JTTEE5 9:241-244

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Duplex Coating of Electroless Nickel and HVOF (High-Velocity Oxygen Fuel) Sprayed WC-Co

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(Submitted 28 June 1999; in revised form 10 December 1999)

The porosity of thermal sprayed coatings is usually a problem when coatings are used in wet corrosion application. The porosity allows media to penetrate to the surface of the base material. Corrosion spreads rapidly and the coating loses contact with the substrate and delaminates. This problem can be initiated by impregnating different polymers into the pores. An alternative approach has been tested in the present work to prevent corrosion of cemented carbide coated carbon steel in wet corrosion environments.

Carbon steel substrates were coated with a thin film of electroless nickel (electroless nickel plating) and then HVOF (High-Velocity Oxygen Fuel) sprayed with cemented carbide. Reference specimens without electroless nickel were sprayed at the same time. The microhardness of the specimens was measured and the coating structure examined using optical microscopy and X-ray diffractometry (XRD). The bond between the layers and the base material was examined by means of a bend test. A salt chamber test was also performed for the specimens.

The structure of the electroless nickel layer was crystalline as a result of the HVOF spraying. There were no cracks in the nickel layer, if the layer was about 20 μm thick. According to the results of the bend test, the adhesion between coatings and substrate was good, and there was no difference between the duplex-coated specimen and the reference specimen. A sample with a thin nickel layer under an HVOF sprayed cemented carbide did not exhibit corrosion after 8 h in the salt chamber test.

Keywords corrosion, HVOF spraying, cemented carbide, chemical nickel, electroless nickel

1. Introduction

Different layers under thermally sprayed coatings have been examined to obtain better adhesion or high-temperature oxidation resistance. These layers have been formed by various methods, such as thermal spraying or electroplating.[1] A new approach is to use layers under thermally sprayed coatings to prevent corrosion. Chemical (electroless) nickel plating is normally used because of its excellent corrosion and good wear resistance. Electroless nickel coatings also demonstrate excellent adhesion to the substrate.[2]

The wear resistance of HVOF (High-Velocity Oxygen Fuel) sprayed cemented carbides is similar or better than that of electroless nickel.[4,5] Also, the coating thickness achieved by HVOF spraying is greater than for electroless plating; the HVOF coating can be as thick as 2 mm, while electroless plated coatings are typically up to ~50 μm thick. The features of these two coatings can be combined to obtain excellent corrosion and wear resistance properties.

As the electroless nickel is heated to temperatures above 220 to 260 °C, structural changes begin to occur. The electroless nickel begins to crystallize and lose its amorphous structure at 320 °C. Heat treatment at temperatures above 320 °C radically affects the hardness and the tensile strength. As a result of structure changes, shrinking and loss of corrosion resistance will occur as the phosphor precipitates from the nickel matrix to the grain boundaries. Shrinking and associated cracking can result, and these cracks form passageways for the electrolyte to penetrate through the coating to the substrate. Temperatures above 320 °C can be reached during thermal spraying, and therefore, structural changes may affect the adhesion of electroless nickel.[2,3]

The objective of this work was to investigate the microstructure, the microhardness, the adhesion, and the corrosion behavior of duplex systems consisting of coatings of electroless nickel after being sprayed with cemented carbide using HVOF spraying.

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2. The Experiment

Three specimens were coated using an autocatalytic chemical (electroless) reduction of nickel ions. One specimen was left without electroless nickel and used as a reference. The nickel coating was deposited in an acidic bath using sodium hypophosphite as a reducing agent. The deposition temperature was about 90 °C. The thickness was measured by a microscopic method according to standard EN ISO 1463[6] (Table 1).

All specimens were sprayed simultaneously using an HV-2000 (Praxair Surface Technologies, Inc., Appleton, WI) torch with hydrogen gas as a fuel. The possible moisture on the surface of specimens was removed by preheating them to 70 °C immediately with the HVOF flame. The coating process was started immediately after preheating by turning the powder feeding on. The powder (AI-1173-TG) was tungsten carbide with 17% cobalt as a matrix and was made by Alloys International Inc. (Baytown, TX). The spraying parameters were optimized according to the microhardness and density of the coating made

Table 1 Coating thicknesses of the specimens

Specimen number	Size of the specimen	Ni thickness	WC-Co thickness
1	$2 \times 30 \times 110$ mm	20 μm	190 μm
2	$2 \times 30 \times 110$ mm	40 μm	210 μm
3	$2 \times 30 \times 110$ mm	60 μm	190 μm
4 (Ref)	$2 \times 30 \times 110$ mm	0 μm	220 μm

Table 2 Results of the salt spray test

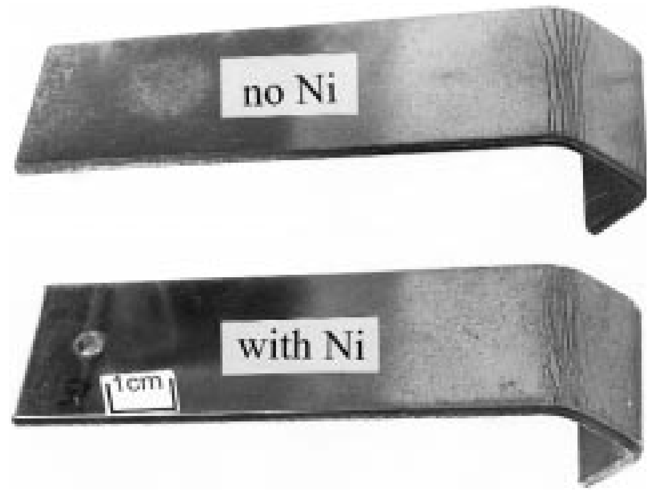
Specimen number	Number of pitting holes
1	0
2	20
3	40
4 (Ref)	∞

by this specific torch. The hydrogen flow rate was 600 L/min, the oxygen flow rate was 230 L/min, and the spraying distance was 200 mm. While being sprayed, the specimens were standing on a table and the traverse speed was 300 mm/s. The thermal spray coating was applied onto an as-plated nickel coating. The reference plate was sand blasted before spraying, and cooling air was applied to the front of specimens to maintain a low surface temperature during the spraying.

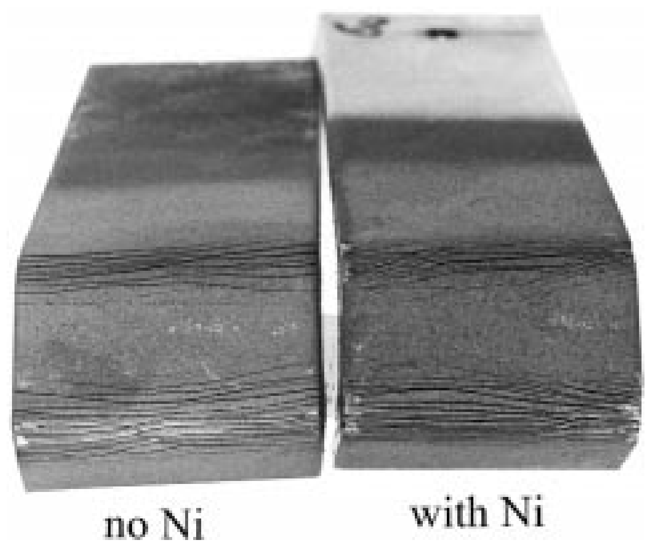
The microstructure of the nickel layer was measured using X-ray diffraction before and after the thermal spray process in order to find any structural differences resulting from the heat flow of the HVOF spraying. The crystal structure was defined by X-ray diffractometry (XRD) using Mo K_α radiation. The measurements of microhardness on these coatings were made according to the ISO 4516 standard "Metallic and related coatings. Vickers and Knoop microhardness tests"[7] by applying a load of 100 g for nickel and 300 g for WC-Co. The microstructure of the coating system was investigated using optical microscopy.

The adhesion between the coatings and the substrate was examined by bending the specimens to angles of 45 and 90°. The behaviors of the specimens were compared visually to each other (Fig. 1).

In order to measure the corrosion resistance, a salt chamber test according to standard ISO 9227[8] was used. The test was made using an acetic acid salt spray test (AASS) procedure. The specimens were tested with the acetic acid salt spray test. The duration of the test was 8 h, and afterward, the specimens were taken from the chamber for examination.



(a)



(b)

Fig. 1 Specimens after bending test. (a) Specimen size and bending angle. (b) Details of bent surfaces

3. Results and discussion

3.1 X-ray Diffraction

The as-deposited nickel has exhibited amorphous microstructure showing only pure nickel (Fig. 2a). The matrix of the deposited nickel from the electroless bath always contained some phosphor, and this would account for an increase in the corrosion resistance. Because the phosphor content is lower than 5%, the diffraction spectra do not show any traces of phosphorous. The diffraction spectra of the coating after the thermal spraying process show the transformation of the amorphous structure (NiP) to crystalline nickel with Ni_3P precipitate. As mentioned above, similar transformations occur by annealing as-deposited electroless nickel at a temperature of more than 300 °C. It seems that is also sufficiently high to cause a microstructural transformation.

3.2 Microhardness

The microhardness of as-deposited electroless plated nickel coating is about 550 HV and its microstructure is amorphous. By heat treatment at, for example, 400 °C, the microhardness can increase to 1000 HV. This arises from transformation of the amorphous structure to a crystalline structure in which there is precipitation of nickel phosphide (Ni_3P_2).[3]

The hardness coating of the thermal sprayed coating was measured and the electroless plated nickel was measured (Table 3). The microhardness of the nickel specimens is between 790 and 890 HV0.1. These results support the XRD analysis; *i.e.,* the specimen structure is crystalline.

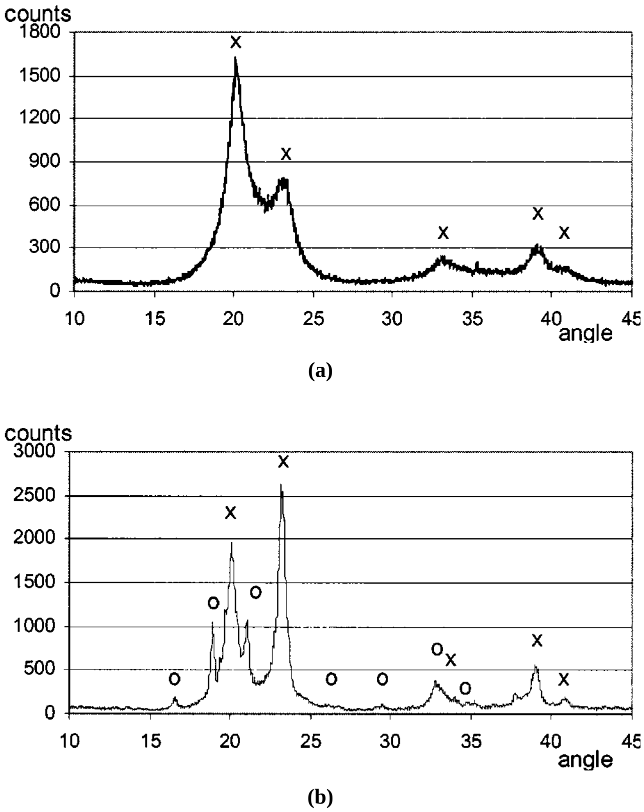


Fig. 2 X-ray diffraction spectra of electroless plated nickel: (a) as-plated and (b) after HVOF spraying with WC-Co top layer

3.3 Adhesion

A simple bend test was performed for each specimen. The test plates were bent to an angle of 45°, and then 90°. The adhesion of the coating was determined according to the degree of coating spallation from the surface of the bent area (Fig. 1).

No spalling was observed on the bent area of the nickel plated or reference plate while bending to an angle of 45°. On the other hand, while bending to an angle of 90°, spalling takes place on the edges of the plate where the stress distribution is more complex. There were as many cracks on the surface of the nickel plated specimens as on the reference plate. These results indicate that the adhesion of tungsten carbide coating on the nickel plated surface is as good as it is on the sand blasted carbon steel surface.

3.4 Microstructure

Optical microscopy showed that in all specimens the tungsten carbide-cobalt coating was porous and had cracks parallel to boundaries (Fig. 3 to 6). Also, more cracks were evident when the nickel layer thickness was increased. In our case, 20 μm was the optimal thickness of the nickel layer that showed no cracks (Fig. 3). If the nickel layer is more than 20 μm, then more cracks are created during the thermal spray process due to crystallization of the nickel. These cracks may penetrate though the HVOF sprayed coating to the substrate surface (Fig. 4 and 5) and can reduce the corrosion behavior of the coating system.

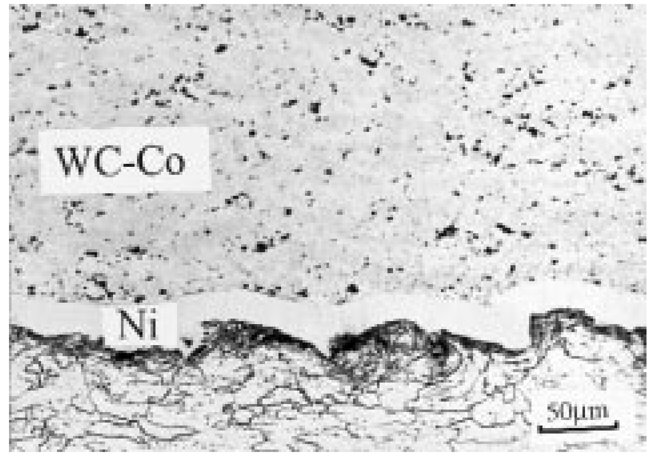


Fig. 3 The microstructure of specimen 1

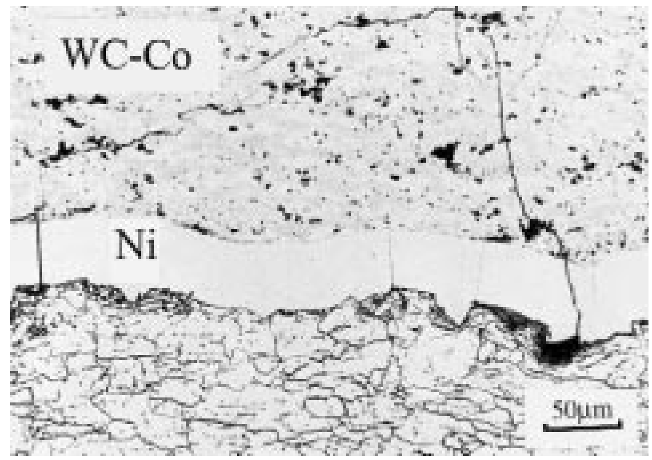


Fig. 4 The microstructure of specimen 2

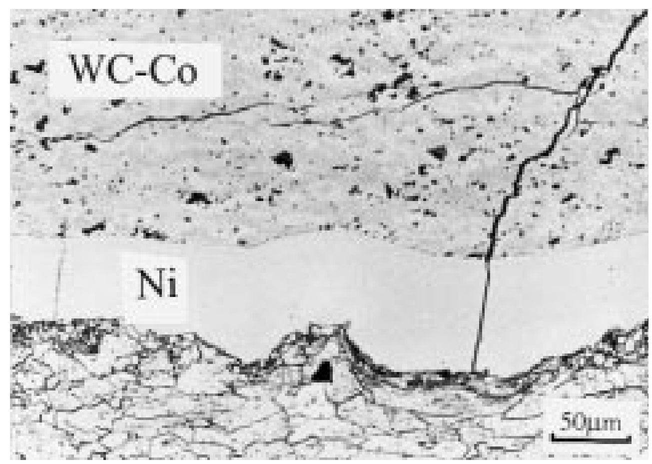


Fig. 5 The microstructure of specimen 3

3.5 Salt Chamber Test

During the AASS test, corrosion occurs rapidly on the reference coating. The duplex coating with the 20 μm underlayer (electroless nickel) showed no pitting. The specimens with 40 and 60 μm

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Table 3 Average hardness of the coatings

Microhardness	Specimen 1	Specimen 2	Specimen 3	Specimen 4
Nickel AVR	786 HV0.1	869 HV0.1	893 HV0.1	. . .
STDEV	92	52	73	. . .
WC-Co AVR	1321 HV0.3	1266 HV0.3	1304 HV0.3	1298 HV0.3
STDEV	98	118	111	98

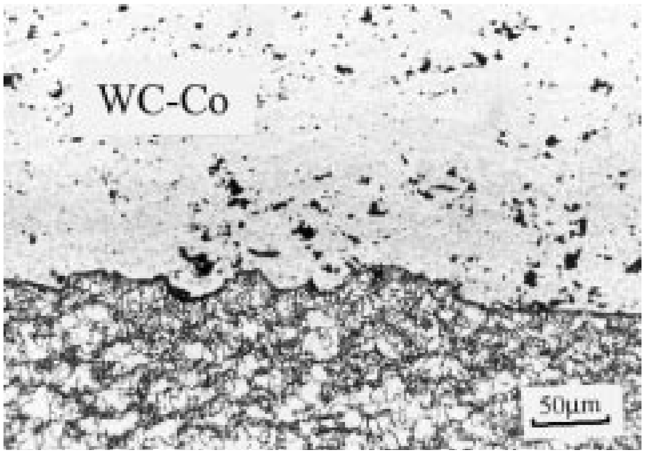

Fig. 6 The microstructure of specimen 4

underlayers, however, exhibited many pitting holes on their surface (Table 2). The poor corrosion resistance of these coatings is due to the cracks observed after the thermal spraying. These cracks allow media to penetrate into the substrate material.

4. Conclusions

Electroless nickel plated carbon steel specimens were thermal sprayed by the HVOF process. The microstructure, microhardness, corrosion resistance, adhesion of the duplex coating of electroless nickel, and HVOF sprayed tungsten carbide-cobalt were examined. Three thicknesses, 20, 40, and 60 μm, of electroless plated nickel were used under the thermal sprayed coating. A specimen without electroless nickel was examined as a reference.

The results of the X-ray diffraction and microhardness tests of the electroless nickel layer after thermal spraying show that the microstructure of the nickel layer is crystalline with Ni_3P precipitations. This behavior indicates that the heat treatment generated by the HVOF spray process can change the amorphous nickel to a crystalline character.

This work shows that it is possible to produce duplex coatings of electroless plated nickel and thermal sprayed cemented carbide without cracks or reduction of adhesion. In order to succeed in producing such coatings, the thickness of electroless nickel must by less than 20 m and the operator of the HVOF process must take care to provide sufficient cooling of the substrate surface. These duplex coatings can confer good corrosion and wear resistance to the substrate material.

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