

Structural inhomogeneity in ceramics produced by oxidizing of one-dimensional zirconium samples in a nitrogen atmosphere

*A. V. Shokodko¹, S. V. Shevtsov¹, A. I. Ogarkov¹, I. A. Kovalev¹, S. V. Kannykin²,
A. A. Konovalov¹, D. V. Prosvirnin¹, E. A. Shokodko³, A. S. Chernyavskii¹, V. M. Ievlev^{2,4},
K. A. Solntsev^{1,4}*

¹Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences, Moscow, Russia

²Voronezh State University, Voronezh, Russia

³Moscow State University of Civil Engineering (National Research University), Moscow, Russia

⁴Lomonosov Moscow State University, Moscow, Russia

imet@imet.ac.ru

In accordance with SEM images of a transverse fracture surface of samples, zirconium nitridation for 4 min leads to the formation of a layered structure. Two polycrystalline nitride layers and one layer of a nitrogen solid solution in zirconium can be distinguished morphologically with certainty according to their thickness and grain size. The surface nitride layer formed as a steady-state synthesis temperature was approached is 4 to 10 μm in thickness, with a grain size of 5 μm . In the 35- to 40- μm -thick nitride layer formed at a constant temperature, the grain size approaches its thickness. The layer of the nitrogen solid solution in zirconium consists of grains up to 15 μm in length and up to 2.5 μm in width. Synthesis for 6 min increases the thickness of both the surface and inner nitride layers (to 20 and 100 μm , respectively), which is accompanied by an increase in the grain size of the polycrystalline phase of the nitrogen solid solution in zirconium to 150–170 μm . At synthesis times from 6 to 16 min, the thickness of the solid solution layer decreases, whereas its surface morphology experiences no significant changes. After 21 min of synthesis, no nitrogen solid solution in zirconium was detected in the central part of the sample in an SEM image. The surface nitride layer grows slowly, reaching 40 μm . The inner nitride occupies the entire residual volume of the sample. The central part of the sample has a sharp interface, which resulted from coalescence of two reaction fronts that moved across the flow direction. Heating for 60 min is only accompanied by the growth of the surface nitride layer, which reaches 80 μm in thickness. Note that the grain size in this layer remains smaller than that in the inner nitride layer. The disappearance of the sharp interface in the central part of the sample is caused by recrystallization processes in the bulk of the nitride and also possibly by the sintering of grains similar in orientation. The thickness of the inner nitride layer increases linearly with time up to 21 min, that is, as long as the solid solution of nitrogen in zirconium persists in the bulk of the sample. The surface nitride layer grows over a period of 6 min. At synthesis times from 6 to 16 min, its thickness varies little. Subsequently (after 21 min), the thickness of this layer increases linearly on account of the depletion of the inner nitride layer. This work was supported by the Russian Science Foundation, project no. 14-13-00925 (federal state budget funded science institution Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences).