



METODOLOGIE AVANZATE DI SINTESI E CARATTERIZZAZIONE DI MATERIALI NANOSTRUTTURATI

CHIM/03 - 8 CFU - 2° Semester

Teaching Staff

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LEARNING OBJECTIVES

- **Knowledge and understanding: Knowledge and understanding:** To acquire theoretical and experimental knowledge of the synthetic procedures of materials in the form of films, ultrathin films and nanostructured systems. To acquire knowledge on the main structural characterization techniques, morphological and compositional materials.
- **Applying knowledge and understanding:** Ability to apply what students have learned during the lectures on the synthesis and characterization of materials in the experiments carried out in the laboratory.
- **Making judgments:** Students learn to evaluate in an objective way how much they understand the lectures and the laboratory activities.
- **Communication skills:** Students acquire communication skills that are developed both during lectures and lab activities thanks to a continuous verbal dialogue with the teacher, and furthermore during the oral exam.
- **Learning skills:** Learning skills are assessed through oral examination and laboratory reports that are an important part of the course.

COURSE STRUCTURE

The course will be done through lectures and some laboratory experiments which allow students to observe experimentally some theoretical topics covered during the course.

If lectures are given in a mixed or remote way, some changes may be introduced from what has been stated above, in order to comply with the programme envisaged and reported in the syllabus.

DETAILED COURSE CONTENT

Course syllabus: Deposition techniques of thin films through chemical (chemical vapor deposition and metal-organic CVD, atomic layer deposition, chemical beam epitaxy, sol-gel) or physical processes (molecular beam epitaxy). Syntheses of nanostructures through vapour phase and solution approaches: effects of process parameters and the use of hard and soft templates. Materials/properties relationship for particular crystalline structures. Perovskite based materials and conduction properties: superconductors, dielectrics and ferroelectrics. Solid oxide fuel cells (SOFCs): electrodes and oxide and proton based conduction electrolytes. Characterization techniques in the fabrication of materials. X-ray diffraction (XRD) of powders, oriented and epitaxial films: phase identification, domain size, rocking curves and pole figures. Electron microscopies: principles and applications of SEM and base concepts of TEM. Energy dispersive X-ray analyses and wavelength dispersive X-ray analyses.

FRONTAL LESSONS (6 CFU)

ADVANCED SYNTHETIC METHODOLOGIES

Atomic layer deposition (ALD)

- Theoretical principles: temporal ALD and spatial ALD
- Case studies: simple oxide deposition (Al_2O_3); photonic crystal synthesis; deposition of multi-component systems.

Chemical Vapor Deposition (CVD) and Metal-Organic-CVD (MOCVD)

- Theoretical principles: transport phenomena; mechanisms and reaction kinetics; nucleation and growth.
- Case studies: deposition of simple oxides and multi-component oxides; fluoride film deposition.
- Processes of industrial interest: deposition of GaN, AlN and SiC.

Molecular beam epitaxy (MBE)

- Theoretical principles.
- Applications to the growth of epitaxial films.

Chemical Beam Epitaxy

- Theoretical principles and applications.

Sol-gel deposition techniques

- Theoretical principles.
- Applications to film synthesis, hybrid systems, nanoparticles by Stober process.

Precursors for vapor phase and solution processes

Synthesis of nano structured materials

- Definition of nanostructures.
- Synthetic approaches for the synthesis of nanostructures.
- Nanorod synthesis and vapor phase nanotubes.
- Synthesis of nanoparticles and solution nanostructures

Monolayer synthesis

Molecular layer deposition for the deposition of monolayers.

Notes on the synthesis of self-assembled monolayer from solution.

TYPES OF MATERIALS

- Perovskite structure
- Structure description and tolerance factor

Structure-property relationship

- Perovskites with ionic conduction properties
- Description of ionic conductivity of intrinsic and extrinsic type.
- Solid oxide fuel cells.

Perovskites with dielectric properties

- Piezoelectric, pyroelectric and ferroelectric.
- Applications in energy harvester.
- Notes on piezotronic, piezophotonic and piezo-phototronic

Perovskite-based materials with superconducting properties

- Basic and superconducting principles I and II type. -
- Outline of BCS theory.
- Case studies of systems based on high T_c superconductors

CHARACTERIZATION METHODOLOGIES

X-ray production.

- Information obtainable from a diffractogram: amorphous or crystalline nature, phase identification, grain size.

- Crystalline lattices and systematic absences.
- Diffractions from powders and thin films: calculation of cell parameters.
- Notes on the single crystal: Ewald sphere.
- Advanced characterization of materials: rocking curves and polar figures in the characterization of oriented and epitaxial samples

Electronic scanning microscopy

- General principles.
- Tungsten filament electron beam, LaB6 crystal, or FEG (Field Emission Gun).
- Volume of interaction, elastic and inelastic events, species produced.
- Detector of secondary and backscattered electrons.

EDX Microanalysis (Energy Dispersive X-ray Analysis)

- Detector type
- Qualitative and quantitative analysis (ZAF method).
- Maps and scan-lines via EDX.

WDX (Wavelength Dispersive X-ray Analysis) microanalysis

- Spectrometer and crystal type description
- Qualitative and quantitative analysis.
- Advantages and disadvantages of the two microanalysis

LABORATORY EXPERIMENTS (2 CFU)

Experiments carried out:

- 1) syntheses of precursors: Sr(hfa)₂tetraglyme, La(hfa)₃diglyme, Ni(tta)₂tmeda, Eu(tta)₃phen;
- 2) Characterization of physico-chemical properties of synthesized precursors (m.p., FT-IR and UV-Vis spectra; thermogravimetric analyses);
- 3) Fabrication of oxides and fluoride or oxyfluoride films through MOCVD;

- 4) Application of the europium complex to the formation of a luminescent hybrid layer as solar concentration;
- 5) Morphological (SEM) and compositional (EDX) characterization of synthesized materials;
- 6) Structural characterization through XRD of synthesized materials.

SEMINAR ACTIVITIES:

Lanthanides and rare earths: unknown elements in everyday life

TEXTBOOK INFORMATION

- 1) A. R. West "Basic Solid State Chemistry and its Applications" Wiley, 2012;
- 2) B. D. Fahlman "Materials Chemistry" 3rd Edition, Springer, 2018;
- 3) Editor R. Fisher, "Precursor Chemistry of Advanced Materials: CVD, ALD and Nanoparticles", Springer, 2010.
- 4) L. V. Interrante e M. J. Hampden-Smith *Chemistry of Advanced Materials* Wiley-VCH, 1998.

Lecture handouts.
