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EFFECT OF TWO-STAGE HEAT TREATMENT ON THE PROPERTIES OF Al/NiCr DUPLEX COATING

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Abstract

There is always a presence of characteristic pores and microcracks inside thermal spray coatings that would bring harmful effects on the coatings. Post spray heat treatment aims to stabilize the structure of the coatings. For metallic coating, in dependence of annealing temperature, an intermetallic diffusion between the coatings and the diffusion between the coating and the substrate may happen. This paper presents our study on the microstructure, mechanical properties and erosion corrosion resistance of an electric arc thermal spray Al/NiCr duplex coating on C45 steel substrate after consecutive heat treatments at 600 °C and 950 °C in 15 minutes. The obtained results show positive effects of the heat treatment: the porosity of the coating decreased from 1.99 % to 0.72 %, the adhesion strength increased 8 times, the microhardness varied in a range of 190-800 HV and the erosion corrosion resistance increased 2 times when testing in acidic slurry containing hard particles.

Keywords. thermal spray, NiCr alloy, heat treatment.

1. INTRODUCTION

Thermal spray has been commonly applied in creating metal, ceramic and composite coatings for improving abrasion-corrosion resistance of machine parts used in industries [1]. However, it always exist pores and defects inside the coatings that might bring harmful effects, e.g. reduction in the adhesion and corrosion resistance of the coatings. [2, 3]. Heat treatment is a common method for thermal spray coatings. Its aim is to stabilize the microstructure, to reduce the porosity and improve some functional properties of the coatings.

According to the research of H.L. Huang et al [4] in case of using aluminum coating on NiCr alloy coating after two-stage heat treatment at 600 °C in 1 hour and 900 °C in 0.25 hours, the formation of intermetallic phases of NiAl in the coating structure such as γ'-Ni_3Al, β-NiAl and mixture of β-NiAl + α-Cr phases significantly improved the abrasion resistance of the coating. The results of J.M. Brossard et al [5] also showed that, $NiAl_3$, δ-$NiAl_3$, β-NiAl, γ'-Ni_3Al, Ni (Al) alloys can be formed on NiCr alloy coatings.

The structure and properties of Al / NiCr alloy duplex coating after two-stage heat treatment at 600 °C and 950 °C in 15 minutesby acetylene combustion gas were investigated in this study. During the first the duplex coating was heat treatments at 600 °C, to form intermetallic phases due to aluminum has been diffused into NiCr alloy [6] that increases the hardness of the coating surface and improves the abrasion resistance. The second heatingat 950°C facilitated the interdiffusion of coating layers of NiCr alloys and the steel substrate; hence, the coating adhesion would be ameliorated.

2. EXPERIMENT

2.1. Preparation of NiCr coating

NiCr20 alloy in form of Φ=2 mm wires with the composition (wt%): 79.39 % Ni, 18.16 % Cr, 0.26 % Ti; 0.9 % Si, 0.73 % Mn; 0.56 % Fe was sprayed on the C45 steel cylinder samples with diameter of 30 mm and height of 5 mm. The thickness of the NiCr20 coating was about 1000 μm. Next, we sprayed pure aluminum coating with

a thickness of about 200 μm. An electric-arc thermal spray equipment OSU Hessler 300 A (Germany) was used to fabricate the coatings with the following technical parameters: voltage 33 V; current 200-250 A; compressed air pressure 4.2-4.5 atm; spray distance 150-200 mm.

2.2. Heat treatment

Two-stage consecutive heat treatment at 600 °C and 950 °C with each stage in 15 minutes by acetylene combustion gas was realized for the NiCr20/Al coating samples. After the heating, the samples were let to cool down to room temperature at ambiance and then put sample into storage cabinet before analyzing the properties of the coating.

2.3. Characterization of the coatings

The adhesion of the coating was determined by JIS H8666 standard. The microhardness on the cross-section was measured from the coating surface into the NiCr alloy coating; using the AVK-Co / Mitutoyo hardness measuring instrument with a loading weight of 300 g for 15 s.

The porosity of the coatings was determined by image analysis on the cross-sectional structure according to ASTM B276. The percentage of porosity was calculated by using AxioVision phase analysis software.

The erosion corrosion resistance of the coating is tested in H_2SO_4 acid solution pH=2 containing 3 % SiC solid grain causing motion erosion with the rotational speed of 1040 m/min (4m/s) and a test time of 168 hours [7]. The mass loss during erosion corrosion test was monitored over time with analytical balance of 0.0001 g accuracy. After heat treatment the phase composition on the coating's surface was investigated by X-ray diffraction (XRD) analysis using X-RAY D5005/ SIEMENS equipment of the University of Science - Vietnam National University (VNU). The phase composition analysis of the selected samples was performed as follows: temperature 25 °C, the sweep angle 2θ in a ranges from 10° to 60°, measurement step angle of 0.03°/s, using Cu radiation.

3. RESULTS AND DISCUSSION

3.1. Porosity of coating

The cross-sectional image of the coating before and after heat treatment was shown in Figure 1.

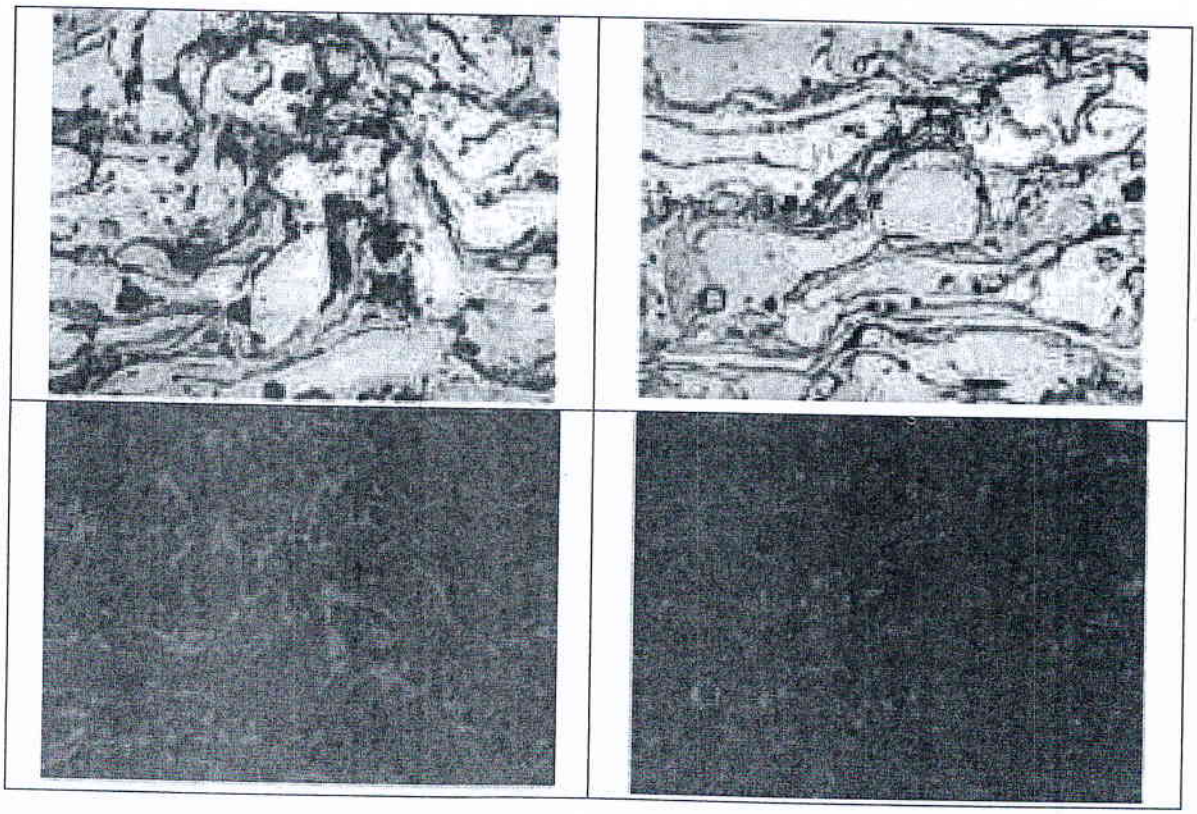

Fig. 1. Optical microscopy micrographs taken on the cross sections of the coating: pores inside the coating (in blue); Grain border (in green); NiCr alloy coating substrate (in red)

After being processed by AxioVision image analysis software, the grain boundary and pore-shaped images in the coating have been shown. Analysis results showed that, after the heat treatment the pores and cracks inside the coating were significantly reduced. The porosity of the coating reduced from 1.99 % to 0.72 % after heat treatment.

3.2.Phase composition

The phase composition on the surface of the coating after heat treatment was analyzed by X-ray diffraction (XRD) method (Fig. 2). The results showed that, on the coating surface, in addition to the aluminum crystalline phase (Al), Al_3Ni intermetallic crystalline phase appeared with relatively high intensity .This proves that after two-stage heat treatment, a large amount of aluminum on the surface of the NiCr alloy interacted with nickel to form Al_3Ni new phase.

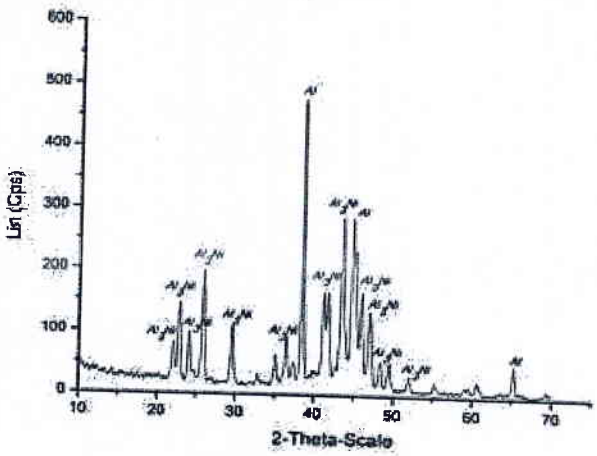

Fig. 2. XRD spectrameasured on the surface of NiCr / Al coating after heat treatment

3.3. Adhesion of the coating

The obtained results of the adhesion measurement between the coating system (Fig. 3) show that, the adhesion of the coating on C45steel substrate prior to the heat treatment was relatively low (8.2 MPa). After heat treatment, the coating's adhesion increased sharply, reached a value of approximately 66 MPaand increased about 8 times in comparison with untreated coating. This proves that, at high treatment temperature, the steel substrate and the coating diffused into each other, forming intermetallic phases of $FeNi_3$to increase the adhesion between the coating and the substrate. The appearance of $FeNi_3$in the boundary between the coating and the substrate was shown recently in the XRD result by Tuan Nguyen Van [7].

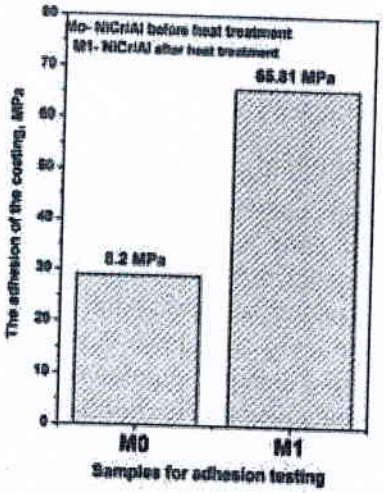

Fig. 3. Adhesion of the coating before (M_o) and after heat treatment (M_1)

3.4. Microhardness

The microhardness was measured on the cross-sectional structure of the coating after heat treatment. Fig. 4a presents the image of the indentation defining the microhardness on the cross-section of the NiCr alloy coating. Fig. 4b is image of the indentations defining the microhardness measured on the cross-section of the coating from the outer surface to the lateral depth and to the interface between the aluminum coating and NiCr alloy coating. The microhardness values of the coating are shown in Table 1.

Table 1 showed that, the microhardness measured on the part of the aluminum coating (about 50 μm from outer surface) was significantly higher than that measured on the other parts of the coating. The highest measured microhardness value is 799 HV, equivalent to 64 HRC. This result shows that, the formation of Al_3Ni intermetallic phase has made the microhardness on the surface of the coating significantly increase compared to the microhardness of the NiCr alloy coating. The coating with high microhardness will have high abrasion and erosion resistance. The results of the erosion corrosion resistance test of the coating are presented in the next section.

3.5. Erosion corrosion resistance

The erosion corrosion resistance of the coating before and after the heat treatment was tested on our self-designed test system. The samples were tested

for 168 hours (1 week). Corrosion resistance of the samples is evaluated by change in surface appearance and mass loss measurement with time. Fig. 5 presents the photos of the sample surface during the test.

According to surface observations, the non-heat-treated coating exhibited reddish spots on the surface after 120 hours of test. However, the aluminum coating remains on the surface of the NiCr alloy coating. For heat-treated samples, red rust spots began to appear on the surface after 168 hours of test. The results show that, the corrosion protection possibility for steel substrate in the acidic environment containing the erosive particles of the heat-treated coating was better than that of the untreated coating. The mass loss of the coating over the tested period was shown in Fig. 6.

The results show that in the first 24 h of test, the erosion corrosion resistance of samples before and after heat treatment was not much different. After 24 hours of test, the erosion corrosion resistance of the heat treated coating sample was higher than that of untreated one. Specifically, the weight loss of the untreated coating was 966.2 mg after 168 h of testing, equivalent to 5.751 mg per hour. Meanwhile, for the heat-treated coating, the weight loss was 441.9 mg, equivalent to 2.63 mg/h. It was lower about two times lower than the one of untreated sample. This can be explained there was formation Al_3Ni on the surface of Al/NiCr coating and simultaneously the porosity of the coating reduced. The results show that the heat treatment process improved the corrosion protection and erosion resistance of the coating.

Table 1. Microhardnessmeasured on the cross-sectional structure of the coating after heat treatment

Location of indentations	Microhardness, HV
Near to outer surface	541
Distance from outer surface: 50μm	799
Distance from outer surface: 100μm	730
Distance from outer surface: 150μm	409
Distance from outer surface: 200μm	577
Distance from outer surface: 400μm (NiCr coating)	181
Distance from outer surface: 420μm (NiCr coating)	190

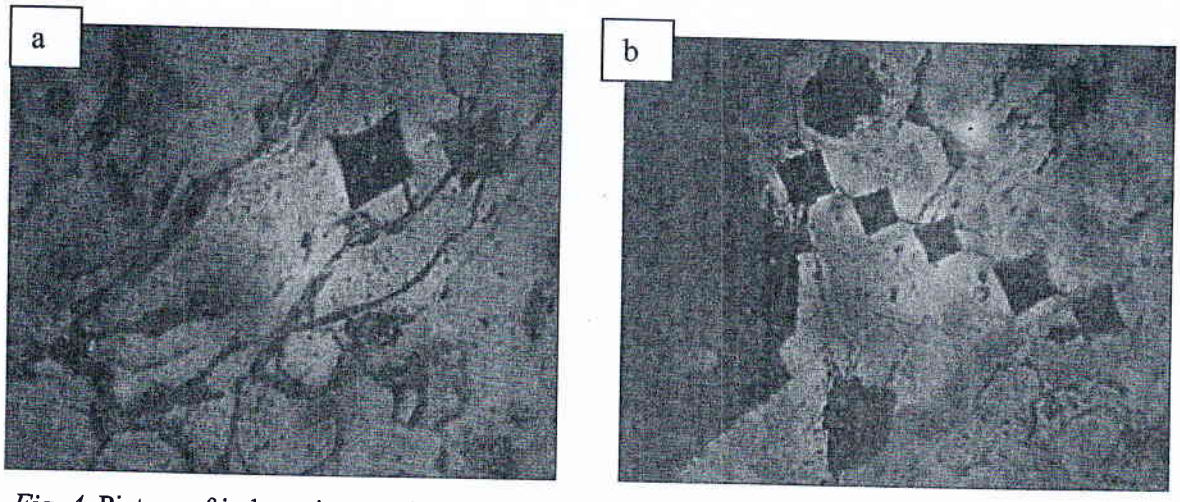

Fig. 4. Picture of indentations to define microhardness on the cross-sectional structure of the coating

a-Measurement on NiCr coating; b- Consecutive measurements from the outer surface towards the steel substrate

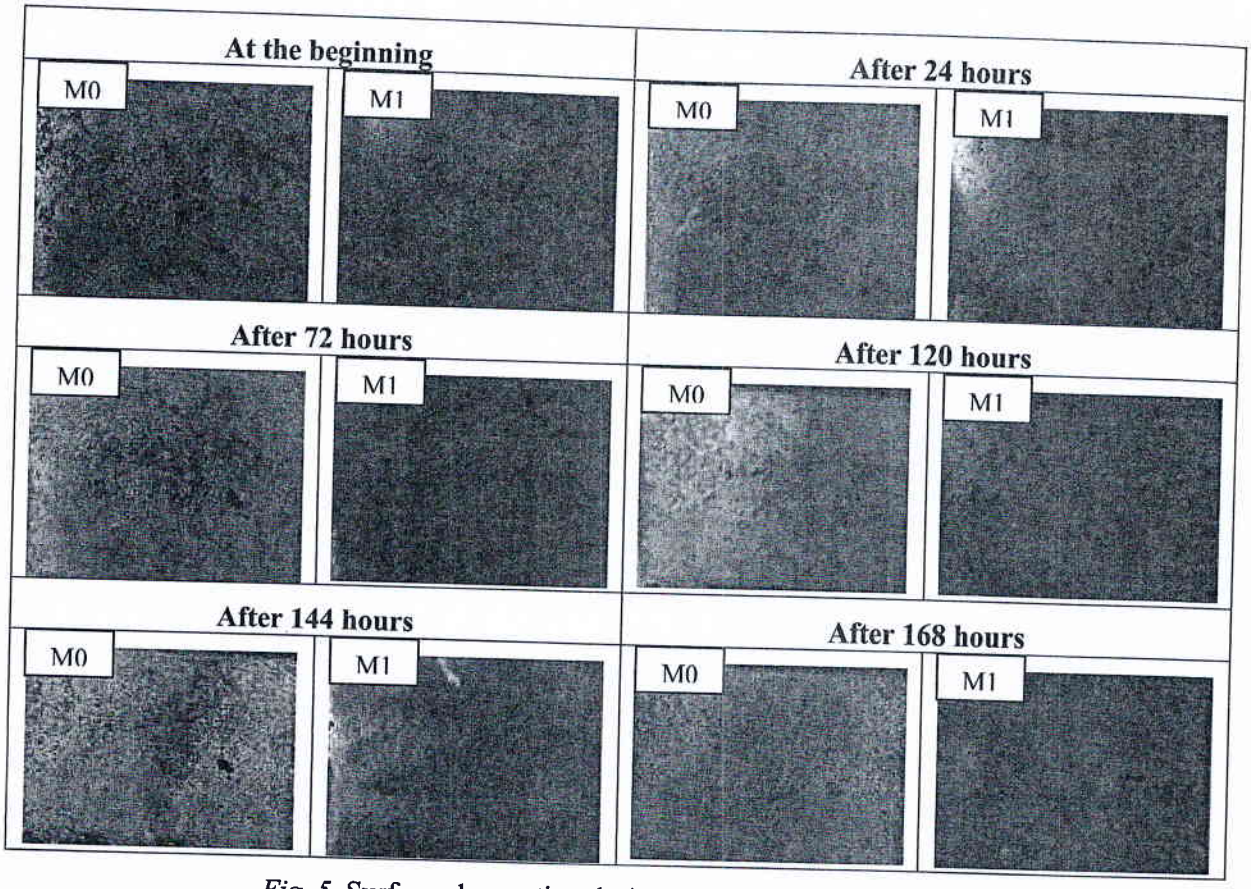

Fig. 5. Surface observation during the erosion corrosion test

M0: the coating before heat treatment; M1: the coating after heat treatment

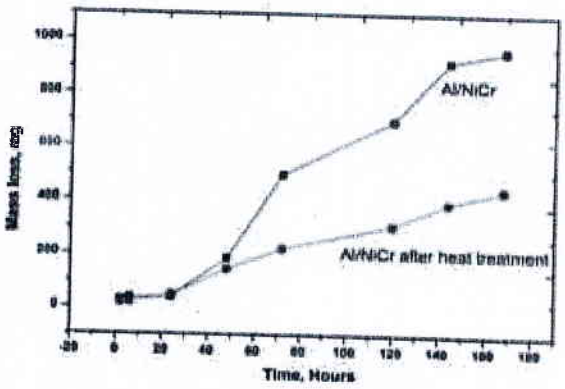

Fig. 6. Mass loss during the erosion corrosion test

4. CONCLUSION

Based on the obtained results, we make some conclusions as follows:

- The porosity of the coating after two-stage heat treatment decreased from 1.99 to 0.77 %.

- After the heat treatment, the intermetallic crystalline phase of Al_3Ni forms on the coating surface with relatively high specific strength peaks, contributing to increase in the microhardness of the coating system, compared to the original NiCr alloy coating.

- The adhesion of the coating on the C45 substrate after the heat treatment increased by 8 times compared with untreated coating from 8.2 to 65.81 MPa.

- After the heat treatment, the erosion corrosion resistance of the coating increased by more than 2 times than that of the untreated coating.

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