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Rola mechanizmów deglomeracji nanorurek węglowych w kształtowaniu mikrostruktury i właściwości kompozytu z osnową magnezową

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ABSTRACT

The role of carbon nanotubes deglomeration mechanisms in magnesium matrix composite microstructure and properties formation

Thesis analyze the possibility of Multi-Walled Carbon Nanotubes (MWCNTs) nanocomponent deglomeration in magnesium matrix composites fabrication process in the form of sinters and composite rods obtained from sinters. Study of the issue have shown that a key problem in the consolidation of composites reinforced with nanocomponents including MWCNTs is their tendency to agglomerate. In order to limit this problem, solutions based on sonication and plastic forming in technological processes are used. A new solution, so far not described in the literature is the possibility of using SHS reaction and extrusion assisted by cyclic rotation of a die as an effective method of nanocomponents deglomeration, and these issues were the main focus in research.

Experimental work included the binary (Mg-MWCNTs) and ternary (Mg-MWCNTs-nanometric Si) mixtures preparation of micrometric Mg powders with nanometric by the sonication method in the liquid, followed by their subsequent consolidation by sintering under pressure in vacuum. For Mg-MWCNT sinters, extrusion with a reversible die (KOBO method), which is one of the SPD methods allowing fabrication of fine grain material, was additionally applied. During sintering of Mg-MWCNT-(n)Si mixture, the highly exothermic reaction of Mg with Si took place and Mg_2Si silicide was formed which was the additional phase in the composite.

In order to determine the most favorable procedure for deglomeration of nanocomponents and formation of powder mixtures, several variants of sonication were tested. It was shown that separate deglomeration of nanocomponents and subsequent mixing with Mg was the most effective method for obtaining ordered mixtures of micrometric and nanometric powders.

Composites with a multiphase skeleton were obtained from sintered powder mixtures consisting of magnesium powder coated with a mixture of MWCNTs and (n)Si with MWCNT:(n)Si ratios of 1:2, 2:4 and 3:6. In the Mg-based matrix, a Mg_2Si-MWCNT-MgO-Mg skeleton was formed, along with individual Mg_2Si particles of various sizes including those containing MWCNTs. The intense SHS reaction between (n)Si and Mg induced the deglomeration of MWCNTs. The proportion of 6 vol.% (n)Si and 3 vol.% MWCNTs in the

magnesium-based powder mixture resulted in a two times increase in the hardness of the composite compared to the reference magnesium sinter. In addition, a reduction in Mg_2Si particle size was found with an MWCNTs and (n)Si volume fraction increase. The presence of MWCNTs in both the Mg_2Si phase and the multiphase skeleton was shown. No chemical degradation of MWCNTs and formation of SiC possible during Mg_2Si synthesis of SHS type was observed during composite and isolate synthesis by XRD and HREM methods.

Plastic forming of Mg-MWCNT sinters and reference materials by the KOBO method without preheating and after heating to 150°C produced 8 mm diameter rods. The composite rods were characterized by lower porosity compared to materials extruded from Mg powder, and heating up the material before extrusion to 150°C further reduced it.

Strength tests of the composite rods showed their increased stiffness and hardness compared to all reference magnesium rods and higher tensile strength compared to magnesium powder-based rods. No increase in strength was found with an increase in the volume of MWCNTs in the material. Increasing extrusion temperature to 150°C resulted in increase of composite bar strength and their ductility.

During mixing of Mg with MWCNTs, secondary controlled agglomeration of MWCNTs on the surface of Mg powder occurred. A MWCNT/MgO skeleton was formed in the sintered composite which under the influence of KOBO was transformed. In addition to elongation of skeleton's cells the possibility of its segregation in the Mg matrix on the cross-section was demonstrated depending on the process conditions. MWCNTs were oriented and fragmented as a result of extrusion. A small amount of MWCNTs migrated into the metal matrix under the influence of magnesium flow during plastic deformation.

Both the presence of the MWCNT/MgO skeleton and the presence of MWCNTs in the MgO matrix affected the recrystallization effects of metal matrix. The Mg grain size was smaller than in the reference materials, and its shape was determined by the vicinity of the MWCNTs.