

NANOSIZED AlN POWDER SUSPENSIONS FOR ADVANCED CERAMICS AND THEIR RHEOLOGICAL PROPERTIES

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Use of fine nanosized powders for processing the advanced ceramics permits lowering significantly the sintering temperature, which reduce the production costs and makes possible the broadening of ceramics market. For producing fine nanosized ceramic powders on a commercial scale, a plasma technique was developed in the Latvian Institute of Inorganic Chemistry. Evaporation of raw materials in a thermal plasma jet with subsequent condensation of particles in a controlled environment allows us obtain fine powders of a number of compounds including aluminium nitride (AlN). For AlN ceramics parts, using processing by injection moulding, a composition on the basis of paraffin doped with surfactants was found. A volume fraction of 48-52 % of a fine powder in suspension was reached and its use for producing the various parts, for example, crucibles for metal melting was shown. An interaction between the fine particles leads to complicate behaviour of such suspensions. Therefore, the objective of this work was an extended study of rheological peculiarities of the suspensions mentioned.

The rheological properties of an injection moulded suspension prepared from nanosized AlN powder and paraffin wax were studied at 90°C using a rotational viscometer RHEOTEST 2 (cone-plate system, diameter of 36 mm, cone angle of 0.3°) and a Weissenberg Rheogoniometer R 18 (cone-plate working unit, diameter of 25 mm, cone angle of 4°). The use of two cone-plate systems with different angles allows measuring the viscosity in a range of shear rates 0.07-1350 s⁻¹. The viscoelastic properties were investigated in the range of the deformation amplitude of 0.66 - 20% and frequencies of 0.1-80 s⁻¹.

Oscillatory shear strain (γ_A) sweep measurements at $\omega = 1.6 \text{ s}^{-1}$ in a range of $\gamma_A = 0.66 \div 20\%$ show that the region of linear deformation (at the lowest deformation amplitude $\gamma_A = 0.66\%$) exists only for the powder loading of 43 vol.% in the suspension. The phase lag and frequency dependence of the complex modulus components were determined at the lowest deformation amplitude, and the dynamic viscosity was calculated.

At a steady shear flow of the suspensions investigated, two regions of a supernormalous viscosity giving the different shear ordering structures were observed after reaching the apparent yield stress. The steady shear flow becomes metastable and a flow spurt occurs in these regions. The dynamic viscosity $\eta'(\omega)$ was approximately four times greater than the effective viscosity $\eta(\dot{\gamma})$ at $\dot{\gamma} = \omega$ in the range of ω investigated. This allows to assume that a spatial thixotropic structure persist in the suspensions treated. The frequency dependence of the dynamic viscosity lacks an expressed viscosity plateau and shows a significant change in the suspension structure during a steady shear flow. It allows us to consider that

this structure is destroyed at low shear rates. The agglomerates were destroyed at higher values of shear rates ($\dot{\gamma} > 100 \text{ s}^{-1}$), when the separate powder particles were involved in the suspension flow. Such a property of the feedstock promotes its flow through the canals of technological equipment and form filling by the injection moulding.

A considerable change in structure of high-loaded suspension during the shear treatment was observed. It was caused by orientation of the particle packing structure under a stress and reflected in a hysteresis on the flow curve. The same rearrangement in the suspension structure is also seen after the ultrasound treatment, which results in the dependence of rheological properties on the pretreatment history of suspensions.