# Application of sol-jets in preparation of different shape metal oxide materials.

**Tanel Tätte**[1], Marko Part[1], Keijo Riikjärv[1], Medhat Hussainov[1], Kelli Hanschmidt[1], Ioannis Chasiotis[2], Valter Kiisk[1], Kaupo Kukli[1], Aile Tamm[1], Vadim Kessler[3], Ants Lõhmus[1]

[1]Institute of Physics, University of Tartu, Riia 142, 51014, Tartu, Estonia
[2]Aerospace Engineering, University of Illinois at Urbana-Champaign, Urbana, IL-61801, USA
[3]Uppsala BioCenter SLU, Dept. of chemistry, Box 7015, Arrheniusplan 8 750 07, Uppsala, Sweden
tanelt@fi.ut.ee

Sol-gel technology is based on science of nanocolloidal suspensions known as sols. When the purpose is to prepare metal oxide materials in a controlled manner, the corresponding metal alkoxides are the primary choice as precursor compounds. During the last decade, the majority of researches have started to agree that chemistry behind the transformation of metal alkoxides to oxides is different compared to the chemistry of widely used Si-alkoxides that undergo hydrolysis and polymerization processes after reaction with water. When set into contact with water, metal alkoxides directly form oxide nanoparticles as a result of one step chemical process [1]. Therefore, by adding certain amount of water and controlling the parameters like concentration, acidity, temperature etc., it is possible to obtain metal oxide nanocolloidal systems, suitable for sol-gel processing.

High surface energy of nanoparticles should naturally lead to their dissolution or coagulation inside the liquid phase. To achieve stability of sols, stabilising layer is used on the surface of particles. In the case of alkoxides, the stabilizing role is played by monolayer of alkoxy groups on the surface of particles. The stabilising effect of the layer depends largely on its thickness, the length and shape of alkoxy groups have high impact on the properties of sols and their gelation. Probably for the first time, we have shown that at least in the case of $SnO_2$ alkoxy sols, the alkoxy groups can be fully exchanged by -OH groups. In the consequence, it is possible to get highly pure and fully stable colloids that are based just on $SnO_2$ in water. Such liquid systems provide clear cassiterite structure and could be concentrated up to 20-30 %. When more water is removed, these sols transform into gels being perfect materials for sol-gel processing.

In the current presentation we will demonstrate the application of jeting in order to shape the precursor sols and produce metal oxide particles with desired geometries [Fig. 1]. Micro- and nanofibres could be obtained when stable slender jets are solidified [2]. Micro- and nanospheres, capsules and torroids could be obtained when the jets are broken into pieces and the droplets formed solidify under specific conditions. Nanometrically sharp needles could be obtained when jets are pulled into air until break-up. Immediate solidification of break-points is possible due to the high speed of reaction between alkoxide precursors and water [3]. Microtubes could be obtained when losses in volume occurs during the solidification of the jets. Crucial in geting the tubes is that the process starts by formation of rigid shell on the surface of the jet.

All obtained structures can be post-treated by aging and baking to achieve fully dense nanocrystalline oxide materials. When the residual organics is removed from the matter and the size of crystallites is still kept low enough then the eventually formed materials will possess optical transparency, being potentially applicable as waveguides for micro- and nanophotonic devices and sensors [4]. We have shown that microtubes, when made of yttria stabilized zirconia (YSZ), could be used as high temperature ion-conducting membranes. Potential of such tubes could be realized in construction of high temperature (up to 1000 °C) and high pressure (at least 1000 atm.) microfluidic systems and as miniature plasma chambers. Applications of the materials are supported by their high Young modulus, that remains within 100-200 GPa range, while tensile strenght is around 1GPa, being comparable to that of stainless steel. To realise the applications of the materials we have also started studies on atomic layer deposition (ALD) of functional films, such as $TiO_2$, MgO, $Ta_2O_5$, on the surface of materials formed by sol-gel. ALD is inherently a chemical deposition method driven and controlled by the adsorption capability of chemically active surface. ALD of oxides is based on the sequential adsorption and reaction cycles between metal and oxygen precursors, with only one sub-monomolecular material layer deposited in a single cycle. For instance, MgO films can be deposited layer-by-layer from magnesium *beta*-diketonate and ozone [5]. Due to the surface control and the self-limitation of the adsorption process, dense and ultrathin films can be deposited over complex-shaped, three-dimensional, substrates, including micro- and nanostructures created by sol-gel. In this way optical, electrical, mechanical and chemical properties of nanofibres, microtubes, nanoparticles, etc., can be modified.

## References


[1] Vadim G. Kessler and Gulaim A. Seisenbaeva, New Insight into Mechanisms of Sol-Gel Process and New Materials and Opportunities for Bioencapsula-tion and Biodelivery, *NATO Science for Peace and Security Series C: Environmental Security, (book title: Sol-Gel Methods for Materials Processing)*, 2008, 139-153, Springer.

[2] Tanel Tätte, Medhat Hussainov, Madis Paalo, Marko Part, Rasmus Talviste, Valter Kiisk, Hugo Mändar, Kaija Põhako, Tõnis Pehk, Kaido Reivelt, Marco Natali, Jonas Gurauskis, Ants Lõhmus and Uno Mäeorg, *Science and Technology of Advanced Materials*, **12** (2011) 034412.

[3] Tanel Tätte, Madis Paalo, Vambola Kisand, Valter Reedo, Alexander Kartushinsky, Kristjan Saal, Uno Mäeorg, Ants Lõhmus and Ilmar Kink, *Nanotechnology*, **18(2)** (2007) 125301.

[4] Limin Tong and Eric Mazur, Nanophotonics and Nanofibres, *Handbook for Fiber Optic Data Communications: A Practical Guide to Optical Networking*, Ed. Casimer DeCusatis, (2008) 713-728, Academic Press, Burlington, MA.

[5] Matti Putkonen, Leena-Sisko Johansson, Eero Rauhala and Lauri Niinistö, Surface-controlled growth of magnesium oxide thin films by atomic layer epitaxy, *Journal of Materials Chemistry*, **9** (1999) 2449.


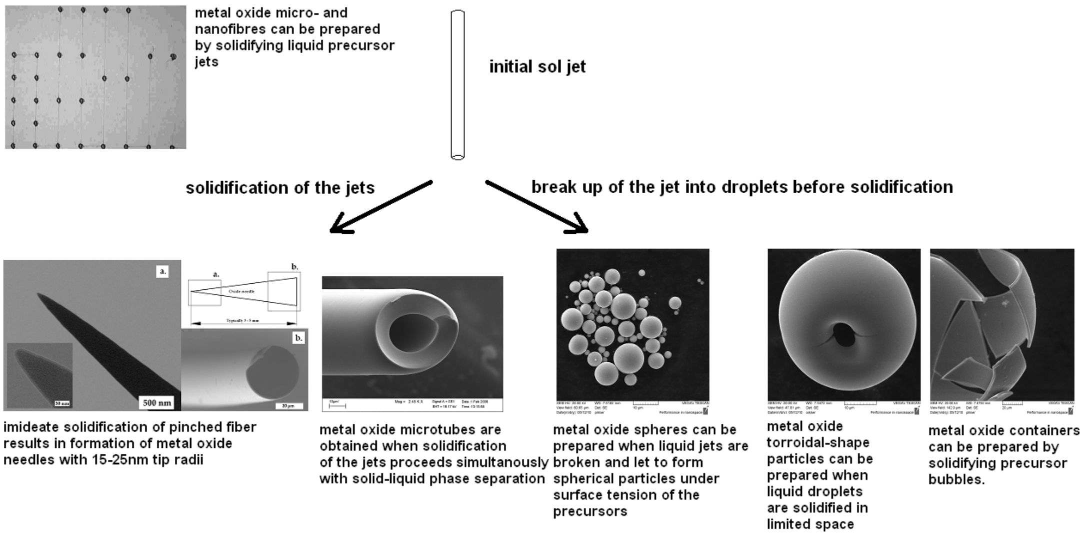


Figure 1 Different shape metal oxide materials that can be prepared from sol-jets.