

DAY 1

Keynote Forum



17th Edition of International Conference on

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David Horwat, Nano Res Appl, Volume:4
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OPTIMIZATION OF AL-DOPED ZNO TRANSPARENT ELECTRODES WITH AND WITHOUT THERMAL ASSISTANCE USING HIPIMS

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Aluminum-doped zinc oxide (AZO) is a transparent conductor that can be used in thin film form as transparent electrode of electro-optical devices. For most of actual applications, a large conductivity is required on large surface areas. The rapid growth of transparent electronics and electro-optical devices on flexible supports calls for the development of methods that enable the synthesis of transparent conducting films without thermal assistance while keeping high electrical and optical performances. Magnetron sputtering has emerged as a reference method for the synthesis of AZO films. It is particularly due to, its scalability to industrial scale. Unfortunately, AZO films usually produced without thermal assistance using magnetron sputtering tend to a strong inhomogeneity of the electrical properties with a large sensitivity to the process parameters (composition of the gas phase, geometry of the experiment). This presentation first highlights the interest of high power impulse magnetron sputtering (HiPIMS) to synthesize AZO films of high electronic conductivity on large surface areas and without thermal assistance. Electronic structure measurements using X-ray absorption spectroscopy evidence a correlation between the distribution of the electrical behavior and dopant activation/inactivation. A deactivation mechanism, complementary to the well-known compensation of dopants, is proposed in the case of conventional sputtering. A model explaining the minimization of the deactivation amplitude is proposed in case of HiPIMS. In a second part we will show how the electrical properties of AZO films degraded after long term exposure to ambient moisture can be restored by low temperature thermal annealing and how thermal assistance during growth can prevent degradation upon exposure to ambient moisture. Finally, self-nanostructured AZO films could be obtained using HiPIMS under certain conditions and their interest for flexible electronics is highlighted.

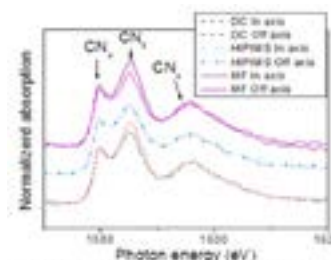


Figure 3: Electronic structure at the Al-K edge as probed by X-ray absorption near-edge structure for direct current (DC), HiPIMS and medium frequency (MF) reactive sputtering of AZO films.

Recent Publications

1. D Horwat and A Billard (2007) Effects of substrate position and oxygen gas flow rate on the properties of ZnO: Al films prepared by reactive co-sputtering, *Thin Solid Films* 515(13):5444-5448.
2. D Horwat, et al. (2010) On the deactivation of dopant and electronic structure in reactively sputtered transparent Al-doped ZnO thin films *Journal of Physics D: Applied Physics* 43, 132003.
3. M Jullien, et al. (2011) Influence of the nanoscale structural features on the properties and electronic structure of Al-doped ZnO thin films: an X-ray absorption study. *Solar Energy Materials and Solar Cells* 95:2341-2346.
4. M Mickan et al. (2016) Room temperature deposition of homogeneous, highly transparent and conductive

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Al-doped ZnO films by reactive high power impulse magnetron sputtering, *Solar Energy Materials and Solar Cells* 157:742-749.

5. M Mickan, M Stoffel, H Rinnert, U Helmersson and D Horwat (2017) Restoring the properties of transparent al-doped ZnO thin film electrodes exposed to ambient air *Journal of Physical Chemistry C* 121:14426–1443.

Biography

David Horwat has an expertise in the physico-chemistry of inorganic functional thin films. He is more particularly interested in understanding how physical properties, more particularly electrical and optical, are related to local structural, chemical states and nanostructures and on ways to modify them using physical vapor deposition methods. His research is based on a multidisciplinary approach involving synthesis, spectrometries/spectroscopies and microscopies. He teaches Materials Science and Engineering at the European School of Materials Engineering (EEIGM) and conducts his research activities in the POEME research group of Institut Jean Lamour at University of Lorraine.

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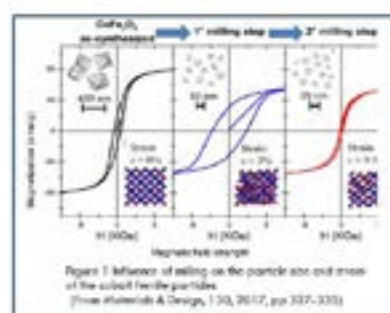


MULTIFERROIC CERAMIC COMPOSITES: PROCESSING AND MICROSTRUCTURE

Carmen Galassi

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Among multifunctional materials magnetolectric multiferroics are a special class with coupled ferromagnetic and ferroelectric orders. They are particularly appealing because they not only show the characteristic of the single ferroic orders where the electric field E , magnetic field H , and stress σ control the electric polarization P , magnetization M , and strain ϵ , respectively, but also because interactions between them lead to additional functionalities. Therefore in a magnetolectric multiferroic, H may control P or E may control M . In composite materials the electric and magnetic degrees of freedom are located in different components and are interfacially coupled to each other. Several applications, including magnetic sensors, high-frequency inductors, memory devices, and high-frequency signal processing devices, (very high-density memory storage media) have been proposed and demonstrated. Nanopowders are mainly synthesized by solid state synthesis, coprecipitation, hydrothermal and sol-gel methods and the magnetic properties vary by changing the processing parameters and/or the stoichiometry. The magnetostrictive and piezoelectric materials used as constituents of magnetolectric (ME) composites are ferromagnetic oxides, including ferrites like cobalt ferrite (CFO), manganites, 3d-transition metals/alloys for the magnetic phase and lead zirconate titanate (PZT), barium titanate (BTO), or lead magnesium niobate-lead titanate (PMN-PT) for the ferroelectric phase. Bulk and thin or thick film structure are produced where the connectivity of the phases plays a critical role; and the 3-0 type particulate structure, 2-2 type laminate structure and the 1-3 type cylinder matrix are the most frequently investigated. The influence of the processing on the microstructure and final properties is shown for PZT-CFO particulate and laminate composites with focus on the role played by the milling and densification treatments that allow producing high- and low-coercivity nanosized cobalt ferrite nanoparticles and fully dense composites.



Recent Publications

1. Srinivasan G, Priya S and Sun N X (ed) (2015) Composite magnetolectrics: materials, structures, and applications, Cambridge: Woodhead, ISBN: 978-1-78242-254-9.
2. Galizia P, Ciuchi I V, Gardini D, Baldisserrri C and Galassi C (2016) Bilayer film based on composite $\text{CoFe}_2\text{O}_4/\text{TiO}_2$ and niobium-doped PZT by electrophoretic Deposition Journal of the European Ceramic Society 36(2):373-380.
3. Galizia P, Baldisserrri C, Capiani C and Galassi C (2016) Multiple parallel twinning overgrowth in nanostructured dense cobalt ferrite. Materials and Design 109:19-26.
4. Galizia P, Ciomaga C E, Mitoseriu L and Galassi C (2017) PZT-cobalt ferrite particulate composites: densification and lead losses control by quite-fast sintering Journal of the European Ceramic Society 37:161-168.
5. Galizia P, Cernea M, Mihalache V, Diamandescu L, Maizza G and Galassi C (2017) Easy batch-scale production of cobalt ferrite nanopowders by two-

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step milling: structural and magnetic characterization.
Materials and Design 130:327–335.

Biography

Carmen Galassi is a Research Director at CNR-ISTEC, Italy. She is the Head of the Research Project – Smart multifunctional ceramic materials: piezoelectrics, ferroelectrics, antiferroelectrics, multiferroics with main expertise in ceramic processing (powder treatments, shaping and densification),

chemico-physical characterization of dispersed ceramic systems, R&D on piezoelectric ceramic materials, magnetic ceramic materials and relative composites, multifunctional materials. Her main activity is research projects coordination and dissemination on ceramic materials development, processing and characterization and tutoring MSc and PhD students. She has got the National Scientific Qualification to function as Full Professor of Materials Science and Technology in Italian Universities. Out of 259 papers co-authored, 168 are published in international, refereed journals.

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NANOMATERIALS AS LUBRICANT ADDITIVES FOR MORE EFFICIENT AND MORE ENVIRONMENT FRIENDLY ENGINE LUBRICANT

Fabrice Dassenoy

Ecole Centrale de Lyon, France

There has been growing interest in nanoparticles for tribological applications over the past 20 years. Studies have shown the remarkable lubricating properties, viz., friction-reducing and anti-wear, of these small objects. This makes them potential candidates for replacing the molecular lubrication additives currently used in automobile lubricants, known to be pollutants and less efficient in some specific conditions. This has not gone unnoticed to the oil companies and the car manufacturers. All of them are faced with the problem of providing lubricants that meet the needs of the technological evolution of engines while respecting ever stricter environmental norms. Among the nanoparticles with proven tribological performance are carbon nanotubes and onions, boron nitride nanoparticles, and inorganic fullerene-like (IF) metal disulfides (IF-MoS₂, IF-WS₂). The latter exhibit the best friction-reducing and anti-wear properties ever observed. These nanoparticles have been the subject of detailed investigation in the lab for more than fifteen years now, and during this time, many key issues have been tackled, such as the conditions leading to these properties, the lubrication mechanisms coming into play, and the influence of parameters such as size, structure, and morphology of the nanoparticles on their tribological properties and the lubrication mechanisms. These years of research have given us a good understanding of the way these nanoparticles behave, and we can now identify the key parameters to be adjusted when optimizing their lubrication properties. During this presentation the performance and the lubrication mechanism of the metal disulfide nanoparticles will be presented and we will see how to move from the nanoparticle to the development of new highly efficient nanolubricants for automotive applications (engine and gearboxes).

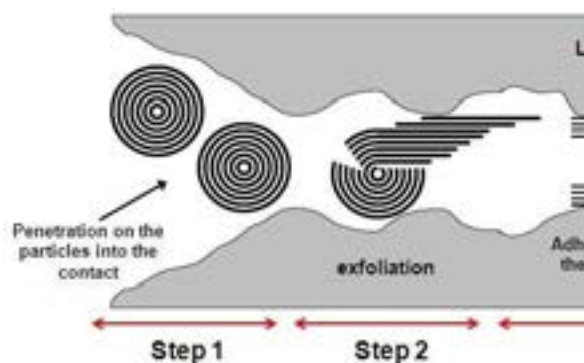


Figure 1: Lubrication mechanism of the inorganic fullerene-like metal disulfide nanoparticles.

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1. Jenei I and Dassenoy F (2017) Friction coefficient measured on a single WS₂ nanoparticle: an *in situ* transmission electron microscope experiment. *Tribology Letters* 65(86):2-8.
2. Ussa Aldana P, Dassenoy F, Vacher B, Le Mogne T and Thiebaut B (2016) WS₂ nanoparticles anti-wear and friction reducing properties on rough surfaces in the presence of ZDDP additive. *Tribology International* 102:213-221.
3. Ussa Aldana P, Dassenoy F, Vacher B, Le Mogne T, Thiebaut B and Bouffet A (2016) Antispalling effect of WS₂ nanoparticles on the lubrication of automotive gearboxes. *Tribology Transactions* 59(1):178-188.

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4. Rabaso P, Dassenoy F, Ville F, Diaby M, Vacher B, Le Mogne T, Belin M and Cavoret J (2014) An investigation on the reduced ability of IF-MoS₂ nanoparticles to reduce friction and wear in the presence of dispersants. *Tribology Letters* 55(3):503-516.
5. Lahouij I, Bucholtz E, Vacher B, Sinnott S and Dassenoy F (2012) Lubrication mechanisms of hollow-core inorganic fullerene-like nanoparticles: coupling experimental and computational works. *Nanotechnology* 23:375701.

Biography

Fabrice Dassenoy has completed his PhD in Materials Science (1999) from the Paul-Sabatier University of Toulouse, France. He spent three years (2000-2002) in Surface Physics Department at the Max-Planck Institute of Berlin, Germany before joining École Centrale de Lyon first as Assistant Professor (2002-2011) and got promoted to Professor in 2012. As an expert in nanomaterials, his research conducted at the Tribology and System Dynamics Laboratory (LTDS) is related to the study of the tribological properties of nanoparticles used as lubricant additives, with a particular interest in understanding the lubrication mechanisms of these nanomaterials and in the optimization of their lubricating properties, especially for automotive applications. He has published more than 50 articles and four patents in this field and got the Taiho Encouraging Award for Young Tribologists in 2009. He teaches Materials Science at École Centrale de Lyon with a focus on the characterization techniques. He is currently in charge of a research group called Understanding Fundamental Phenomena in Tribology.

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DAY 2

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ROOM TEMPERATURE HYDROGENATION IN FUNCTIONAL OXIDE NANOWIRES BY AN ELECTRIC FIELD VIA AIR NANOGAP

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Institute of Scientific and Industrial Research - Osaka University, Japan

Large reversible changes of the electronic transport properties of solid-state oxide materials induced by electrochemical fields have received much attention as a new research avenue in iontronics. The action on time-dependence of conductive modulation is slower. Despite the slow modulation, the emergence of non-linear, plastic and/or memristive behaviors provides an opportunity to obtain new abilities in information processing, like signal flow in brain, in addition to sensing and energy devices. In this conference, dramatic transport changes in VO₂ nanowires were demonstrated by electric field-induced hydrogenation at room temperature. As a suitable device structure to perform transport modulation through electrochemical reactions, we proposed a planar-type field effect transistor with side gates and a nanowire channel separated by air nanogaps (denoted PG-FET), as illustrated in Figure 1. This unique structure allowed us to investigate hydrogen intercalation and diffusion behavior in VO₂ channels with respect to both time and space. Figure 2 shows the reversible, non-volatile resistance changes in a VO₂ nanowire channel with a width (w) of 500 nm obtained by applying positive and negative VG at 300 K under a humidity of around 50%. The normalized resistance (R/R₀), where R and R₀ are the measured resistance and resistance of the pristine device before applying a VG at 300 K, respectively) slowly decreased down to the saturation line at roughly $R/R_0 = 0.75$ during the application of VG = +100 V. This state was held after the removal of the VG. Namely, the device exhibited a non-volatile memory effect. The R/R_0 increased again with applying V_G = -100 V. Our results will contribute to further strategic researches to examine fundamental chemical and physical properties of devices and develop iontronic applications, as well as offering new directions to explore emerging functions for sensing, energy, and neuromorphologic devices combining ionic and electronic behaviors in solid-state materials.



Figure 1: Typical device structure (left) and an atomic force microscopy image of the VO₂ channel area (right). L, D and G indicate the source, drain and gate electrodes, respectively.



Figure 2: The dependence of normalized resistance (R/R_0) on VG with VG of 100 V and -100 V. (Inset: 10³).

Recent Publications

1. Manca N, Pellegrino L, Kanki T, Venstra W J, Mattoni G, Higuchi Y, Tanaka H, Caviglia A D and Marré D (2017) Selective high-frequency mechanical actuation driven by the VO₂ electronic instability. *Advanced Materials* 29, 1701618.
2. Wei T, Kanki T, Chikanari M, Uemura T, Sekitani T and Tanaka H (2017) Enhanced electronic-transport modulation in single-crystalline VO₂ nanowire-based solid-state field-effect transistor. *Scientific Reports* 7, 17215.
3. Kanki T and Tanaka H (2017) Nanoscale electrochemical transistors in correlated oxides. *APL Materials* 5, 042303.
4. Wei T, Kanki T, Fujiwara K, Chikanari M and Tanaka H (2016) Electric field-induced transport modulation in VO₂ FETs with high-k oxide/organic parylene-C hybrid gate dielectric. *Applied Physical Letters* 108, 053503.
5. Sasaki T, Ueda H, Kanki T and Tanaka H (2015) Electrochemicalgating-induced reversible and drastic resistance switching in VO₂ nanowires. *Scientific Reports* doi: 10.1038/srep17080.

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Biography

Teruo Kanki has completed his PhD in Material Physics from Osaka University in 2004. After working as Visiting Researcher in IBM's Almaden Research Center from 2004 to 2006, he became a specially appointed Assistant Professor in Osa-

ka University. Now he is an Associate Professor in Osaka University and works on novel and new concept oxide nano-electronics. He has published more than 80 papers in reputed journals.

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SINGLE DNA SEQUENCING AND VISUALIZATION OF MIR-134 IN NERVE CELLS WITH FORCE-BASED AFM

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Single-molecule sequencing methods have been developed to analyze DNA directly without the need for amplification. Here, we present a new approach to sequencing single DNA molecules using atomic force microscopy (AFM). In our approach, four surface-conjugated nucleotides were examined sequentially with a DNA polymerase-immobilized AFM tip. By observing the specific rupture events upon examination of a matching nucleotide, we could determine the template base bound in the polymerase's active site. The subsequent incorporation of the complementary base in solution enabled the next base to be read. Additionally, we observed that the DNA polymerase could incorporate the surface-conjugated dGTP when the applied force was controlled by employing the force-clamp mode. MicroRNAs (miRNAs) play key roles in controlling various cellular processes, and the expression levels of individual miRNAs can be considerably changed in pathological conditions such as cancer. Accurate and precise quantification of miRNA at the single-cell level will lead to a better understanding of miRNA function. Here, we present a direct and sensitive detection method for a specific miRNA using AFM. A hybrid binding domain (HBD)-tethering tip enabled mature miRNAs to be located individually on an adhesion force map. By scanning several sections of a micron-sized DNA spot, we were able to quantify the copy number of miR-134 from a single neuron and demonstrated that the expression was increased upon the cell activation. Additionally, we visualized individual miR-134s on fixed neurons after membrane removal and observed 2-4 miR-134s in the area of 1.0x1.0 μm^2 of soma. The number increased to 8-14 in stimulated neurons, and this change matches with the ensemble-averaged increase in copy number.

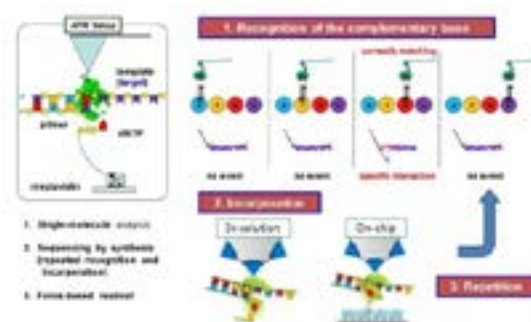


Figure 1: Three steps for the single DNA sequencing with AFM

Recent Publications

- Hyun-Seo Koo, Ikbum Park, Yoonhee Lee, Hyun Jin Kim, Jung Hoon Jung, Joo Han Lee, Youngkyu Kim, Joung-Hun Kim and Joon Won Park (2016) Visualization and quantification of microRNA in a single cell by AFM. *Journal of the American Chemical Society* 138(36):11664.
- Yoonhee Lee, Youngkyu Kim, Donggyu Lee, Dhruvajyoti Roy and Joon Won Park (2016) Quantification of fewer than ten copies of a DNA biomarker without amplification or labeling. *Journal of the American Chemical Society* 138(22):7075.
- Woong Kim, Nara Kim, Joon Won Park and Zee Hwan Kim (2016) Nanostar probes for tip-enhanced spectroscopy. *Nanoscale* 8(2):987.
- Dae Heon Kim, Jae-Eun Lee, Zheng-Yi Xu, Kyeong Rok Geem, Yun Kwon, Joon Won Park, Inhwan Hwang (2015) Cytosolic targeting factor AKR2A

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captures chloroplast outer membrane-localized client proteins at the ribosome during translation, *Nature Communications* 6:6843.

5. Youngkyu Kim, Eung-Sam Kim, Yoonhee Lee, Joung-Hun Kim, Bong Chu Shim, Seong Moon Cho, Jeong Soo Lee and Joon Won Park (2014) Reading single DNA with DNA polymerase followed by atomic force microscopy. *Journal of the American Chemical Society* 136(39):13754

Biography

Joon Won Park has obtained his PhD at California Institute of Technology in

1988, and worked in Northwestern University as a Postdoctoral Fellow for two years, he started an independent professional career at Pohang University of Science and Technology (or POSTECH) in 1990. His initial research was focused on self-assembled monolayer and created a dendron-modified surface that provides regular nanoscaled spacing between immobilized molecules. He combined the proprietary approach with force-based atomic force microscopy. As a result, he was able to increase the reliability, reproducibility and accuracy of the analysis. He has been keen on analyzing various biomarkers including DNA, mRNA, microRNA, and protein of low abundance quantitatively without amplification as well as manipulating single biomolecules and mapping biomolecules at high resolution. He was the Head of Chemistry Department and is a Founder of NB POSTECH Inc. (a Pohang-based venture) and Nanogea Inc. (Los Angeles-based venture).

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