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Preparation and Micro Mechanical Properties of Nano-Al2O3 Particles Strengthened Ni-based Composite Coatings

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Abstract

Ni-based composite solution containing nano-Al2O3 particles was prepared by high-energy mechanical and chemical processes. The microstructure and nano-particle content of nano-Al2O3/Ni composite coatings were determined by SEM, EDS and TEM. The micro mechanical properties were tested by nano-indentation technique, and the strengthening mechanism was analyzed. The results show that 85 percent of particles in the solution are dispersed in size of nano meter, nano-particles co-deposited in the coating increases by a factor of 53 percent and the structure of the composite coating is more compact and uniform than that of Ni coating. Nano-Al2O3/Ni coatings exhibit excellent micro mechanical properties, the nanohardness and Young's modulus are 7.04 GPa and 225 GPa respectively, which are attributed to finer crystals strengthening, dispersion strengthening and high-density dislocations strengthening.

Keywords: nano-particle composite coating; nanoindentation; micro mechanical property; strengthening mechanism

1. Introduction

With a newly developed nanomaterials, ranging in size from 1 to 100 nm available, nano- particles strengthened composite coatings are increasingly attracting scientific and technological interest by virtue of their high hardness, low friction coefficient and superior wear resistance properties. While the dispersion and suspension of nano-particles in the electrolyte and the co-deposition of nano-particles with composite coatings have been being an intractable problem, and the former properties researches were focused on the macro mechanical properties of composite coating (Ding H Y, Chen Z G, Chen C F, 2003;Du L Z, Xu B S, Dong S Y,et al, 2004).the surface and near-surface micro mechanical properties of the nano-composite coatings, which can be critical to their final

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performance, have not been evaluated previously. The rapidly expanding field of depth-sensing nano-indentation technique provides a quantitative method for mapping the micro mechanical properties, such as nanohardness and Young's modulus, of the surface/near-surface region(Wang D G， Zhang S W， Gao M L, 2003;Zheng X.J, Zhou Y.C, Liu J.M, et al, 2003;Kirsten I. Schiffmann, Andreas Hieke, 2003).

In the present study, Ni-based composite solutions containing nano-Al2O3 particles were produced by high-energy mechanical and chemical processes, then nano-Al2O3/Ni composite coatings were prepared on steel by brush plating, and nanoindentation was used to test the micro mechanical properties, including nanohardness and Young's modulus, of the composite coating, the strengthening mechanism was discussed.

2. Experiments

2.1. Sample preparation

Prior to the brush plating, the required quantity of nano-Al_2O_3 particles with average grain size of 30 nm (provided by the Institute of Process of the Chinese Academy of Sciences), were added to typical nickel electrolyte with a special dispersant and treated sufficiently by high-energy mechanical and chemical processes (HMC)(Xu B S, China patent, No.02101196.6). By this method the compound solution in which nano-particles suspended steadily was prepared. The composite coating was brush-plated on mild steel substrate abraded with waterproof paper (800 grit) at a voltage of 12 V, and a relative velocity between the negative and positive poles, of 10 m/min. The coating thickness was about 0.1mm.

Particle size distribution in the compound solution was measured by Mastersizer S laser particle-size analyzer with a lower limit of 50 nm. The surface morphologies of the coatings were examined on a JSM-840 scanning electron microscope (SEM). A H-800 transmission electron microscope (TEM) was employed to analyze the microstructure of the coatings.

2.2 Nanoindentation

The Nano Test 600 was used for the indentation testing. A Berkovich diamond indenter was used throughout and the area function was calibrated by indentations into fused silica. All indentations were load controlled at 15 mN maximum load. Other experimental conditions were : (1) preset initial load : 0.03 mN； (2) loading rate=unloading rate : 0.3 mN s^{-1}； (3) holding period at peak load : 60s. All data were corrected for thermal drift and analyzed with Oliver and Pharr method(Oliver W C, Pharr G M, 1992).A grid of 10 × 10 indentations were produced on the coating surface polished in order to reveal the differences in mechanical properties through the coating. Indentations were spaced sufficiently far apart so that the indentation behavior was not affected by adjacent indentations.

3. Results and Discussion

3.1. Particle size distribution in the compound solutions

Fig.1 shows the particle size distribution of nano-Al_2O_3 in the solutions before and after treated by HMC, the contents of nano-Al_2O_3 are both 20g/L. It can be seen that the nano-Al_2O_3 particles in the untreated solution has wide scope of distribution, and most are in conglomeration with size of over 100 nm. However, in the treated solution, the nano-Al_2O_3 particles are scattered well and over 85% are in size of less than 100 nm. Therefore, high-energy mechanical and chemical processes can effectively disperse and prevent conglomeration of the nano-Al_2O_3 in the Ni-based electrolyte.

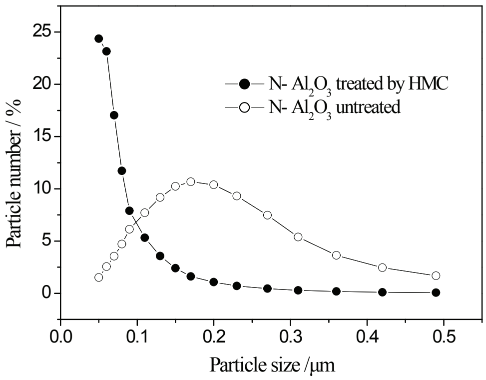

Fig.1 Particle size distribution of nano- Al_2O_3 in the composite solutions

3.2. *Microstructure and particle content of the composite coatings*

Fig.2 depicts the surface morphologies of nano-Al_2O_3/Ni composite coating and Ni coating. It can be seen that both morphologies show typical nodular units composed of many grains, while the surface of the composite coatings is more compact and uniform than that of Ni coating. The higher nucleation, due to nano-Al_2O_3 incorporation, perturbs the growth of nickel matrix and results in a finer and denser microstructure, which is beneficial to improve the properties of the composite coatings.

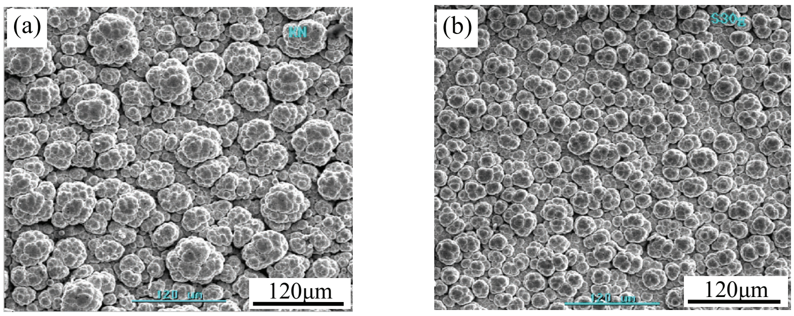

Fig.2 SEM morphologies of Ni coating (a), n-Al_2O_3/Ni composite coating (b)

Fig3 shows the microstructure of nano-Al_2O_3/Ni composite coating. It reveals that the nano-particles are homogeneously distributed and well bonded with Ni matrix with the size of less than 40nm,which is resulting from the selection effect of the deposition of composite coating.

The total amount of nano-particles in the coating treated by HMC is about 2.6 wt%, higher 53% than that of untreated. Although the content does not exceed 3wt%, the nano-particles are uniformly scattered in the coating and the particle sizes are super small, therefore it is sufficient to significantly improve the microstructure and properties of the composite coatings.

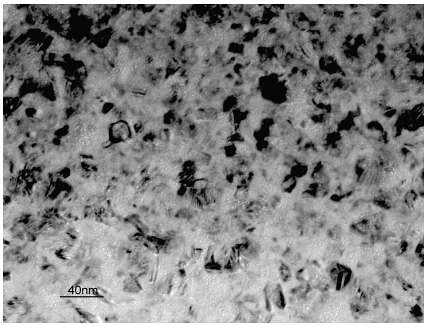

Fig.3 TEM morphology of n-Al_2O_3/Ni coating

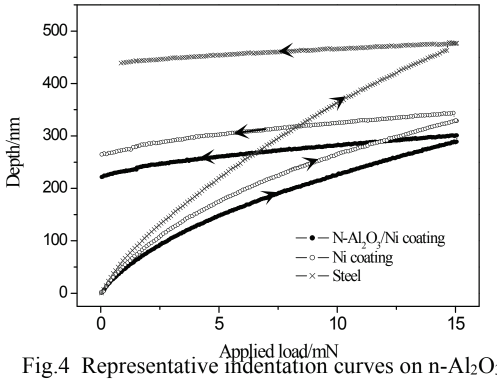

Fig.4 Representative indentation curves on n-Al_2O_3/Ni composite coating, Ni coating and steel

3.3. Nanohardness and Young's modulus of the composite coatings

The typical Load-Depth curves of the nano-Al_2O_3/Ni composite coating, Ni coating and the steel are shown in Fig.4. It can be seen that the composite coating shows much higher resistance to indentation than steel and the Ni coating. Table 1 shows the mean values and standard deviations of hardness and modulus. The data are collected from 100 indentations including those on or near defects in the coating or steel. The average nanohardness and Young's modulus of the composite coatings are 7.04 GPa and 225 Gpa, which are higher 1.2 GPa and 21 Gpa than Ni coating respectively.

Table 1. Distribution of hardness and modulus values of steel, Ni coating and n-Al_2O_3/Ni coating

Material	Hardness H/GPa	Reduced modulus E_r/GPa	Modulus E_s/GPa
Steel	2.70±0.38	185.4±19.6	204
Ni coating	5.80±0.52	188.6±15.4	204
n-Al_2O_3/Ni coating	7.04±0.69	204.7±12.6	225

Fig.5-6 show the maps of hardness and modulus from the grid of indentations on the composite coating. It can be seen that the composite coatings have consistent mechanical properties with the exception of a few areas with low hardness and modulus. These areas are clearly visible in the pores and cracks of Fig.7. Additionally, there are some regions of high hardness and elastic modulus, these indentations may be on or near the nano-particles. To optimize the technique and reduce the defects is therefore the critical factor of improving the over-all properties of nano composite coatings.

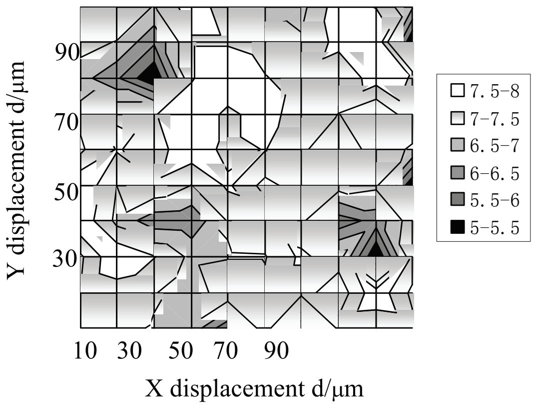

Fig.5 Map of hardness (GPa) of n-Al_2O_3/Ni versus position

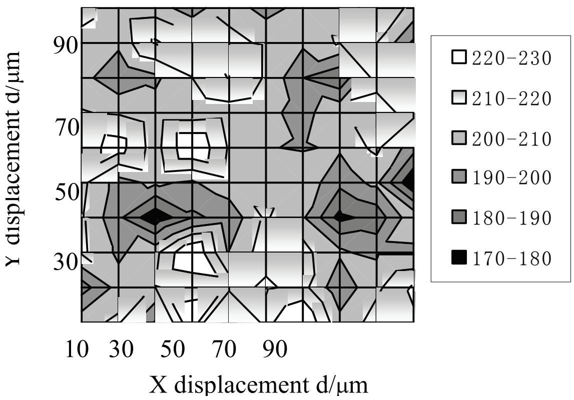

Fig.6 Map of reduced modulus (GPa) of n-Al_2O_3/Ni versus position

3.4 Discussion

The higher micro mechanical properties of the composite coating are due to the finer surface morphology, more compact structure and also as a result of homogeneously distribution of nano-Al_2O_3 particles. On one hand, the nano-particle surfaces with special high activity supply more nucleuses for the deposition of Ni atoms, which leads to thinner grains and denser surface morphology. The finer surface can increase the load-carrying areas and improve the indentation resistance of the composite coating. On the other hand, the nano-Al_2O_3 particles possess superior hardness, which can hinder the migration of dislocations during the deformation. Furthermore, as the nano-particles are compounded to the coating, the crystal defects, such as twins and layer dislocations, increase significantly (Fig.8), which interact with nano-particles and prevent the movement of dislocations ulteriorly(Dong S Y, Xu B S, Ma S N, et al, 2004). Consequently, the composite coatings exhibit excellent mechanical properties owing to the finer crystals strengthening, dispersion strengthening and high-density dislocations strengthening.

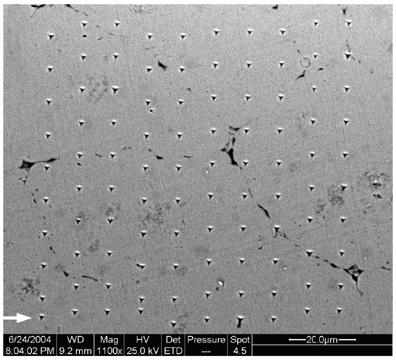

Fig.7 SEM morphology of indentations

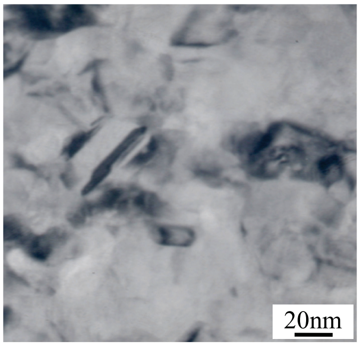

Fig.8 TEM morphologies of twin grains and layer dislocations in n-Al_2O_3/Ni coating

4. Results

(1) The high-energy mechanical and chemical processes can effectively disperse and prevent conglomeration of the nano-Al_2O_3 in the Ni-based electrolyte, and 85 percent of particles in the solution are dispersed in size of less than 100 nm.

(2) The composite coatings possess much finer and more compact morphologies than Ni coatings. Nano-particles are homogeneously distributed and well bonded with Ni matrix in the size of less than 40nm.

(3) Nano-Al_2O_3/Ni coatings exhibit excellent micro mechanical properties, which are attributed to finer crystals strengthening, dispersion strengthening and high-density dislocations strengthening.

Acknowledgements

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References

Ding H Y, Chen Z G, Chen C F, 2003. Abrasive wear resistance of composite coating dispersedly reinforced by nano-Al_2O_3 [J]. Materials for mechanical engineering, 27(4): 38-40

Du L Z, Xu B S, Dong S Y,et al, 2004. Study of tribological characteristics and wear mechanism of nano-particle strengthening nickel-based composite coatings under abrasive contaminant lubrication [J]. Wear, 257: 1058-1063

Wang D G，Zhang S W，Gao M L, 2003. Nano-scratch study of molecular deposition films on silicon wafers using nano-indentation [J]. Tribotest journal, 9（3）：231~237

Zheng X.J, Zhou Y.C, Liu J.M, et al, 2003. Use of nanomechanical fracture-testing for determining the interfacial adhesion of PZT ferroelectric thin films [J]. Surface and Coatings Technology,176：67~74

Kirsten I. Schiffmann, Andreas Hieke, 2003. Analysis of microwear experiments on thin DLC coatings： friction, wear and plastic deformation[J]. Wear, 254: 565~572

Xu B S, China patent, No.02101196.6

Oliver W C, Pharr G M, 1992. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments [J]. J Mater Res., 7(6): 1546~1583

Dong S Y, Xu B S, Ma S N, *et al, 2004*. Properties and strengthening mechanism of brush plated nanoparticle reinforced composite coatings [J]. Transactions of Nonferrous Metals Society of China, 14(Special 2): 182-185