

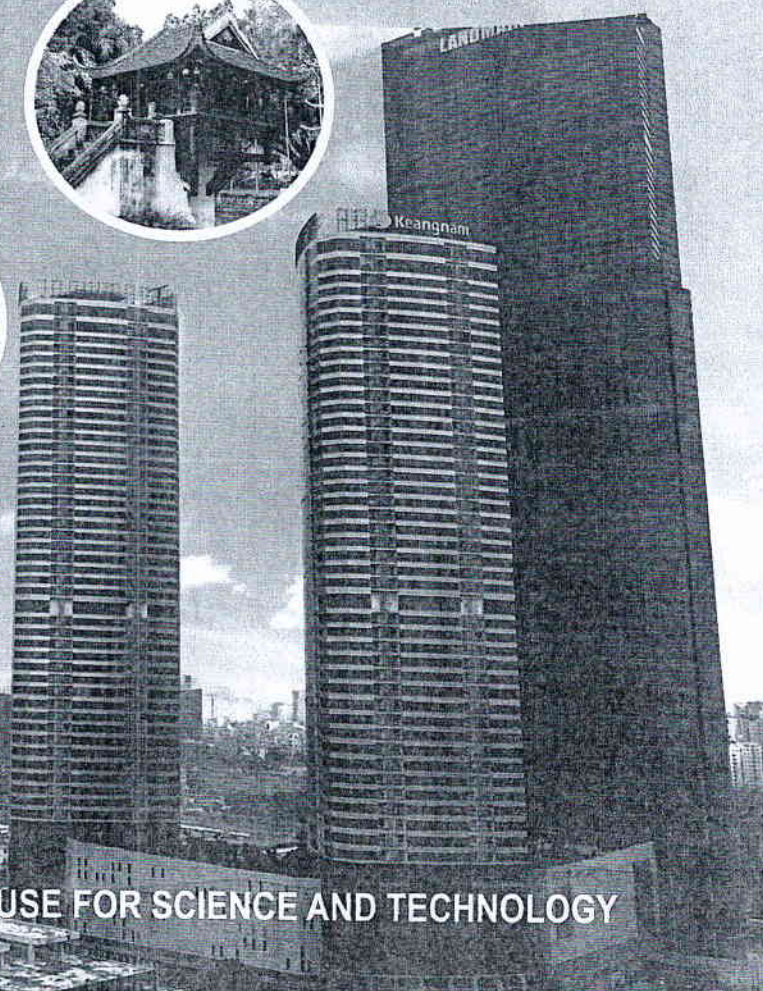
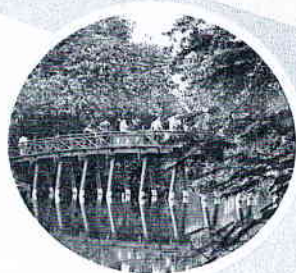


**Proceedings**

**The 6<sup>th</sup> Asian Symposium on Advanced Materials:  
Chemistry, Physics & Biomedicine  
of Functional and Novel Materials**

**ASAM-6**

Hanoi, September 27 - 30<sup>th</sup>, 2017



**PUBLISHING HOUSE FOR SCIENCE AND TECHNOLOGY**

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## INFLUENCE OF ANNEALING ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Al/Ni-20Cr DUPLEX COATING SYSTEM ON STEEL SUBSTRATE

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### Abstract

The electric arc thermal spray duplex Al/Ni-20Cr coating system on carbon steel substrate was heat-treated at different temperatures in a range of 400 - 600 °C during annealing durations of 2 - 8 hours. The cross-sectional microstructure analysis shows a significant reduction of the coating porosity after the heat treatment. The microhardness measurement reveals interdiffusion between the Ni-20Cr and Al layers, as well as between the Al layer and steel substrate. The newly formed intermetallic compounds are characterized by high values of microhardness of 800 - 850 HV that brought a positive effect to increase the wear resistance and to reduce the friction coefficient of the coating system.

**Keywords.** electric arc thermal spray, nickel-chromium alloy coating, aluminium coating, duplex coating, heat treatment, wear resistance.

### 1. INTRODUCTION

Working capability of the machine components depends much on the properties of the surface layer: hardness, abrasion resistance, corrosion resistance heat resistance [1]... In addition, the porosity of the surface, the adhesion between the surface and the substrate material also influences significantly the service lifetime of the machine components [1, 2]. Metallic coatings made using heat-sealing technology are widely applied in the industry for the purpose of protective or restoring coatings. Therefore, the surface layer can be one or more combined layers [3, 4]. The characteristic of bonding between thermal spray coating and base material is mainly mechanical adhesion; therefore, it has low adhesive value. In the coating, there is still residual stress and porosity. Thus, when working in potentially corrosive media, the corrosive species in form of solutions or gases can penetrate into the coating to blister, leading to peeling of the coating [2]. Therefore, in order to

increase the bonding between the coating and the substrate material and the bonding among the coating layers, while reducing the residual stress and porosity within the coating, one of the methods is annealing which facilitates the coatings to diffuse into each other and diffuse to the substrate material [2, 5].

The beneath Ni-20Cr duplex coating annealed in combination with an upper Al coating as a sealant has been studied. Annealing of diffusive Al coating on steel substrate has also been studied [5]. Annealing of Al coating beneath Ni-20Cr coating on steel substrate has not been studied extensively. The Al coating beneath the Ni-20Cr coating will promote the strength of both coatings and the Al coating that attaches to the steel substrate can protect the steel with both methods of barrier and sacrificial anode. The upper Ni-20Cr alloy coating is resistant to abrasion with good thermal resistance [5].

## 2. EXPERIMENT

### 2.1. Preparation of test specimens

#### *Equipments and materials:*

- Cleaning equipment: Abrasive blast machine SPEEDO PBM-100 (Singapore).
- Abrasives: aluminum oxide  $Al_2O_3$ , particle size 0.8-1.2 mm.
- Coating materials: Aluminum wire (99.99 % Al) and Ni-20Cr alloy wire (79.39 % Ni, 18.16 % Cr, 0.9 % Si, 0.26 % Ti, 0.73 % Mn, 0.56 % Fe), with a diameter of 2 mm.
- Arcspray equipment: OSU-HESSLER 300A (Germany).
- The base material is CT3 steel sheet and C45 steel sized  $\Phi 20 \times 5$  mm.

The steel substrate is cleaned from grease with acetone; dried and then cleaned and roughened; then sprayed with an aluminum coating of a thickness of 120-150  $\mu m$ , followed by a coating sprayed with Ni-20Cr alloy in thickness of approximately 200-250  $\mu m$ . The time between two sprayings should not exceed 20 minutes to avoid surface oxidation.

### 2.2. Heat treatment

In this study, we investigated the properties of the coating system heat-treated for 2, 4, 6 and 8 hours at different temperatures of 400, 500, 550 and 600 °C. The description of the annealing process is shown in Figure 1.

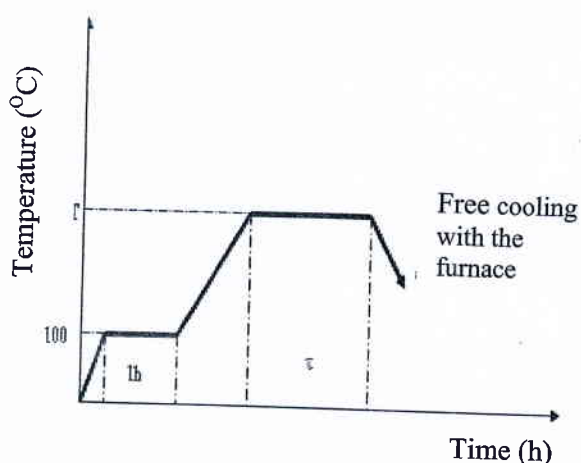


Figure 1. Heat treatment process

Equipment: NABERTHERM furnace.

### 2.3. Experimental methods

The specimens annealed in different modes were filled with epoxy resins in a cylindrical PVC tube of size  $\phi 25 \times 20$  mm high, then abraded to a cross section with two-stone abrasives, followed by grinding on abrasive paper in size between 100 and 2000 and polished on a polishing machine with chromium(III) oxide powder. After that, the specimens were etched with 4 %-HNO<sub>3</sub> alcohol solution.

The assessment of the coating quality and influence of annealing on the properties of the coating is on basis of 3 methods:

- Studying microstructure: It uses Axiovert 25 A optical microscope (Germany) - Hanoi University of Technology.

- Phase microhardness measurement was performed on microhardness tester IndentaMet 1106; the image of the indentation was taken by Axiovert 40 MAT hardness tester at the Institute of Materials Science, Vietnam Academy of Science and Technology. The HV microhardness of the coating is measured under a weight load of 300 g in 15 s.

- Evaluation of wear resistance using specimens of Al/Ni-20Cr duplex coating on C45 steel substrate after annealing at 550 °C/8h and 600 °C/8h. An untreated coating specimen was also tested for comparison purpose. The test was realized in accordance with ASTM G99. For the wearing friction pair of Ni-20Cr/carbon steel, it works in dry mode (without lubrication) with load  $P = \text{const}$ , velocity  $V = \text{const}$ .

## 3. RESULTS AND DISCUSSION

### 3.1. The microstructure and microhardness of Al/Ni-20Cr duplex coating system before heat treatment

#### *Microstructure before heat treatment*

Observation of the pre-annealing microstructure of the Al / Ni-20Cr / CT3 steel substrate in Figure 2 shows that the microstructure clearly distinguishes between coating materials and the substrate. The right layer is the steel substrate, followed by the aluminum coating in the middle, and Ni-20Cr alloy coating. The Al coating has a thickness of 120 - 150  $\mu m$ . The Al coating has a roughness of about 15 - 20  $\mu m$ , allowing the

Ni-20Cr coating to adhere firmly to the Al coating. The Ni-20Cr coating has a thickness of 200 - 250  $\mu\text{m}$ . The coating structure is brighter due to less corrosion after etching. The coatings are structured as superimposed layers, with dark border among layers, some of black porous areas.

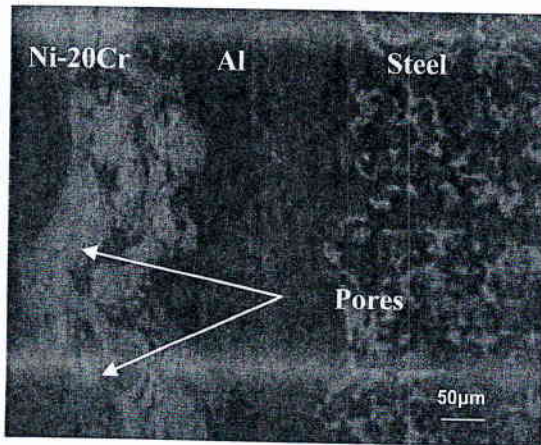


Figure 2. Structure of the coating before heat treatment on the steel substrate CT3 x 200

*Pre-annealing microhardness*

Figure 3 shows pre-annealing microhardness of the Al/Ni-20Cr duplex coating.

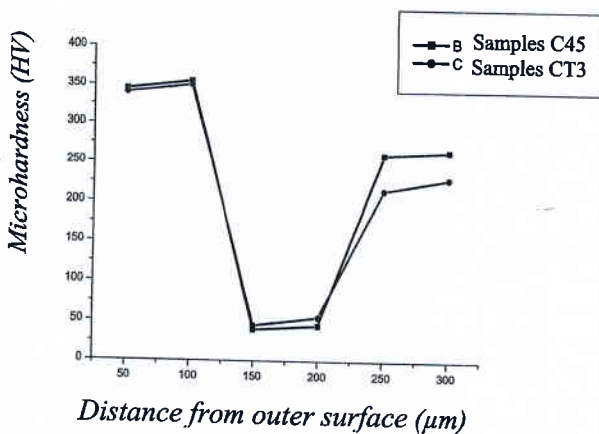


Figure 3. Microhardness measured on the Al/Ni-20Cr duplex coating specimens on the C45, CT3 steel substrates before heat

The hardness test results show that there are three different hardness areas ranging from the outer surface to the steel substrate: Ni-20Cr coating, Al layer and the steel substrate. The Ni-20Cr region near the vertical axis (external area) has the highest hardness of 350 - 400 HV. The Al area (150 - 200

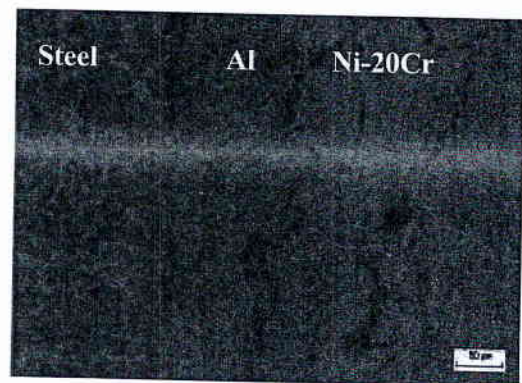
$\mu\text{m}$  from the surface) has the lowest hardness of 45 - 50 HV. The steel substrate has a higher hardness than the Al substrate and varies with steel grade. The C45 substrate has hardness of 260 - 300 HV, the CT3 does hardness of 240 - 280 HV.

**3.2. Influence of annealing on the microstructure, hardness of the Al/Ni-20Cr duplex coating**

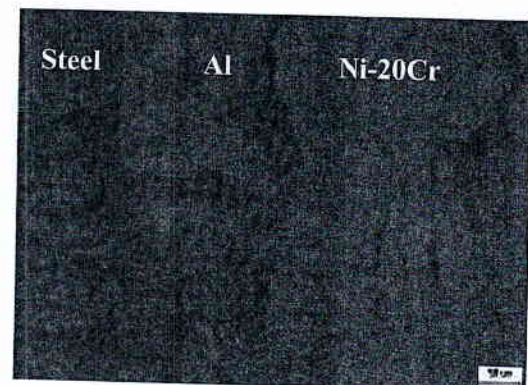
*Post-annealing microstructure*

In annealing regimes, the author presented microstructure figures only in a number of typical modes. The 4-hour thermal retention time was the medium duration among the studied periods (2, 4, 6 and 8 hours), so the 4-hour thermal retention time is selected for the typical annealing mode.

Figures 4 and 5 are respectively microstructures of the Al/Ni-20Cr duplex coating with substrates C45 and CT3 after 4-hour annealing at 500 and 600  $^{\circ}\text{C}$ .



a) C45-500  $^{\circ}\text{C}$ - 4 hours x200



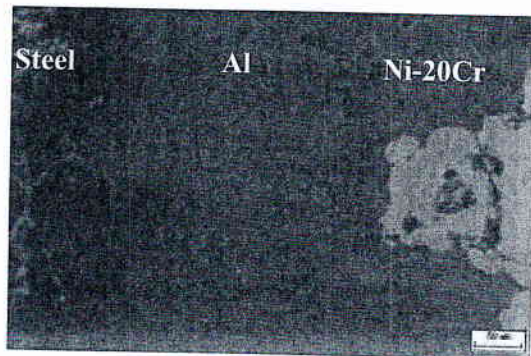
b) C45-600  $^{\circ}\text{C}$ - 4 hours

Figure 4. Microstructure of the Al/Ni-20Cr duplex coating on the C45 steel substrate after heat treatment for 4 hours at 500 and 600  $^{\circ}\text{C}$

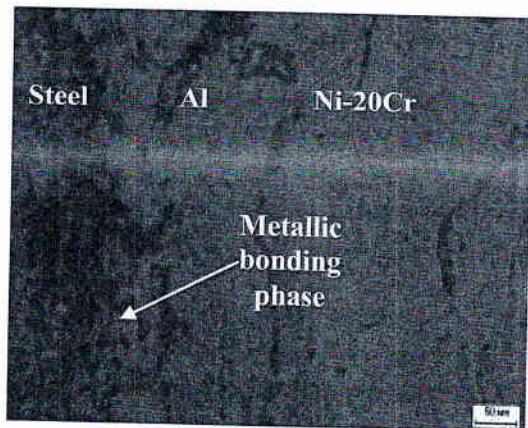
On the figure of the Al/Ni-20Cr duplex coating, there are different dark and bright areas,

consisting of many material components with multiple phases, with varying levels of corrosion. Therefore, it is possible to reflect light differently so there are different colors.

On the microstructure image, from the left to the right are the steel substrate, the Al coating, and the Ni-20Cr coating. After etching the steel substrate (C45, CT3), there are two distinct colors that are the microstructure of the steel with bright white ferrite and pearlite plates (mixture of ferrite and cementite) in dark color. The Al coating is more corroded so they are darker; the more chemical-resistant Ni-20Cr coatings are bright white. Inside the duplex coating, there are black areas which may be porous holes, the interfaces among the coatings, between coatings and steel substrate and among coatings (same material).



a) CT3-500 °C-4 hours x200



b) CT3-600 °C- 4 hours x200

Figure 5. Microstructure of the Al/Ni-20Cr duplex coating on CT3 steel substrate after heat treatment for 4 hours at 500 and 600 °C

Figures 4a and 5a are the microstructure of Al/Ni-20Cr coatings on C45 and CT3 substrates after annealing at 500 °C for 4 hours. Visual

observation of the microstructure shows that, at the interface among the coatings, as well as between the coatings and the steel substrate, a relatively strong, apparent interaction has occurred. Especially at the interface between the Al and Ni-20Cr coatings, there is a scale slightly darker than the Ni-20Cr coating but brighter than the Al coating, which is predicted to be a new phase formed after annealing. This area is approximately half of the width of the Al coating (about 40 - 60 μm).

Figures 4b and 5b are the microstructure of the Al/Ni-20Cr duplex coating after annealing at 600 °C on C45 and CT3 for 4 hours. It shows that the Al coating almost disappeared, and is substituted with a new thicker layer, which is about 80 - 100 μm thick.

To better understand the properties of this phase, we need to measure the microhardness of the phase, and test the wear resistance of the coating, in order to more accurately conclude the newly formed phase.

#### Microhardness of the Al/Ni-20Cr duplex coating

Figures 6 and 7 show the results of the microhardness measurement at two characteristic coating regions. Region I is the Al coating adjacent to steel substrate, and region II is the Al coating area adjacent Ni-20Cr coating. The microhardness measurements was conducted on a specimen of Al/Ni-20Cr duplex coating on C45 steel substrate after annealing at 600 °C for 2, 4, 6 and 8 hours.

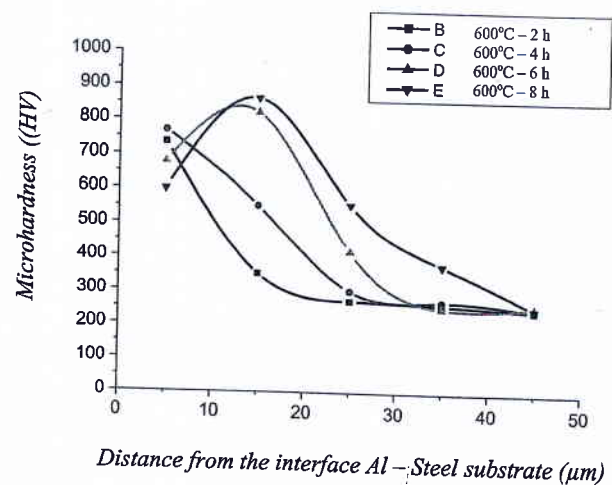


Figure 6. Microhardness measured on the interface Al/ C45 steel substrate after heat treatment at 600 °C

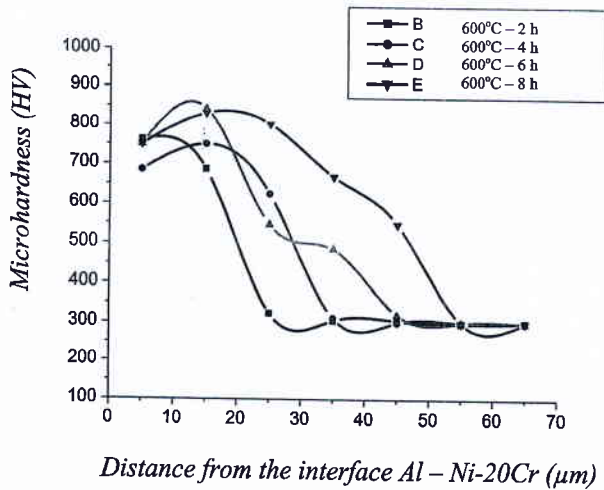


Figure 7. Microhardness measured on the interface Al/Ni-20Cr after heat treatment at 600 °C

The result of the hardness measurement in Fig. 6 revealed a diffusion area between the Al coating and the steel substrate. We realize that the result of the microhardness measurement is quite in accordance to obtained microstructure results at 2-hour retention time of the diffusion area between the Al coating and the C45 steel substrate. The white bright areas have a hardness of 750 HV, and the diffusion thickness is about 20 - 25 µm. When the heat retention time increases to 8 hours, the highest hardness reaches 850 HV, and diffusion thickness is about 40 - 45 µm.

Based on the results of the microhardness measurement on the interface between the Al coating/the steel substrate and the Al coating/Ni-20Cr coating at 600 °C, with different retention times, they show that the longer the retention time, the higher the diffusion layer. The highest diffusion layer is about 40 - 45 µm (corresponding to the 8-hour thermal retention time). Hardness of the diffusion layer with the 2-hour retention time is about 750 HV at the interface area between the Al - the steel substrate. When the thermal retention time increases, the hardness does not increase much, that the highest value reaches 850 HV (thermal retention for 8 hours). Al is capable of diffusing into the steel substrate and also interacts with the Ni-20Cr coating to form high hardness alloyed phases.

*Wear resistance of the coating*

The wear resistance and friction coefficient of the Al / Ni-20Cr duplex coating is measured on the TE97 Friction and Wear Demonstrator at the

Laboratory of Materials - Vinacomin Institute of Mining and Energy.

The abrasion test results of the Al/Ni-20Cr duplex coating are given in Table 1.

Table 1. Results of of wear test

No.	Specimens	Average intensity of abrasion in mass (kg/N.m)	Average friction coefficient
1	Unannealed specimen	4.86 x 10 <sup>9</sup>	0.75
2	Annealed specimen 550 °C - 8 hours	2.15 x 10 <sup>9</sup>	0.65
3	Annealed specimen 600 °C - 8 hours	1.92 x 10 <sup>9</sup>	0.60

The results of the wear test presented in Table 1 show that the wear intensity of the annealed specimen decreases with increasing of annealing temperature and it is lower than that of unannealed specimen. It is due to a fact that annealed samples containing high hardness phases so they have better abrasion resistance than unannealed samples. The coated specimen annealed at 600 °C has higher hardness than the one treated at 550 °C.

As the annealing temperature increases, the coefficient of friction decreases, possibly due to the tighter metal layers, and the coarser grains, thus it would be favourable for the machine parts working under heavy sliding wear conditions.

4. CONCLUSIONS

Some conclusions are made from this study as follows:

- The Al/Ni-20Cr duplex coating once annealed will increase the bondings among the coating layers, as well as between the coating and steel substrate.

- After annealing at suitable temperature (500 - 600 °C), the Al coating may diffuse into the steel substrate and diffuse into the Ni-20Cr coating to

form an intermetallic phase with high hardness of 800 - 850 HV and diffusion area of 40 - 45  $\mu\text{m}$ .

- The Al/Ni-20Cr duplex coating after annealing has higher wear resistance and less friction coefficient than unannealed coating.

#### ACKNOWLEDGMENT

This research is funded by Vietnam National Foundation for Science and Technology Development (NAFOSTED) under grant number 104.05-2011.38.

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