

EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

Proposal to the ISOLDE and Neutron Time-of-Flight Committee

Local investigation of impurities in wide band gap nanostructured oxides with radioactive probes.

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Abstract

Extending the work started with LOI144, we intend to apply radioactive ion implantation techniques to measure hyperfine interactions at implanted ions (acting as dopants) in wide band gap metal oxides nanostructured samples in order to investigate 1) the doping effect on the electronic structure of the oxide and 2) the method of preparation of nanostructured and hierarchical structured oxides. Perturbed Angular Correlations will be used, to locally investigate the doped oxide properties regarding crystallinity, defects, electron density and possible magnetic interactions. We will use the several radioactive ions available at ISOLDE to vary the charge state of the impurity, which will be the PAC probes. With specific experiments, we aim to advance in fundamental research, which can be used to optimize manufacture procedures of devices while strengthening the collaboration between synergetic institutes.

Requested shifts: 9 shifts, (split into 3 runs over 3 years)



Introduction and motivation: Wide band-gap semiconductor oxides as nanostructured or hierarchically structured materials are good candidates for important technological applications [1,2]. The doping with other elements than the native cationic metal can enhance certain properties of these oxides and turn those better materials for specific applications or make them suitable for new ones [3-5]. When a dopant atom replaces the cationic metal in an oxide host, even at very small concentrations, the properties of the host can be dramatically affected producing changes in electronic and transport properties, and altering phase transition temperatures. The goal of the doping is to improve the functional performance of the oxide host. The role of the dopant and its influence in the host oxide is desirable to be understood so that a general model of the connection between dopant–oxide pairing and the functional performance must be developed. Methods of characterization need to answer some important questions. Is the host oxide homogeneously doped? Can one assure that the dopant did not produce small oxide clusters in the oxide host? Is the presence of other phases ruled out? How is the electronic structure in the neighbourhood of the dopant? Are defects, such as oxygen vacancies created by the dopant? However, the lack of quantitative experimental data on the local neighbourhood of the dopant, which can give information about the local structure and the electronic structure in this region, has prevented the formulation of a general theoretical description of the doping phenomenon in oxides. New experimental data of these categories are, therefore, necessary and techniques sensitive enough to identify them from different regions within samples on an atomic scale are welcome. In turn, semiconductor oxides with hierarchical morphologies have been intensively studied with great interest because both fundamental science and potential applications in diverse technological fields [6-8]. Functional materials with such structures offer a very large area of the active surface, which is fundamental for many applications such as gas sensors, for instance. The fabrication of materials with hierarchical structures for new application need to be achieved by low cost and without any effect on the environment. Therefore, to meet these requirements, preparation of hierarchically structured materials requires characterization techniques capable to distinguish different regions inside the material at the atomic scale.

Objectives: The purpose of this project is to investigate the doping mechanism in some selected nanostructured oxides (listed below) using experimental techniques of characterization sensitive to atomic distances as well as first-principles calculations based on density functional theory (DFT). Local modification produced by dopants implanted into samples will be tracked by perturbed angular correlation (PAC) spectroscopy using both the gamma-gamma (γ -PAC) and electron-gamma angular correlation (ϵ^- -PAC) spectroscopies by measuring hyperfine interactions at the implanted nuclei. These nuclear techniques are able to measure both the nuclear electric quadrupole and the nuclear magnetic dipole interactions. Because hyperfine interactions have the short range of the atomic distances, it will be possible to investigate the neighborhood of the dopants (which are the probe atoms) and the local effect of the doping. As ISOLDE offers a variety of radioactive nuclei, we can choose ions with different valences than that of the native cations and investigate the influence of the excess or lack of electrons. In order to obtain a detailed picture of the doping effect, PAC results will be complemented by EXAFS measurements to study the oxidation state, coordination chemistry, and the distances, coordination number and species of the atoms immediately surrounding a selected element into samples. First-principles calculations using full-potential linearized augmented plane waves method (FP-LAPW) based on the DFT will be performed to simulate the doping in the host oxides to interpret mainly the experimental hyperfine interactions results. Our

goal in the present project is to provide a description of the doping effects at an atomic level. In parallel, the mechanism of fabrication of hierarchical structures in microtubes of these oxides will also be investigated. It is important to emphasize that the results of this project can either contribute to the formulation of a general theory about the doping mechanism in semiconductor oxides as well as produce new materials for technological applications.

Justification and Methodology: Results of LOI144 showed that PAC spectroscopy can be successfully used to characterize doped oxides[9-11] and that the technique is suitable to a precise and detailed characterization of nanoscopic materials mainly because it can obtain information at different regions inside the whole sample, differently from most synchrotron techniques. The idea is to dope samples with different probe ions having different valences (the ion of ^{111m}Cd has valence +2, the ion of ^{117}In has valence +3, ^{181}Ta has valence +5, etc.) and investigate the local effect of doping on the charge density, oxygen vacancy occurrence, and electronic structure by measuring the electric field gradient. In addition, intrinsic properties of samples such as crystal phase transition and magnetism (by measuring the magnetic hyperfine field) will be investigated too. The method is complemented by first-principles calculations. This methodology has been used with success by our team [10-14]. PAC spectroscopy will also be used to follow the fabrication of 3D-hierarchical-structured microtube samples of the oxides by thermal oxidation as it successfully helped to unveil the mechanism of nanowire growth in ZnO microtubes[15]. PAC measurements will be carried out at different temperatures to follow the behaviour of the observed parameters as well as dynamic process particularly measured by ϵ^- -PAC.

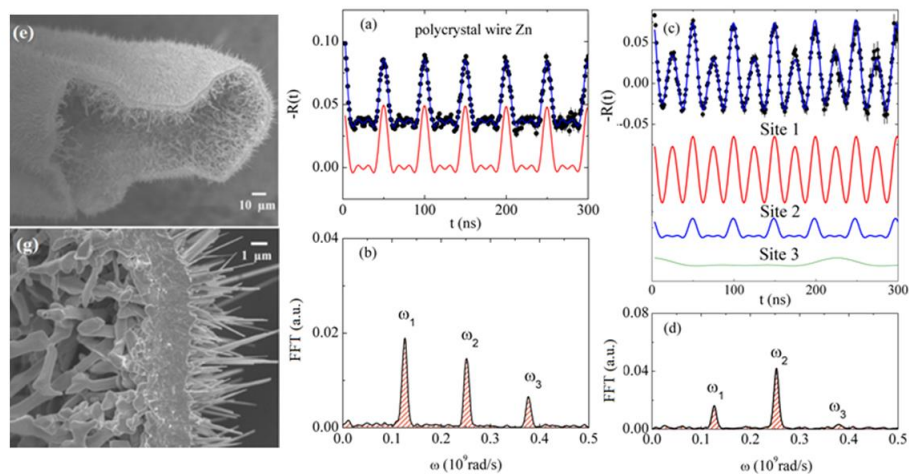


Figure1: TEM images of hierarchical structured ZnO (left) and PAC spectra for pure Zn before and after thermal oxidation. From Ref. 15.

Thin film samples will be prepared by electron beam evaporation in IPEN and by magnetron sputtering in the Engineering College at São Paulo University, where they will be further characterized by x-ray diffractometry (XRD) and scanning electron microscopy (SEM). Hierarchical structured microtube samples will be prepared at IPEN in São Paulo, and UFABC in Santo André by thermal oxidation process and also characterized by XRD and SEM as well as by resistivity measurements. Thin film samples will be characterized by Rutherford backscattering (RBS) and magnetization measurements at IFUSP. EXAFS measurements will be carried out in the synchrotron accelerator in Campinas, SP, Brazil. The team has develop the thermal oxidation process to fabricate hierarchical microtubes of oxides by oxidating microwires of the cation metal[15]. The method is simple and consist in

oxidize microwires of the cation element by heating them in air above their respective melting temperature. For some metals it is necessary to apply a low current to the wire during the oxidation process. That was necessary for the fabrication of microtube of TiO_2 as the preliminary test indicated.

Proposed studies: Vanadium, Titanium, Indium and Gallium oxides: Vanadium oxides exhibits four common oxidation states +5, +4, +3, and +2, respectively, which can be interconverted allowing the fabrication of a vanadium flow battery [16]. V_2O_5 crystallizes in an orthorhombic structure with layers of VO_5 square pyramids sharing edges and corners. These layers are weakly bond along the c-axis and the space between them permits the accommodation of guest ions. This property along with the reversibility of valences of vanadium cations, high abundance, and theoretical high capacity[7] makes this material an attractive candidate for the intercalation of Li-ions producing the Lithium ion batteries (LIB). New electrode materials with higher lithium storage capacity are needed to produce high-performance LIB. VO_2 is a promising material for a wide variety of technological applications [17]. Unexpectedly insulating at room temperature, VO_2 undergoes a sharp metal-to-insulator transition (MIT) at 341 K along with a structural phase transition from monoclinic to the rutile structure. The origin of the transition is not a consensus yet prevailing the scenario described as Peierls assisted orbital selective Mott transition [18]. Pure V_2O_3 is a paramagnetic metal at room temperature with a first-order transition to an antiferromagnetic insulating phase occurring near 160 K accompanied by a transition from rhombohedral (corundum) to monoclinic structure[19]. The MIT in transition metal oxides is characterized by a change in the resistivity along with structural and even magnetic ordering transitions, which may lead to revolutionary applications. However, these phenomena are still a challenge to a theoretical description[20]. Nanostructured TiO_2 , which crystallizes in two main phases with the rutile and anatase structures and is a wide band gap n-type semiconductor, has become an important material for environmental and energy applications [21-23]. TiO_2 film is employed as a substrate in the next-generation of solar cells, assisting in capturing electrons from an excited sensitizer or quantum dot and transporting the electrons to the collecting electrode surface[21]. High-conversion performance is achieved when the electron diffusion length is much greater than the TiO_2 film thickness. The enhancement of the electron diffusion length by reducing the dimensionality of the TiO_2 photoanode has been successfully demonstrated by means of vertically aligned TiO_2 nanotubes or nanowires. Hierarchical architectures combining efficient light trapping and high surface area are used for dye absorption too[8]. In_2O_3 (with a cubic bixbyite structure) and Ga_2O_3 have received attention as a new class of wide-band-gap semiconductors. Ga_2O_3 , a relatively unexplored material shows polymorphism with α -, β -, γ -, δ -, and ϵ - phases [24]. These phases are not only different in their space group but also in the coordination number of Ga atoms. Very recently, the research is focusing on single-crystalline oxide layers with low defect densities and semiconducting behaviour [25]. The β - Ga_2O_3 (having monoclinic structure with the same number of two inequivalent sites with 4 and 6 coordination) is the most attractive representative of this class of materials due to the large optical band-gap and high breakdown field promising applications in transparent electronics [26]. Alloying Ga_2O_3 with In_2O_3 offers an opportunity to tailor the band gap and other properties, greatly increasing the range of potential applications, enabling electronic and optical confinement. The two oxides have very different ground-state crystal structures and it is not clear which structure the alloys would assume and how this structure would affect stability and

electronic properties. Theoretical calculations in $(\text{In}_x\text{Ga}_{1-x})_2\text{O}_3$ alloys show that for x up to 50% the structure is monoclinic and bixbyite for higher In concentrations[27]. PAC can be used to experimentally verify the structure of these alloys since the quadrupole frequencies and asymmetry parameters for pure compounds are well known.

Justification for each radioactive ion: $^{111}\text{In}(^{111}\text{Cd})$ will be used to simulate doping with +2 valence impurity. $^{117}\text{Cd}(^{117}\text{In})$ will be used to simulate doping with +2 valence impurity. $^{111m}\text{Cd}/^{111}\text{Cd}$ will be used to carry out PAC measurements on the implanted element without element transmutation ($^{111m}\text{Cd}/^{111}\text{Cd}$) to compare with results obtained with $^{111}\text{In}(^{111}\text{Cd})$. Only at CERN-ISOLDE it will be possible to perform this investigation on semiconductor oxides using different PAC probes. Our priority of using the CERN facilities are for the following probe nuclei: ($^{111m}\text{Cd}/^{111}\text{Cd}$), $^{111}\text{In}(^{111}\text{Cd})$, and $^{117}\text{Cd}(^{117}\text{In})$ probes and subsequent PAC measurements. CERN-ISOLDE measurements will complement PAC measurements from IPEN where $^{181}\text{Hf}(^{181}\text{Ta})$ can be produced in the IEA-R1 research reactor.

Summary of requested shifts:

We estimate the total amount of ISOLDE beam time needed to accomplish the above-described tasks to be 9 shifts, distributed according to table I:

Table I: Beam time request

Required isotope	Implanted beam	PAC experiment	Intensity [at/ μC]	Target / Ion source	Comments	n° of shifts
^{111m}Cd	^{111m}Cd	$\gamma-\gamma$	10^8	molten Sn, plasma		3
^{117}Cd	^{117}Ag	$\gamma-\gamma$	10^8	UC ₂ , RILIS (Ag) UC ₂ – only used with surface ionization (In)	Nb or Ta ion source cavity to decrease In surface ionization contamination for Ag. Any UC2 target using W or Ta transfer line can be used for In.	3
^{111}In	^{111}Cd	$\gamma-\gamma$	10^8			3

All beam times consist of collections to be measured off-line and can in this way be easily shared with other users, particularly the case of the ^{111m}Cd beam time, where collections should run day and night with a period of about 4-5 hours between collections that usually last for 15-30 min. There are actually four PAC setups co-shared during beam times and the samples can be implanted on the same collective sample holder used with other users which are also doing PAC experiments. For these PAC experiments, the number of implanted atoms per sample range from $5 \cdot 10^8$ up to 10^{11} , depending on half-lives, coincidence efficiency and on the fluence limit for proper recovery of the implantation damage. All isotopes will be collected in the general-purpose implantation chambers at GLM and/or GHM new collection point at the ISOLDE hall, building 170. All $\gamma-\gamma$ PAC and ε^- -PAC measurements will be performed off-line, outside the ISOLDE hall, in the new Solid State Laboratory in building 508. Several furnace systems exist already at ISOLDE for annealing treatments under vacuum or gas flow at atmospheric pressure at the new SSP lab.

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- Appendix

DESCRIPTION OF THE PROPOSED EXPERIMENT

The experimental setup comprises: *(name the fixed-ISOLDE installations, as well as flexible elements of the experiment)*

Part of the Choose an item.	Availability	Design and manufacturing
SSP-GLM chamber	<input checked="" type="checkbox"/> Existing	<input checked="" type="checkbox"/> To be used without any modification
Existing equipment on the solid state labs in building 508	<input checked="" type="checkbox"/> Existing	<input checked="" type="checkbox"/> To be used without any modification <input type="checkbox"/> To be modified
- 6 detector PAC standard setups - annealing furnaces - glove boxes	<input type="checkbox"/> New	<input type="checkbox"/> Standard equipment supplied by a manufacturer <input type="checkbox"/> CERN/collaboration responsible for the design and/or manufacturing

HAZARDS GENERATED BY THE EXPERIMENT

(if using fixed installation) Hazards named in the document relevant for the fixed SSP-GLM chamber and building 508 installations.

Additional hazards:

Hazards	SSP-GLM	Building 508	[Part 3 of the experiment/equipment]
Thermodynamic and fluidic			
Pressure	[pressure][Bar], [volume][l]		
Vacuum	10-6 mbar at SSP chamber 10 during collections		
Temperature	295 K, room temperature collections		
Heat transfer	-		
Thermal properties of materials	-		
Cryogenic fluid		Liquid nitrogen, 1 Bar, few litres used during the PAC measurements on appropriate dewar	
Electrical and electromagnetic			
Electricity	[voltage] [V], [current][A]		
Static electricity			
Magnetic field	[magnetic field] [T]		
Batteries	<input type="checkbox"/>		
Capacitors	<input type="checkbox"/>		
Ionizing radiation			
Target material	[material]		
Beam particle type (e, p, ions, etc)			

Beam intensity			
Beam energy			
Cooling liquids	[liquid]		
Gases	[gas]		
Calibration sources:	<input type="checkbox"/>		
• Open source	<input checked="" type="checkbox"/> Produced at ISOLDE 111mCd (48m) 117Cd(2.5h)	Sources to be measured at 508	
• Sealed source	<input checked="" type="checkbox"/>	22Na sources provided by RP services at CERN, used at 508	
• Isotope	111mCd (48m) 117Cd(2.5h)		
• Activity	111mCd (48m) < 3 e 7 Bq 117Cd(2.5h) < 8 e 6 Bq		
Use of activated material:	none		
• Description	<input type="checkbox"/>		
• Dose rate on contact and in 10 cm distance	[dose][mSV]		
• Isotope			
• Activity			
Non-ionizing radiation			
Laser	none		
UV light	none		
Microwaves (300MHz-30 GHz)	none		
Radiofrequency (1-300MHz)	none		
Chemical			
Toxic	[chemical agent], [quantity]		
Harmful		Acetone (ICSC: 0087), ethanol (ICSC: 0044) and methanol (ICSC: 0057). Less than few centilitres per chemical, used on cleaning samples on ventilated fume hood on building 508. The respective ICSC forms have been printed and will be handled during preparation and experiments.	
CMR (carcinogens, mutagens and substances toxic to reproduction)	[chemical agent], [quantity]		
Corrosive	[chemical agent], [quantity]		
Irritant	[chemical agent], [quantity]		
Flammable	[chemical agent], [quantity]		
Oxidizing	[chemical agent], [quantity]		
Explosiveness	[chemical agent], [quantity]		
Asphyxiant	[chemical agent], [quantity]		
Dangerous for the environment	[chemical agent], [quantity]		
Mechanical			
Physical impact or mechanical energy (moving parts)	[none]		
Mechanical properties (Sharp, rough, slippery)	[none]		
Vibration	[none]		
Vehicles and Means of	[none]		

Transport			
Noise			
Frequency	[frequency],[Hz] Ambient noise at the ISOLDE Hall, building 170		
Intensity	Ambient noise at the ISOLDE Hall, building 170		
Physical			
Confined spaces	[none]		
High workplaces	[none]		
Access to high workplaces	[none]		
Obstructions in passageways	[none]		
Manual handling	All samples and sample holders are manually handled either by long tweezers to insert and extract the sample holder into and out of the SSP implantation chamber at GLM, or when manipulating the samples and sample holders inside glove boxes or fume houses on building 508 r-007	All samples and sample holders are manually handled either by long tweezers to insert and extract the sample holder into and out of the SSP implantation chamber at GLM, or when manipulating the samples and sample holders inside glove boxes or fume houses on building 508 r-007	
Poor ergonomics	[none]		

0.1 Hazard identification

3.2 Average electrical power requirements (excluding fixed ISOLDE-installation mentioned above):
(make a rough estimate of the total power consumption of the additional equipment used in the experiment)

There is no additional equipment with relevant power consumption on these small-scale experiments.