Nanocomposite materials based on carbon nanotube reinforced alumina matrix

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Due to high chemical resistance and perfect mechanical properties carbon nanotubes (CNT) are a promising addition for production of composite materials with improved properties. Reinforcing by CNT even in a small content drastically alters the properties of polymers and ceramics: impart the electro conductivity, improve the thermal conductivity, and increase the mechanical, chemical properties, in particular the fracture toughness.

The present work is devoted to the development of alumina – CNT composite fabrication using two different technologies, namely vacuum sintering and spark plasma sintering (SPS) techniques. The study included the investigation of type of CNT, their concentration and sintering parameters influence on the microstructure of fabricated material.

The multiwall carbon nanotubes (MCNT) prepared by different ways were using for implantation into ceramic material. CNT concentrations up to 24% vol. and 50 % vol. were used for composite fabrication by vacuum sintering and SPS techniques correspondingly. The CNTs were treated in acids to remove particles of the catalyst and amorphous carbon impurities.

MgO-doped Al_2O_3 was used as the ceramic matrix. The addition of MgO, which is widely used in practice in the amounts 0.2 – 1.0 wt.% for preparing strong corundum articles, does not lower the sintering temperature, but it has a considerable effect on the crystallization of alumina, slowingdown grain growth, and also facilitates the acquisition of a more isometric shape by corundum crystallites.

The charge for the ceramic matrix composite Al_2O_3 doped with 0.25 wt.% MgO was fabricated by solid-phase synthesis at 1100°C from aluminumchloride and magnesium nitrate and subjected to mechanical activation in a planetary mill. Carbon nanotubes were introduced in amounts up to 2 vol.% in the form of a suspension by pouring into the comminuted Al_2O_3-MgO mixture. To achieve effective separation of the bundles and a uniform distribution in the material the nanotubes were dispersed by ultrasound (20 kHz) in ethanol with addition of sodium laureth sulfate (concentration 3 g/liter) as the surfactant.

The experimental samples were formed by semi-dry uniaxial pressing (pressure 150 MPa) in the form of small slabs with dimensions 40∗4∗4mm in a hydraulic press. Different combinations of the concentrations of the CNTs introduced and the calcination regimes for the samples were used to obtain the composite. The calcinations was performed in vacuum under residual pressure 10^{-4} mm Hg, varying the heating regime and the maximum calcination temperature in the range 1700 – 1730°C.Process of sintering also includes the holding time at 1450°C for one or two hours.

Spark-plasma sintering was another technique used in this study. SPS is considered the leading technique for composite consolidation. This technique is a pressure assisted fast sintering method based on a high-temperature plasma (spark plasma) that is momentarily generated in the gaps between powder materials by electrical discharge during on-off DC pulsing. Fully dense multi-wall CNT/Al_2O_3nanocomposites were fabricated using SPS. CNT concentrations up to 50% vol. were used for fabrication of composites by SPS method. The heating rate during sintering varied from 200 to 380°C/min. Maximum temperature of calcination was 1500?1600°C with isothermal exposure at maximum temperature during 3 min. As a calcination atmosphere inert gas (argon) and vacuum were using.

Ceramic matrix composites fabricated by vacuum sintering without application of pressure demonstrated uniform morphology with a small grain sizes (3 – 5 microns), zero open and closed porosity and flexural strength increasing up 2 times compared to initial alumina and achieves 400 -500 MPa depending on particular type of CNT and their concentration; at the same time fracture toughness increases about 2.5 times exceeding8MPa$*$m$^{1/2}$ for highest CNT content.

Composite samples prepared by SPS technology also demonstrated a uniform distribution of CNTs in alumina matrix. The flexural strength and fracture toughness of composites howed an increase with enhancing of CNTs fraction. Stress intensity factor (fracture toughness) for samples containing 50 % vol. CNT appeared to be about 3.3 times higher (reaching 8.9 MPa$*$m$^{1/2}$) compared to this characteristic of pure alumina, and elastic modulus rose about 2.8 time sand reached 780 GPa. The enhancing of composite mechanical properties attributes by the reticular carcasses, the bridges between alumina grains and presence of CNTs in boundaries of alumina grain that proved by electron microscopy images.

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