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Fine structure features of Ni-Al coatings obtained by high velocity atmospheric plasma spraying

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ABSTRACT

Introduction. Development of Ni-Al intermetallic compounds is one of the priority directions of modern machine building. Due to such characteristics as high heat resistance, high temperature strength, and low density, nickel aluminides are used as functional coatings in the aerospace industry. The main methods of Ni-Al coating surfacing are High-Velocity Oxygen-Fuel and High-Velocity Air-Fuel spraying (HVOF and HVAF), atmospheric plasma spraying (APS) and its modification such as High-Velocity Atmospheric Plasma spraying (HV-APS) which provides non-equilibrium cooling conditions. Since there are eight different intermetallic compounds, as well as martensite transformation, Ni-Al coatings is quite interesting to study. The work purpose is to study the features of the martensitic structure in HV-APS coatings, and also to establish the effect of heating temperature on its decomposition. Materials and methods. Ni-Al coatings were surfaced onto a low-carbon steel substrate using the HV-APS method. Studies of the fine structure of the coatings were carried out using transmission electron microscopy (TEM). In addition, the influence heating temperature on structural transformations of the coatings was analyzed. Results and discussion. Two types of particles are formed in HV-APS coatings: with a dendritic and granular structure. The most part of HV-APS coatings consists of particles with a two-phase grain structure (Ni_xAl_{xx} and γ '- Ni_xAl grains). Only Ni₂Al₁, grains undergo martensitic transformation at cooling. Martensite in large grains (sizes greater than 500 nm) has a lamellar structure, while small grains are completely transformed into one martensite plate. In addition, the coatings contain grains in which martensite plates $(N_{l_{x}}Al_{l_{x}})$ and β -phases alternated. It is shown the behavior of martensitic plates at colliding with each other, as well as with the γ' -Ni, Al grain. Heating up to 400 °C contribute the begins of martensite decomposition in individual grains with the release of a secondary phase; after heating up to 600 °C all martensite dissolves.

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Introduction

Nowadays, intermetallic materials design for structural purposes is one of the priority areas for the development of modern mechanical engineering. Due to the combination of characteristics such as high heat resistance and thermal conductivity, the ability to maintain strength and rigidity at high temperatures and relatively low density [1–3], nickel aluminides are used as materials for components of aircraft engines, gas turbines and heat exchangers [4–6]. It is vital that *Ni-Al* system alloys, being high-temperature materials, have low ductility and fracture toughness at room temperature [6] and this limits its use as bulk parts. In turn, one of the solutions to this problem is the use of nickel aluminides as functional coatings. In general, the main methods of applying *Ni-Al* coatings are high-velocity oxygen-fuel spraying (*HVOF*) [7–9], high-

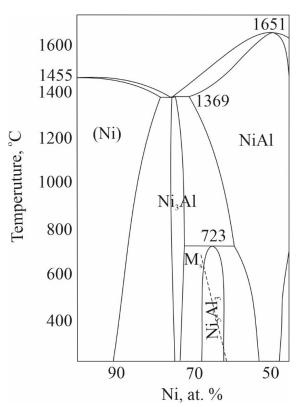


Fig. 1. Part of Ni-Al phase diagram

velocity air-fuel spraying (*HVAF*) [9, 10], atmospheric plasma spraying (*APS*) [11–14] and also its modification such as high-velocity atmospheric plasma spraying (*HV-APS*).

There are eight stable and metastable intermetallic compounds in the Ni-Al system [15], the most promising of which are aluminides located in the nickel-rich part of the phase diagram, such as γ' -Ni₃Al and β -NiAl (Fig. 1) [3, 16, 17]. β-NiAl solid solutions have a wide range of homogeneity (43-70 at. % Ni at 1,400 °C), which decreases to 45–60 at. % Ni at a room temperature [3, 16]. Cooling of the β-phase in the range of high Ni concentrations is accompanied by the formation of a mixture of β - and γ' -phases, while grains of the β -NiAl phase often have different chemical compositions. The martensitic transformation $B2 \rightarrow L1_0$ occurs in β-phase crystals containing more than 62.3 at. % Ni. The onset temperature of this transformation (M_s) varies, according to various sources, from -200 to ~ 650 or 900 °C [17–19] depending on the Ni concentration. Subsequent heating of alloys from 62.5–68.0 at. % Ni promotes the separation of the Ni_5Al_3 phase or the metastable Ni_2Al phase [20–22].

As a rule, coatings with a similar composition are often used as a bonding layer between the base material and a ceramic heat-shielding coating (YSZ) [23]. Chen et al. [24] discovered that the martensitic transformation occurring in the

metal sublayer can cause the destruction of the ceramic coating due to changes in volume during the transformation of the β -phase into martensite. Thus, the study of the structural-phase state, as well as the understanding of structural transformations are priority tasks in the *Ni-Al* coatings design, since both functional and mechanical, as well as technological properties will depend on this.

The purpose of this work is to study the features of the martensitic structure of *Ni-Al* coatings obtained by the *HV-APS* method. To achieve this purpose, the following tasks were solved:

- study of the coatings structure;
- study of the features of the martensitic structure depending on the grain size;
- study of the behavior of martensitic plates when colliding with other structural components;
- study of the influence of heating temperature on the structure of the coatings.

Materials and methods

Ni-Al coatings 500–600 µm thick were spraying on discs from low carbon steel with a diameter of 20 mm and a thickness of 8 mm. The particle size of powder was 40–100 µm, chemical composition was 75 at. % Ni and 25 at. % Al. To apply the coatings, we used the Termoplasma~50 plasma spraying installation,





equipped with an *HV-APS* plasma torch. The supersonic spraying mode using air as the working gas ensures the speed of the sprayed particles at a level of 500 m/s and higher. Optimal modes for high-velocity spraying *Ni-Al* powder are presented in our previous work [25].

To analyze the structural state of the coatings, a scanning electron microscope (*Carl Zeiss EVO50 XVP* with an *EDS X-Act* microanalyzer) and a transmission electron microscope (*FEI Tecnai G2 20 TWIN*) were used. Transverse sections of coatings were the samples for *SEM* as well as foils, cut from the middle of the coatings, were the samples for *TEM*.

Ni-Al coatings were kept for 1 hour at temperatures of 300, 400, 500 and 600 °C (air cooling) to study the structural transformations that occur upon heating.

Results and discussion

Previously, we have shown that HV-APS coatings are characterized by the presence of several zones that differ in structure [25]. Fig. 2 shows SEM image and scheme of the microstructure of the HV-APS coating in the initial state. The chemical composition of all areas was determined using micro-X-ray spectral analysis. According to the data obtained, there are particles, the central part of which is the β -NiAl intermetallic compound (section 1 in Fig. 2), surrounded by a single-phase layer of the β -NiAl phase enriched in Ni (referred to as the Ni_XAl_{I-x} phase) (section 2 in Fig. 2). The structure of section 3 in Fig. 2 is dendritic: the chemical composition of the dendrites coincides with the composition of the layer (section 2), and the chemical composition of the interdendritic space corresponds to the γ - Ni_3Al phase. We considered the fine structure of these areas more thoroughly in our work [25]. As a rule, the particles with a similar structure are not common for HV-APS coatings. Predominant particles are particles with two-phase structure consisting of Ni_XAl_{I-x} and γ '- Ni_3Al grains (section B in fig. 2).

Fig. 3 shows TEM images of section 4. It can be seen that Ni_xAl_{1-x} grains undergo a shear martensitic transformation, during which the high-temperature B2 structure transforms into the low-temperature $L1_0$ structure, while the γ' - Ni_3Al grains do not change. There are also one-phase areas consisting only of grains of the γ' - Ni_3Al phase (fig. 3, b). The shape of the grains in section 4 is non-equiaxial, which is typical for material cooled under non-equilibrium conditions. The grain sizes usually do not exceed 500 nm, although sometimes larger γ' - Ni_3Al grains are formed. Such grains often have deformation twins (Fig. 3, c) and stacking faults (Fig. 3, d).

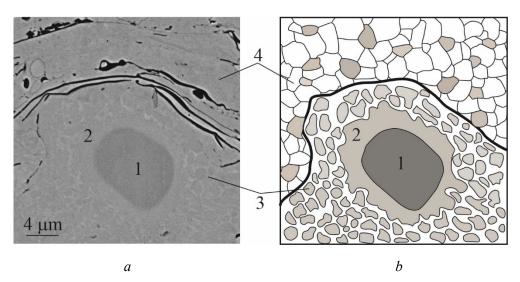


Fig. 2. SEM image (a) and scheme (b) of HV-APS coatings: $1 - \beta$ -NiAl phase; 2 - layer of $Ni_x Al_{l_x}$; 3 - area with dendritic structure:

 Ni_xAl_{l-x} dendrites, interdendritic region (γ' - Ni_3Al phase); **4** – area with grain structure: both Ni_xAl_{l-x} and Ni_3Al grains



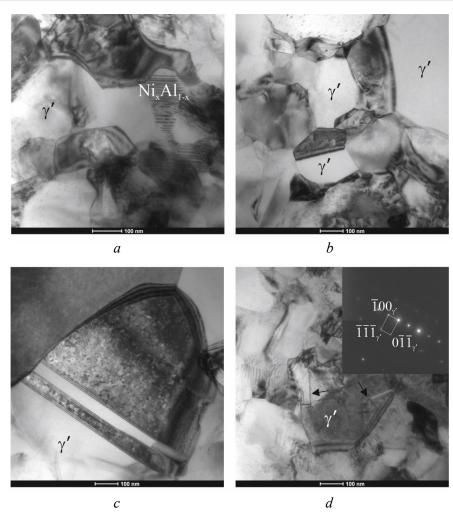


Fig. 3. Bright field *TEM* images of *HV-APS* coatings: a – two-phase area of $Ni_xAl_{l-x} + \gamma' - Ni_3Al$; b – one-phase area of $\gamma' - Ni_3Al$; c) twins in $\gamma' - Ni_3Al$; d – stacking faults in $\gamma' - Ni_3Al$ with diffraction pattern

Martensite in HV-APS coatings is lamellar (Fig. 4), but depending on the size of the grains in which the transformation occurs, it looks different. For example, martensite formed in large Ni_xAl_{l-x} grains consists of plates located in a twinned orientation relative to each other (Fig. 4, a). The distance between microtwins may be from 0.5 nm (Fig. 4, b) to several nanometers (fig. 4, a). The martensite-martensite interface can be located both inside the former Ni_xAl_{l-x} grain and outside it (Fig. 4, c). Unlike large grains (greater than 500 nm), fine grains completely transform into one microtwinning plate (Fig. 4, d). Sometimes there are martensitic grains in which, even with the use of a dark field, it is not possible to detect microtwins in pairs of parallel plates, and these plates appear to be single crystals (Fig. 5). According to data of local chemical analysis, adjacent plates have different chemical compositions. The Ni content in plates with microtwins (type 1) is 77.4 at. %, which corresponds to the Ni_xAl_{l-x} phase, while the Ni content in plates without microtwins (type 2) is 52.5 at. %, which corresponds to the P-phase.

Martensite plates can behave differently when collide with each other or with other phases. For example, growth of individual thin plates growing in different directions often does not stop. These plates pass through each other and only the area of its intersection is rearranged (Fig. 4, a). Fig. 6, a, b shows that when a martensitic plate collides with a γ' - Ni_3Al grain, it does not penetrate into it, but continues to transform. On the other hand, Fig. 6, c shows martensitic plates that seem to have grown inside the γ' - Ni_3Al grain. Apparently, in this case, the Ni_xAl_{l-x} plates appeared first, around which the γ' - Ni_3Al phase later formed. Some plates change the direction of its growth, deviating to the side at collision with obstacle (Fig. 6, d). Deformation and elastic distortions occur in areas near curved plates, which contrast is visible near the bend.



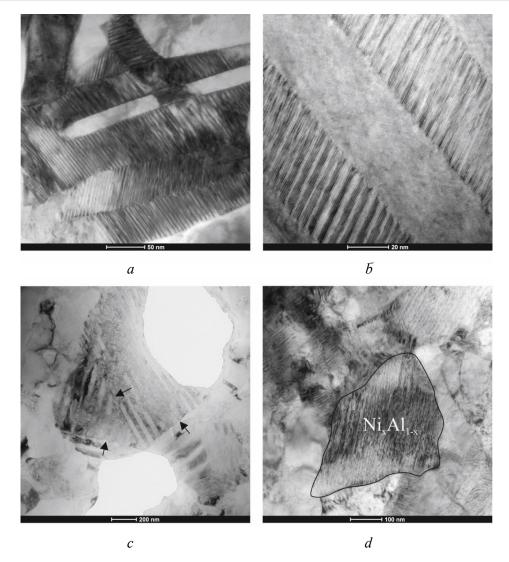


Fig. 4. Bright field TEM images of martensite: a–c – lamellar martensite in coarse grains of Ni_xAl_{l-x} ; d – lamellar martensite in fine grains of Ni_xAl_{l-x}

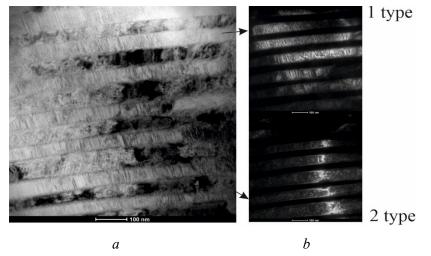


Fig. 5. TEM image of martensite with different types of plates: a – bright field image; b – dark field image



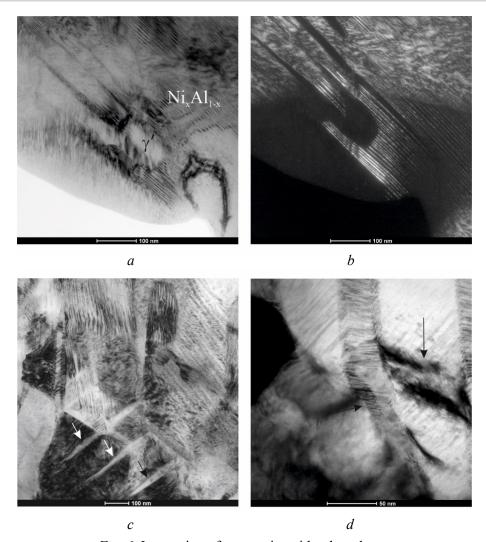


Fig. 6. Interaction of martensite with other phases: a, b – collision of martensite plate with grain γ' - $Ni_3Al; c$ – growth of martensite plates into grain; d – martensite plate deformation; a, c, d – bright field; b – dark field

It was shown above that the temperature of martensitic transformation in Ni-Al alloys is determined by the chemical composition of the material. Coating heating at 400–600 °C allows obtaining information about structural changes in the material. We do not observe any noticeable changes at lower heating temperatures. Fig. 7 demonstrates TEM images of the microstructure of HV-APS coatings after heating at 400 and 500 °C. According to structural studies, the beginning of the reverse transition of $L1_0$ martensite to the B2 structure is observed at 400 °C. In addition, a secondary phase in the form of elongated disks segregates along the boundaries of microtwins (Fig. 7, a). In some cases, only part of the martensite plate undergoes transformation. This can be explained by differences in the chemical composition within the same crystal. An increase in temperature to 500 °C leads to further decomposition of martensite plates and the growth of an already precipitated secondary phase (Fig. 7, b). The secondary phase in grains, where martensite has completely transformed, is oriented in one direction. The formation of the secondary phase is characteristic only for Ni_xAl_{l-x} grains and is absent in grains of the γ' - Ni_3Al phase (Fig. 7, b) and β -NiAl plates (fig. 7, c). The internal structure of β -NiAl plates is characterized by a relatively uniform distribution of dislocations. Finally, heating to these temperatures does not lead to any noticeable structural changes in small grains of the Ni_xAl_{l-x} phase (fig. 7, d).

A significant increase in the width of grain boundaries is observed in two-phase regions after heating at 600 °C (Fig. 8, a, b). The shape of the γ' - Ni_3Al and Ni_xAl_{l-x} grains approaches equiaxial, which indicates the occurrence of recrystallization processes. A growth of the secondary phase with increasing temperature





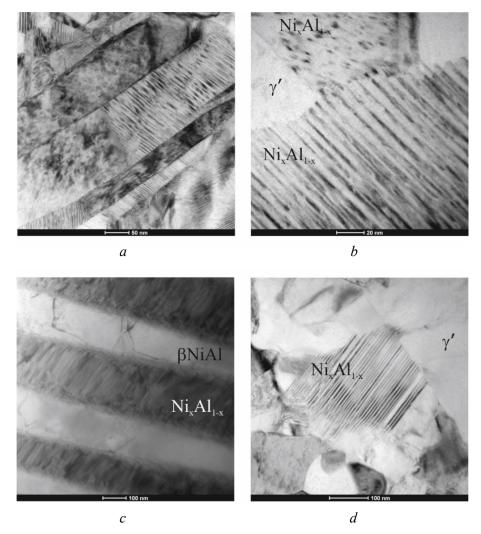


Fig. 7. Bright field images of coating structure after heating 400 (a, c, d)and 500 °C (b)

is not observed. The structure also contains martensite crystals in which the $L1_n \rightarrow B2$ transformation has completely occurred. Fig. 8, c, d shows bright-field and dark-field images of former martensite plates. Data of dark-field analysis confirms the presence of the secondary phase in former martensite plates, the chemical composition of which corresponds to the $Ni_xAl_{1,x}$ phase (Fig. 8, d). The completely transformed plates are separated from each other by low-angle boundaries.

Conclusions

There are two types of particles in HV-APS coatings: with a dendritic and grain structure. The center of particles with a dendritic structure consists of the β-NiAl phase surrounded by a one-phase layer of the Ni_xAl_{l-x} phase and a layer of dendrites (Ni_xAl_{l-x}) with interdendritic space $(\gamma'-Ni_3Al)$. Most of the coatings are particles with a grain structure (Ni_xAl_{l-x} and γ '- Ni_3Al grains).

Only Ni_xAl_{1-x} grains experience martensitic transformation when the particles are cooled. In the large grains, larger than 500 nm, martensite consists of plates in a twinned orientation relative to each other, while small grains are completely transformed into one microtwinning plate. In addition, there are grains in which martensite and β -phase plates alternated.

The collision behavior of martensitic plates is different. Thin plates at collision pass through each other and only the area of its intersection is rearranged. When a martensitic plate collides with an already formed γ' -Ni₃Al grain, the plate continues to transform without penetration. If martensitic plates were formed first, the γ' - Ni_3Al phase is formed around it. In addition, thin plates at collision with an obstacle can be deflected.

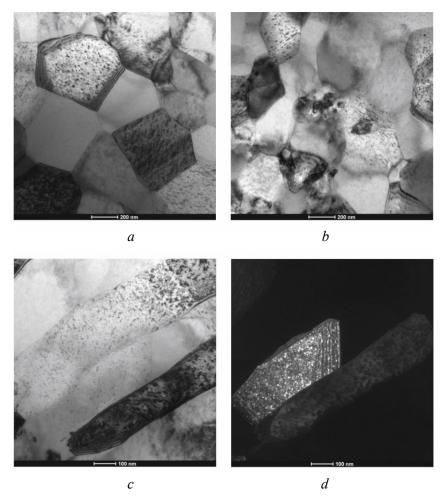


Fig. 8. TEM images of coating structure after heating 600 °C: a, b – two-phase area; c, d – prior martensite plates; a, b, c – bright field; d – dark field

When heated to 400 °C in large grains of Ni_xAl_{1-x} , the beginning of the reverse transition of $L1_0$ martensite to B2 structure with the secondary phase formation along microtwins is observed. No changes are observed in small grains of the Ni_xAl_{1-x} phase, $\gamma'-Ni_3Al$ grains and $\beta-NiAl$ plates. After heating to 600 °C, the shape of the $\gamma'-Ni_3Al$ and Ni_xAl_{1-x} grains approaches equiaxial, which indicates the occurrence of recrystallization processes. The secondary phase is oriented in one direction in Ni_xAl_{1-x} grains. The martensite crystals in large grains are completely transformed into the B2 structure, although it retained its orientation.

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Conflicts of Interest

The authors declare no conflict of interest.

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