Study of diffusion oxide hardening on intermetallic materials

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Abstract. In the last decade, there has been a tendency to increase the use of titanium ortho-
allloys on an intermetallic basis for the manufacture of critical parts. The technology of diffusion
oxide hardening on intermetallic materials to increase hardnes
s is proposed. The results of a
metallographic study of the structure and phase composition of diffusion oxide hardening are
presented. According to the results, the hardness of the VIT-1 intermetallic material in the initial
state is 1250 MPa, and with hardening it is 1563 MPa, which is 25% higher than the base
material.

1. Introduction

Recently, in industry, all attention has been focused on a new class of materials based on titanium
aluminides with high specific strength characteristics, heat resistance, and heat resistance.

In these materials, the main alloying element is niobium, which increases strength, ductility,
toughness and heat resistance. In addition to niobium, these materials also contain Mo, Ta, W, and Zr.
This material is recommended for the manufacture of aircraft engine parts with an operating temperature
of up to 700° C [1-6].

Despite a large number of studies on orthorhombic alloys, the mechanism of ortho-phase formation,
and the study of the occurrence of phase transformations of ortho-alloys over a wide temperature range,
the formation mechanism remains controversial. A set of service properties is formed not only by
obtaining a specific phase composition, but also due to structural characteristics - morphological features
of the structure of phase components, their distribution over the grain body, the presence of grain-
boundary precipitates, etc.

Intermetallic compounds formed by two or more metals having a special crystal lattice, structure and
properties are different from the properties of the starting components. Most intermetallic compounds
have an ordered super structural lattice, the formation of which is associated with the interatomic
interaction of components at temperatures below the melting point. In intermetallic compounds, there
can be metallic, covalent and mixed types of atomic bonds in the crystal lattice. Intermetallic compounds
with a metal bond of atoms are materials with properties similar to those of ductile metals that do not
have high heat-resistant characteristics, and materials with mixed and covalent types of bonds are most
interesting to increase heat-resistant characteristics [7-12].

Thus, these materials, which by their nature have a special crystal lattice, structure, and
physicomechanical properties, are promising heat-resistant materials intended for use in rocket and
space technology and in the field of aviation and engine building, but further research is also required
to study the formation of strong directional bonds, structures with new physicochemical properties [13-
16].
Based on this, in this work, the task was set to study the morphological changes of hardened intermetallic materials on physico-chemical properties.

The aim of the work is to study the diffusion oxide hardening of intermetallic materials on the structure, phase composition, and also on the study of hardness after oxide hardening.

2. Materials and methods
Discrete diffusion hardening was carried out on equipment, which consists of the UIV-1 device with a unipolar positive crown, from a work bed, fasteners, electromechanical units, electrical wiring, air duct, as well as a pressure regulator and flexible hoses (figure 1). Additionally, included is a monitor, computer, driver unit and system software. The UIV-1 device - is a nozzle - an ionizer combining the organization of a directed air flow and its activation by positive ions [17-20].

![Figure 1. Scheme and photo of equipment with a sample mounting mechanism for hardening with the UIV installation: 1 - linear displacement module; 2 - control unit of the stepper motor; 3 - air duct; 4 - case; 5 - fixing rack; 6 - guide.](image)

Samples are fixed in (2) the rotary axis with the control unit of the stepper motor (figure 1). Then set the predetermined frequency of its rotation or the specified feed linear displacement, according to a computer program. The processed sample can perform both rotational and reciprocating motion.

For research used samples from intermetallic materials, in particular VIT-1.

Metallographic studies of diffusion oxide hardening were carried out: the phase composition of the hardening was performed on a Dron 6 diffractometer in Fe-Kα radiation and the chemical composition of the hardening was determined using a JSM-5610 LV electron microscope. The presence of a low-vacuum operating mode of the microscope allows us to study non-conductive inorganic and organic objects without samples of preparation and application of conductive coatings, i.e. receive the image from a real surface. The presence of two types of detectors allows obtaining images in the modes of secondary and backward reflected electrons. The JSM-5610 LV scanning electron microscope is equipped with the EDX JED-2201 chemical microanalysis system, which allows simultaneous automatic qualitative and quantitative chemical analysis of up to 99 areas of interest in the image of the object under study.

Micro X-ray spectral analysis of the chemical composition allows one to determine the content of elements of diffusion oxide hardening with step-by-step scanning of the sample with an electron probe in the direction perpendicular to the side surface of the sample in the cross section of the microsection. The number of scan steps determines the thickness of the coating. X-ray diffraction analysis allows to determine the phase composition. Hardness was measured using a Solver Next microscope with a nanosclerometric head (nanoindenter) in sclerometry mode.
3. Results and discussion
As a result of hardening of the sample from the VIT-1 intermetallic material, the presence of oxides is present on the surface, as evidenced by spectral chemical analysis (table 1).

Table 1. The chemical composition of the hardened sample.

| Spectrum  | O   | Al | Si | Ti  | Zr  | Nb  | Ta  | W   | Total |
|-----------|-----|----|----|-----|-----|-----|-----|-----|-------|
| spectrum 8| 7.83| 9.72| 0.37| 40.65| 2.05| 37.05| 1.16| 1.18| 100.00|
| spectrum 9| 11.02| 9.32| 0.44| 39.49| 1.97| 35.85| 0.93| 0.99| 100.00|
| spectrum 10| 11.47| 9.18| 0.44| 39.31| 2.02| 35.79| 0.81| 0.98| 100.00|
| spectrum 11| 12.49| 9.03| 0.32| 38.91| 1.85| 35.22| 1.05| 1.13| 100.00|
| spectrum 12| 12.95| 8.90| 0.35| 38.69| 1.96| 35.03| 1.01| 1.11| 100.00|
| source base| 0.05| 11.12| 0.19| 43.95| 2.28| 39.9 | 1.16| 0.91| 100.00|

The content of Si, W, Zr, Mo, Ta practically did not change, while it was found that oxygen is present only in the surface layer (12%) (table 1).

The increase in the oxygen content in the coating is explained by the ionization effect achieved by using a corona discharge.

Thus, it can be argued that the decrease in the content of Ti, Al and Nb in the surface layer is due to the oxidation and formation of oxides: titanium oxide (anatase) TiO₂(α), aluminum oxide (corundum) Al₂O₃(α) and the formation of intermetallic compounds Ti₃Al(α₂-phase) and Ti₂AlNb(β₂) having an ordered superstructural crystal lattice.

Intermetallic Ti₃Al (α₂-phase) has an ordered tetragonally curved face-centered structure. It should be noted that oxides have vacancies at the lattice sites, there is a cationic vacancy due to energy fluctuations, one of the atoms can receive enough energy from neighboring atoms to leave the lattice site, i.e. in place of one ion, two ions are formed at two other sites. Thus, any crystal located at a temperature other than zero will always contain a certain number of the indicated thermal defects.

The characteristic microstructure of diffusion oxide hardening on the samples is shown in figure 2.

Figure 2. Microstructure of the surface of the sample with diffusion oxide hardening (x 2000).
The macrostructure showed that this hardening obtained by the diffusion method has a polycrystalline structure. In the mode of back-reflected electrons, the oxygen-saturated surface of the sample has a uniform color (see figure 2), which indicates uniform coverage.

In this case, the structure of the main alloy does not undergo noticeable changes in the surface layer, since the process occurs during ionization of the corona discharge, oxygen ions interact with the atoms of the alloy and form a thin oxide film.

X-ray diffraction analysis obtained the phase composition of the sample from the VIT-1 intermetallic compound (figure 3).

![Figure 3](image-url)

**Figure 3.** Phase composition of the sample: a- before hardening; b- after hardening. X-ray analysis confirmed the presence of oxides: TiO$_2$ (anatase) and α-Al$_2$O$_3$ (corundum) and the formation of intermetalides Ti$_{3}$Al(α$_2$-фаза) and Ti$_2$AlNb(β$_2$).
Metallographic studies were carried out on the colors of maturity; as a result, it was found that the composition of the hardened sample from the VIT1 intermetallic material includes the following oxides: titanium oxide (anatase) TiO$_2$(α); aluminum oxide (corundum) Al$_2$O$_3$(α) and intermetallic compounds Ti$_3$Al(α$_2$-phase) and Ti$_2$AlNb(β$_2$), as evidenced by color discoloration (figure 4).

Thus, it can be argued that the decrease in content – Ti, Al, Nb in the surface layer due to the oxidation process and the formation of oxides: TiO$_2$(α) (anatase) and Al$_2$O$_3$(α) (corundum) and the formation of intermetalides Ti$_3$Al (α$_2$-phase) and Ti$_2$AlNb (ortho-alloy β$_2$).

The oxygen content in the material affects the mechanical characteristics of the coating, in particular hardness. 4 scratches were made on a sample of VIT-1 intermetallic material. Each section was measured in several cross sections (blue stripes) (figure 5).

The measurement results are presented in figure 6.

According to the results, the hardness of the VIT-1 intermetallic material in the initial state is 1250 MPa, and with hardening it is 1563 MPa, which is 25% higher than the base material.
4. Conclusion

The proposed technology of diffusion oxide hardening on an intermetallic material can be different, since for each material the formation of the structure of the hardened material is individual in nature, depending on the chemical composition of the material. Diffuse oxide hardening prevents the propagation of cracks, and, therefore, contributes to an increase in resistance during operation.

5. References

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