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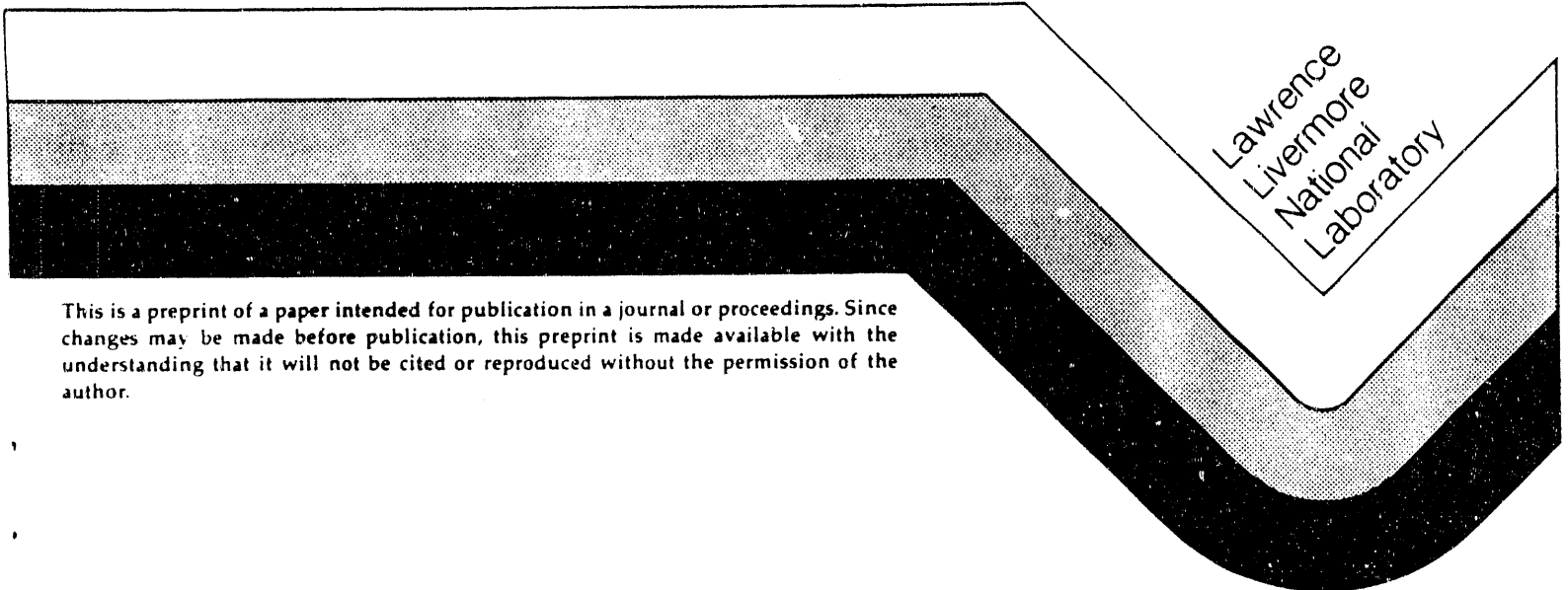
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DIAMOND TOOL WEAR OF ELECTRODEPOSITED NICKEL- PHOSPHORUS ALLOY

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Abstract

Nickel-Phosphorus alloys are attractive materials for diamond turning applications such as fabrication of large optics and other high precision parts. Although the mechanism is not understood, diamond tool wear is minimized when the phosphorus content of the deposit is greater than 11% (wgt). In recent years, increased attention has been directed at electrodeposition as an alternate to electroless deposition for producing Ni-P alloys. One principal advantage of the electrodeposition process is that alloys with 14-15% P can be obtained; another is that an order of magnitude greater deposition thickness can be provided if necessary. This paper compares diamond turning results for electrodeposited and electroless Ni-P alloys and shows that the electrodeposited coatings provide promising results.

Introduction

Diamond turning is the use of a single point diamond tool on a precision lathe under very carefully controlled machine and environmental conditions to fabricate high-precision components including reflective optics. Coatings offer significant advantages for diamond turning

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applications inasmuch as they can be applied to substrates that are not diamond-turnable such as beryllium, molybdenum or glass. One of the most frequently employed coatings for diamond turning applications is electroless nickel. Typical applications include fabrication of large optics and other precision parts(1-3). An excellent surface finish can be obtained; e.g., a surface roughness value of about 10 Å has been measured with a 10X WYKO optics profiler(4). To minimize diamond tool wear, electroless nickel deposits need to contain at least 11% phosphorus (Figure 1). A stress relief treatment at 200 degrees C for 2 hours even further enhances the cutting characteristics of deposits containing greater than 11% phosphorus(5).

Although electroless nickel deposits have worked well for diamond turning applications, they have some limitations. Typically, a minimum coating thickness of 3 mils is required and often as much as 10 mils is desired. This requirement taxes the capacity of many electroless nickel processes. Secondly, plating rates are slow; e.g., about 0.5 mil/hour; thirdly, and very important, the coating must be free of defects such as pinholes. This latter requirement has been the most difficult to meet for many applications. With all this in mind and with the increased technical coverage being devoted to electrodeposited Ni-P, we decided to investigate this deposit for diamond turning applications.

Details on Electrodeposition of Ni-P

Electrodeposited Ni-P alloys were first produced by Brenner in the early 1950s at the National Bureau of Standards(6,7). These early solutions were not widely used for a variety of reasons: nickel content rose steadily with use, phosphorus acid was converted to phosphoric acid, deposit stress was high, deposits were very brittle and solution life was indeterminate but usually rather short. In recent years, interest in deposition of Ni-P alloys has noticeably increased(8-24) and variations in solution composition and operating parameters have resulted in stable solutions capable of producing good deposits(20).

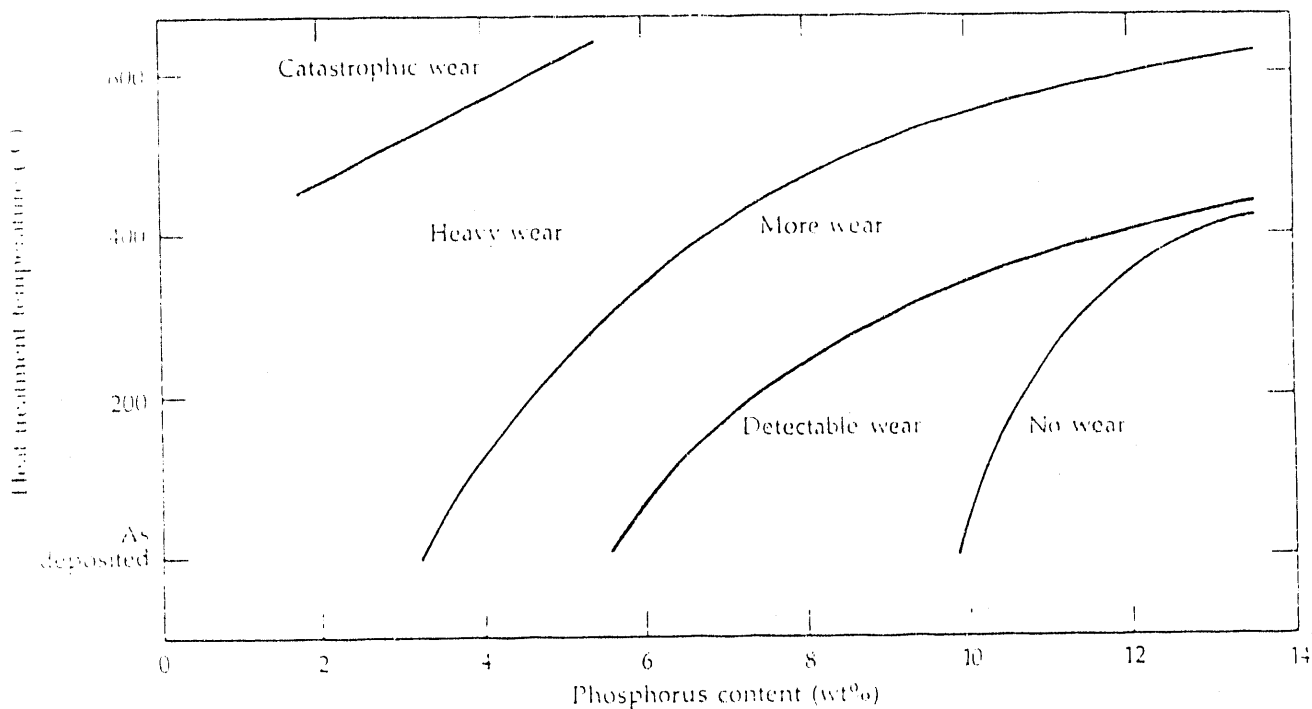


Figure 1 Map of diamond-tool flank-face wear and damage vs phosphorus content and heat treatment condition, based on scanning electron microscopy (from Ref. 5).

Plating Conditions

The deposits evaluated in this work were produced in a solution containing nickel chloride, nickel carbonate, phosphorous acid, phosphoric acid and a wetting agent (Table 1). Nickel chloride served as the primary nickel source. This salt is preferred for better solution conductivity and deposit properties. The total acidity of the solution is lowered with additions of nickel carbonate.

Free acid normality is maintained between 0.9 - 1.1 in the low speed formulation and 2.5 - 3.0 in the high speed formulation. Phosphorous acid acts as the phosphorus source in the solution, while phosphoric acid acts as a leveling, brightening agent. Solutions with phosphoric acid plate smoother, brighter deposits. It is possible that the acid provides a buffering effect in the diffusion layer, preventing the deposition of nickel hydroxides. A minimum concentration of 0.3 moles/liter is used in all

Table 1 Formulation and Operating Conditions for Solutions used to Electrodeposit Ni-P Alloys

	Low Speed*	High Speed**
Nickel (as nickel chloride)*	1.0 M	1.0 M
Phosphorous Acid (H ₃ PO ₃)	1.25 M	2.5 - 3.0 M
Phosphoric Acid (H ₃ PO ₄)	0.3 M	0.3 M
Temperature	75 C	75 C
Free Acid Normality**	0.9 - 1.1	2.5 - 3.0
Current Density***	100 - 150 asf	50 - 125 asf

*Total acidity of the solution is lowered with additions of nickel carbonate.

**Determined by titration with standard base solution using a methylorange endpoint.

***The low speed formulation deposits at 40-45% efficiency, while the high speed formulation is about 75% efficient.

formulations. A wetting agent can be added, if needed, to control pitting. Since the solution plates at less than 100% cathode efficiency, the wetting agent may be required to reduce pitting on complex shapes.

The temperature of the solution is maintained at 75 ± 5 degrees C. Higher temperatures induce a tensile stress in the deposit and reduce the phosphorus content. Lower temperatures provide compressively stressed deposits and increase the phosphorus content while lowering the plating rate. When operated within the recommended range, the deposit is neutral to slightly compressive in stress. The process will plate at current

densities from 50 to 500 asf. Higher current densities decrease the phosphorus content slightly.

Deposit Properties

Composition of the deposit, determined by energy dispersive x-ray analysis, was 14% phosphorus. Work by others(3,24) has shown the density of electrodeposited alloys to be consistently higher than those produced by electroless deposition (Figure 2). We suspect that the electrodeposited alloys contain a lower void volume than the electroless deposits; e.g., the nickel and phosphorus are more closely packed. This reduces the amount of porosity in the plated coatings and is of particular interest for diamond turning applications since it potentially leads to fewer microdefects in the material being machined, resulting in improved surface finish.

The as-plated hardness of the deposits used in this work was 580 VHN (100 gm load). Although the samples used in this work were not heat treated, exposure to 400 degrees C for 1 hour would have increased the hardness to approximately 1000 VHN (100 gm load).

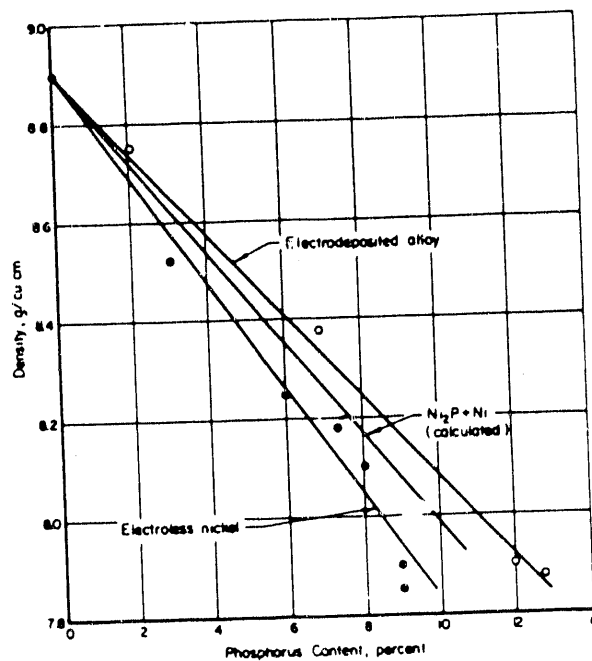


Figure 2 Density of Ni-P Alloys as a Function of the Phosphorus Content(3,24)

Description of Experiment

1. Initial sample preparation

The sample substrates were oxygen-free high-conductivity copper discs, 1.5 inches in diameter and 0.25 inches thick, precision machined to a flatness of 50 microinches on both faces. They were plated on one face with 8-10 mils of Ni-P alloy at a current density of 100 asf, which provided a deposition rate of 2.3 mils/hour.

2. Initial machining

After electroplating, the samples were prepared for the test cuts using a conventional precision lathe and a carbide tool. First, any roughness and non-uniform buildup was removed from the face and diameter until no uncut areas remained. Next, the center of the disk was relieved, cutting through the Ni-P layer to expose the underlying copper out to a diameter of 0.5 inches, leaving an annular ring of Ni-P. Finally, the unplated face of the disk was lapped flat to 20 microinches to mate with the vacuum chuck of the diamond turning machine.

3. Diamond tools

Single crystal diamond tools having a 60 mil nose radius and a -0.5 degree rake angle were used. The diamond crystals were oriented with the rake and clearance faces in the $\langle 110 \rangle$ direction. Cutting was done at the middle of the 110 degree total arc.

4. Machine tool

The cutting tests were performed with the Precision Engineering Research Lathe (PERL), a small (CNC) diamond turning lathe at Lawrence Livermore National Laboratory that was designed for high stiffness and low vibration amplitude. The PERL design employs two orthogonal slides, independently mounted on a heavy granite base, and a 4 inch, air-bearing spindle mounted with a brushless dc spindle motor. The PERL slides employ hydrostatic bearings and capstan-roller servo drives, and the granite base is mounted on pneumatic vibration isolators. Further details have been given previously(25).

5. Cutting tests

It has been found previously(26) that an effective technique for the quantitative measurement of diamond tool edge wear is to make plunge cuts into copper at fixed intervals during the tool wear tests, thereby creating grooves containing replicas of the tool nose that could be probed subsequently with a high-resolution stylus profiler. In the present test, after an initial copper plunge cut to establish the starting condition of the tool edge, a sequence of 60 facing passes was made across the Ni-P layer, each being 100 microinches deep, thereby removing a total thickness of 0.006 inches. After each 10 passes, another plunge cut groove was made (at a sufficiently different diameter to prevent overlap), yielding 6 more grooves.

The entire sequence was programmed to execute without interruption. The feed per revolution was 100 microinches resulting in a linear cutting distance of 0.25 miles per pass, or 2.5 miles between plunge-cut grooves and 15 miles total. The spindle speed was constant at 1000 rpm, the grooves were 50 microinches deep, and a light synthetic (polyalphaolefin) oil was used as a cutting fluid.

The above test conditions were selected to simulate the tool wear in a single pass for a 20 inch diameter annular mirror, to be cut at 75 rpm with a 1300 microinch feed per revolution, also yielding 15 miles of cutting distance.

Results

1. Talystep profiles of plunge-cut replicas

Figure 3 shows a composite of talystep profiles taken across the bottoms of the 7 successive tool signature replication grooves, labeled as to cumulative cutting distance. The leading edge is toward the left in Figure 3 and for a perfectly round tool cutting the deposit in an ideal manner, the tool-work contact would end at one-half of the feed-groove width, or 50 microinches, to the left of the lowest point on the profile. The tool develops wear zones of increasing width and depth with cutting distance.

For comparison purposes, Figures 4 and 5 present data obtained with electroless nickel containing 13% (wgt) phosphorus and heated at 200

degrees C for 2 hours in vacuum. This composition and treatment provided optimum diamond turning properties in previous studies(26,27). The electroplated Ni-P (Figure 3) compares quite favorably with the results obtained with the electroless Ni-P deposits (Figures 4 and 5).

2. Talystep profiles of Ni-P surface roughness

Figure 6 from earlier work shows a collection of surface roughness profiles, 500 microinches in length at cutting distances from 15 to 60,000 ft for electroless Ni-13P, illustrating the variety of profile shapes and amplitudes that occur(27). Figure 7 shows a similar measurement for the electrodeposited Ni-P at the end of the test after a cutting distance of 81,000 ft. Comparison of the two figures reveals that for the tools used in these studies, the electrodeposited Ni-P was not as damaging to the tool as the electroless Ni-P, yielding an rms roughness of 15.7 Å at 81,000 ft versus 83.2 Å for electroless nickel at about 60,000 ft.

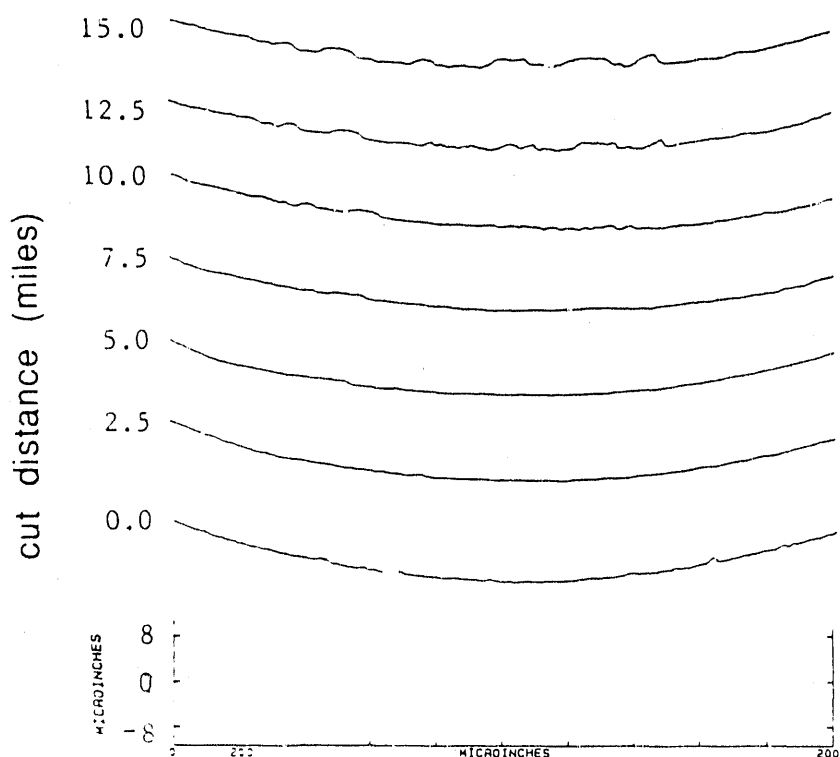


Figure 3 Talystep profiles of replication grooves for electrodeposited Ni-P labeled with cutting distance in miles. The leading edge of the tool is toward the left.

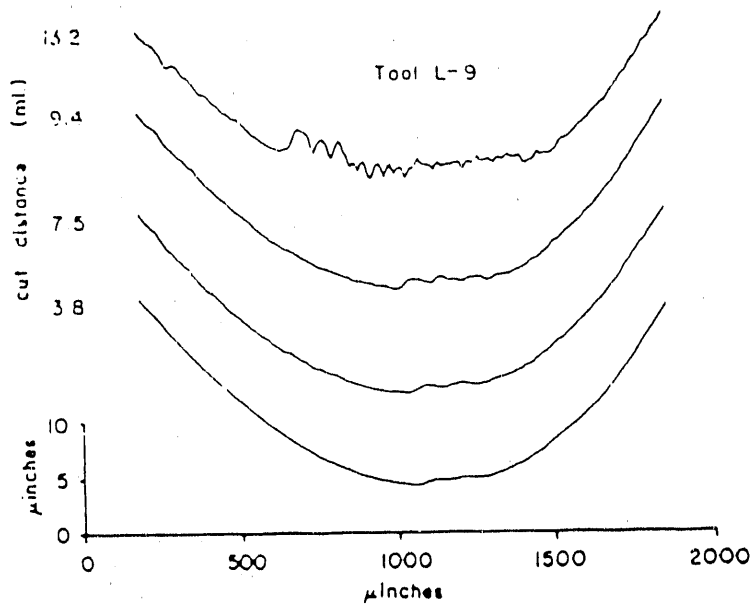


Figure 4

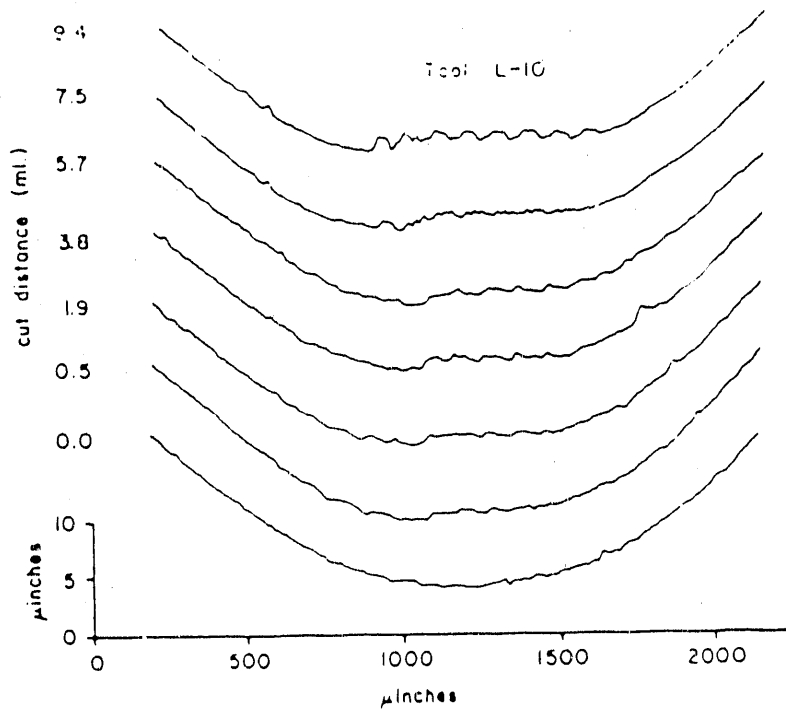


Figure 5

Talystep profile of replication grooves for electroless nickel labeled with cutting distance in miles (from Ref 27). The leading edge of the tool is toward the left.

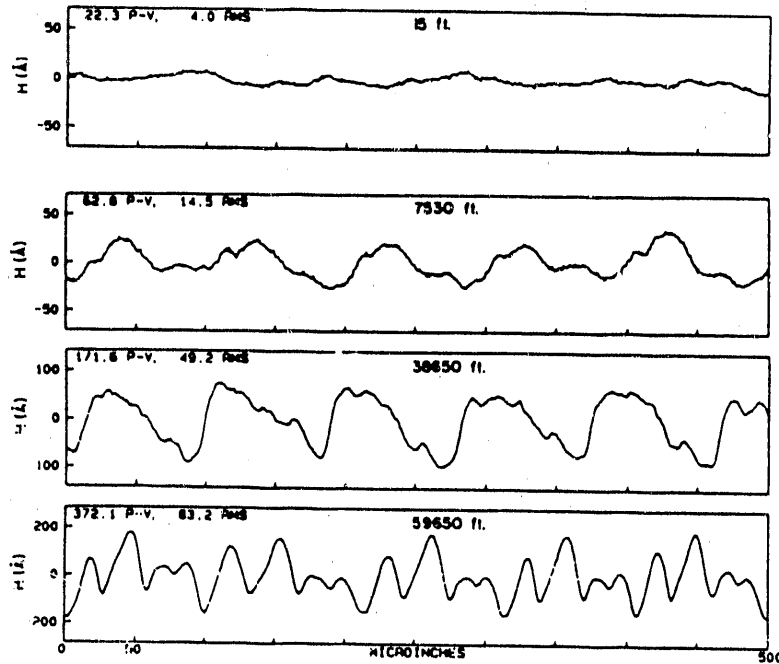


Figure 6 Quadratically detrended feedmark profiles at various cutting distances for electroless Ni-13P. Note vertical scale change in the two bottom scans; tool advance was from right to left (from Ref 27). Each trace spans five feedmarks.

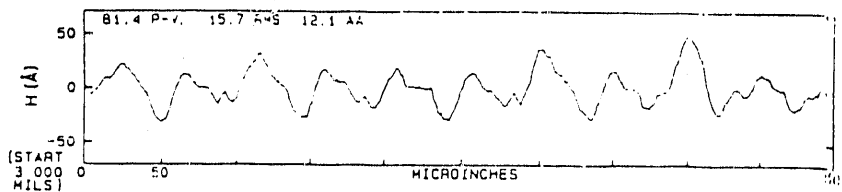


Figure 7 Quadratically detrended feedmark profile, for electro-deposited Ni-P after 81,000 ft of cutting. The trace spans five feedmarks.

Discussion

These preliminary results showing that electrodeposited Ni-P alloys perform quite similarly to electroless Ni-P deposits for diamond turning applications should not be surprising since many of the metallurgical characteristics of the alloy are independent of the deposition process.

Coatings produced by either electrodeposition or electroless deposition both exhibit a laminar structure upon etching(10). These laminations reflect differences in the phosphorus content, that is, phosphorus-rich and phosphorus-depleted zones, according to the findings of Ogburn and Johnson(28). Films with P contents greater than 7 to 8 weight percent have been shown to be amorphous by many studies, regardless of the deposition process(9). One difference between the alloys is hardness. Microhardness of pulse and dc plated Ni-P alloys was consistently higher than electroless deposited alloys, with the highest hardness occurring at the highest current density. It is suggested that this is caused by two different amorphous configurations in the electrodeposited alloys(8).

A very important observation is that phosphorus content of the electrodeposited alloys can be increased to greater than 20 weight percent(8). This is done by pulsed current deposition and could offer even further improvement in reducing diamond tool wear. As shown earlier in this paper (Figure 1), deposits containing greater than about 10 weight percent P exhibited the least tool wear during machining. It would be interesting to evaluate deposits with 20% P, or greater for diamond tool wear.

The electroless deposition process is slow but has the advantage of providing uniform coverage. It is an expensive, batch process and quite difficult to utilize for coating thicknesses greater than about 5 mils. Electrodeposition is faster but it cannot provide coverage on odd shapes uniformly. Utilization of this process with unusual shapes would require the assurance that P content is not a function of current density or require creative shielding to assure that current density is uniform during deposition. Electrodeposition offers the ability to deposit coatings considerably thicker than 5 mils with much more ease than via the electroless route.

Summary

Based on this preliminary work, electrodeposited Ni-P coatings offer promise as a substitute for electroless Ni-P coatings for applications requiring diamond machining. An electrodeposited Ni-14P coating compared quite favorably in terms of low tool wear and achievable surface roughness with electroless nickel coatings even after a cutting distance of 81,000 ft (15.3 miles). Since the electrodeposition process offers the potential for providing coatings thicker than 5 mils less expensively, and with potentially fewer defects than electroless nickel, it is suggested that consideration be given to the process for diamond turning applications.

References

1. J. M. Casstevens and C. E. Daugherty, "Diamond Turning Optical Surfaces on Electroless Nickel", in Precision Machining of Optics, T. T. Saito, ed., Proc. SPIE 159 109 (1978).
2. J. W. Dini, "Electroless Nickel: An Important Coating for Diamond Turning Applications", Proc. Electroless Nickel Conference II, Gardner Publications, Cincinnati, OH (1981).
3. G. M. Sanger and J. W. Dini, "A Perspective on Electrodeposited and Electroless Nickel Coatings Used in Optical Applications", Proc. SUR/FIN 82, American Electroplaters Society (1982).
4. E. L. Church and P. Z. Takacs, "Survey of Finish Characteristics of Machined Optical Surfaces", Opt. Eng., 24 (3), 396 (1985).
5. C. K. Syn, J. W. Dini, J. S. Taylor, G. L. Mara, R. R. Vandervoort and R. R. Donaldson, "Influence of Phosphorus Content and Heat Treatment on the Machinability of Electroless Nickel Deposits", Proc. Electroless Nickel Conference IV, Gardner Publications, Cincinnati, OH (1985).
6. A. Brenner, D. E. Couch and E. K. Williams, "Electrodeposition of Phosphorus with Nickel or Cobalt", J. Research, National Bureau of Standards, Research Paper RP 2061, 44, 109 (Jan 1950).

7. A. Brenner, D. E. Couch and E. K. Williams, "Electrodeposition of Alloys of Phosphorus and Nickel or Cobalt", *Plating* 37, 36-42, 161, 162 (1950).
8. D. S. Lashmore and J. F. Weinroth, "Pulsed Electrodeposition of Nickel-Phosphorus Metallic Glass Alloys", *Plating & Surface Finishing*, 69, 72 (Aug 1982).
9. E. Vafaei-Makhsoos, E. L. Thomas and L. E. Toth, "Electron Microscopy of Crystalline and Amorphous Ni-P Electrodeposited Films: In-Situ Crystallization of an Amorphous Solid", *Metallurgical Transactions A*, 9A 1449 (1978).
10. A. W. Ruff and D. S. Lashmore, "Dry Sliding Wear Studies of Nickel-Phosphorus and Chromium Coatings on 0-2 Tool Steel", Selection and Use of Wear Tests for Coatings, ASTM STP 769, R. G. Bayer, Ed., American Society for Testing and Materials, 134 (1982).
11. D. S. Lashmore, L. H. Bennett, H. E. Schone, P. Gustafson and R. E. Watson, "Polymorphism of Nickel-Phosphorus Metallic Glasses", *Physical Review Letters*, 48, No. 25, 1760 (21 June 1982).
12. C. Rajagopal, D. Mukherjee and K. S. Rajagopalan, "Electrodeposition of Corrosion Resistant Amorphous Nickel Alloys on Mild Steel", *Metal Finishing*, 82, 59 (Jan 1984).
13. R. Narayan and M. N. Mungole, "Hardness Control in Electrodeposited Nickel-Phosphorus Coatings", *Metal Finishing*, 83, 55 (Jan 1985).
14. "Studies of Electrodeposited and Electroless Nickel Phosphorus Alloys", UDR-TR-85-142 (Nov 1985).
15. R. Narayan and M. N. Mungole, "Electrodeposition of Ni-P Coatings", *Surface Technology*, 24, 233 (1985).
16. A. Mayer, K. Staudhammer and K. Johnson, "Electroformed Bulk Nickel-Phosphorus Metallic Glass", *Plating & Surface Finishing*, 72, 76 (Nov 1985).

17. M. Ratzker, D. S. Lashmore and K. W. Pratt, "Electrodeposition and Corrosion Performance of Nickel-Phosphorus Amorphous Alloys", *Plating and Surface Finishing*, 73, 74 (Sept 1986).
18. I. Kim, R. Weil and K. Parker, "Comparison of Some Properties of Electroless and Electrodeposited Ni-P Deposits", *Proceedings SUR/FIN 87, AESF*, 1987.
19. P. K. Ng, D. D. Snyder and J. LaSala, "Structure and Crystallization of Nickel-Phosphorus Alloys Prepared by High-Rate Electrodeposition", *J. Electrochem. Soc.*, 135, 1376 (June 1988).
20. D. J. Sugg, "Electrodepositing Electroless Nickel", *Products Finishing*, 53, 66 (Sept 1989).
21. N. E. Myers, R. L. Gamblin and D. J. Sugg, "Commercial Nickel Phosphorus Electroplating", US Patent 4,673,468, June 1987.
22. R. L. Gamblin, N. E. Myers and D. J. Sugg, "Nickel Phosphorus Electroplating and Bath Therefor", US Patent 4,767,509, August 1988.
23. D. J. Sugg, "Electroforming with Electrolytic Ni- and Co-P Alloys", *Proceedings AESF Electroforming Symposium, Las Vegas, NV, Oct 1989*.
24. W. H. Safranek, The Properties of Electrodeposited Metals and Alloys. A Handbook, Second Edition, American Electroplaters and Surface Finishers Society, Orlando, FL 1986.
25. R. R. Donaldson and D. C. Thompson, "Design and Performance of a Small Precision CNC Turning Machine", *CIRP Annals*, Vol. 35, No. 1, 1986.
26. J. S. Taylor, C. K. Syn, T. T. Saito and R. R. Donaldson, "Surface Finish Measurements of Diamond-Turned Electroless Nickel Plated Mirrors", *Optical Engineering*, 25, No 9, 1013 (1986).
27. C. K. Syn, J. S. Taylor and R. R. Donaldson, "Diamond Tool Wear vs Cutting Distance on Electroless Nickel Mirrors", Lawrence Livermore National Laboratory, UCRL-95513, Oct. 14, 1986.

28. F. Ogburn and C. E. Johnson, "Banded Structure of Electroless Nickel", *Plating & Surface Finishing*, 60, 1043 (1973).

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