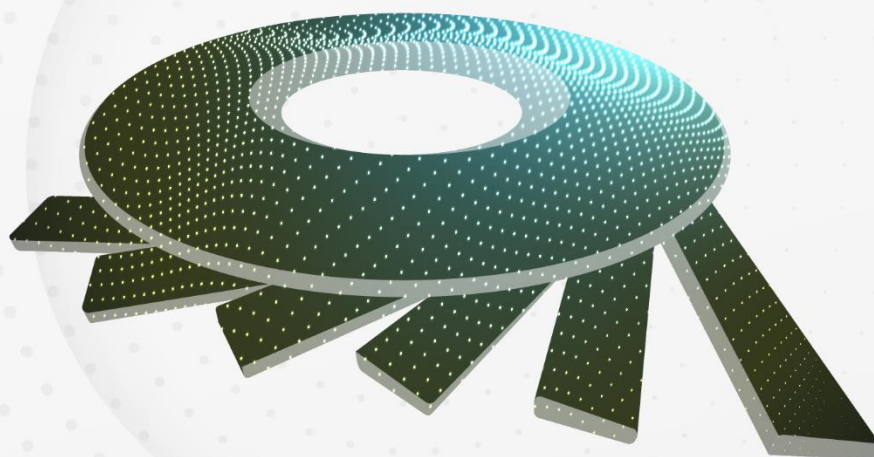


8-9th
OCTOBER '19
LNLS | CNPEM
CAMPINAS | BRAZIL

CARNAÚBA

WORKSHOP

1st International Sirius Workshop
on X-ray Nanospectroscopy
Nanodiffraction and
Nanoimaging (Xn³)



ABSTRACT BOOK



MINISTRY OF
SCIENCE, TECHNOLOGY,
INNOVATION AND COMMUNICATION



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FOREWORD

The Brazilian Synchrotron Light Laboratory (LNLS) is proud to host the first Sirius Workshop on X-ray Nanospectroscopy, Nanodiffraction and Nanoimaging (CARNAÚBA Workshop). This seminal workshop will bring together the LNLS community – external users, staff scientists and engineers – and experts working in the field of X-ray spectroscopy, diffraction and imaging at leading facilities around the world to present and discuss the new scientific opportunities offered by X-ray nanoprobe with scanning energy capabilities.

The CARNAÚBA beamline will be the first Sirius X-ray nanoprobe exploring the coherence and the spatial resolution to study a wide range of materials. This instrument will offer X-rays in the 2 to 15 keV energy range, with a beam spot down to 30 nm, for multimode analysis including techniques such as: X-ray fluorescence – XRF, X-ray absorption spectroscopy – XAS, X-ray excited optical luminescence – XEOL, X-ray diffraction – XRD and ptychographic X-ray coherent diffractive imaging – ptyCDI – in small angle scattering and Bragg geometries. When coming into operation in 2020, this beamline will offer state-of-the-art research tools for nanoscience and nanotechnology.

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INVITED TALKS

INVITED TALK**A MATERIALS SCIENCE VIEW OF
COHERENT SYNCHROTRON TECHNIQUES**

Angelo Malachias

UFMG, Belo Horizonte, Brazil

In this seminar we discuss current challenges in coherent scattering techniques (CDI, ptychography, etc..) from a materials science perspective. Besides the direct application of coherent X-ray beams in isolated objects, several limiting issues still need to be overcome in order to bring such techniques to common grounds with respect to Transmission Electron Microscopy (TEM) and Scanning Probe Microscopy (SPM) results. In particular, we discuss scientific cases of objects with large strain fields, the observation of lattice mismatch in novel two-dimensional materials systems and the use of anomalous (resonant) conditions, which require positional stability over selected energy ranges.

INVITED TALK

**ANCIENT LIFE COMES TO LIGHT,
NEW APPROACHES FOR OLD QUESTIONS**

Paula Sucerquia

UFPE, Recife, Brazil

Study of fossils allow paleontologists to know both biological and ecological characteristics of ancient life, but also its biodiversity and several times, the geological processes necessary to preservation. The fossil record is very odd and fragmented, some fossils can be very rare museum specimens, some preservation processes are exceptional occurring few times in the geological record, or fossils can come from remote and difficult to access localities, defying paleontologists to look for methods of study that preserve the specimen integrity without losing of data quality. Modern paleontology lead with issues beyond classic descriptions of organisms and interpretations about its ecological relationships or their position in geological timescale. Nowadays, fossils are valuable pieces in the puzzle of life's history, and its insertion in an ecological and evolutionary context, requires the ability to integrate diverse sciences i.e., geology, biology, chemistry, physics, and so on, this increasingly interdisciplinary area is reaching a more sophisticated level of knowledge by the integration of data from several areas. In recent years, several approaches based on Paleometry techniques have been contributed to understand the ancient life, solving old questions and raising new ones, these approaches using quantitative and/or qualitative non-destructive or less invasive analytical techniques has proved its relevance in Paleontology. Imaging and 3D modelling based on synchrotron micro CT can reveal hidden fossil structures inside rock matrix, geochemical techniques as X-ray Fluorescence (XRF), X-ray Diffraction (XRD) and, X-ray absorption near edge structure (XANES) of microfossils, fossil plants, animal carcasses, or preserved soft tissues, have allowed the characterization of morphology, chemistry, physiology, and ecology of extinct organisms and the understanding of fossilization processes thorough the understanding of the geobiological mechanisms and interaction of decaying organisms and its surrounding environment including interaction with microorganisms.

INVITED TALK

**AuPd BIMETALLIC NANOPARTICLES:
CORRELATIONS BETWEEN COMPOSITION,
LOCAL STRUCTURE AND CATALYTIC ACTIVITY**

Liane Rossi

USP, São Paulo, Brazil

Bimetallic nanoparticles may present different and superior catalytic properties to their monometallic constituents but are much more complex and difficult to characterize systems. The understanding of the effects of compositional variation (bulk and surface), morphology and possible restructurings suffered by bimetallic nanoparticles under reaction conditions, as well as correlations with their catalytic properties, is fundamental for the design of increasingly active and selective catalysts. In this seminar we will discuss the studies with bimetallic nanoparticles of AuPd composition and varied structure against oxidation and hydrogenation reactions. The samples were prepared and characterized by X-ray absorption (XAS), X-ray diffraction (XRD) and x-ray photoelectron spectroscopy (XPS) techniques. Samples with structure ranging from homogeneous alloys to core-shell structures were obtained. For the oxidation of benzyl alcohol an increase in catalytic activity of up to five times was observed after the calcination of the AuPd alloy type catalysts, with higher activity for palladium rich samples. When preparing core-shell Au @ Pd type catalyst, the amount of palladium required for maximum activity is much lower. For hydrogenation reactions, amounts as low as 1% palladium are enough to develop active and selective catalysts. All systems were compared with monometallic catalysts.

TED TALK

**COHERENT DIFFRACTIVE IMAGING TECHNIQUES
IN BIOLOGICAL SYSTEMS**

Carlos Sato B. Dias

LNLS-CNPEM, Campinas, Brazil

Coherent Diffractive Imaging (CDI) is a collection of techniques dating back to 1999 when a simple inorganic mask was imaged proving the concept. Since then, significant developments have been done branching on multiple techniques such as ptychography, plane wave-CDI, Bragg-CDI, among others. Today, those techniques are ever improving and had already proved as a viable tool with tridimensional imaging capabilities (tomographic imaging) a resolution between optical and electron microscopy, thus working as a bridge between the two. Within this context, biological samples are notorious for being highly hierarchical and to present a strong relationship between structure and function. Such a structured sample is typically tridimensional in space and beneficiates from both the tridimensional imaging of x-ray and the resolution of the CDI techniques. Here, we discuss the perspectives for studying biological samples using CDI. We will present some of the main results found in the literature that would have a significant impact on biological imaging experiments. Also, we shall present some of the current developments on sample preparation for the proper design and execution of tridimensional CDI-based biological imaging at the Carnaúba beamline.

INVITED TALK

**COHERENT X-RAY DIFFRACTION IMAGING
UNRAVELLING STRUCTURE-ACTIVITY
RELATIONSHIPS OF GOLD NANOCRYSTALS
UNDER CO OXIDATION CONDITIONS**

Amélie Rochet

LNLS-CNPEM, Campinas, Brazil

Chemical properties of catalytic materials are dependent on dynamic changes of the three-dimensional structure of the catalysts as well as the reactive environment. The identification of the active sites formed under reaction conditions remains a great challenge. Besides, defects and lattice strain dynamics of nanocrystals directly tuned their catalytic properties. To reveal the relationship between catalysts structure and activity, the study of catalysts under operando conditions is crucial. Bragg coherent X-ray diffraction imaging (Bragg CDI) provides a unique opportunity to follow in 3D under realistic reaction conditions the strain and defects dynamics in the surface and inner core of the nanomaterials. In this talk, I will present our in situ/operando Bragg CDI investigations of gold catalysts during CO oxidation. Under the catalytic reaction conditions, we investigate the defects dynamics of gold nanocrystals with twin domains and show the *in situ* formation of a nanotwin network that correlates with the catalytic properties. Besides, we follow at the single nanoparticle-level, the highly dynamic 3D strain distribution during the hysteresis phenomenon occurring through CO oxidation reaction cycles.

INVITED TALK

**DEVELOPMENT OF TOOLS TO LABEL AND IMAGE
PROLIFERATIVE CELLS AND NEURONS USING
X-RAY IMAGING TECHNIQUES WITH
SYNCHROTRON RADIATION**

Hozana Castillo

LN BIO-CNPq, Campinas, Brazil

Zebrafish have an exquisite capacity to regenerate damaged neurons following spinal cord injury (SCI). The activation of quiescent ependymal cells that serve as endogenous stem cells is required to initiate neural regeneration in zebrafish, but little is known about the cellular mechanisms these cells undergo or the signaling pathways that activate them. Therefore, understanding the cellular behavior and manipulating signaling pathways that activate the stem/progenitor cells holds great promise in the treatment of SCI. The aim of this project is to develop tools to label and image newborn neurons during the regenerative process in zebrafish using X-ray imaging techniques with Synchrotron Radiation (SR), such as X-ray Fluorescence microscopy (XFM) and X-ray Computed Tomography (XRCT). More specifically, this project involves the development of chemical probes that label proliferative cells with heavy metals or other chemical elements and a technique to label specific neurons with nickel, allowing the identification of newly formed neurons during the regenerative process using the X-ray imaging techniques with SR mentioned previously.

INVITED TALK

**GLYCANS AND METALS FROM MARINE
INVERTEBRATES TO MAMMALIANS: ANCIENT
REGULATORS OF CELL FATE IN HEALTH AND DISEASE**

Mariana Stelling

IFRJ, Rio de Janeiro, Brazil

Glycosaminoglycans are ancient molecules found from early invertebrates to humans that have evolved in complexity through time. In particular, they interact with many metals and co-operate to regulate diverse biological processes, such as cell growth, differentiation, and migration in development and disease. Our work focuses on how glycosaminoglycans regulate metallic elements' activity within mammalian systems and how this process is disrupted in cancer. We also study how ancient glycosaminoglycans produced by marine invertebrates present antitumoral properties in mammalian systems, bringing to light an evolutionary link on how evolving organisms dealt with multicellularity in continuously and profoundly changing environments. Our perspectives for the new light source, Sirius, include higher magnification imaging of isolated mammalian normal and cancer cells, as well as marine invertebrate cells, in order to unveil subcellular metals distribution. We also intend to develop techniques to couple protein and glycans imaging with multi-elemental imaging in the same sample, allowing for more robust correlations in biological systems.

INVITED TALK**HYPERSPECTRAL XEOL IMAGING AT THE NANOSCALE**

Gema Martinez-Criado

ICMM, CSIC, Spain

Owing to the sensitivity (i.e., signal to background ratio), temporal and spatial resolutions, pulsed synchrotron X-ray nanobeams are promising tools for correlative light and X-ray chemical analysis of functional nanodevices. However, their full potential is ultimately dictated by our ability to detect multiple property-function relationships taking place at the nanoscale in the spatial and time domains. Only a combination of high-resolution X-ray excited optical luminescence techniques can provide a comprehensive understanding of their complex functionalities. Here we describe how a multimodal hard, polarized and pulsed X-ray nanoprobe addresses fundamental questions in nanowire research about the carrier dynamics, relaxation, recombination and light polarization within single wires at the nanoscale. Selected topics ranging from cluster formation, dopant segregation, and phase separations to quantum confinement effects are investigated with sub-100 nm spatial resolution and sub-50 ps temporal resolution. This approach opens also new avenues for structural, composition and optical studies with broad applicability in materials science.

INVITED TALK

**NANOPARTICLES-CELL INTERACTION:
INTRACELLULAR DYNAMICS AND CELL STRUCTURE
RESPONSE PROBED BY X-RAY MICROSCOPY**

Mateus Cardoso

LNNANO-CNPEM, Campinas, Brazil

The understanding of the mechanisms behind the interaction of nanoparticles and living cells is the cornerstone for the development of future nanomedicine. Improvements on nanoparticles efficiency and their safety regarding applications on diagnostics and therapeutics can fundamentally be traced back to the nanoparticle distribution within a cell. However, probing nanoparticles internalization and trafficking into cells with subcellular resolution remains limited by conventional 2D imaging or low-resolution 3D information. At the same time, the recent development of x-ray microscopes has pushed x-ray 3D imaging down to an isotropic nanometer resolution is now possible. In this way, the Carnaúba beam line and their two experimental stations, named Tarumã and Sapoti, will represent a turning point for nanoparticle visualization and characterization. Multiple techniques such as XRF, XAS and specially ptychography will provide 3D chemical and structural analyses of in situ nanoparticles; there is inside a cell or tissue. At this work, we discuss the perspectives for studying nanoparticles-cells systems using Ptychographic Tomography (PXCT) at the Carnaúba beamline how it will allow the high-resolution imaging of multiple nanoparticles at various cell incubation stages unveiling the fundamentals behind both nanoparticle uptake and long-term exposure to advanced nanomedicines.

INVITED TALK

**NANOPHOTONIC MATERIALS FOR ENERGY
HARVESTING AND STORAGE: LOOKING CLOSER WITH
CARNAUBA COHERENT BEAM**

Lucas Rodrigues

USP, São Paulo, Brazil

New applications of different photonic materials arise every year, especially for harvesting and storing energy. For example, applications like photosynthesis at night, photodynamic therapy in the dark and bioimaging without in-situ excitation sources (or radioactivity) could sound controversial a few years ago, but can be achieved now using light storage compounds, e.g. persistent luminescence materials. These materials store energy in defects releasing it with the aid of thermal energy. On the other hand, applications very well established like solar energy harvesting from up- and down-conversion materials urges for more efficient processes. To optimize these processes, as well to think in new applications for photonic materials, it is important to look deep into the materials. Nano and micro domains are fundamentals for the emitting, absorption and energy transfer processes. The Carnauba beamline will aid the understanding and optimization of these phenomena allowing us to investigate the material domains during the electronic processes with e.g. coupled micro and nano XEOL, XRD, XAS and XRF.

INVITED TALK**NEW CHALLENGES IN NANOMATERIALS
CHARACTERIZATION TO BRIDGE THE “SIZE GAP”**

Felix Requejo

UNLP, Argentina

Historically, spectroscopic techniques represent a powerful tool for electronical or chemical characterization, being a complementary method of image-based techniques to create the strongest scientific description of physical or biological systems. However, the combination of those strategies results not enough for actual demands in scientific research, in particular at the Nanoscience field. The emerging new instrumentation based on nano-spots of X-rays using synchrotron sources, as the Carnauba beamline at SIRIUS synchrotron, represent new opportunities for new rational answers and even revisit old and relevant fields. In this presentation we will present few examples of nanomaterials characterization, like nanoparticles, nanofibers, atomic quantum clusters, interphases, etc, analyzing the possible impact of the new instrumentation for characterization at the Nanoscience field.

INVITED TALK

**OPPORTUNITIES FOR SOIL AND PLANT SCIENTISTS
AT THE SIRIUS-LNLS CARNAÚBA BEAMLINE**

Dean Hesterberg

Dept. of Crop and Soil Sciences, North Carolina State University, Raleigh, North Carolina, USA

Optimal performance of soil-plant systems is essential for high agricultural productivity and conservation of natural ecosystems. The element specificity of synchrotron X-ray techniques provides unprecedented mechanistic information about chemical and structural properties of soils and plants, which serves as a foundation for improved strategies for managing natural resources. The CARNAÚBA beamline is designed with unique and complementary analytical capabilities of that are well suited for studying soils, plants, and soil-plant interfaces. Soils themselves comprise complex assemblages of minerals, organic matter, non-crystalline inorganic solids and microorganisms; and plant roots alter the chemical and microbial properties of the root-soil interface, i.e., the rhizosphere. Functional insights to such complex, multi-component systems are enhanced by characterization of the same sample areas with multiple, spatially resolved analytical techniques. For multi-element imaging, the two experimental stations at CARNAÚBA with different beam spot sizes will facilitate rapid imaging of larger sample areas or volumes to define overall spatial patterns of chemical elements, followed by high-resolution imaging of chemical substructure on features of interest using a smaller beam (down to 30 nm). Moreover, heterogeneous spatial patterns of chemical elements in soils and at soil-root interfaces enhances identification of less abundant chemical species of a given element by micro- or nano-scale X-ray absorption spectroscopy (μ -XAS, n-XAS). Similarly, in plant sciences, access to X-ray beams of multiple spot sizes at a single beamline will enable rapid and high-resolution imaging of important biological elements at tissue, cellular, and sub-cellular levels. The CARNAÚBA beamline also covers a broad energy range from tender to hard X-rays. Soils contain numerous chemical elements. Ideally, all elements of concern to a particular problem could be imaged simultaneously to augment chemical speciation analysis via knowledge of element co-localization. At low-energies, the more abundant soil matrix elements (silicon and aluminum) can be imaged along with important plant nutrients - especially phosphorus, sulfur, potassium, and calcium. Remarkably, the beamline is also designed for imaging and spectroscopy of heavier soil-matrix elements and biological micronutrients (e.g., iron, manganese, zinc, copper, selenium), and toxic metal(loids) of environmental concern in soils, water, and the food chain (e.g., arsenic, mercury, cadmium and lead). Finally, spatially resolved diffraction should identify mineral phases at matrix contents far below those needed for detection by bulk-sample diffraction. The CARNAÚBA beamline will provide complementary X-ray techniques that are of great value for addressing a broad range of problems in soil and plant sciences.

INVITED TALK**PTYCHOGRAPHIC RECONSTRUCTION
USING MULTI GPUS**

Eduardo Miqueles & Giovanni Baraldi

LNLS-CNPEN, Campinas, Brazil

In this talk, we will describe the main challenges for ptychographic reconstruction using an array of GPUs within the context of high-performance computing. A reconstruction pipeline for SIRIUS will be discussed, presenting algorithmic perspectives for the beamline Carnauba.

INVITED TALK

SIRIUS STATUS

Harry Westfahl Jr.
LNLS-CNPEM, Campinas, Brazil

INVITED TALK

**TESTING BIOGENICITY OF ANCIENT ROCKS –
HOW NANOMETRIC RESOLUTION CAN SOLVE
THESE PROBLEMS**

Flávia Callefo

LNLS-CNPq, Campinas, Brazil

Microbial life may have colonized Earth already before 3.5 billion years. However, no fossil evidences from this period have been universally accepted so far, and any evidence of life around these ages is contestable or dubious. Samples related to the earliest signs of life on Earth are rare and often require non-destructive techniques, as well as the highest resolution as possible. One way to detect or support biogenicity in ancient rocks is to correlate the distribution patterns of trace elements and minerals commonly associated to modern microbial metabolisms with the morphological biosignatures. Here, we present some of these approaches to solve questions about biogenicity using synchrotron-based X-Ray techniques, such as X-ray Fluorescence, X-ray absorption and X-ray diffraction, in which nanometric resolution is of prime importance. The examples of applicability include 1 billion years old Brazilian stromatolites, in which traces of a putative microbial ecological interactions may have been preserved, as well as a candidate of the oldest record of life on Earth consisting of putative preserved biofilms in rocks of 3.7 billion years old from Isua Greenstone Belt, Greenland.

INVITED TALK

**THE CARNAÚBA BEAMLINE:
STATUS AND FUTURE OPPORTUNITIES**

Hélio Tolentino

LNLS-CNPEM, Campinas, Brazil

The status of the CARNAÚBA beamline will be presented, as well as future scientific opportunities.

INVITED TALK**UNDERSTANDING ENERGY STORAGE MATERIALS
WITH IN-SITU COHERENT DIFFRACTION IMAGING**

Andrew Ulvestad

Tesla Inc., USA

Coherent diffraction continues to develop into a mature technique capable of imaging single nanoparticles and thin film grains in a variety of in-situ environments. Its unique sensitivity to structural perturbations, and dislocations, has made it the premier imaging tool for crystalline specimens with sizes of 100 nm to 1 micron. In this talk, I will introduce coherent diffraction in both Bragg and transmission geometries and then spend the majority of the time on applications of the techniques to two energy storage systems that share many similarities in their underlying thermodynamics: advanced battery cathodes and hydrogen storage materials.

INVITED TALK

**USING SYNCHROTRON-BASED TECHNIQUES TO
PROBE MICROBE-MINERAL INTERACTION: FROM LIFE
DETECTION TO BIOTECHNOLOGICAL APPLICATIONS**

Douglas Galante & Lara Maldanis

LNLS-CNPEM, Campinas, Brazil

Microorganisms were the first life forms to appear on the planet and nowadays they are present on almost all environments, even the most extreme ones. They have a large variety of metabolisms and adaptative strategies to survive, being responsible for global changes on the planet. A special group of microorganisms are of those capable of interacting with inorganic species to obtain energy, to reduce the toxicity of the medium and others. This area of knowledge, called biogeochemistry, geomicrobiology, or also microbe-mineral interactions, can be important, for example, to study ancient or modern registers of life, since microorganisms are responsible for the precipitation of biogenic minerals, and to the development of biotechnological applications, like biomining, bioprecipitation of functional nanoparticle, bioremediation of contaminated environments, etc. Synchrotron-based techniques play an important role on the study of the formed species as well as on the understanding of the processes involved. In this presentation, it will be showed a general overview of this field and the importance of some spectroscopic techniques, focusing on some of the problems that are being carried out using the facilities of LNLS and which will take a leap further with Sirius.

INVITED TALK**X-RAY SPECTROMETRY IN AGRICULTURAL SCIENCES**

Hudson de Carvalho

CENA-USP, Piracicaba, Brazil

In this presentation, we will approach some uses of X-ray spectrometry in agricultural sciences. We will outline the state-of-the-art for chemical speciation and elemental mapping in plant and soil samples. Currently, XRF allows tracing the movement of elements through plant tissues, which is an alternative to the use of radioisotopes. Additionally, XAS can reveal the interaction between fertilizers and herbicides in tanks and probe the chemical environment of nutrients within seeds and plants. The main challenges in the field consist in developing sample environments for in situ measurements, coupling of techniques and dealing with radiation damage during in vivo analysis.



ABSTRACTS

POSTER PRESENTATION

**AEROSOL INTEGRATED ANALYSIS
IN WEST ANTARCTICA: CHARACTERIZATION
AND CHEMICAL IMAGING**

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Mary Gilles³, Johannes Weis³, Alex Laskin⁴

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Changes in Antarctica ice sheets and ice shelves are of primary concern to the regional and global climate. We hypothesize that the West Antarctic warming can be related to the aerosols transported and/or formed in this region. Rather than being inert, snow is highly active, with snowpack impurities being photolyzed to release reactive trace gasses such as OH, NO/NO₂, and O₃ into the troposphere. The impact of solar radiation, enhanced by O₃ depletion, creates the optimal conditions for heterogeneous gas-to-particle reaction, modifying the chemical and physical properties of aerosols substantially. This study shows evidence of atmospheric processing, via microscopic and molecular speciation of individual aerosol by Scanning Transmission X-ray microscopy with near-edge X-ray absorption fine structure spectroscopy (STXM/NEXAFS). Specifically, STXM reveals an accurate fraction of internally mixed particles with NaCl cores and nitrate coatings. The scattering efficiency of sea salt particles may decrease as a consequence of the external nitrate covering since the hygroscopicity of a mixed nitrate-salt particle is weak. The unique signal of Cl_xO_y-type-molecules revealed could be possibly associated with OH radical oxidation products of NaCl particles. Considering the perchlorate (ClO₄) has a supercooling property, it is possible to assume that Antarctica successively underwent interglacial warming phases, creating salty crusts. The salty influx combined with the geothermal heat flux and the overlying ice sheet may preserve water in its liquid state at the subglacial environment.

ORAL PRESENTATION

**ANGULAR DEPENDENCE OF FERROMAGNETIC
RESONANCE IN IrMn₃/PY**

J. Holanda^{1*}, Hilal Saglam^{1,2}, Vedat Karakas³, Michael Vogel¹, Yi Li¹, Ralu Divan⁴,
Yuzi Liu⁴, Ozhan Ozatay³, V. Novosad¹, John E. Pearson¹, Axel Hoffmann¹

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Antiferromagnetic materials with non-collinear spin structures, such as IrMn₃, are of increased current research focus. Non-collinear antiferromagnets have additional spin Hall effects due to the net chirality of their magnetic spin structure, which makes them more complex than ordinary non-magnetic materials. In this work, films of IrMn₃ were grown using magnetron sputtering on (100)MgO substrates. These were integrated into MgO/IrMn₃/Ni₈₀Fe₂₀/Ti heterostructures. The Ti(2 nm) layer is used to protect the surface properties of IrMn₃ or of the permalloy (Ni₈₀Fe₂₀). The Ni₈₀Fe₂₀ and Ti layers were deposited at room temperature. The IrMn₃ was characterized by X-Ray Diffraction (XRD), Superconducting Quantum Interference Device (SQUID) magnetometry, magnetotransport measurements using a Physical Property Measurement System (PPMS) and Transmission Electron Microscopy (TEM). We use dc current modulation of ferromagnetic resonance, which creates a spin accumulation at the interfaces of the magnetic heterostructure. This enables us to explore spin-orbit torques and magnetic damping in individual nanoscale samples with uniform magnetization profiles. Due to the microscopic non-collinearity of the antiferromagnetic spin structure, there is a magnetic spin Hall effect, which gives rise to a change of the magnetic damping as a function of the magnetic field direction, which also defines the polarization of the spin accumulation due to the magnetic spin Hall effect. The variation of damping was 41% which represents one new form for control of information at the interface antiferromagnetic/ferromagnetic heterostructures.

ACKNOWLEDGEMENTS:

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POSTER PRESENTATION

**APPLICATION OF IRON OXIDES IN ADSORPTION FOR
THE STUDY OF DYE-OXIDE INTERACTION**Ketlyn Wolfart Borth^{1*}, Verônica de Carvalho Teixeira², Fauze Jacó Anaissi¹¹Universidade Estadual do Centro-Oeste, Brazil; ²Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM), Brazil.

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Iron oxides are found in natural or synthetic form and can stabilize in different crystalline structures depending on the chemical reactions involved. Digestion acid methodology can be used to mitigate environmental problems by the recycling of metallic waste, such as steel [1]. Steel, as a metal material of great demand, can be recycled without losing its properties or being transformed in new iron compounds. These compounds can be applied as adsorbents to remove harmful substances from water used in textile process and thus creating a cycle to control contaminants through materials previously called pollutants. In this work, two iron oxides were synthesized, LCNH e TCNH, from steam steel and carbon steel, through acid digestion with HCl ($1 \text{ mol} \cdot \text{L}^{-1}$) followed of co-precipitation with NH_4OH ($5 \text{ mol} \cdot \text{L}^{-1}$), for application in adsorption of the congo red dye and, after, study of the interaction dye – oxide. The oxides were characterized through X-ray diffraction (XRD), before and after the adsorptive process. The diffratograms of the pure oxides presented the main peaks at 7.5° , 14.6° and 23.7° , characteristic of iron oxychloride, goethite and magnetite or maghemite, respectively. The ordinary phases of iron oxide were expected, however, the formation of iron oxychloride occurred due to the excess of the chloride ion, from the digestion acid, to advantage the transformation of the iron oxide (III) to FeOCl [2]. After the adsorption, the most intense peaks in the XRD referred to congo red dye. However, the iron oxide phase in 23.7° was still identified. Therefore, the interaction dye-oxide did not create a compound with a modified structure through chemical bondings. Besides, the physical-chemical studies defined that the adsorption occurred in monolayer (Langmuir Isotherm), in which each site holds one molecule [3]. That is, the interaction is simply among the charges in the solid-liquid surface, in other words, between the anionic dye and the cationic oxides.

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POSTER PRESENTATION

**CHARACTERIZATION OF $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ POWDERS
MODIFIED WITH W^{6+}**

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$\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ (CCTO) has been studied due to its several properties, such as dielectric [1], non-ohmic [2], gas sensing [3] photoluminescent (PL) [4] and photocatalytic [5]. It exhibits a pseudo-cubic perovskite structure with stoichiometric formula $(\text{A})\text{BO}_3$, in which the ions Ca^{2+} and Cu^{2+} share the site A and Ti^{4+} occupies the site B. While the Ca^{2+} is in a body centered cubic structure, the Cu^{2+} occupy the center of the faces and edges. As result, the structure becomes distorted. The structural flexibility and metal-oxygen polyhedral disorders are key to understand PL emission on CCTO [6]. CCTO/ $x\%\text{W}^{6+}$ powders, prepared via solid state reaction, were characterized using a combination of in-situ techniques: X-ray diffraction (XRD), Raman spectroscopy and X-ray Absorption (XAS). XRD was performed at the XRD1 from the LNLS to investigate the long-range order structure and, all samples showed single phase. The crystalline and unit cell parameters were calculated using the Rietveld method and the experimental data were successfully fitted using the Split Pseudo-Voigt (SPV) function. The Rietveld quality factors (R_p , R_{wp} , R_{exp} , R_{bragg} and χ^2) indicated an effective refinement in accordance to the literature. Short-range order was characterized using Raman spectroscopy. XAS (XANES and EXAFS) measurements were performed at the XAFS1 and XAFS2 beamline from the LNLS, around the Ti, Ca and Cu K-edge. Crystallochemistry (geometry and oxidation state) of Ti, Ca and Cu ions was studied through XANES. On the Ti K-edge spectra, the peaks identified on the pre-edge region are associated to the octahedral coordination of the Ti^{4+} ions on each of the samples [7]. All samples present pre-edge features A, B and C associated to the transition of excited 1s electrons into the bands t_{2g} and e_g of the octahedral field. Information regarding bond length and the coordination number was determined using EXAFS.

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POSTER PRESENTATION

COMPARATIVE STUDY ON LOCAL STRUCTURE AND OPTICAL PROPERTIES OF Y₂O₃:ND NANOPARTICLES PREPARED BY ALTERNATIVE SOL-GEL ROUTES USING NATURAL COMPOUNDS

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Nanomaterials doped with Nd³⁺ ions have attracted the interest for optical applications as laser media and photonic devices due to its efficient four laser-level system at room temperature. In particular, the intrinsic features of yttrium oxide (Y₂O₃) host such as its high thermal conductivity, broad transparency window and small nonradiative relaxation rate, can help to improve the infrared emission of Nd³⁺ ions. Currently, there is a scientific interest in develop synthesis routes more adequate with respect to improvement of the properties, as well as viability of production in terms of good reproducibility, low toxicity and environment impact and low costs. In this sense, this work aims to present a comparative study about structural and optical properties of Nd-doped Y₂O₃ nanoparticles produced by two different sol-gel routes. The first one employs coconut water as polymerizing agent and the other uses the complexation capability of natural organic matter (NOM) present in aquifers to chelate the metal ions in the precursor solution. The viability of these routes was recently reported [1,2]. Samples with different Nd concentration and thermal treatment were obtained. Their local structural and optical properties were investigated by means of X-ray absorption and VUV spectroscopy techniques. From preliminary analysis of EXAFS measurements, it was observed that the samples prepared by using NOM presented a longer range ordering of the structure around Y atoms in comparison with the samples prepared with coconut water treated at the same temperature. This feature can indicate that the complexation capacity of the NOM is higher than the proteins of coconut water. In addition, the PL emission spectra showed typical emission of Nd³⁺ in the infrared range for all the samples more efficiently under excitation at around 5.0 eV.

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ORAL PRESENTATION

CRYSTALLOGRAPHIC AND MAGNETIC BEHAVIOR OF $Gd_5(Si,Ge)_4$ NANOPARTICLES PRODUCED BY A NS PLD

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The $Gd_5(Si,Ge)_4$ family compounds have been intensively investigated since the discovery of the Giant Magnetocaloric Effect near room temperature in 1997 [1], for magnetic refrigeration applications. From these extensive researches many other important properties were revealed, such as giant magnetoresistance and colossal magnetostriction [2,3], that rises from an extreme sensitivity to variation of external and internal parameters. However, only recently, the studies on the $Gd_5(Si,Ge)_4$ system in reduced dimensionality have gain attention since the achievement of thin films and nanostructures with good crystalline features. For instance, a recent study performed by Hadimani et al. have shown that $Gd_5Si_{1.3}Ge_{2.7}$ ($x=0.325$) nanogranular thin film produced by femtosecond laser ablation have a giant thermal expansion of 8000 ppm, showing its potential for sensing/actuating applications [4]. In this work, a more conventional and accessible KrF Excimer laser was chosen to produce $Gd_5(Si_{1-x}Ge_x)_4$ with $x = 0.45$ and 0.6 nanoparticles using Ar atmosphere. Synchrotron X-ray Diffraction analysis shows that the particle size reduction to 8-30 nm lead to a change from a Monoclinic to an Orthorhombic-I structure for $x = 0.45$ system and a 40 K increase in its Curie temperature. Rietveld calculations for both compositions revealed a shortening between the Si/Ge bonds, being the responsible for the higher T_c . Furthermore, the nanoparticles presented a relative unit cell volume reduction $\Delta V/V_{bulk}$ of 1.59% and 0.94% for $x = 0.45$ and 0.60 samples, respectively, which is closely related with their bulk counterpart volumetric compressibility values that reduces for higher Si contents. Large microstrain values (in the order of 20 ppm), obtained by Williamson-Hall fitting, can be responsible for these nanostructures distinct features. In resume, these findings reveal that the $Gd_5(Si,Ge)_4$ compounds also present a sensitivity to dimensionality reduction in its structural and magnetic behaviors.

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POSTER PRESENTATION

**DECIPHERING PAST ENVIRONMENTS THROUGH
SYNCHROTRON RADIATION**

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In the last few years, Synchrotron Radiation (SR) has proven to be an intense and versatile source for geological investigations. The X-rays generated have a brilliance (thus flux) that is many orders of magnitude higher than that of standard laboratory sources. Furthermore, monochromatic beam at a specific energy, which corresponds to a determined element, enables detailed and in situ investigations of geological materials. This can be performed with high spatial resolution and in a non-destructive way. For instance, through SR, geoscientists are able to identify elements in trace amounts as well as their physicochemical states. Consequently, subjects that were once considered obscure or inconceivable, are now being resolved by adopting this approach. Despite that geosciences are subdivided into several branches, many hold similar objectives, which is the understanding and reconstruction of the processes that acted on ancient environments. The Paraná-Etendeka Magmatic Province (PEMP) is a Large Igneous Province (LIP) where ca. 1 GKm³ of lava was expelled during a short interval of time (ca. 1 Ma) in the Cretaceous Period. Although it had the potential to generate considerable amounts of gases and volatiles, it was not able to produce significant environmental changes. Curiously, and different to other LIPs, no mass extinction is associated with this event. This paradox raises several important questions, but the main thing is why this volcanic event is so different than all others that provoked changes in the biosphere? To resolve this, SR became a crucial approach, as some beamlines are able to resolve the presence of the remaining volatile elements that stay trapped within the magma, such as sulfur. Among these techniques available at UVX, XANES (X-Ray Absorption Near-Edge Structure) can provide information about the oxidation states of these elements. Consequently, we are able to recognize the processes that acted during the volcanic events and their influence in th

ORAL PRESENTATION

**DEMANDS ON SYNCHROTRON X-RAY IMAGING FOR
ENGINEERING MATERIALS**

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X-ray imaging is traditionally used in medicine for the 3D characterization of biological matter, but it has demonstrated over the last 20 years an increasing relevance in the non-destructive characterization of microstructures and damage in engineering materials. While micro and nanotomography performed with laboratory X-rays as well as synchrotron light are already well-established tools in materials research in developed countries, the reality of in- and ex-situ studies of engineering materials based on X-ray imaging in Brazil reveals a very scarce use of this powerful technique. Synchrotron X-ray imaging within the 2 to 15 keV energy range at Carnauba will allow to analyze, with sub-microscopic resolution, lightweight and low absorption engineering materials, such as magnesium- and aluminum-base alloys and composites, as well as ceramics and polymers. Nanotomography allows for quantifying the volumetric fraction and 3D arrangement of phases, reinforcement particles, porosity and cracks, as well as their variations in micro and nanometric scale throughout the investigated body. Compared to traditional 2D techniques, 3D volume analysis offers several advantages, such as: I) Quantification of sizes and distribution of objects with complex geometry, which can only be obtained from 3D images, such as hierarchical microstructures; II) Determination of the connectivity of phases, particles, porosities or cracks, which cannot be determined from 2D measurements; III) Determination of local heterogeneities, such as the distribution of mechanical damage and/or corrosion, particle growth or crack propagation, which can only be clearly observed and quantified using 3D volumes; IV) Determination of the influence of boundary conditions regarding mechanical stress states on the materials performance.

POSTER PRESENTATION

**DEVELOPMENT AND ELECTROCHEMICAL BEHAVIOR
OF A VERSATILE SPECTROELECTROCHEMICAL CELL
FOR IN SITU EXPERIMENTS IN ELECTROCHEMISTRY**

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Electrochemical processes are involved in many fields of science and technology as they directly impact the sectors of energy (batteries, supercapacitors, etc.), health (electrochemical sensors), environment (water treatment, biomass conversion, etc.), among others. The rational development of materials for electrodes requires of a deep knowledge of the process that occurs in the electrode-solution interface. Thus, the use of spectroscopic techniques coupled to electrochemistry, are mandatory in order to get information about these processes. The development of versatile, cheap and easy to build spectroelectrochemical cells (SEC) contribute to the progress of the field of electrochemistry and its impact in science and technology. Therefore, in this work, we describe in detail the development of a SEC that can be used for several in situ techniques (FTIR, Raman, XAS, XRD and some using femtosecond lasers, among others) just by changing commercially available windows. Different to most of the contributions in the literature, we carefully analyze the electrochemical response of the SEC, which must be the same than the obtained in a electrochemical system. Otherwise, the spectroscopic data can only be connected with the electrochemical results obtained only in the SEC.

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POSTER PRESENTATION

**DEVELOPMENT OF TOOLS TO LABEL AND IMAGE
PROLIFERATIVE CELLS AND NEURONS USING X-RAY
IMAGING TECHNIQUES WITH SYNCHROTRON
RADIATION**

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Zebrafish (*Danio rerio*) have an exquisite ability to regenerate damaged neurons following spinal cord injury (SCI), while mammals fail to do so. Some steps of this process are known, such as the necessity of resident stem cells activation, but little is known about the cellular mechanisms these cells undergo or the molecular control of this process. To understand the cellular mechanisms of spinal cord (SC) regeneration in zebrafish, we are developing tools to label and image newborn neurons and axonal process using imaging techniques with Synchrotron Radiation (SR), such as X-ray Fluorescence Microscopy (XFM). We developed an immunohistochemistry (IHC) protocol with metal deposition that allows the identification of specific cell types using XFM. Performing BrdU incorporation and IHC protocols in zebrafish larvae post injury, we detected nickel deposition in the lesion site, an area with high concentration of proliferative cells. We also performed IHC against glial fibrillary acidic protein, a glial marker, in adult SC sections and XFM indicated intense nickel deposition in the SC. Another approach we are developing is the metal deposition upon the expression of APEX2, a peroxidase. We transfected HEK 293T cells with APEX2 followed by nickel deposition and detected high nickel signal in the transfected cells. These data show that this protocol generates a specific and XFM detectable signal in different cell types and approaches. This assay is a promising tool to label different cell types in a broad range of biological samples, giving them enough contrast to be imaged by XFM with high specificity. To deepen our understanding in the cellular process driving motoneuron (MN) regeneration, we are currently generating a zebrafish transgenic reporter line that drives APEX2 expression in motoneuron. This transgenic line will allow us to trace MN regeneration in the adult SC in a non-destructive and tridimensional manner using X-ray imaging techniques with SR.

POSTER PRESENTATION**FABRICATION AND CHARACTERIZATION OF SYSTEM
MAGNETOELECTRIC HETEROSTRUCTURES
 $\text{Pb}(\text{Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3/\text{CoFe}_2\text{O}_4$ GROWN BY RF-SPUTTERING**

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Over the past decade, nanostructured systems with magnetoelectric coupling have aroused great interest from researchers and high-tech industries, because these materials integrate two properties of high technological applicability, ferromagnetism and ferroelectricity. Through a systematic experimental approach, in the recent work, it was possible to investigate the fabrication by RF-Sputtering, and characterization of magnetoelectric composite thin films systems $\text{Pb}(\text{Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3/\text{CoFe}_2\text{O}_4$ (PZT / CFO) with connectivity 2-2 (laminate). In this work it was possible to verify the presence of the magnetodielectric and magnetoelectric coupling were correlated with the interaction via strain/stress between the phases. In this case, a tensile stress appeared on the CFO phase due to the presence of the PZT phase, resulting in the improvement of the magnetic properties when applied external magnetic field perpendicular to the film surface. On the other hand, it was seen that the magnetodielectric coupling arises in the maximum dielectric dispersion frequency spectrum of the heterostructure also influenced by compressive stress on the ferroelectric phase. Thus, advances on the analysis of the influence of strain on the magnetoelectric properties of this system will be expected by the X-ray scattering technique with coherent light (in the presence of external electric field and/or magnetic field).

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ORAL PRESENTATION

INTERACTION OF SUNSCREENS WITH SYNCHROTRON RADIATION: PHOTODISSOCIATION OF THE PABA MOLECULE FOLLOWING CORE IONIZATION

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The interaction of sunscreens with high energy photons can be uniquely explored with tunable synchrotron radiation. It constitutes a powerful broad spectrum light source that can probe molecules at the valence and inner shell levels [1]. We have studied the interaction of the first chemical sunscreen that was commercially available, the 4-aminobenzoic acid (paba), with synchrotron radiation employing time of flight mass spectrometry. The study was conducted at the Brazilian Synchrotron Facility, using high purity photons of the TGM and SGM beam-lines [2,3]. Photoabsorption spectra were obtained at the C 1s, N 1s and O 1s edges and the mass spectra were obtained around the edges. The results point out to a complex fragmentation patterns following core-level excitation in contrast with low fragmentation observed in low energy excitation and valence ionization. For instance, site-selective fragmentation mechanisms were observed at the O 1s resonances. The photoabsorption spectrum around the O 1s edge presents two resonances with energy spacing of 3 eV, 530 eV and 533 eV, reflecting the different chemical environments of the oxygen atoms. Theoretical calculations are in progress to assign the main resonances of the photoabsorption spectrum.

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POSTER PRESENTATION

INVESTIGATION OF INTERACTION OF IRON MAGNETIC NANOPARTICLES IN TOMATOES SEED TREATMENT

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The use of nanotechnology in agriculture can revolutionize food production techniques by leveraging the productive sector [1], but it still raises questions about the potential effects on the environment and human health, typically related to translocation to food sources [2]. This study presents unpublished results regarding the effect of magnetic iron oxide nanoparticles (MNP) applied to seed treatment. MNP were prepared by co-precipitation method and subsequently functionalized with 3-aminopropyltriethoxysilane (APTES) and coated with polycaprolactone (PCL). The MNP were characterized by thermogravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM) and photon correlation spectroscopy (DLS) techniques. The results confirm the functionalization and coating of the MNP, which presented an average size in the 9-11 nm range evaluated by TEM. Similar results were obtained by DLS study for unfunctionalized-MNP and APTES-MNP, with an average size of 4 ± 0.5 nm, while PCL coated MNP showed an increase in size (76 ± 0.26 nm) which is probably due to aggregation and crosslinking. Tomato seeds (*Solanum Lycopersicum*) were treated with MNP at different concentrations and seed cover, toxicity was evaluated in terms of germination index and content of mucilage. The penetration and translocation of MNP was probed by electron microscopy and X-ray fluorescence microanalyses. We concluded that MNPs are adsorbed on the surface of the seeds, but did not penetrate them. The study also revealed that the MNP did not interfere in the germination and growth of tomato seedlings because there is no MNP translocation to shoots or roots. Therefore, the results show that MNP may be a safe nanotechnology with strong potential for application in agriculture. In principles, MNP can carry active substances and delivery them in specific targets, therefore contributing to pest control and/or plant nutrition.

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ORAL PRESENTATION

**INVESTIGATION OF INTERACTION OF ZnO
NANOPARTICLES IN *PHASEOLUS VULGARIS* SEED
COAT USING SYNCHROTRON MICROANALYSIS**

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Nanoecotoxicology is a growing field of science driven by the extensive use of nanomaterials in commercial products and agriculture. Their use in agriculture is to become a reality through fertilizers and pesticides. On the other hand, little is known about possible collateral effects that of nanomaterials and therefore they may pose a threat to human health and the environment. To shed light on these topics, one must better understand the mechanisms of interaction between nanoparticles (NP) and living beings. Seed treatment is a common practice that provides micronutrients and protection, enhancing germination and plant development. This study investigated the interaction of zinc sources (ZnSO₄ and ZnO 40 nm) in common bean seed coat after germination. The common bean seed treatments and synchrotron analysis were performed at the ID21 beamline in European Synchrotron Radiation Facility (ESRF), Center for Nuclear Energy in Agriculture (CENA-USP) and XRF beamline in the Brazilian Synchrotron Light Laboratory (LNLS). Scanning electron microscopy (SEM) images showed ZnO nanoparticles clusters in the edge of the hilum and around the micropyle. X-ray fluorescence microanalysis (μ -XRF) for seed coat samples treated with both sources revealed a concentration gradient of Zn from the middle layers towards the cuticle (most external layer) and parenchyma (most internal). X-ray absorption spectroscopy (XAS) showed that the Zn incorporated by the seed coat was biotransformed into a mixture of Zn-citrate and Zn-malate, regardless of the Zn source used in priming. Attenuated total reflectance Fourier transform infrared (ATR-FTIR) and FTIR microscopy (μ -FTIR) analysis allowed to identify differences mainly in carbohydrate and protein groups in the layers of common bean seed coat samples exposed to ZnO 40 nm and ZnSO₄ at 100 and 1000 mg.L⁻¹. Altogether, these results presented an overview of the interaction of the tested Zn sources with the seed coat after five days of germination.

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ORAL PRESENTATION

**LIMITATIONS AND COMPLEMENTARITY OF
SPECTROSCOPIC TECHNIQUES ON GEOCHEMICAL
CHARACTERIZATIONS: THE CASE OF PERMIAN
MESOSAURIDS**Júlia D'Oliveira¹, Natália Cantuária¹, Mírian L. A. F. Pacheco^{1,2*}¹*Departamento de Biologia, UFSCar-Sorocaba, Brazil;*²*Departamento de Física Nuclear (USP), Brazil.***forancelli.ufscar@gmail.com*

Mesosaurids were Permian aquatic reptiles that sometimes display histological details preserved on its bones. Spectroscopic analysis on these fossils can produce data that help to understand the paleoenvironmental conditions in which these organisms were preserved. We test the complementary parameters and limitations between energy-dispersive spectroscopy (EDS) and synchrotron micro-X-ray fluorescence (micro XRF) on the investigation of labile tissues in the bone marrow of mesosaurids. Fossils were preserved in carbonate-enriched rocks rescued from outcrops of Angatuba, SP (ca. 290 Ma). We verified dark spots (organic matter?) filling the bone marrow channels in a fossilized vertebra. EDS were better for the detection of lighter elements, as C (intensities at 5 and 15 keV), as well as other heavier ones correlated to the preservation of organic matter in the dark spots: S (intensities at 5 and 15 keV) and P (higher intensities at 20 keV). Ca was detected in bone marrow channels also by EDS (higher intensities at 5 keV), though at lower intensities when compared to the bone matrix. The difference of relative intensities of Ca in bone and marrow channels might have obscured the signal intensity of this element in micro XRF analysis, highlighting the intensities on bone matrix. Strontium was at levels below the sensitivity range of EDS. We were able to detect this trace element on the bones only by micro XRF. It is known that Sr substitutes Ca ions in bone apatite, and it is incorporated during life and diagenesis as well. Here, it is also present on the marrow, possibly replacing Ca on carbonate infillings. The present contribution corroborates the importance of testing parameters and limitations among analytical techniques. We reinforce that synchrotron light has been shown to be more powerful in analysis that seek sensitivity in order to reveal undisclosed data about fossils and, consequently, extinct biospheres.

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POSTER PRESENTATION

LOCAL STUDY OF THE FORMATION THE ZrO_6 OCTAHEDRAL CLUSTER IN THE Tb AND Yb DOPED AND PURE BaZrO_3 MATRIX BY THE MICROWAVE ASSISTED HYDROTHERMAL METHODRafael U. Fassbender^{1*}, Verônica C. Teixeira², Mario L. Moreira¹¹Universidade Federal de Pelotas, Brazil; ² Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM), Brazil.

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Currently, several nanostructured materials of type ABO_3 where $\text{A} = \text{Ba}^{2+}$; Sr^{2+} ; Pb^{2+} and $\text{B} = \text{Zr}^{4+}$; Ti^{4+} ; Ca^{4+} are being studied. These compounds have many properties of scientific interest and can be applied in the field of catalysis, solar cells, the construction of capacitors and superconductors and also as scintillating materials. These perovskite type compounds present energy band with a gap of about 4 to 6 eV. The diversity of properties presented by this class of materials motivates the investigation of pure and doped matrices such as Barium Zirconate BaZrO_3 (BZO). In general, the BZO has a cubic crystalline structure, formed by a unit cell composed of two clusters (ZrO_6) and (BaO_{12}). The ZrO_6 cluster is more sensitive to local distortions that can modify and/or generate new optical and electronic properties. In this work, we present the first study on the changes in the ZrO_6 cluster environment as a result of co-doping via the Microwave Assisted Hydrothermal (MAH) method, replacing Ba and Zr by rare earths (RE) in the BZO matrix. The dopants used were Terbium (Tb^{3+}) and Ytterbium (Yb^{3+}) inserted in the matrix under follow stoichiometry $\text{Ba}_{1-x}\text{Tb}_x\text{Zr}_{1-y}\text{Yb}_y\text{O}_3$, with ($x=1\%$; $y=1,2,4$ and 8%) in solid solution. For the structural investigation of the samples were impregnated the techniques of, X-ray Diffraction and Raman Spectroscopy. A complementary technique of X-ray absorption near-edge structure (XANES) which is able to identify chemical environment around the atom absorbed. XANES also informs characteristics of the crystalline fields from observation of e_g and t_{2g} group orbitals as well it's distortions caused by the presence of the dopants in the BZO matrix. As well as the Raman Spectroscopy allows to investigate the polarized states of the symmetrical and asymmetric vibration of the crystalline structure. And the experimental results of the first order vibrational modes allow us to simulate computationally the local and global distortions of the crystal structure

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POSTER PRESENTATION

**MAGNETIC-LUMINESCENT NANOPLATFORM:
TOXICITY ASSESSMENT AND SYNCHROTRON X-RAY
FLUORESCENCE MICROSCOPY
IN ZEBRAFISH EMBRYOS**

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Multifunctional nanomaterials co-assembling optical and magnetic properties in single-entity nanostructures have high potential in several biotechnological and environmental applications. Indeed, a great number of studies concerning the technological applications of those nanomaterials are published every year. However, toxicity assessment is mandatory towards safe innovations in nanotechnology. In this sense, we evaluated the toxicity of a nanoplatform with integrated photoluminescence and magnetic properties ($\text{Fe}_3\text{O}_4@\text{SiO}_2$ GdOF: Ce^{3+} , Tb^{3+}). The nanoplatform was synthesized combining magnetic iron oxide nanoparticles with Ce^{3+} - and Tb^{3+} -doped with GdOF luminophore and concurrent capping with chitosan biopolymer. In vivo toxicity assessment of the nanoplatform was evaluated using Zebrafish embryos; it was not observed acute toxicity to the zebrafish embryos up to 100 mg.L^{-1} , suggesting in vivo biocompatibility. Biodistribution of nanoparticles using synchrotron X-ray fluorescence (SXRF) imaging was reported. It was observed that the distribution of Gd and Fe had occurred in a roughly constant relative ratio in the whole embryo. Furthermore, it was revealed by the elements mapping data a predominant localization of Gd and Fe in the gastrointestinal tract. This result demonstrated that the uptake of the particles by embryos was mostly through oral exposure rather than the dermal pathway. Finally, these results demonstrated the great potential of SXRF technique associated with nanotoxicity testing for monitoring the internalization and biological transformation of nanoparticles in the emerging field of biological and environmental nanosciences.

POSTER PRESENTATION

MODULATING THE MAGNETIC PROPERTIES OF IRON OXIDE NANOPARTICLES WITH MOLECULAR COATING

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Metal Oxide magnetic nanoparticles (NPs), especially magnetic spinel ferrites nanoparticles are interesting materials for a wide variety of applications. Therefore, the understanding and the control of their magnetic properties are attractive issues. We report the magnetic properties of Fe₃O₄ nanoparticles prepared by thermal decomposition method with oleic acid as the surfactant and having different coating via ligand exchange (amino acid propyl phosphonic, APPA, oleylamine and dopamine) [1]. The nanoparticles have an average size of 4.4 nm. The *M(H)* measurements at room temperature show a superparamagnetic behavior for all samples and suggest a spin ordering of the Fe₃O₄@APPA nanoparticles. We explained the presence of magnetic interactions through the mean field approximation, and considering the surface and magnetic relaxation effects to understand the magnetic behavior. AC magnetic susceptibility measurements suggest a stronger magnetic interaction and spin glass behavior in bare and Fe₃O₄@OAM nanoparticles as expected, and a weaker behavior for all other coatings. The surface contribution to the magnetic properties is evaluated by X-ray absorption spectroscopy (XAS) and X-ray circular magnetic dichroism (XMCD) measurements. Preliminary results indicate different Fe cation occupancy of octahedral and tetrahedral sites and magnetic moments extent for each NP. This study opens new perspectives in the use of molecular coating for tuning the magnetic properties of nanoparticles [2].

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POSTER PRESENTATION

**MONITORING OF Li-AIR BATTERIES REACTION
PRODUCTS BY OPERANDO SYNCHROTRON XRD**

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Li-air batteries are promising substitutes of Li-ion batteries. They can provide ten times more electrical capacity by the reaction of metallic lithium and atmospheric O₂. The main reaction product is Li₂O₂, but side reaction can take place if electrode surface and atmosphere conditions are not rigorous controlled. In this way, unique cell design must be developed. In this work, two different cells were designed for in situ and operando analysis of Li-air batteries by synchrotron radiation X-ray diffractometry. The cells were 3D-printed in stainless-steel by additive manufacturing by direct metal laser sintering. The cells were assembled in a dry box with argon atmosphere and then transferred in hermetic containers. A potentiostat and gas valves were attached to the XRD equipment. The charge and discharge process were performed in purified synthetic air chambers, with air humidity below 5%. In both cells a stainless-steel mesh with 0.2 g.cm⁻² of carbon nanotubes was used as work electrode. The diffractograms indicated progressive formation and subsequently degradation of different amounts of LiOH and Li₂O₂ after a 16h cycle of discharge/charge. The formation of hydroxide can be justified by the presence of undesirable water in the atmosphere and in the electrolyte. Nevertheless, this technique can provide useful information about the performance of different cells, electrodes and electrolytes in Li-air batteries.

ORAL PRESENTATION**nanoCT APPLIED ON ENDODONTIC
TREATMENT AND IMPROVEMENT**

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This work aims to optimize the application of microCT in teeth seeking the best image quality from the acquisition to thresholding, arguing over the spatial resolution, contrast and noise [1]. For this, the resulting images of three different micro-tomography/nano devices were compared qualitatively and quantitatively, with different acquisition energy powers and arrangements, after the use of different image processing algorithms, as filters and thresholding methods. It was demonstrated that the simple use of the same acquisition parameters is not sufficient for the correct interpretation, comparison, and replication of the results obtained between different devices, requiring an standard approach. A positive relationship between the decrease of the beam voltage and the quality and amount of visualized structures was shown. The use of the Unsharp mask filter significantly improved the segmentation quality of the root canals, in addition to reducing their variance in relation to the raw image. The best images were provided by a semi-automatic thresholding method. However, more consistent results were shown using the automatic method developed by Otsu in three-dimensional space.

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POSTER PRESENTATION

**NiVCE LAYERED DOUBLE HYDROXIDE
NANOPARTICLES AS HIGH-PERFORMANCE
ELECTROCATALYST FOR OXYGEN EVOLUTION
REACTION**

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Production of clean energy in a sustainable way is of utmost importance in modern society, but reactions such as the oxygen evolution reaction (OER), well-known for its sluggishness and energy requirements, play a key role in fuel cells technology and conversion of energy. Thus, efficient water oxidation catalysts (WOCs) based on abundant elements are attracting lots of scientific and technological interest[1]. Recent advances in the design of heterogeneous WOCs show that the preparation of trimetallic double lamellar hydroxides (LDHs) is paving the way for high performance WOCs. In this sense, the objective of this work is to synthesize and characterize trimetallic NiVCE-LDH nanoparticles (NPs) based WOCs and evaluate their performance against OER. For this, the nanostructured LDH containing 5 mol% of Ce^{3+} and V^{3+} was prepared by sol-gel method and characterized by TEM, XRD, VC and EIS. The XRD results confirm the formation of LDH, characterized by the presence of the 003 reflection plane at low angle ($2\theta=10.4^\circ$). In addition, a higher structural disorder was observed in HRTEM images for NiVCE-LDH, probably due to the incorporation of Ce^{3+} ion. The electrocatalytic activity of NiVCE-LDH was revealed by VC and EIS, as the Nyquist diagram after 100 successive redox cycles showed two semicircles. The first high-frequency activated process was attributed to the $\text{Ni}^{2+}/\text{Ni}^{3+}$ process, while the second lower-frequency process was associated to the electron transfer process of the NPs catalyzed OER. Interestingly, the impedance of the second decreased significantly, indicating increased electrocatalytic activity as a function of the number of voltammetric cycles. The results obtained so far clearly indicate the potential of NiVCE-LDH as an electrocatalyst for OER. However, new NPs with different proportions need to be prepared and tested to achieve synergistically improved properties to modulate the binding energy of oxygen intermediates and lower OER overpotential.

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ORAL PRESENTATION

**ON-DEMAND ACTIVATION OF
ARTIFICIAL CERIUM-BASED PROENZYMES**

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CeO_(2-x) nanoparticles have emerged as a promising redox-like enzyme for biomedical applications. [1] Despite their potential benefits to treat various diseases caused by oxidative damage, they can be deleterious to healthy tissues. [2] Considering this, we proposed an *in situ* synthesis of CeO_(2-x) nanoparticles from Ce(OH)₃ dispersed in liquid crystal using H₂O₂ as trigger. In this context, Ce(OH)₃ nanoparticles could behave as a proenzyme-like system able to be converted to the active form only under oxidative stimulus. The reactions between H₂O₂ and Ce(OH)₃ were carried out at pH = 5.5 (buffered). The CeO_(2-x) nanoparticles started to be released from the liquid crystal medium just after H₂O₂ was added to the medium. The Ce(III) to Ce(IV) conversion was evidenced by X-ray absorption spectroscopy measurements. The CeO_(2-x) nanoparticles were evaluated by X-ray diffraction and transmission electron microscopy (TEM). The first technique confirmed the fluorite structure of CeO_(2-x). The analysis of the TEM images showed ultra-small nanoparticles. CeO_(2-x) nanoparticles showed superoxide dismutase-like activity in contrast to the inactive Ce(OH)₃ form. This is the first report in the literature [3] of nanoparticles as artificial proenzymes. The present study is a proof of concept that extends the frontiers of biomimetic chemistry and nanomedicine, providing a perspective on the future design of smart nanoparticles as functional artificial proenzymes that may be activated specifically at the site of action *in vivo*. Measurements at Carnauba beamline would allow us to investigate this *in vivo* activation into cell organelles, which is not possible with conventional imaging techniques.

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POSTER PRESENTATION

**PROTECTIVE EFFECT OF LOSARTAN IN
CORONARY ARTERIES OF RATS SUBMITTED
TO RADIOTHERAPY FOR BREAST CANCER.**

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Radiotherapy for breast cancer often involves some incidental exposure of the heart to ionizing radiation. Women with breast cancer who are treated with adjuvant radiation have a decreased risk of local recurrence but an increased risk of mortality from coronary artery disease (CAD). [1] The possible reason to occurrence of CAD after radiotherapy is the unfavorable remodelling of the coronary artery due the radiation injury. [2] Losartan is an angiotensin II type 1 receptor antagonist widely used for the treatment of hypertension as well as other cardiovascular diseases [3]. Previous studies showed the efficacy of losartan to correct the altered structure and endothelial dysfunction of resistance arteries from patients with essential hypertension [4], and also prevent the progression of coronary atherosclerosis in patients with significant atherosclerotic disease. [5] The present study aims to clarify how the radiotherapy for breast cancer affects the coronary artery and to determine if losartan can be used to minimize the side effects of the irradiation in this artery. Using LEXRF technique, at TwinMic beamline (Elettra Synchrotron), the effects of irradiation and the protective effects of Losartan were evaluated through the distributions maps of O, Na and Mg in the coronary arteries of the rats. CARNAÚBA beamline provides two separated experimental stations, one with sub-micrometer resolution and another with nanometer resolution, to cover various analysis techniques, including X-ray fluorescence. The all achromatic CARNAÚBA optics will covers continuously the energy range from 2 to 15 keV. [6] With this energy range, it will be possible to analyze K-lines of light elements, like phosphorus and sulfur, and also heavier elements, like calcium and zinc. The use of this beamline will provide both submicrometer spatial resolution and elemental mapping of important elements in coronary artery of hypertensive rats allowing analyze the tissue in a cellular level.

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POSTER PRESENTATION

**SURFACE FUNCTIONALIZATION EFFECTS
ON RARE-EARTH DOPED PERSISTENT
LUMINESCENT MATERIALS**Leonardo H. C. Francisco^{1*}, Maria C. F. C. Felinto¹, Hermi F. Brito²¹*Instituto de Pesquisas Energéticas e Nucleares (IPEN/USP), Universidade de São Paulo, Brazil;*²*Instituto de Química (IQ/USP), Universidade de São Paulo, Brazil.*

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Recently, research on persistent luminescent materials leans towards high-quality nanoparticles with specific requirements on design methodology, morphology and functionalization [1, 2]. In this work, rare earth doped strontium aluminate persistent phosphor (i.e. $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+/3+}$, Dy^{3+}) was prepared by the Pechini method and post-annealed on carbon monoxide reducing atmosphere. Furthermore, the prepared luminescent material was coated with 3-aminopropyltrimethoxysilane (APTMS) and β -diketonate europium complexes *via* microwave assisted synthesis, resulting in highly luminescent complex/silica coated particles. Structural analysis by X-ray powder diffraction of the pure strontium aluminate phosphors revealed a stable monoclinic phase which undergoes several structural changes after the functionalization and coating processes; while energy dispersive X-ray spectroscopy results support the expected surface alteration effects. Moreover, luminescence spectroscopy results acquired on the TGM beamline showcased the increasing intensity of the characteristic broad emission band assigned to $4f^65d^1 \rightarrow 4f^7(^8S_{7/2})$ transition of europium (II) compared to narrow emission bands, $^5D_0 \rightarrow ^7F_J$ ($J = 1, 2$ and 4) of non-reduced europium (III). Finally, all of the silica coated materials exhibited a distinguished spectral profile, suggesting an effective interaction between the inorganic host-matrix, the silica network and the β -diketonate complexes.

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POSTER PRESENTATION

SYNCHROTRON BASED INVESTIGATION OF CELL/DENDRITE STRUCTURES IN SOLAR GRADE SILICON

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The growing demand for silicon in photovoltaic applications triggered the development of alternative more economic routes to produce solar grade silicon [1-4]. The metallurgical route is a sequence of processes including unidirectional solidification, which can be used to remove most of metallic impurities as these segregate to the top of the silicon ingots. The pile-up of impurities in front of solidification interface can lead to the formation of cells and dendrites at the solid-liquid interface, which hinders the impurity segregation and ingot purification [5]. Therefore, understanding the morphology of the solid-liquid interface is essential to develop more efficient purification processes and better grain control of multicrystalline silicon. The solubility of most metallic impurities is very small in solid silicon, making the variations in their impurity concentrations too small to be detected by chemical etching or by backscattered electron contrast observed in the electron microscope. Consequently, dendrite structures that existed during solidification require more sensitive techniques to be detected. X-ray fluorescence microscopy (μ -XRF) has been frequently used to detect iron and others metallic impurities in a silicon matrix [6-9] to investigate their effects on the electrical performance of solar cells. In the present work, the distribution of concentration of impurities will be measured by μ -XRF and an attempt will be made to identify the existence of cell/dendrite structures in samples extracted from directionally solidified silicon ingots.

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POSTER PRESENTATION**SYNTHESIS OF NANOWIRES FOR APPLICATION IN THE
REDUCTION REACTION OF CARBON DIOXIDE**

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New technologies that mimic photosynthesis, a complex reaction that occurs in living organisms, are vital in today's society since they bring the possibility of converting carbon dioxide into energy-rich molecules such as methane and ethylene. The development of new methodologies for control and rational design of materials that can be used as catalysts must be carried out keeping in mind the challenge of promoting the highest selectivity and conversion efficiency possible. It is common to observe multiple reactions happening simultaneously in electrochemical systems, so in order to understand in detail the processes that occur in the system it is necessary to use rapid monitoring techniques that, combined with the electrochemical information, lead to a deeper understanding of the system. This project proposes to establish relationships between new materials for catalytic purposes with information about the molecular kinetic chemistry involved and, consequently, to selective obtain molecules of interest through the knowledge of the mechanistic aspects. The electrocatalytic material to be studied proposed in this work consists of multilayer copper/copper oxide nanowires, in which the conductive and electrocatalytic characteristics can be combined with synergistic effects. The oscillatory electrodeposition reaction will be used in order to perform the growth of the nanowires on anodized alumina oxide substrate containing self-organized nanopores. On-line mass spectrometry coupled to the electrochemical cell will be used for the monitoring of volatile reaction intermediates, thus evaluating the performance of the proposed catalyst.

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POSTER PRESENTATION

**THE LOCAL ATOMIC ENVIRONMENT INSIDE
A SHEAR BAND IN METALLIC GLASSES**

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The strain localization in Metallic Glasses (MGs) is due to the formation of shear transformation zones (STZ) that evolve into shear bands (SB). Therefore, a great deal of effort has been made to try to understand the relationship between short range order and strain localization. In this work we studied the deformation of a $\text{Cu}_{45}\text{Zr}_{45}\text{Al}_{10}$ metallic glass in compression using molecular dynamics. A $\text{Cu}_{45}\text{Zr}_{45}\text{Al}_{10}$ metallic glass model (50 nm x 50 nm x 50 nm in size) was produced by the quenching method, and then uniaxially compressed at a strain rate of 108 s^{-1} for up to 20% strain. All the simulations were performed using the Large-scale Atomic Molecular Massively Parallel Simulator (LAMMPS) software, and a time step of 1 fs. The Voronoi analyses indicate that the most significant structural change as a function of compression strain was a decrease in the amount of Cu-centered $\langle 0, 0, 12, 0, 0 \rangle$ icosahedra. In order to complement this theoretical study, we will perform nanobeam X-ray absorption experiments, EXAFS and XANES, which will be used to analyse the structural changes inside a shear band, and compare them to those obtained in the molecular dynamics simulations.



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