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# PREPARATION AND OPTICAL CHARACTERISATION OF SOL-GEL DERIVED ZIRCONIUM OXIDE FIBERS AND MICROTUBES

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For applications one of the very needed tools is structuring of materials in nano- and microscale. Sol-gel method offers big flexibility in preparation and shaping of oxide materials such as  $ZrO_2$  and  $TiO_2$  [1]. This method also allows doping of the host materials with optically active inclusions (e.g. rare earth ions) and other impurities (e.g.  $Y_2O_3$ ) [2].

The aim of this work was to prepare  $ZrO_2$  based waveguiding structures. Keeping in mind applications as lighting materials, the samples were doped with rare earth ions.

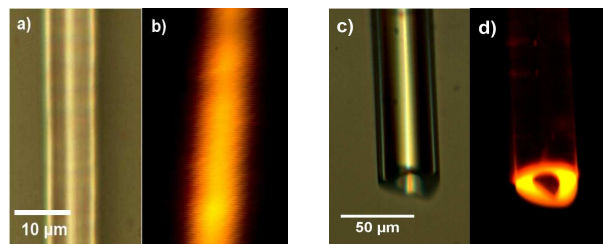
We prepared Sm-doped  $SiO_2(70\%)-ZrO_2(30\%)$  microfibers by direct drawing method from precursor material with suitable viscoelastic properties. Prepared fibers were annealed at temperatures up to  $1000^\circ C$  to achieve development of Sm-doped  $ZrO_2$  nanocrystals inside the amorphous silica matrix (i.e. transparent nanoceramics) [3]. As a result we obtained light guiding fibers with bright luminescence of  $Sm^{3+}$  ions (Fig.1:a,b). XRD measurements confirm formation of  $ZrO_2$  nanocrystals inside the silica matrix while luminescence study suggests that emitting  $Sm^{3+}$  ions remain distributed both in amorphous silica and  $ZrO_2$ .

We also worked out a novel sol-gel preparation method for  $ZrO_2$  microtubes. For luminescence applications we doped the material with 1 mol% of  $Sm^{3+}$  or  $Eu^{3+}$  ions. To avoid cracking of the microtubes during annealing (which is present in the case of sol-gel-prepared microrolls, for example [4]) we added 8 mol% of  $Y_2O_3$  to the material. The obtained microtubes were  $\sim 10$  mm long, had an average diameter of  $\sim 40$   $\mu m$  and wall thickness of  $\sim 10$   $\mu m$ . Prepared microtubes remained crack-free even after annealing up to  $1100^\circ C$  and microtubes annealed at  $800-900^\circ C$  exposed also excellent luminescent and light guiding properties (Fig.1:c,d).

*Fig. 1: On the left: Optical micrographs of  $Sm^{3+}$  doped  $SiO_2(70\%)-ZrO_2(30\%)$  fiber annealed at  $1000^\circ C$ ; a) illumination with white light; b) propagation of luminescence observed through a 550 nm cutoff filter (excitation wavelength 473 nm).*

*On the right: Optical micrographs of  $Sm^{3+}$  doped YSZ microtube annealed at  $900^\circ C$ .*

*c) illumination with white light; d) luminescence observed through a 550 nm cutoff filter (excitation wavelength 405 nm).*



## References

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