Doping of optical fiber preforms

by
oxide and metal nanoparticles

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Scope of presentation

- Interest
- Fabrication method
- Experimental
- Results
- Conclusions

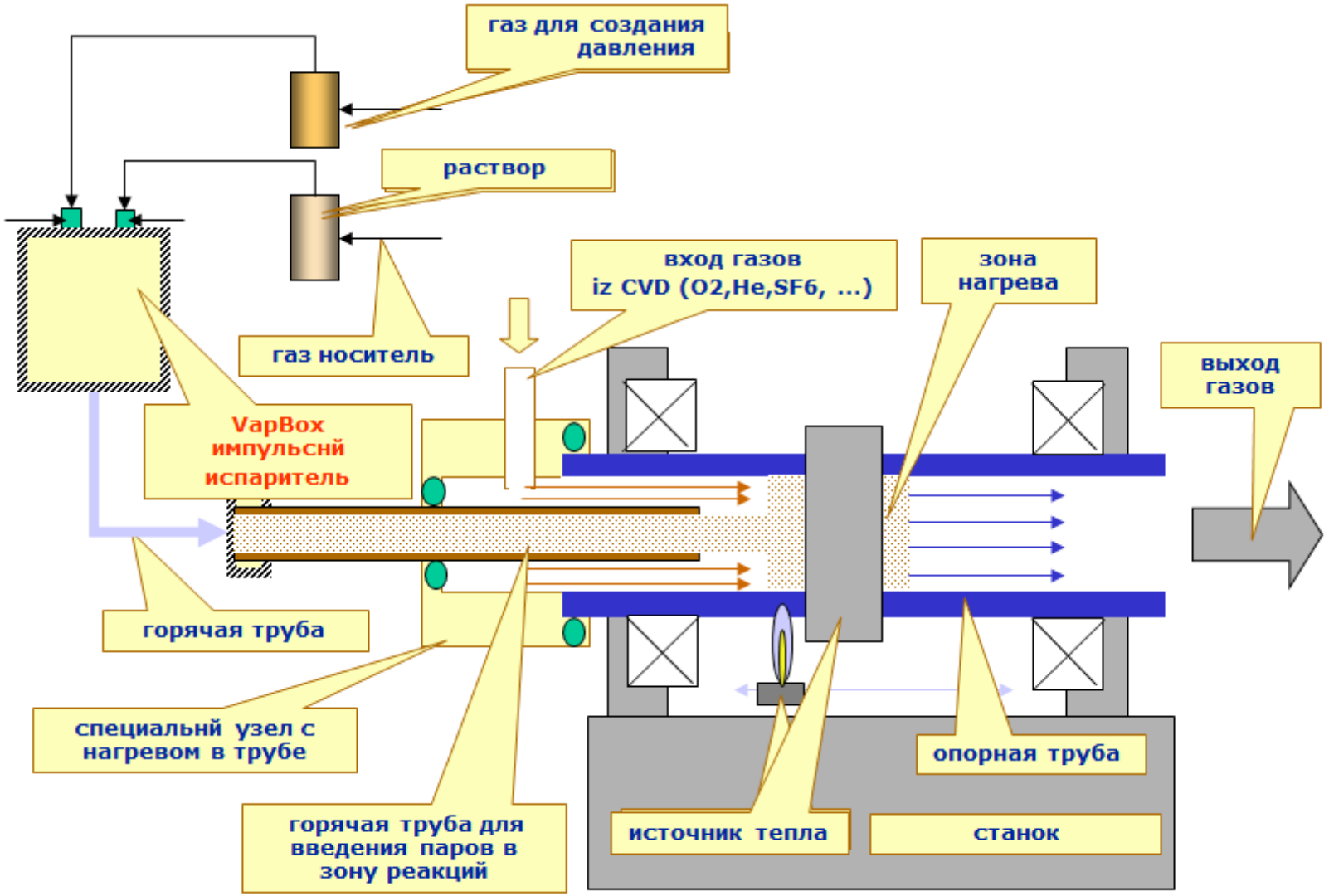
Interest

- Interest
  - fabrication of layers inside optical fiber, containing metal nanoparticles or even thin metal layers
  - fabrication of silica-based optical fibers, containing a crystalline or amorphous phase of a different (oxide) material
  - (i.e. Optacore‘s project to fabricate OF with magneto-strictive characteristics under MANUNET programme)
  - fabrication of rare earth- or metal-doped optical fibers with dopants distributed as nanoparticles or agglomerations with limited size

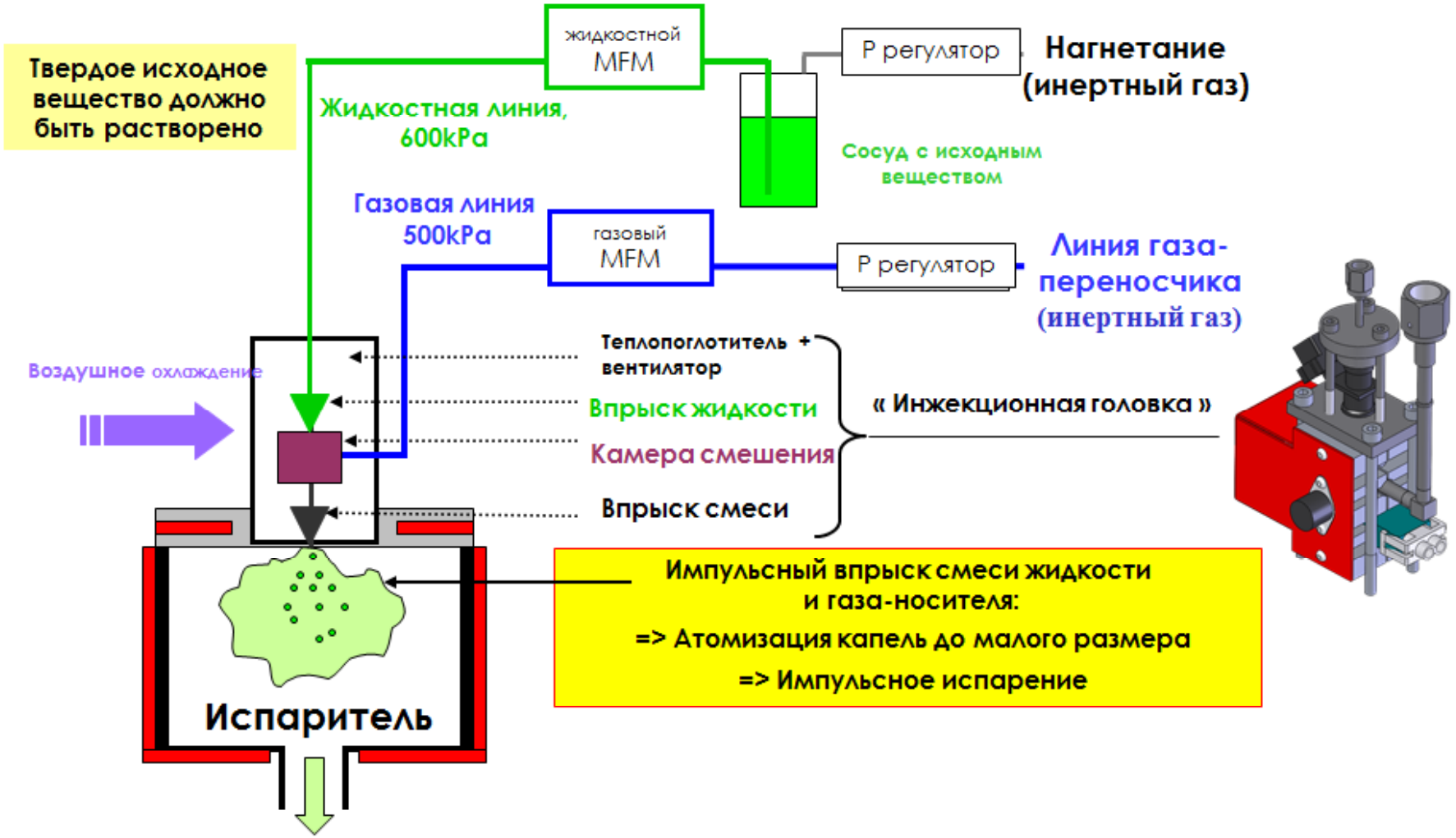
Fabrication method

- Flash vaporization and aerosol preform fabrication methods were used
- Optacore‘s FVS and AES devices connected to MCVD system were used
- Nanoparticle dopants were either commercial products or custom developed suspensions with low agglomeration characteristics
- Standard preform analysis tools were used combined with SEM, TEM microscopy and EDAX probe

процесс импульсного испарения

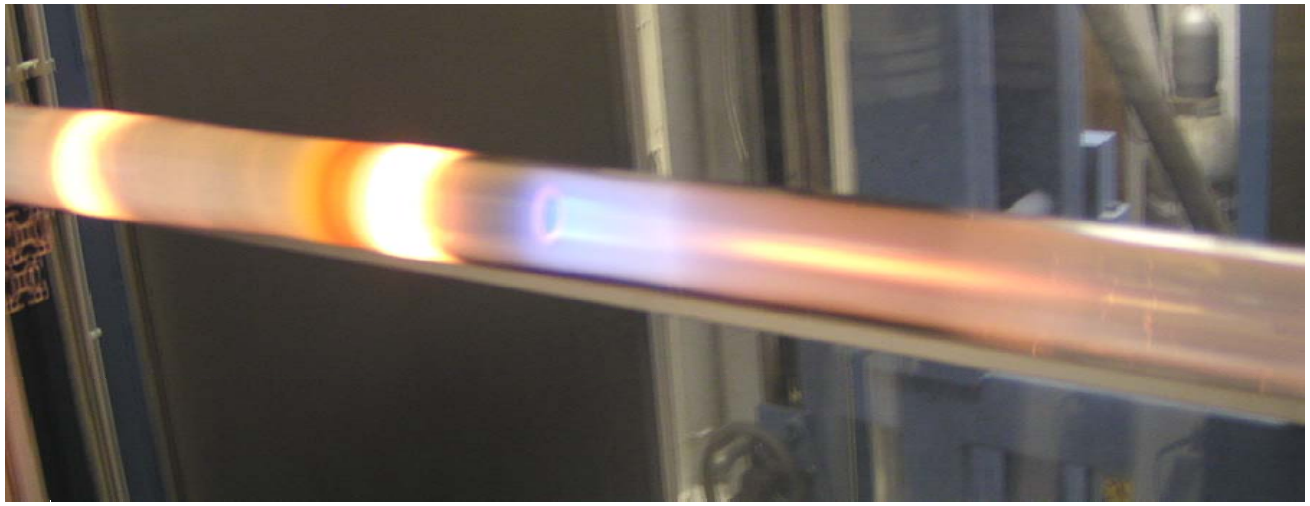


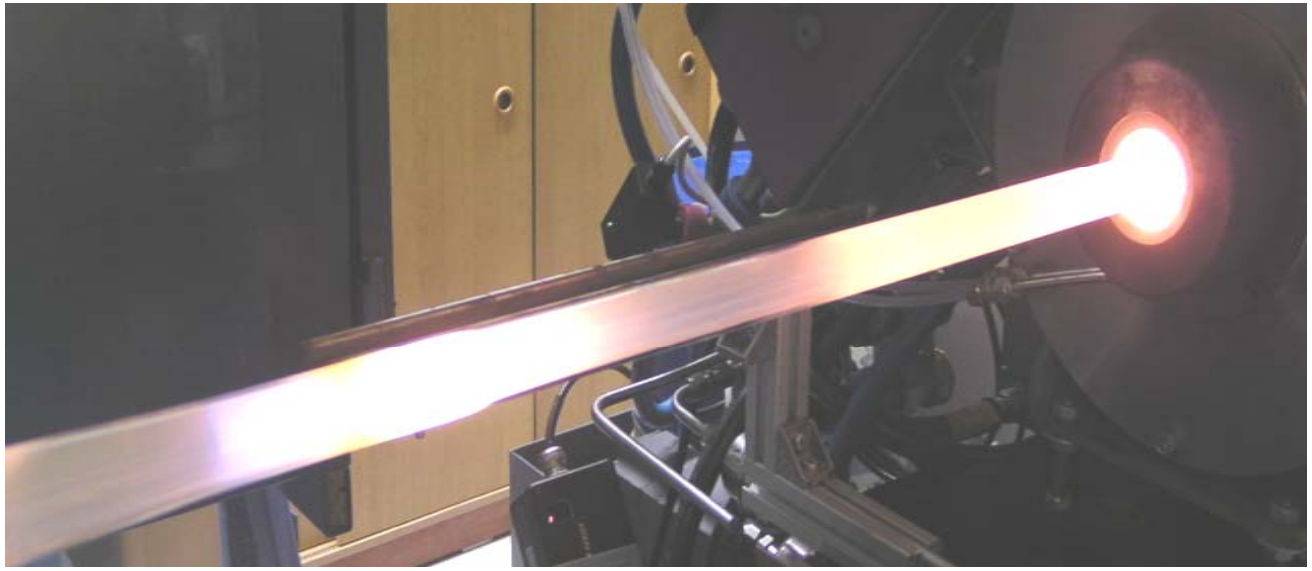
VAPBOX - Принцип Действия



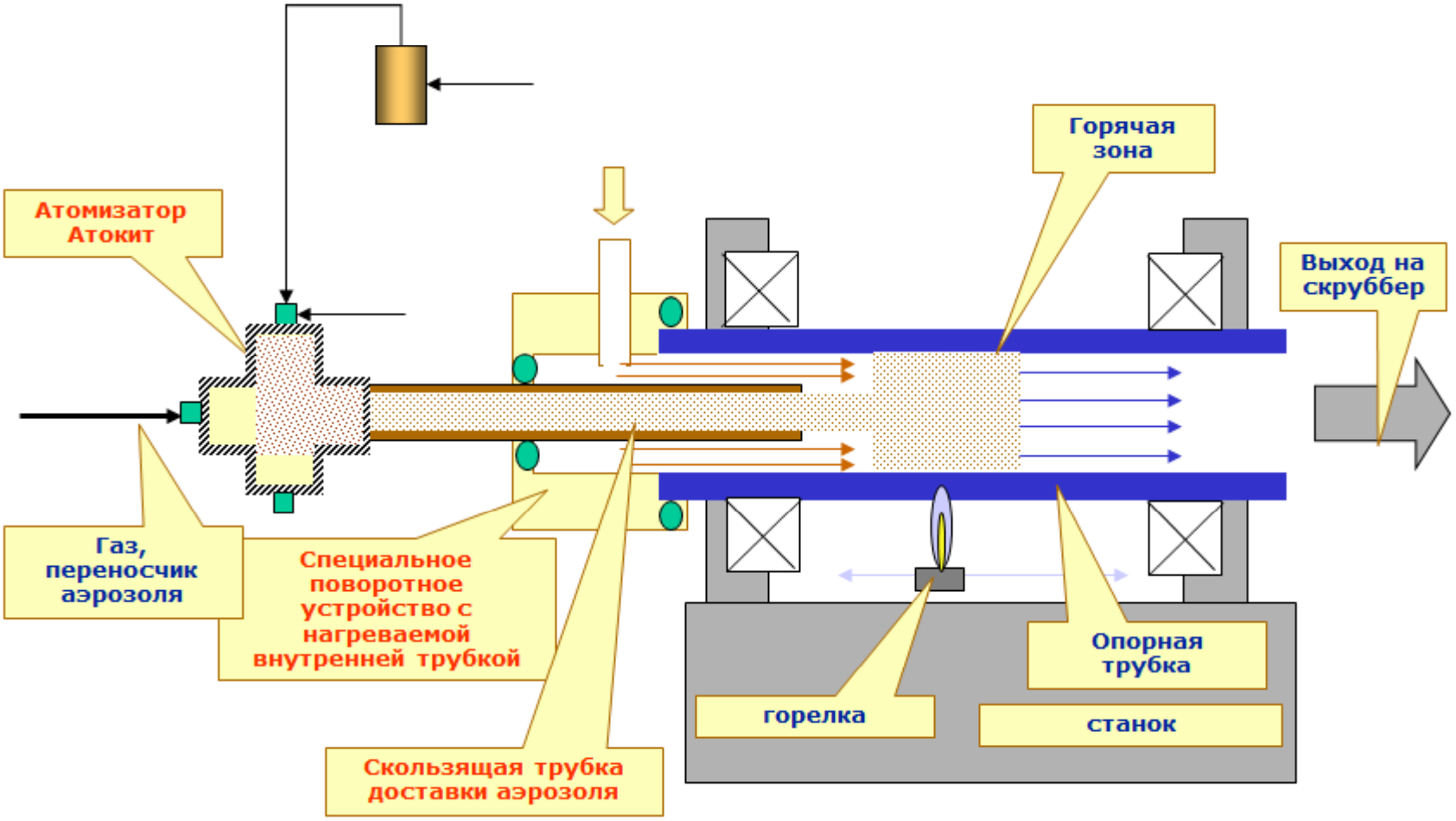
MFM = Mass Flow Meter

Процесс Осаждения





Устройство FVS для осаждения аэрозольным методом



Experimental

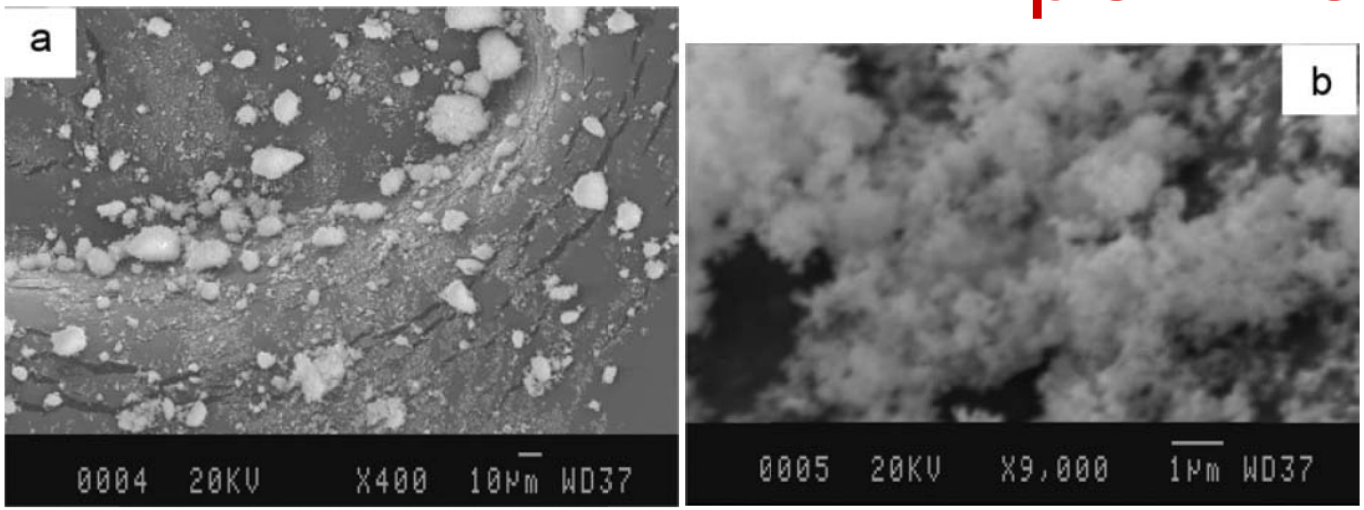
- Nanoparticles
  - Al2O3, cat.no. 544833, avg. particle size <50 nm, Sigma Aldrich
  - Er2O3, cat.no. 903581, avg. particle size 41-53 nm, MaTecK GmbH
  - Cu, avg. particle size 25-70 nm, Tekna
  - Fe-Co ferrite custom developed by Department for Material Synthesis of JSI, Ljubljana, part of FOMS project
  - Gold nanorod suspension, prod. code 716855, nanorods 25 nm OD and approx. 600 nm long Aldrich Chemistry

- Preform deposition materials
  - standard MCVD materials (tubes, chlorides, gases)
  - in most cases TEOS was the Si-precursor
  - TEOGe and TEPO replaced GeCl4 and POCl3 precursors, when used in combination with TEOS

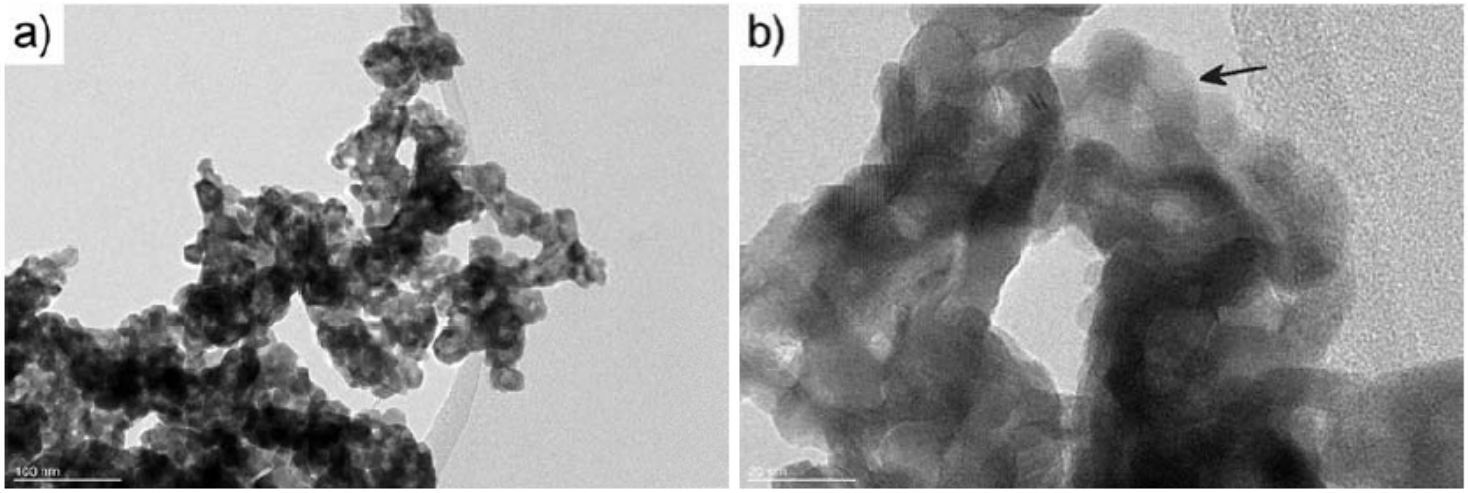
Experimental

PREFORM	INJECTOR 1		INJECTOR 2		Deposition Conditions
	solvent	particles	Si precursor	dopant	
No.	(ml)	(g)	(g)	(g)	
P0348	solvent 1	copper	TEOS	TEPO	layer of SiO2, followed by nanoparticle layer, vitrification after SiO2 deposition
	150	1.5	150	24	
P0349	solvent 2	Er/Al oxide	TEOS	-	combined deposition of SiO2 and nanoparticles, forward direction
	378	0.26 / 0.35	196	-	
P0350	solvent 2	Er/Al oxide	TEOS	-	separate SiO2 porous layer deposition, followed by several layers of nanoparticle deposition
	167.5	1.04 / 1.35	167.5	-	
P0351	solvent 2	Cu	TEOS	TEPO	as P0350
	282.6	5.99	228.2	24	
P0411	solvent 3	Fe/Co ferrite nanosusp.	TEOS	GeCl4	separate SiO2 porous layer deposition, followed by several layers of nanoparticle backward deposition + Ge-doped core
	282.6			from MCVD	
P0418	solvent 3	Fe/Co ferrite nanosusp.	TEOS	TEOGe	separate SiO2 porous layer deposition, followed by several layers of nanoparticle backward deposition + Ge-doped core
	282.6		55	4.3	
P0433	solvent 3	Fe/Co ferrite nanosusp.	TEOS	GeCl4	combined deposition of SiO2 and nanoparticles in backward direction, low temperature + Ge-doped core
	282.6			from MCVD	
P0436	SigmaAldrich	Au nanorods suspension	SiCl4	GeCl4	separate & combined backward deposition SiO2/ nanoparticles + Ge-doped core
	water		from MCVD	from MCVD	
P0439	SigmaAldrich	Au nanorods suspension	SiCl4	-	combined deposition of SiO2 / nanoparticles in backward direction, no core
	water		from MCVD	-	

Experimental

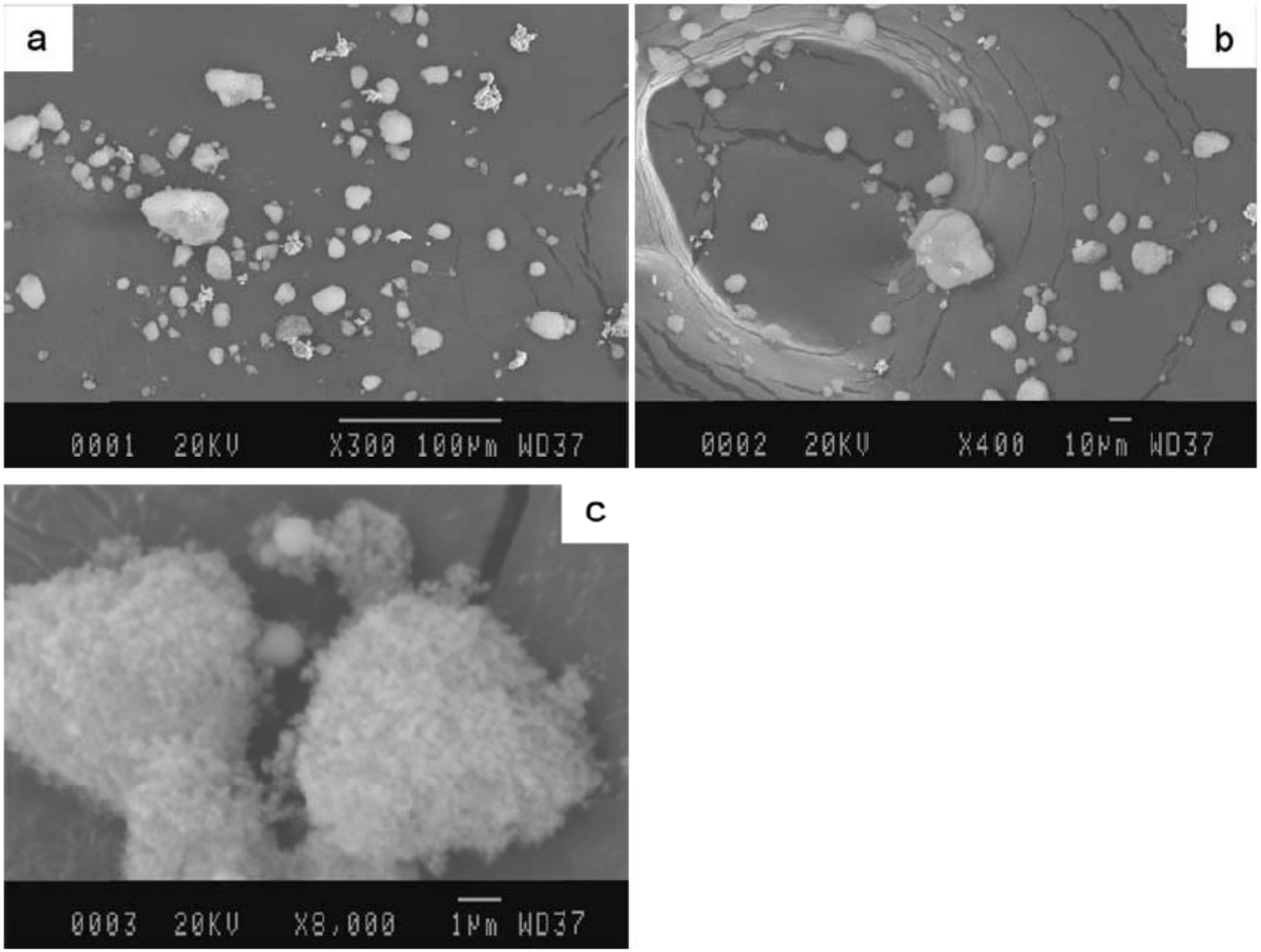


SEM image of Er2O3 nanoparticles – agglomerates of approx. 250 nm



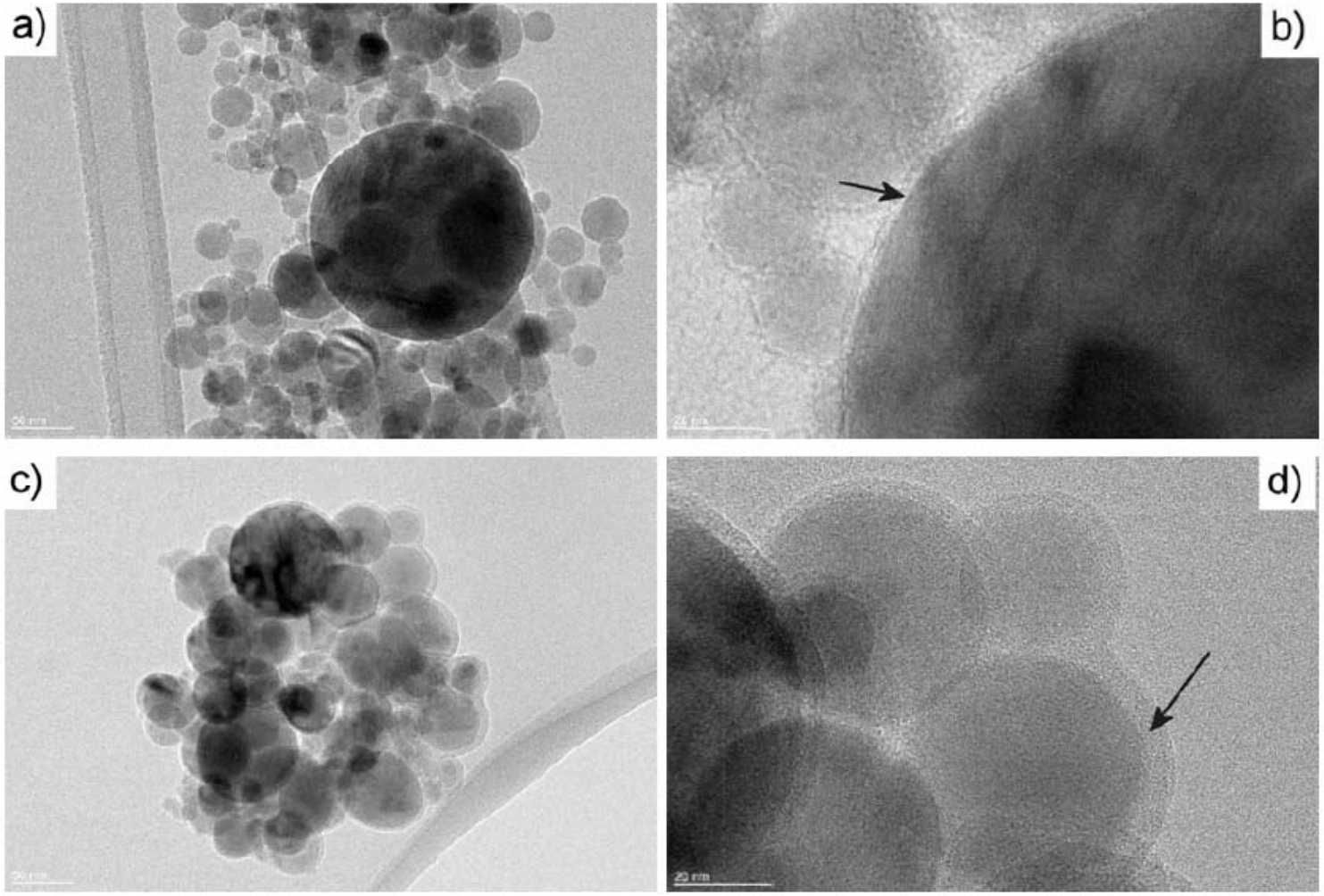
TEM image of Er2O3 nanopraticles, ave. 15-25 nm, amorphous la[illegible]2.

Experimental



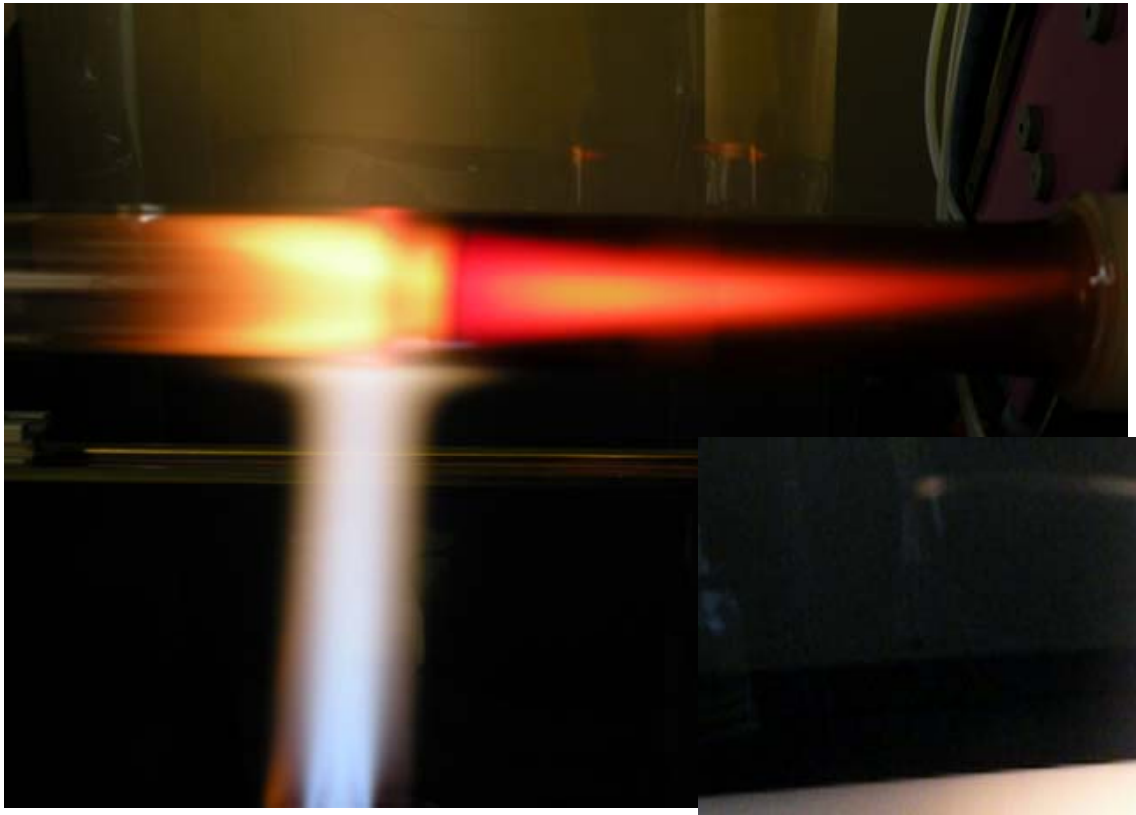
SEM image of Al2O3 nanoparticles – agglomerates of approx. 100 - 500 nm

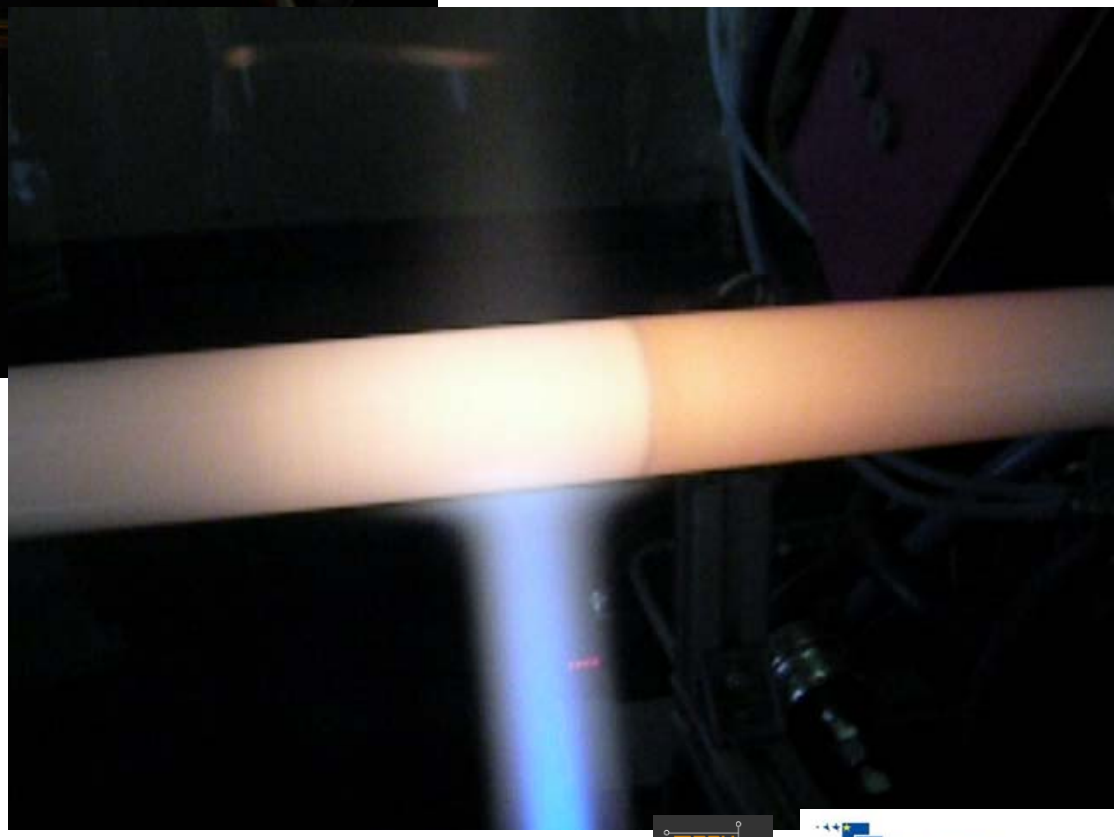
Experimental



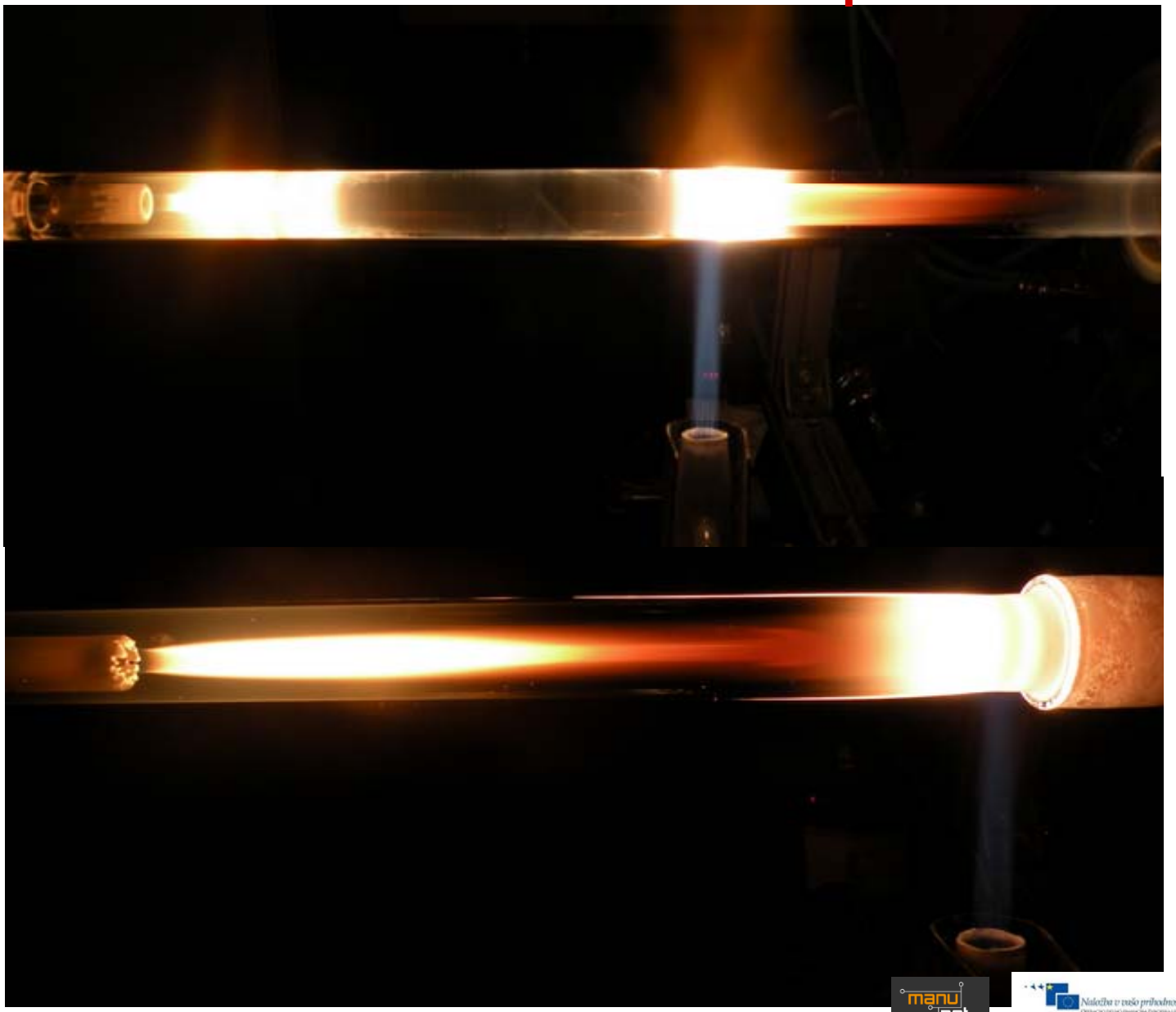
TEM image of Al2O3 nanopraticles, aggl. 100 - 290 nm, amorphous layer 4-8 nm

Experimental





Experimental



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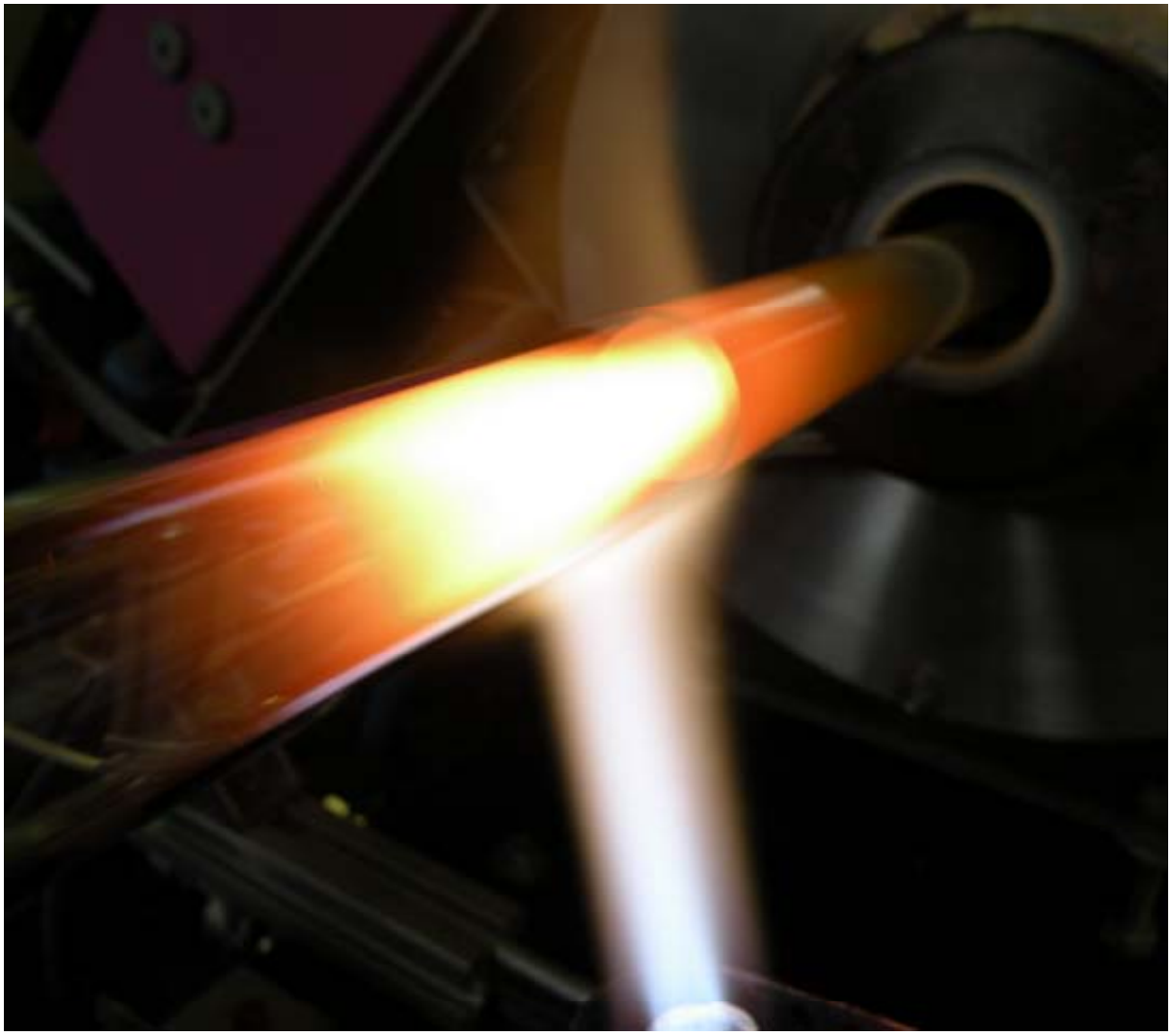
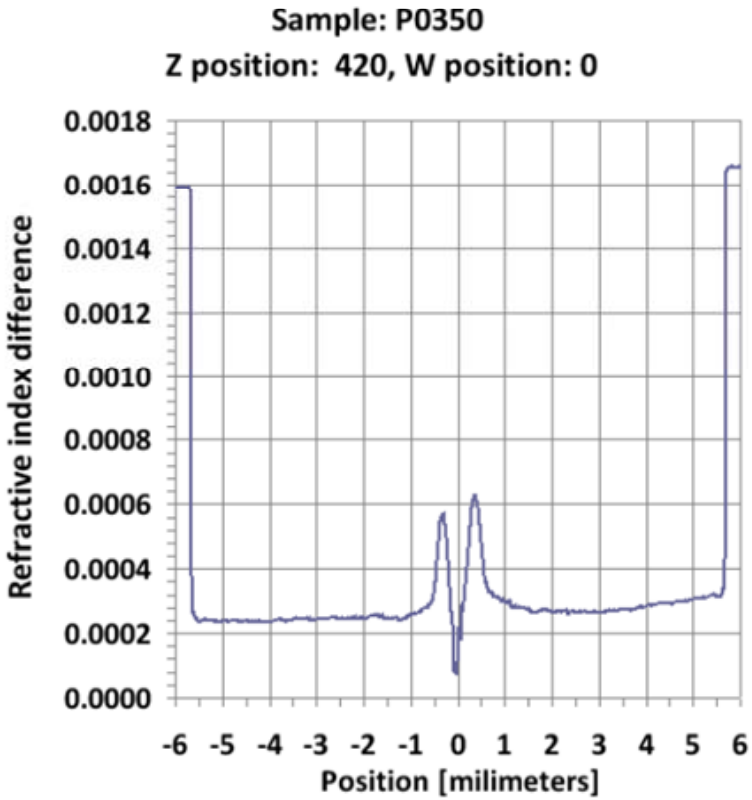
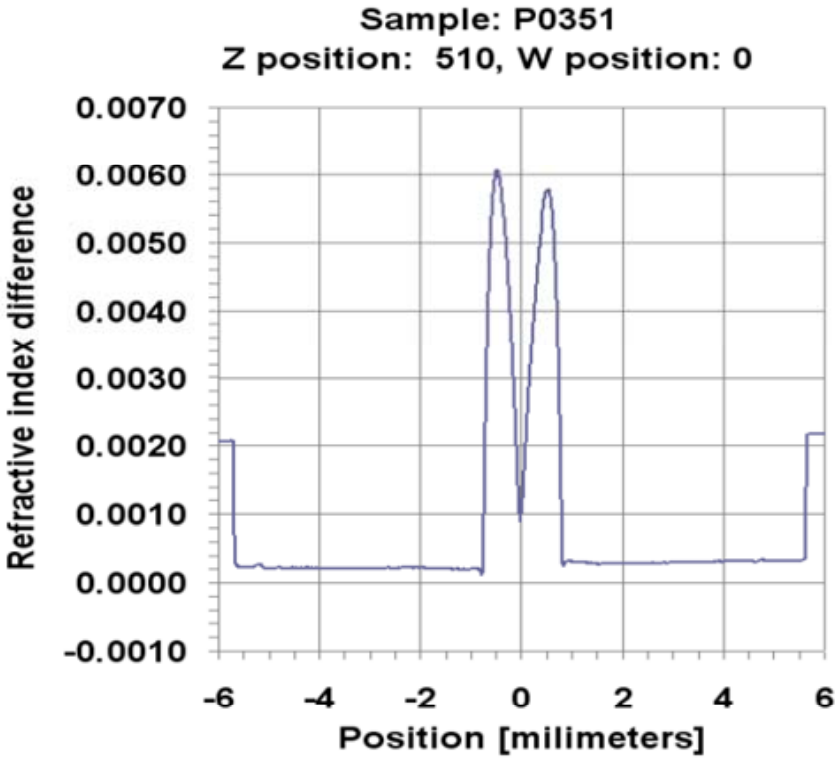


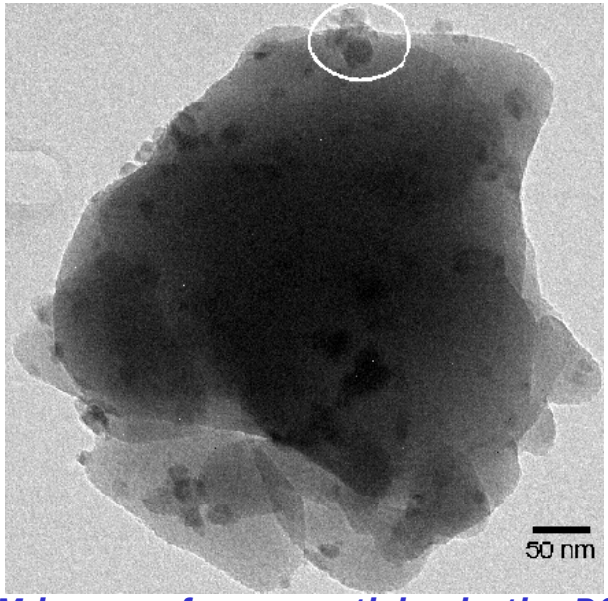
Results

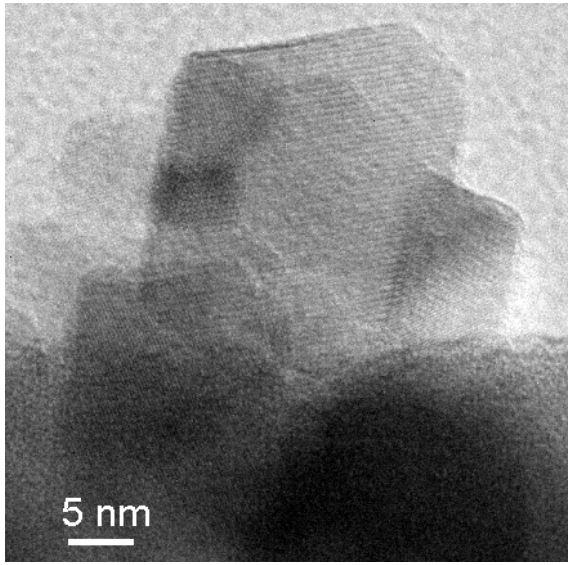




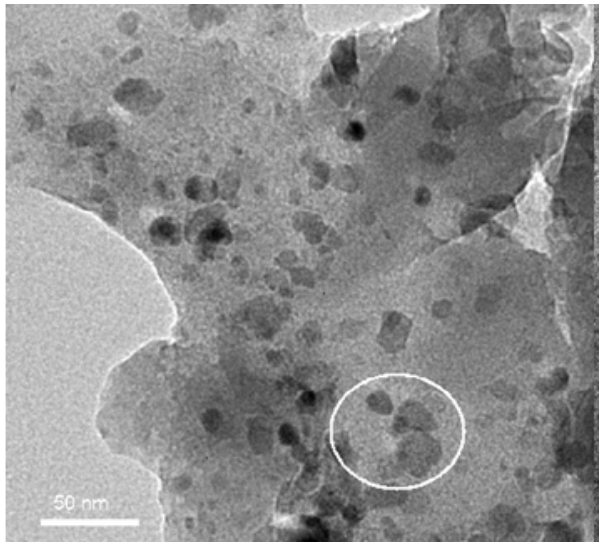
Refractive index profile of P0350 preform (pos. 420, angle 0°) and P0351 preform, both doped by Er2O3/Al2O3 nanoparticles

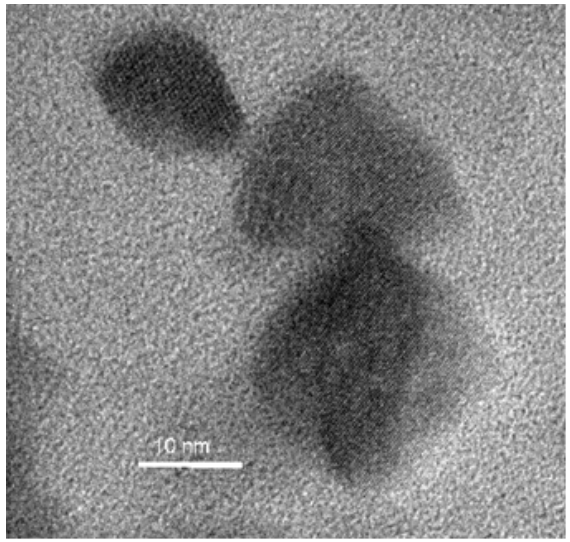
Results





TEM image of nanopraticles in the P0350 with the enlargment of the circled area



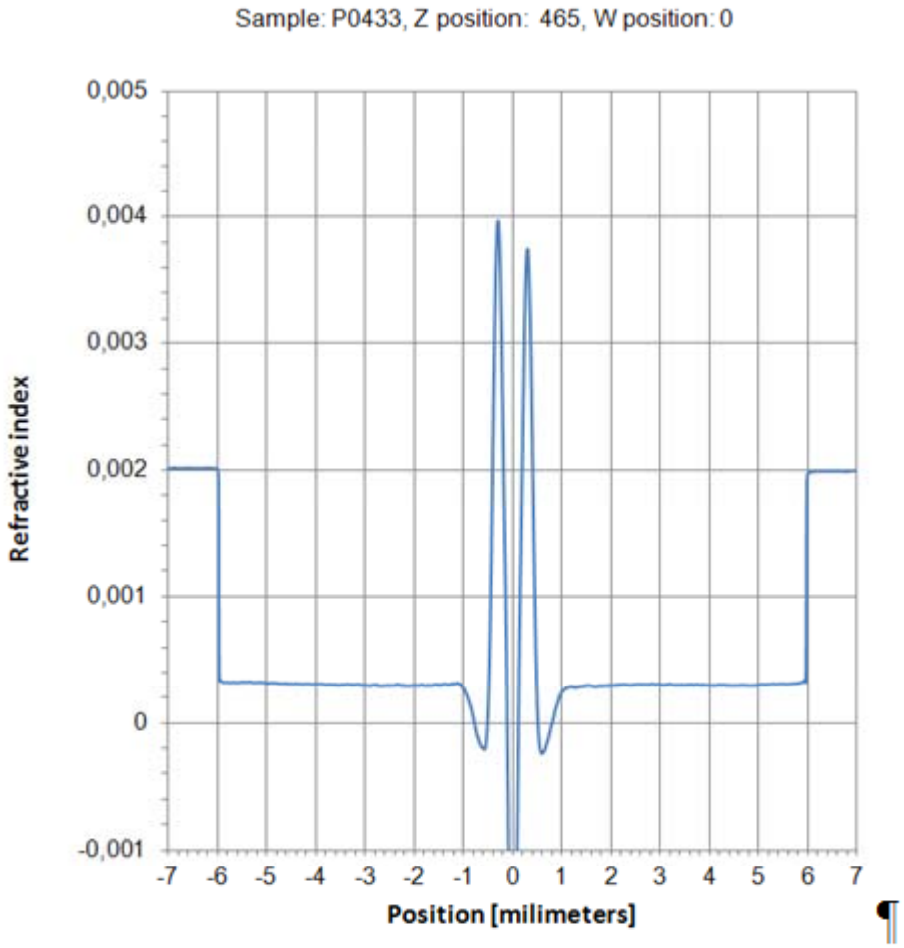


TEM image of nanopraticles in the P0351 with the enlargment of the circled area

Results – Er/Al nanoparticles

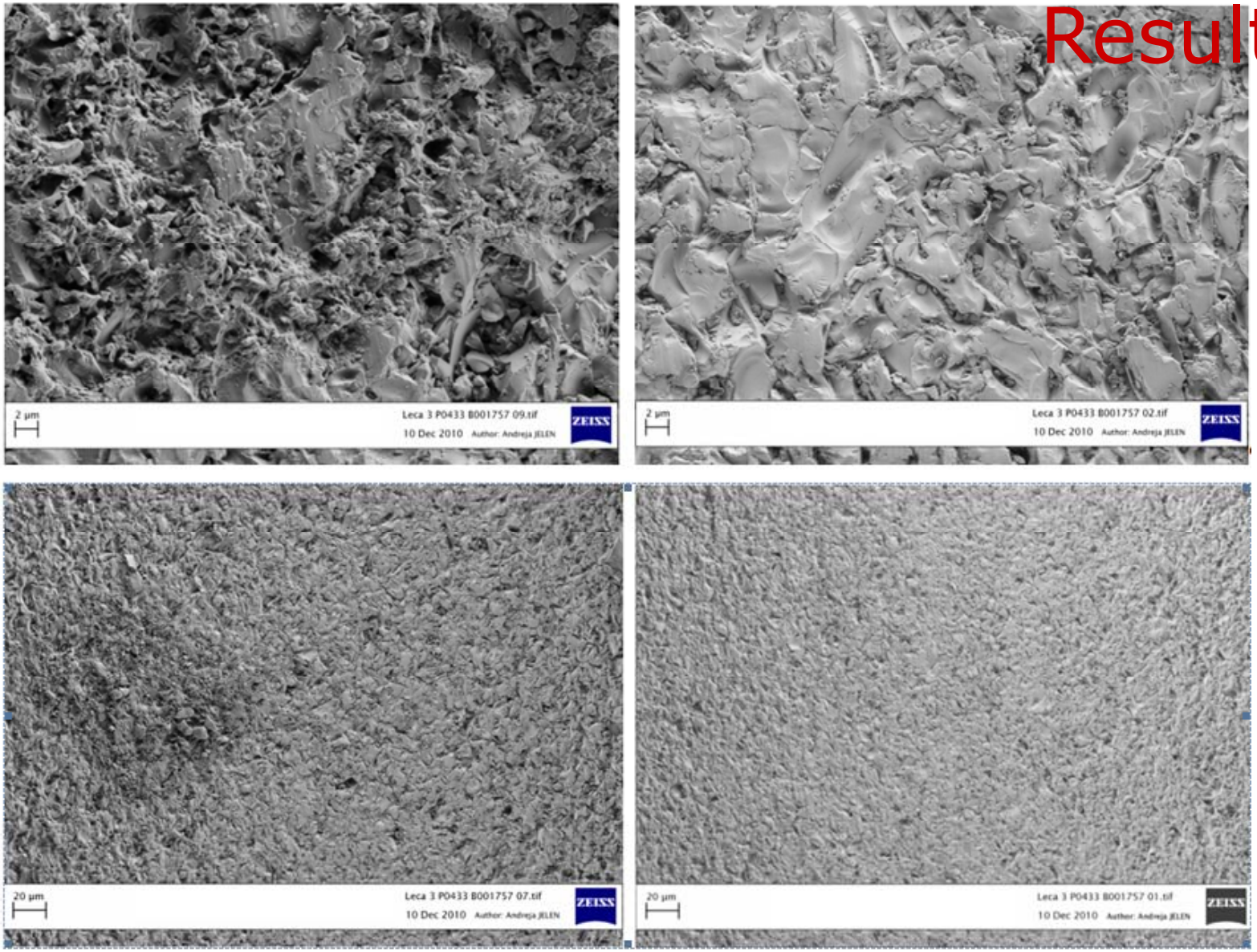
- Electron diffraction pattern (EDP) of pure silicon matrix showed no presence of crystalline phase
- However, an EDP of the matrix containing nanoparticles suggested the presence of crystalline nanoparticles
- EDP was too weak due to the small crystallite sizes and the presence of an amorphous matrix and no conclusive finding on the structure could be drawn from it
- Crystalline nature of the particles was undoubtedly confirmed with high-resolution images showing crystal-lattice
- EDXS spectrum showed a very weak Al and Er peaks together with the peaks of other elements, present in the matrix (Si, O) and in the supporting foil (Ni, Cu, C). It has to be noted that Er peaks overlap with the Co peaks. Since the sample contamination with the Co source cannot be entirely excluded the presence of Er in the P0350 sample can only be assumed from the initial composition

Results



Refractive index profile of P0433 preform doped in cladding by ferrite nanoparticles, core doped by GeO2

Results



Figures show photo of P0433 Fe/Co- ferrite doped preform sample surface at two different magnifications, nanoparticle-doped region on the left, and undoped region on the right

Conclusions

- It was demonstrated that fully vitrified, transparent, nanoparticle-doped preforms without inclusions or bubbles can be fabricated using flash vaporization process and device. Preforms were fabricated *in-situ* and in relatively short time, handling of nanoparticles' and other precursors is straightforward and simple

- Metal nanoparticles oxidize at high temperature in the presence of oxygen. This can be prevented by providing reducing or inert environment or they have to be replaced by precious metals.

- Nanoparticle suspensions need to be prepared properly, otherwise particles may agglomerate. Nanoparticles density in deposited layer needs to be increased significantly to achieve the desired level, by increasing their content in the suspension

- Recent results using alternative approaches in preparation of nanoparticle suspensions and use of aerosol are promising and further results shall be presented in specialty fiber conferences

Affiliations & Acknowledgements

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