Diamond-Dispersed Electroless Nickel Coatings

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iamond powder incorporated tools have become important industrial materials, enhancing productivity in metalworking, materials production, grinding and finishing, precision cutting, and machining. Diamond coatings have entered into commercial markets via tool inserts, audio speakers, X-ray windows, and heat sinks, and directly interface with the heavy automotive and high-tech areas as well as the stone processing industries for cutting, slicing, polishing, and finishing.¹

Diamond powders are available in several forms. Broadly, there are two types: monocrystalline and polycrystalline. Single crystals of diamond fracture along cleavage planes result in sharply defined edges and points. Natural diamond and certain types of synthetic diamond belong in this category. Shock-synthesized diamond, on the other hand, is polycrystalline. The choice of the type of the powder that can be used for making composites depends upon the end use of the composite.^{2,3}

Diamond particles incorporated into a metal matrix are usually designed for at least one of several purposes such as grinding or abrasive applications, wear resistance or surface protection properties, controlled surface topography involving specific ranges of roughness or surface friction qualities, antigalling or lubricating uses, and corrosion resistance.

By codepositing nickel with very fine particles or polycrystalline diamond, composite coatings with high wear resistance have been produced, adopting both electrolytic and electroless techniques. The wear properties of these coatings will depend on how firmly the particles are anchored in the matrix.^{4,5}

Electroless nickel solutions are found to be suitable for composite coatings resulting in consistent, homogeneous concentration of the particulate material in the composite. The electroless nickel plating technique is well known for its simplification of electroplating in avoiding problems of current distribution, throwing power, and anode-cathode orientation within the bath. Its inherent problem is that the stability is aggravated by the inclusion of par-

In this article the authors share some of their experiences in this area.

EXPERIMENTAL TECHNIQUES

A solution of the composition shown in Table I was used for the preparation of the diamond composites. The pH of the solution was adjusted electrometrically to 5.0 with caustic or dilute sulfuric acid. The volume-to-area ratio was maintained as 16 and fresh solutions were taken for each experiment.

ticulate matter in the bath. Despite this problem,

the process has become a commercial reality.⁶⁻⁷

Plating was done on 4-cm diameter cups made of aluminum. Various pretreatment procedures for the substrate were tested and the best treatment was selected based on the ring shear test for adhesion (Table II).

Diamond powder (8 to 12 μ m) of natural variety was used for the study. The diamond powder concentration was varied from 2 to 10 g/L and the temperature from 70 to 90°C. The volume percent of incorporation was estimated by knowing the total mass of the deposit obtained in one hour; that of the powder incorporated into the deposit, as estimated by stripping the deposit and gravimetric determination of the powder in the above solution; and the density of nickel.

The deposits were heat treated at 250°C for 2 hours in air to improve the hardness, estimated by the Vicker's indentation method. Wear index was assessed in a Tabor abraser at a 1-kg load with a No. 10 wheel. Metallographic examination was made using a metallurgical microscope at $400 \times$.

RESULTS AND DISCUSSION

Preliminary experiments were based on identifying a suitable pretreatment procedure for plating on aluminum. The modified zincating process⁸ was

Table I.	Electroless	Nickel	Bath	Composition	
and the second s					

Nickel sulfate	26.0 g/L
Sodium hypophosphite	24.0 g/L
Sodium acetate	30.0 g/L
Lactic acid	27.0 g/L
Propionic acid	2.2 ml/L
Lead	2.0 ppm

Treatment	Shear Strength, N/m ²	Bent Test	Quenching
Soak clean, acid dip, alloy zincate, single copper strike, EN plate	$33.439 imes 10^6 \ 29.600 imes 10^6 \ 36.624 imes 10^6$	Peels	Blisters
Soak clean, acid dip, alloy zincate, double copper strike, EN plate		Adherent	Adherent
Soak clean, acid dip, double alloy zincate, EN plate		Peels	Peels
Soak clean, acid dip, double alloy zincate, copper plate, EN plate, heat treat		Adherent	Adherent

Table II. Adhesion of Deposits on Aluminum

Table III. Effect of Powder Content on the Deposition Rate

Concentration	Thickness	Vol. %
<u>(g/L)</u>	(µm/hr)	in Deposit
0.0	26.42	
2.0	22.27	10.90
2.0^{a}	21.90	27.70
5.0	20.26	15.60
8.0	13.00	17.80
10.0	4.74	18.40

"Horizontal mode.

found to be the best in producing adherent deposits even after heat treatment. Adhesion of the deposits was tested by ring sheer test, bend test, and quenching test.

Most common practices appear to focus on 25 to 30% by volume of particulate material in the deposit. Experiments conducted with varying concentrations of dispersoid in the solution, as shown in Table III, indicate that electrodeposition plays a major role in deciding the composition of the deposit. When deposited under a vertical condition the incorporation was less and hardly exceeded 20%, even when the powder concentration in the solution was

 Table IV. Effect of Operating Variables on the Deposition Rate

 and Hardness

pН	Temperature (°C)	Deposition Rate (µm/hr)	Hardness (Kp/mm ²)
4.0	90	4.79	466
5.0	90	22.27	720
6.5	90	21.65	585
5.0	80	2.30	_

Table	V.	Hardness	of	Nickel	Com	posites

Concentration (g/L)	Condition	Hardness (Kp/mm ²)
0.5	Vertical	223
1.0	Vertical	508
2.0	Vertical	720
5.0	Vertical	757
2.0	Heat treated	862
2.0	Horizontal	830
2.0	Horizontal and heat treated	1,033
0.0	Vertical	493
0.0	Vertical and heat treated	711

Load 100 gms.

Table VI. Wear Resistance of Electroless Composites

Material	Condition	Wear Index (g/dm ²)
Aluminum		1.759
EN	As plated	0.098
EN	Heated	0.081
EN composite	Vertical as plated	0.076
EN composite	Vertical heated	0.065
EN composite	Horizontal as plated	0.053
EN composite	Horizontal heated	0.030

EN-electroless nickel.

more than 10 g/L. Moreover, the deposits obtained were noncoherent in nature with a very low rate of deposition. In the horizontal plane the required volume percentage could be obtained at 2-g/L powder concentration and the deposits obtained were smooth, adherent, and coherent.

The operating variables have their own influence in affecting the powder incorporation, as shown in Table IV. The deposition almost ceased to occur at temperatures below 80°C and pH below 3.5, and correspondingly affected particle incorporation.

Hardness of the deposits, as given in Table V, increased with the volume percent incorporation, and showed a higher value for deposits obtained by the horizontal technique. Heat hardness measurement showed some inconsistency with deposits having higher volume percent.

Wear resistance of the deposits, as shown in Table VI, registered a maximum value for those plated in the horizontal plane and subsequently heat treated. Considerable improvement in the wear index of the specimens was observed when diamond particles were incorporated in the deposit.

CONCLUSION

Adherent electroless diamond composites could be produced successfully on aluminum substrates with suitable pre- and posttreatment. The hardness of the deposits was similar to hard chromium. Using the horizontal technique, it is possible to get diamond-nickel composites containing around 30% powder incorporation with a very low amount of powder in suspension.

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