

ASNAEM-2021 Workshop

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Book of Abstracts

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XRD, USAXS, SAXS and WAXS Investigations of PZN-4.5PT nanoparticles thin Films

Author: Rémi Ndioukane¹

Co-authors: Abdoul Kadri Diallo¹; Diouma KOBOR¹; Jan Ilavsky²; Laurent Lebrun³; Moussa Touré¹; Ndèye Coumba Yandé Fall¹; Tabbetha Dobbins⁴

¹ LCPM/UASZ

² Advanced Photon Source, Argonne National Laboratory, Chicago USA

³ LGEF/INSA-Lyon, France

⁴ Rowan University

Corresponding Authors: r.ndioukane1532@zig.univ.sn, n.fall1530@zig.univ.sn, diallokhadreabdoul@yahoo.fr, laurent.lebrun@insa-lyon.fr, dobbins@rowan.edu, ilavsky@anl.gov, dkobor@univ-zig.sn, touremoussa1986@gmail.com

The $\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-}4.5\text{PbTiO}_3$ (PZN-4.5PT) single crystals showed very large ferroelectric and piezoelectric properties compared to traditional ferroelectric ceramics (BaTiO_3 and PZT) used presently as active material in medical imaging, detection and sonars. However, despite these excellent properties, the greatest difficulty to use PZN-4.5PT single crystals on electronic devices is to achieve them in thin layers form because of their incongruent melting property. To overcome this difficulty, we deposit them as thin layers by dispersing their nanoparticles in a gel containing a matrix that can maintain at least their bulk properties. After this size reduction at nanoscale and the annealing process following the deposition, changes and structural transformations would occur. We fabricate with success thin films by dispersing these nanoparticles in a gel. The materials show some agglomeration at the surface of the silicon substrate films (from SEM images) and non-identified hexagonal microcrystals, which could be at the origin of their excellent properties.

In this paper we use the combined USAXS/SAXS/WAXS instrument at 9ID beamline at APS-ANL for in situ characterization of undoped and 1% Mn doped PZN-4.5PT inorganic perovskite nanoparticles thin films deposited on nanostructured silicon to understand the phases transitions and determine the observed hexagonal microcrystals structure. It revealed a hexagonal structure of the nanoparticles thin films, which could be explained by the new phase that can be assigned to the $\text{Pb}_3(\text{PO}_4)_2$ based component. The peak at 31° indicates the presence of the rhombohedral phase perovskites assigned to the nanoparticles. XRD spectra, Raman and EDX mapping are compared to the USAXS, SAXS and WAXS results.

Keywords: Perovskite, nanoparticles, thin film, Synchrotron, Xray

Figure 1: EDX mapping for PZN-4.5PT thin film on silicon substrate after annealing.

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Surfaces and buried interfaces characterization of Mn/ZnO and $\text{Cu}_2\text{ZnSnS}_4$ based device: lessons learned from lab to synchrotron facilities

Author: Mac MUGUMAODERHA Cubaka¹

¹ Physics Departments, ISP-Bukavu and University of Namur

Corresponding Author: mugcubaka@gmail.com

A detailed picture of the thermally activated processes occurring at the Mn/ZnO interface was obtained by a combination of wide range of techniques. The low energy electron diffraction (LEED), the scanning tunnelling microscopy (STM) and spectroscopic techniques based on Auger electron (AES) and on high energy X-ray: X-ray photoelectron and absorption spectroscopies (HAXPES and

XANES) and the kinematical X-ray standing wave method (KXSW) allow not only to investigate the chemistry at the heterojunction but also to describe in detail the thermal structure transformations and diffusion process.

We also highlighted the structural and electronic properties of ZnO/CdS/Cu₂ZnSnS₄/Mo interfaces in different photovoltaic cells by the combination of SEM and destructive XPS depth profiles. The observed composition changes play a key role in the cell performances. The results may also drive a better control of the interfaces in quaternary based photovoltaic cells. Instead of destructive XPS depth profiling, a sounder understanding is possible using bulk sensitive hard X-ray photoemission at various detection angles.

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Solar cell simulations made easy

Author: Alex Quandt¹

Co-author: Robert Warmbier²

¹ *Wits*

² *University of Johannesburg*

Corresponding Authors: rwarmbier@uj.ac.za, alex.quandt@wits.ac.za

Numerical device simulations are a very powerful tool for the development of new types of highly efficient solar cells. The underlying theoretical framework describes many details of the complex flow of light through a photovoltaic device down to the atomic level. We will give a general overview of state-of-the-art device simulation methods and show that the most crucial simulation parameters can be taken from first principles quantum mechanical numerical calculations. This allows for the development of novel types of photovoltaic devices entirely on a computer. However, it also turns out that in many cases, simpler types of simulations involving the Shockley diode equation plus a realistic set of materials parameters are often sufficient to get a first idea about the performance a novel type of photovoltaic device. We will present some of these simplified approaches and describe how to combine them with experimental studies to develop more efficient types of photovoltaic devices.

3

Hybrid Organic–Inorganic Perovskites Materials: A review on Architecture and Stability for Power Conversion Efficiency

Author: Sidiki ZONGO¹

Co-authors: Antoine BERE²; Bouchta SAHRAOUI³; Jacques NEBIE⁴; Malik Maaza⁵; Sié Zacharie KAM²; Tizane DAHO²

¹ 1. *Department of Physics, Université Joseph KIZERBO, 03 PO. BOX 7021 Ouagadougou 03- Burkina Faso*// 2. *Materials Research Department, iThemba LABS, PO. BOX 7131, Cape Town-South Africa*

² 1-*Department of Physics, Université Joseph KIZERBO, 03 PO. Box 7021 Ouagadougou 03- Burkina Faso*

³ *Institute of Sciences and Molecular Technologies of Angers MOLTECH Anjou - UMR CNRS 6200, University of Angers, UFR Sciences 2 Bd Lavoisier 49045 ANGERS cedex 2, France*

⁴ 1. *Department of Physics, Université Joseph KIZERBO, 03 PO. BOX 7021 Ouagadougou 03- Burkina Faso*

⁵ *Materials Research Department, iThemba LABS, PO. BOX 7131, Cape Town-South Africa*

Corresponding Authors: maaza@tlabs.ac.za, sidiki.zongo@yahoo.fr, szachkam@gmail.com, jacquesnbi@yahoo.fr, bouchta.sahraoui@univ-angers.fr, berebiya@yahoo.fr, tizane_daho@yahoo.fr

Abstract

In the past decade, solar technology has emerged with the three competing technologies of crystalline semiconductors, polymeric and carbon nanostructures based. These technologies face to challenges that include cost and conversion efficiency. More recently, hybrid organic-inorganic perovskite (HOIP) materials has been proven as promising for new technologies of cost effective solar cells devices. Due to their tunable band gap, low temperature processing and abundant elemental constituents, in combination with their flexibility and simplicity of fabrication methods, hybrid perovskite materials allow us to reach an impressive power conversion efficiency (PCE) of 35.3%. In this review, we discuss the importance of perovskite film preparation, characterization and properties in achieving high performance HOIP photovoltaic cells. The review also focuses on highlighting the post-deposition and thermal annealing treatments.

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Bio-synthesized P2-Na_{0.57}CoO₂ nanoparticles as cathode for aqueous sodium ion battery

Author: Assumpta Nwanya¹

¹ *University of South Africa*

Corresponding Author: chinwe.nwanya@unn.edu.ng

We used aqueous extract obtained from the dry silk of zea mays lea plant to synthesize Na_{0.57}CoO₂ nanoparticles and studied same for use as cathode in aqueous Na ion battery (SIB). Structural characterizations by X-ray diffraction (XRD) show that the Na_{0.57}CoO₂ can be indexed to the hexagonal structure of the P 63/mmc (no 194) space group with P2 packing phase. The scanning electron microscope (SEM) micrograph of the Na_{0.57}CoO₂ nanoparticles (NPs) revealed an agglomerated sheet-like NPs of various sizes mixed with smaller sized spherical and quasi spherical NPs with uneven dimensions. Charge and discharge capacities of about 68 and 57 mA h g⁻¹ were respectively obtained when cycled at a C-rate of 0.7C using the bio synthesized Na_{0.57}CoO₂ and activated carbon as the positive and negative electrode and respectively. The cell retained about 79% of its inceptive capacity at the end of 1000 charge-discharge cycles and maintained about 98% Colomby efficiency from the 200th to the 1000th cycle when cycled at C-rate of 7C. Hence, the bio-synthesized Na_{0.57}CoO₂ NPs is a prospective positive electrode material for SIB.

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Exploring hematite as a choice material for photoelectrochemical water splitting.

Author: Mmantsae Diale¹

¹ *University of Pretoria*

Corresponding Author: mmantsae.diale@up.ac.za

Thus far, materials explored to achieve theoretical solar to hydrogen efficiency (STH), included cadmium selenide (CdSe), zinc oxide (ZnO), copper(I)oxide (Cu₂O), tungsten trioxide (WO₃) and hematite (α -Fe₂O₃). All these failed due to their band edge alignments that do not straddle water oxidation and reduction potentials. Of these materials, hematite has received much attention for photoelectrochemical water splitting attributed to its stability in aqueous solution, a small band gap of 1.90 eV-2.20 eV, non-toxicity and abundance. However, it has associated limitations such as high electron-hole recombination rate, short hole diffusion length (2-4 nm) accompanied by short excited lifetime of 10 ps, and poor minority charge carrier mobility of 0.1 cm² V⁻¹s⁻¹, leading to low photocurrent during water splitting. Furthermore, hematite promises a maximum theoretical STH efficiency of ~16.8 %, with photocurrent densities above 12 mAcm⁻², but the reported findings are far below 10 mAcm⁻². In this work, results produced from efforts to improve hematite as a choice material for water splitting included nanostructuring, doping, underlayers and use of PBS/LHCII to improve the photocurrent will be presented.

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The enhancement of the ionic conductivity of Sn-doped LiTi₂(PO₄)₃ by formation of an Al, Sn co-doped system

Author: Gugulethu C. Nkala¹

Co-authors: Sikhumbuzo M. Masina¹; Caren Billing¹; Roy P. Forbes¹; David G. Billing¹

¹ *Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, Johannesburg*

Corresponding Authors: 721270@students.wits.ac.za, dave.billing@wits.ac.za, 1762101@students.wits.ac.za, caren.billing@wits.ac.za, roy.forbes@wits.ac.za

NASICON-type LiTi₂(PO₄)₃ (LTP) has been studied as a potential solid-state electrolyte material in Li ion batteries (LIBs), owing to its thermal stability and fast ionic mobility. 1 The structure of LTP consists of TiO₆ octahedra corner-linked to PO₄ tetrahedra, forming a helix about the c-axis. Li can occupy two sites in the structure: the more stable six-fold O coordinated M1 and the eight-fold O coordinated M2, which is less stable. The net ionic movement is described as M1-M2-M1. The 3D network allows for the migration of alkali ions through the structure, making the material a candidate as an electrolyte in LIB. However, its room temperature conductivity in the order of 10⁻⁷ S/cm is too low for practical applications in LIBs. 1,2 Research around this class of materials has been focused on ways to increase their conductivities, including tuning the bottleneck size by substituting Ti⁴⁺ with other cations such as Zr⁴⁺ and Hf⁴⁺, and increasing Li⁺ concentration by lattice site substitution with M³⁺ cations as in Al-doped LTP. 3, 4 In the former case, substitutions in the framework with cations of larger ionic radii increase the lattice constants a and c, resulting in a bigger bottleneck size, thus higher conductivity of the mobile cations, Li⁺. In Al³⁺-doped systems, the presence of excess Li⁺ required for charge balance, increases the amount of charge carriers.

In this work, we investigate the effect of Al³⁺/Sn⁴⁺ co-doping at the Ti⁴⁺ site on the room temperature conductivity of LTP. The structural properties have been studied using synchrotron PXRD and pair distribution function (PDF). Raman spectroscopy provided insight on the changes in local order around the substitution sites as well as confirming the phase composition of the material in question. LATSP showed improved ionic conductivity of 8.04 × 10⁻⁶ S/cm.

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Summary:

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Growing a user base for an African Synchrotron facility

Author: George Clerk^{None}**Corresponding Author:** george.clerk@bucks.ac.uk

The glaring absence of a synchrotron facility in Africa is a growing concern, which has triggered efforts leading towards the establishing of such a facility, creating the opportunity for developing the knowledge base, skills, train and maintain the personnel for transforming the renewable energy landscape. The African Synchrotron Network for Advanced Energy Materials (ASNAEM) conducted a study to assess the potential user base for a future pan-African synchrotron light source.

This study includes a survey of scientists to provide insight into the objectives, opportunities and challenges of their research, their experience with stakeholders (for e.g. government and industry) and recommendations for improving their experience doing research. Also, it included a qualitative study of the current state of Synchrotron use in Africa, and the opportunities, challenges and the potential impact on society. The project focuses on obtaining data through open-ended and conversational communication.

The findings underpin the need for a synchrotron facility on the continent and the need for boosting the developing a research culture, providing broad infrastructure for supporting the work of scientists, which includes.

Summary:

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Enigmatic Structure Property Behaviour in SOFC & SOEC electrolyte materials.

Author: David Billing¹¹ *University of the Witwatersrand***Corresponding Author:** dave.billing@wits.ac.za

Solid Oxide Fuel Cells (SOFCs) and Solid Oxide Electrolyser Cells (SOECs) are exciting electrochemical devices that provide unique and revolutionary solutions to some of the renewable energy challenges facing society. The archetype materials used as solid electrolyte in most devices include YSZ (Yttrium stabilised Zirconia) and CaSZ (Calcium stabilised Zirconia) with the Y or Ca dopants present at around 8 to 10% level. As the performance characteristics of these materials are not completely satisfactory, there is a definite need for improved alternatives. Particularly Doped Cerate and Bis-muthate are being investigated as alternatives. Although it is well established that most conducting phase of these is cubic with average structure features consistent with the Flourite structure, there have been only a few no reports of studies into the nano-structure or local structures of these materials. The nuanced structural details of these materials are thus not yet clear, and certainly play a significant role in the important properties of the materials. Within this context our research has

focused on gaining a fundamental understanding of the mechanisms governing the transport properties of these and closely related materials such as δ -Bi₂O₃ which has the highest reported oxides ionic conductivity for the (Bi_{0.5})_{0.88}(Dy_{0.5})_{0.08}(WO₃)_{0.04} case [1], as well as the role of the various doped variants in these structure-property relationships. Typically the cubic forms of these materials exhibit higher oxygen ionic conductivity due to the presence of vacant anionic sites, and exists only at elevated temperatures. In most cases doping results in only a meta-stable cubic phase that slowly transitions to a less conducting phase. From a collection of almost 400 distinct chemical compositions we have learnt that the nature, number and concentration of the dopants used, all affect the conductivity and stability of the desired phase in a complicated manner.

I will present a selection of our results to date. Including our PDF & PXRD analysis of the scattering data we measured at ID-22 at the ESRF, as well as at 28-ID-1 at NSLS-II. Variations in the respective thermoresponsive behaviours clearly shows structural variations when comparing the structure as perceived on the nano-scale with the bulk average structure.

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Summary:

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Probing the Local Structure of Bi₂O₃ Chemical Derivatives: The Tale of the Neglected Cation Sublattice

Author: Sikhumbuzo M. Masina¹

Co-authors: Gugulethu C. Nkala²; Kevin H. Stone³; Caren Billing¹; David G. Billing¹

¹ *Molecular Science Institute, School of Chemistry, University of the Witwatersrand, Private Bag X3, Johannesburg, 2050, South Africa*

² *DSI-NRF Centre of Excellence in Strong Materials, University of the Witwatersrand, Private Bag X3, Johannesburg, 2050, South Africa*

³ *Stanford Synchrotron Radiation Lightsource, SLAC National Accelerator Laboratory, 2575 Sand Hill Road, Menlo Park, CA, 94025, USA*

Corresponding Authors: 1762101@students.wits.ac.za, dave.billing@wits.ac.za, 721270@students.wits.ac.za, caren.billing@wits.ac.za

Comprehensive understanding of the average and local structure of materials plays a major role in formulating reliable deductions about the structure-property correlations. It may lead to complex structure engineering and physical property enhancement. One of the functional materials that have found extensive use in the fabrication of devices of technological importance is Bi₂O₃ [1]. Bi₂O₃ exists in many different polymorphs. Its δ -polymorph has the highest ionic conductivity (approximately 1 S cm⁻¹) ever reported for an oxide ion conductor at 730 °C [2]. However, it is only stable within a narrow range (730 – 824 °C) [2]. To stabilise δ -Bi₂O₃ like phases back to room temperature, hence forth referred to as stabilised δ -phases, isovalent and aliovalent cations have been used to substitute for the Bi³⁺ cations with a moderate decrease in ionic conductivity associated with the substituents [3].

The stabilised δ -phases obtained by substituting Bi³⁺ with lanthanides have been shown to be highly conductive. However, these are metastable and exhibit phase separation upon prolonged annealing at temperatures around 600 °C [4]. The oxide ions and oxygen vacancies order in a process called ageing in these systems. These problems are normally circumvented by adding low concentrations of cations such as W⁶⁺, and Nb⁵⁺ [3]. Systems fabricated in this way exhibit both high conductivity and stability relative to the singly substituted systems. In this work, the systems (Bi₂O₃)_{1-x-y}(Dy₂O₃)_x(Er₂O₃)_y, (Bi₂O₃)_{1-x-y-z}(Dy₂O₃)_x(Er₂O₃)_y(WO₃)_z, (Bi₂O₃)_{1-x-y-z}(Dy₂O₃)_x(Er₂O₃)_y(Nb₂O₅)_z, and (Bi₂O₃)_{1-x-y-2z}(Dy₂O₃)_x(Nb₂O₅)_y(WO₃)_{2z} were fabricated to stabilise the δ -phase and study the effect of the substituents in the local structure.

The local structure of the stabilised δ -phases is a subject of intense research [5]. However, the research focus is on the distribution of the oxide ions around the metal cations, i.e., the metal-oxygen (M–O) correlations. Very few studies have been done on the metal-metal (M–M) correlations. Therefore, this work seeks to bridge this gap and provide a complementary picture of the local structure of the stabilised δ -phases. To this end, total scattering data, the x-ray pair distribution function (xPDF) technique and x-ray absorption spectroscopy have been used to probe the metal-metal correlations

and the local environment around a specific substituent to get insight into the local structure of the material around the metal cations.

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Summary:

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Nanostructured Copper Oxide: Preparation and Characterisation

Author: DODOO-ARHIN DAVID¹

Co-authors: Matteo Leoni ²; Paolo Scardi ²

¹ UNIVERSITY OF GHANA

² University of Trento

Corresponding Authors: ddodoo-arhin@ug.edu.gh, paolo-scardi22@unitn.it, matteo-22@unitn.com

Nanostructured copper oxide has attracted several research interests over the years due to their interesting optical properties and their potential use in several electronic applications such as solar cells and gas sensors.

In this work, reverse micelle microemulsions (a bottom-up approach) and high energy milling (Top-down approach) have been employed for the production of defect-free and highly defective nanocrystals.

The produced defect-free nanocrystals show good crystallinity with the nanometric nature of the primary domains (20 nm – 4 nm) leading to quantum confinement phenomena highlighted by photoluminescence measurements. Models have been proposed for the nanocrystal formation and growth validated by means of several techniques such as X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM), UV-Visible and Fourier Transform InfraRed spectroscopy (UV-Vis and FTIR).

The effect of high energy milling on the microstructure evolution was investigated using synchrotron radiation XRD line profile analysis supported by High Resolution TEM and SEM. The reduction in the average size of coherently scattering domains - and simultaneous narrowing of the size distribution - occurs in the first minutes. A proper nanocrystalline microstructure is obtained for a milling of ca. 20 min with asymptotic limit of ca. 10 nm. The reduction in size occurs at the expenses of introducing a massive quantity of dislocations in the system, reaching a limit of ca. $4 \times 10^{-16} \text{ m}^{-2}$.

Keywords: Nanostructured materials, Copper Oxide, Synchrotron radiation XRD, Electron microscopy.

Summary:

NA

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Thermal expansion behaviour of BPO4 studied by X-ray thermodiffraction

Authors: Mashikoane Mogodi¹; David Billing²

¹ *University of Cape Town*

² *University of the Witwatersrand*

Corresponding Authors: dave.billing@wits.ac.za, wilson.mogodi@uct.ac.za

This talk presents the crystal structure dynamics as a function of temperature of non-porous borophosphates. Much of the work published on these materials has focused on investigating the synthetic approaches as well as exploring their fascinating structural chemistry. Little has been reported about their thermal expansion behaviour. This thermoresponsive behaviour revealed their thermal stability while serving as a predictive measure of the effects of temperature on other materials properties and subsequent application. The thermal expansion behaviour of the non-porous cristobalite BPO4 phase is will be highlighted. BPO4, which crystallizes in the tetragonal lattice, showed a substantial anisotropic expansion in the lattice. This contrasting thermal expansion behaviour along the various axes of the material is related to the temperature variation of the inter-polyhedral angle between adjacent polyhedral in the crystal structure. This talk will also include total scattering data collected at the European Synchrotron Radiation Facility (ESRF), beamline ID22. This analysis is sensitive not only to the average (local-range) material structure but also the local distortions away from the average structure. Results will be reported relating to the total scattering investigation of the thermal expansion of the B-O and P-O bond-lengths in comparison with the values obtained by conventional X-ray diffraction measurements.

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The X-TechLab platform in Benin: a regional feeder facility for the future African Light Source Facility

Author: Thierry d'Almeida¹

¹ *Sèmè City*

Corresponding Author: tdalmeida@semecity.com

X-TechLab is a regional training platform that aims to provide the region with skills and tools to use X-ray techniques for developing innovative solutions to critical issues in Africa. The initiative is the result of an interaction between the Lightsources for Africa, the Americas, Asia, Middle East, and the Pacific (LAAAMP) and the Sèmè City hub, one of Benin Government's flagship projects, which aims to create a world-class knowledge and innovation centre in Africa. The goals are to: 1) provide hands-on experience with the use of cutting-edge X-ray equipment, 2) develop X-ray-based problem-solving skills targeting specific socioeconomic issues, 3) meet the requirement for Feeder Facilities that allow the preparation of samples to be studied at world advanced light sources and 4) contribute to the emergence of a community of experts who will be active users of the future African Synchrotron.

The X-TechLab platform was established in 2019 within Sèmè City, one of Benin government's flagship project which aims to create a world class knowledge and innovation hub, focused on the challenges facing the African continent. The overall mission of X-TechLab is to provide the region with the skills and tools that are needed to use the potential of X-ray techniques in order to design innovative solutions to critical issues in various fields such as health, agriculture, energy, and the environment.

The initiative is the result of an interaction between the Lightsources for Africa, the Americas, Asia, Middle East, and the Pacific (LAAAMP) project and the Sèmè City hub. The specific goals are to: 1) provide hands-on experience with the use of cutting-edge X-ray equipment, 2) develop X-ray-based problem-solving skills targeting specific socioeconomic issues, 3) meet the requirement for Feeder Facilities that allow the preparation of samples to be studied at world advanced light sources and 4)

contribute to the emergence of a community of experts who will be active users of the future African Synchrotron.

Learners participating in the X-TechLab are trained around 2 parallel, interrelated yet distinct, tracks: Crystallography and X-ray diffraction techniques, including both single and powder diffraction applied to structural studies; and Absorption and phase contrast X-ray imaging (Microtomography) using mathematical tools for research on sustainable and ecological materials. Started in 2019, X-TechLab training sessions gathered many scientists from several countries and scientific disciplines. About 20 Experts from several academic institutions worldwide (Africa, Europe, USA) are involved in the training sessions. This talk will emphasize the unique potential of X-ray techniques as a multidisciplinary tool for development in Africa. We will present the facility, the achievements so far and future prospects.

Summary:

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Update on the AfLS project

Author: Simon Connell¹

¹ *University of Johannesburg*

Corresponding Author: shconnell@uj.ac.za

The African Light Source (AfLS) Foundation is engaged in developing the case for an advanced Light Source (adLS) to be designed, constructed and operated on the African continent. The motivation is the enormous benefit to the continent from Science, Technology, Innovation and Development, especially in areas of crucial interest to Africa. There are several areas of progress recently, to note, which are in addition to the baseline activities of human capacity development. In all cases, more details are available at the AfLS website. The AfLS has established its International Advisory Committee. The Interim Chair is Prof Khotso Mokhele, the Chancellor of a SA University, formerly CEO of the SA National Research Foundation, and well versed in large scale research infrastructure advocacy and leadership in a Pan African Context. There are now two MoUs (LAAAMP and SESAME) and many letters of support for the AfLS from African National, Pan African and International organisations and institutions. Another important step in our roadmap is the production of a comprehensive Conceptual Design Report (CDR) for the modern proposed adLS. The editor of the 5 volume CDR is Prof Sekazi Mtingwa. The volumes cover the full spectrum of drivers for this initiative, and also details the science case, as developed from a consultative process within Africa, from its diaspora and from friends and stakeholders in the wider community. The science (including the innovation and business imperatives) then informs the technical specifications of the machine and its instruments. The CDR also includes aspects of finance and governance. We believe that the CDR will concretise the conversation, thus the developing commitment from African Governments and the Pan African diplomatic nature of the project. In fact, the CDR is a most significant milestone to be achieved. Of course, the concept of an AfLS is simultaneously a concept of much advanced human capacity development and also development of local and regional research infrastructure. It also implies a step forward in the capacity of Africa to innovate and have competitive businesses. As such, for example, in a future pandemic, Africa would make a strong contribution in vaccine discovery and production. The AfLS-CDR Town Hall launch event for the Letters of Interest is on Tuesday 11 Oct 2021 from 15:00 – 18:30. There will be the next edition of the AfLS Conference series, AfLS3 2021, from 15-19 November 2021. It is a co-organised event together with the Pan African Conference on crystallography (ePCCr) and also the African Physical Society (AfPS), as well as a cohort of other African scientific organisations. A landmark feature of this conference is several sessions involving African Government Leaders and the African Union Commission, discussing the strategic policy towards the AfLS, in addition to the normal scientific discourse.

Summary:

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Two-dimensional Doping of Proton Conductors

Author: Prosper Ngabonziza¹

¹ *Max Planck Institute for Solid State Research*

Corresponding Author: ngabonzizap72@gmail.com

Ionic conducting heterostructures are of interest to explore interfacial effects in solid state ionics and to foster their potential deployment in clean energy technologies such as solid oxide fuel cells. How to achieve ion conduction in heterostructures is therefore a fascinating and relevant question.

In this presentation, we will report on the first realization and study of a two-dimensionally doped ion conductor. This work is based on epitaxial BaZrO₃-BaYO_x heterostructures [1] in which entire ZrO₂-layers of the BaZrO₃ crystal are replaced by heterovalent layers (YO_x). The resulting charge carriers reside in the immediate vicinity of the substituted layer. These heterostructures show – if hydrated - significant proton conductivities increasing with the number of interfaces. They are comparable, yet somewhat lower than those of hydrated Ba(Zr,Y)O₃ ceramics. Pros and cons of 2d versus conventional 3d doping are discussed.

To explore the potential of inelastic electron tunneling spectroscopy to study ionic species at high-temperatures, we then use the same BaZrO₃-based heterostructures as proton conductors and electron tunnel barriers in tunnel junction devices [2]. These junctions yield high-resolution inelastic tunneling spectra of protons diffused along the interfaces in BaZrO₃-BaYO_x-based tunnel barriers up to at least 400 K, breaking the previously established fundamental resolution limit by a factor of nine. By analyzing O-H bond vibrations, the existence of protons in the tunnel barriers is confirmed.

[1] P. Ngabonziza, R. Merkle, Y. Wang, P. A. van Aken, T. S. Bjørheim, J. Maier, and J. Mannhart, 2D Doping of Proton Conductors: BaZrO₃-Based Heterostructures., *Adv. Energy Mater.* 11, 2003267 (2021).

[2] P. Ngabonziza, Y. Wang, P. A. van Aken, J. Maier, and J. Mannhart, Inelastic Electron Tunneling Spectroscopy at High-Temperatures., *Adv. Mater.* 33, 2007299 (2021).

Summary:

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ASNAEM Project

Corresponding Author: m.c.newton@soton.ac.uk

Introduction to the project

Summary: