European Network on New Sensing Technologies for Air Pollution Control and Environmental Sustainability - *EuNetAir*

COST Action TD1105

WGs and MC Meeting at LINKOPING, 3 - 5 June 2015

Action Start date: 01/07/2012 - Action End date: 30/06/2016

Year 3: 1 July 2014 - 30 June 2015 (***Ongoing Action***)

Complex nanostructured perovskite systems developed by chemical processes

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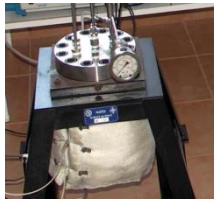
Chemical method for the development of new nanostructured systems

Sol-gel procedures

Organic synthesis

Colloidal Chemistry



Hydrothermal procedure

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Comparing different techniques for nanostructured film deposition

Sol-gel		H-E (hydrothermal electrochemical process)		RF- Sputtering		Screen - printing	
Advantages	**Disadvantages**	**Advantages**	**Disadvantages**	**Advantages**	**Disadvantages**	**Advantages**	**Disadvantages**
Thick film	Futher thermal of treatment is nesessary	Thin film	Low kinetics	Thin film	Targets not available on the market	Rapid process	Thick film

Motivation:

- **reducing the raw material consumption**
- **obtaining thin films with enhanced properties**

PZT films deposited by H-E (for actuators)

BST films deposited by H-E, RF-Sputtering, Screen printing (sensors)

Hydrothermal procedure

There is no unanimity regarding the definition of this procedure

Definition 1 **(according to O.Schaf, H.Ghobarkar and P.Knauth, book chapter, Nanostructured Materials 2004):**
A non-conventional method to obtain inorganic nanocrystalline materials

Definition 2 **(according to K.Byrappa and Masahiro Yoshimura, Handbook of hydrothermal technology 2012)**
Any heterogeneous reaction in the presence of a solvent (aqueous and non-aqueous) which takes place in a closed system at a pressure > 1 atm and temperature > room temperature.

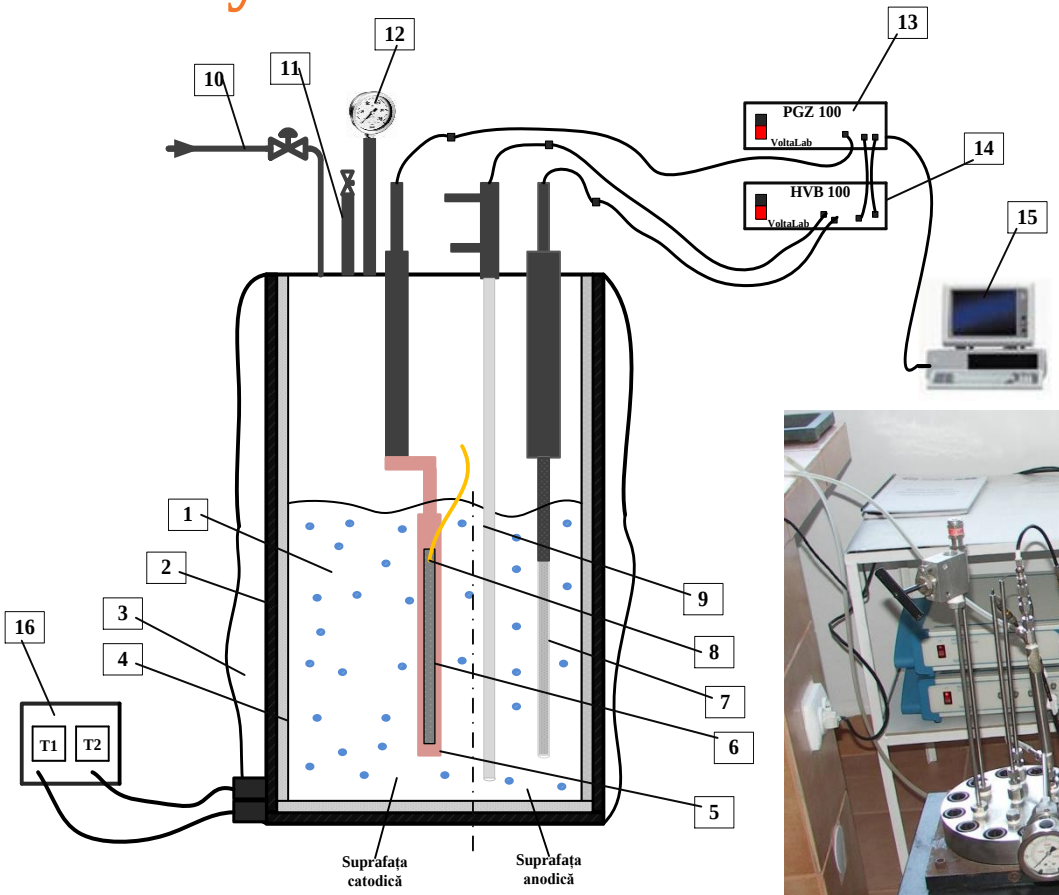
Current trends in hydrothermal technology

- Materials processing in soft and environmental friendly conditions
- Solvent behavior should be understand in correlation with pressure and temperature (e.g: structure in critical, supercritical or sub-critical conditions, dielectric constant, pH change, viscosity, density)
- Modeling of the hydrothermal reactions based on thermodynamic principles to enable to control phase purity, particles sizes, particles sizes distribution, particles morphology

New concepts in hydrothermal technology

- Additional external energy (microwave, ultrasound, mechanic-chemical, electrical and magnetic energy)
- Instantaneous hydrothermal reactions to obtain nanoparticles
- Organic synthesis in hydrothermal conditions
- Organic-inorganic hybrid materials, core of the nanotechnology, in situ synthesized in hydrothermal conditions
- Inorganic materials functionalized with biomolecules (proteins, organic ligands, ADN, amino-acids)

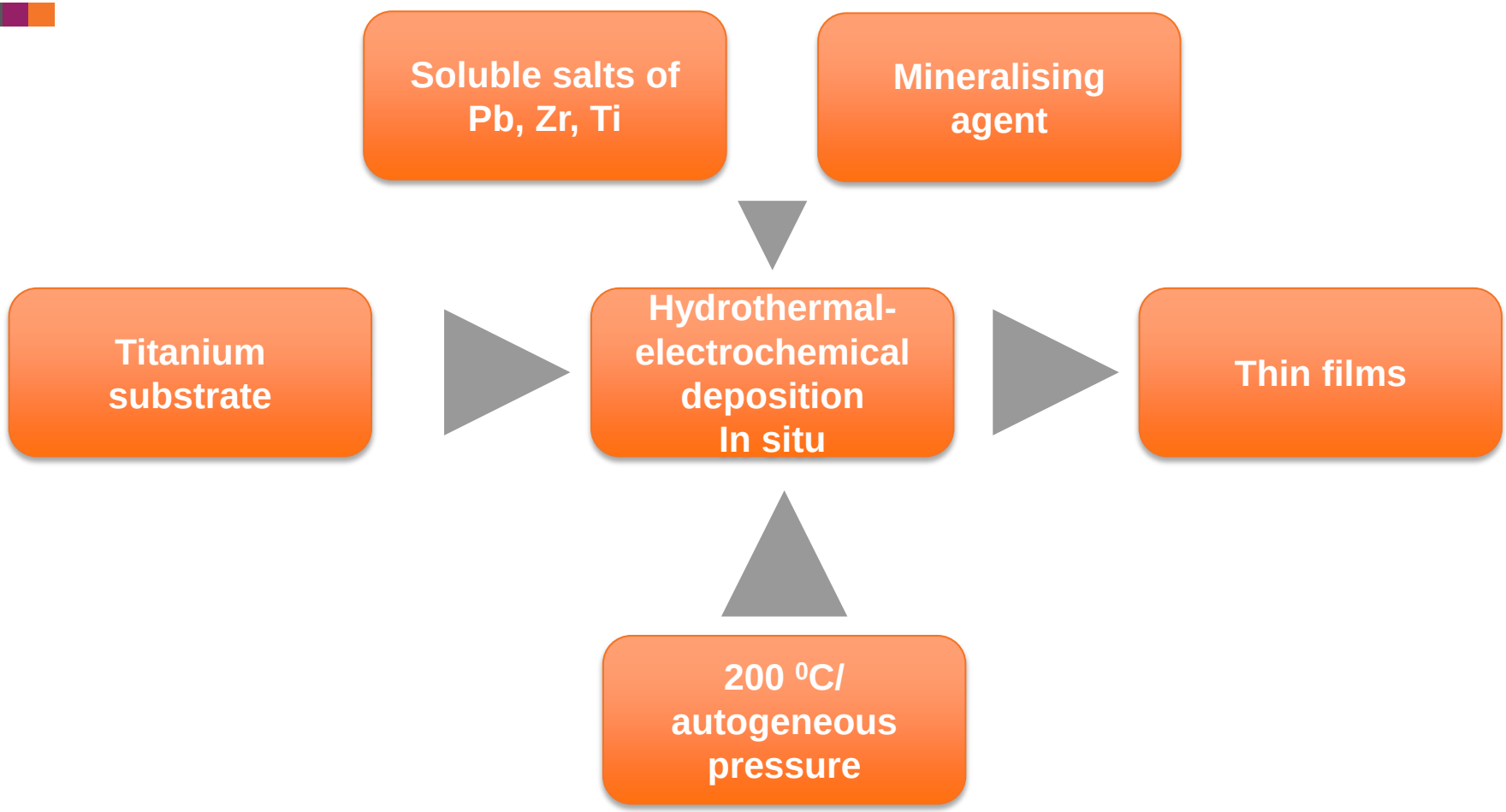
Hydrothermal - electrochemical (H-E) procedure



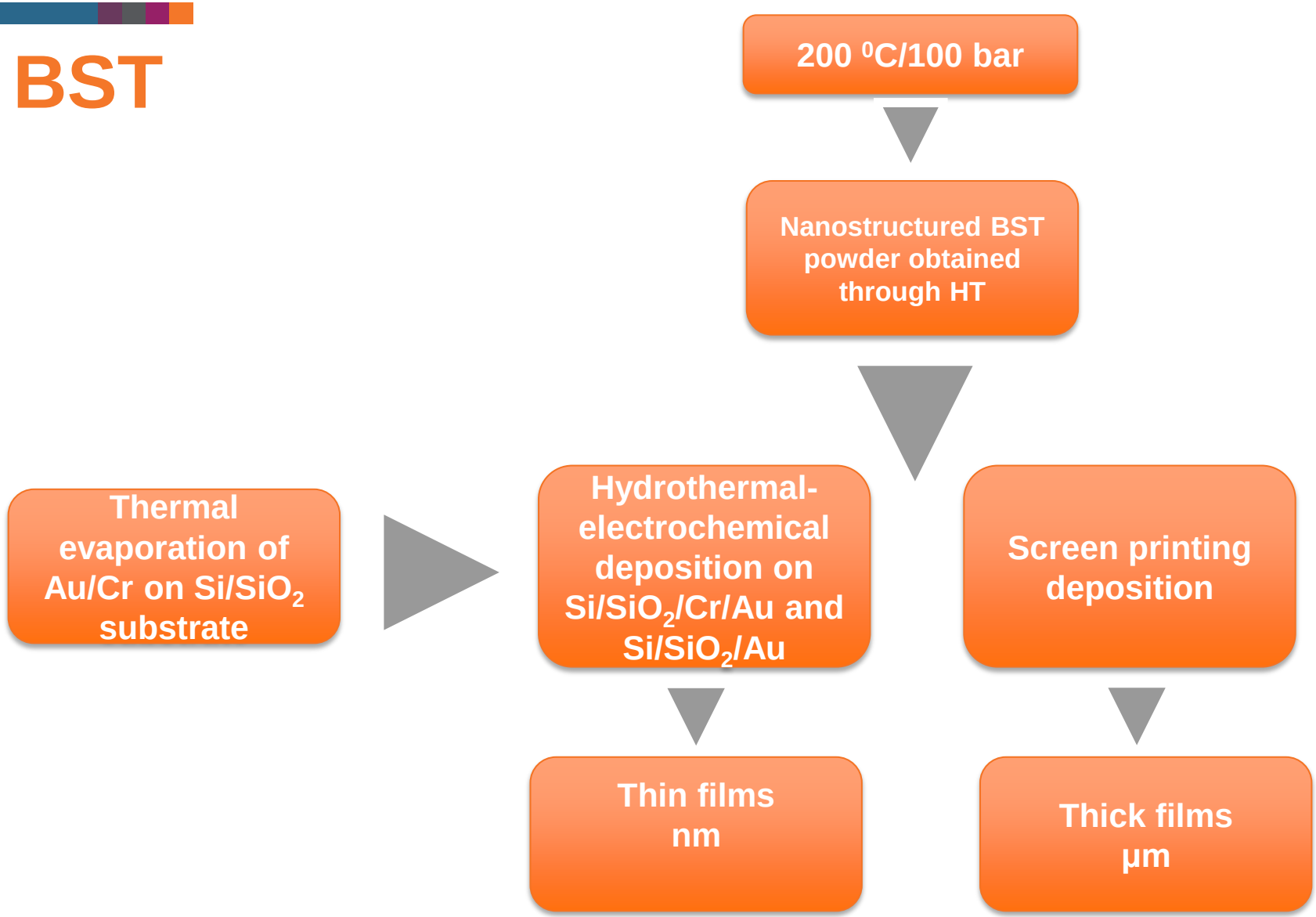
Hydrothermal-Electrochemical system:

1. Coloidal solution;
2. Cortest autoclave 300 °C, 250 bar;
3. Autoclave thermal insulation;
4. Teflon vessel;
5. Working electrode conector (copper);
6. Working electrode (Si;Ti);
7. Auxiliary electrode (Pt coated Nb);
8. Au wire (electrical contact);
9. Reference electrode (Ag/AgCl);
10. Pressure compensation valve;
11. Bledder;
12. Air gauge;
13. PZG 100 potentiostate/galvanostate;
14. booster HVB 100;
15. Computer, VoltaMaster 4 software;
16. PID programmer.

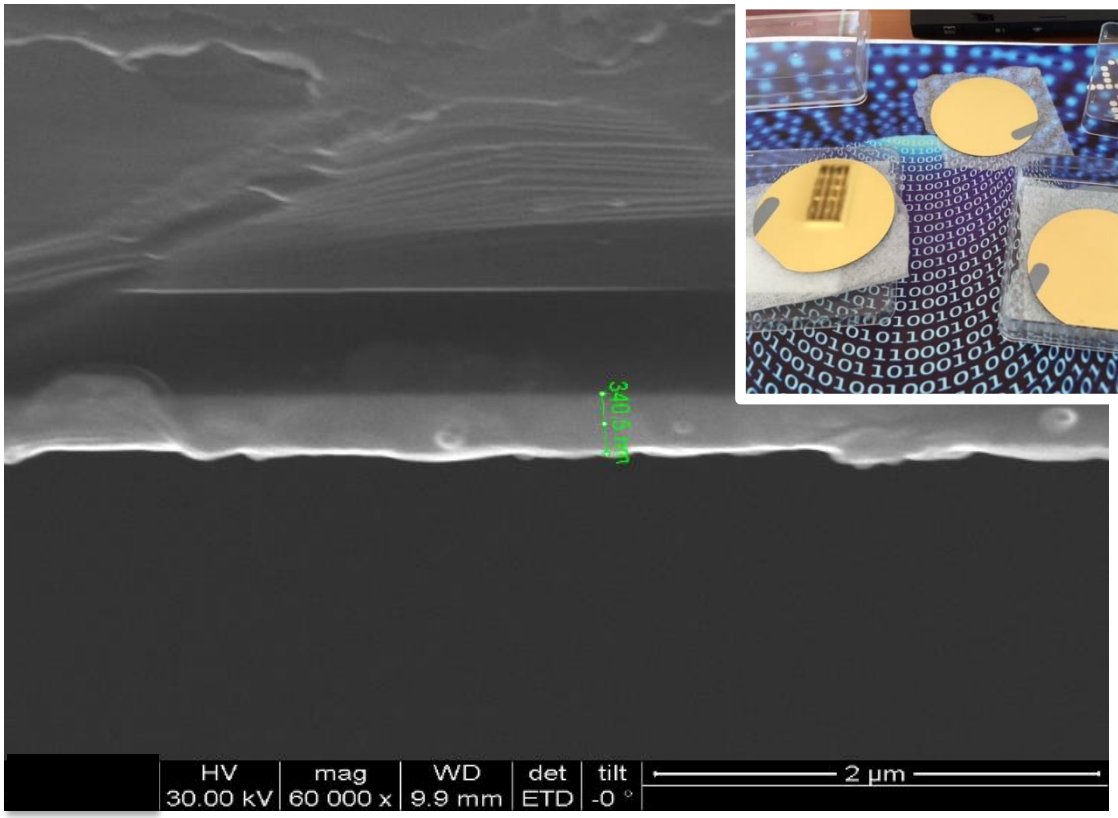
PZT

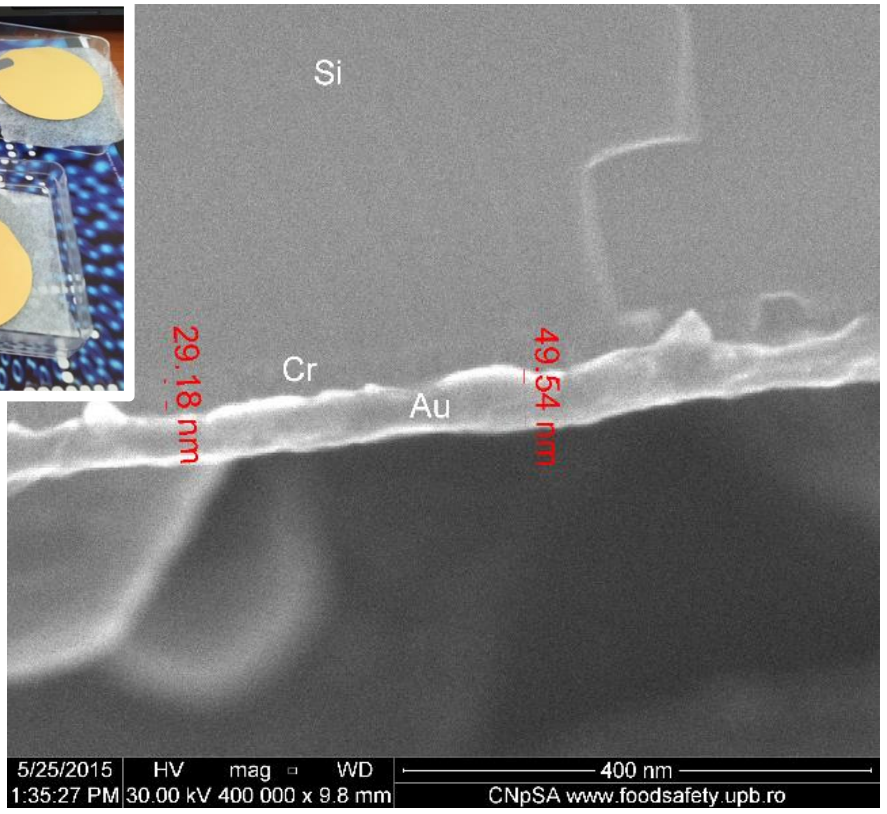

Soluble salts of Pb, Zr, Ti
Mineralising agent
Titanium substrate
Hydrothermal-electrochemical deposition In situ
Thin films
200 ^{0}C/ autogeneous pressure

BST


200 ^{0}C/100 bar
Nanostructured BST powder obtained through HT
Thermal evaporation of Au/Cr on Si/SiO_2 substrate
Hydrothermal-electrochemical deposition on Si/SiO_2/Cr/Au and Si/SiO_2/Au
Screen printing deposition
Thin films nm
Thick films µm

Original results on substrates manufacturing

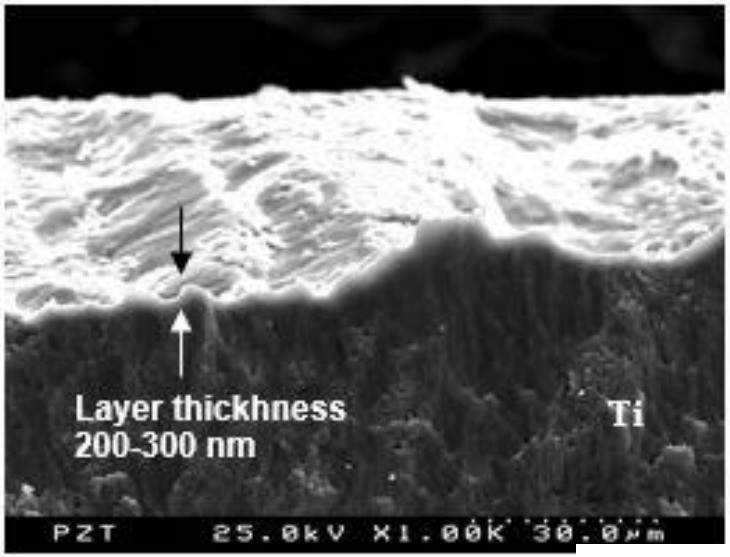




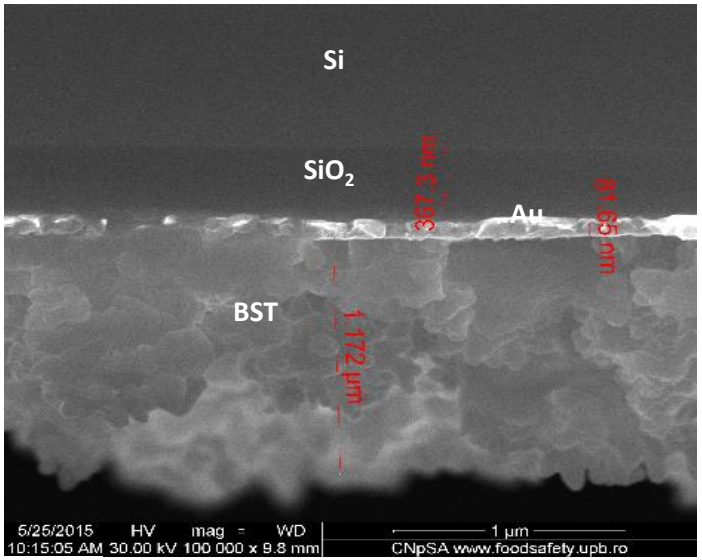
SEM images of the Si/SiO_2/Au substrate (E-beam deposition)

SEM images of the Si/SiO_2/Cr/Au substrate (Thermal - evaporation deposition)

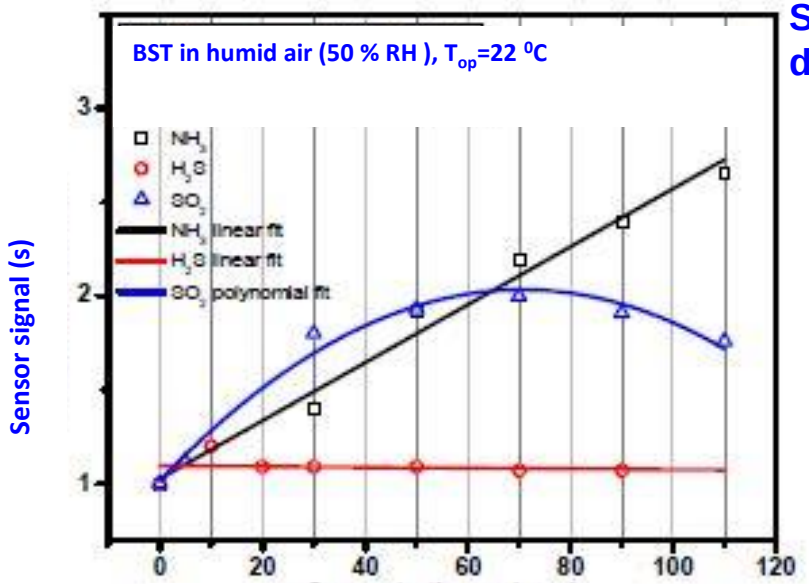
Original results on nanostructured films deposition



SEM images of PZT layer H-E deposition



SEM images of BST layer H-E deposition on $Si/SiO_2/Ti/Au$



Gas concentration influence on sensor signal for un-doped BST (thick films on screen printing)

Performed at National Institute of Materials Physics
(A. Stanoiu and C. Simion)

Scientific context and objectives in the Action

IMNR research work in the field of advanced materials also involves gas sensor materials.

IMNR is member in WG1: Sensor Materials & Nanotechnologies.

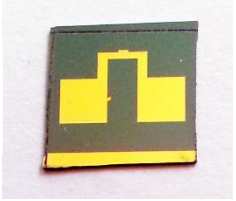
Objectives of WG1:

- **Protocols for synthesis of gas-sensitive nanomaterials;**
- **Protocols for synthesis of functionalized nanostructures for enhanced gas detection at part per- billion (ppb) level, stability and selectivity;**
- **Report on nanomaterials characterization for AQC gas sensors;**
- **Protocols for integration of nanomaterials into micromachined devices and gas sensors;**
- **Protocols for development of gas nanosensors, microsensors and sensors-array.**

Current research activities of the Partner (1/2)

- Nanostructured perovskite materials for SO_2, NH_3, H_2S gas detection (environmental application)
- Nanostructured hybrid materials (organic-inorganic) for VOC detection (medical application)

HE of biomolecules, hydrophobic organic compounds



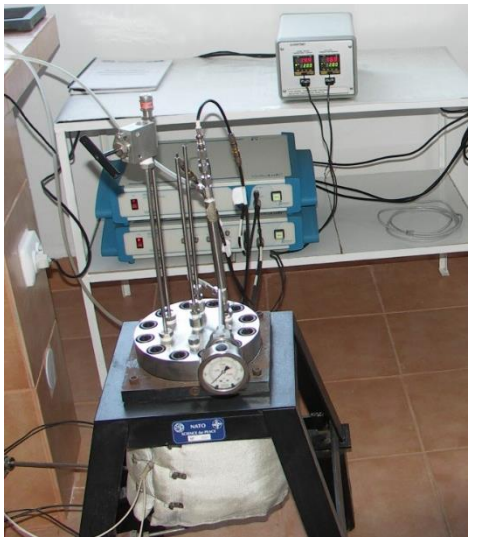
Ongoing Projects with topic in the frame of COST TD 1105:

- Ctr. : 198/2012 SENSGAS - UEFISCDI Romania (Co: IMNR);
- TROPSENSE H2020-RISE (Co: Universitat Rovira i Virgili- Spain, dr. Radu Ionescu);
- COST MP 1402 – HERALD - Hooking together European Research in atomic layer deposition (Co: Tyndall National Institute - Ireland, Dr. Simon Elliott

Research Facilities available, related to EuNetAir subject

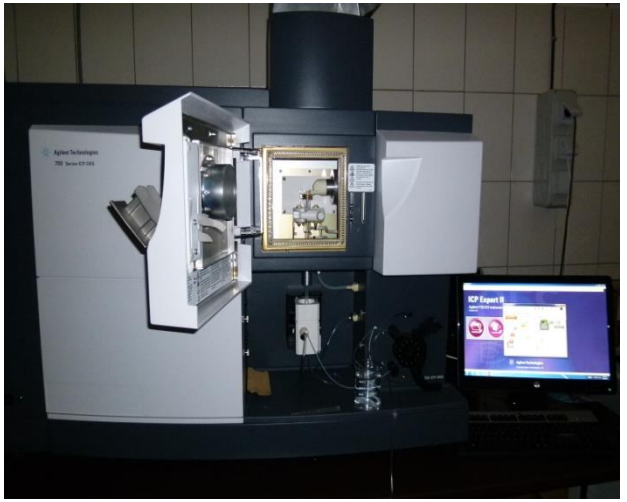
Hydrothermal synthesis autoclave (Berghof)

High Pressure Autoclave (HP Systems)



Cortest autoclave, H-E deposition

E-beam/ thermal evaporation system (Torr Int. Inc.)



ICP-OES System with ablation laser for solids analysis (Agilent Technology)

Spray-drier (LabPlant)

Suggested R&I Needs for future research

- Electrical measurements on thin film based on nanostructured BST, TEM/SEM, XPS on films, it is necessary to establish a mechanism for gas adsorption.
- Standardization on NMs and NPs research.
- Our research is at TRL 4 - the need to translate it to TRL 5-6 as much as possible.

- Acknowledgment:

- Ctr. : 198/2012 SENSGAS - UEFISCDI Romania;
- TROPSENSE H2020 - RISE;
- COST MP 1402 – HERALD - Hooking together European Research in atomic layer deposition.
- PN 09 24 02 07 – ANCSI- Romania

Thank you!

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