Int. J. Electrochem. Sci., 7 **(2012)** 10818 - 10824

International Journal of
ELECTROCHEMICAL
SCIENCE
www.electrochemsci.org

Short Communication

Jet Electrodeposition of Bulk Nanocrystalline Nickel with Real-Time Polishing

Gui-Feng Wang[1,*], *Li-Da Shen*[2], *Yin-hui Huang*[2]

[1] State Key Laboratory of Coal Resources and Safe Mining, China University of Mining and Technology, No. 1 University Road, Xuzhou, Jiangsu 221116, PR China.
[2] College of Mechanical and Electrical Engineering, Nanjing University of Aeronautics and Astronautics, No. 29 Yudao Street, Nanjing, Jiangsu 210016, PR China.
[*]E-mail: wgfskl@163.com

Received: 29 September 2012 / *Accepted:* 17 October 2012 / *Published:* 1 November 2012

The synthesis of bulk nanocrystalline nickel by a novel polishing jet electrodeposition (PJED) technology with real-time polishing for deposit surface was studied. The morphology, surface roughness, microstructure and microhardness of deposits produced by PJED without any organic additives were examined compared with traditional JED. The results indicated that PJED could significantly smoothen the deposit surface by removing pits and nodules with real-time polishing of free particles. The structure of nanocrystalline deposits was more uniform with refined grains and the microhardness was significantly improved by PJED. A nickel thin-walled part with a complex profile was experimentally produced by PJED which had mirror-like surface and excellent hardness uniformity.

Keywords: jet electrodeposition; nanocrystalline nickel; microhardness

1. INTRODUCTION

Comparing with the conventional electrodeposition, jet electrodeposition (JED) provides the advantages of selective and high-speed plating, and the grain refining effect is more efficient since a much higher overpotential of cathodic substrate can simultaneously be used with much higher current density [1-2]. However, until now, it is very difficult for JED to produce high-quality and large bulk of nanocrystalline. This is because, the high current density and electrolyte flow rate of JED can severely promote outward growth of adatom clusters perpendicular to the surface and tend to generate large amount of nodules or dendrites, causing deposit surface always irregular during JED, and eventually yielding a nonuniform deposit [3-6]. Furthermore, with the large current of JED, many additives tend to be largely consumed and codeposit with adatom clusters, which may reduce the Levelling power of additives, evenly affect the structure and decrease some properties of deposits [7-8].

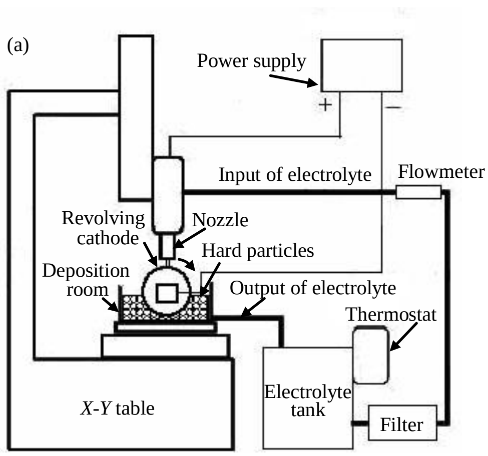
In this paper, a novel polishing jet electrodeposition (PJED) technology is developed to overcome the above-mentioned drawbacks of JED and produce bulk nanocrystalline nickel deposits by introducing real-time polishing of dynamical free particles to the process of electrodeposition without any additives using direct current. The structure and properties of the deposits were investigated.

2. EXPERIMENTAL

Experimental system. Fig. 1 shows a schematic diagram of the PJED system and electrodeposition room. A revolving cathode was half buried in a heap of isolating and insoluble little free particles, and rotated under the control of CNC unit, thus the particles were forced to move around and slightly polish the deposit surface continuously during PJED. The electrolyte was injected locally onto the cathode surface from the nozzle which moved along the cathode profile in accordance with the CNC commands, and was continuously recirculated to electrolyte tank. Further details similar to the geometry of traditional JED system can be found elsewhere [9-12].

Deposition experiments. The experiments were carried out in the solution containing $Ni_2SO_4 \cdot 6H_2O$ 280 g/L, $NiCl_2$ 40 g/L, H_3BO_3 38 g/L, pH 4–5, T= 40 ± 1 ºC. No organic additives were used. Graphite sticks with revolving and complex profile were used as cathodes. Spherical ceramic beads with diameter of Ø 1–2 mm were employed as the free particles. Nickel deposit samples were prepared in the cathode rotational speed range 2–10 r/min and current density range 40–120 A/dm^2.

Deposit analysis. The surface morphology of deposits was characterized by Hiroxkx-7700 optical microscopy, and the surface roughness was measured by Mahr Perthometer M1 roughness tester. A JEM-2000EX transmission electron microscope (TEM) was used to characterize the microstructure and grain size of the samples at an operating voltage of 160 kV. The microhardness of samples was measured with a HXS-1000A Vickers microhardness measuring device.



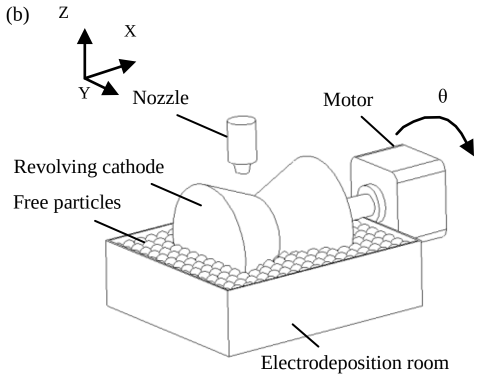


Figure. 1 Schematic diagram of (a) PJED system and (b) electrodeposition room

3. RESULTS AND DISCUSSION

Fig. 2 shows the surface morphologies of nickel deposits produced by JED at different times. As shown in Fig. 2a, b and c, surface protrusions develop more faster and larger as deposition time prolonging, and merge with the neighboring ones leaving some holes inside, and eventually reducing the uniformity and compactness of deposits produced by JED [13-15].

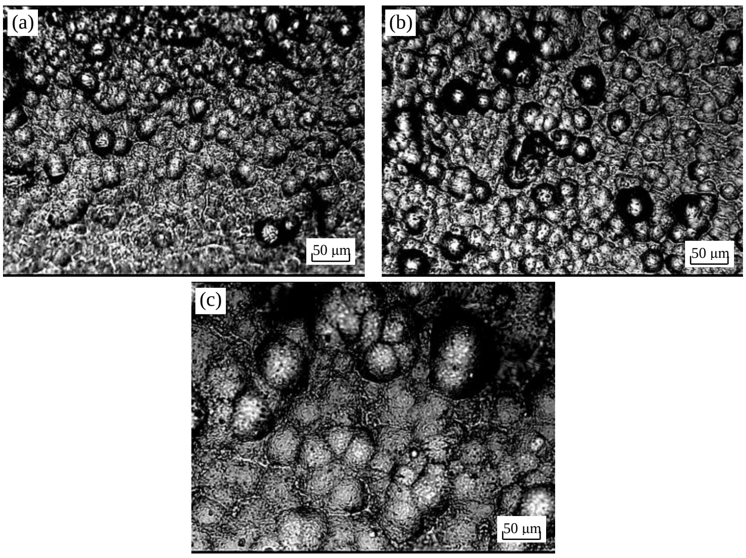


Figure 2. Surface morphologies of nickel deposits electrodeposited by JED at different times: (a) 40 min, (b) 80 min, (c) 120min.

Due to the polishing of free particles, a refinement of the surface morphologies appeared as shown in Fig. 3. The existence of marks on the deposit surface proves the polishing effect of free particles during PJED. The polishing became more important especially in the late stages of deposition, in which large nodules with polished tops began to emerge (see Fig. 3b and c).

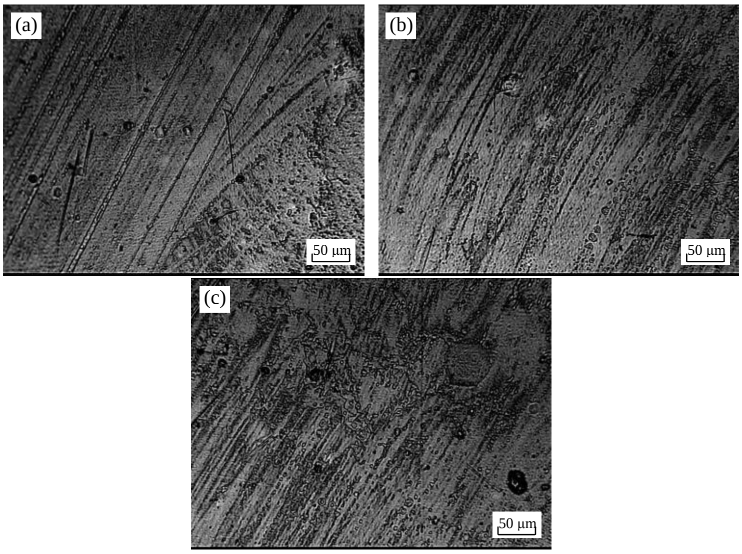


Figure 3. Surface morphologies of nickel deposits electrodeposited by PJED at different times: (a) 40 min, (b) 80 min, (c) 120min.

Fig. 4 shows the variation of surface roughness of deposits with cathode rotational speed and current density. As it is expected, the surface roughness has relation with the polishing effect of free particles, and the effect decreases with current density and increases with cathode rotational speed, which leads to the variation of surface roughness as shown in the fig. 4a and b.

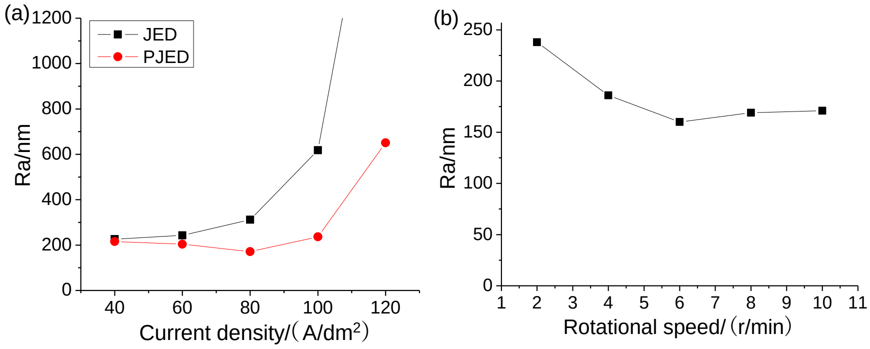


Figure. 4 Variation of surface roughness of deposits with (a) cathode rotational speed and (b) current density.

Fig. 5 shows the photos of typical deposits produced by JED and PJED. The deposits became smooth and mirror-like without pits or nodules by the use of PJED. Fig. 6 shows the TEM micrograph of the deposits. The result suggests that nanocrystalline deposits with very fine-grained and uniform structure can be obtained by PJED. This is probably because the polishing of free particles may produce more active sites increasing the number of nucleation, polish the tops of crystallites and slow their growth [16-18].

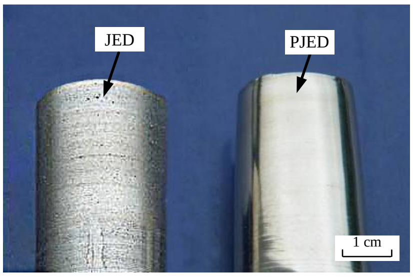


Figure 5. Photos of nickel deposits produced by JED and PJED

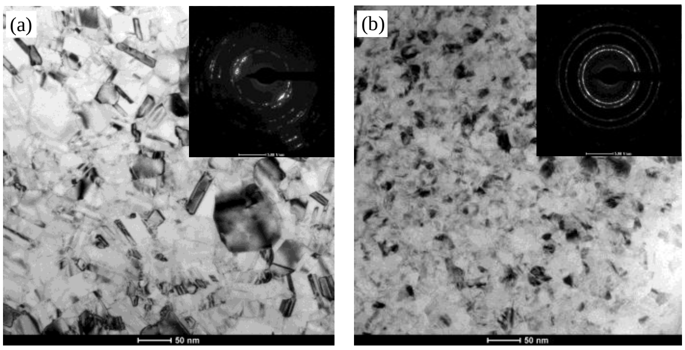


Figure 6. TEM micrographs of nickel deposits produced by (a) JED and (b) PJED.

The test results of microhardness are shown in Fig. 7. It suggests that the polishing of free particles can significantly improve the hardness of deposits (see Fig. 7a). The highest value is about 650 HV, which can be obtained in a competitive balance between the polishing effect of particles and the growth of surface protrusions. The variation of hardness, surface roughness and abrasion mark provide strong evidence for beneficial polishing effect of free particles.

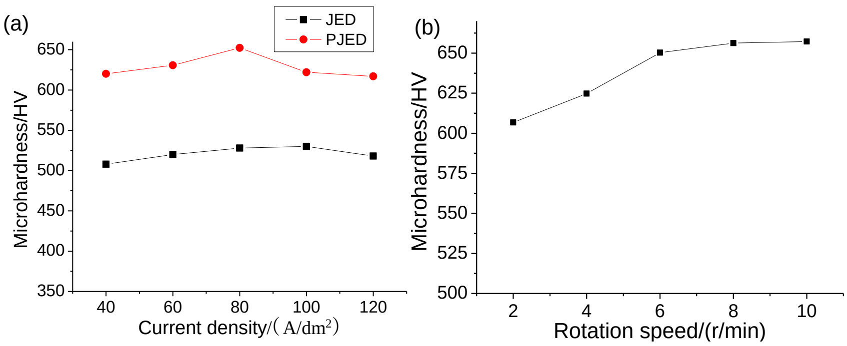


Figure 7. Variation of microhardness of deposits with (a) cathode rotational speed and (b) current density.

Fig. 8 shows the photo of a typical thin-wall revolving part with complex profile produced by PJED at current density of 120 A/dm^{2}and cathode rotational speed of 10 r/min, and the thickness of the nickel deposits obtained was about 1.8 mm. The deposit surface remained smooth and bright during

PJED. Fig. 9 shows the test results of hardness and surface roughness measured along the thin-wall part profile, both of which show small variations indicating a uniform structure.

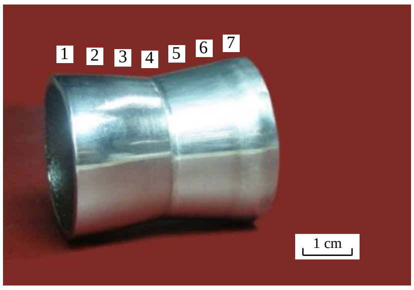


Figure 8. Photo of the nickel thin-walled part prepared by PJED.

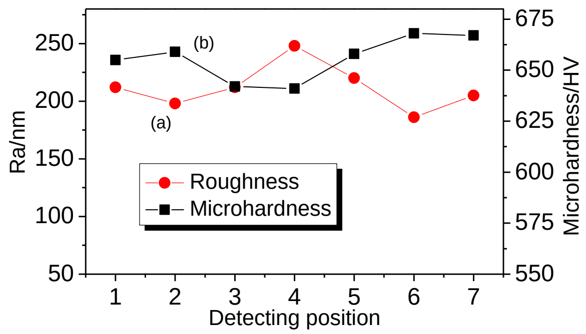


Figure 9. Distributing graph of (a) surface roughness and (b) microhardness.

4. CONCLUSION

During PJED, the particles were driven to do compound movements and polish the surface of cathode continuously. It is evident that the polishing of the particles can significantly smoothen the deposit surface without any organic additives by removing the pits and nodules of deposit in real time. The surface roughness and microhardness improved significantly compared with traditional JED. A nickel thin-walled part with a complex profile was experimentally produced with mirror-like surface and excellent hardness uniformity.

ACKNOWLEDGEMENTS
Authors acknowledge financial support from the Academic Program Development of Jiangsu Higher Education Institutions of China (Grant No. SZBF2011-6-B35).

References

1. S. M. Silaimani, S. John, *Bull. Electrochem*. 17 (2001) 553-560.
2. C. Richard, *J. Electrochem. Soc*. 192 (1982) 2424-2432.
3. C. Bocking, *Trans. Inst. Met. Finish*. 69 (1991) 119-127.

4. T. R. Bergstresser, H. D. Merchant, *Proc. TMS Fall Meet*. (1995) 115-167.
5. Y. D. Gamburg, *Elektron. Obrab. Mater.* (2003) 4-14.
6. E. Garfias-Garcia, M. Romero-Romo, M. T. Ramirez-Silva, M. Palomar-Pardave, *Int. J. Electrochem. Sci.* 7 (2012) 3102-3114.
7. J. A. McGeough, M. C. Leu, K. P. Rajurkar, A. K. M Rajurkar, Q. Liu, *CIRP Ann*. 50 (2001) 499–514.
8. L. P. Bicelli, B. Bozzini, C. Mele, L. D'Urzo, *Int. J. Electrochem. Sci*. 4 (2008) 356-408.
9. C. Bocking, B. Cameron, *Trans. Inst. Met. Finish*. 72 (1994) 33-40.
10. D. Thiemig, J. B. Talbot, A. Bund, *J. Electrochem. Soc*. 154 (2007) 510-515.
11. M. Kunieda, R. Katoh, Y. Mori, *CIRP Ann. Manuf. Technol*. 47 (1998) 161-164.
12. M. Karnik, A. Ghosh, R. Shekhar, *Trans. Inst. Met. Finish*. 87 (2009) 264-271.
13. S. Shivareddy, S. E. Bae, S. R. Brankovic, *Electrochem. Solid-State Lett*. 11 (2008) 13-17.
14. C. P. Chen, J. Jorne, *J. Electrochem. Soc*. 138 (1991) 3305-3311.
15. E. A. Mamontov, L. A. Kurbatova, A. P. Volenko, *Sov. Electrochem*. (1987) 166-170.
16. Z. W. Zhu, D. Zhu, N. S. Qu, W. N. Lei, *Mater. Des.* 28 (2007) 1776–1779.
17. Z. W. Zhu, D. Zhu, N. S. Qu, *Mater. Sci. Eng*., A. 528 (2011) 7461–7464.
18. Z. W. Zhu, D. Zhu, N. S. Qu, K. Wang, J. M. Yang, *Int. J. Adv. Manuf. Technol*. 39 (2008) 1164–1170.