



IV Brazilian MRS Meeting

16-19 October 2005

Recife, Pernambuco

**PLENARY AND
INVITED TALKS
SUMMARIES**



Anderson S. L. Gomes
Local Chair
Physics Department
Universidade Federal
de Pernambuco



Celso P. de Melo
National Chair
Physics Department
Universidade Federal
de Pernambuco

Welcome Address by IV Brazilian MRS Meeting National and Local Chairs

Dear IV Brazilian MRS Meeting Participant,

It is with great pleasure that we would like to give you a warm welcome to the IV Brazilian MRS Meeting, (IV SBPMat) in Recife from 16th-19th October 2005.

Among the several participants in the technical sessions, we will have 04 talks and 33 invited speakers, in a total number of 793 contributions to the different Meeting Symposia.

This part of the proceedings contains all the available summaries of the plenary and invited talks, as well as a short biography of each speaker.

We would like to use this opportunity to thank all the invited and plenary speakers for accepting the program coordinators' invitation.

Again, a very warm welcome to Recife to all of you.

Anderson S. L. Gomes and Celso P. de Melo
Physics Department
Universidade Federal de Pernambuco
IV MRS Meeting Local and National Chairs

Plenary Sessions

Plenary Sessions

Room Manuel Bandeira I, II, III and IV

Monday	2:15/3:15pm	Plenary I: (Title to be announced)	Page 4
Oct. 17		Dr. Barry Carter - Department of Chemical Engineering and Materials Science University of Minnesota	
	6:00/7:00pm	Plenary II: Construction of Molecular Nano-Structures and Electronic Properties	Page 4
		Dr. Takuji Ogawa - Center for Molecular-Scale Nanoscience, Institute of Molecular Science – IMS/Okazaki	
Tuesday	2:15/3:15pm	Plenary III: Multifunctional Organic/Inorganic Hybrids	Page 5
Oct. 18		Dr. Luis A. Ferreira M. Dias Carlos - Departamento de Física da Universidade de Aveiro.	
	6:00/7:00pm	Plenary IV: From High-Temperature Superconductivity to Complex Electron Systems Science	Page 6
		Dr. H. Takagi - Magnetic Materials Laboratory, RIKEN'S Discovery Research Institute.	

Plenary I (Title to be announced)

Dr. Barry Carter - Department of Chemical Engineering and Materials Science University of Minnesota

Plenary II

CONSTRUCTION OF MOLECULAR NANO-STRUCTURES AND ELECTRONIC PROPERTIES

Dr. Takuji Ogawa - Center for Molecular-Scale Nanoscience, Institute of Molecular Science – IMS/Okazaki

Construction of molecular nano-structures and their electronic properties

Takuji Ogawa

¹ Research Center for Molecular-Scale Nanoscience, Institute for Molecular Science, Okazaki, Aichi 444-8787, Japan

² CREST, Japan Science and Technology Corporation (JST), Kawaguchi, Saitama 332-0012, Japan

³ The Graduate University for Advanced Studies, Okazaki, Aichi 444-8787, Japan

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Molecular electronics is a fairly new and fascinating area of research that is firing the imagination of scientists. However, most organic molecules are not “conductive” in a classical meaning, and long range electronic transfer through the molecules can not be so effective to realize practical electronic circuits. Our research center on (1) construction of nano-structures made from conductive materials such as carbon nanotubes, metal particles or rods, with functional organic molecules, and (2) measurements of their electric or photonic properties while observing their nanometric images. Fig.1 shows a schematic diagram of self-assembled film consisted of dithiol and Au nano-particles between 1 μ m gap Au electrodes. Fig.2 exhibits AFM image of single wall carbon nanotube covered with porphyrin molecules. Electric properties of these systems will be discussed.

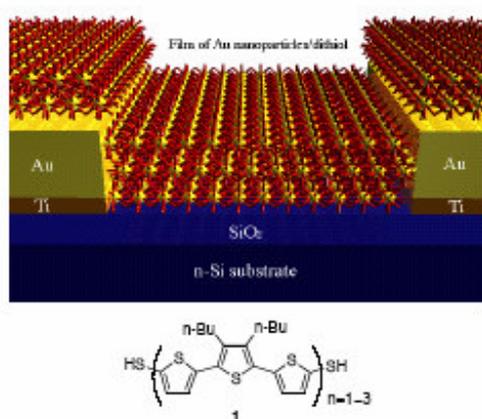


Fig. 1 Schematic diagram of self-assembled film consisted of dithiol **1** bridged active Au-NPs between 1 μ m gap Au electrodes.

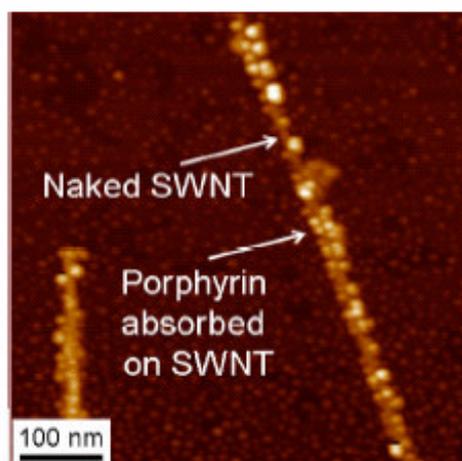


Fig. 2 Atomic force microscopic image of SWNT covered with porphyrin molecules.

Plenary III

MULTIFUNCTIONAL ORGANIC/INORGANIC HYBRIDS

Dr. Luis A. Ferreira M. Dias Carlos - Departamento de Física da Universidade de Aveiro.

Luís António Ferreira Martins Dias Carlos was born in Coimbra, Portugal, in September 1964. Following his undergraduate studies in Physics at Coimbra University he became Assistant in the Physics Department of Évora University in 1987. He received his PhD degree on physics from this University in June 1995 with a thesis entitled: "Optical and Structural Features of Polymer Electrolytes Incorporating Lanthanide Salts". At 1996 joined the Department of Physics at the University of Aveiro and since 1998 he is Professor Associado there. His current scientific activities include photoluminescence of (i) sol-gel derived organic/inorganic hybrids incorporated lanthanide ions, (ii) lanthanide-based micro and mesoporous materials, (iii) lanthanide coordination complexes, and small-angle X-ray scattering of organic/inorganic nanostructured multifunctional materials.

Dr. L.D. Carlos is co-author of over 120 scientific papers and is member of the American Physical Society, European Physical Society, Portuguese Physical Society, Materials Research Society, Electrochemical Society and European Rare Earth and Actanide Society

<http://sweet.ua.pt/~lcarlos/curriculum/curriculum.html>

Multifunctional organic/inorganic hybrids

L. D. Carlos

Departamento de Física e CICECO, Universidade de Aveiro, 3810-193 Aveiro, Portugal

Abstract –The main applications of organic/inorganic hybrids (OIHS) is reviewed with a particular focus on the discussion of the multifunctionality of a series of amine and amide-functionalized OIHS.

The drive to miniaturization that occurred during the last two decades — with the corresponding demand for smaller machines and components using less resources and energy — have been rapidly pushing industry into the atomic and nanometer scale. The development of new synthesis strategies for advanced materials with enhanced properties and affording an effective control at the nanometer level is therefore required. Amongst the available synthetic methods employed for the development of nanosystems, the "bottom-up" approach allows the creation of organic/inorganic and/or inorganic structures using atoms or molecules as building blocks to design and assembly nanoarrangements of atoms in a functional form to give macroscopic systems. The birth of "soft" inorganic chemistry processes, in particular the sol-gel via, allows the chemical design of pure and well-controlled multifunctional organic/inorganic hybrid materials. The resulting synergies open up exciting directions in materials science research and related technologies with implications in the processing of novel multifunctional advanced materials with innovative performances. Moreover, the ability to tailor materials properties over broad length scales suggests that research on hybrids can significantly impact diverse fields, such as nanophotonics, magnetism, catalysis, separation techniques, smart coatings, sensors, biomedical and ceramic and polymer composite applications¹⁻³.

Increasing attention has been focused in the last few years on the photonic features of stable and efficient amine- and amide-functionalized organic/inorganic hybrids, such as those obtained from 3-aminopropyltrimethoxysilane (APTES) with carboxylic acids and urea (di-ureasils), urethane (di-urethanesils) and amide-based (amidosils) cross-linked xerogels, due to their intrinsic and efficient room-temperature white light emission with significant quantum yields (QY) (20-35 %) ^{4,5}.

After a review of some of the main applications of OIHS (for protective and decorative coatings, for barrier systems, for biomedical and dental applications, for microelectronics and micro-optics, for batteries, for proton-exchange membrane fuel cell applications and for photovoltaic cells)³, the talk will be focused on the properties of a series of di-ureasils, di-urethanesils, APTES-based OIHS (aminosils) and amidosils. Samples with polymer chain of different molecular weights and with cross-linkages in one or in the two sides of the polymer segments were investigated. All the OIHS are room temperature white-light emitters, due to the convolution of a blue component associated with donor-acceptor (D-A) recombinations occurring in the NH groups of the urea, urethane and amide linkages with a purplish-blue D-A emission originated by oxygen related defects (*O–O–Si≡(CO₂)) in the siliceous nanodomains⁶. Particular attention will be devoted to examples that show the subtle relation between modifications in the local structure of these materials and the concomitant alterations on their emission features. The multifunctionality of these organic/inorganic hybrids is nicely illustrated by the discussion on the magnetic features of iron-based OIHS.

References

- [1] C. Sanchez, B. Lebeau, F. Chaput, J.-P. Boilot, *Adv. Mater.* **15**, 1969 (2003).
 - [2] L. D. Carlos, R. A. Sá Ferreira, V. de Zea Bermudez, in Handbook of Organic-Inorganic Hybrid Materials and Nanocomposites, ed. H. S. Nalwa, Eds., American Scientific Publishers, Morth Lewis Way, California, Vol. 1, Chapter 9, 2004, 353.
 - [3] C. Sanchez, B. Julian, P. Belleville, M. Popall, *J. Mat. Chem.* **15**, 3559 (2005).
 - [4] W. H. Green, K. P. Le, J. Grey, T.T. Au, M.J. Sailor *Science*, **276**, 1826 (1997).
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 - [6] L. D. Carlos, R. A. Sá Ferreira, R. N. Pereira, M. Assunção, V. de Zea Bermudez, *J. Phys. Chem. B.* **108**, 14924 (2004).
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Plenary IV

FROM HIGH-TEMPERATURE SUPECONDUCTIVITY TO COMPLEX ELECTRON SYSTEMS SCIENCE

Dr. H. Takagi - Magnetic Materials Laboratory, RIKEN'S Discovery Research Institute.

From High-Temperature Supeconductivity to Complex Electron Systems Science

Dr. H. Takagi - Magnetic Materials Laboratory, RIKEN'S Discovery Research Institute.
Department of Advanced Materials, University of Tokyo, Kashiwa 277-8651, Japan
RIKEN (The Institute of Physical and Chemical Research), Wako 351-0198, Japan

An overview is given on the recent progress in understanding the mechanism of high temperature superconductivity in layered cuprates. The topics include competing orders, hidden order in pseudo-gap phase, universality of magnetic excitations and electron-phonon coupling. The fascinating physics of correlated electrons discovered in cuprates has been stimulating the exploration for the many faces of strongly correlated electron systems. Selected topics along this line will be also discussed.

+supported by CREST-JST

INVITED TALKS

Symposium A - Synthesis and Characterization of Nanocomposites **Page 10**

MoSA-I-1 **8:00/9:00am**
NANOPARTICLES SINGULARITIES: ADHESION EFFECTS IN NANOCOMPOSITES INTERFACES
Fernando Galembeck, UNICAMP/SP

TuSA I-2 **8:00/9:00am**
GLASS DYNAMICS IN THE EXCHANGE BIAS PROPERTIES OF NANOGANULAR MAGNETIC SYSTEMS
Dino Fiorani, CNR, Roma

WeSA I-3 **8:00/9:00am**
PROPRIEDADES CATALÍTICAS E MAGNÉTICAS DE NANOPARTÍCULAS METÁLICAS DISPERSAS EM LÍQUIDOS IÔNICOS
Jairton Dupont, UFRGS, SP

Symposium B - Supramolecular Materials and Organic Devices **Page 11**

MoSB-I-1 **8:00/9:00am**
ORGANIC LIGHT EMITTING DIODES BASED ON RARE EARTH COMPLEXES
Marco Cremona, IF-PUC/RJ

MoSB-I-2 **9:45/10:15am**
CONDUCTING POLYMERS, SURFACTANTS, COLLOIDS
Celso P. de Melo, DF-UFPE/PE

MoSB-I-3 **10:30/11:00am**
ORGANIC LIGHT EMITTING DIODES: TECHNOLOGICAL CHALLENGES AND APPLICATIONS
Roberto Faria, USP/São Carlos

TuSB -I-5 **8:00/9:00am**
HYBRID METAL BASED TRANSISTORS
Ivo Hummelgen, DF/UFPR, PR

TuSB -I-6 **9:45/10:15am**
SENSORS AND BIOSENSORS MADE OF LAYER-BY-LAYER FILMS
Osvaldo Novaes Jr.

TuSB -I-7 **10:30/11:00am**
MOLECULAR NANODEVICES DEVELOPED AT THE LAND-FOTON/DQF/UFPE
Petrus Santa Cruz, DQF/PE
Biography and Summary Not Available

TuSB -I-8 **11:45/12:15am**
SUPRAMOLECULAR MATERIALS AND DEVICES
Koiti Araki, IQ-USP

Symposium C - Biocompatible Materials **Page 17**

TuSC-I-1 **8:00/9:00am**
INJECTABLE BIOMATERIALS FOR BONE REGENERATION
Pedro Granja INEB- Univ. do Porto, Portugal

TuSC-I-2 **10:30/11:30am**
BIORESORBABLE SCAFFOLDS PREPARED BY PARTICULATE LEACHING METHOD FOR TISSUE ENGINEERING
Samuel H. Barbanti, UNICAMP, SP

WeSC -I-5 8:00/9:00am
CONTRIBUTION WITHDRAWN

WeSC-I-4 10:30/11:30am
RAPID PROTOTYPING APPLICATIONS IN THE TREATMENT OF CRANIOMAXILLOFACIAL DEFORMITIES – UTILIZATION OF BIOMATERIAIS
Jorge L. Silva, CenPRA/Campinas/SP

Symposium D - Structural materials: Processing Properties and Applications Page 20

MoSD-I-1 8:00/9:00am
MATERIALS FOR THERMAL PROTECTION OF SPACE VEHICLES
Carlos Alberto Alves Cairo, IAE-CTA/SP

TuSD I-2 8:00/8:30am
RECICLAGEM DE REJEITOS DA INDÚSTRIA CERÂMICA TRADICIONAL COMO AGREGADOS PARA ARGAMASSA DE ALVENARIA E CONCRETO DO TIPO PORTLAND
João Batista Baldo, UFSCar, SP

TuSD I-3 8:30/9:00am
NON-EQUILIBRIUM SOLIDIFICATION OF UNDERCOOLED METALLIC MELTS
Walman Benicio, DEM – UFCG, PB

WeSD-I-4 8:00/9:00am
PROCESSING VS. STRUCTURE VS. MECHANICAL AND CORROSION BEHAVIOUR OF ZRNXYOY THIN FILMS
Luís Augusto Rocha, Universidade do Minho, Portugal

Symposium E - Advances in Photonics Materials and applications Page 22

MoSE-I-1 8:00/9:00am
CONTRIBUTION WITHDRAWN

MoSE-I-2 10:30/11:30am
HOLOGRAPHIC TECHNIQUES TO STUDY PHOTSENSITIVE MATERIALS
Lucila Cescato, UNICAMP, SP

MoSE-I-3 12:00am/12:30pm
RARE EARTH DOPED TRANSPARENT FERROELECTRIC CERAMICS FOR PHOTONIC APPLICATIONS.
Andrea Simone Stucchi de Camargo, IF-USP, São Carlos

TuSE-I-4 8:00am/9:30pm
RECENT ADVANCES AND CHALLENGES IN THE PHYSICS OF SOLID STATE LIGHTING.
Fernando A. Ponce, IF-USP, São Carlos

MoSE-I-5 12:00am/12:30pm
LASER-INDUCED REFRACTIVE INDEX CHANGES IN PHOTONIC MATERIALS
Tomaz Catunda, IF-USP, São Carlos

Symposium F- Magnetic Materials: Preparation, Characterization and Applications Page 27

MoSF-I-1 8:00/9:00am
PERMANENT MAGNETS - RECENT DEVELOPMENTS
Frank P. Missell Universidade de Caxias do Sul, RS

MoSF-I-2 10:30/11:30am
NANOSCALE PROPERTIES OF MAGNETIC COLLOIDS
Jérôme Depeyrot UnB, DF

TuSF-I-3 **8:00/9:00am**
THE MAGNETOCALORIC EFFECT: MATERIALS AND APPLICATIONS
Sergio Gama, UNICAMP, SP

Symposium G - Superconductor Materials **Page 29**

TuSG-I-1 **10:30/11:00am**
RESEARCH AND DEVELOPMENT OF HIGH-TC SUPERCONDUCTORS IN CEPEL-ELETROBRÁS
Alexandre Polasek, CEPEL-Eletróbrás,RJ

WeSG I-2 **8:00/8:30am**
ON THE PROPERTIES AND CHARACTERISTICS OF SUPERCONDUCTORS WITH ARTIFICIALLY INTRODUCED NANOMETRIC-SCALE PINNING CENTERS
Durval Rodrigues

WeSG – I-3 **8:30/9:00am**
STRUCTURAL, MICROSTRUCTURAL, ELECTRICAL AND MAGNETIC PROPERTIES OF RUTHENIUM PHYROCLORES
José Albino Aguiar

WeSG-I-4 **9:00/9:30am**
UNIVERSAL ASPECTS OF THE MAGNETIC RESPONSE OF GRANULAR SPECIMENS HAVING A NARROW WEAK-LINK CRITICAL CURRENT DISTRIBUTION
Wilson Ortiz

Symposium H - Sol-Gel Materials **Page 34**

TuSH-I-1 **8:00/8:30am**
DFB EMISSION OF RHODAMINE CONTAINING ORGANIC-INORGANIC HYBRID MATERIALS
Sidney J.L. Ribeiro

TuSH-I-2 **9:45/10:15am**
X-RAY ABSORPTION SPECTROSCOPY AND SOL-GEL CHEMISTRY
Valerie Briois

TuSH-I-3 **10:30/11:00am**
SOL-GEL COATINGS ON METALLIC SUBSTRATES
Wander Vasconcelos

WeSH-I-4 **9:45/10:15am**
DESIGN OF INORGANIC MATERIALS BASED ON DYNAMICS OF SOFT MATTER
Eric Prouzet

WeSH-I-5 **10:30/11:00am**
SILICA-CONTAINING PROTON CONDUCTING MATERIALS FOR FUEL CELL APPLICATION
Marcos Lopes Dias

INVITED TALKS**Symposium A - Synthesis and Characterization of Nanocomposites****MoSA-I-1****8:00/9:00am****NANOPARTICLES SINGULARITIES: ADHESION EFFECTS IN NANOCOMPOSITES INTERFACES**

Fernando Galembeck, UNICAMP/SP

Biography and Summary Not Available

TuSA I-2**8:00/9:00am****GLASS DYNAMICS IN THE EXCHANGE BIAS PROPERTIES OF NANOGRANULAR MAGNETIC SYSTEMS**

Dino Fiorani, CNR, Roma

Biography

O Professor Dino Fiorani coordena o Grupo de "Materiais Magnéticos Nanoestruturados" do Instituto de Estrutura da Matéria, ISM-CNR, Área da Pesquisa de Roma. É membro da IUPAP e, entre outras atividades, foi chairman da 16ª Conferência Internacional de Magnetismo (ICM-International Conference on Magnetism), promovida pelo Conselho Nacional das Pesquisas su delega da Comissão de Magnetismo dell'Unione Internazionale di Fisica pura e Applicata (IUPAP). O Professor Dino possui mais que cento e oitenta publicações, todas em revistas internacionais indexadas na base de dados "Web Of Science".

Professore Dino Fiorani

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WeSA I-3**8:00/9:00am****PROPRIEDADES CATALÍTICAS E MAGNÉTICAS DE NANOPARTÍCULAS METÁLICAS DISPERSAS EM LÍQUIDOS IÔNICOS**

Jairton Dupont, UFRGS, SP

Biography and Summary Not Available

Symposium B - Supramolecular Materials and Organic Devices

MoSB-I-1

8:00/9:00am

ORGANIC LIGHT EMITTING DIODES BASED ON RARE EARTH COMPLEXES

Marco Cremona, IF-PUC/RJ

Biography

Affiliation and official address:

Pontifícia Universidade Católica do Rio de Janeiro

Address: PUC-Rio, Depto. Física, Rua Marques de São Vicente 225, Rio de Janeiro, RJ, Brazil

Date and place of birth: 02/03/1963, Rome, Italy **Nationality:** Italian

E-mail: cremona@fis.puc-rio.br

Home Page: <http://www.fis.puc-rio.br/html1/docentes/cremona/cremona.htm>

Education (*degrees, dates, universities*)

Graduated in Physics 28/09/1989 at University of Rome "La Sapienza", Italy

PhD in Physics 19/09/2000 at PUC-Rio, Brazil

CNPq Research Group: Espectroscopia Óptica e Optoeletrônica Molecular

CNPq Fellowship

Career/Employment (*employers, positions and dates*)

Dep. of Physics PUC-Rio	Professor	2000-present
Dep. of Physics PUC-Rio	Senior Researcher	1999-2000
Dep. of Materials Science	Senior Researcher	1996-1999
ENEA, Frascati (RM), Italy	research fellow	1989-1994
INFN-CNR, Rome, Italy	research fellow	1995-1996

Specialization (*specify*)

(i) main field

Integrated optics, organic electroluminescent devices (OLEDs), thin films, spectroscopy, material characterization, laser optics.

(ii) other fields

Optical fibers, sensors,

(iii) current research interest

Integrated optical devices, OLEDs, waveguide in new materials.

Honours, Awards, Fellowships, Membership of Professional Societies

Member of SIF (Italian Society of Physics)

Member of SBF (Brazilian Society of Physics)

Member of APS (American Physical Society)

Bunshah Award - Best paper in Thin Solid Films in 2004 (American Vacuum Society).

Publications

- **Number of papers in refereed journals:** about 50
- **Number of communications to scientific meetings:** about 60
- **Other technical publications:** 5
- **Patents:** 3 (1 pending)

Organic Light Emitting Diodes based on rare earth complexes

Marco Cremona

Laboratório de Optoeletrônica Molecular (LOEM) - Departamento de Física
Pontifícia Universidade Católica do Rio de Janeiro, PUC-Rio

Solid state miniaturized light sources are very important as much for the scientific point of view as for the industrial one. As an example, today's handled electronics commonly incorporate LCD and TVs displays with white LEDs as dominant backlight solution. Due to their applications in many different areas and manufacturing simplicity, the OLEDs (Organic Light Emitting Diodes) represent a promising research in the development of new optoelectronic and photonic devices. These electroluminescent (EL) devices have the advantage of ease of fabrication, low operating voltages, and the possibility of a wide selection of emission colors through the molecular design of organic materials. Generally, OLEDs are assembled using an heterojunction architecture between three or more organic molecular materials: an electron injection layer, the emitting one and finally the hole injection layer. The research in new electroluminescent organic materials comes earning, therefore, a great impulse in searching new molecules or organic complexes that increase the OLED stability, making possible new applications. Trivalent rare earth ions (RE^{3+}) have been used as emitting centers and the range of materials now available for organic molecular-based light-emitting diodes (OLEDs) with lanthanide complexes is such that by selecting an appropriate compound for the emission layer, electroluminescence (EL) can be obtained throughout all the visible spectrum. Among the RE-complexes those based on β -diketones are very interesting since they present high luminescence intensity. The ligand excitation (by UV light or electrical) is followed by efficient intramolecular energy transfer from the ligand excited states to the lanthanide emissive levels, yielding characteristic and strongly luminescent narrow band of the $f-f$ electronic transitions. In this talk the fabrication and the characterization of different kind of OLED using a β -diketones binuclear complexes as emitting and transporting layer is reported and discussed.

MoSB-I-2

9:45/10:15am

CONDUCTING POLYMERS, SURFACTANTS, COLLOIDS

Celso P. de Melo, DF-UFPE/PE

Biography



Celso Pinto de Melo received the Laurea degree in Chemical Engineering (1973) and a MSc in Physics (1975) from the Universidade Federal de Pernambuco (UFPE, Brazil) and a PhD in Physics (1980) from the University of California in Santa Barbara. At present he is Graduate and Research Dean of UFPE, where he has a permanent position at the Physics Department. He has supervised several graduate students and authored more than 90 theoretical and experimental articles on different aspects the electronic structure, nonlinear optical properties and electrical characterization of conducting polymers, thin organic films and colloidal systems. In 1986–1987 he has been a Fulbright visiting scholar at the Massachusetts Institute of Technology and is an associated member of the Brazilian Academy of Sciences. In the period 1999–2002 he was Scientific Director of the Brazilian National Science and Technology Council (CNPq). He has served in several national science committees and in 2002 received the Brazilian National Order of Scientific Merit for his contributions to Physics in Brazil. At present, his main research interests are concentrated in the investigation of the electrical and spectroscopic properties of conducting polymer/metal nanocomposites and in their possible uses as sensors and as photoactive materials.

Conducting Polymers, Surfactants, Colloids

Celso P. de Melo

Departamento de Física, Universidade Federal de Pernambuco, Brazil, 50670-901

Abstract – Polymeric blends with controlled conductivity and metal/(conducting polymer) nanoparticles are two specific examples of the use of the technique of emulsion polymerization to prepare micelles of conducting polymers and surfactants. The physical characteristics of these

colloidal systems, taken as representative examples of the organization of “soft matter” at the mesoscale, are investigated by electrical impedance spectroscopy. Possible uses of these systems in electronic nose instruments and as active materials in electroluminescent devices and photovoltaic cells are discussed.

Each one of the different examples of complex systems that constitute the rich diversity of “soft matter” can be understood as a particular point in a triangular phase diagram that has as independent vertices the fundamental characteristics of polymers, surfactants and colloidal systems, respectively. In the last few years, the Non-Conventional Polymers (PNC) Group at the Physics Department of UFPE has expanded its original expertise on the electronic structure and transport properties of conducting polymers and thin organic films to exploit the flexibility in structural arrangements displayed by polymers, proteins, surfactants and colloids. We investigate the underlying physical principles operating when the soft matter is organized at the mesoscopic scale and propose new supramolecular structures involving conducting polymers and metallic aggregates. Frequently, in these systems at least one of the dimensions belongs to the nanometric scale, and non-covalent interactions (such as hydrogen bonds, polar attractions, van der Waals forces and competition between hydrophilic-hydrophobic forces) are the ones determining the long-range order.

At the PNC laboratory we have taken advantage of the technique of emulsion polymerization to prepare surfactant micelles that can act as microreactors for the selective preparation of polymeric structures. The balance between the forces contributing to the collapse of the polymeric chains as a whole and to their stabilization by the interaction with the surfactant molecules results in an optimum dimension for which the metallic agglomerates are stabilized by the amount of surfactant present. The technique of electrical impedance spectroscopy has been developed as a convenient tool to characterize the process of micelle formation.

In this presentation we will examine some recent work done along two possible directions.

Polymeric blends with controlled conductivity: When oxidizing agents are incorporated into the interior of a dielectric matrix it becomes possible to induce a controlled polymerization of conducting polymers of interest by exposition to the corresponding monomer in vapor phase. We have adopted this procedure to prepare blends of polypyrrole that find convenient use as active materials in electronic nose equipments. When micelles comprising metallic particles enveloped by conducting polymer chains are dispersed in the interior of the dielectric matrix, convenient systems of hybrid nature are produced. We are using this technique to prepare heterojunctions involving photoactive materials with promising properties for the development of electroluminescent devices and photovoltaic cells.

Metal/Polymer Nanoparticles: Whenever the dimensions of noble metals particles are reduced beyond the mean free path for the electrons, an intense absorption in the near UV-visible region arises, as demonstrated by the characteristic bright color of the colloidal suspensions of these metals. We have used conducting polymers (such as polypyrrole) to stabilize the metallic clusters and control the resulting luminescence of silver and gold aggregates prepared in this manner. Due to the exquisite sensitivity of their spectroscopic properties to the preparation conditions, metal/(conducting polymer) nanocomposites appear as promising systems to be used as biological sensors.

Several possibilities of further development of above line of research will be discussed

References

- [1] M. Gavarelli, F. Bernardi, M. A. Robb and M. Olivucci, *Journal of Molecular Structure (Tetrahedron)* 463, 59 (1999).
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- [4] R. J. Sengwa, R. Chaudhary and S. C. Mehrotra, *Polymer* 43, 1467, (2002).
- [5] A. C. Tenorio, H. P. de Oliveira, A. M. S. Silva, et al., *Physica Status Solidi B-Basic Research* 232, 50 (2002).

MoSB-I-3

10:30/11:00am

ORGANIC LIGHT EMITTING DIODES: TECHNOLOGICAL CHALLENGES AND APPLICATIONS

Roberto Faria, USP/São Carlos

Biography

Brazilian, married, born in May 26, 1952

Present Position: Full Professor at Institute of Physics of São Carlos, University of São Paulo

Address: Av. Do Trabalhador São-carlense, 400, C. P. 369, 13560-970, São Carlos, Brazil

Telephone: 55-16-33739758; fax: 55-16-33713365; e-mail: faria@if.sc.usp.br

Education and Degrees

B. in Physics (1975); Master in Physics (1981); PhD (1984)

Teaching

Extensive experience of teaching graduates and undergraduates at IFSC/USP.

Graduate and pos-doc supervisions (Completed)

14 M.Sc., 15 Ph.D., and 5 Pos-doc

Administrative Positions

President of the Undergraduate Council of IFQSC/USP, 1992-1995

Chief of the Department of Physics and Material Science, IFSC/USP, 1994-1996 and 1998-2000

President of the Graduate Council of IFSC/USP, 1999-2001

Director of the IFSC/USP, 2002-

Editorial and Advisory Boards

Material Science

Publications

130 papers published on indexed international Journals (since 1981)

2 Chapters in books

Research Interests

Electrical and optical properties of Polymers; Organic electronic and optoelectronic devices; Supramolecular materials and nano-organized systems

Meetings organized

12 th International Symposium on Electrets (2005)

I, II, III and IV Meetings of Instituto Multidisciplinar de Materiais Poliméricos (2002-2005)

III Brazilian Material Research Society Meeting (2004)

Referee for:

Journal of Non-Crystalline Solids, Applied Physics Letters, Journal of Applied Physics, Journal of Physics D, Synthetic Metals, Thin Solid Films, Physical Review B, etc.

Organic Light Emitting Diodes: Technological Challenges and Applications

Roberto Faria

Institute of Physics of São Carlos, University of São Paulo, C. P. 369, 13560-970
São Carlos, Brazil

Polymeric materials, which have historically been classified exclusively as electrical insulators, after the discovery of polymeric conductors in the 1970s are now finding varied applications as both conductors and semiconductors. Within a very short time organic conductors have been developed with the conductivity of metals such as copper, while organic electronics has evolved sensors, photovoltaic diodes, light-emitting diodes, and transistors. Organic light-emitting devices (**OLEDs**), in particular, have attracted considerable interest in the last fifteen years and are now viewed as an important competitor with liquid crystals for a wide variety of display applications. Current research focuses on the use of small-molecule and polymer materials to make organic electroluminescent displays with both passive and active-matrix technologies. This talk will discuss the advances and the challenges imposed by the polymer thin film morphology on OLEDs technology. We show that ionomers and other transport layers have shown an impressive increase in the device efficiency and an enhancement in the injection equilibrium of both carriers; and that polarized emissions are generated by stretched films. Finally, we will comment the use of ink-jet printing for high-resolution patterning of red, green, and blue light-emitting polymers.

TuSB -I-5
HYBRID METAL BASED TRANSISTORS**8:00/9:00am**

Ivo Hummelgen, DF/UFPR, PR

Biography

Bacharel em Física (UFPR, 1985), mestre em Física Aplicada (USP - São Carlos, 1987), doutor em Física (Universidade de Göttingen, 1991), Pós-Doutorado (Universidade de Göttingen, 1991). Atua em Física Aplicada a Dispositivos Eletrônicos e Optoeletrônicos Orgânicos. Orientou 4 doutorados e 11 mestrados, co-orientou um doutorado. Participou da criação de duas empresas de base tecnológica e é autor de 73 artigos científicos e 6 patentes.

HYBRID METAL BASE TRANSISTORS

Ivo Alexandre Hümmelgen

Lab. de Dispositivos Optoeletrônicos Orgânicos, Dep. Física, UFPR

Hybrid metal base transistors are semiconductor-metal-semiconductor devices, which can operate as transistors and are suitable for fast switching. We developed hybrid semiconductor-metal-semiconductor devices, which are constructed in vertical architecture using an inorganic semiconductor as collector; a metal, metal multilayer or conducting polymer as base; and an organic semiconductor as emitter. Low molecular weight conjugated molecules and conjugated polymers are used as emitter materials. Our transistors present nearly ideal base-transport factor and devices having electrons, as well as holes, as majority charge carriers were developed. When magnetic multilayers are used as base, the transistors transport characteristics are magnetic field dependent.

TuSB -I-6
SENSORS AND BIOSENSORS MADE OF LAYER-BY-LAYER FILMS**9:45/10:15am**

Osvaldo Novaes Jr.

Biography

Osvaldo N. Oliveira Jr. is an associate professor at the Instituto de Física de São Carlos, Universidade de São Paulo. He got a BSc and MSc. in Physics, 1982 and 1984, respectively, from Universidade de São Paulo in São Carlos, and a PhD at the University of Wales, Bangor, in 1990. Prof. Oliveira has led research into the fabrication of novel materials in the form of ultrathin films obtained with the Langmuir-Blodgett and self-assembly techniques. Most of this work has been associated with fundamental properties of ultrathin films with molecular control, but technological aspects have also been addressed in specific projects. This is the case of an electronic tongue, whose response to a number of tastants is considerably more sensitive than the human gustatory system. Prof. Oliveira Jr. has helped establish the Núcleo Interinstitucional de Linguística Computacional (NILC), which is a leading institute for natural language processing of Portuguese. Research and development activities at NILC include the development of a grammar checker for Brazilian Portuguese, now available worldwide through Microsoft Word, and participation in the Universal Networking Language (UNL) Project. He published approximately 230 papers in refereed journals, which have received over 1,700 citations according to the Institute of Scientific Information.

Sensors and biosensors made of layer-by-layer films

Osvaldo N. Oliveira Jr.

Instituto de Física de São Carlos, USP, Brasil

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The layer-by-layer (LbL) method, based on the physical adsorption of oppositely charged layers, was exploited to produce nanostructured films used as sensors and biosensors. Examples will be provided of sensing that does not require specific interactions between the analyte and the film-forming molecules. In this class are included sensor arrays referred to as "electronic tongues" that make use of global selectivity. With impedance spectroscopy as the principle of detection, the data are analyzed with equivalent electrical circuits and then treated with statistical or artificial intelligence methods. The electronic tongues allow detection of small amounts of substances responsible for the basic tastes, with a much higher sensitivity than the human tongue, in addition to distinction among complex liquids such as different types of wine or coffee. With regard to the biosensors, results demonstrated a high sensitivity due to the molecular

recognition capability of film-forming molecules. For instance, LbL films of immobilized enzymes, whose activity was preserved for weeks after immobilization, were employed to detect catechol and cholesterol, which could be performed either by electrical or optical means. Emphasis will be given to the reasons why the LbL method is suitable for the immobilization of biomolecules, and to the methods currently employed to investigate the molecular-level interactions with the analyte molecules.

TuSB -I-7**10:30/11:00am****MOLECULAR NANODEVICES DEVELOPED AT THE LAND-FOTON/DQF/UFPE**

Petrus Santa Cruz, DQF/PE

Biography and Summary Not Available

TuSB -I-8**11:45/12:15am****SUPRAMOLECULAR MATERIALS AND DEVICES**

Koiti Araki, IQ-USP

Biography

Koiti Araki is an associate professor at the Institute of Chemistry of University of São Paulo since 2000. He got the BSc in Chemistry in 1986 from University of São Paulo in Ribeirão Preto and the MSc and PhD in Inorganic Chemistry by Institute of Chemistry of University of São Paulo, respectively, in 1989 and 1994, under the supervision of Prof. Henrique Eisi Toma. He was a visiting scientist at California Institute of Technology in 1990/91 (USA); Massachusetts Institute of Technology, in 1995/96 (USA) and at Research Center for Molecular Nanoscience at Institute for Molecular Science of Okazaki National Research Institutes in 2003/2004 (Japan). Is associated to the Supramolecular Chemistry and Nanotechnology Group since 1989, leading research on polymetallated porphyrin chemistry and materials, as well as its applications in molecular devices. His major interest is in supramolecular chemistry and nanotechnology, particularly coordinative assembly of molecular and organic-inorganic hybrid materials and its applications in sensors and photoelectrochemical devices. Supervised 2 master and 4 doctoral thesis and authored or co-authored approximately 70 papers, receiving over 800 citations according to the Institute of Scientific Information.

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Supramolecular materials and devices*Koiti Araki**Lab. Química Supramolecular e Nanotecnologia*

Porphyrins, metalloporphyrins and transition metal complexes exhibit remarkable spectroscopic, electrochemical, electrocatalytic, electronic and photochemical properties and have been used as building-blocks of supramolecular systems and materials. In fact, we have successfully built-up and explored the properties of supramolecular systems obtained by binding ruthenium-polypyridines and trinuclear μ_3 -ORu₃(OAc)₆ clusters, to the pyridyl-groups of metallotetrapyrrolylporphyrins. In such systems, the ancillary metal complexes can play a wide role, modifying the local environment around the porphyrin ring, acting in parallel or as cofactor in redox processes, as well as electron donor-acceptor groups and relays, modifying the intrinsic activity of the metalloporphyrin center by induced electronic interactions. Furthermore, they are important sites for intra- and intermolecular interactions, allowing the occurrence of supramolecular effects and the assembly of higher order supramolecular structures and materials as ultrathin films. In this presentation, we briefly review the characteristics of such polymetallated porphyrin films and their possible applications in molecular electronics and devices.

Symposium C - Biocompatible Materials**TuSC-I-1****8:00/9:00am****INJECTABLE BIOMATERIALS FOR BONE REGENERATION**

Pedro Granja INEB- Univ. do Porto, Portugal

Biography

Born on March 11, 1970, in Porto, Portugal.

Academic qualifications: PhD in Engineering Sciences, Faculty of Engineering of the University of Porto (FEUP), 2001; Metallurgical Engineer. FEUP, 1993.

Recent relevant academic and scientific activities

- Present: Auxiliary Researcher at INEB (Instituto de Engenharia Biomédica). Principal Investigator of the Cell Encapsulation Systems research area, of the Biointerfaces Group. Founder and Editor-in-Chief of the Biomaterials Network (Biomat.net). Member of the Special Education Interest Group, from the European Society for Biomaterials, to create an EU Doctorate in Biomaterials.
- 2004-2005: Lecturer at the PhD and MSc Programme in Biomedical Engineering (UP), and Lecturer of Tissue Engineering at the MSc Programme in Biomedical Engineering, U. Aveiro.
- 2004: Visiting Assistant Research Scientist at the Dept. Biologic and Materials Sciences, School of Dentistry, University of Michigan, USA.
- Refereeing for journals: Acta Biomaterialia (2004-2005); Journal of Bioactive and Compatible Polymers (2004); Biomaterials (2003); Applied Surface Science (Special Issue of APHYS2003, 2003).

Area of scientific activity: Tissue engineered injectable biomaterials for bone regeneration

Tissue Engineering can be defined as the persuasion of the body to heal itself, through the delivery to the appropriate sites of molecular signals, cells and supporting structures. At present, no biomaterials match all required characteristics for bone regeneration. Moreover, usual surgical procedures are highly invasive, making it highly painful to patients and contributing to long patient recovery. Hence, injectable systems constitute promising alternatives since they are able to fill-in defects and provide minimally invasive surgical procedures. Polymers of natural origin are under investigation for developing these injectable systems, as viscous gels or microspheres. Usually, their derivatives are biocompatible and adequate for the encapsulation of cells, as well as for the immobilization and subsequent release of bioactive molecules capable of playing an active role in bone regeneration. The ongoing research work is primarily focused on developing encapsulation strategies for cocultures of bone cells with other cells influencing their differentiation.

Supervision of scientific works: Currently supervises 1 post-doctoral researcher, 4 PhD students and 1 MSc student.

Participation in scientific projects: Principal Investigator of 1 ongoing national project and also participates in 1 other national and in 2 international projects. Has participated in 4 previous national (one as PI) and 2 international projects.

Organization of scientific events: Organizing Committee of 15 international events, including 5 editions of the annual Advanced Course in Cell-Material Interactions (Porto), 6 editions of the Young Scientists Forum (YSF), integrated in the European Conferences on Biomaterials (Arcachon, Barcelona, Stuttgart, Évora, Sydney and Sorrento), the II Iberian Congress on Biomaterials (2004), the Biocities (2002), the 12th European Conference on Biomaterials (1995), and the meeting Establishing a Materials Science Education Network (1995).

Membership to societies: European Society for Biomaterials (ESB), Portuguese Materials Society, SPM (Polymers and Renewable Resource Materials Groups), Portuguese Chemistry Society, SPQ (Carbohydrates Chemistry Group).

Contact

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Injectable biomaterials for bone regeneration

P.L. Granja,¹ C.C. Barrias,¹ M.B. Evangelista,^{1,2} C.C. Ribeiro,^{1,3} M.A. Barbosa^{1,2}

¹ INEB - Instituto de Engenharia Biomédica, Laboratório de Biomateriais, Porto, Portugal

² Universidade do Porto, Faculdade de Engenharia, Dept. Eng. Metalúrgica e Materiais, Porto, Portugal

³ ISEP - Instituto Superior de Engenharia do Porto, Dept. Física, Porto, Portugal

The regeneration of bone tissue is still a major concern in orthopedic surgery and, as a consequence, remains an active area of basic and clinical research. Injectable systems have been attracting increasing attention due to their ability to provide minimally invasive surgical procedures, thus resulting in less patient discomfort, faster recovery times and lower overall costs. However, their effectiveness needs to be improved, which can be achieved through their association with bioactive molecules and/or cells which, in this way, can be targeted and locally delivered to accelerate the regeneration process.

In our group, injectable systems have been prepared using several materials, in the form of microspheres, incorporated or not in a polymeric vehicle. Furthermore, materials were either associated with a therapeutic agent or with cells which, in turn, were either encapsulated within materials or colonized their surface. Materials tested thus far were: ceramic microspheres of hydroxyapatite (HAp) or calcium titanium phosphate (CTP); alginate polymeric microspheres; or composite polymer/ceramic microspheres of these materials. Microspheres were prepared by droplet extrusion and ionotropic gelation with Ca^{2+} for every system tested. CTP and HAp microspheres were prepared by sintering polymer/ceramic microspheres, and thus burning-off the polymer and bonding together the ceramic granules.

Alginate polymeric microspheres were used for the entrapment of a recombinant enzyme. Enzyme-loaded microspheres were analyzed in terms of enzyme activity, stability, release profile, and internalization by cultured enzyme-deficient cells. The enzyme retained full activity, and its stability at physiological pH was improved. Composite polymer/ceramic microspheres, prepared by adding HAp or CTP powders to alginate, were used to modulate the enzyme release-kinetics.

The behavior of bone cells cultured on the surface of microspheres of different compositions was investigated. Cells were unable to interact with alginate microspheres, probably due the high hydrophilic nature of the polymer, and were only able to spread and adopt a flattened morphology on polymer/ceramic microspheres with a high percentage of ceramic. Some preliminary studies with ceramic HAp microspheres showed that bone cells were able to adhere and proliferate on their surface. Bone marrow stromal cells cultured on CTP microspheres, were able to attach, adopted a typical osteoblast-like morphology and gradually proliferated. Moreover, cells synthesized a fibrillar extracellular matrix and expressed bone phenotypic markers, which confirmed that differentiation along the osteoblastic lineage occurred.

In another approach, bone cells were encapsulated within alginate conjugated with oligopeptides including the Arg-Gly-Asp (RGD) sequence. Alginate was further irradiated and oxidized to improve its biodegradability. After encapsulation and dynamic culture conditions, microspheres of alginate conjugated with RGD peptides were able to promote bone cells' viability, proliferation and differentiation.

TuSC-I-2

10:30/11:30am

BIORESORBABLE SCAFFOLDS PREPARED BY PARTICULATE LEACHING METHOD FOR TISSUE ENGINEERING

Samuel H. Barbanti, UNICAMP, SP

Biography



Samuel Hilsdorf Barbanti concluiu o doutorado em Engenharia Mecânica pela Universidade Estadual de Campinas com ênfase em polímeros bioreabsorvíveis como suportes na engenharia de tecidos. Tem

publicado artigos em periódicos especializados e trabalhos em anais de eventos, com participação nos principais eventos nacionais em ciência e engenharia de materiais. Atua na área de Engenharia Biomédica e de Biomateriais. Em suas atividades profissionais interagiu com 18 colaboradores em co-autorias de trabalhos científicos. Em seu currículo Lattes os termos mais frequentes na contextualização da produção científica e tecnológica são: Engenharia de Tecidos, Biomateriais, Polímeros Bioreabsorvíveis, Membranas, Biodegradação, Cultura de Células e Degradação *in vitro*.

WeSC -I-5
CONTRIBUTION WITHDRAWN

8:00/9:00am

WeSC-I-4
RAPID PROTOTYPING APPLICATIONS IN THE TREATMENT OF CRANIOMAXILLOFACIAL
DEFORMITIES – UTILIZATION OF BIOMATERIAIS

10:30/11:30am

Jorge L. Silva, CenPRA/Campinas/SP



Biography

Formado em Engenharia Elétrica pela Universidade Federal do Espírito Santo e Mestre em Engenharia Elétrica e Computação pela Universidade Estadual de Campinas. Foi Pesquisador Visitante no Instituto de Automática Industrial, Espanha. Foi professor do Curso de Especialização em Engenharia Mecânica da Universidade Metodista de Piracicaba. Foi professor da curso de especialização em Engenharia Mecânica da Universidade Estadual de Campinas. É candidato ao título de Doutor no Departamento de Engenharia Química da Universidade Estadual de Campinas. É pesquisador do Centro de Pesquisas Renato Archer - CenPRA, onde desenvolveu trabalhos nas áreas de automação industrial e robótica, criando e coordenando a Divisão para Desenvolvimento de Produtos do CenPRA, um dos Institutos do Ministério da Ciência e Tecnologia. Seus trabalhos têm se concentrado nas aplicações de tecnologias para desenvolvimento de produtos, em especial para as pequenas e médias empresas. Tem ministrado palestras em Hospitais, conferências, universidades e órgãos de classe sobre aplicações de tecnologias da informação. Desenvolve pesquisa aplicada e divulgação de tecnologia da informação na área médica, em especial, na área de imagens médicas e aplicações de Prototipagem Rápida na medicina incluindo vários trabalhos em parceria com hospitais brasileiros e ênfase na área de reconstrução de anomalias faciais.

Rapid Prototyping Applications in the Treatment of Craniomaxillofacial
Deformities – Utilization of Biomateriais

Jorge L. Silva, CenPRA/Campinas/SP

"O papel da prototipagem rápida e biomateriais na reconstrução tecidos ósseos" Os processos de prototipagem rápida, originalmente desenvolvidos para a indústria como uma ferramenta para a produção de protótipos físicos diretamente de imagens computacionais tridimensionais, já ultrapassam as suas fronteiras, permitindo que biomateriais possam ser utilizados direta ou indiretamente na produção de próteses personalizadas para reconstrução de lesões ósseas ou mesmo na engenharia de tecidos através da produção de "scaffolds" que possam ser colonizados com células e posteriormente implantados. Serão abordados nesta palestra os mecanismos de obtenção destas próteses e "scaffolds" que se constituem no estado-da-arte na moldagem de biomateriais.

Symposium D - Structural materials: Processing Properties and Applications**MoSD-I-1****8:00/9:00am****MATERIALS FOR THERMAL PROTECTION OF SPACE VEHICLES**

Carlos Alberto Alves Cairo, IAE-CTA/SP

MATERIALS FOR THERMAL PROTECTION OF SPACE VEHICLES

Thermal protection systems (TPS) are essential for the successful launch and operation of all spacecraft. The obvious goal of a thermal protection system is to keep the excessive heat from destroying or damaging a vehicle or its contents. While the temperature capability is often a major concern, the goal remains to protect the internal components at a minimal weight. TPS is a system composed of appropriate materials which the selection is based upon heat dissipation needs and environmental constraints. In radiation cooling method for heat dissipation, much of the heat flux is reflected back to the black body of space by a high emissivity coating on the protected substrate. Hot structures used in these systems are selected based on expected temperature regime and mechanical loads, and generally require insulation to protect underlying components. Carbon-carbon composites have one of the highest temperature capabilities of all material systems and become stronger and stiffer at elevated temperatures, but its main disadvantage is the poor oxidation resistance. Ceramic matrix composites (CMC) used for thermal protection systems include carbon reinforced silicon carbide (C/SiC) and silicon carbide reinforced silicon carbide (SiC/SiC). These materials are used where the superior strength and stiffness at elevated temperature, oxidation resistance, high emissivity and high temperature capability are critical. This exhibition intends to present actual researches on C/SiC and SiC/SiC composites materials at Aerospace Technical Center/CTA – Brazil.

TuSD I-2**8:00/8:30am****RECICLAGEM DE REJEITOS DA INDÚSTRIA CERÂMICA TRADICIONAL COMO AGREGADOS PARA ARGAMASSA DE ALVENARIA E CONCRETO DO TIPO PORTLAND**

João Batista Baldo, UFSCar, SP

Biography and Summary Not Available

TuSD I-3**8:30/9:00am****NON-EQUILIBRIUM SOLIDIFICATION OF UNDERCOOLED METALLIC MELTS**

Walman Benicio, DEM – UFCG, PB

Biography

Personal Information

Job: Professor Adjunto IV

Job Address: Department of Mechanical Engineering
Center of Sciences and Technology
Aprígio Veloso, 882 – Bodocongó – P.A. Box 10069
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Federal University of Campina Grande

ACADEMIC BACKGROUND

Bachelor of Science (B. Sc.) – Mechanical Engineering - Federal University of Paraíba - UFPB - Campina Grande – PB, Brazil, 1988.

Master of Science (M. Sc.) - Mechanical Engineering - Federal University of Paraíba - UFPB – Campina Grande – PB – 1992.

Doctor of Science (D. Sc) – Materials Engineering - Federal University of São Carlos – UFSCar – São Carlos-SP, Brazil, 1997.

RESEARCH INTERESTS

Foundry and Rapid Solidification;
Physical Metallurgic;

Materials and Fabrication Process;
Phase Transformation.

PROJECTS

Science and Technology of Rapidly Solidified Metals- PROCAD 0101/01-9.
Study of the influence undercooling in shape memory alloy.
Rapidly solidified alloys using in ceramics brazing.

Non-equilibrium solidification of undercooled metallic metls

Walman Benicio de Castro
UFCG – CCT – DEM – Caixa Postal 10069 – Campina Grande – Paraíba – Brazil.
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Rapid solidification processing provides a typical case of non-equilibrium solidification, therefore microstructure refinement, solid solubility extension, and metastable phases formation will take place under this condition. The non-equilibrium state of an undercooled melt allows the investigation of various phenomena involved in the formation of metastable solid phase. The present work reviews the present state of the art in this field. In detail, the nucleation and the thermodynamics of the undercooled melt are discussed. The rapid solidification techniques for investigation of non-equilibrium solidification phenomena in undercooled melts are discussed, and their potential for such investigations is evaluated. The effect of metastable phases from undercooled melts on mechanical properties and magnetic contributions to alloys are too evaluated.

WeSD-I-4

8:00/9:00am

PROCESSING VS. STRUCTURE VS. MECHANICAL AND CORROSION BEHAVIOUR OF ZRNxOy THIN FILMS

Luís Augusto Rocha, Universidade do Minho, Portugal

Processing vs. structure vs. mechanical and corrosion behaviour of ZrN_xO_y thin films

L. A. Rocha^{1,2}, S.C. Ferreira¹, E. Ariza¹, P. Carvalho³, A.C. Fernandes³, F. Vaz³
¹Research Centre on Interfaces and Surface Performance, Azurém, 4800-058 Guimarães, Portugal
²Universidade do Minho, Dept. Eng. Mecânica, Azurém, 4800-058 Guimarães, Portugal
³Universidade do Minho, Dept. Física, Azurém, 4800-058 Guimarães, Portugal

Coloured thin films possess a high potential for being applied in high-quality consumer products, such as eyeglass frames, wristwatch casings and wristbands. In fact, while enhancing the appearance of the pieces by lending attractive colorations to their surfaces, these films are supposed to provide simultaneously scratch-resistance and improved corrosion resistance.

Up to now, decorative films are mostly based on elemental materials and also binary nitrides (TiN, golden yellow; ZrN, greenish yellow; HfN, pale greenish yellow) or titanium carbonitrides. The colour tones attainable are, however, largely restricted to golden yellows, various shades of grey and black, although some attempts have been made in order to obtain other colours, based on borides.

Recently a new class of materials is gaining importance for these decorative applications, the so-called metal oxynitrides, MeN_xO_y (Me = early transition metal). In fact, the presence of oxygen allows the tailoring of film properties between those of metallic nitrides, MN_y, and those of the correspondent insulating oxides, MO_x. Tuning the metallic/covalent and ionic bonding characteristics allows one to tune the mechanical, electrical, chemical and optical properties of materials, including colour.

In this work an overview on the interrelation between the processing parameters, obtained structure, and the mechanical and/or corrosion resistance of single layered zirconium oxynitride, ZrO_xN_y, is presented. Particular focus is given to the functional aspects of the obtained films, in particular, to their corrosion and tribocorrosion behaviour. The influence of the coating structure and microstructure and/or chemical composition originated by the variation of the deposition parameters is correlated with the corrosion or tribocorrosion mechanisms occurring in each system.

Symposium E - Advances in Photonics Materials and applications
**MoSE-I-1
CONTRIBUTION WITHDRAWN**
8:00/9:00am
**MoSE-I-2
HOLOGRAPHIC TECHNIQUES TO STUDY PHOTOSENSITIVE MATERIALS**
10:30/11:30am

Lucila Cescato, UNICAMP, SP

Biography

Lucila Cescato is head of the Optics Laboratory of the IFGW - Unicamp. She received the Master and PhD degree. in 1980 and 1987 respectively, both from Unicamp and was Humboldt fellow from 1988-1990 at the University of Erlangen-Nürnberg in Germany. She is author of more than 50 scientific papers in international journals. Her research subjects are Diffractive Optics, Holography, Photosensitive Materials, Photonic Crystals and Processes of Micro and Nanofabrication.

Holographic Techniques to study Photosensitive Materials

Lucila Cescato, UNICAMP, SP

Holographic techniques have been successfully employed to study photosensitive materials. They ally, the high sensitivity of the diffraction, with the high spatial frequencies possibilities of interference patterns and, in case of volume photosensitive materials, they directly fulfill the Bragg condition. If the measurements are performed in real time, dynamic gratings or optical modulations, not bound with the permanent changes in the material, could be measure. Even for materials that need a development, small changes in the optical constants are produced during the exposure of a proper wavelength. In this presentation we will give an overview about the use diffraction and holographic techniques in the study of photosensitive materials and we describe phase sensitive holographic technique that allows the measurement of real time optical modulations as small as 10⁻⁵, induced by light in photosensitive materials. We apply this technique to study solid-state kinetic reactions in photopolymers.

**MoSE-I-3
RARE EARTH DOPED TRANSPARENT FERROELECTRIC CERAMICS FOR PHOTONIC APPLICATIONS.**
12:00am/12:30pm

Andrea Simone Stucchi de Camargo, IF-USP, São Carlos

Rare Earth Doped Transparent Ferroelectric Ceramics For Photonic Applications

 Andrea S. Stucchi de Camargo^{1,2}, Luiz Antonio de O. Nunes¹, Ériton R. Botero², Érika R. M. Andreetta², Ducinei Garcia², José Antonio Eiras².

¹Instituto de Física de São Carlos – Universidade de São Paulo, CEP 13566-590, São Carlos - SP

²Departamento de Física - Universidade Federal de São Carlos, CEP 13565-905, São Carlos – SP

Abstract – Rare-earth doped transparent ceramics are new and promising materials for photonic device applications. In this work we present structural, microstructural and spectroscopic characterization of PLZT ferroelectric ceramics.

Rare-earth ion-doped transparent ceramics are emerging optical materials that hold promising applications for photonic devices as laser active media, amplifiers, waveguides, optical sensors, etc. As compared to optical glasses and crystals, which are normally used for these purposes, ceramics present important advantages as lower cost and simpler fabrication, the ability to incorporate higher ionic concentrations, and they can be obtained in a much larger variety of compositions, shapes and sizes. Due to their usually superior thermal and mechanical properties they are especially suitable for the construction of near-infrared (1.0 - 3.0 μm), high power laser active media.^{1,2} Among the examples that have most recently been studied for this purpose, are neodymium doped YAG transparent ceramics.²

The ability to obtain transparent ferroelectric ceramics brought us motivation to study rare-earth ion (RE = Nd³⁺, Er³⁺, Yb³⁺, Tm³⁺) doped ferroelectric samples in order to characterize their potential application as laser active media and other devices.³⁻⁵ Lead lanthanum zirconate titanate (PLZT), with La/Zr/Ti proportion 9/65/35 host presents many of the desired characteristics of an efficient laser active media such as an extensive transmission window, as it can be seen in Figure 1, fairly low phonon energy

(750 cm^{-1}) that implies small nonradiative decay rates, negligible OH^- absorption which could constitute a significant energy loss, and a high refractive index (2.5) that leads to increased emission cross sections of the RE ions. Furthermore, the piezoelectric/ ferroelectric nature of PLZT gives the possibility of increased functionality by mechanical-optical or electro-optical effects, which are particularly interesting for both, bulk and thin films forms, and their applications in telecommunications.

In this work we present the interesting structural, microstructural, optical and spectroscopic results we have obtained in recent years, while studying several Nd^{3+} , Er^{3+} , Yb^{3+} or Tm^{3+} doped PLZT bulk samples.

The samples were obtained through conventional solid state reaction of high purity PbO , ZrO_2 , TiO_2 , La_2O_3 , and the dopants Nd_2O_3 , Er_2O_3 , Yb_2O_3 or Tm_2O_3 oxides, followed by isostatical hot pressing at $1250\text{ }^\circ\text{C}$ for 3 h, under 5 MPa, in O_2 atmosphere. The structures of powders and densified samples were verified by X-ray diffraction and microstructural characterization was done at fractured surfaces with a scanning electron microscope. UV-VIS and IR absorption spectra were taken in commercial spectrophotometers, and luminescence signals were measured using as excitation sources 800 or 980 nm diode lasers. Thermal properties were evaluated using the Thermal Lens technique and stimulated emission and excited state absorption measurements were also done using the pump probe experimental setup. Radiative properties were analyzed using the Judd Ofelt theory and energy transfer models.

Results indicate that PLZT ferroelectric ceramic is a very promising laser host with appropriate thermal and mechanical properties. $\text{PLZT}:\text{Er}^{3+}$, $\text{PLZT}:\text{Tm}^{3+}$ and $\text{PLZT}:\text{Yb}^{3+}$ samples are shown to be potential laser active media with intense emissions at 1.55, 2.8, 1.8 and $1.0\text{ }\mu\text{m}$, as long as segregation of oxide phases at their grain boundaries can be avoided to enhance their optical quality. Neodymium doped samples present the most promising characteristics (see Fig. 2) with fairly high stimulated emission, negligible excited state absorption at $1.06\text{ }\mu\text{m}$, and impending laser action. Furthermore, this emission was found to be modulated by an electric field.

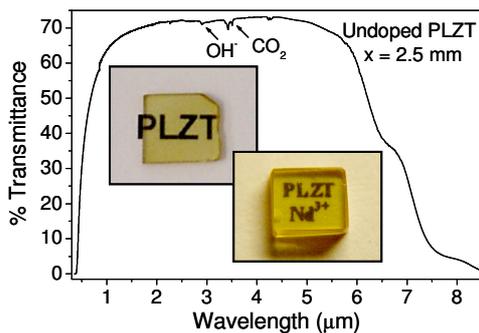


Figure 1: Transmission spectrum and photographs of an undoped and a 2.0 mol% Nd^{3+} doped PLZT sample

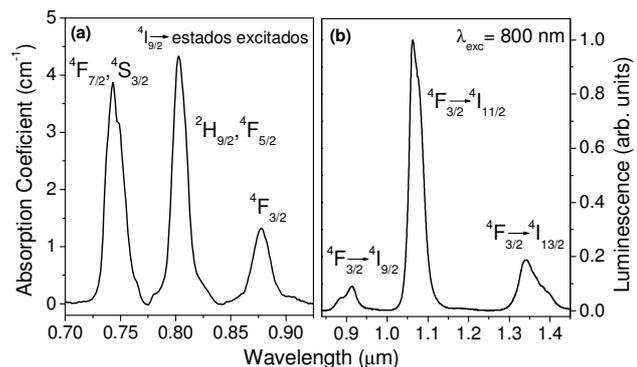


Figure 2: Absorption (a) and luminescence (b) spectra of a 2.0 mol% Nd^{3+} doped PLZT transparent ceramic sample.

References

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TuSE-I-4**8:00am/9:30pm****RECENT ADVANCES AND CHALLENGES IN THE PHYSICS OF SOLID STATE LIGHTING.**

Fernando A. Ponce, IF-USP, São Carlos

Biography**FERNANDO A. PONCE**

Department of Physics and Astronomy
Arizona State University, Tempe, Arizona 85287-1504
Tel. (480) 965-5557, Fax (480) 965-7954, Email: ponce@asu.edu

WORK EXPERIENCE

Professor, Department of Physics, Arizona State University, Tempe, AZ (Since 1999).
Xerox Palo Alto Research Center, Palo Alto, CA. Member of Research Staff. (1984 - 1998).
Hewlett-Packard Labs, Palo Alto, CA. Member Technical Staff. (1980 -1984).
Xerox Palo Alto Research Center, Palo Alto, CA. Research Associate. (1978 -1980).

EDUCATION

Ph. D., Materials Science and Engineering, Stanford University, 1976-1981
M. S., Solid State Physics, University of Maryland, 1973-1975
B. S., Physics, Universidad Nacional de Ingenieria, Lima, Peru, 1967-1971

HONORS

Honorary Doctor Degree, Universidad Ricardo Palma, Lima, Peru, 2005
Vice-Chair, Intel International Science and Engineering Fair, Phoenix, Arizona, 2005
Chair, International Conference on the Physics of Semiconductors (ICPS-27), July 2004
Fellow American Physical Society, elected 2002.
Honorary Professor, Universidad Nacional de Ingenieria, Lima, Peru, 2002
Chair, Materials Research Society, 1999 Fall Meeting
Medal "Eduardo de Habich", Lima, Peru, 1999
Honorary Professor, National University, Cuzco, Peru, 1990
Ross M. Tucker AIME Electronics Materials Award, 1981
Organization of American States Fellow, 1974-1975
Graduated Summa Cum Laude, U. N. I., Lima, Peru, 1971

PROFESSIONAL AFFILIATIONS

American Association for Crystal Growth, American Physical Society, American Vacuum Society, Electron Microscopy Society of America, Materials Research Society, ECS.

AREAS OF TECHNICAL EXPERTISE AND INTEREST

1. Structure of interfaces and defects in crystalline materials.
2. High-resolution transmission electron microscopy.
3. Microscopic determination of optical and electronic properties of materials.
4. Materials for photonics and optoelectronics.
5. Thin film growth by molecular beam epitaxy and chemical vapor deposition.
6. Materials analysis, including x-ray diffraction, microanalysis, and TEM.
7. III-V compounds and related epitaxial systems
8. Double heterojunction laser diodes, optical coatings for optoelectronic applications.

PUBLICATIONS

1. Editor of 8 books, co-inventor in 5 patents.
2. More than 220 publications, > 160 in refereed journals, > 4200 citations (Web of Science).
3. More than 300 technical talks, over 150 invited.

Recent advances and challenges in the physics of solid state lighting

F. A. Ponce

Department of Physics and Astronomy, Arizona State University, Tempe, AZ 85287-1501, USA

Abstract – The most important current challenge in the physics of solid state lighting is in understanding why the internal quantum efficiency of InGaN-based light emitting diodes is much lower than in other systems. The answer lies in the nature of the quantum wells, where piezoelectric fields play a critical role.

GaN-based semiconductors are widely used for visible light emitters used as lasers for optical recording, and as light emitting diodes (LEDs) for lighting applications. Blue-violet LEDs with very high efficiency were demonstrated more than 10 years ago, which were shortly followed by green LEDs. Their efficiency and lifetime significantly exceed the monochromatic performance of other light sources. Much excitement has existed about achieving very high efficiencies across the full visible spectrum that would open the way for high energy efficient solid state lighting [1-4].

The internal quantum efficiency (IQE) is the capability to combine electrons and holes to produce light. It is a property of the active region of the device (i.e the quantum well region in LEDs). In the case of the nitride semiconductors it peaks at around 410nm and drops significantly towards red, and very rapidly toward the UV range. In spite of much effort over the last decade, the advances in improvement of IQE have not been significant. Several factors have been considered in order to explain this behavior. First is the large density of threading dislocations, a characteristic of these epilayers that appears to affect the UV range but not the visible range. Second is the compositional stability of the InGaN alloy; the phase diagram shows a significant gap in compositional stability. Third is the large lattice mismatch between InGaN alloys and the GaN underlayer; this lattice mismatch can be as large as 11%. Fourth is the strong piezoelectric field that develops in InGaN layers in the presence of large compressive strains that develop due to mismatch in thermal expansion and lattice parameter.

We have performed detailed studies of the microscopic properties of InGaN alloys using various techniques. With high resolution transmission electron microscopy we observe with atomic resolution the morphology and thickness variations of the quantum well (Fig 1). Electron tomography is used to distinguish compositional and strain related fluctuations. Electron holography is used for determination of distribution of electrostatic fields and charges [5] (Fig. 1). Cathodoluminescence imaging is used for spatially resolving the spectral emissions, and time-resolved CL is used to understand the nature of the optical transitions [6]. In this talk, the variation of the properties of InGaN layers grown on various substrates will be presented. Emphasis will be given on the nature of the light emission and on the effect that the internal electrostatic fields have on the internal quantum efficiency. It will be shown that these materials have a quite different behavior than other semiconductor systems, mostly due to the strong piezoelectric fields. The latter reduce the capture cross section of the quantum wells which is accentuated with In composition.

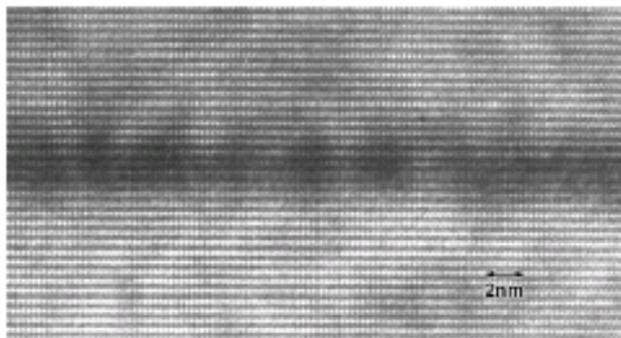


Fig. 1. High resolution TEM image showing the structure of an InGaN quantum well. The image is taken in a $\langle -1-100 \rangle$ projection. Notice the inhomogeneous contrast of the QW. [3]

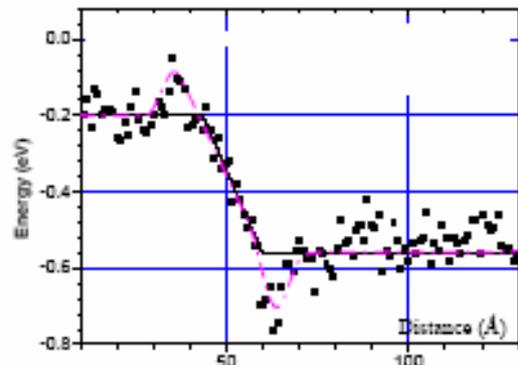


Fig. 2. Electrostatic potential distribution across an In_{0.18}Ga_{0.82}N quantum well (x=0.18). The quantum well is 2.5nm thick. The field is 2.2 MeV/cm. [3]

References

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- [5] M. Stevens, F. A. Ponce, et al., *Appl. Phys. Lett.*, 85, 4651 (2004)
- [6] A. Bell, F. A. Ponce, et al., *Appl. Phys. Lett.*, 84, 58 (2004)

MoSE-I-5
12:00am/12:30pm
LASER-INDUCED REFRACTIVE INDEX CHANGES IN PHOTONIC MATERIALS

Tomaz Catunda, IF-USP, São Carlos

Laser-Induced Refractive Index Changes In Photonic Materials

T. Catunda

Instituto de Física de São Carlos, S.Carlos, SP, Brazil

Abstract – In this work we'll discuss laser-induced refractive index and optical path changes in photonic materials, focusing rare earth doped crystals and glasses. These studies are important to the characterization of thermo-optical, nonlinear and spectroscopic properties of materials.

Pump-induced thermal focusing is of primary importance optical materials because of its consequences in laser efficiency, beam quality, resonator stability, etc. The dual beam mode-mismatched Thermal Lens (TL) spectrometry have been lately applied to the study of photonic materials, been applied to the determination of thermo-optical parameters, thermal diffusivity, the temperature coefficient of optical path (ds/dT), heat efficiency (the fraction of absorbed energy converted into heat) and the fluorescence quantum efficiency (η) [2]. In this work we'll discuss several approaches to determine η and study Energy Transfer mechanisms in laser materials, mostly glasses and crystals, doped with Nd^{3+} , Yb^{3+} , Tm^{3+} and Er^{3+} [2].

The TL transient signal amplitude is proportional to its phase shift, Θ . Since, in any photothermal method, the signal is proportional to the absorbed power (P_{abs}) it is usually convenient to use a normalized parameter, Θ/P_{abs} . This parameter is proportional to the heat efficiency, which decreases linearly with excitation wavelength. Figure 1 shows the linear decrease of Θ with the excitation wavelength (λ_{exc}) for 1 at. % doped Nd^{3+} :YAG crystal. From the linear fit of Fig.1.a data we obtained $\eta = 0.95 \pm 0.02$ and $ds/dT = (13.7 \pm 0.9) \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ [1].

Although less extensively studied, refractive index changes due the difference in polarizability of active ion in metastable and ground state can be comparable with the thermal ones [3,4]. We investigated the physical origin of this change analyzing resonant and nonresonant contribution to this electronic contribution to refractive index change (Δn) in laser materials doped with Cr^{3+} , Nd^{3+} and Yb^{3+} . We have measured the nonlinear refractive index, $n_2(\lambda)$, lineshapes using variation of the Z-scan technique where the excitation laser is continuously tuned [3]. The n_2 spectrum of Nd^{3+} :YAG crystal can be explained by the interference effect between the resonant and nonresonant contributions. These lineshapes change drastically at low temperatures, where n_2' acquires a dispersive feature characteristic of resonant interactions (Fig.2). This behavior can be attributed to the narrowing of line, which is phonon broadened. So this increases Δn_g (which is proportional to the resonant interaction) while Δn_g (the nonresonant interaction) is expected to be nearly temperature independent.

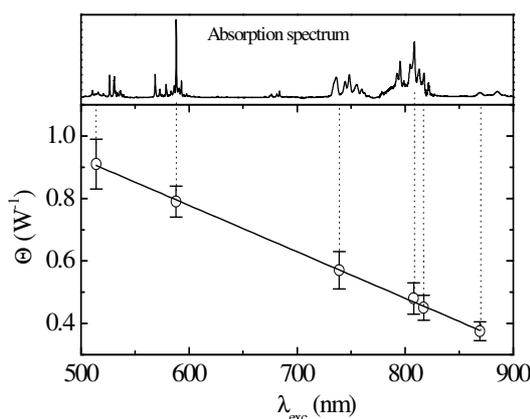


Fig. 1 Plot of the normalized TL amplitude, Θ , versus the excitation wavelength (λ_{exc}) for Nd^{3+} :YAG crystal. The inset shows the optical absorption spectrum of the crystal [1].

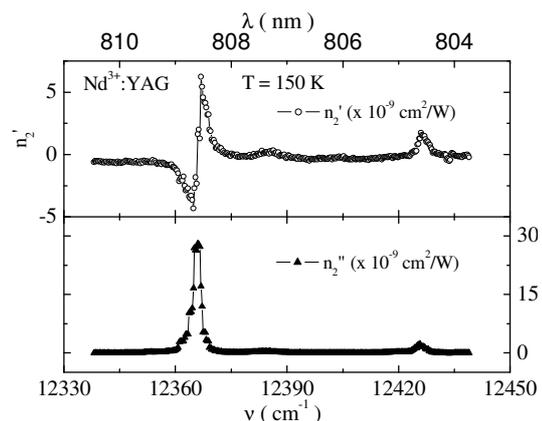


Fig.2- Nd^{3+} :YAG complex nonlinear refractive index ($n_2 = n_2' - n_2''$) spectra around $^4F_{5/2}$ absorption line

References

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Acknowledgements: This work was supported by the Brazilian Agencies FAPESP, CNPq and CAPES

Symposium F- Magnetic Materials: Preparation, Characterization and Applications

MoSF-I-1

8:00/9:00am

PERMANENT MAGNETS - RECENT DEVELOPMENTS

Frank P. Missell Universidade de Caxias do Sul, RS

Biography

Programa de Pós-Graduação em Materiais
Departamento de Física e Química
Universidade de Caxias do Sul
Cidade Universitária/ Bloco V
Rua Francisco Getúlio Vargas, 1130
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Rua Nossa Senhora das Graças, 50 – Xerém
25245-020 Duque de Caxias, RJ, Brazil
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Professional Experience

INMETRO – DUQUE DE CAXIAS, RJ, BRAZIL 2005
Scientific Consultant – Magnetic materials

UNIVERSITY OF CAXIAS DO SUL – CAXIAS DO SUL, RS, BRAZIL 2003-2005
Invited Professor – Friction materials, non-destructive evaluation (NDE), magnetic properties of steels, alloys and permanent magnets

WESTERN DIGITAL TECHNOLOGIES – FREMONT, CA 2003
WD produces hard drives for computers.
Senior Principal Engineer – Responsible for experimental determinations of pole tip protrusion

READ-RITE CORPORATION (RDRT) – FREMONT, CA 2001-2003
One of the world's largest independent suppliers of magnetic recording heads for the hard disk drive and tape drive market. RDRT designed, manufactured and marketed recording heads as head gimbal assemblies.

Staff Engineer (2001-2003) - Conducted development work in the area of magnetic data storage: Developed experimental techniques for quantifying head-disk interactions related to pole tip protrusion (PTP). Spin stand tests were developed to measure PTP due to ambient temperature increase and write current heating. Filed patent for material to reduce pole tip protrusion. Developed experimental techniques to measure thermo-mechanical material properties (coefficient of thermal expansion and elastic modulus) related to PTP for use in numerical simulations.

UNIVERSITY OF SÃO PAULO (USP) – SÃO PAULO, BRAZIL 1971-2001
As the largest and oldest university in Brazil, USP is a major center for physics research.

Full Professor (1986-2001) - Developed World Class independent research on magnetic and superconducting materials; conducted collaborative research; developed research infrastructure, including thin film laboratory and surface analysis facilities. Authored more than 100 papers stemming from these projects, which have been cited in over 600 journal publications worldwide.

LABORATOIRE LOUIS NÉEL – GRENOBLE, FRANCE 1992-1993
European center for the study of magnetism and magnetic materials.
Developed research program on coercivity mechanisms in rare-earth permanent magnets and magnetism of rare-earth transition metal thin films.

ENERGY CONVERSION DEVICES, INC. – TROY, MI 1981-1982
Innovative company carrying on contract research in the fields of amorphous silicon solar cells, hydrogen storage, as well as superconducting and magnetic materials.
Conducted research on amorphous thin film superconducting alloys and their applications.

MIT FRANCIS BITTER NATIONAL MAGNET LABORATORY – CAMBRIDGE, MA 1976-1977
Center for high magnetic field research.
Conducted research on the hydrostatic pressure dependence of magnetic phenomena.

EDUCATION

PH.D. PHYSICS
MASSACHUSETTS INSTITUTE OF TECHNOLOGY – CAMBRIDGE, MA
Thesis: Laser magnetoreflexion studies of the optical de Haas-Shubnikov effect.
Thesis Advisors – MILDRED S. DRESSELHAUS, ALI JAVAN

A.B. PHYSICS
PRINCETON UNIVERSITY – PRINCETON, NJ
Thesis: The design and construction of a continuous-wave helium neon laser.

PERMANENT MAGNETS – RECENT DEVELOPMENTS

Frank P. Missell
Universidade de Caxias do Sul

Permanent magnets based upon $\text{Nd}_2\text{Fe}_{14}\text{B}$ have now been around for more than 21 years and have led to revolutionary advances in the development of magnetic devices. Other magnetic materials employed in permanent magnets have also made important progress. This presentation will outline recent developments in hard magnetic materials. La-Co substituted M-type ferrite has raised the coercive force H_c of conventional Sr ferrite and holds promise of automotive applications. The development of $\text{Sm}(\text{CoFeCuZr})_z$ magnets for applications above 400°C , led to the discovery of an anomalous temperature dependence of H_c whose origin is related to the complex nanostructure of these materials as well as the metallurgy of the quinary alloys. Nanostructured NdFeB alloys have found widespread application in bonded magnets. MAGnetic Micro-Actuators and Systems (MAGMAS) present outstanding performance for powerful integrated electrical-to-mechanical energy conversion. Recent developments in these areas will be reviewed.

MoSF-I-2**10:30/11:30am****NANOSCALE PROPERTIES OF MAGNETIC COLLOIDS**

Jérôme Depeyrot UnB, DF
Biography and Summary Not Available

TuSF-I-3**8:00/9:00am****THE MAGNETOCALORIC EFFECT: MATERIALS AND APPLICATIONS**

Sergio Gama, UNICAMP, SP
Biography and Summary Not Available

Symposium G - Superconductor Materials**TuSG-I-1****10:30/11:00am****RESEARCH AND DEVELOPMENT OF HIGH-TC SUPERCONDUCTORS IN CEPEL-ELETRÓBRÁS**

Alexandre Polasek, CEPEL-Eletróbrás,RJ

Biography

Dr. Polasek received his DSc. in Materials Science and Engineering from Pontifical Catholic University of Rio de Janeiro (PUC-Rio), in 2002, having worked under the supervision of Prof. Rizzo. He developed part of his thesis work on melting relations in the Bi-Sr-Ca-Cu-O system at the Max-Planck-Institut für Metallforschung, in Stuttgart, Germany, with Dr. Peter Majewski, renowned for his investigations on phase diagrams, crystal chemistry and processing of high-T_c superconductors. Dr. Polasek has published more than 30 works in international journals and conferences, and has participated in the organization of events like the Seventh International Conference on Materials and Mechanisms for Superconductivity and High Temperature Superconductors (M2S-RIO), held in Rio de Janeiro, May 2003. Since 2002 he has been working in CEPEL (Electric Power Research Center / ELETROBRÁS), where he is in charge of the Superconductivity Laboratory. His researches focus on the development of superconducting materials for electric power applications.

Researcher, Materials Science and Engineering

CEPEL – Electric Power Research Center, C.P. 68007, CEP: 21944-970, Rio de Janeiro-RJ, Brazil e-mail: polasek@cepel.br

Research and Development of High-T_c Superconductors in CEPEL - ELETROBRÁS

Alexander Polasek and Eduardo Torres Serra

CEPEL – Centro de Pesquisas de Energia Elétrica, C.P. 68007, CEP: 21944-970 Rio de Janeiro-RJ-Brazil, e-mail: polasek@cepel.br

Abstract – An overview of the main R&D activities undertaken in the Superconductivity Laboratory of CEPEL is presented. Our work has been focusing on the investigation and development of High Temperature Superconductor (HTSC) materials for electric power applications.

According to the International Superconductivity Industrial Summit 2002, “The electric power applications are where superconductivity has the potential to have the greatest impact”. The market for such products can be worth billions of dollars by 2010-2020 [1]. Besides, superconducting equipments like fault current limiters, SMES, and superconducting cables can improve the stability, reliability and power quality, preventing congestion and costly blackouts. Such applications became much more feasible with the advent of ceramic materials superconducting above the liquid nitrogen boiling point (77 K), since liquid nitrogen is a cheap and available coolant. Before this breakthrough, mostly of the potential applications of superconductivity in the electric power sector were hampered, due to the need of liquid helium (4.2 K) for cooling metallic Low Temperature Superconductors. Nowadays, large-scale prototypes of superconducting equipments are being successfully demonstrated. However, the main technical and economical challenges still rely on the improvement of processing and properties of HTSC materials.

In 1996, with the aim of developing HTSC materials and transferring such knowledge to the Brazilian electric power sector, CEPEL established its Superconductivity Laboratory (LAS), with funds of the Ministry of Mines and Energy. Initially, our work focused mainly in studying and processing superconducting Bi₂Sr₂Ca₂Cu₃O_{10+x} (Bi-2223) tapes. The process consists of packing a pre-reacted precursor powder into a silver tube, which is further drawn, rolled and sintered. The investigation of the whole process - from the precursor powder preparation [2] to the most critical thermal and mechanical parameters [3] - allowed the development of the Continuous Cooling Sintering method by our group (patent pending) [3]. This method is suitable for large-scale production, since the total processing time is reduced, whereas the thermal processing window is considerably enlarged without decreasing the performance of the superconducting tapes. Critical current densities above 30 kA/cm², at 77 K, were achieved.

Presently, the optimization of the process of bulk Bi₂Sr₂CaCu₂O_{8+x} (Bi-2212) has been focused in LAS / CEPEL. This material can be obtained via the so-called melt-processing, by which a precursor is melted and further slow-cooled. The Bi-2212 phase recrystallizes during slow cooling, forming a dense and textured bulk material, allowing the transport of relatively high critical currents. This material has been shown to be suitable for several applications, e.g., in superconducting fault current limiters (SCFCL). The SCFCL is a low impedance device that can limit huge fault currents by means of the fast transition from the superconductor to the normal state. After the current peak, the system can return to normal operation, without human intervention. Owing to the potential of SCFCL for reducing damage and losses occasioned by fault currents, the processing of Bi-2212 blocks by partial melt processing has been investigated in CEPEL. Partially melted Bi-2212 blocks transporting over 1 kA/cm², at 77 K, were obtained [4].

By contrast with the Bi-2212 phase, there isn't a well established melt-processing route for synthesizing the Bi-2223 phase, due to its narrow stability range and sluggish formation kinetics. It is thus a hampering factor for achieving

high quality bulk Bi-2223. However, the Bi-2223 phase presents a higher critical temperature ($T_c = 110$ K) than Bi-2212 ($T_c < 96$ K) and than the $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ (Y-123, $T_c = 93$ K) superconductor. Therefore, a melt-processing route could be very interesting for producing bulk Bi-2223. Thus, we have been studying the viability of melt-processing Bi-2223 [5] and promising results have been attained. The elucidation of the Bi-2223 / melt equilibrium is a key issue for achieving Bi-2223 directly from the melt.

Since the very beginning, the works on HTSC superconductors carried out in CEPEL have involved an invaluable partnership with the Department of Materials Science and Metallurgy and with the Department of Physics from PUC-Rio. On the other hand, cooperation with other institutions, as well as with the industry, is essential for promoting innovation and knowledge transferring to the Brazilian electrical sector.

References

- [1] R. M. Stephan, R. Andrade Jr. and A. Polasek, to be published in *EletoEvolução*, CIGRÉ-Brazil, September (2005).
- [2] A. Polasek, S.K. Xia, M.B. Lisboa, M.A. Sens, E.T. Serra, F. Rizzo and H. Borges, *IEEE Trans. on Appl. Supercond.*, v.9, n.2, p. 2573, (1999).
- [3] S.K. Xia and E.T. Serra, in *Studies of High Temperature Superconductors*, ed. A. Narlikar, Nova Science, v. 43, p. 63, (2002).
- [4] B.A. Marinkovic, S.K. Xia, E.T. Serra and F. Rizzo, *Mat. Chem. and Phys.*, v. 91, p. 301, (2005).
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WeSG I-2

8:00/8:30am

ON THE PROPERTIES AND CHARACTERISTICS OF SUPERCONDUCTORS WITH ARTIFICIALLY INTRODUCED NANOMETRIC-SCALE PINNING CENTERS

Durval Rodrigues

Biography

DURVAL RODRIGUES JUNIOR concluiu o doutorado em Física pela Universidade Estadual de Campinas (UNICAMP), Campinas, SP, Brasil, em 1997, após realizar parte do trabalho no Massachusetts Institute of Technology (MIT), em Cambridge, MA, EUA. Atualmente é professor titular da Faculdade de Engenharia de Lorena (FAENQUIL) trabalhando no Departamento de Engenharia de Materiais (DEMAR), onde leciona Física básica e aplicada, Eletromagnetismo, Estrutura dos Materiais, Supercondutividade, e Microscopia Eletrônica e Microanálise. Ele trabalhou como Chefe do DEMAR-FAENQUIL de 1997 a 2001 e participa de Conselhos Institucionais desde 1995. Ele é o coordenador do Laboratório de Microscopia Eletrônica do DEMAR-FAENQUIL. Publicou 35 artigos em periódicos especializados e 140 trabalhos em anais de eventos. Possui 4 capítulos de livros publicados. Possui 1 software, 5 processos ou técnicas e outros 20 itens de produção técnica. Participou de 9 eventos no exterior e 41 no Brasil. Orientou 1 tese de doutorado, 5 dissertações de mestrado, além de ter orientado 9 trabalhos de iniciação científica. Ele está orientando 3 teses de doutorado, 2 dissertações de mestrado e 2 alunos de Iniciação Científica nas áreas de Engenharia de Materiais e Metalúrgica, Física da Matéria Condensada e Engenharia Elétrica. Ele participou de 50 bancas de avaliação de trabalhos de pós-graduação. Ele coordenou ou ajudou a coordenar 9 eventos científicos, tanto nacionais como internacionais. Entre 1989 e 2004 participou de 17 projetos de pesquisa, sendo que coordenou 11 destes. Atualmente participa de 10 projetos de pesquisa, sendo que coordena 9 destes, além de manter colaborações com grupos de pesquisa no país e no exterior. Atua nas áreas de Ciência e Engenharia de Materiais e de Física da Matéria Condensada. Em suas atividades profissionais interagiu com 154 colaboradores em co-autorias de trabalhos científicos. Em seu currículo Lattes os termos mais frequentes na contextualização da produção científica, tecnológica e artístico-cultural são: supercondutividade, caracterização microestrutural, microscopia eletrônica, aprisionamento de fluxo magnético, propriedades de transporte, materiais supercondutores, fios e fitas supercondutoras, cerâmicas supercondutoras, texturização e tratamentos térmicos, caracterização elétrica e magnética em baixas temperaturas.

On the properties and characteristics of superconductors with artificially introduced nanometric-scale pinning centers

Durval Rodrigues Jr., and Carlos A. Rodrigues

Superconductivity Group, Department of Materials Engineering, FAENQUIL, Lorena, SP, Brazil

Abstract – The present work describes the influences of nanometric-scale artificially introduced pinning centers on the transport properties of A15 superconductors. The superconducting and microstructural characterization of the samples enabled the understanding of the pinning

mechanisms acting in those materials, leading to results useful to the optimization of their critical current densities.

ONE of the most important applications of superconductivity is the generation of high magnetic fields in systems for materials characterization, accelerators to study particle collisions (like the Large Hadron Collider, CERN, Geneva, Switzerland) or reactors to test nuclear fusion (like the International Thermonuclear Experimental Reactor, Cadarache, France), among others. These high magnetic fields are obtained through the transport of high currents in the superconductors, determined by the optimization of the performance of these materials. The optimization of flux line pinning in superconductors is one of the most efficient ways to improve their transport properties. The pinning is due to the interaction of the flux lines with the inhomogeneities created in the superconducting phase.

The generation of pinning centers in a controlled way and with a previously projected distribution can contribute to the estimation of the pinning acting on the flux lines and to the improvement of the critical current densities J_c . These projected microstructures, called Artificial Pinning Centers (APC), can be artificially introduced into the superconducting phase through successive steps of bundling and mechanical deformation. This APC technique tries to enhance the flux pinning (and the critical currents) through the introduction of normal phases inside the already existing superconducting phase [1]. The introduction of APCs enables the generation of structures comparable to the superconductor coherence length ξ of the material (≈ 3.5 nm for Nb_3Sn). When the thickness of the APCs is comparable to ξ , the proximity effect induces superconductivity in these APCs and they are transformed into internal defects of the superconducting phases [2,3]. This produces highly efficient magnetic flux pinning and increases J_c .

The present work shows the development and characterization of several Nb_3Sn superconducting wires with nanometric-scale Cu(Sn) artificial pinning centers introduced in a controlled manner into the superconducting phase to enable the analysis of the flux pinning in this new type of composite. The multifilamentary APC wires were produced with different APC volume fraction and they were mechanically deformed to different final diameters, enabling the comparison of the APC dimensions to the Nb_3Sn coherence length.

These nanometric APC regions change the properties of the superconducting phase, mainly due to the proximity effect and to the inelastic scattering of the electrons on the interfaces. These effects will act on the pinning characteristics of the new composite [4]. The Cu(Sn) APCs are introduced in the Nb matrix through successive bundling, followed by swaging and wire drawing. During all fabrication process the Nb, Sn and Cu are kept in the pure state. After several bundlings to form multifilamentary wires, the final conductors were heat treated to form the Nb_3Sn phase and the Cu(Sn) APC regions [5]. Samples with different APCs volume fraction and different external diameters were taken from the wires for superconducting and microstructural characterization, enabling the determination of the flux pinning behavior with the variation of the Cu(Sn) APC dimensions, in comparison to the superconducting coherence length.

The results are analyzed under the microscopic point of view trying to determine the influence of the Cu(Sn) presence on the superconducting properties. The understanding of the pinning mechanisms acting in these superconductors can lead to important results useful to the optimization of J_c in superconductors.

Financial support by FAPESP, CAPES and CNPq, Brazil.

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WeSG – I-3
8:30/9:00am
STRUCTURAL, MICROSTRUCTURAL, ELECTRICAL AND MAGNETIC PROPERTIES OF RUTHENIUM PHYROCLORES

José Albino Aguiar

Structural, microstructural, electrical and magnetic properties of Ruthenium pyrocloros

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We present the structural, microstructural, electrical and magnetic characterization of rare earth (RE) based Ruthenium pyrocloros compounds with RE = Y, Gd, Eu and Sm. X-ray studies with *Rietveld* analysis reveal that RE-113 has a cubic structure belonging to the spatial group *Fd3m* with lattice parameter $a = 1.0152$ nm, 1.0242 nm, 1.0261 nm, and 1.0286 nm for Y, Gd, Eu, and, Sm based Ruthenium pyrocloros, respectively. The electron scanning microscopy studies show that the sample has homogeneous surface with grain size of the order of 2000 to 3 000 nm. The electrical resistivity as a function of temperature results showed that the compound presents a metal-insulator transition governed by a Coulomb gap mechanism and a new mechanism of conduction, starting at a temperature T^* dependente on the compound (for example, $T^* \sim 80$ K for the Y based system). The magnetic-susceptibility measurements show the onset of a magnetic transition at T_M followed by a spin-glass behavior at lower temperature T_{SG} . Both T_M and T_{SG} are compound dependent, for example $T_M = 170$ K and $T_{SG} = 80$ K for the Y based system.

Work financed by CNPq and CAPES.

WeSG-I-4
9:00/9:30am
UNIVERSAL ASPECTS OF THE MAGNETIC RESPONSE OF GRANULAR SPECIMENS HAVING A NARROW WEAK-LINK CRITICAL CURRENT DISTRIBUTION

Wilson Ortiz

Universal Aspects of the Magnetic Response of Granular Specimens Having a Narrow Weak-Link Critical Current Distribution

Wilson Ortiz

Grupo de Supercondutividade e Magnetismo

Departamento de Física

Universidade Federal de São Carlos

The magnetic response of granular specimens of superconducting materials has long been recognized as composed by two independent contributions, one related with the grains (intragranular) and the other with the linking matrix (intergranular). In general, transport and magnetic properties of such samples are treated as extrinsic, in the sense that the overall response is sample-dependent. This means that even those specimens prepared with highly homogeneous grains will have their relevant features strongly dependent on the intergranular contribution which, in turn, depends on the processing conditions. If the significant physical properties of the linking matrix are quite inhomogeneous, as is usually the case for ordinary ceramic samples of high-temperature superconductors, modeling of bulk properties will have to be done on the basis of average quantities. Nonetheless, resolving both contributions separately might be of relevance in a number

of cases, either because one wants to study the physics of each individual component of the system or, as in the present case, for the purpose of understanding how both parts interact with each other. To be able to address this problem, one has to deal with quite special granular samples, refined in both aspects: a collection of grains with very similar properties and a linking matrix formed by weak-links possessing nearly equal features. To study vortex matter dynamics in the presence of a minimally ordered mesh of defects, we have chosen a number of different superconducting systems which have in common a characteristic microstructural feature: an atypical, very narrow distribution of weak-link critical currents, $N(J_{cinter})$. For this class of samples, prepared through specially selected routes, we have detected a field-dependent threshold temperature, $T^*(H)$, at which the vortex dimensionality changes, as revealed by measurements of AC-susceptibility at different excitation fields and also by magneto-transport experiments in a 6-terminal flux-transformer arrangement. As one would expect, the threshold line $H^*(T)$ is extremely dependent on sample preparation and, among others, the above mentioned narrowness of $N(J_{cinter})$ is a crucial requirement for its observation. Although extrinsic in nature, $H^*(T)$ is a characteristic border on the magnetic phase diagram of such class of samples, being, in fact, the line at which the intergranular critical current vanishes. Moreover, data collected in this study demonstrate that vortices penetrated in the weakly linked region lose their longitudinal coherence at the $H^*(T)$ frontier. Unquestionably, it is of practical interest to locate, in the magnetic phase diagram, this upper bound to the regime of efficient flux pinning. Results on a number of systems are already available, among which we discuss here four examples: Mg-deficient MgB_2 ; multi-filamentary BSCCO-2224, Nb powder pressed in a pellet, and YBCO-123 prepared by a sol-gel route.

Symposium H - Sol-Gel Materials

TuSH-I-1

8:00/8:30am

DFB EMISSION OF RHODAMINE CONTAINING ORGANIC-INORGANIC HYBRID MATERIALS

Sidney J.L. Ribeiro

Biography

Sidney José Lima Ribeiro, 45 anos. (ISI- web of science- SJL Ribeiro)

Instituto de Química-UNESP, Araraquara (tel. 16-33016631, e-mail- sidney@iq.unesp.br)

Professor Livre-docente (UNESP-1999)

Doutorado- IQ-UNESP, 1992 (orientador- Prof. Dr.Gilberto F. de Sá)

Pós-doutorado- École Centrale Paris- 1994 (Dra. Jeannette Dexpert-Ghys)

Pós-doutorado- CNET- France Telecom- 1995 (Dr. François Auzel)

Interesses-

Química Inorgânica- Compostos de íons lantanídeos e espectroscopia eletrônica Materiais- Vidros e vitrocerâmicas, guias de luz (fibras ópticas, filmes finos e guias canais), Óptica integrada, biocelulose.

Ensino de química- Vidros como tema gerador em cursos básicos de química e física.

DFB emission of rhodamine containing organic-inorganic hybrid materials

Sidney J.L. Ribeiro¹, Daniela C. Oliveira¹, Karim Dahmouche¹, Rogéria R. Gonçalves², Adrien Vésperini³, Denis Gindre³, Jean-Michel Nunzi³, Younes Messaddeq¹

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²Depto De química, FFCLRP, USP, 14040-901, Ribeirão Preto-SP, Brazil

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Abstract We present a study of Distributed Feedback (DFB) Laser emission in organic-inorganic thin films prepared from ureapropyltriethoxysilane and methacrylic acid modified zirconium(IV) n-propoxide (ZPO) precursors.

In order to control the refractive index and improve the optical and mechanical properties, different Zr-Si molar ratios were used: Zr:Si: 75:25, 50:50 and 25:75. Thin films were obtained by spin-coating technique on glass substrates. Refractive index vary from so 1.55 to 1.51 depending on the stoichiometry. Rhodamine 6G containing stable films were prepared and devices are optically pumped using a Lloyd-mirror interferometer. Fig. 1 shows the schematic experimental set-up used in order to observe laser emission. Fig. shows the interference pattern obtained by the two overlapping beams. The period Λ of the pattern depends on the incident angle θ between the incident beam and the normal to the layer. Emission is observed by an optical fiber connected detector. Optical feedback is provided by distributed Bragg gratings formed in the film by interference patterns from the pump beam. This laser scheme permits efficient control of the stimulated emission in dye doped hybrid materials and Fig. 2 shows the laser wavelength as a function of the incidence angle θ . We report on particular features of the thin film DFB lasers. For a given DFB grating period, the number of lasing modes depends on film thickness (fig. 2 shows two emission modes). The effect of optical confinement was studied by varying the difference of refractive indices between the hybrid film and the substrate. .Spectral content of the devices can be analyzed using planar waveguide theory. Good agreement between the theoretical transverse electric mode structure and the laser emission spectrum is found.

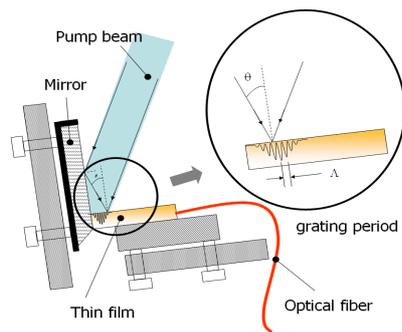


Fig. 1- Lloyd mirror interferometer. One part of the pump beam reaches directly the film while the other part overlaps the first one after reflection on the mirror. Inside the beams superposition region, an interference pattern with a period of Λ is created in the polymer thin film. This period Λ depends on the incident angle θ between the incident beam and the normal to the layer.

angle

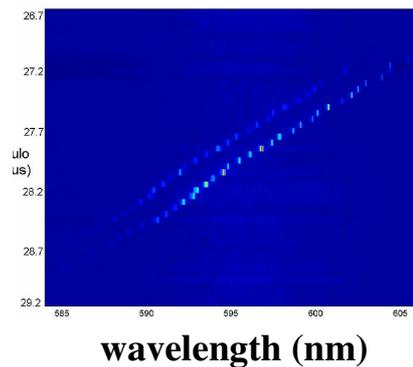


Fig. 2- Laser emission through the dye emission profile as a function of the incidence angle θ

TuSH-I-2

X-RAY ABSORPTION SPECTROSCOPY AND SOL-GEL CHEMISTRY

Valerie Briois

9:45/10:15am

TuSH-I-3

SOL-GEL COATINGS ON METALLIC SUBSTRATES

Wander Vasconcelos

10:30/11:00am

Sol-gel coatings on metallic substrates

Federal University of Minas Gerais - Dept. of Metallurgical and Materials Engineering

Laboratory of Ceramic Materials - Rua Espírito Santo, 35 - sala 206 - Centro

CEP 30160-030 - Belo Horizonte - MG - Brazil

Wander L. Vasconcelos

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Abstract – We described the formation of different sol-gel coatings on metallic substrates and the structural characterization of the films.

Sol-gel coatings can be used in a variety of applications, depending upon the composition of the film, the type of substrate and on processing conditions. We describe in this work some examples of sol-gel coatings on different substrates. We carried out the making of films based on silica, alumina, titania and mixed compositions. The major substrates that we used were stainless steels, either ASTM 316 or ASTM 409. Dip-coating was performed at withdrawal speed of 6 mm/min, 30 mm/min, and 60 mm/min. Deposited gel films were heat treated in air in a furnace at 100 °C, 300 °C, and 400 °C. We also describe in this work structural characterization using scanning electron microscopy, Fourier transform infrared spectroscopy, atomic force microscopy and thermal analyses

WeSH-I-4
9:45/10:15am
DESIGN OF INORGANIC MATERIALS BASED ON DYNAMICS OF SOFT MATTER

Eric Prouzet



Chemistry and Materials Science

Present position : Group Leader of the "Advanced Materials for Analysis and Separation" team (7 fulltime researchers, 2 technicians, 10 students in average)

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Personnel :

Born : October 28th 1958; french citizen, married, two children

Position Held

Senior Research scientist ("Directeur de Recherche") in Chemistry of Materials at the French National Scientific Research Center (CNRS) since 1988.

Education

1994-1996 : Visiting Scientist at Michigan State University, USA (Pr. T.J. Pinnavaia) and University of California, Santa Barbara, USA (Pr. C. Bunton) (NATO grant)

"Synthesis and characterisation of new ordered mesoporous materials"

1989-1990 : Post-doctoral period at the French Synchrotron Factory LURE (Laboratoire pour l'Utilisation du Rayonnement Synchrotron), Orsay University.

"EXAFS in-situ study of the V2O5-based lithium battery"

1988 : Ph. D. in Inorganic and Materials Chemistry , Nantes University.

"New nickel thiophosphates through soft chemistry reaction and lithium intercalation"

1986 : Upper Study Diploma (D.E.A) in Ceramic and Surface processes, Limoges University.

"Lower temperature sintering boehmite-derived transition alumina seeded with corundum."

1986 : Engineering Diploma in Ceramic, Ecole Nationale Supérieure en Céramique Industrielle, University of Limoges , France.

1984 : Master of Science in Organic and Inorganic Chemistry, University of Nantes , France.

Outlines of the Field of Research

- . Synthesis of organic and inorganic systems structured by soft matter (micelles, liquid crystals, biopolymers,...).

- . synthesis of mesoporous silica (powder and monoliths) with nonionic surfactants and copolymers ("proprietary" two-step process).

- . Nanostructured systems prepared within "Swollen Liquid Crystals" (oxides, polymers, metals).

- . Synthesis and study of hybrid nanobricks.

- . Synthesis of composite or mineral porous pearls prepared with alginate.

- . Synthesis and study of ceramic sheath of electrical insulation of superconductor Nb3Sn wires.

- . Preparation of membranes (ultrafiltration, gas permeation) and powders for HPLC chromatography.

- . Preparation of chemical sensors with Surface Acoustic Waves (SAW) systems.

- . Characterization of materials, especially for ill-ordered materials, with techniques ranging from solid state chemistry to soft matter physics.

Design of inorganic materials based on dynamics of soft matter

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Abstract – We described a new approach to structure inorganic materials based on the organization of inorganic matter through dynamics of soft matter.

Since chemists were more and more interested by the surface properties of materials, they tried to develop new syntheses, especially those of "Chimie Douce" (Soft Chemistry), that are particularly well dedicated to this purpose. This led to new classes of materials, such as highly porous materials (zeolites, molecular sieves, "mesoporous" materials,...) as well as hierarchically structured materials. This field of research helped to develop "bio-inspired" or "bio-mimic" syntheses based on soft matter concepts, where, even if the process by itself is by far much more simple than actual biomineralization, interactions at the molecular level govern the total structure and the whole complexity of the material organization at almost any scale from nanoscopic to microscopic. Compared with the "old-fashioned" solid state chemistry where bulk properties were achieved through the thermodynamic stabilization of phases by high temperature processes that led to ionocovalent bondings, soft chemistry may work with kinetically governed processes and intermolecular weak interactions.

Soft matter, including surfactants, liquid crystals (...) exhibits this kind of interaction and may lead to well-organized systems. Among the different ways available for the synthesis of compounds with designed structure, micellar objects were first used by a Mobil Co. group for the synthesis of mesoporous silica (known as MCM-41) with a well-defined honeycomb architecture, a high surface area and a narrow pore size distribution. Since, reactions based on close interactions between surfactants and inorganic species opened a new field of research where the dynamics of soft matter is exploited through an assembly process in order to structure inorganic materials.

We expanded this approach based on the organization of inorganic matter through dynamics of soft matter, which allowed us to explore different ways that will be described:

- (i) Mesoporous silica was synthesized with the help of nonionic surfactants through a two-step synthesis involving the intermediary preparation of hybrid micelles. These micelles behave like usual micelles and their structure is sensitive to slight changes in the synthesis process. This helped to tune pore size as well as particle size and shape by only monitoring the synthesis condition.
- (ii) Sodium alginate is well known for its properties of gelation when mixed with divalent cations such as calcium. If alginate is added dropwise in such a solution, drops form pearls that are usually used as substrates for biological reactions. We used this property by mixing different inorganic precursors able to gelify (sodium silicate, boehmite...). This allowed us to prepare spherical monodisperse particles in the millimetric scale with a continuous inorganic network. Depending on the precursors and the process, different microstructures, including highly opened with a honeycomb framework, were obtained.
- (iii) Hexagonal or cubic swelled liquid crystals were developed to be used as reacting mediums. In that case, the centre of surfactants cylinders is swelled up to a diameter of 30 nm with an organic solvent whereas they are regularly spaced by a continuous aqueous network. Examples of several syntheses, either in the inner cylinder organic phase or in the aqueous network, will be shown. The swelled liquid crystal acts as a mold and modifies the nanostructure of materials, compared with those prepared in bulk.

WeSH-I-5
10:30/11:00am
SILICA-CONTAINING PROTON CONDUCTING MATERIALS FOR FUEL CELL APPLICATION

Marcos Lopes Dias

Silica-containing proton conducting materials for fuel cell application

Marcos L. Dias(1), Marlon S. Santos(1) and Chiaki Azuma(2)

Instituto de Macromoléculas Professora Eloisa Mano (IMA), Universidade Federal do Rio de Janeiro (UFRJ),(1) Centro de Tecnologia, Bloco J, CP68525, 21945-970 Rio de Janeiro Brazil - mldias@ima.ufrj.br; (2) University of the Air, Setagaya Gakushu Center, 4-1-1, Shimouma, Setagaya-Ku, Tokyo 154, Japan

Abstract – Preparation, characterization and performance evaluation of proton conducting membranes based on sol-gel silica and crosslinked poly(vinyl alcohol) (PVA) was investigated. Two routes were used. In the first, sulfonated-modified nanosilica synthesized from chlorotriphenylsilane-terminated TEOS sol-gel reaction and subsequent sulfonation was used as proton conducting elements in formaldehyde crosslinked PVA/TEOS system. In the second route, poly(styrene sulfonic acid-co-maleic acid) was reacted with TEOS in a PVA solution. Membranes obtained by the second route containing about 30wt% of Si presented SEM detectable ionic domains and showed conductivity higher than Nafion at low hydration.

Commercial proton conducting materials used in Polymer Electrolyte Membrane Fuel Cell (PEMFC) are characterized by containing large amounts of sulfonic groups bonded to the polymer that constitute the membrane. In the 70's, the fully fluorinated polymer membrane NAFION (DuPont) was introduced and has served as standard material for fuel cell. Although this material shows high performance in PEMFC, especially due to high oxidative resistance and ionic conductivity, it is very expensive and typically presents limitations for high temperature operation. In addition, it shows limitations when operating with alternative fuels like methanol due to alcohol crossover in the large nanochannels produced by interconnected sulfonic group clusters resulting from phase separation. In 1997, NASA's Jet Propulsion Laboratory disclosed the development of an improved proton conductive membrane composed of sulfonated poly(ether ether ketone) (H-SPEEK). Due to a rigid nature of the polymer chain, phase separation of ionic groups in the polymer bulk is reduced resulting in channels of small size which decrease fuel crossover.

In Brazil there is a large interest in use ethanol as fuel in PEMFC. Investigation on new nanostructured proton conducting polymeric materials for membranes to be used in this kind of device is in progress in the Institute of Macromoléculas of University of Rio de Janeiro. The aim of this work was to investigate routes for preparation of proton conductive nanostructured membranes based on silica hybrids obtained by sol-gel reaction and crosslinked polymers with broad conductive nanochannels and high thermal resistance. Two routes were investigated: (1) crosslinked poly(vinyl alcohol) (PVA) nanocomposite membranes containing acid sulfonic surface modified silica nanoparticles obtained by sol-gel process; (2) crosslinked poly(styrene sulfonic acid-co-maleic acid)/PVA/silica hybrids.

Organically-modified nanosilica with average size diameter of 30 nm determined by light scattering was typically obtained from Route 1. These nanosilica which presents characteristic phenyl group bands at ~ 1430 e 730 cm^{-1} , has these bands shifted to 1037 cm^{-1} after sulfonation overlapping with SiOSi $800\text{-}1080\text{ cm}^{-1}$ band. UV-VIS phenyl band at 254nm in the non-sulfonated silica disappears after sulfonation. The chemically crosslinked composite membranes containing these sulfonated silica nanoparticles showed low water uptake (29%).

Crosslinked membranes from Route 2 showed good thermal resistance with TGA peak degradation under N_2 at near 320°C . These membranes are less homogeneous than Nafion, showing different domains that present different sulfur content as seen by EDS x-ray microscopy analyses of external and internal surfaces (N_2 fractured). Small white domains from SEM contain higher sulfur content which suggests that, although the material is highly crosslinked, phase separation also takes place. It seems, however, that due to the higher amount of proton conductive elements, the conductivity is maintained at high level. Proton conductivity measured with an AUTOLAB Potentiostat Galvanostat by using films sandwiched between Al electrode showed that at low hydration this membranes present superior ionic conductivity in comparison with Nafion.

As a conclusion, good proton conducting membranes for PEMFC based on silica/crosslinked hydrosoluble polymer hybrids are obtained by sol-gel process using TEOS as monomers. These polymers can be used to improve mechanical properties and presence of the silica improves thermal properties. The same phenomenon of phase separation was observed for membranes obtained from Route 2 which gave the best

proton conduction. Work to establish the extent of alcohol crossover through these materials is in progress.

References

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