Influence of titanium and chrome on the structure and heat resistance of layered composites from a multicomponent Nb-alloy and intermetallic compounds of aluminium

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Abstract. Investigations of the structure and measurements of the strength characteristics of layered composites of niobium alloys prepared by diffusion welding under pressure have been carried out. It was found that titanium has a strong effect on the formation of microstructure and strength of composites. Higher values of short-term strength and fracture toughness are demonstrated by layered composites with a lower titanium content.

1. Introduction
In several structural materials of modern science, a special place is occupied by heat-resistant alloys, among which alloys based on niobium and its intermetallic compounds are promising. They can possess high mechanical properties up to 1400–1500 ºC. Alloys based on the Nb–Al system stand out.

One of the most promising methods for producing heat-resistant alloys based on the Nb–Al system are technologies for creating composites with a layered structure, developed by foreign [1–5] and Russian scientists [6, 7]. The widespread methods are high-speed magnetron sputtering [1–6] and combined methods for obtaining layered structures [3, 5]. But in all layered composites, layers consisting of strong intermetallic compounds alternate with layers of a relatively plastic Nb solid solution. In addition to the above-mentioned methods of obtaining (Nb–Al) alloys, there is a method that combines diffusion welding and rolling of already welded packages.

For heat-resistant composites, it is important to combine high strength at elevated temperatures with satisfactory crack resistance, which characterizes their viscous-plastic state under normal conditions. On the example of alloyed steels, it has long been known that toughness and plasticity are acquired by them with the addition of vanadium. For this purpose, similar studies have been undertaken in this work. This should be facilitated by the Nb–V phase diagram, which is characterized by continuous mutual solubility of the components in liquid and solid states.

2. Materials and experimental technique
The studied composites made of Nb alloys foils of two compositions, differing in the contents of Ti, Cr, Zr, Mo and Si, but to a greater extent in the contents of titanium (Table 1). The fact is that for smelting of both alloys, we used charges of the same composition. The compositions became different after the alloys were smelted by different methods. The first initial niobium alloy (Nb1) (see Table 1) was obtained by melting in suspension (MiS) with pouring the melt into massive cylindrical Cu-crystallizers
and subsequent 2-fold electron-beam remelting (EBR) in a stream water-cooled Cu-crystallizer. The second alloy (Nb)2 was obtained using only the MiS-method with pouring the melt into a flat Cu-mold.

It turned out that after MiS the ingots contained 85.44 wt.% Nb, and after MiS+EBR – 87.70 wt.% Nb. An increase in the concentration of niobium in the alloy was due to the almost complete evaporation of titanium during electron-beam remelting, complete evaporation of chromium and partial evaporation of silicon, as, like aluminum, an intermetallic-forming element with metals. After smelting, the resulting ingots 6.5–8.0 mm thick were rolled on a vacuum rolling mill at 1000–1300 °C with 20% reduction to a thickness of 2 mm. Further rolling of strips to 0.1 mm was carried out already at room temperature.

| Alloy | Method     | at. or wt.% | Nb  | Ti  | Cr  | Zr  | Mo | Hf | Si |
|-------|------------|-------------|-----|-----|-----|-----|----|----|----|
| (Nb)1 | MiS+EBR   | at.%        | 89.08 | 0.27 | 0.01 | 0.73 | 5.40 | 2.76 | 1.03 |
|       |            | wt.%        | 87.70 | 0.14 | 0.01 | 0.71 | 5.49 | 5.22 | 0.31 |
| (Nb)2 | EBR       | at. %       | 82.11 | 7.72 | 0.79 | 0.19 | 3.26 | 2.53 | 3.00 |
|       |           | wt. %       | 85.44 | 4.14 | 0.46 | 0.20 | 3.50 | 5.06 | 0.94 |

From pieces of rolled strips of Nb-alloys with a thickness of 100 μm and strips of alloy Al–2 wt. % Si 25 μm thick, multilayer packages (Nb)1/AlSi and (Nb)2/AlSi 2 mm thick were assembled.

The formation of the layered structure of the composite was carried out in the process of diffusion welding under pressure (Nb)/AlSi-packages. Welding was carried out in an installation, which has a water-cooled body and a high-strength graphite heater, designed for temperatures up to 1700 °C.

To obtain the creep characteristics, a series of experiments was carried out in which the deformation of the sample was measured under the action of discretely increasing loads. The sample was held for 10–15 h at each load. The calculation was carried out according to the method proposed in [8, 9].

Microstructure studies and local X-ray spectral analysis were performed on Tescan VEGA-II XMU and CamScan MV230 digital scanning electron microscopes. Tests of specimens for short-term, with a loading rate of 0.5 mm / min, and long-term strength were carried out according to the 3-point bending scheme in a vacuum chamber combined with an Instron testing machine in an atmosphere of high purity argon.

3. Experimental results

3.1 Microstructure of composites

The melted and rolled alloys had a homogeneous structure of solid solutions based on niobium without any other structural components.

The layers inheriting the alloy foils, after diffusion bonding, are identified as an Nb-solid solution of mainly aluminum and silicon in niobium. Its layers contained light inclusions of regular shape 1–2 μm in size (shown by arrows, see Figure 1a), identified as HfO2, and a large number of pores of various configurations formed in place of brittle inclusions, the composition of which remained unknown.

The microstructure of the (Nb)2/AlSi composite prepared from alloy 2 (Figure 1b) significantly differed from the structure of the composite with the (Nb)1/AlSi alloy. At this stage of solid-phase interaction, the composite contains two structural components that do not have a sharp boundary between them. These are “layers” of the Nb solid solution (points of analysis 3, 4, 8–12, and 15) and 2-phase “layers” (points of analysis 1, 2, 5–7, 13, and 14), consisting of intermetallic compound (Nb,Me)2Al in the form of a gray matrix and light threadlike or cellular formations from an Nb-solid solution.

Summing up the results of studying the microstructure of the (Nb)2/AlSi composite, we can say that it consists of two alternating 2-phase structural components (structural formation) – (Nb,Me)3Al + Nb-s. s. and Nb-s. s. + (Nb,Me)3Al. One is the (Nb,Me)3Al aluminide matrix, which contains elongated thin sections of the Nb-solid solution inclusions. Another structural component is large grains consisting of
an Nb-solid solution and (Nb,Me)$_3$Al, with boundaries also from an intermetallic compound (Nb,Me)$_3$Al.

![Microstructures of layered composites](image)

**Figure 1.** Microstructures of layered composites (Nb)1/AlSi (a) and (Nb)2/AlSi (b): 1–18 and 1–14 – points of analysis.

The third structural object, as in the (Nb)1/AlSi composite, includes finely dispersed HfO$_2$ inclusions, which in this composite are mainly localized in the (Nb,Me)$_3$Al compound.

### 3.2 Mechanical test results

The dependences of the short-term strength of composites on the test temperature in the range from room temperature to 1300 °C are shown in Figure 2. The changes in strength of the (Nb)1/Al and (Nb)2/Al composites differ significantly. The ultimate strength $\sigma_B$ of the 1st composite at room temperature was 930 MPa, retained a high value equal to 850 MPa at 1100 °C and only at 1300 °C fell to 420 MPa. Composite (Nb)2/AlSi already at room temperature had an average strength of only 370 MPa and practically the same – 375 MPa – at 1300 °C.

Crack resistance $K^*$ of composites with alloys 1 and 2 at room temperature turned out to be equal to 12.5–13.0 and 8.5–9.0 MPa·m$^{1/2}$, respectively. The $K^*$ values differ so much that the fracture patterns are clearly different.

The results of creep tests of composites at 1300 °C are shown in Figure 3. For the (Nb)1/AlSi composite, the 100-hour strength or stress value causing 1% deformation per 100 hours is 8 MPa with an exponent $n = 0.42$. The composite 2 had a lower 100-hour strength of 7 MPa with a higher $n$ value of 0.77.

### 4. Discussion of the results

The analysis of the results obtained is reduced to the effect of Ti and Cr (and, perhaps, Si) on the structure and strength characteristics of composites based on niobium alloy. The alloy, from the foils of which the packages for (Nb)1/AlSi composite were assembled, contained ~0.3% Ti, ~0% Cr, and 1 at. % Si. The (Nb)2/AlSi composite was made from Nb alloy foils with 7.7% Ti, ~0.8% Cr, and 3 at. % Si (see Table 1). It followed from microstructural studies that the presence of these elements accelerated the reactive diffusion between the layers of the Nb alloy and the Al–Si alloy. This resulted in a relatively low volumetric content in the (Nb)2/AlSi composite of the structural component with the Nb-solid solution. According to the results of processing the color image of the surface area of a thin section in the composite instead of Nb-alloy tapes after diffusion welding, about 30 volume percent of the structural component [Nb-solid solution + (Nb,Me)$_3$Al], partially containing viscous plastic (Nb)-solid solution. This amount was extremely insufficient to resist crack propagation in the composite. Hence – the brittle nature of the destruction of the (Nb)2/AlSi composite, its low crack resistance and underestimated, in relation to (Nb)1/AlSi, strength values. The composite became so brittle that it lost its ability to resist bending deformation not only at room temperature, but also at temperatures up to 1300 °C. Therefore, we observed the "independence" of the ultimate strength of the composite from the
test temperature, except for an increase in the average value of $\sigma_B$ by 5 MPa at 1300 °C in relation to the ultimate strength at room temperature.

The layered composite (Nb)1/AlSi, containing ~50 volume percent of the Nb-solid solution, in contrast to (Nb)2/AlSi, was destroyed by the mechanism of the formation of longitudinal cracks, which propagated along the intermetallic layers as less crack-resistant structural components.

![Figure 2. Dependences of short-term bending strength $\sigma_B$ on the test temperature for composites (Nb)1/AlSi (1) and (Nb)2/AlSi (2).](image1)

![Figure 3. Dependences of the creep strain rate on stress for the (Nb)1/AlSi (1) and (Nb)2/AlSi (2) composite at a temperature of 1300 °C.](image2)

The mechanical properties of layered composites during short-term tests and during creep tests turned out to be noticeably lower than those of highly alloyed eutectic alloys of the Nb–Si system under similar conditions. Crack resistance of layered composites and melted Nb–Si alloys had, in general, the same values [10]. However, if in cast Nb-alloys the results are maximally achieved, then in composites they were obtained for the first time and there are opportunities to significantly improve them.

5. Conclusions
1. It was found that titanium and chromium accelerated the reactive diffusion of aluminum in a solid solution based on niobium. The layered structure of the (Nb)2/AlSi composite with a Nb alloy containing slightly more than 4% Ti and ~0.5 wt. %Cr had two structural components separated by diffuse boundaries. One of them consisted of the (Nb,Me)3Al intermetallic compound with extended inclusions of an Nb-solid solution. Another structural component was from coarse grains of the Nb-solid solution, which contained the compound (Nb,Me)3Al in the form of inclusions and along the grain boundaries.

2. The nature of the change in strength depending on temperature in composites was significantly different. The ultimate strength of the composite with a low titanium content at room temperature was 930, at 1100 °C – 850, and only at 1300 °C it fell to 420 MPa. Composite with 4.14 wt. % Ti already at room temperature had a strength of only 370 and practically the same – 375 MPa – at 1300 °C.

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