Preparation research of Nano-SiC/Ni-P composite coating under a compound field

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Abstract. In this paper, the preparation process of Ni-P-SiC composite coatings on 45 steel surfaces with the assistance of magnetic and ultrasound fields was researched. The influence of external field on the surface morphology and performance of the composite layer is also discussed. Experimental results showed that when prepared under magnetic and ultrasonic fields, composite layers are significantly more dense and uniform than coatings made without external fields. Nano-SiC particles, dispersed uniformly in the layer, significantly improve the hardness of the composite layer, and the composite layer under the external field had the highest hardness at 680 HV. The external fields can also accelerate deposition and increase the thickness of the layer. Compared to layers processed without the assistance of external fields, the thickness of the layers increased by nearly ten μm.

1. Introduction

With the addition of nanoparticles [1-5], the wear resistance, hardness, corrosion resistance and other properties of electroless composite coatings (ECCs) have been improved [6-9]. The preparation process of nano ECCs has therefore become one of the research hot spots in this field [10-14]. An amorphous Co-Ni-B-Ce coating with fine grains was prepared by Xuan et al. [10] under the condition of a magnetic field. This coating showed an increased ability to resist deformation as well as improved toughness and wear resistance. Fan et al. [11] improved the deposition rate, grain refinement, and inhibition of nanoparticle aggregation of layers by using an ultrasonic assisted preparation method. As a result, the hardness and corrosion resistance of the coating were significantly improved. A smooth and flat Ni-P/SiC coating surface of low porosity and a fine cellular structure was prepared by Wang et al. [12] by utilizing ultrasonic mechanical stirring composite preparation technology. Even so, there have been few studies to date on fabrication technology under an extra field for ECCs.

Because of its excellent corrosion and erosion resistance properties, SiC is also a useful kind of electronic material. Therefore, nano-SiC particulates are commonly used as reinforcement phases for

electroless deposited Ni-P alloys, and the fabrication and performance of electroless Ni-P-SiC composite coatings have been widely investigated [2, 15-16]. In the present work, nano-SiC/Ni-P coatings on the carbon steel surface prepared with the assistance of an ultrasonic and magnetic field were studied. Based on micro-morphology observation, structure analysis, and thickness and micro-hardness determination, the role of the compound field in the preparation process is summarized.

2. Experimental process and characterization methods

2.1. Experimental

45 steel matrix samples were polished and placed in 15% HCl solution for l min. The plating solutions were made according to the formula in Table 1 with a pH value of approximately 10. Then, the solutions were stirred for 5 min by an ultrasonic generator. Finally, the samples were processed at 25 ℃ under the conditions of Table 2.

Table 1. Solution composition of electroless nano-composite plating.

Reagent name	Content(g/L)
$NiSO_4 \cdot 7H_2O$	25
$NaH_2PO_2 \cdot H_2O$	25
$NH_3 \cdot H_2O$(ml/L)	22
$Na_4P_2O_7 \cdot 10H_2O$	50
20 nm-SiC	5

Table 2. Fabrication processes of different samples.

Sample number	SiC	Magnetic field	Ultrasonic field	Process time
1#	-	-	-	1 h
2#	5g/L	-	-	1 h
3#	5g/L	7.5T/m	-	1 h
4#	5g/L	7.5T/m	-	1.5 h
5#	5g/L	7.5T/m	40 Hz, 100 W	1 h
6#	5g/L	7.5T/m	40 Hz, 100 W	1.5 h

2.2. Characterization methods

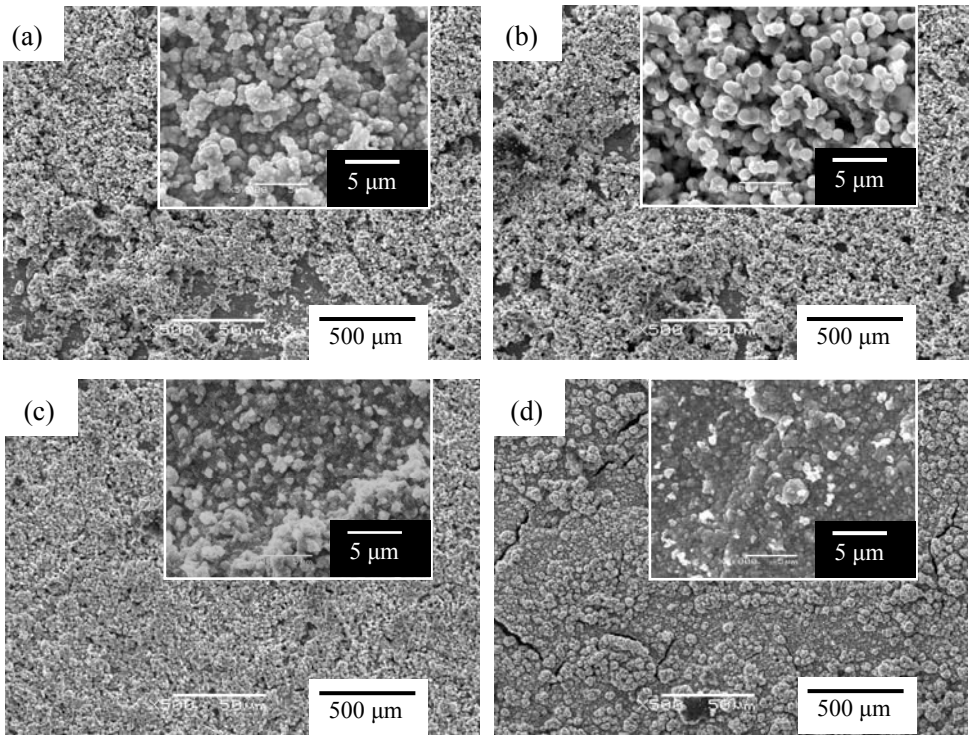
The coating samples' micro-morphology was observed by SEM (JSM-6460lv). The ECCs' composition was analysed by EDS (GENESIS 2000XMS60). Also, the phase was detected by XRD (UltimaIV) with the scanning angle from 10 DEG to 90 DEG, scanning step 0.02 DEG and scanning speed 1.2 DEG/min. The thickness of the samples was also measured by TT206 coating thickness

tester. The hardness was measured with an hvs-1000 digital micro hardness meter under a holding pressure of 100g for 10s.

3. Results and analysis

3.1. Microstructure of the coating samples

Figure 1 shows SEM photos of the coating samples. Figure 1 (a) and (b) indicate that the surfaces of sample coatings 1# and 2# are incompact microstructures. From the high magnification SEM photos in Figure 1 (a) and (b), it also can be seen that the surface coating of the 2# sample has a nonuniform and loose particle aggregation state with many gaps, which results in the coating surface being very rough. On the other hand, from the high magnification SEM photos in Figure 1 (c), (d), (e), and (f), we can see that with the addition of the different extra fields, the surfaces of the coating samples became more and more continuous and compact. The coating surfaces of samples 3# and 4# were denser than sample 2#, and the surface morphologies of 5# and 6# were more compact and smooth as well. This also shows that the extra ultrasonic and magnetic field played a very important role in the coating deposition and was helpful in forming more compact and flat nano-composite coatings.



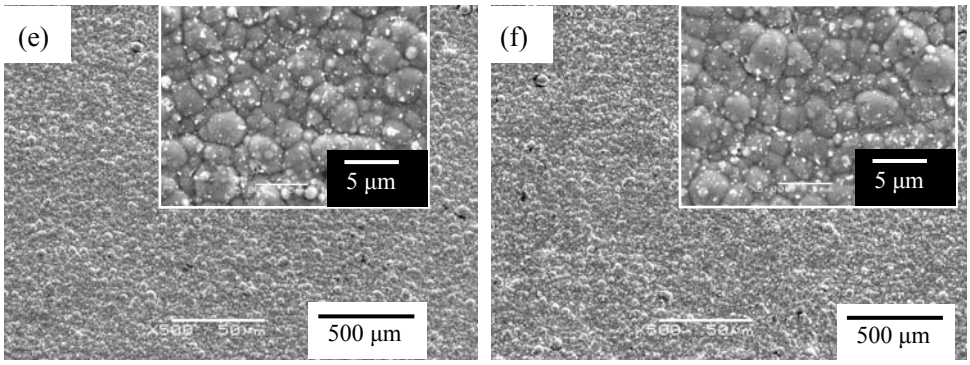


Figure 1. SEM morphology of coatings
(a) 1#, (b) 2#, (c) 3#, (d) 4#, (e) 5#, (f) 6#.

3.2. Composition and phase of the coating samples

Because samples 3# and 4# and samples 5# and 6# differed only in deposition time, only samples 1#, 2#, 3# and 6# were selected as targets for EDS and phase analysis.

Figure 2 shows the EDS analysis results of the samples made under different preparation conditions. From Figure 2 (a), it is clear that no nano-SiC particles existed in the 1# sample, and the coating composition was mainly dominated by Ni and P. From Figure 2 (b), (c), it is clear that the nano-SiC particles added into the solution deposited in the coating with the Ni, P elements, and the deposition of Ni, P element remained mostly unchanged. Thus, it may be concluded that the added SiC particles, just as an ideal nucleation point for Ni-P do not affect the deposited ratio of the Ni, P elements. Figure 2 (d) shows the EDS analysis results of the high magnification SEM morphology on Figure 1 (d). The Fe element signal is from the matrix.

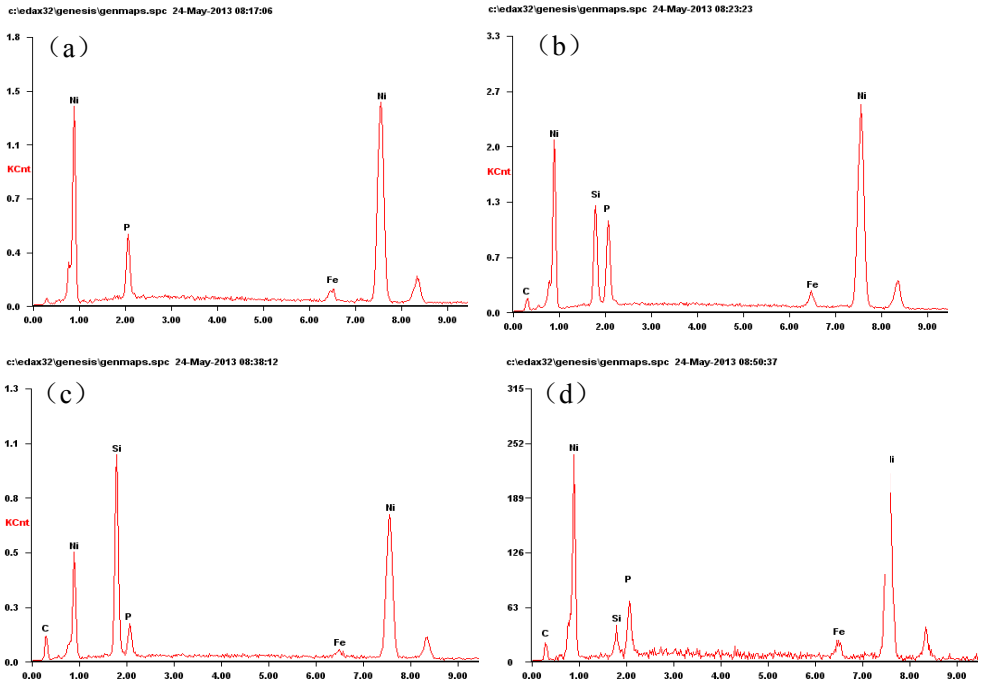


Figure 2. EDS analysis results of the coatings (a)1#, (b)2#, (c)3#, (d)6#.

Figure 3 displays the XRD analysis results of the coatings prepared under four different conditions. In the same way, it can be seen from the XRD result of the 1# sample that there were no SiC particles. On the other hand, the results of the 2#, 3#, 6# sample show diffraction peaks of the SiC phase (JCPDS-ICDD: 00-001-1118), which complies with the experimental conditions. According to JCPDS-ICDD: 00-003-0953, the peaks at 35.351˚, 44.613˚, 57.392˚ and 72.721˚ can be ascribed to the Ni_2P phase. Compared with the 1# sample, the position of the Ni-P's peaks of 2#, 3#, 6# samples do not change much, which indicates that the addition of SiC particles did not change the crystallization behavior of the Ni-P alloy, and that the intensities of the Ni_2P phase diffraction peaks were higher than those of the 1# sample. This clearly indicates that the addition of the external fields has contributed to the crystallization of Ni-P. The diffraction peaks of Fe (JCPDS-ICDD: 00-001-1252) in the samples derive from the matrix.

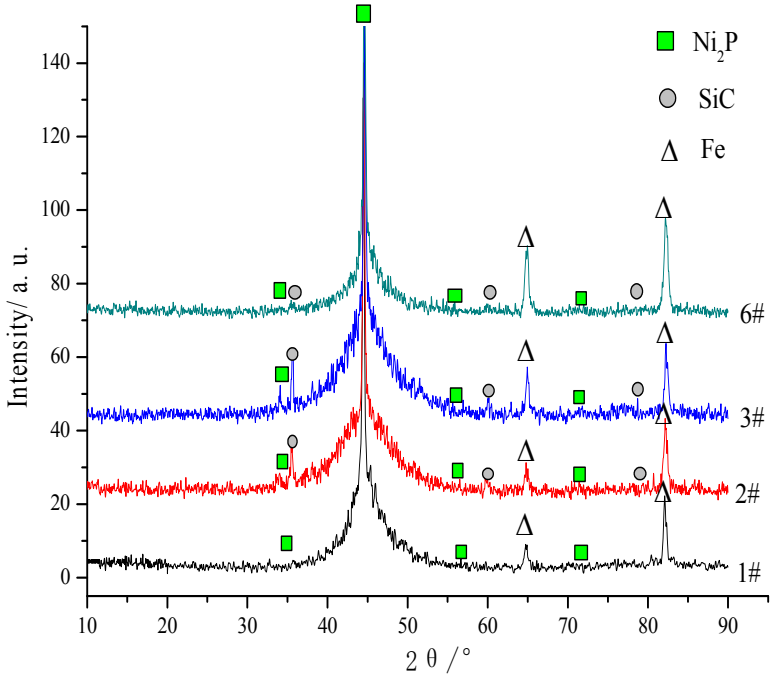


Figure 3. XRD spectra of the coatings.

3.3. Thickness of the coating samples

Figure 4(a) shows the average thickness values of the coating samples. It is clear from Figure 4(a) that the thickness values of the coatings underwent an increasing trend from 17.2 μm to 29.6 μm. Compared with the thickness value of 2# coating, the thickness values of the 3#, 4#, 5#, 6# samples all increased. This result shows that the addition of the external fields not only accelerated the deposition of the nano-composite coatings, but also thickened the composite coatings. It can also be found from the thickness values of 3# and 4# samples that the coatings' thicknesses increased as the process time was extended.

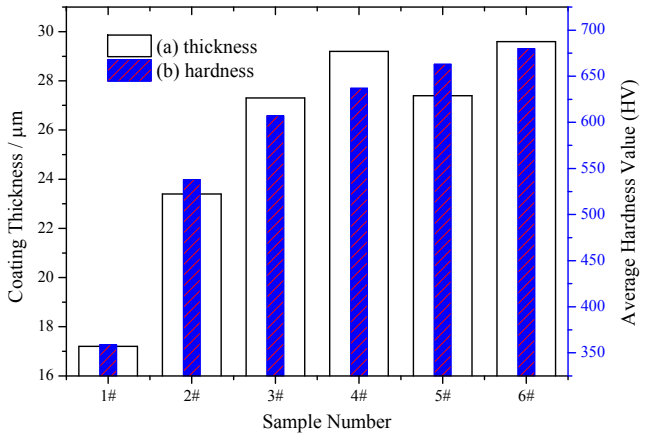


Figure 4. Average thickness and hardness values of coating samples
(a) average thickness values, (b) average hardness values.

3.4. Hardness of the coating samples

Figure 4(b) shows the mean micro-hardness values curve of the different coating samples. All data were averaged over the selected five dispersed points on the each coating's surface. In Figure 4(b), it can be seen that the Victorinox hardness values of the 2#, 3#, 4#, 5# and 6# samples were significantly higher than the value of the 1# sample. In particular, the hardness difference between the 1# and 2# samples was near 200 HV, which shows that the addition of nano-SiC can significantly improve the hardness of the coatings. By this method, the wear resistance of the nano-composite coatings can also be greatly improved because the SiC particles themselves are hard particles. Also, the exotic SiC particulates improve the ability of the plastic deformation of the Ni-P matrix. As the main point of contact in the friction process, the protruding SiC particles bear the loading and shear force. Thereby, the wear abrasion of the composite coatings will be reduced. At the same time, the hardness values of samples 5# and 6# were higher than those of samples 3# and 4#. These results not only illustrate that the thicker the coatings are, the greater the hardness value and wear resistance are, but they also indicate that the hardness values correspond to the uniformity and compactness of the composite coatings.

4. Conclusions

In this work, nano-SiC/Ni-P coatings were prepared with the help of a magnetic and ultrasonic compound field. Then, the surface micro-morphology and microstructure of the composite coatings were observed and analyzed. The thickness and micro-hardness of the coatings were also measured. Therefore, the following conclusions can be drawn.

1) The surface density and flatness of the composite coatings that were prepared under the conditions of an external magnetic and ultrasonic field were better than that of the composite coatings prepared without any external field.

2) The composite coatings were mainly composed of Ni_2P and SiC phases.

3) The compound field can effectively promote electroless deposition and increase the thickness of the coatings. The thickness of the composite layer made under the external field showed an increase of nearly ten μm.

4) The addition of nano-SiC significantly increased the micro-hardness of the composite coatings. The maximum micro-hardness value of nano-SiC/Ni-P coating reached nearly 680 HV.

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