STUDY OF GELATION DURING THE FORMATION OF SOL-GEL FIBRES

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Ceramic materials, including metal oxides, have wide range of outstanding properties like high hardness, refractive index, resistance for thermal shocks etc. Still, the promising real applications of the materials are often limited by their fragile nature and difficulties in preparing them in desired final shape structures. The origin of the problems is the fact that usually bulk ceramics decompose instead of melting at very high temperatures. Therefore melt molding is not suitable for shaping of metal oxides. The problems are classically overcome by using sintering of micro level compressed powders. Since early works in 1970`s, different sol-gel approaches have also become routine in many labs and industry in order to prepare ceramic materials [1].

Compared with powder ceramics sol-gel method has several advantages in preparation of low-dimensional materials. The main advantages like easiness to dope the materials by different additives (even viruses and DNA), structural homogeneity, low-cost is possible as preparation of these materials starts from fully homogeneous precursors. If the processes are carried out correctly then the homogeneity is not lost until reaching final dense materials. Real mastery of sol-gel technology is achieved if chemistry is combined with mechanical shape processing of the materials. Direct drawing of suitable sol-matters enables for example to prepare different shape nano and micro materials as nanofibres, microtubes and nanometer level sharp needles [2]. Tape casting enables to prepare micro stripes on flat surfaces [3]. Colloidal generation if applied at suitable conditions results in formation of oxide micro spheres.

However in our recent works we have introduced an alternative approach for alkoxide based sol-gel preparation: oligomeric concentrate route [4, 5]. It is based on utilizing solvent-free oligomeric concentrates of primary alkoxides such as Ti, Sn, Hf, Zr, Ce, etc. The materials that have viscous fresh honey-like nature are stable in dry atmosphere for a period longer than a year. Drawing of these materials enables to obtain optical quality nanocrystalline metal oxides in shape of microtubes, (micro)nanofibres and nanometrically sharp needles. Characteristic for these structures is their high uniformity. AFM measurements have proved that surface roughness of the fibres remains in 1-2 nm range. First test using some micron diameter fibres have proved that the structures are applicable as optical waveguides with possible sensor applications. Materials can be heat-treated for transforming to final dense ceramics.

Current presentation focuses on solidification kinetics of freshly formed sol-jet surfaces after pulling into humid air. Thickening of the shell-structure is directly measured and presented as a function of time in case of alkoxides of different metals. Evolution of elastic-plastic behavior of the core is monitored in-time by measuring the tensile strength of the structures. Calculations of the results show that shell thickening is limited by diffusion of water molecules that initiate polymerization reaction. Discrete transition of viscoelastic core into fragile shell proves that chemical reactivity is many orders of magnitude higher compared to diffusion of water in the core. Moreover, a method to reliquify the shell, giving therefore a possibility to joint the structures is introduced. Current project has great cognitive value as it enables to understand sol-gel processes more deeply, having also direct value for sol-gel synthesis of low dimensional structures.

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References:

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Figures:

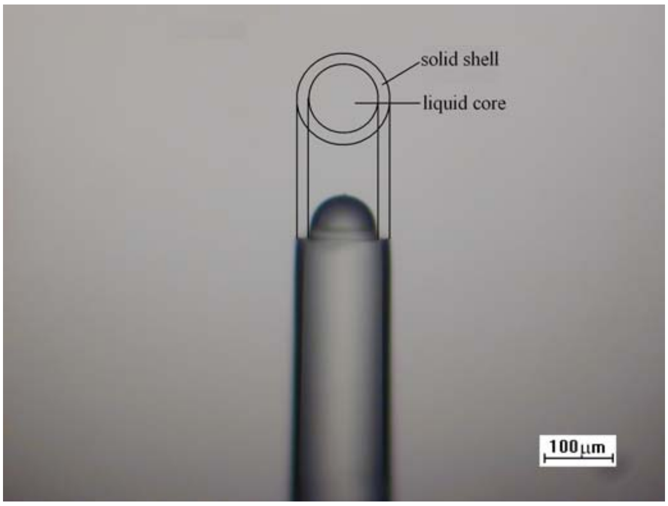

Image of core-shell sol-gel fiber structure
Sketch on the image illustrate structure of the fiber.