HEAT RESISTANT DIFFUSION COATING FOR NIOBIUM ALLOY

by

G.V. Zemskov, R.L. Kogan, V.M. Luk'yanov
HUMAN TRANSLATION

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*yё* initially, after vowels, and after Ь, Ъ; ё elsewhere. When written as ё in Russian, transliterate as yё or ё.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

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Russian English

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HEAT RESISTANT DIFFUSION COATING FOR NIOBUM ALLOY

G. V. Zemskov, R. L. Kogan, and V. M. Luk'yanov

The question of protecting niobium and its alloys from high-temperature corrosion is a current one at the present time. Diffusion coatings [1, 2] are receiving ever more widespread application for this purpose. Silicide diffusion coatings possess high durability [3]. Much attention is being given abroad to a coating of chromium, titanium and silicon obtained by the method of vacuum treatment in an alloy of titanium with chromium, with subsequent siliconization [4, 5]. However, the indicated works do not present the results of the study for the purpose of selecting the optimal technological process ensuring a diffusion layer of maximal durability.

We studied the possibility of using a simpler technological process as compared with that described [4] for obtaining a tri-component coating containing titanium, chromium and silicon and protecting the niobium against oxidation at a temperature of 1100-1200°C.

Diffusion saturation is obtained from powdered mixtures of elements in containers with seal in the absence of a vacuum.

The tri-component layer was obtained by a sequential method: First the surface was saturated jointly with Cr + Ti in a definite ratio of these elements. The second stage in the technological process was siliconization.
Fig. 1. Dependence of depth of two-component (Cr+Ti) coatings on the ratio of Cr and Ti in the saturation mixture (t=1200°C).

1 - \( \gamma = 5 \); 2-10; 3 - 15 hrs.

The chromium-titanium coating was performed at temperatures of 1200°C* and duration of 5, 10 and 15 hours. The saturation mixture contained 70% metallic component (Cr+Ti), 25% Al₂O₃ and 5% NH₄F. The application of ammonium fluoride ensured a high-quality surface layer.

The change in basic characteristics of the obtained diffusion layer on the content of chromium and titanium in the saturation mixture was studied, and a composition was selected which would ensure the simultaneous diffusion of these elements in the alloy.

* The temperature of the process was selected based on the temperature of recrystallization of the alloy.
Fig. 1 shows the dependence of the depth of the diffusion layer on the ratio of titanium and chromium in the saturation mixture. With increase in the content of titanium in the charge, the depth of the diffusion layer increases. The samples treated in mixtures containing from 20 to 80% titanium were distinguished by their microstructure, microhardness, and by their phase composition from chrome-plated ones, as well as from titanium-coated samples. This confirms the joint diffusion of chromium and titanium in them. In subsequent tests, a holding time of 15 hours was taken for chromium-titanium coating. This ensured obtaining a coating with maximal depth of 25-30μm.

Silicon coating was performed at a temperature of 1170°C from a mixture containing 60-70% Si. To select the optimal conditions of the siliconization process, we studied the effect of the amount of activator (NH₄Cl) on the depth of the diffusion layer. Fig. 2 presents this dependence for a process duration of 1 and 5 hours. With increased content of halogenide in the saturation mixture, we observed an increase in the depth of the layer. In subsequent tests, in order to avoid silicon coatings with a depth of over 30-40μm, we introduced no more than 1% NH₄Cl into the saturation mixture, and limited the holding time during siliconization to 1 hour.
Fig. 3. Dependence of basic characteristics of tri-component coatings (Cr+Ti, Si) on the ratio of Cr and Ti in the saturation mixture during chromotitanization.

1 - depth of layer after chromotitanization \((t=1200^\circ \text{C}; \gamma = 15 \text{ hrs.})\); 2 - depth of layer after chromotitanization and subsequent siliconization \((t=1170^\circ \text{C}; \gamma = 1 \text{ hour})\).

- **a**: Depth of layer, \(\mu \text{m}\);
- **b**: Microhardness, \(\text{Mn/m}^2\);
- **c**: Weight gain, \(\text{kgm} \cdot \text{sec} \cdot 10^6\);
- **d**: Content of Cr and Ti in saturation mixture, weight \%;
- **e**: Distance from surface, \(\mu \text{m}\);
- **f**: Content of Cr and Ti in saturation mixture.
Fig. 4. Microstructure of tri-component coating (Cr+Ti, Si), obtained according to optimal technology. Magnification x340.
a - before testing; b - after testing.

The high heat resistance of the complex coating containing chromium, titanium and silicon is provided by the complex silicides which are formed during siliconization of the chromotitanized layer. Our studies showed that the heat resistance of the obtained tri-component coatings (Cr+Ti, Si) depends primarily on the ratio of chromium and titanium in the layer, obtained during joint chromotitanization. The effect of this ratio on the basic characteristics of the tri-component diffusion coatings (Cr+Ti, Si): on the depth, microstructure, microhardness distribution by depth of layer and heat resistance* is illustrated in Fig. 3.

* Heat resistance was tested at a temperature of 1200°C for 12 hours and determined by weight gain.
Fig. 5. Dependence of weight gain of samples on time of testing at 

$\theta=1250^\circ \text{C}$.

1 - Cr+Ti--Si+15% V, in saturation mixture; 2 - Cr+Ti--Si+20% V, in 
saturation mixture; 3 = Cr+Ti--Si. $a$ - Weight gain, kg/m $\cdot$ sec $\cdot 10^6$. 
b - hours.

From the presented graphs, we see that the most heat resistant 

tri-component coating is obtained by means of siliconization after 

chromotitanization in a charge containing 60% Cr and 40% Ti. By its 

microstructure (Fig. 4,a) and microhardness distribution (Fig. 3), this 

coating is distinguished from coatings obtained with analogous 

technology, but with a different ratio of chromium and titanium in the 

charge during chromotitanization.

Three zones are clearly delineated in the microstructure and on 

the curve showing change of microhardness by layer depth. The middle 

and inside zones of the coating possess the greatest hardness. The 

hardness of the middle zone with increase in titanium content in the 

saturation mixture to 40-50% increases, and then declines. The 

hardness of the outside zone of the tri-component coating does not 
depend on the conditions of the chromotitanization process, while that 
of the inner zone declines with an increase of titanium in the charge.
In the microstructure of the coating obtained with optimal ratio of chromium and titanium, after testing (Fig. 4,b) we found an oxidized zone which encompasses less than half its depth. As a result of testing, the depth of the coating increased from 56 to 75 μm. The microhardness of the coating after testing comprises $H_v = 1389 \text{ Mn/m}$, i.e., it does not differ from the initial value.

We also studied the effect of adding vanadium on the heat resistance of the studied tri-component diffusion layer (Cr, Ti and Si). For this purpose, vanadium was introduced into the saturation mixture during the siliconization process, in the amount of 5-30% of the amount of silicon. The obtained four-component layers on alloy VN-2 (Cr, Ti, Si and V) were subjected to heat resistance testing at a temperature of 1250 °C until the appearance of superficial defects which led to disintegration. Fig. 5 presents the dependence of the change in weight gain of the samples on the time of testing for layers obtained from the saturation mixtures without vanadium and with its optimal content of 15-20% V. The introduction of vanadium, as we can see from the graph, reduced the number of point defects appearing in the process of testing the samples, and therefore increased their durability.

Conclusions

1. The process of complex surface alloying of alloy VN-2 with chromium, titanium and silicon was studied. The optimal ratio of saturating elements ensuring maximal heat resistance of the alloy was determined.
2. A technology was developed for obtaining a complex diffusion coating on alloy VN-2 with chromium, titanium and silicon in powder
mixtures of elements, in containers with fusible seals in the absence of a vacuum, which considerably simplifies the existing technology.

3. It was found that the introduction of vanadium into the saturation mixture with tri-component alloying of the VN-2 alloy (Cr, Ti and Si) increases the durability of the obtained diffusion layer.

Literature