

Nanocomposites and nanomaterials

Synthesis of single crystal alumina whiskers

**A.M. Verkhovliuk, O.A. Shcheretskiy, R.A. Sergiienko, V.V. Apukhtin,
D.S. Kanibolotsky**

*Physico-Technological Institute of Metals and Alloys, Natl. Acad. of Sci. of
Ukraine. Akad. Vernadsky Avenue, 34/1, Kiev-03680, Ukraine.*

E-mail: kanibolotsky@univ.kiev.ua

Metal oxide single crystal whiskers are used as reinforcements in composites. One of the most perspective materials for the whiskers production is α -alumina. It is reported a theoretical and experimental investigation of the α -alumina single crystal whiskers synthesis using interaction between pure molten Al and one of the oxides: Al_2O_3 , SiO_2 , Fe_2O_3 and NiO. The mechanism of the synthesis includes oxidation of pure Al to form gaseous Al_2O and subsequent disproportionation of the latter with Al_2O_3 and Al formation. The Gibbs free energy of reactions $2n\text{Al} + \text{MeO}_x = n\text{Al}_2\text{O} + \text{MeO}_{x-n}$ and $2n\text{Al} + 3\text{MeO}_x = n\text{Al}_2\text{O}_3 + 3\text{MeO}_{x-n}$ has been calculated using reference thermodynamic data (Me is Al, Si, Fe or Ni, x and n are integers, $n \leq x$). It was taken into consideration, that the Fe_2O_3 and Fe_3O_4 are unstable above 1566 °C and 1538 °C, respectively, and decompose to form FeO. The Gibbs free energy of the Al_2O formation is negative in all the cases at 1500 °C and 1600 °C, but the Al_2O_3 formation is more thermodynamically favorable than the Al_2O if the NiO is used as an oxidizer. Powder mixtures of pure Al with Fe_2O_3 , NiO, Al_2O_3 and SiO_2 were pressed into tablets, which were placed into alumina crucibles, and annealed at 600 °C for 2 hours, finally heated in an induction furnace up to 1600 °C and soaked for 15 minutes. The experiments were conducted under argon atmosphere as well as under air with prevalence of CO. In the second case the alumina crucible with the tablets was placed in a massive graphite crucible which is not tightly closed by a graphite cap. A white vapor (presumably of Al_2O) was escaping during the experiments with Fe_2O_3 and SiO_2 and amorphous Al_2O_3 was deposited on the graphite cap and on the internal walls of the furnace chamber. After the furnace was cooled down to room temperature, the tablets with Fe_2O_3 and NiO were etched by 10 % HCl and the specimens with Al_2O_3 and SiO_2 were etched by 5 % NaOH. Then the specimens were analyzed using REMMA-102 Scanning Electron Microscope. Single crystal Al_2O_3 whiskers 0.05-7 μm in width and 10-3000 μm in length have been revealed in the experiments with Fe_2O_3 and SiO_2 . A larger number of the whiskers have been obtained using the Fe_2O_3 as an oxidizer. The whiskers were not synthesized in the experiments with Al_2O_3 , despite the process is thermodynamically favorable. Probably, kinetic impediments occurred and a higher temperature is required for the reaction.