 700 mA/cm², desorption of $Ni(OH)_2$ crystals and/or H_2 molecules may occur and result in large grain size. Based on the results of the pilot experiment, three different levels for each factor are set for further experimental investigations and these are shown in Table 1.

Experimental Design Using Orthogonal Arrays
To construct an orthogonal array, the total number of degrees of freedom must be determined; this gives the minimum number of experiments that must be performed. As a rule of thumb, one degree of freedom is associated with the overall mean, regardless of the number of control factors to be studied. In general, the number of degrees of freedom associated with a controlling factor equals the selected number of levels minus one. Based on these, the total number of degrees of freedom was obtained and is shown in Table 2. The results show that at least 9 experiments must be conducted to

Table 4
Relative Significance of Each Controlling Factor

Factors	Units	Levels I	Levels II	Levels III	Degrees of freedom	Sum of square from factors	Mean square	F Significance
A Cathodic Time of Mandrel (T_{on})	msec	11.97253	13.63838	13.33333	2	4.71989	2.35994	0.96051
B Anodic Time of Mandrel (T_{off})	msec	12.80456	11.32647	14.81232	2	18.36614	9.18307	3.73757
C Positive Peak Current Density (i_p)	mA/cm²	8.14566	10.79859	20.00000	2	232.22939	116.11469	47.25940
D Negative Peak Current Density (i_n)	mA/cm²	13. 97940	12.80546	12.15939	2	5.10797	2.55399	1.03949
Error					0	0	—	
Total					8	260.42338	32.55292	
(ERROR)					4	9.82786	2.45696	

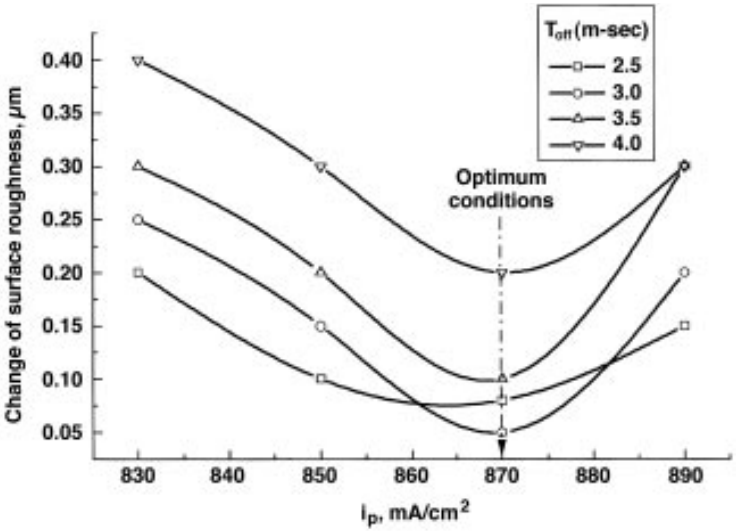

Fig. 8—Effect of i_p and T_{off} on surface roughness.

estimate the full effect of each factor, and a standard Taguchi orthogonal array[13] $L_9(3^4)$ was used in the experimental design.

Results of the Matrix Experiment

According to the Taguchi method,[14] the relative merits of each controlling factor can be evaluated using the signal-to-noise ratio (S/N ratio or -) which is given by

$$n = -10 \log_{10} \left[\frac{1}{n} \sum_{i}^{n} y_i^2 \right]$$

Where y_i represents the value of surface roughness at a controlling factor level *i*. Table 3 shows the results of the matrix experiment according to this equation. By averaging η of each controlling factor for different experiments, the S/N ratio for each factor was obtained and is given in Table 3. A more complete evaluation of the effect of the controlling factors on the surface finish can be obtained by performing the analysis of variance (ANOVA).[15] The results of the analysis of ANOVA are shown in Table 4 and are graphically presented in Fig. 6. The figure shows the relative significance of the various factors. It was found that the positive peak current density was the most significant factor in controlling the surface roughness of the deposit, whereas the anodic time of the mandrel was the second important factor.

Interaction between Positive Peak Current Density & Anodic Time

Before identifying the optimum levels for each factor from the ANOVA table, the interaction between positive peak current and anodic time, (the two most important controlling factors in the process), was analyzed. The interaction between i_p and T_{off} in terms of the S/N ratio is shown in Table 5. From Fig. 7, it can be seen that a certain degree of synergistic interaction exists. Although the curves were not parallel, their trends were similar and the direction of improvement was consistent. According to the Taguchi method, the optimum levels identified from the present analysis are valid and adequate.

Prediction of Optimum Level for Each Controlling Factor

Based on the Taguchi method, the optimum level of each controlling factor is represented by the level at which the factor has the largest mean S/N ratio. Therefore, with reference to Tables 1 and 4, the predicted optimum level for factor C was level III (850 mA/cm^2), which has a mean S/N ratio of 20 dB. Similarly, for factors A, B, and D, the optimum levels were level II (5 msec), level III (3 msec), and level I (650 mA/cm^2), respectively. The corresponding mean S/N ratios were 13.63838 dB, 14.81232 dB, and 13.9794 dB.

Final Experiment

Having predicted the optimum levels for each factor, a set of final experiments was performed to determine the optimum surface finish. Because the positive peak current density and the anodic time were found from the pilot experiment to be the two most critical factors in controlling surface finish, their effects on surface finish were more closely examined in a range very close to the predicted levels. In the final experiment, the settings used for each factor are shown in Table 6. Sixteen experiments were conducted and the results are summarized in Table 7 and Fig. 8. An optimum surface finish with a ΔR value of 0.05 m was obtained at a positive peak current density of 870 mA/cm^2, an *off* time of 3 msec, an *on* time of 5 msec and a negative peak current density of 650 mA/cm^2.

Table 5
Interaction between i_p and T_{off}

S/N (db)	C_1	C_2	C_3
B_1	7.95880	10.45757	20.00000
B_2	6.02060	7.9588	20.00000
B_3	10.45757	13.9794	20.00000

C_1, C_2 & C_3 represent i_p at level I, II & III, respectively.
B_1, B_2 & B_3 represent T_{off} at level I, II & III, respectively.

Table 6
Settings Used for Each Factor in Final Experiments

Factor	Settings
A Cathodic Time of Mandrel (T_{on})	5 m-sec
B Anodic Time of Mandrel (T_{off})	2.5, 3, 3.5 and 4 m-sec (the finest possible steps can be set within the optimum range)
C Positive Peak Current Density (i_p)	830, 850, 870 and 890 mA/cm^2 (the finest possible steps that can be set within the optimum range)
D Negative Peak Current Density (i_n)	650 mA/cm^2

Table 7
Result of Final Experiments

i_p	ΔR (μm) at	ΔR (μm) at	ΔR (μm) at	ΔR (μm) at
830	0.2	0.25	0.3	0.4
850	0.1	0.15	0.2	0.3
870	0.08	0.05	0.1	0.2
890	0.15	0.2	0.3	0.3

Summary

In this study, the Taguchi method was successfully applied to pulse reverse current electroforming to identify the dominant factors in controlling surface roughness. Moreover, the conditions to obtain an optimum surface finish of nickel electroforms have been established. The positive peak current density was found to be the most critical factor in controlling surface roughness in pulse reverse current electroforming. Better control of this parameter is therefore highly recommended for the process. Under the conditions of this study, the optimum surface finish obtained was 0.05 m, which occurred at a positive peak current density of 870 mA/cm^2, an *off* time of 3 sec, an *on* time of 5 msec and a negative peak current density of 650 mA/cm^2.

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