Effect of auxiliary friction on nickel electroforming deposits

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Abstract. The nickel deposits were obtained with friction assisted, and the porosity, microstructure, texture and tensile properties of nickel deposits under different parameters were studied. The experiment shows that when the cathode velocity reaches a certain value, non-porous nickel deposits can be obtained under the friction of the microbeads. In addition, the microstructure characteristics of nickel deposits under different parameters were tested and analyzed, and the mechanical properties of nickel deposits before and after annealing were tested.

1. Introduction

Now nickel electroforming has been employed in fabricating precision dies, rocket engine thrust chamber, shaped charge liner, and other special structure parts[1-4].The microstructure and mechanical properties of electroformed nickel are related to electroforming process. In the nickel electroforming, sulfamate electroforming solution is usually used because a higher current density can be used, and the internal stress of the nickel electroforming layer is smaller. However, there are some drawbacks in the traditional electroforming process, such as pinholes, pits and nodules. As deposits grow progressively thicker, the defects on the surface will become worse. The researchers used additives in the solution, pulse power, vibration, ultrasound and other ways to improve the electrodeposition process. But these methods can not completely solve the problem [5-7].

In this paper, electroforming with abrasion-assisted have been applied to the nickel electroforming process to overcome the drawbacks. The nickel deposits were electroformed with attrition assisted and their structures and mechanical properties were examined.

2. Experimental

Figure 1 illustrates the schematic diagram of the experimental apparatus. The system consists of power supply, cathode unit, anode basket, heater with temperature control equipment and an electrolyte circulating system. The electrolyte contained $Ni(NH_2SO_3)_2 \cdot 6H_2O$ 400g/l, H_3BO_3 30g/l, $NiCl_2$ 15g/l. Nickel deposition was controlled at pH=4 in the electrolyte.

All solutions were prepared by freshly distilled water. No sulfur-containing Nickel beads were used as the anode. A stainless cylinder mandrel was used as the cathode, and the deposit area was Ø48 mm×80 mm. Ceramic beads with diameter of Ø1-2mm were chosen as the hard particles.

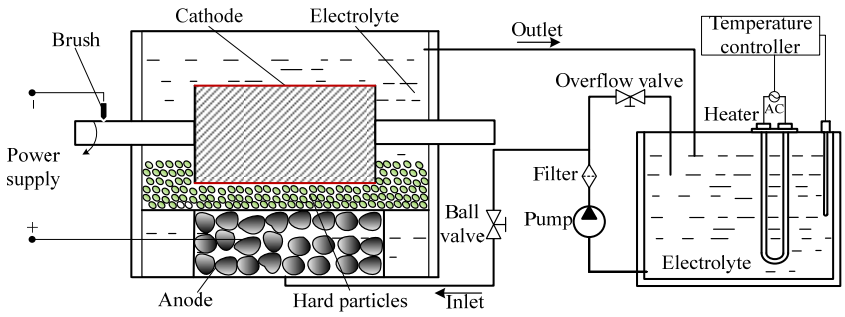

Figure 1. Schematic diagram of the experimental apparatus.

The cathode mandrel was connected to a motor, the speed of the cathode was varied from 0.0025 to 0.08m/s at a fixed current density of 4 A/dm^2 and a fixed temperature of 43℃.

Porosity: the porosity of electroformed nickel is measured by filter paper. Formula: potassium ferricyanide 1g/l, sodium chloride 10g/l, agar 10g/l, T=82~94℃, time 10min. The wetting test solution against filter paper on the sample surface, and after the required time, observe the number of colored spots. In about 30μm thick electroformed layer on the detection, each sample three times, respectively, according to the following formula to calculate the porosity, whichever is the average.

$$\text{Porosity} = \text{pore number (n)} / \text{measured area (s)} \tag{1}$$

When the spot diameter is less than 1mm, a spot is calculated by one; when the spot diameter is 1 ~ 3mm, a spot is calculated by three, when the pore diameter is 3～5mm, a spot is calculated by ten.

The structure of the samples was measured by a BRUKER D8 ADVANCE X-ray diffraction (XRD) using Cu Kα radiation. Preferential orientation of the samples was characterized by the orientation index (M) for each plane (hkl), and the formula is shown in Formula (2).

$$TC_{(hkl)} = \frac{I_{(hkl)}/I_{0(hkl)}}{\sum_{i=1}^{n} I_{(hkl)}/I_{0(hkl)}} \times 100\% \tag{2}$$

I (hkl) represents the X-ray diffraction relative intensity of the crystal plane of the deposited, I0(hkl) represents the X-ray diffraction relative intensity of the standard powder(hkl) crystal plane, and n is the number of diffraction peaks.

3. Results and discussions

3.1. Porosity

In the traditional electroforming technology of electroforming, the surface of nickel deposit is dark gray and covered with pitting. The friction assisted electroforming can solve the problem of pinhole formation in nickel deposit, and can adopt high current density in electroforming process. Figure 2 is the graph of the number of porosities at different velocities of cathode, while the current density of cathode is 4A/dm^2. The surface of the nickel deposits is smooth. It can be seen from the curve that the porosities decrease with the increase of velocity of cathode. When the velocity is above 0.0025m/s, the porosity of the nickel deposits is zero.

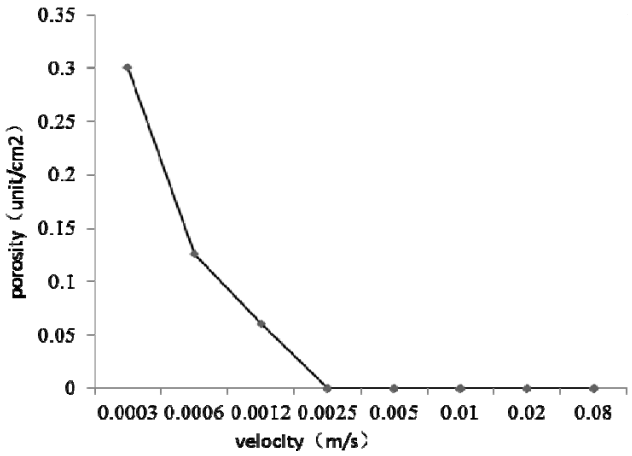

Figure 2. Porosity quantity of nickel deposits under different velocities

3.2. TEM

Figure 3 shows the TEM micrograph of four nickel deposits. As can be seen from the graph, the grain size of nickel deposits is gradually decreased with the increasing of linear velocity of cathode. As shown in Figure 3, when the linear velocity of cathode is 0.0025m/s, the grain size of nickel deposit is coarse, and when the linear velocity of cathode increased to 0.08m/s, the grain size of nickel deposit is significantly decreased. The above results also indicate that hard particles have a role in grain refinement. When speeding up the velocity of cathode, the polishing effect of hard particles can be strengthened.

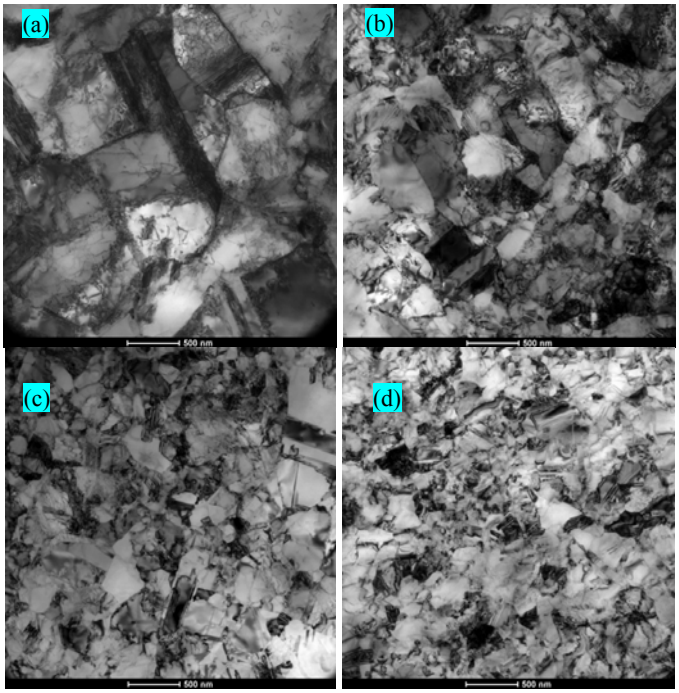

Figure 3. TEM morphology of nickel deposits under different velocities
(a)0.0025m/s; (b)0.01m/s; (c)0.02m/s; (d)0.08m/s

3.3. Texture coefficient

According to the formula (2), the texture coefficient of the nickel deposit prepared by the traditional electroforming technique is shown in Table 1.

Table 1. The TC (hkl) of nickel deposit prepared by traditional electroforming technology

crystal surface	(111)	(200)	(220)
$TC_{(hkl)}$ (%)	4.79	89.21	6

According to the formula (2), the change trend of texture coefficient of the nickel deposits is obtained at different cathode velocities by using attrition assisted electroforming is shown in Figure 4.

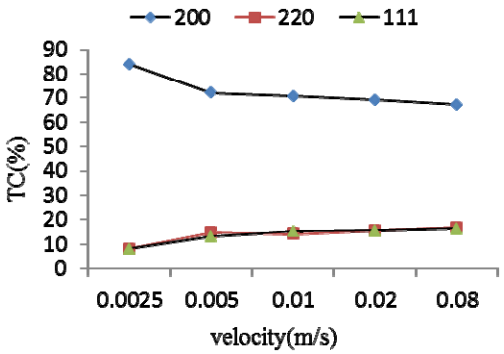

Figure 4. Texture coefficient of nickel deposits with cathode velocities

When using attrition assisted electroforming technology, the change of cathode velocity will affect the texture coefficient of nickel deposit. As shown in Figure 4, with the increase of the cathode velocity, the texture coefficient of (200) crystal surface decreases, while the texture coefficients of (111) and (220) crystal surface are increasing. The above results show that the attrition of the surface layer during the electroforming process can affect the growth of the crystal, and the relative growth rate of each surface is reduced.

3.4. Mechanical property

Electroforming products in some occasions often need to use after annealing, so it is necessary to test and analyze the performance of nickel deposit after annealing. The annealing process used in this paper is as follows: annealing in high temperature vacuum furnace, the temperature rises to 280℃ in 10 minutes, then keep 2 hours at constant temperature, and finally removed after the furnace cooling.

It can be seen from Figure 5 and Figure 6 that the tensile strength of the electroformed nickel increases with the increase of the cathode velocity and the elongation decreases gradually. The tensile strength and the elongation of nickel before and after annealing are the same, but the tensile strength of nickel after annealing has a certain degree of reduction, while the elongation is increased. When the velocity is 0.0025m/s, the tensile strength of nickel decreases from 525MPa before annealing to 452MPa after annealing, and the elongation increases from 24% to 36%, and When the velocity is 0.08m/s, the tensile strength of nickel decreases from 1020MPa before annealing to 841MPa after annealing, and the elongation increases from 4% to 10%.

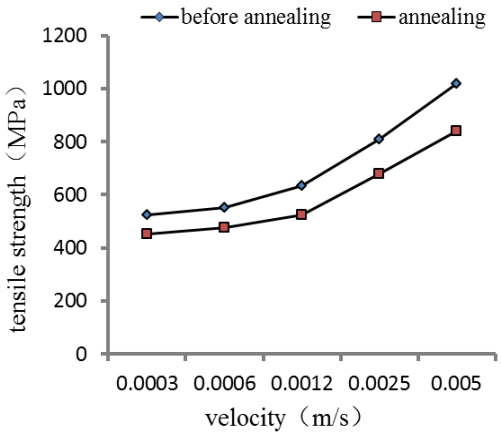

Figure 5. Effect of velocity on tensile strength of electroformed nickel

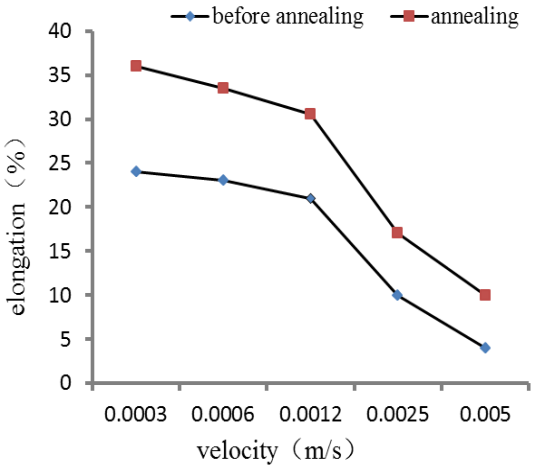

Figure 6. Effect of velocity on elongation of electroformed nickel

4. Conclusion

The nickel deposits were electroformed with attrition assisted and their microstructure were examined. The experimental results show that the attrition of particles could obviously have a role in grain refinement, and when speeding up the velocity of cathode, the grain refinement is enhanced.

It was found that the attrition of hard particles affect the growth of crystallites and their relative speed of growth, which leads to the change of surface diffraction density and orientation index. And it was also found that when the velocity of the cathode was increased, the tensile strength of the electroformed nickel was increased and the elongation was decreased.

Acknowledgments

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