January 2014

CNMS User Newsletter

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Important Dates:

- Call for User Proposals: *Deadline April 30*
- 2014 User Meeting September 15-19, 2014

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We encourage feedback and suggestions for the content of future newsletters. We are especially interested in receiving research highlights from CNMS users that may be featured in future issues of this newsletter. Please email us any time you have an important paper that is accepted for publication.

Message from the UEC Chair

2014 CNMS UEC Chair:
Vivek Prabhu, NIST

As your elected 2014 User Executive Committee (UEC) Chair, Welcome to the CNMS! This year already has an exciting start for us with our new CNMS Director, Hans Christen, the addition of the world-class ShaRE electron microscopy and atom probe tomography facility into the CNMS user program, and the rollout of the new website. The CNMS provides users with access to a complete suite of nanoscience research and fabrication capabilities. The UEC was established as an impartial body to facilitate an active dialog between you, the User community, and the CNMS leadership. The UEC is here to voice your questions and comments and we value your input. We are continually looking for new ways that the User community can become engaged with the CNMS for mutual benefit. Feel free to contact me or any of the UEC members directly, or anonymously via the "Feedback" button on the CNMS website. Your feedback improves the User experience and helps make the most of the CNMS resources to advance science and technology. Here are a few comments we recently received during our annual survey:

- *"As an undergraduate research assistant, this was a unique opportunity for me to get microfabrication experience at a world-class facility. Thank you for furthering my educational experience."*
- *"Because I come from a small university without the infrastructure to support some of our research projects, the access to the facilities at the CNMS is critical to our success. Moreover, the training from the staff and the annual workshops provide opportunities for my students that they would not have otherwise."*
- *"I am impressed by the scientific quality of support given by the facility staff. My group has had user projects at CNMS since 2007. This collaboration has become very valuable for us…"*

The UEC is responsible for planning the annual CNMS User Meeting with Workshops, so please hold the week of September 15-19, 2014 and visit the website for more information.

Lastly, I wish to thank our outgoing UEC Chair, Tony Hmelo (Vanderbilt University), who will continue to serve as Past Chair, ex officio and Tony Haynes the CNMS User Program Manager for making 2013 a success for the CNMS as we look forward to 2014.

Vivek Prabhu
National Institute of Standards and Technology
Gaithersburg, Maryland

About the UEC
-Contact information
-Web links

CNMS 2014 User Executive Committee

The UEC elections have concluded and our user community has elected a new committee to serve us.

Chair (succession from 2013 Vice-Chair)– Vivek Prabhu (NIST)
Vice-Chair—Nazanin Bassiri-Gharb (Georgia Tech)
Secretary—Milan Buncick (Aegis Technologies Group)

At-Large Members –
- Nina Balke (ORNL/CNMS)
- Zheng Gai (ORNL/CNMS)
- Marian (Molly) Kennedy (Clemson U.)
- Martyn McLachlan (Imperial College, London)
- Megan Robertson (U. Houston)
- Ray Unocic (ORNL/CNMS)
- Rafael Verduzco (Rice U.)

Past Chair, ex officio member: Tony Hmelo (Vanderbilt University)

Watch for upcoming
Call for Proposals

News from CNMS

Hans Christen named CNMS Director

Hans M. Christen

Hans M. Christen has accepted the position of Director of the Center for Nanophase Materials Sciences, effective January 1, 2014.

Hans joined ORNL in 2000 and led the Thin Films and Nanostructures group from 2006 to 2013. In 2013, he became Associate Director within the Materials Science and Technology Division and has managed the DOE Materials Sciences and Engineering Program from 2011 through 2013.

He received a doctorate in physics from the Ecole Polytechnique Fédérale, Lausanne, Switzerland, in 1994 for research performed at the IBM Zurich Research Lab. Hans' research has focused on the effects of epitaxial strain, spatial confinement, and interfacial mechanisms on the properties of complex-oxide thin films, in particular ferromagnetic, ferroelectric, and multiferroic perovskites. He has authored over 150 scientific publications and several patents, is a fellow of the American Physical Society, and has served on a number of NSF and DOE review panels.

“I’m very honored to be given the opportunity to serve the CNMS in this role,” Hans says. “The CNMS is a fantastic place to perform cutting-edge research, and the user community is what makes

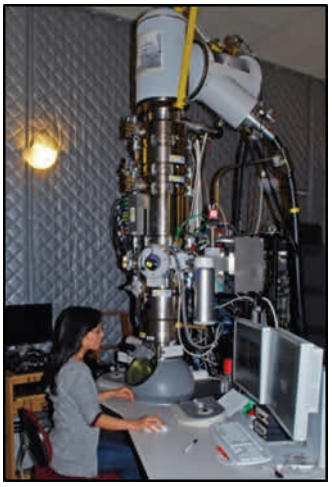
us different from other research institutions. In fact, the strong interaction between engaged users and dedicated CNMS staff leads to much more than a few isolated research projects – it defines who we are as a Center and determines the direction we're taking within the field of nanoscience. I'm really looking forward to getting to know many of the users and their research, and I'm committed to the success of the CNMS as a facility that helps advance the research goals of a broad range of productive users."

New equipment/capabilities

Microscopy Capabilities added from merging Shared Research Equipment (ShaRE) User Facility with CNMS

A suite of world-class electron microscopy and atom probe tomography capabilities were added to CNMS and now users can take full advantage of the unparalleled capabilities for sub-Å imaging, chemical analysis, and in situ/operando S/TEM characterization . The figure illustrates some of the TEM/STEM instruments added to CNMS and further details can be found on the CNMS website.

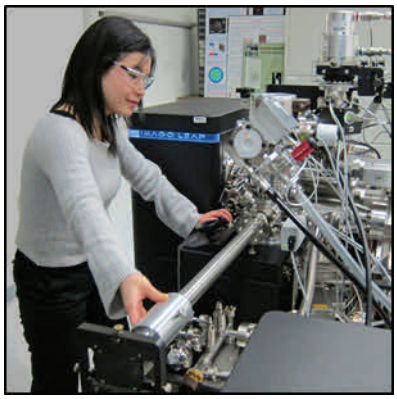
There is also the opportunity to apply atom probe tomography (APT) resources to nanostructured materials. The figure below shows the CAMECA LEAP 4000X HR instrument used for APT and a *Materials Today* highlight from E. Marquis from University of Oxford [E. Marquis et al., *Mater. Today* **13**, 34–36 (2010)] showing how APT has proven very powerful to analyze the atomic structure of bulk oxides.



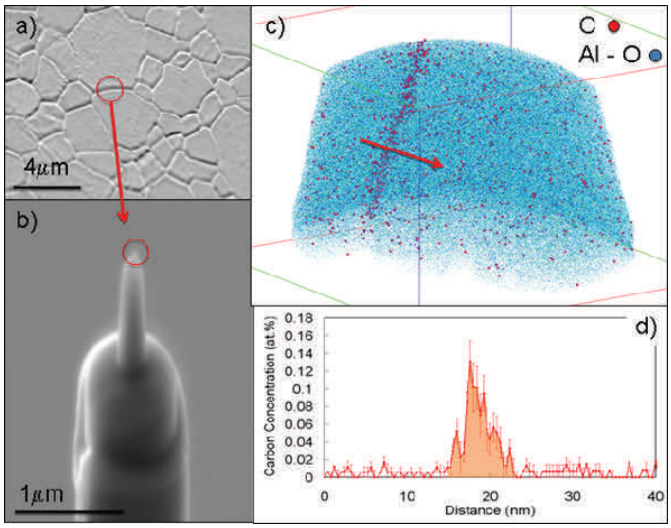
FEI Titan S 60-300

Hitachi HF 3300 Nion

Ultra STEM 100



Cameca LEAP 4000X HR

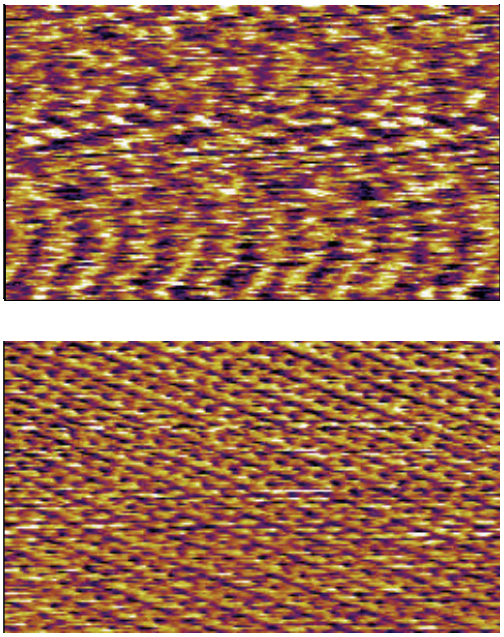


Probing the improbable: imaging C atoms in alumina: Scanning electron microscopy image of the etched polycrystalline alumina surface revealing grain boundaries; (b) Focused ion beam prepared atom-probe tomography specimen containing a grain boundary near the apex of the specimen; (c) atom-probe tomography reconstruction showing carbon segregation at this particular grain boundary; (d) Concentration profile across grain boundary along the red arrow shown in b).

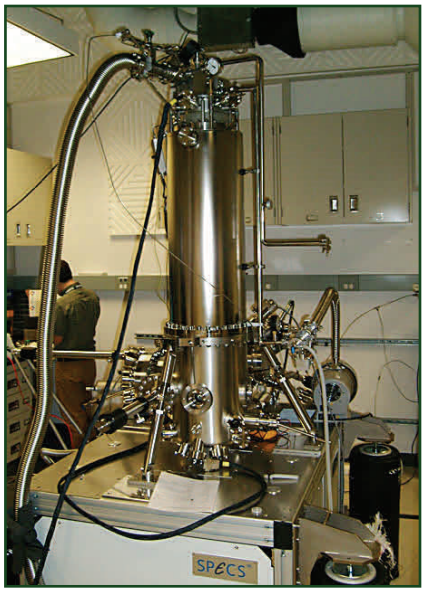
Improvements We Want to Keep Quiet

The Imaging and Nanoscale Characterization group is making a few improvements to their imaging capabilities which they would like to keep quiet. Specifically, they have moved three scanning probe microscopes to the ultra-quiet environment of the Advanced Materials Laboratory (AML). The AML was designed for high-resolution microscopy and houses ORNL's top electron microscopes. Both electron and now scanning probe microscopes in this exceptional environment are available through the CNMS user program.

The three scanning probe microscopes include two atomic force microscopes from Asylum Research (Cypher and MFP-3D) for ambient and liquid imaging and a SPECS Joule-Thomson combination scanning tunneling and non-contact atomic force microscope for cryogenic imaging in ultrahigh vacuum. The new environment for the Cypher and MFP-3D microscopes has demonstrated particular improvements for force distance and electrochemical dilatometry experiments at liquid-solid interfaces. Measurement techniques presently implemented for the SPECS combination microscope include tunneling microscopy, scanning tunneling spectroscopy, force-distance spectroscopy and Kelvin probe force microscopy. The accompanying UHV system is equipped for UHV surface preparation and organic thin film/molecular deposition. The low-noise environment particularly adds the ability to study a single molecule for extended times and enables spectroscopy to measure local electronic density of states.



Atomic imaging in liquids is demonstrated for calcite in deionized water using the Cypher atomic force microscope in its previous home (upper) and its new low-noise (lower) environment.



New Joule-Thomson cooled STM/nc-AFM for atomic imaging and spectroscopy at 1 K.

Joule-Thomson Low Temperature Scanning Tunneling and Non-Contact Atomic Force Microscope

This scanning probe microscope is a commercial microscope from SPECS GmBH, fitted with a custom-designed molecular beam deposition system. Operations temperatures range from 1-77 K with intermediate temperatures using a heater at the sample stage. The microscope head has an independent tip and sample exchange, provides optical access to the tip-sample junction, and has four contacts on the sample holder for *in-situ* transport experiments. It offers excellent drift stability at 4K of <100 pm/hr (parallel to the surface) or better. The microscope operates as a Scanning Tunneling Microscope (STM), a frequency-modulated non-contact Scanning Force Microscope (FM-ncAFM) or a combination thereof. For FM-ncAFM, a sharp metal tip is mounted at the end of a length-extension quartz resonator with Q-factors above 50000. Measurement techniques presently implemented include tunneling microscopy, scanning tunneling spectroscopy, force-distance spectroscopy and Kelvin probe force microscopy. The microscope is controlled by a flexible Nanonis/SPECS controller that allows incorporation of custom-designed imaging and spectroscopy modes.

2014A Proposal Call Results

For the 2014A Proposal Call, there were 135 proposals submitted and 104 were approved. There are also projects from prior cycles that have been granted extensions bringing our active project count to more than 290.

Postdoctoral Opportunities at the CNMS

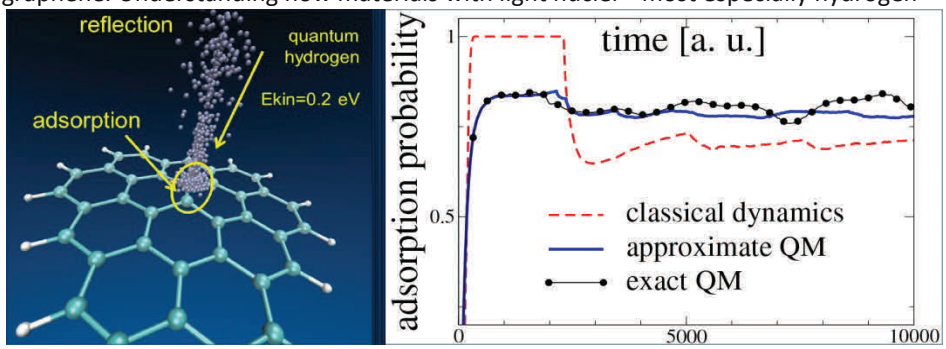
The program is open to all qualified U.S. citizens and non-U.S. citizens. Visit the website for more information and links to view other opportunities at ORNL.

CNMS in the News

New Capability Helps Overcome Limitations in the Study of Energy and Materials Applications

Researchers at the National Institute for Computational Sciences (NICS), the Center for Nanophase Materials Sciences at Oak Ridge National Laboratory (ORNL), and the University of South Carolina (USC) have developed a new computational capability to study the dynamics of prospective energy materials under diverse environmental situations. This new method employs quantum mechanics to understand how nuclear effects change the dynamics of nanoscale materials. The researchers used the NICS-managed Kraken supercomputer to perform massively parallel modeling and simulation studies in which a graphene (carbon) flake was bombarded by an ensemble of 1,000 hydrogen atoms to examine the likelihood of accumulation (adsorption) of the hydrogen on the porous graphene surface. They found that quantum nuclear effects are responsible for increased adsorption selectivity of deuterium over hydrogen on graphene. Understanding how materials with light nuclei—most especially hydrogen—behave on nanoporous surfaces is essential in the design of new materials for energy science applications.

This work has been highlighted at the University of Tennessee website and was recently published in the *Journal of Chemical Theory and Computation.* Along with Jacek Jakowski of NICS, the research team members and co-authors of the article were Sophya Garashchuk and Lei Wang of the Department of Chemistry and Biochemistry at USC, and Bobby Sumpter of the Center for Nanophase Materials Sciences and Computer Science and Mathematics Division at ORNL.

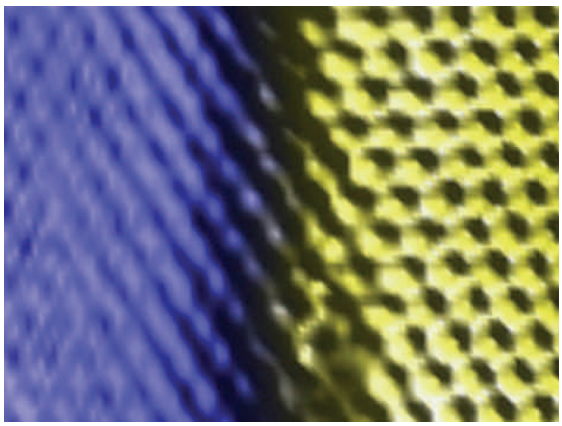


Left: A snapshot of a graphene (carbon) flake under bombardment by an ensemble of 1,000 hydrogen atoms. Right: Graph of the time-dependent absorption probability of hydrogen on the graphene flake. The results were obtained from classic dynamics, computational quantum-mechanical approximations, and from exact quantum evolution.

New Twist on Epitaxial Growth Opens New Possibilities for Two-Dimensional Materials

Researchers at Oak Ridge National Laboratory (ORNL) and the University of Tennessee (UT) have developed a new technique for creating a two-dimensional hybrid material of graphene and boron nitride that has a seamless boundary. “People call graphene a wonder material that could revolutionize the landscape of nanotechnology and electronics,” ORNL’s An-Ping Li said in a press release. “Indeed, graphene has a lot of potential, but it has limits. To make use of graphene in applications or devices, we need to integrate graphene with other materials.”

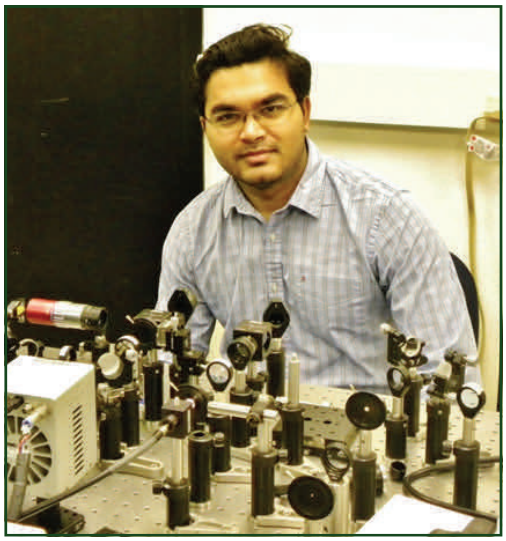
This latest ORNL/UT work, which was published in the journal *Science* (“Heteroepitaxial Growth of Two-Dimensional Hexagonal Boron Nitride Templated by Graphene Edges”) was also highlighted in the *IEEE Spectrum*.



Research Highlights

User Spotlight

Parikshit Moitra – Graduate Research Assistant at Vanderbilt University

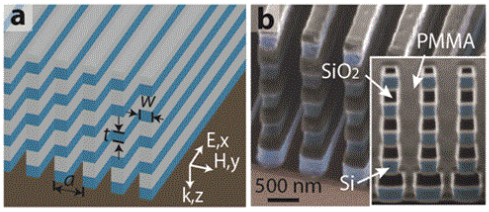


"I am currently performing research in the field of electromagnetic metamaterials, composites in which structuring is used to engineer the effective optical properties of the material. I find this field fascinating as metamaterials can exhibit novel optical properties such as negative refractive index, ultrahigh index, and zero index, properties not available in naturally occurring materials. In my recent project conducted at CNMS, I experimentally realized a zero-index metamaterial (ZIM) at optical frequencies using all-dielectric constituents, allowing us to avoid the absorption loss that is typically present in metal-based metamaterials. ZIMs are particularly interesting as they can be used to realize directional transmission and coherent spontaneous emission from incoherent emitters embedded within the material. The fabrication of the ZIM required expertise in silicon growth and patterning, including etching through thick multilayer films. Due to both the knowledge base and tool availability, CNMS was the ideal place to perform this research. The ZIMs created at CNMS had tight dimensional tolerances and the CNMS staff provided expert guidance and assistance in their realization. I especially want to acknowledge the guidance from Ivan Kravchenko, Nick Lavrik, and Dayrl Briggs, all of whom I worked closely with in accomplishing this research."

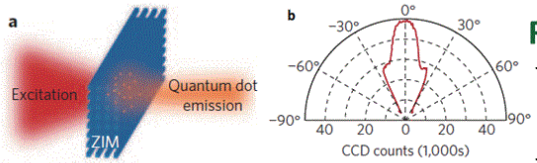
Parikshit Moitra graduated with Bachelor of Engineering from Jadavpur University, India and is currently pursuing his Ph.D. at Vanderbilt University under the guidance of Prof. Jason Valentine. The goal of his research is to explore novel low-loss metamaterials and photonic devices using all-dielectric implementations.

User Project Highlight

Realization of an All-Dielectric Zero-Index Optical Metamaterial



a) Diagram of the zero-index metamaterial structure with a unit cell period of a = 600 nm and w = t = 260 nm.
b) False color focused ion beam image of the real sample. The inset shows a cross-section of the structure after a polymer filling. The fabricated sample has 11 alternating Si/SiO_2 layers.



a) Schematic of laser pumped PbS semiconductor quantum dot emission from within the ZIM structure.
b) Experimental polar diagram of the quantum dot emission distribution within the ZIM, showing enhanced directivity of spontaneous emission.

Scientific Achievement

The first all-dielectric zero-index metamaterial (ZIM) has been experimentally demonstrated at optical frequencies, which exhibits a nearly isotropic low-index response for a particular polarization, resulting in angular selectivity of transmission and spontaneous emission.

Significance and Impact

The realization of impedance-matched zero-index metamaterials at optical frequencies allows development of angularly selective optical filters, directional light sources and large area single-mode photonic devices. Furthermore, the advent of such metamaterials provides a new route to designing novel optical media with both low absorption loss and isotropic optical properties.

Research Details

- **CNMS Capability:** ZIM fabrication began with a multilayer stack of 11 alternating layers of α-Si and SiO_2 followed by patterning using an advanced e-beam lithography system and plasma reactive ion etching.
- The optical ZIM serves as an angular optical filter while also enhancing the directivity of spontaneous emission from quantum dots embedded inside the structure. The experimental results, together with numerical calculations, serve as direct evidence of impedance-matched, near-zero index within the metamaterial.

Work was performed at the Center for Nanophase Materials Sciences and Vanderbilt University.

P. Moitra, Y. Yang, Z.Anderson, I.I.Kravchenko, D.B.Briggs, J.Valentine, *Nature Photonics*, **2013**

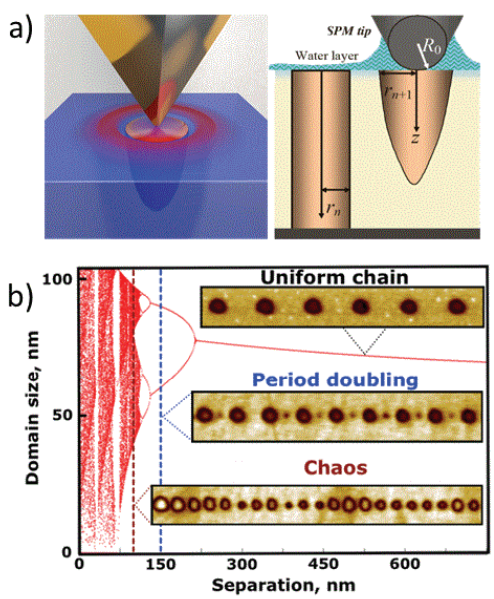
CNMS Staff Spotlight

Anton Ievlev – Postdoctoral Researcher at the Center for Nanophase Materials Sciences

"I study processes of polarization reversal in ferroelectric materials. In my research, I use Scanning Probe Microscopy which reveals processes occurring at the nanometer scale. Results of this research can be used for development of a new generation of non-linear optical devices and ultrahigh density data storage devices. CNMS has a great set of scanning probe microscopes with outstanding characteristics and unique techniques that allows advanced investigation and provides excellent experience for users. Moreover CNMS offers great opportunities from theoretical modeling to experimental investigations using neutron scattering. All these factors create an attractive atmosphere for scientists all around the world."

Anton Ievlev received his B.S., M.S. and Ph.D in Physics of condensed matter from Ural Federal University, Ekaterinburg, Russia. For his graduate research, he studied ferroelectric single-crystals of lithium niobate and strontium-barium niobate using scanning probe microscopy. Part of his graduate research was done at the CNMS as a user project. Anton is currently a Postdoctoral Associate in the CNMS Imaging and Nanoscale Characterization group.

Chaotic physics in ferroelectrics hints at brain-like computing



a) Schemes of the switching by SPM tip and long-range domain-domain interaction
b) Bifurcation diagram of the domain size in the chain vs period in the chain

Work was performed at the Center for Nanophase Materials Sciences, ORNL

Scientific Achievement

Complex spatial dynamics observed during scanning probe microscopy (SPM) tip-induced polarization reversal on the surface of single-crystalline $LiNbO_3$ enables a new generation of memristive, memcapacitive and neuromorphic devices.

Significance and Impact

Ferroelectric materials are widely used in optic, acoustic and data storage devices. Intermittency, quasiperiodicity and chaos revealed during writing 1D chains of domains reveals that polarization switching is a coupled physical and surface electrochemical process mediated by long-range ionic flows. These long-range interactions enable new ferroelectric-based architectures with potential for information processing.

Research Details

- SPM tip-induced writing of 1D domain chains has been studied on the surface of a 20-µm-thick $LiNbO_3$ single crystal with dependence on the period in the chain, temperature and relative humidity.
- Observed long-range interaction has been explained by the influence of the depolarization field of the initial domains in the chain mediated by ionic flows.

A.V. Ievlev, S. Jesse, A.N. Morozovska, E. Strelcov, E. A. Eliseev, Y. V. Pershin, A. Kumar, V. Ya. Shur, S. V. Kalinin, *Nature Physics* **2013**, 10.1038/nphys2796

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