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# Reaction mechanism of Ni-Al during thermal spraying

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In this paper, the microstructure and element distribution of the Ni-Al coating prepared with the nickel coated aluminum composite powder, aluminum coated nickel composite powder and Ni-Al composite wire are studied by Scanning Electron Microscopy. Energy dispersive spectrometer element mapping was employed to obtain elemental composition at different areas of the powder and coating cross-section. X-ray diffraction and thermogravimetry differential thermal analysis are used to qualitatively analyze the phase. Finally, the reaction mechanism of Ni-Al in thermal spraying process is studied. It is found that the quality of Ni-Al coating obtained by aluminum coated nickel powder is better than that of nickel coated aluminum powder. Compared with Ni-Al composite powder, the spraying efficiency of Ni-Al composite wire flame spraying is higher.

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*Keywords:* Thermal spraying, Ni-Al coating, Ni-Al composite wire, Reaction mechanism

## 1. Introduction

Thermal spraying is a surface processing method for heating solid-state materials through a special spray gun to melted or partially melted to the surface of the treated material to form a coating. Adding special coatings on the surface of common materials by thermal spraying can achieve corrosion protection, wear resistance, wear reduction, high temperature resistance, oxidation resistance, heat protection, insulation, electric conduction, microwave radiation protection and other functions. Thermal spraying technology is an important part of surface engineering technology. It has a wide range of applications, accounting for about 1/3 of surface engineering technology [1].

Thermal spraying technology has the characteristics of flexible process, wide application range, small heating area and high production efficiency [2-3]. It plays an important role in modern aviation and atomic energy industry [4-6]. Ni-Al coating has always been a protective coating for high temperature components of aeroengine. It has excellent properties such as oxidation resistance, thermal shock resistance, wear resistance and atmospheric corrosion resistance. This

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coating is mainly prepared by thermal spraying. It is often used as the bond coat, which can not only provide protection to the substrate, but also increase the bonding strength between the top coat and the substrate. Its surface is more irregular than the surface of the matrix treated by sandblasting coarsening, so the top coat can form stronger mechanical chimerism with it. The Ni-Al bond coat plays a shielding role between the top coat and the substrate, which can minimize the oxidation or corrosion of the substrate caused by the inherent pores of the thermal spraying coating. In addition, the expansion coefficient of Ni-Al is between the substrate material and the top coat. It has enough toughness under mechanical and thermal load, which can "buffer" the stress caused by the difference of the thermal expansion coefficient between the substrate and the top coat. Ni-Al coatings are often prepared by flame spraying and plasma spraying [7].

During the spraying process, when nickel and aluminum are heated to the melting point of aluminum, the reaction between nickel and aluminum occurs violently, and a lot of heat is released at the same time. The intermetallic compounds $Ni_3Al$ and NiAl with uniform composition are formed, which makes the micro metallurgical bonding between the coating and the substrate [8]. Ni-Al can produce a strong bonding layer with steel, aluminum and aluminum alloy, cast iron and copper. The substrate of these materials is sprayed with a layer of nickel aluminum material as the bottom layer [9]. Ni-Al compounds have the properties of metals and ceramics [10].

Nickel coated aluminum powder is a kind of powder with aluminum powder as the core, which is coated with a layer of metal nickel uniformly on the periphery, so as to form a kind of tightly combined powder[11]. Aluminum coated nickel powder is a typical composite powder with nickel as the core and a layer of aluminum powder is uniformly coated on the surface of nickel by agglomeration method. It is used as bond coat or intermediate layer at medium and high temperature. As a bond coat material, Ni-Al powder occupies a special position in thermal spraying and is known as a synergistic material [12].

## 2. Methods and procedure

### 2.1. Preparation of coating

The feedstock powders of nickel coated aluminum powder (Metco 404NS) and aluminum coated nickel powder (Metco 450NS) and Metco 405NS (80% Nickel / 20% Aluminum) composite wire were obtained from Oerlikon Metco (Winterthur, Switzerland). The composite coatings were fabricated by atmospheric plasma spraying (APS) and wire flame spraying. Before APS or wire flame spraying, the substrates (45 steel) were sand blasted with corundum grits and then cleaned by ultrasonic cleaner with acetone. The process parameters of plasma spraying Ni-Al coating are shown in Table 1. Wire flame spraying process parameters are shown in Table 2.

The X-ray diffraction (XRD) patterns of the samples were obtained on D8-ADVANCE (AXS, Germany). Thermogravimetric differential thermal analysis (TG-DTA) of powder was performed on a STA 449F3 synchronous thermal analyzer (NETZSCH, Germany). GeminiSEM500/VP (CARL ZEISS, Germany) ultrahigh resolution emission scanning electron microscope (SEM) was used to observe the surface microstructure of the powder and coating before and after spraying. The powders and coatings were impregnated with epoxy. After hardening of the resin, the sample was ground with SiC abrasive paper and polished on soft disks with diamond suspension. A cross section of the sample was investigated by SEM and Digital Microscope VHX-5000 (KEYENCE, Japan).

*Table 1. Plasma spraying process parameters.*

| Powder | Metco 404NS | Metco 450NS |
|---|---|---|
| Current (A) | 495 | 450 |
| Voltage (V) | 68 | 65 |
| Primary gas pressure(Ar,PSI) | 75 | 75 |
| Primary gas flow (Ar,SCFH) | 90 | 110 |
| Power feed rate (% r.p.m) | 35 | 25 |
| Spray distance (mm) | 105 | 115 |

*Table 2. Wire flame spraying process parameters.*

| | |
|---|---|
| Acetylene pressure (PSI) | 15 |
| Acetylene gas flow (SCFH) | 45 |
| Oxygen pressure (PSI) | 33 |
| Oxygen gas flow (SCFH) | 38 |
| Compressed air pressure (PSI) | 70 |
| Compressed air gas flow (SCFH) | 72 |
| Spray distance (mm) | 200 |

## 3. Results and discussion

### 3.1. Surface morphology and composition of powders

#### *3.1.1. Metco 404NS*

Figure 1 shows the surface morphology of nickel coated aluminum powder (Metco 404NS) prepared by chemically clad. The main composition is 80% Nickel / 20% Aluminum. Chemically clad method refers to the process of metal precipitation by chemical method without external current. Chemically clad is mainly used for coating metal or composite coating on the surface of ceramic powder to realize the uniform mixing of ceramic and metal, so as to prepare cermet composite materials. Manufactured using the hydrometallurgy process, the chemically clad composite particles in these materials demonstrate consistent chemistry and nickel shell thickness surrounding the aluminum core. It can be seen from the figure that most of the Ni-Al powder particles are regular spherical. The fluidity of the powder is good, which can improve the powder feeding efficiency. The coating effect of nickel layer is very good and complete and continuous. The surface is free of voids and the dispersion is good.

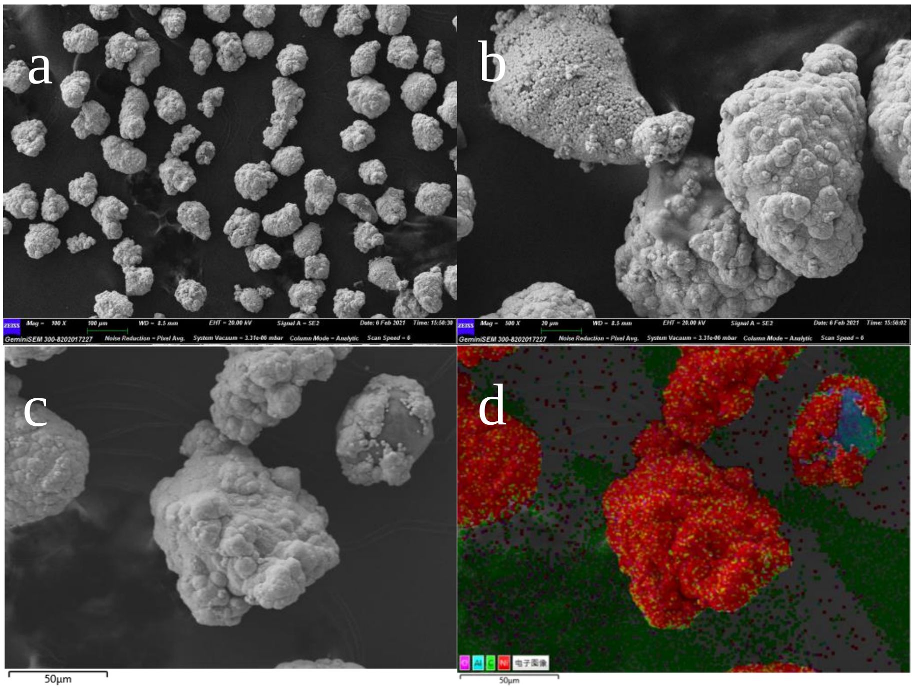


*Fig. 1. SEM micrographs of Metco 404NS powder.*

Figure 1d shows the Energy dispersive spectrometer (EDS) element mapping of nickel coated aluminum powder. It is proved that the powder surface is almost exclusively nickel. It also shows that the coating effect is good, the core-shell structure is complete, and the complete coating structure is realized. In addition, there are occasional powder particles that are not completely coated.

***3.1.2. Metco 450NS***

Figure 2 shows the surface morphology of aluminum coated nickel powder (Metco 450NS) prepared by mechanically clad. The main chemical composition is Ni5Al (95% Nickel / 5% Aluminum). Mechanically clad method uses mechanical forces such as extrusion, impact, shear and friction to evenly distribute the modifier on the outer surface of powder particles, so that various components infiltrate and diffuse into each other to form coating. However, this method is only used for the coating of micron sized powder, and the powder is required to have a single dispersion.

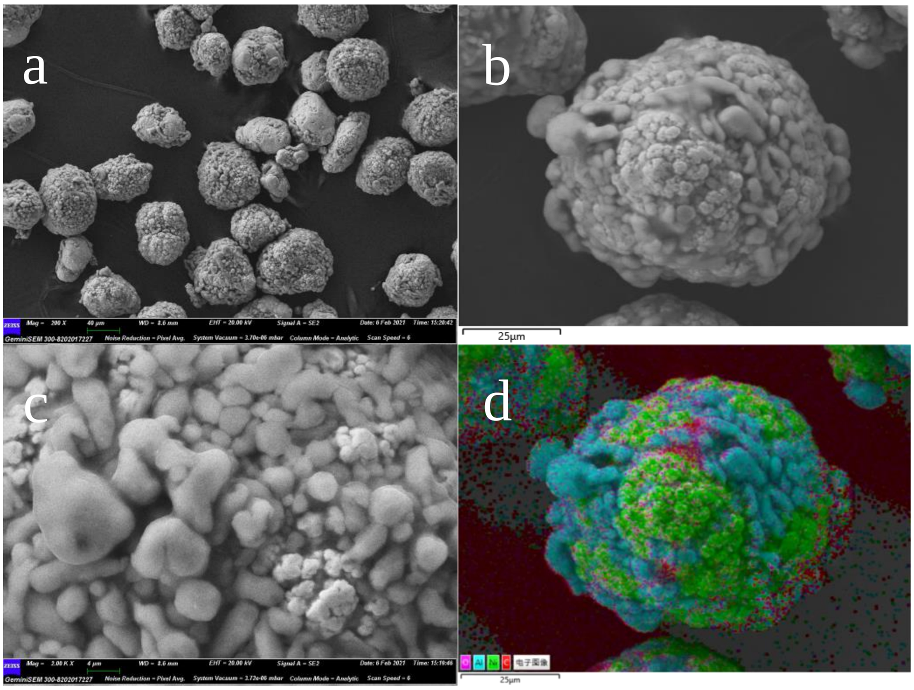


*Fig. 2. SEM micrographs of Metco 404NS powder.*

It can be seen from Figure 2b and Figure 2c that the structure of nickel is different from that of aluminum. EDS element mapping (Figure 2d) indicates that the main composition of the outer agglomerate layer is Al, and the main composition of the powder core is Ni. The surface of agglomerated Al is smooth, while the surface of Ni in the the core is rough. It can be seen that the surface of nickel powder is coated with a layer of aluminum powder, forming a similar "sesame ball" structure. But it is not completely covered.

### 3.2. Cross section and EDS analysis of powders

#### *3.2.1. Metco 404NS*

Figure 3 shows the image of Metco 404NS powder cross-section. It can be seen that in the powder the aluminum core is completely encapsulated in nickel shell. Region A is nickel while region B is aluminum. It is proved that Metco 404NS powder is completely coated by chemically clad. Figure 3d shows the EDS element mapping of the aluminum coated nickel powder cross section. The results show that the outer layer is almost Ni and the inner core is almost Al. The outer layer of nickel completely covers the inner core of aluminum.

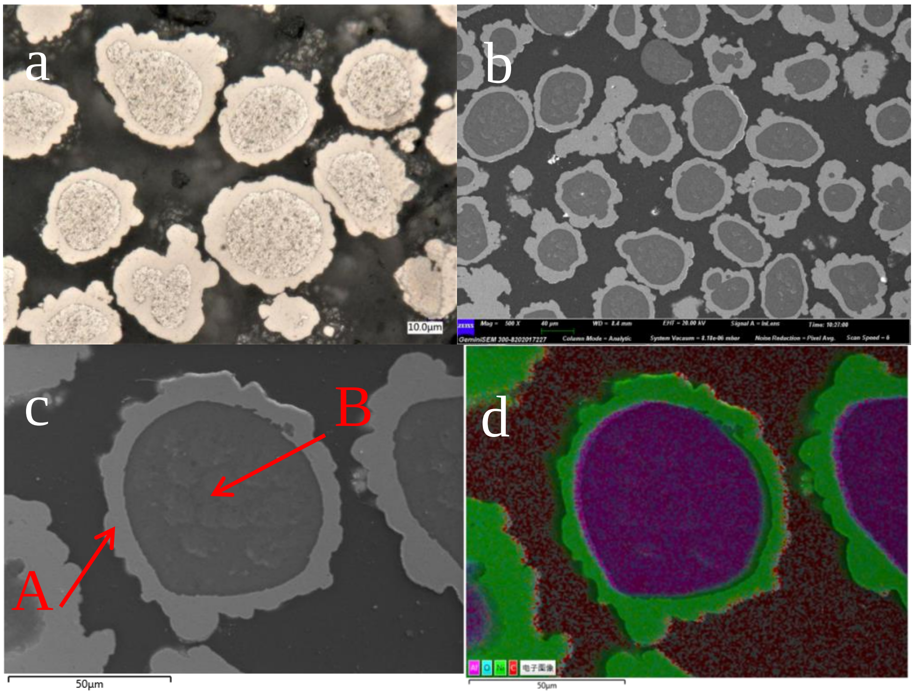


*Fig. 3. Cross-section of Metco 404NS powder.*

### *3.2.2. Metco 450NS*

Figure 4 shows the image of Metco 450NS powder cross-section. It can be seen that nickel core is not completely covered by aluminum shell. Region A is aluminum while region B is nickel. It is proved that Metco 450NS powder is not completely coated by mechanically clad. Figure 4c shows the EDS element mapping of the Metco 450NS powder cross section. It can also be seen that the mechanically agglomerated Al is not completely coated with Ni.

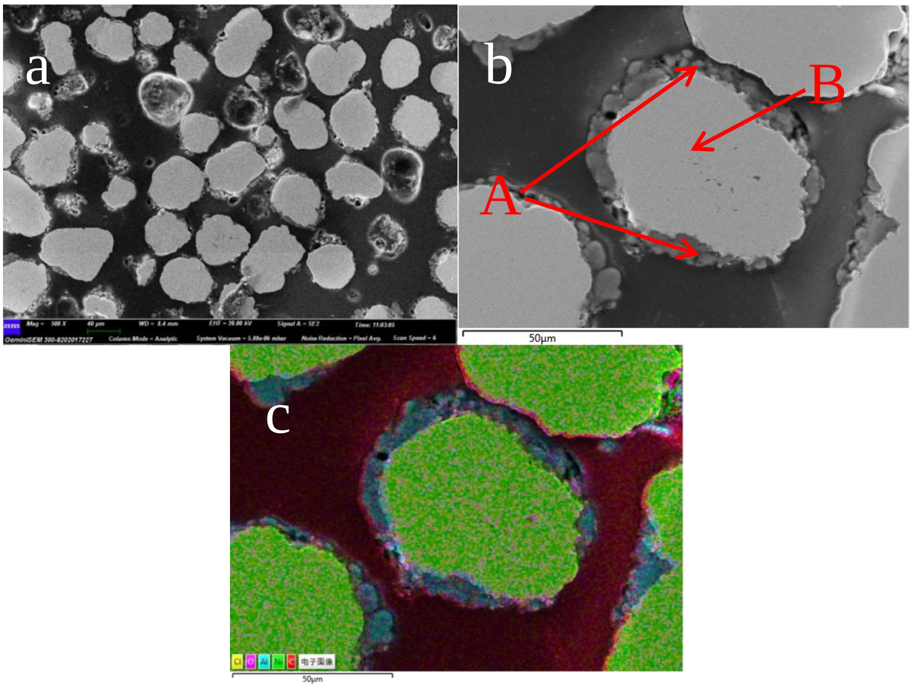


*Fig. 4. Cross-section of Metco 450NS powder.*

### 3.3. Surface morphology and composition of coatings

#### 3.3.1. Metco 404NS

Figure 5 shows the morphology of the Metco 404NS coating surface and its different areas. The coating has a typical layered structure and is composed of layered or irregular granular melted particles piled on top of each other. It meets one of the basic characteristics of plasma-sprayed coating. EDS indicates that the main composition of the surface smooth region (Figure 5c) are Al. The atomic percentage of Ni and Al in the flocculent region (Figure 5d) is similar and the content of oxygen element is relatively high. The surface of the globule (Figure 5e) contains more Ni, and the main composition of the block (Figure 5f) is alumina.

There are unmelted particles on the surface and the coating is oxidized. The surface morphology of the coating includes a surface flat area and a particle rich area. The surface flat area is formed by the deformation of fully melted Ni-Al particles. During the thermal spraying process, particles fly at high speed to the substrate along with the flame flow and collide to form a strip structure parallel to the surface of the substrate. During the spraying process, particles reach a high temperature to form nickel alloy droplets, at which time Al will precipitate out of the Ni solid solution. Due to the high temperature and the strong activeness of the precipitated aluminum, the Al precipitated during spraying can easily react with the oxygen in the air to form $Al_2O_3$, and $Al_2O_3$ on the surface will be sandwiched between the sediments when the particles are deposited. At the end of spraying, aluminum will continue to react with oxygen in the air due to the high temperature of the coating. The unmelted particles and pores on the surface of the coating are caused by the surface melting of the powder due to the short heating time of the powder [13].

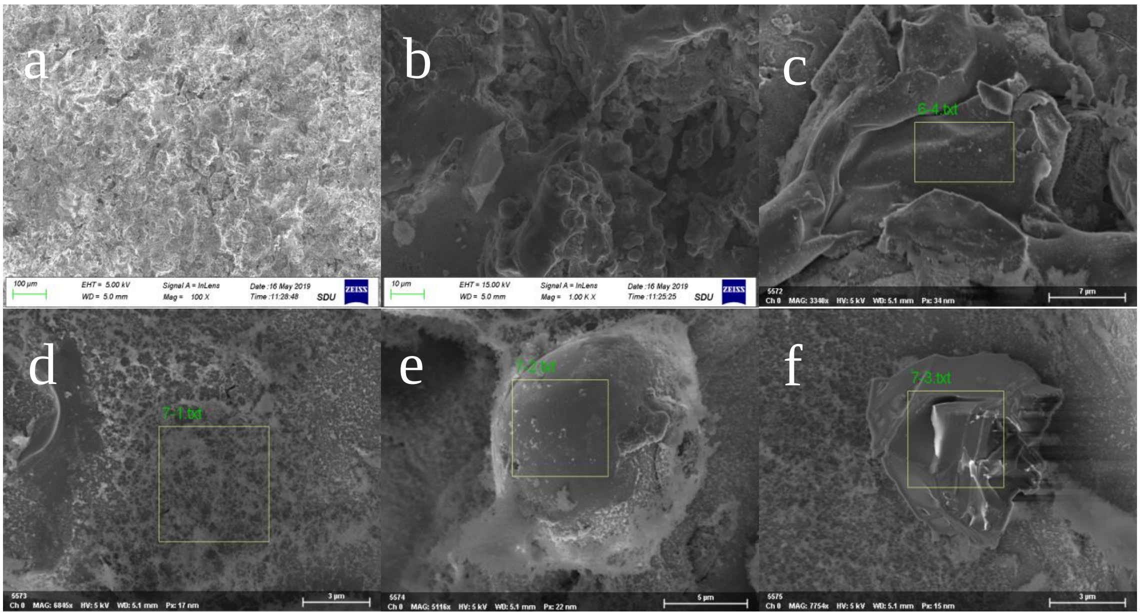


*Fig. 5. SEM images of Metco 404NS coating surface morphology and its different areas: (c) smooth surface area, (d) flocculation area, (e) globular region, (f ) block area.*

*Table 3. Chemical composition of smooth area in Figure 5c.*

| Element | Al | O | Ni | Matrix |
|---|---|---|---|---|
| Wt% | 95.68 | 4.16 | 0.16 | Correction |
| At% | 93.10 | 6.82 | 0.07 | ZAF |

*Table 4. Chemical composition of flocculent region in Figure 5d.*

| Element | Al | O | Ni | Matrix |
|---|---|---|---|---|
| Wt% | 29.15 | 9.08 | 61.77 | Correction |
| At% | 40.01 | 21.02 | 38.97 | ZAF |

*Table 5. Chemical composition of globular region in Figure 5e.*

| Element | Al | O | Ni | Matrix |
|---|---|---|---|---|
| Wt% | 75.68 | 1.71 | 22.61 | Correction |
| At% | 57.71 | 4.78 | 37.50 | ZAF |

*Table 6. Chemical composition of block areas in Figure 5f.*

| Element | Al | O | Ni | Matrix |
|---|---|---|---|---|
| Wt% | 64.37 | 31.99 | 3.64 | Correction |
| At% | 53.65 | 44.96 | 1.40 | ZAF |

Figure 6 shows the SEM images of Metco 404NS coating surface morphology and EDS element mapping. In Figure 6b, there is more Ni and less Al in the field of view, and the powder used is nickel coated aluminum, which meets the coating composition standard. The distribution of O element in Figure 6d and Figure 6e is similar to that of Al element, but different from the distribution of Ni element, indicating that there is oxygen on aluminum and aluminum oxidation is serious. The main element is Ni.

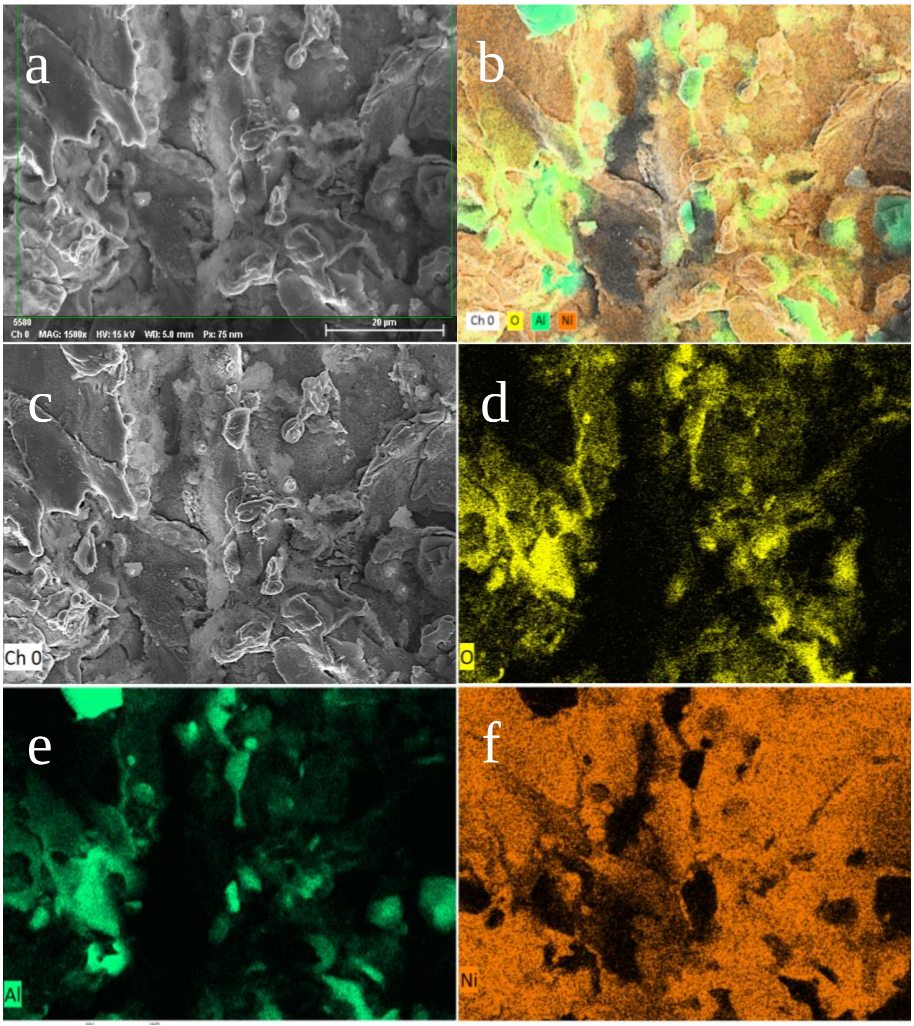


*Fig. 6. SEM images of Metco 404NS coating surface morphology and EDS element mapping.*

### *3.3.2. Metco 450NS*

Figure 7 shows the morphology of the Metco 450NS coating surface and its different areas. There are pores in the coating, the melting effect of the coating material is good, it is a typical layered staggered structure. There is a small amount of spherical structure, which is formed by collision when particles splash. There are no large unmelted particles. It can be divided into smooth area, block area and unmelted particle area. The main composition of smooth area (Figure 7c) is Ni element, the main composition of block (Figure 7d) is alumina, and the unmelted particle (Figure 7e) is mainly Ni-Al. The spraying process of Ni-Al powder is a comprehensive exothermic process in which various reactions occur between nickel and aluminum, thus forming intermetallic compounds such as NiAl and $Ni_3Al$, which form a micro-metallurgical bonding between the coating and the substrate [14-16]. After the aluminum coated nickel material is heated during spraying, the surface agglomerated aluminum powder reacts with oxygen in the air to form $Al_2O_3$. At the same time, exothermic reactions happen between nickel and aluminum to form nickel-aluminum intermetallic compounds.

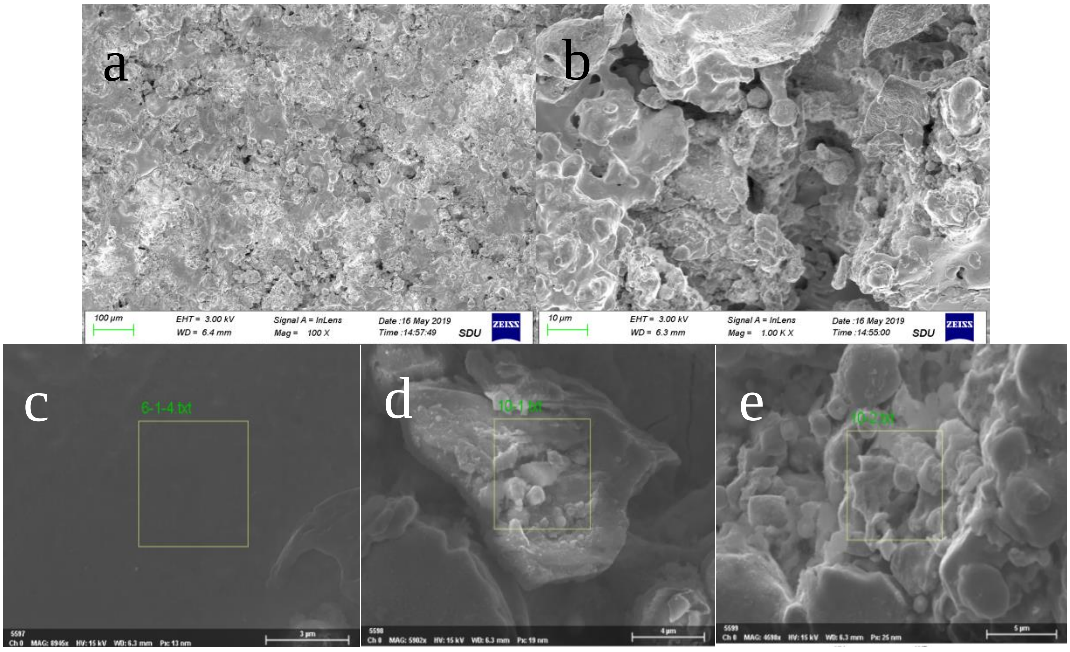

*Fig. 7. SEM images of Metco 450S coating surface morphology and its different areas: (c) smooth surface area, (d) block area, (e) unmelted particles.*

*Table 7. Chemical composition of smooth area (Figure 7c) of Metco 450NS coating.*

| Element | Al | O | Ni | Matrix |
|---|---|---|---|---|
| Wt% | 7.77 | 0.55 | 91.68 | Correction |
| At% | 15.29 | 1.81 | 82.90 | ZAF |

*Table 8. Chemical composition of block area (Figure 7d) of Metco 450NS coating.*

| Element | Al | O | Ni | Matrix |
|---|---|---|---|---|
| Wt% | 44.38 | 39.70 | 15.92 | Correction |
| At% | 37.40 | 56.43 | 6.17 | ZAF |

*Table 9. Chemical composition of unmelted particles (Figure 7e) of Metco 450NS coating.*

| Element | Al | O | Ni | Matrix |
|---|---|---|---|---|
| Wt% | 69.34 | 2.66 | 28.01 | Correction |
| At% | 79.98 | 5.17 | 14.85 | ZAF |

### 3.4. Cross section and EDS analysis of coatings

#### *3.4.1. Metco 404NS*

Figure 8 shows the image of Metco 404NS coating cross-section. No round aluminum ball is found in the coating, which indicates that the melting effect of the powder is good. It also contains pores. It can be seen from Figure 8 that there are a large number of strip-shaped black phases scattered in the coating, which are Al with incomplete reaction [17]. Because in the spraying process, when the particles are heated to the melting point of aluminum, there is a violent combination reaction between nickel and aluminum. At the same time, a large amount of heat is released to form NiAl, $Ni_3Al$ and other intermetallic compounds, which prevent the diffusion of Al into Ni [18-20]. Figure 8d shows the EDS element mapping of Metco 404NS coating cross section. It can be seen that the coating is oxidized.

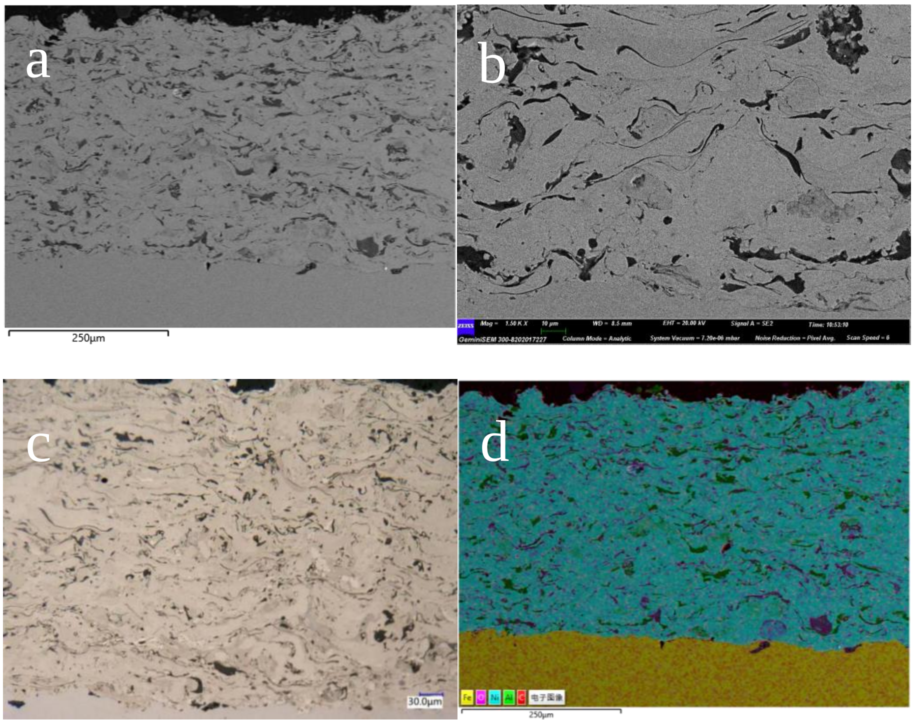


*Fig. 8. Cross-section SEM images of Metco 404NS coating.*

#### *3.4.2. Metco 450NS*

Figure 9 shows the image of Metco 450NS coating cross-section. It can be seen that the distribution of each component is uniform, the melting effect of the coating material is good, it is a typical layered staggered structure. The oxide content at the boundary of strip structure is high. The strip boundary of Ni-Al is mainly the aluminum oxide. Figure 9c shows element mapping of the cross section of Metco 450NS coating. It can be seen from the figure that the main components of the coating is Ni. The content of aluminum in the coating is very little. The coating also contains oxygen. It indicates that the coating is oxidized. As can be seen from the surface morphology and cross section images of the coating, the melting effect of Metco 450 NS is better, the unmelted particles are less, and the coating is denser. The reason is that the content of Al in Metco 450NS powder is lower than that of Metco 404NS, which leads to less reaction between Ni and Al in the spraying process and better spraying effect. The reduced aluminum content decreases the aluminide-forming exothermic reaction during the spray process, but increases the service temperature.

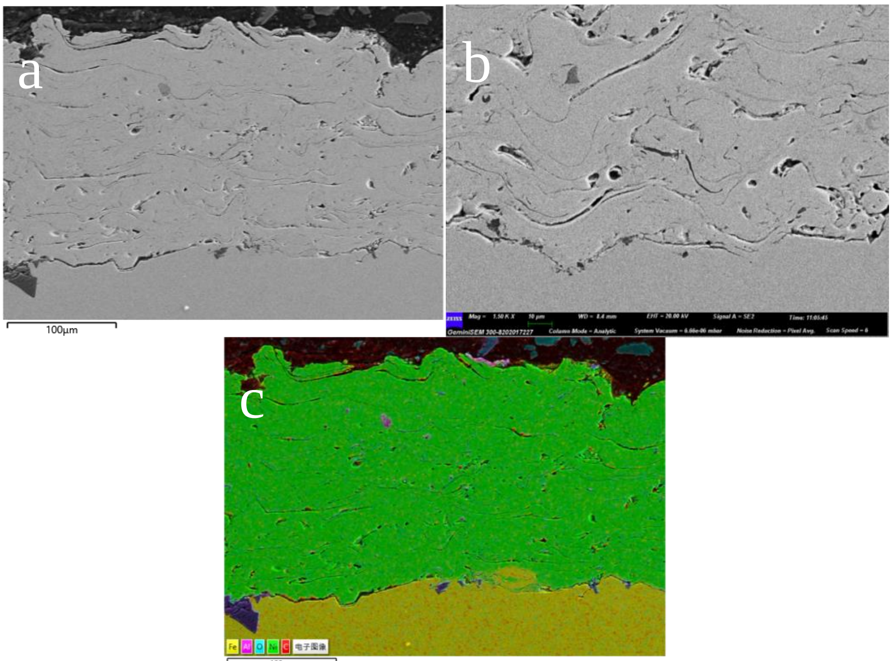


*Fig. 9. Cross-section SEM images of Metco 450NS coating.*

### 3.5. TGA Analysis

#### *3.5.1. Metco 404NS*

Figure 10 shows the TG-DTA curves of the Metco 404NS powder. The melting point of Al is 660°C, and that of Ni is 1453°C which is much higher. When Ni-Al particles are heated, Al reaches liquid state earlier than that of Ni. The endothermic peak at 657°C of the DTA curve could be attributed to the melting of aluminum core encapsulated in nickel shell. Therefore, when the melting effect of Ni is slightly worse and it is still in a plastic state, Al becomes liquid state, like the liquid Al wrapped in a soft "eggshell" [21]. Weight increase in TG curve from 700°C to 1600°C is the result of aluminum and nickel oxidation after melting of aluminum.

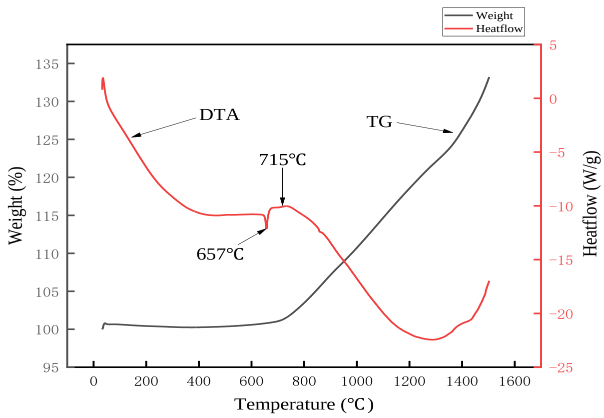


*Fig. 10. The TG/DTA curves of the Metco 404NS powders.*

### *3.5.2. Metco 450NS*

Figure 11 shows the TG-DTA curves of the Metco 450NS powder. It can be seen from the figure that when the temperature reaches 630°C, it begins to release heat. The exothermic peak appears at 667°C. Since the melting point of aluminum powder is 660°C, when aluminum is close to the melting point, violent exothermic reaction between Ni and Al occurs. At the same time, because of the huge heat generated by the exothermic reaction between nickel and aluminum, the endothermic phenomenon of aluminum melting is not obvious on the DTA curve. After the chemical reaction lasts for a period of time, that is, after 720°C, the differential heat curve goes down [22]. 1442°C is close to the melting point of nickel, so the endothermic peak at 1442°C of the DTA curve corresponds to the melting of nickel. Weight increase in TG curve from 600°C to 1600°C is the result of aluminum and nickel oxidation after melting of aluminum. The weight gain starting temperature of Metco 450NS is 600°C while for Metco 404NS it is 700°C. Because Metco 404NS is aluminum core encapsulated in nickel shell while for Metco 450NS aluminum is on the powder surface, when aluminum is melted it react with oxygen easily and earlier than Metco 404NS.

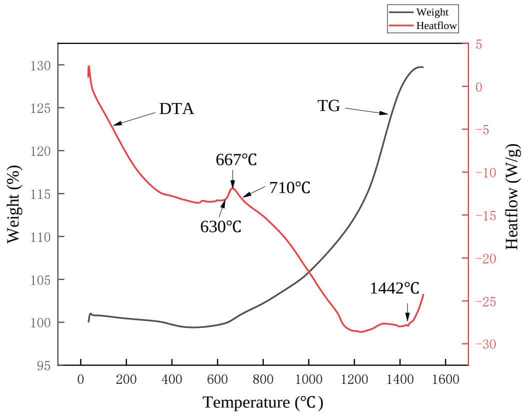


*Fig. 11. The TG/DTA curves of the Metco 450NS powders.*

## 3.6. XRD Analysis

### *3.6.1. Metco 404NS*

Figure 12 shows the XRD pattern of Metco 404NS powder and coating prepared by APS, as well as the standard card of Ni (PDF#65-2865) and Al (PDF#65-2869) for references. The analysis show that the powder and coating contain Al and Ni. The diffraction peaks of the Metco 404NS powder and coating match with the Al and Ni standard card. The peaks intensity decreases after spraying. There is a weak peak at 43.45° in the coating, which is a newly formed phase NiO. In the process of thermal spraying, the melted particles of Ni and Al are not completely isolated from the air, and some of them will be oxidized by oxygen in the air. Therefore, the XRD pattern of the coating shows that there are a small amount of NiO phase and $Al_2O_3$ phase besides the Ni and Al phases. The diffraction peak intensity of the coating is lower than that of the powder, which is due to the oxidation during the spraying process, resulting in the reduction of the precipitation of Ni and Al phases.

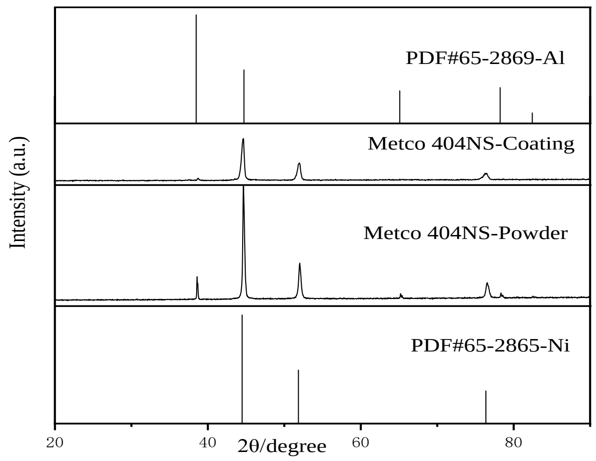


*Fig. 12. XRD spectra of Metco 404NS coating and powder.*

### *3.6.2. Metco 450NS*

Figure 13 shows the XRD pattern of Metco 450NS powder and coating prepared by APS, as well as the standard card of Ni (PDF#65-2865) and Al (PDF#65-2869) for references. The analysis shows that the powder and coating also contain Al and Ni. The diffraction peaks of the Metco 450NS powder and coating match well with the Al and Ni standard card. The peaks intensity decreases after spraying. There is a weak peak at 43.20° in the coating, which is a new phase NiO. The composition of nickel aluminum alloy with less than 5% aluminum is homogeneous Ni solid solution, which is consistent with that reported by Khor K A [23]. This result is in accordance with Ni-Al phase diagram (Fig. 14). In addition, a small amount of $Al_2O_3$ and NiO are also included.

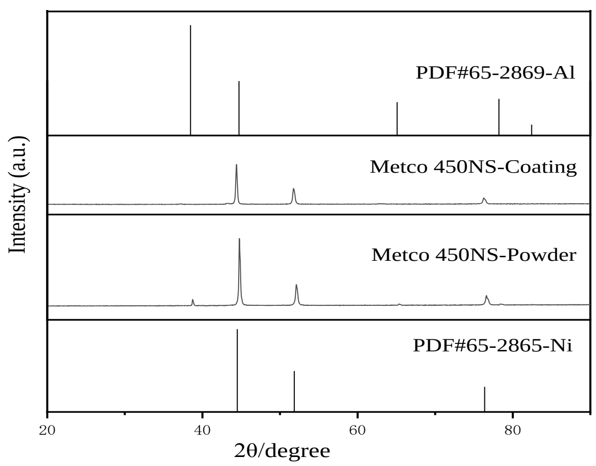


*Fig. 13. The XRD spectra of Metco 450NS coating and powder.*

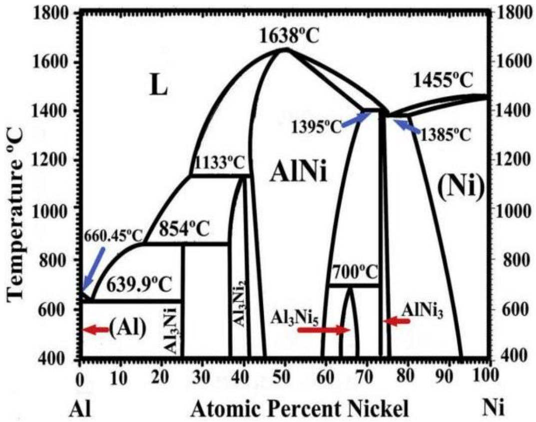


*Fig. 14. Ni-Al phase diagram* [24].

**3.7. Metco 405NS (Ni-Al 20%)**

Figure 15 shows the image of Metco 405NS (Ni-Al 20%) composite wire cross-section. It is a kind of spraying wire which is made by filling different metal or alloy powder into metal or alloy pipe, and then extruded and drawn. Due to the low strength of the wire, the wire is easy to bend, twist and break during spraying operation. Ni-Al 20% composite wire is a self-adhesive material. In the process of thermal spraying, Ni reacts with Al to form intermetallic compound and releases a lot of heat. This reaction process can last until the powder collides with the substrate surface. The bonding strength between the particles and the substrate is greatly improved [25].

Compared with Ni-Al composite powder, the spraying efficiency of Ni-Al composite wire flame spraying is higher. The obtained coatings compositions are mainly $Ni_3Al$ and NiAl [26]. The coating of Ni-Al composite wire has good wear resistance. The coating has excellent high temperature oxidation resistance under 815°C [27].

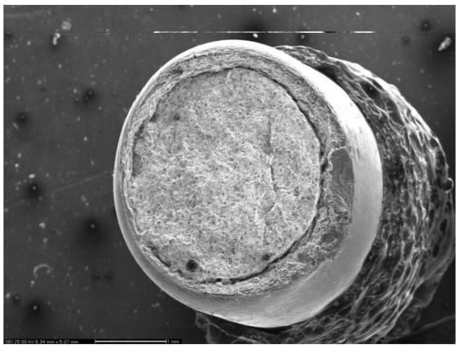

*Fig. 15. Section photo of Metco 405NS (80% Nickel / 20% Aluminum) composite wire.*

Figure 16 shows surface of coating prepared by Metco 405NS (80% Nickel / 20% Aluminum) Ni-Al composite wire flame spraying. It can be seen the surface of the Ni-Al coating is basically flat and continuous, with good surface forming quality, and the coating interior is continuous. Ni-Al coating formed is more ideal [28].


*Fig. 16. Photo of coating surface formed by flame spraying Metco 405NS (80% Nickel / 20% Aluminum) composite wire.*

Figure 17 is the coating cross section formed by flame spraying Metco 405NS (80% Nickel / 20% Aluminum) composite wire. It can be seen that the coating is typical lamellar. There are no large unmelted particles on the coating surface, and the fusion particles are well bonded. The lamellar formed in the Figure 17a is due to the splashing of some melted particles when they impact the substrate surface, and then rapidly cooled and solidified. Figure 17b is the SEM micrograph of coating cross section. It can be seen that there are a large number of strip-shaped black phases scattered in the coating, which are Al with incomplete reaction.

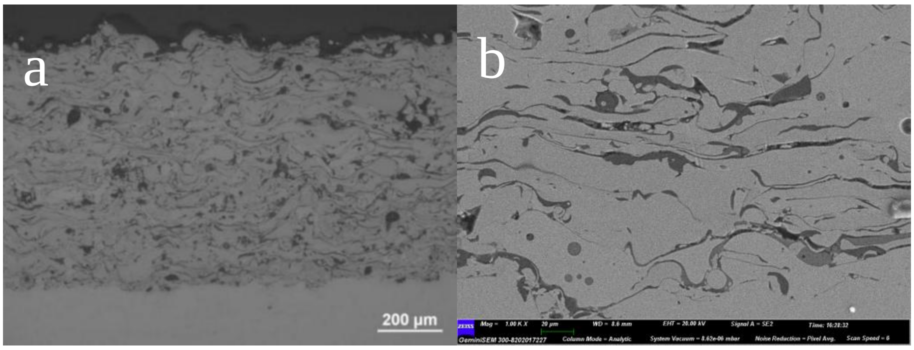


*Fig. 17. Coating section formed by flame spraying Metco 405NS composite wire.*

## 4. Conclusions

The Ni-Al coating was prepared by plasma spraying and flame spraying. The Ni-Al coating prepared from Ni-Al powder and Ni-Al composite wire was studied by SEM and XRD. The analysis shows that when the Ni-Al powder is heated to the melting point of aluminum, on the one hand, aluminum will be melted and absorb heat, on the other hand, nickel-aluminum will react to form nickel-aluminum intermetallic compound and release a lot of heat. The increase of powder weight during heating is mainly due to the oxidation of aluminum. The XRD patterns show that the main phases in the two coatings are Ni and Al, but a small amount of new NiO phase will be formed. Compared with Ni-Al composite powder, a large amount of Ni-Al compounds will be formed by the reaction of Ni and Al during the spraying of Ni-Al composite wire. This increases the area of metallurgical bonding with the substrate. Ni-Al composite wire has higher spraying efficiency.

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