IDENTIFICATION OF DEFECTS ON NiDi COATING

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Abstract

The nickel diamond coating NiDi is made up of two materials: electroless nickel (Ni-P) and diamond. Because of the electroless chemical method of deposition, nickel diamond coating covers all surfaces of the work piece with essentially perfect uniformity. A standard coating should contain 20 to 30 % diamond by volume with a thickness of around 20 µm. On wire samples several defects were identified by 3D optical microscopy and by SEM equipped with EDX analysis. The cause of the defects in electroless nickel plating can be divided into two main categories:chemistry problems and deposit problems.

Keywords: NiDi coating, electroless deposition, coating thickness, diamond particle size, defects, analysis

1. INTRODUCTION

Surface coatings are widely used in various industries to improve the functional performance and robustness of substrate materials. Electroless nickel coatings have excellent corrosion and wear resistance, as well as other specific properties [1]. The electroless nickel plating technique is well known for its simplification of electroplating in avoiding problems of current distribution, throwing power and anode-current orientation within the bath [2]. Electroless nickel coatings are ideal for steel, aluminium, copper, and polymers because they improve hardness and wear and corrosion resistance. In order to achieve better hardness, wear and corrosion resistance, electroless Ni–P coatings are best suited for steel, aluminium, copper, and plastics [3]. However, the desired characteristics are dependent on the percentage of the phosphorus concentration in the coating. The ability to co-deposit particulate matter in a matrix of electroless nickel has led to a new generation of composite coatings. Mechanical and tribological properties of the coating can be further enhanced by reinforcing the second phase particles at micron, submicron and nano sizes to the Ni–P lattice of the electroless coating, such as ceramics, diamond and fluoropolymers and carbides of silicon, tungsten and chromium [1]. Different hard particles with Ni-P coatings improve various properties of different substrate [4].

Nickel–diamond composite powders with nickel layer on the surface of synthetic diamond have been used for as cutting wheel, slicing, polishing and finishing of stone processing industries. Composite diamond coating (DLC) is a regenerative layer of fine diamond particles dispersed in a hard electroless nickel matrix. It was found that adherent electroless diamond composites could be produced on various substrates with suitable pre- and post-treatment [5, 6]. Their incorporation in electroless nickel brings modifications of the coating morphology, with diamond particles often protruding from the coating, leading to an aspect that can be described as foggy and rough [7]. The most significant effect of the addition of diamond in electroless Ni-P is the increase in hardness that is followed by an increased wear resistance, and also by an increase in elastic modulus. The Ni-P + DLC multilayer coating can be an optimal solution to increase the tribological and mechanical performance of the substrates: the hard interlayer (Ni-P) enhances the load-bearing capacity of the substrate, improving the top-coating DLC adhesion, which in turn reduces the wear rate and the coefficient of friction in the system.

The electroless Ni-P coatings with 5 to 8 % of phosphorous may contain small (3 – 6 µm), medium (6 – 12 µm), and large (20 – 40 µm) size diamond particles with the variation in the phosphorus content of the matrix

(**Figure 1**). The deposits with lower phosphorus content have very little integrity with the diamond particles compared to the deposits with higher phosphorus content in the as-deposited condition. The density of diamond particles within composite diamond coating is typically from 20 to 30 % by volume.

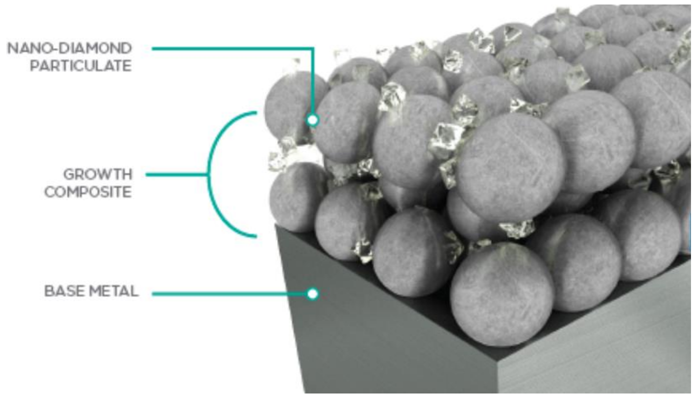

Figure 1 The NiDi coating structure [8]

The Ni–P coating exhibits a typical cauliflower-like morphology (**Figure 2a**) with a smooth surface. Comparing (**Figure 2a** with **Figure 2b**) shows that the incorporation of diamond nano particles (DNP) with Ni–P coating caused a great change on the surface morphology. In the Ni–P deposition, nickel grains have a cauliflower-like growth, while the microstructure of composite coating shows fine-grained granular due to nucleation and growth of Ni grains on the DNP in various directions. Surface of co-deposited DNP provide new and appropriate sites for nucleation of nickel grains, so that the amount of nucleation sites for Ni–P/DNP is higher than Ni–P.

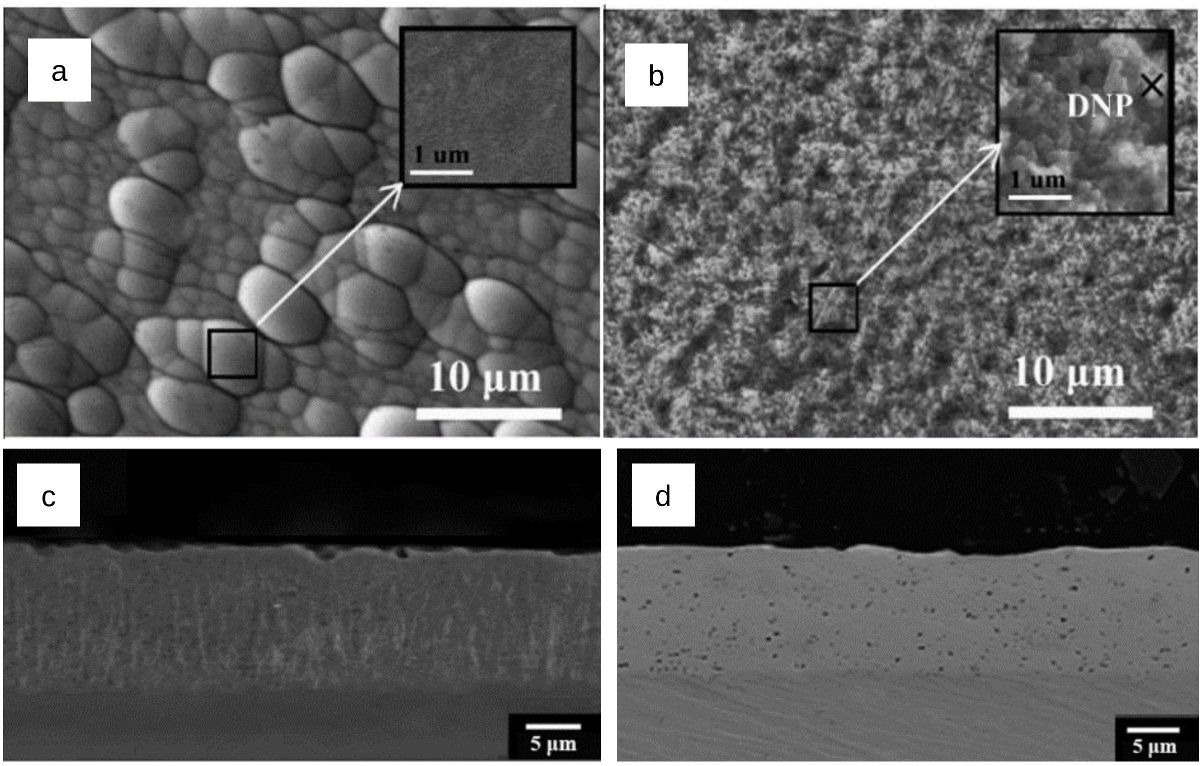

Figure 2 SEM images and cross-section of the surface for prepared coatings by electroless deposition: (a, c) Ni–P; (b, d) Ni–P/ diamond nano particles [9]

Due to the electroless chemical method of deposition, composite diamond coating covers all surfaces of the substrate with essentially perfect uniformity. There is no build up on edges, corners, inner diameters, or any other surface. Any thickness from 5 μm to 625 μm can be produced, although most applications use a standard coating thickness of 25 μm [10].

2. NiDi COATING ON WIRE FOR TEXTILE MACHINERY

Metallic wire is a wire made of metal which has saw tooth shape, and combs fibres on the process of carding in manufacturing of fabric (**Figure 3**). Surfaces in contact with fibres, in particular, are protected effectively against wear. The wires are treated with special smooth coating to obtain corrosion protection for the use in humid conditions. Electroless nickel and electroplated nickel dispersion coatings have been used successfully in textile machinery construction for decades [11].

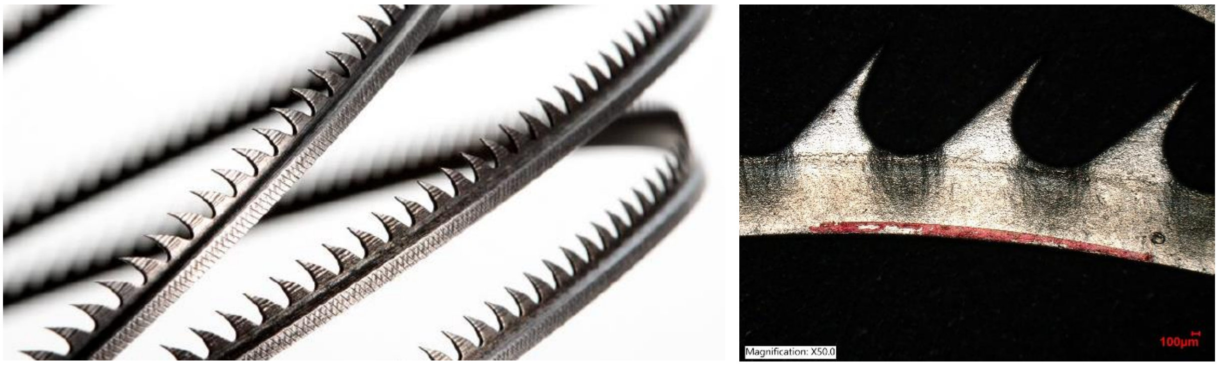

Figure 3 Example of wire and detail of coating

The NiDi coating was applied on special wire for textile industry. Two samples were evaluated: reference **sample A** without defects and **sample B** on which the coating showed a lot of defects (**Figure 4**) documented with 3D optical microscope Keyence VHX 5000:

- black spots and stains (**Figure 4a**),
- blisters (**Figure 4b**),
- overlapping layers which partly delaminated (**Figure 4c**).

Figure 4 Examples of defects on NiDi coating (sample B)

2.1 Thickness and structure of coating layer

The coating layer thickness was measured on the cross sections of wires. On wire without defect (A) the coating layer was distributed uniformly **(Figure 5a**). The coating thickness of evaluated coating varied from 10 to 30 μm. The structure of the NiDi coating guarantees the good adhesion.

On wire sample B with defects the coating was distributed nonuniformly (**Figure 5b, 5c**). The layer thickness was reduced to 3 – 4 µm within the areas of black stains. Non-uniform deposits can be the result of out-of-range electroless bath operating parameters and insufficient surface preparation.

The overlapping was connected with doubled layer. This defect is caused by bath contamination – that can be prevented by bath filtration each 1 to 2 weeks.

The height of numerous spots with grape-like "blisters" reached approximately 55 µm. Deposit blisters can arise from poor surface preparation, an over-stabilized electroless bath or dragging contaminated rinses into the bath.

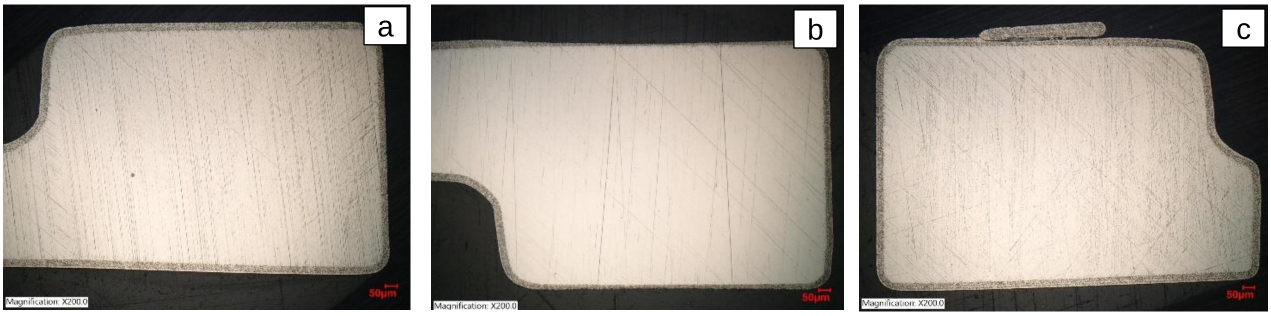

Figure 5 Examples of NiDi coating cross sections

The detailed distribution of diamond particles in Ni-P coating are shown on **Figure 6**. The diamonds´ particle dimensions are small, ca 4 µm. On the top of coating on sample B there was more pronounced layer without diamond particles (bright layer).

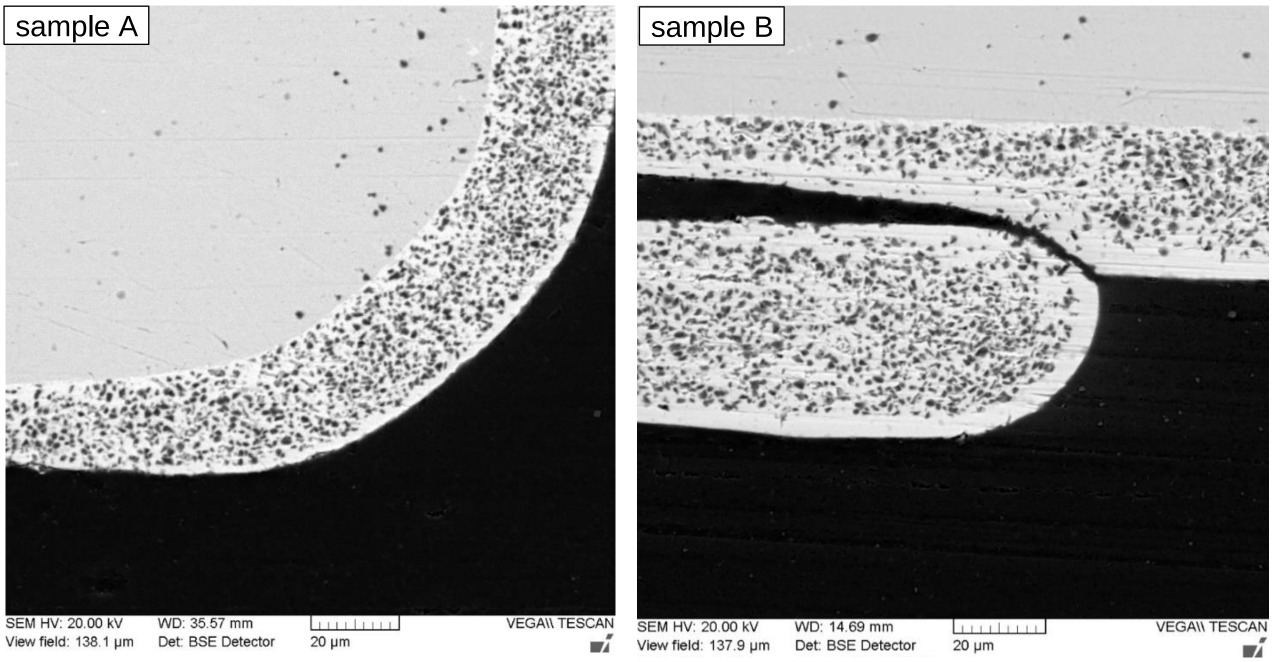

Figure 6 Distribution of diamond particles

2.2 Chemical composition of coating layer and contamination

Electroless nickel is not pure metal but includes other elements also derived from the reducing agent, such as phosphorus, or derived from other bath additives. Microstructural analysis was performed using VEGA II

(TESCAN, CR) with Energy-Dispersive X-ray Spectroscopy (EDX, X-Max 50 SDD, Oxford, UK) on cross sections extracted from specimens. An EDS analysis revealed an average P content in the Ni-P layer equal to ca 8 wt. % and diamond particles as carbon (ca 5.5 wt. %) – **Table 1**. The phosphorus content in the deposited coating effected its morphology. With increasing phosphorus content, the fineness of the structure increased as well.

The surface of the samples was covered with gray or gray and white layer. There was a lot of white “powder” inside of the bubbles and under the delaminated overlapping layers, which is probably related to contamination by residual of chemical bath. EDX was used to determine the chemical composition of the coatings´ contamination (**Figure 7**).

The content of dominant element of Ni-P/Di coating differs on the wire samples A and B. On sample B there is higher content of carbon (ca 10 wt. %) and phosphorus (ca 9 wt. %). Spots with very high content of carbon show also high content of oxygen; together with visual evaluation (see indication on Figure 7) they seem to be residua of carbonates which are used in bath for autocatalytic application of coating.

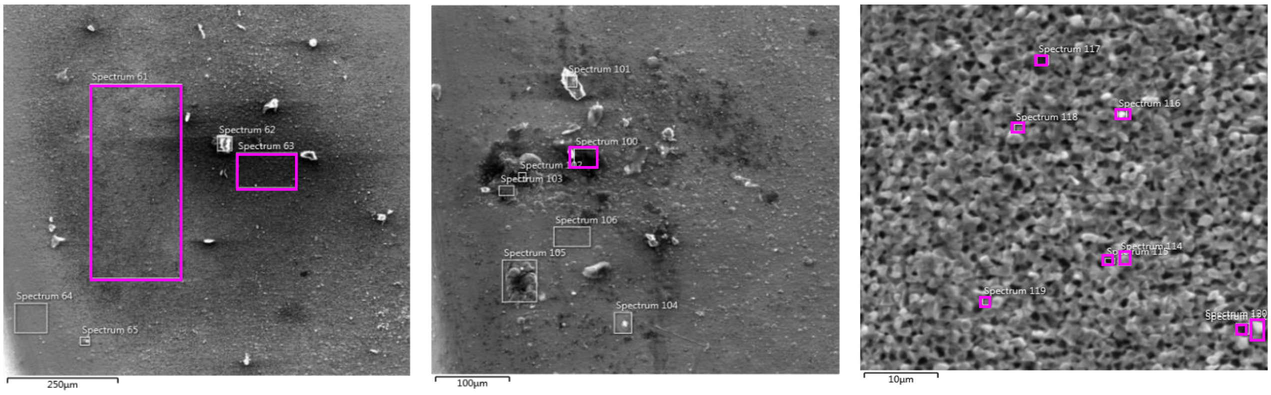

Figure 7 The contamination identification on NiDi coating layer (sample B)

Table 1 The EDX analyses of NiDi coating (selected elements)

sample	average element content (wt%)				
	C	**N**	**P**	**Ni**	**O**
A, reference	5.50	-	7.70	85.21	1.22
A, Di particles	93.51	-	-	1.07	-
B, reference	10.35	-	8.98	76.97	2.28
B, Di particles	84.38	-	-	11.98	-
B, stains	45.52	2.80	2.30	22.83	9.92

The stains contain significant amount of nitrogen – on spots to 8 wt. %. at 100 mg.L^{-1} of nitrates can significantly affect the Ni-P coating adhesion. The main cause of the problems such as streaks, discoloration, slow or no deposition is poor rinsing after a nitric acid pretreatment - activation. Accurate test papers can be used to confirm nitrates in the bath. Correction is either replace the bath or improve rinsing between acid desmut and the electrolytic bath.

Sporadically the contamination by chlorine, iron, silica (quartz) and other elements were found on the coating surface. Some insoluble contaminants, such as iron, co-deposit with the electroless nickel and lower the corrosion protection. Iron, calcium, and other metals may combine with the chelates, which reduces the amount of chelates available for the nickel.

3. CONCLUSION

Nickel–diamond composite coatings were successfully synthesized by using an electroless plating method. The Ni–P–C composite coatings incorporating finer diamond particles are more wear resistant compared to those incorporating coarse diamond particles. By applying diamond coating the metal parts can increase their lifetime and minimize maintenance related downtime due to the replacement of high wear parts.

The evaluated electroless nickel-coated wires showed some defects - roughness, lack of adhesion, poor coverage or lack of uniformity on sample B. The cause of the defects in NiDi coating can be divided into two main categories; chemistry problems and deposit problems:

- non-uniform thickness of coating – thickness varies from 10 to 30 μm – the black spots on wire surface correspond to areas with minimal coating thickness – locally 3 – 4 μm,
- non-uniform distribution of diamond particles,
- overlapping of coating layers - some overlapped layers delaminating from surface – remaining areas show reduced thickness (black spots) and black spots with white powder (residual contamination from bath),
- contamination of surface and gaps between overlapped layers by salts from chemical bath (carbonate, nitrate, etc.) – not delaminated layers,
- blisters - the bubbles from gases emitted during chemical process (hydrogen).

The wire steel surface and the pretreatment of steel wire before electroless plating should not be the reason of defects, because the layers show good adhesion to steel surface.

Electroless nickel is a barrier and cathodic coating on steel. Any defects in the coating will lead to poor corrosion protection of substrate steel.

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