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Effects of process parameters on mechanical properties of abrasive-assisted electroformed nickel

Ren Jianhua, Zhu Zengwei *, Zhu Di

College of Mechanical and Electrical Engineering, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China

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Abstract A cathode mandrel with translational and rotational motion, which was supposed to obtain uniform friction effect on surface, was employed in abrasive-assisted electroforming for revolving parts with complex profile. The effects of current density, translational speed and rotational speed on the deposit properties were studied by orthogonal test. The tensile strength, elongation and micro hardness value were measured to find out how the factors affected the properties. The optimized results show that changes of current density affect the tensile strength of nickel layer most, while translational speed has the most remarkable influences on both elongation and micro hardness. The low rotational speed affects the properties least. In this experiment, a smooth nickel layer with tensile strength 581 MPa, elongation 17% and micro hardness 248HV is obtained by the orthogonal test.

1. Introduction

Metallic thin-wall parts of complex surface manufactured by electroforming are heavily required by modern industry, for electroforming is a precision manufacture technology or by far the most efficient way to produce these parts. By employing the principle of electrodeposition, electroforming can copy microscopic detail, reproduce accurate dimensions, and form components of controllable material property for desirable functions in applications such as precision mould, shaped charge liner, cryogenic upper stage main engine,[1–5] etc.

However, the applications of traditional electroforming process always go with some drawbacks at the electroformed layer, for example, pinholes and nodules on the surface, coarse grain size and long electroforming cycle. So far, a majority of researchers have been engaged in various kinds of additives, some of which could really reduce the grain size and enhance the strength of the deposits in electroforming process, and others could eliminate the pits and make the deposited layer surface smooth.[6,7] However, it is difficult to maintain the electrolyte baths because the additive agents are consumed during the electrodeposited process by decomposition and being absorbed on the cathode which leads to code position of sulfur and carbon.[8,9] Researchers have blamed the high-temperature

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* Corresponding author.
E-mail addresses: renjianrun@163.com (J. Ren), zhuzw@nuaa.edu.cn (Z. Zhu), dzhu@nuaa.edu.cn (D. Zhu).

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ductility losses in nickel on sulfur and carbon that are supposed to give rise to the embrittlement of deposits.[10]

Studies made by researchers have shown that abrasive-assisted nickel electroforming process could effectively eliminate pinholes, remove nodules, positively affect crystal nucleation, and refine the grains of layer, and thus near-mirror electroformed layer was obtained without any organic additives.[11,12]

Generally speaking, there are two independent cathode motions in the abrasive-assisted electroforming process to obtain friction effect on cathode surface. A pure rotational motion was designed for revolving parts electroforming, and a designed translational cathode in horizontal type was employed in complex shaped non-rotating parts electroforming. However, for revolving parts with complex surface in abrasive-assisted nickel electroforming process, neither of the two single cathode motions would be carried out. Firstly, there are different linear velocities along the axial direction of cathode because of diverse curvature radius in pure rotational motion. Secondly, the varying electric field intensity on cathode surface for pure translational motion should be taken into consideration because it will lead to the nonuniformity in thickness of deposits. Naturally, the friction effect would also be inhomogeneous. To solve the problems mentioned above, a horizontally positioned cathode with complicated movement in combination of translation and rotation is proposed in this paper. The complex effects of current density, translational speed and rotational speed on the deposit microstructure and properties were studied by orthogonal test. The tensile strength, elongation and micro hardness values were also measured.

2. Principle and experiments

Fig. 1 shows the scheme of experimental principle of abrasive-assisted electroforming process with moving cathode. $\boldsymbol{n}_1$ and $\boldsymbol{n}_2$ are the rotation directions of ceramic beads. V is translational speed. V' is rotational speed. A cylinder mandrel is translated as well as rotated in horizontal type. In the nickel electroforming process, nonconductive hydrogen bubbles usually adhere to cathode surface impeding nickel ion deposition. The free ceramic beads filling in the space between cathode and anode were forced to polish the growing deposited layer slightly and uniformly, driving the hydrogen bubbles away, during the electroforming process.[13,14] The rotation of cathode was set at low speed, while the translational speed was set much higher and played the key role in driving the beads. The orbital movement of cathode mandrel was a circle. The reasons are presented below.[11,12] On one hand, different parts of the cathode have the same linear velocity during the cathode's translation to make sure the polishing effect on the whole surface in uniformity. On the other hand, the rotation of cathode could maintain the cathode surface in the same electric field intensity, and achieve uniform polishing effect on circumference surface.

Fig. 2 illustrates the schematic diagram of the experimental apparatus. The cathode's translational movement was carried out by a planar worktable driven by stepper motor in X/Y axis linkage. And a speed control motor was employed to drive the rotation of the cathode via transmission mechanism. Nickel pellets were used as anode. A stainless steel cylinder mandrel was used as cathode in horizontal type whose deposit area was $\varnothing 70$ mm × 100 mm. Free ceramic beads in 0.8–1.2 mm diameter were chosen as the abrasive medium filling the space between the electrodes to maintain continuous friction on the cathode's surface in the process of electroforming. The electrolyte was pumped from the storage bath to the electroforming unit and flowed through the gap between cathode and anode. Both the electrolyte flushing and the moving cathode motion served as the agitation of the electrolyte. The electrolyte's temperature was controlled by a heater and temperature controller. Before deposition, the cathode was

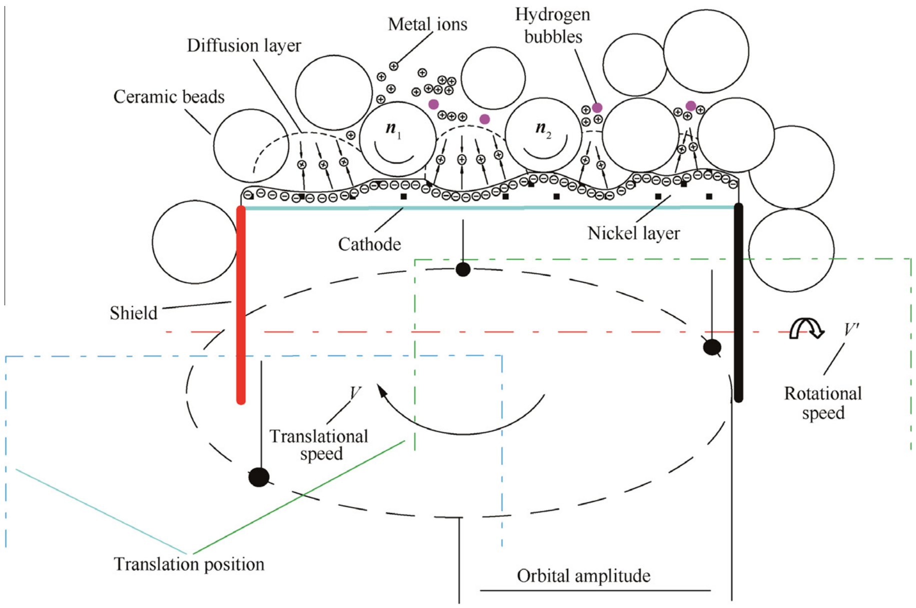


Fig. 1 Scheme of experimental principle of abrasive-assisted electroforming process with moving cathode.

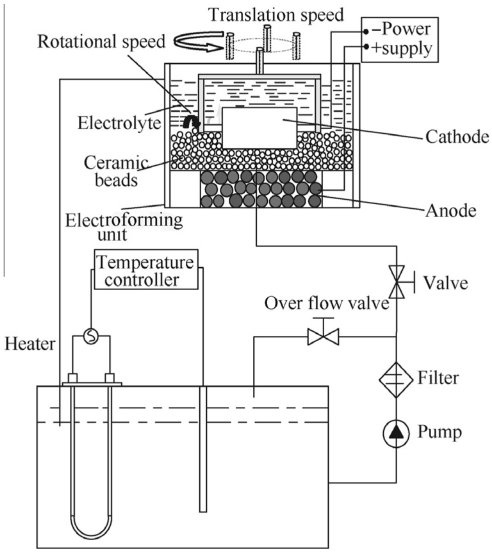


Fig. 2 Schematic diagram of experimental apparatus.

mechanically polished, degreased with organic solvent, and rinsed with ethanol.

An orthogonal method was used to study the effects of the three factors: current density (*A*), translational speed (*B*) and rotational speed (*C*) on the mechanical properties of nickel layers, and each factor had three levels. Thus, the variable factors and levels formed an orthogonal array (Tables 1 and 2). A L9 orthogonal array was selected for optimization of the process parameters.[15,16]

All solutions were prepared by deionized water and analytical reagents were used. No additive was used. The solution content and the experiment condition are shown in Table 3. At the end of the process, the cathode was immediately withdrawn, rinsed with deionized water, dried and then detected.

A HITACHI S3400N SEM was used to observe the surface morphology of the samples. A HXS-1000A Vickers micro hardness tester was used to measure the micro hardness of the nickel deposits at room temperature. A load of 0.49 N was applied and kept for 15 s. The final value quoted for the hardness of each deposit is the average of six times. A Instron 5566 tensile strength tester was used to measure the strength property, the thickness of the samples was kept at about 0.7 mm by controlling the current density and deposition time, and the tensile rate was kept at 0.2 mm/min.

3. Results and discussions

As Fig. 3(a) shows, there are distinct groove marks on the surface of nickel deposit with purely rotating cathode in a very long electroforming cycle, while Fig. 3(b) shows smooth surface of nickel deposit with complicated moving cathode. The linear speed in Fig. 3(a) is a little higher than that in Fig. 3(b), so the surface in Fig. 3(a) is brighter than Fig. 3 (b).[17,18] There is no doubt that the friction effect of ceramic beads with ordinary rotating cathode is too strong to obtain smooth-surfaced deposit layer when the cathode linear speed is at about 20–100 mm/s. The marks on the surface are just the same as the purely rotational movement of cathode. However, when a complicated moving cathode was employed, the ceramic beads were forced to move in a complicated way, achieving uniform polishing effect on circumference surface.

Table 1 Orthogonal factors and levels.

Factor	Level		
	Level 1	Level 2	Level 3
A: Current density (A/dm^2)	2	4	6
B: Translational speed (mm/s)	10	20	40
C: Rotational speed (r/min)	0.5	1	2

Table 2 L9 orthogonal array.

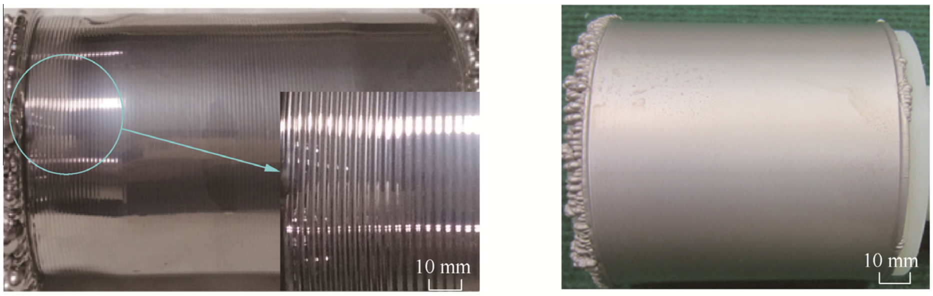
Serial No.	*A* (A/dm^2)	*B* (mm/s)	*C* (r/min)
1	2	10	0.5
2	2	20	1
3	2	40	2
4	4	10	1
5	4	20	2
6	4	40	0.5
7	6	10	2
8	6	20	0.5
9	6	40	1

Table 3 Bath composition and process conditions of nickel electroforming.

Composition and process condition	Quantity
$Ni(NH_2{\cdot}SO_3)_2{\cdot}6H_2O$	400 g/L
H_3BO_3	30 g/L
$NiCl_2$	15 g/L
PH	4.0–4.5
Temperature	(43 ± 2) °C

Then the results of orthogonal test are shown in Table 4, among which I, II, III show the average value of each factor under different levels respectively. The range value in Table 4 shows the degree of the effect of factors on mechanical properties; the maximum value is written in bold; the larger the value is, the more significant the effect is. Table 4 shows the tensile strength, elongation and micro hardness values of each orthogonal array group. As can be seen from Table 4, the factors influencing tensile strength arranged by the order from major to minor are current density, translational speed, and rotational speed, namely *A*, *B*, and *C*. In addition, the influence of current density is the most significant, and translational speed is the second. As to influence on elongation, translational speed is the most significant factor, current density is the second, and rotational speed is the last, namely *B*, *A*, and *C*. There is no significant difference between the first two factors. The factors with their influence on micro hardness listed from the most to the least important are translational speed, current density, and rotational speed, namely *B*, *A*, and *C*, and also the significance levels of first two factors are nearly equal.

The effects of current density, translational speed and rotational speed on the properties of nickel deposits are discussed as follows.



(a) Rotational cathode with groove mark (b) Complicated moving cathode

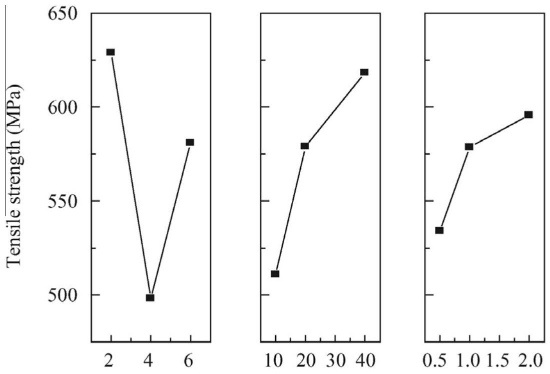
Fig. 3 Abrasive-assisted nickel electroforming process when deposit grows thick at current density of 3 A/dm^2.

Table 4 Tensile strength, elongation and micro hardness of electroformed nickel orthogonal array.

Serial No.	*A*	*B*	*C*	Tensile strength (MPa)	Elongation (%)	Micro hardness (HV)
1	1	1	1	530	13.7	227.4
2	1	2	2	675	12.1	245.1
3	1	3	3	682	7.7	252
4	2	1	2	427	22.2	197.1
5	2	2	3	529	14.85	218.2
6	2	3	1	539	13.75	225.2
7	3	1	3	576	16	204.7
8	3	2	1	533	16.5	232.3
9	3	3	2	634	12.2	237.2
I/3	629	511	534	Tensile strength		
II/3	498.3	579	578.7			
III/3	581	618.3	595.7			
Range	**130.7**	107.3	61.7			
I/3	11.2	17.3	14.7	Elongation		
II/3	16.9	14.5	15.5			
III/3	14.9	11.2	12.9			
Range	5.7	**6.1**	2.6			
I/3	241.5	209.7	228.3	Micro hardness		
II/3	213.5	231.9	226.5			
III/3	224.7	238.1	225.0			
Range	28	**28.4**	3.3			

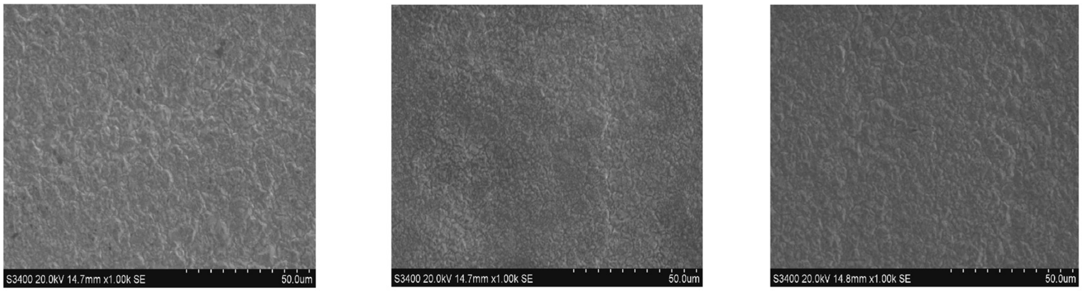
3.1. Effects of process parameters on tensile strength of nickel deposits

Fig. 4 shows the effect of each factor on tensile strength of electroformed nickel with moving cathode that is obtained from the results of Table 4. As Fig. 4(a) presents, tensile strength value of nickel electroformed decreases at first, and then rises when current density increases from 2 A/dm^2 to 6 A/dm^2. There is a big fluctuation in the tensile strength values with the increase of current density. At the same time, there is a quick increase of tensile strength value with the increase of translational speed, as plotted in Fig. 4(b). In addition, there is a positive correlation between tensile strength and rotational speed in Fig. 4(c). It seems that current density is the most significant factor influencing the variation of tensile strength, either increase or decrease. However, the translational speed might be another significantly positive factor to increase the tensile strength. As shown in Fig. 5, when the moving speed of cathode increases, the abrasion strength becomes more distinguishable and grains grow smaller where current density stays the same. When the cathode speed increases, the polishing can be strengthened and help to obtain smooth nickel layers with fine mechanical properties by increasing the activated points on cathode and refine the grain size. Thus, the tensile



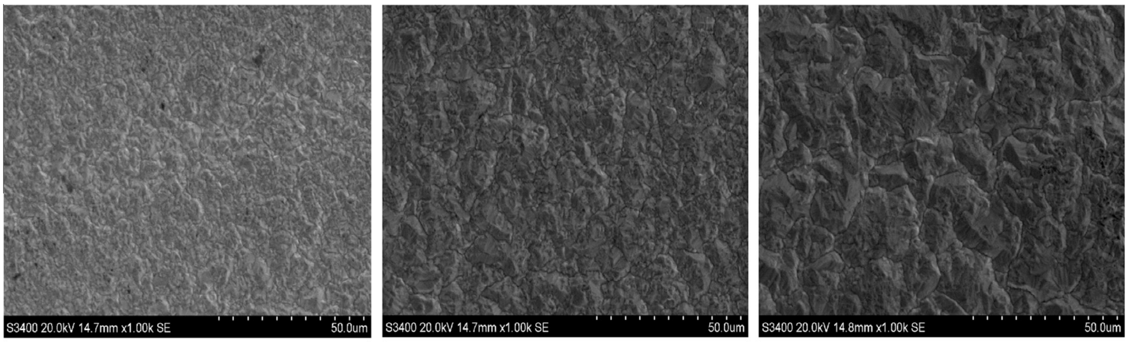
(a) Current density (A/dm^2) (b) Translational speed (mm/s) (c) Rotational speed (r/min)

Fig. 4 Effect of each factor on tensile strength of electroformed nickel.



(a) NO.1[111]-(2 A/dm², 10 mm/s, 0.5 r/min) (b) NO.2[122]-(2 A/dm², 20 mm/s, 1 r/min) (c) NO.7[133]-(2 A/dm², 40 mm/s, 2 r/min)

Fig. 5 Effect of moving speed on SEM morphology of electroformed nickel with increasing translational speed and rotational speed at the same current density (No. 1,2,3 from orthogonal array of Table 2).



(a) NO.1[111]-(2 A/dm², 10 mm/s, 0.5 r/min) (b) NO.4[212]-(4 A/dm², 10 mm/s, 1 r/min) (c) NO.7[313]-(6 A/dm², 10 mm/s, 2 r/min)

Fig. 6 Effect of current density on SEM morphology of electroformed nickel with the same translational speed but increasing rotational speed (No. 1,4,7 from orthogonal array of Table 2).

strength increases with increasing translational speed as well as rotational speed because there is a negative correlation between tensile strength and grain size. The rotational speed is so low that it can be supposed to have uniform abrasion effect on revolving parts with complicated profiles and maintain the circumference surface in the same electric field intensity.

As shown from Fig. 6, higher current density may lead to coarser crystal grains and loose structure. When current density increases, the abrasion effect is not really obvious and the grains grow bigger. So the SEM images are the evidence that low tensile strength can be sourced in coarse crystal grains and loose structure. Tensile strength of 4 A/dm^2 is lower than that of 6 A/dm^2 in Fig. 4(a) maybe because the moving speed at 6 A/dm^2 is higher than that at 4 A/dm^2. The tensile strength is strengthened by the free beads' polishing effect. The influence of current density on property of electroformed nickel is remarkable. Hence, for obtaining nickel layer with proper property, the current density requires tight control for slight change in it will cause much change in tensile strength.

3.2. Effects of process parameters on elongation of nickel deposits

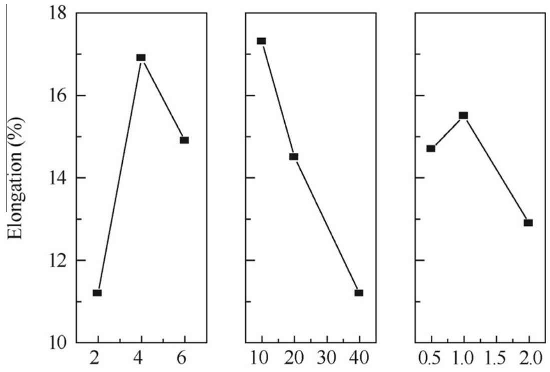
Fig. 7 shows the effect of each factor on elongation of electroformed nickel with moving cathode that is obtained from the results of Table 4. As plotted in Fig. 7(a), the elongation of electroformed nickel first goes up, and then goes down with current density increasing from 2 A/dm^2 to 6 A/dm^2, just contrary to the influence on tensile strength in Fig. 4(a). As shown in Fig. 6, higher current density may lead to coarser crystal grains and loose structure. When tensile strength goes low, the elongation will increase. Data on the effect of current density show that a 4 A/dm^2 current density should be used for good elongation and high tensile strength.

When translational speed increases, the dominant trend of effect on deposits is a quick decrease of the elongation in Fig. 7(b). The influence of translational speed on elongation of electroformed nickel is remarkable. As shown in Figs. 4 and 5, faster translational speed may lead to higher tensile strength, and the corresponding elongation gets lower. Hence, for obtaining nickel layer with proper property, the translational speed requires tight control because slight change in it will cause much change in elongation.

In Fig. 7(c), there is a small positive and then a small negative correlation between elongation and rotational speed. Data on the effect of rotational speed are so small that the rotational speed has really loose correlation with elongation. Well, the rotational speed at low range is supposed to have uniform abrasion effect. The rotational speed should be controlled low enough to get higher elongation.

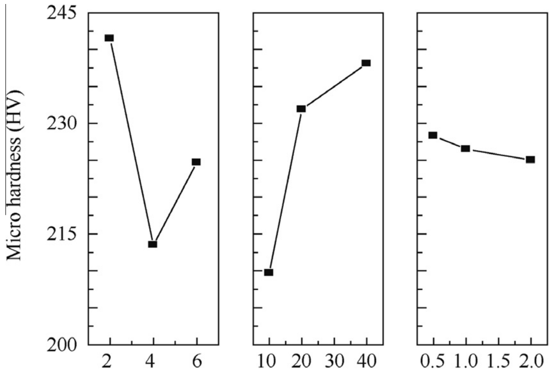
3.3. Effects of process parameters on micro hardness of nickel deposits

As Fig. 8(a) presents, micro hardness values of electroformed nickel decrease at first, and then rise when current density increases from 2 A/dm^2 to 6 A/dm^2. But there is a downward



(a) Current density (A/dm²) (b) Translational speed (mm/s) (c) Rotational speed (r/min)

Fig. 7 Effect of each factor on elongation of electroformed nickel.



(a) Current density (A/dm²) (b) Translational speed (mm/s) (c) Rotational speed (r/min)

Fig. 8 Effect of each factor on micro hardness of electroformed nickel.

trend overall. In Fig. 8(b), the dominant trend is a quick increase of micro hardness value of electroformed nickel with the increase of translational speed. Generally, hardness has the same effect tendency as tensile strength does. And there is a negative correlation between tensile strength and grain size. So the higher hardness might be attributed to the compact and fine-grained deposit structure. There is a small negative correlation between micro hardness and rotational speed in Fig. 8(c). The effect of rotational speed is non-significant and negligible.

3.4. Process parameters optimization procedure

The optimization analysis process by overall equilibrium of the properties[19,20] is shown as follows.

As Table 4, Figs. 3, 6 and 7 show, translational speed has the most remarkable influences both on elongation and micro hardness. Level 20 mm/s is good in factor *B* in view of relatively high elongation value. To tensile strength, the influence of translational speed is not the largest, so level 20 mm/s is also the best.

Rotational speed is not a remarkable influence to any of the three properties. As shown in Figs. 3, 6 and 7, level 1 r/min is good in factor *C* in view of tensile strength and elongation. Furthermore, level 1 r/min in factor *C* is not the lowest to micro hardness, so level 1 r/min is the best in factor *C*.

Current density is the most remarkable influence factor to tensile strength as shown in Table 4. Fig. 3(a) shows that level 2 A/dm^2 is the best in factor *A*, and 6 A/dm^2 is the second. But level 6 A/dm^2 is better to elongation and it is not the lowest to micro hardness. Thus, level 6 A/dm^2 is the best in factor *A*.

4. Conclusions

(1) An orthogonal test of abrasive-assisted nickel electroforming process was carried out with moving cathode in combination of translation and rotation. The influences of current density, translational speed and rotational speed on the microstructure and properties of deposits were studied.

(2) In order to realize high-speed nickel electroforming by using high current density, faster translational speed should be applied to obtain higher mechanical properties, such as tensile strength and micro hardness.

(3) To achieve uniform friction effect on deposits for the abrasive-assisted electroforming of revolving parts with complex surface profile, both translational and rotational motion are necessary. For most of the friction effects were attributed to translational cathode, the rotation could achieve uniform polishing effect at circumferential direction of cathode surface and maintain the surface in the same electric field intensity.

(4) Changes of current density affected the tensile strength of nickel layer most, while translational speed had the most remarkable influences both on elongation and micro hardness. Slow rotational speed had the smallest influence on properties of deposits. In this experiment, best factors with $A_3B_2C_2$ (6 A/dm^2, 20 mm/s, 1 r/min) were obtained by using the optimized analysis: overall equilibrium of the properties. A smooth nickel layer with comprehensive properties of tensile strength 581 MPa, elongation 17% and micro hardness 248HV was obtained.

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REN Jianhua is currently a Ph.D. candidate at College of Mechanical and Electrical Engineering, Nanjing University of Aeronautics and Astronautics. His research interests include electrochemical machining, electroforming, and micro-electrochemical machining.

ZHU Zengwei is currently a professor in Nanjing University of Aeronautics and Astronautics. His research interests include electrochemical machining, electroforming, and micro-electrochemical machining.

ZHU Di is currently a professor in Nanjing University of Aeronautics and Astronautics. His research interests include electrochemical machining, electroforming, and micro-electrochemical machining.