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MULTILAYER STRUCTURES AND HIGH-TEMPERATURE STRENGTH OF REFRACTORY MATERIALS BASED ON NIOBIUM COMPOUNDS WITH ALUMINUM AND SILICON OBTAINED FROM Nb–Al- AND Nb–Si-COMPOSITES

Diffusion method for production of multi-layer artificial heat-resistant materials with the structure consisting of alternating layers of intermetallic compound of niobium with aluminum or silicon and solid solutions based on niobium [Nb(Al) or Nb(Si)] was proposed. Layers of high-strength, but brittle in nature, intermetallic are able to give the material strength at high temperatures, and the layers of the solid solution provide crack resistance under normal conditions. This layered structure was obtained by heat and pressure treatment of packages of thin Nb and Al foils or packages of Nb foils with one- or donble-sided Si coating. The desired structure was formed as a result of inter-diffusion between niobium and aluminum or silicon. In fact, diffusion welding (DW) of the package occurred with the formation of layers of heat-resistant compounds of Nb_3Al or Nb_5Si_3. Welding temperature was ~ 1700°C, the time was as little as 30 min, and the pressure was from 6 to 12 MPa. Welding was performed in vacuum. The package was placed between two punches of high-resistant graphite. In the case of Nb–Al packages, DW was combined with the method of packet rolling (PR), so the welding was applied to layered niobium-aluminum foils, originally containing thinner (of the order of hundreds of nanometers) layers of Nb and Al. For more effective quenching of cracks, the material composed by intermetallic multilayers, located between the layers of almost pure niobium with the thickness of ~ 90 μm was obtained. Composites of Nb–Al produced by the DW and «PR + DW» and composites of Nb–Si in the end are massive plates of size ~35 × 40 mm^2 and of 2.5–3.5 mm thick. The formation of chemical compounds in the Nb–Al-composites occurred in the sequence from the intermetallic compound with higher content of Al to intermetallic compounds with lower content – $NbAl_3 \rightarrow Nb_2Al \rightarrow Nb_3Al$. With limited sources of Nb and Al, the completed result of diffusion was the structure with intermetallic compounds containing less aluminum. It was found that $NbAl_3$ was easily and quickly formed at temperatures below 1000°C, and at >1500°C it was completely consumed. Process of mutual diffusion was the more complete, the more exposure time, and the faster, the higher the temperature. Therefore, the evolution of the multilayer structure from $Nb_2Al + Nb_3Al + Nb(Al)$ to $Nb_3Al + Nb(Al)$ at DW temperature increasing from 1500 to 1700°C seemed regular. Bending tests at temperatures up to 1400°C showed that the material with such a structure can have the strength of 200–300 MPa at 1200–1350°C. The proposed technological approach provides promising prospects for heat-resistant material on the basis of niobium alloys with Al and Si, able to operate at 1300°C and higher.

Keywords: refractory materials, layered composites, diffusion welding, package rolling, intermetallic compound, bending strength

Fig. 1. Microstructure (I) and the results of local X-ray analysis (II) of multi-layer Nb–Al composite after DW: *а* – 550°C/1 h + 1500°C/30 min, ~ 6 MPa; *б* – 550°C/1 h + 1700°C/30 min, ~ 12 MPa; *в* – 550°C/1 h + 1700°C/30 min, ~ 6 MPa

Fig. 2. The cross-section structure of multilayer composites Nb–Al of two constructions after diffusion welding at 1700°C for 30 min under pressure of ~6 MPa

Fig. 3. General view of the structure of the composite in a section parallel to the rolling direction (*a*) and the microstructure with the results of local X-ray analysis (*b*)

Fig. 4. Test temperature dependences of short-term bending strength: *а* – a composite obtained by DW, Fig. 1,*в* and 3,*а*, (1700°C, 30 min, 6 MPa); *б* – multilayer plates produced under the same conditions by the method of «PR + DW» (12NbAl1 (■) and 12NbAl2 (□))

Fig. 5. Macrostructure of multilayer composite Nb–Si after DW

Fig. 6. Test temperature dependences of short-term strength in the 3-point bending tests of multi-layered composites (12NbSi2 (□), 12NbSi3 (■), 12NbSi6 (◆) and 12NbSi7 (◊) – obtained by DC of packages of Nb-foils with one-sided Si-coating of average thickness in the range of 35–50 μm, under the same conditions