

The solution precursor plasma spraying process for making zirconia based electrolytes

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Aim

Developing of zirconia based electrolyte layers for solid oxide fuel cells by solution precursor plasma spraying (SPPS)

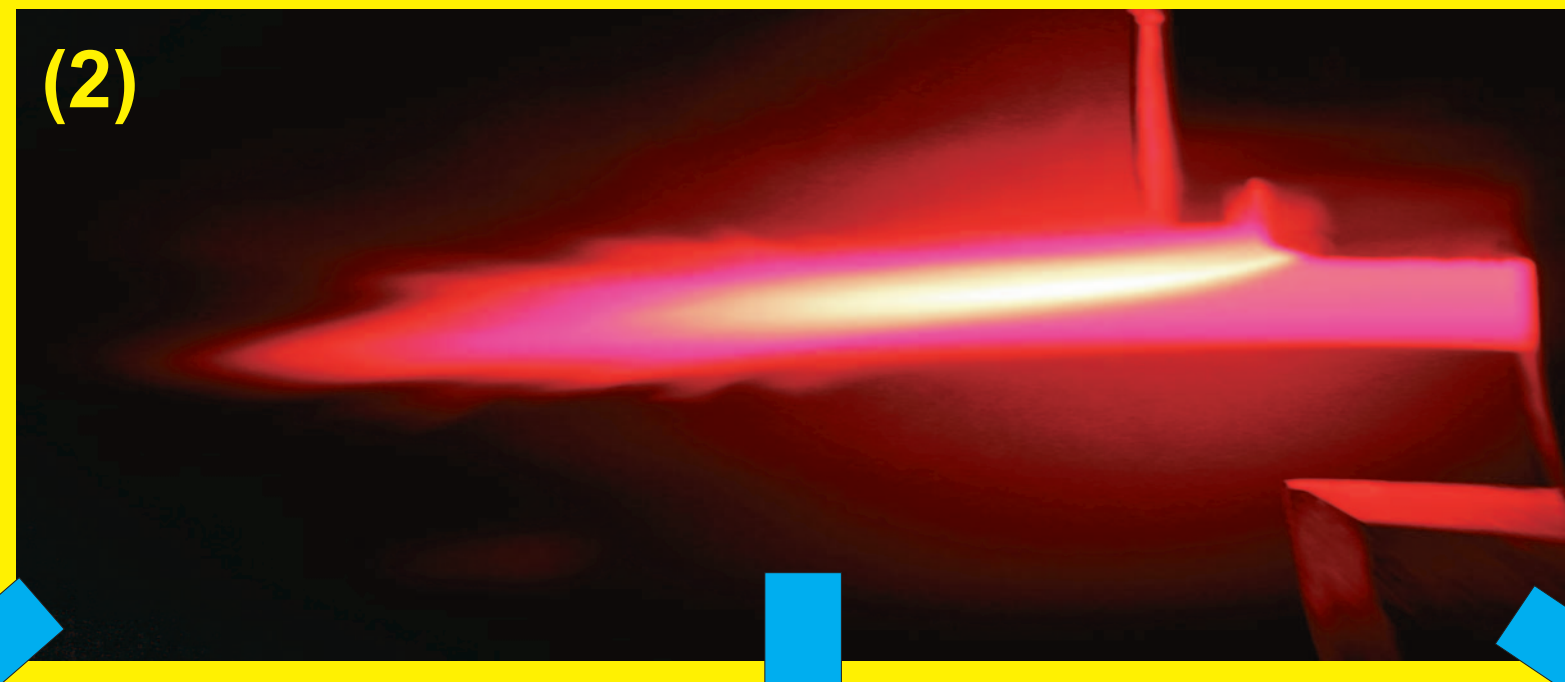
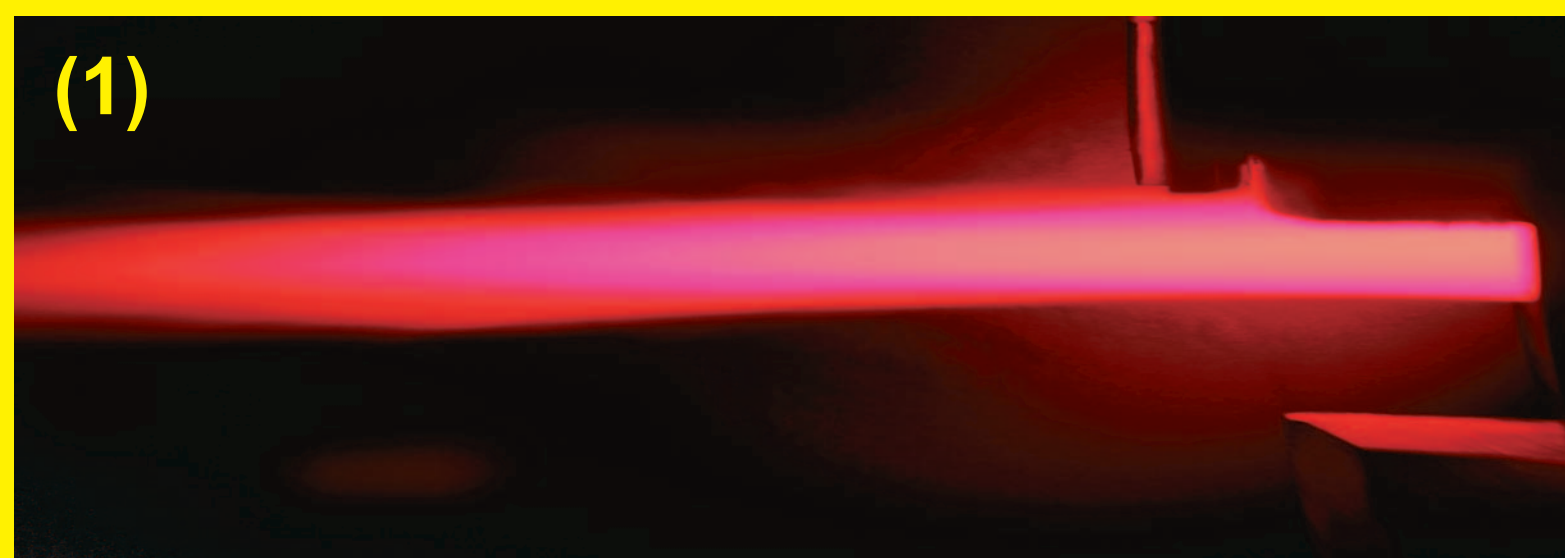
Advantages of SPPS

- Nitrates of metal precursors (zirconyl nitrate and scandium nitrate hydrate) dissolved in water or ethanol can be used
- Fast and easy formation of new precursor compositions
- Formation of nano-particles in-flight

Development steps needed

- Developing of an atomization nozzle for SPPS
- Identification of the plasma parameters for metal precursor solutions by use of TriplexPro200 dc plasma torch (Sulzer Metco, Wohlen, Switzerland)
- Identification of intermediate products (e.g. partially-pyrolized or unpyrolized droplets) deposited in the coatings according to the plasma parameters
- Identification of the influence of different solvent types on coating microstructure

Shadowgraphy of plasma jet without (1) and with (2) injection



Identification of Raman modes

SPPS coatings

- Band structure from 150 - 800 cm^{-1} : cubic stabilized zirconia phonon modes (150, 308, 551, 616 cm^{-1})
- Dominant peak at 616 cm^{-1} is characteristic for F_{2g} phase of cubic stabilized zirconia
- 150 cm^{-1} : Zr-O translations
- Tetragonal ZrO_2 is present: 308, 483 cm^{-1}

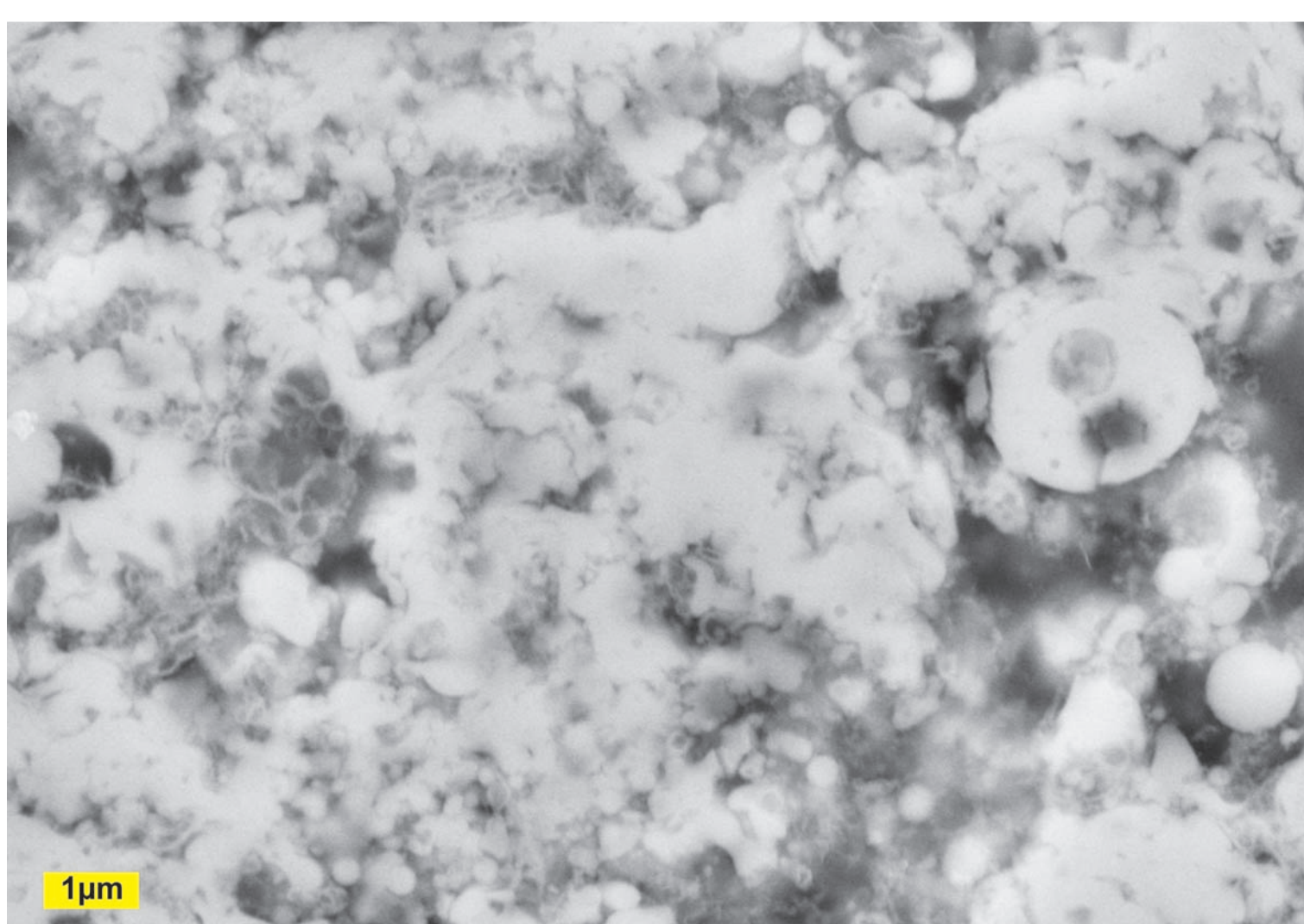
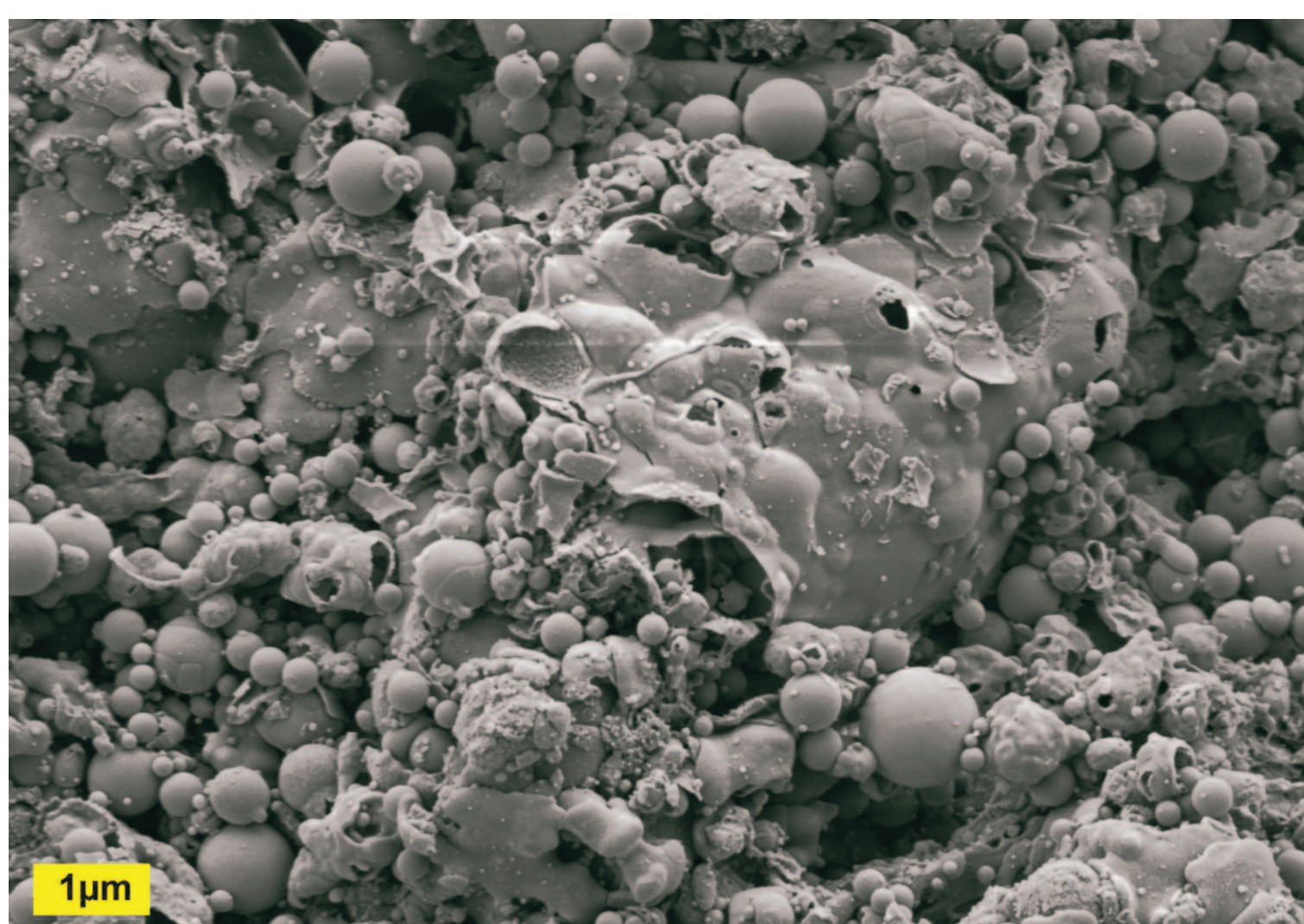
Nitrates

- NO_3^- modes present at: 388 (429), 576, 765, 1030 to 1048 cm^{-1} , only 429 cm^{-1} is present in SPPS coating

Still to be identified

- 242, 388 cm^{-1} present in scandium nitrate and in SPPS coating
- 478 cm^{-1} present in both nitrates and in SPPS coating

Results for water-based metal precursor solution (Sc/Zr- H_2O) (14.3 wt. %)



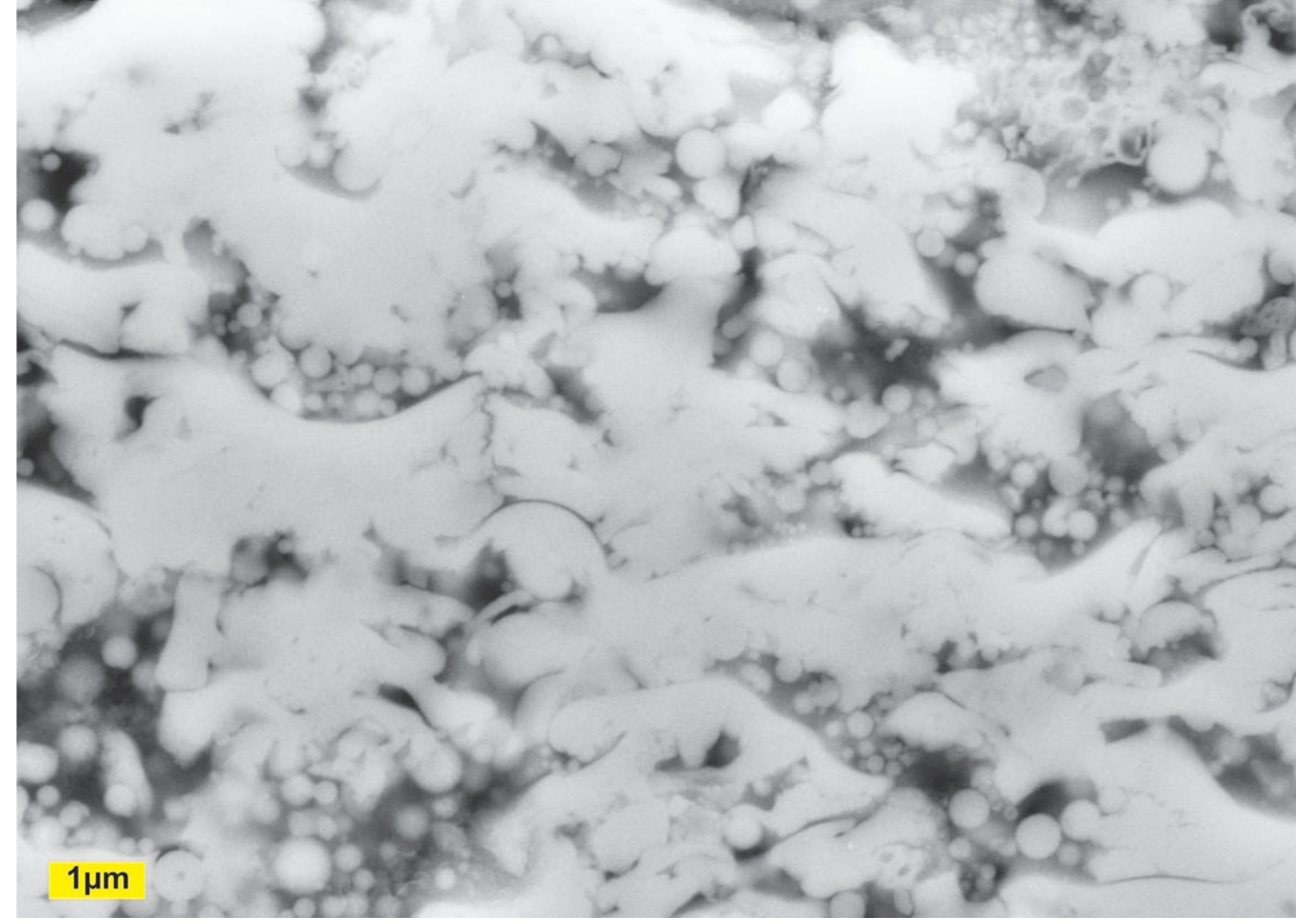
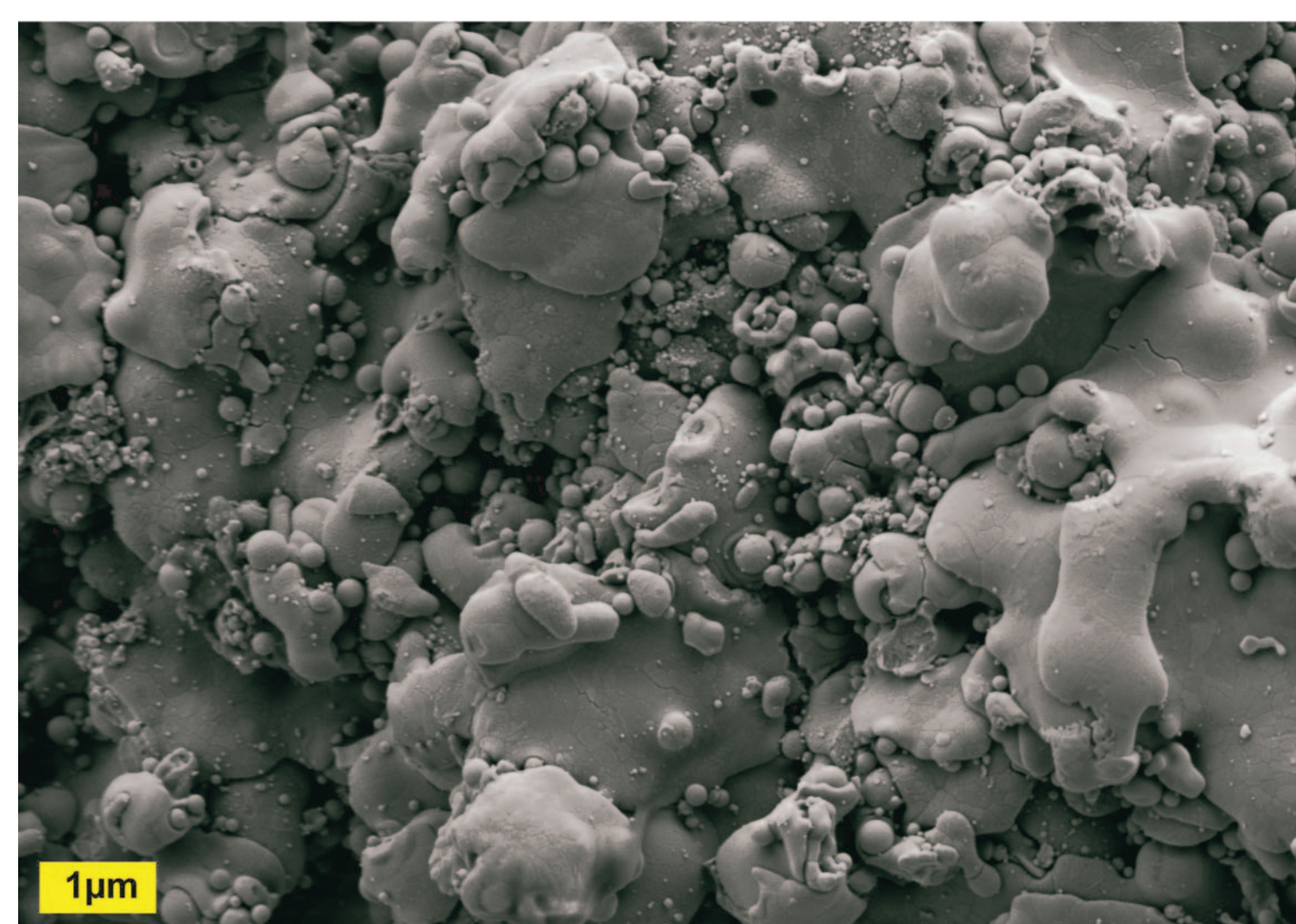
top: surface view ; bottom: cross section, (x 8000, SE images)

Structure of coating

Water-based precursor concentration leads to formation of

- Splats
- Unmelted nano- and micro-particles
- Ruptured bubbles ('cobwebbed areas')
- Porosity of coating: 33.25 +/- 3.84 %
- Structure of coating consists of larger dense zones interrupting the network of porosity by use of higher concentration of the precursor

Results for ethanol-based metal precursor solution (Sc/Zr-EtOH) (15.9 wt. %)



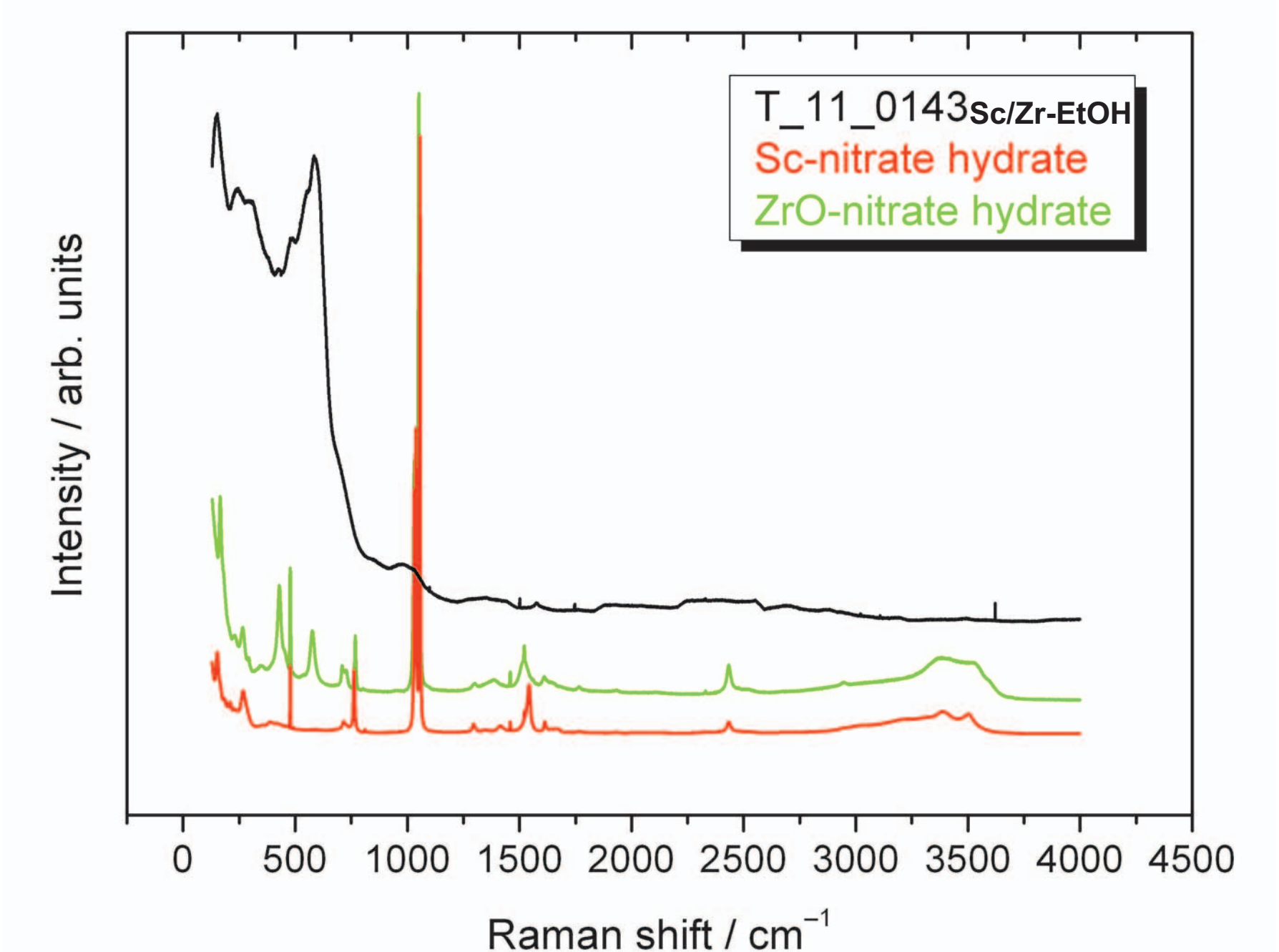
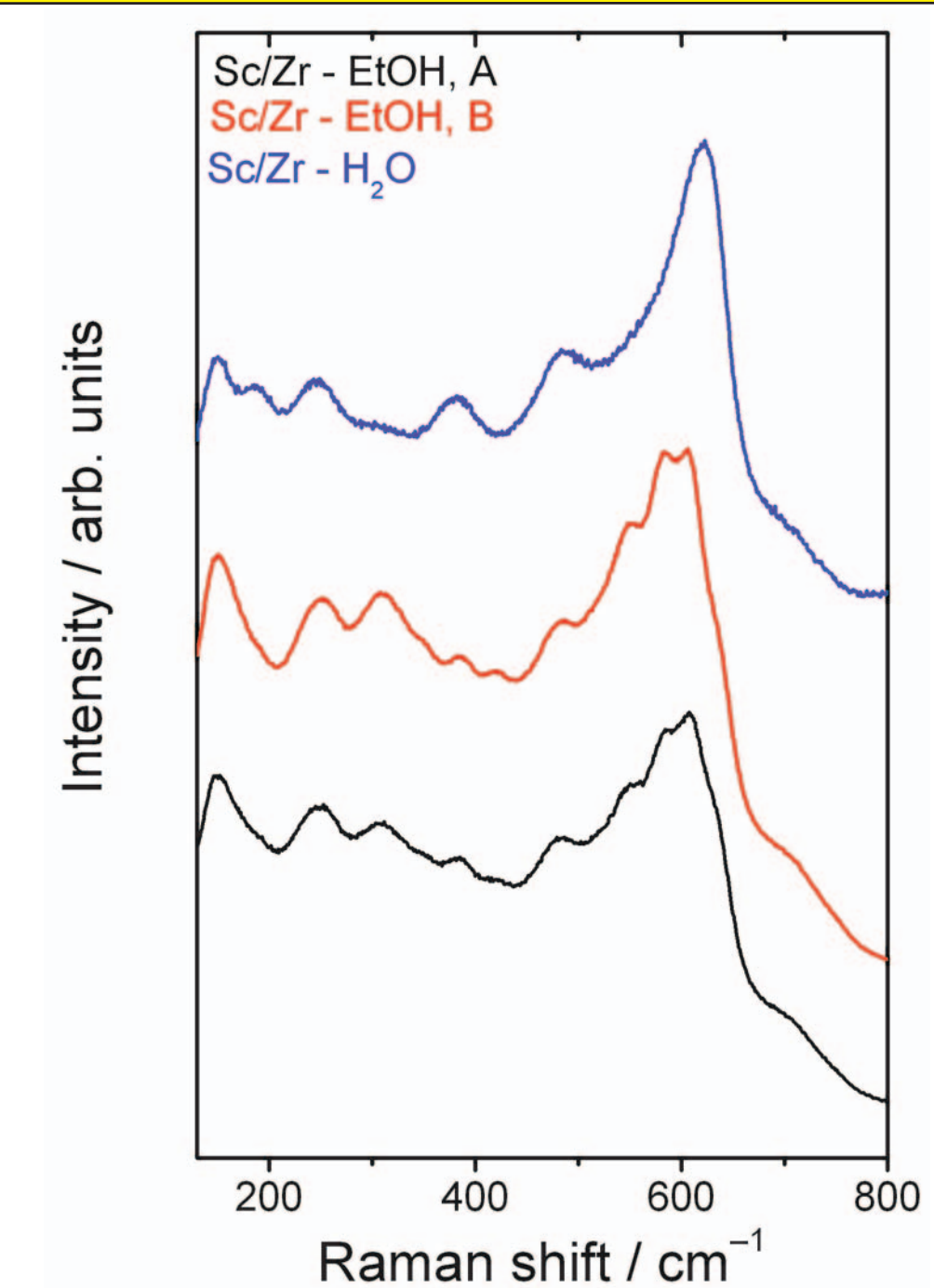
top: surface view; bottom: cross section, (x 8000, SE images)

Structure of coating

Ethanol-based precursor concentration leads to formation of

- Splats
- Enhanced microstructure caused by lower surface tension of solvent, lower boiling point and lower enthalpy of evaporation compared to water
- Unmelted nano-particles
- Low amount of ruptured bubbles ('cobwebbed areas')
- Reduced porosity, value: 23.58 +/- 5.01 %

Characterization of coatings by Raman spectroscopy



Summary

- Coatings deposited by use of solution precursors of metal salts have been prepared successfully
- Structure of coating consists of larger dense zones interrupting the network of porosity by use of higher concentration of precursor or by use of ethanol as solvent
- Characterization by Raman spectroscopy yields information about coating structure and intermediate products due to partially-pyrolized or unpyrolized droplets