

Nanomaterials

Room Naupaka Salon 4 - Session NM-WeM1

Nanocharacterization

Moderator: Byron Gates, Simon Fraser University

8:00am NM-WeM1-1 Phase Transition Study of 2D NbSe₂ by in-situ TEM/STEM, *Moon Kim*, The University of Texas at Dallas

Two-dimensional (2D) transition metal dichalcogenides (TMDs) are a family of layered materials with an X-M-X structure, where M and X are transition metal and chalcogen, respectively. TMDs have attracted tremendous interest due to their unique electronic, magnetic, and optical properties with an atomic layer limit depending upon structural elements. The stacking sequence of layers leads to the formation of polytypes such as 1T, 2H, 4H, and 3R, sometimes resulting in significantly different electronic and optical properties.

NbSe₂ is one of the attractive 2D TMDs. It is a superconducting material with a high-superconducting-transition temperature (T_c) of about 7.0 K and shows metallic characteristics at room temperature. The weak van der Waals force between layers allows superconductivity and charge density wave (CDW) with a transition temperature of about 33K. Interestingly, its phase alters its magnetic and electrical properties. However, its thermal stability, which is important for phase engineering and synthesis, has not been intensively examined.

This study investigated the defect dynamics and thermal evolution of NbSe₂ under vacuum by in-situ heating Scanning Transmission Electron Microscopy (STEM). Low thermal stability of the NbSe₂ was confirmed by direct observation of 2H to 1T phase transition and defects induced by inversion domain boundaries. Interlayer gap expansion, Se atom desorption, and intercalation to the atomic layer gap were also observed. Experimental details will be presented and discussed considering its potential applications.

8:20am NM-WeM1-2 Development of Atomic Holography Microscope CoDELMA, *Hiroshi Daimon*, Toyota Physical and Chemical Research Institute, Japan; *H. Momono*, National Institute of Technology, Japan; *H. Matsuda*, Institute for Molecular Science, Japan; *L. Tóth*, University of Debrecen, Hungary; *Y. Masuda*, *K. Moriguchi*, *K. Ogai*, APCO Ltd., Japan; *Y. Hashimoto*, *T. Matsushita*, Nara Institute of Science and Technology, Japan

INVITED

Atomic resolution holography [1] is a powerful technique that can analyze the local stereoscopic atomic arrangement around not only constituent atoms in a crystal but also isolated atoms such as dopants, which has been impossible to be analyzed so far. In photoelectron holography, the photoelectrons emitted from the target atom are used as the reference wave, and the waves scattered by the surrounding atoms are used as the object waves, and the angular distribution (hologram) of their interference patterns can be used to directly derive the three-dimensional atomic arrangement around the emitter atom. Because the atomic-resolution holography required synchrotron radiation facilities so far, its application has been limited. Hence we are developing Atomic Holography Microscope CoDELMA, which can make atomic holography experiment anywhere using a scanning electron microscope (SEM) electron beam.

Atomic-resolution holography microscope is realized by a combination of a SEM and a new two-dimensional electron spectrometer CoDELMA [2]. CoDELMA is the only two-dimensional electron spectrometer that can analyze the angular distribution of high-energy electrons with a high-energy-resolution width of $\Delta E/E = 1/2000$ over a wide two-dimensional angular range of $\pm 50^\circ$ at once. This new microscope enables us to easily measure the atomic resolution holography at each nano region observed with SEM.

References:

- [1] H. Daimon, "Atomic-resolution holography for active-site structure", *Jpn. J. Appl. Phys.* 59, 010504 (2020).
- [2] H. Matsuda, L. Tóth, and H. Daimon, "Variable-deceleration-ratio wide-acceptance-angle electrostatic lens for two-dimensional angular and energy analysis", *Rev. Sci. Instrum.* 89, 123105 (2018).

9:00am NM-WeM1-4 Development of a Nanocomposite Based Films with Antifungal Properties and Containing Encapsulated Nanoemulsion Based on Essential Oils: Effect of Combined Treatment with γ -Irradiation, *Monique Lacroix*, INRS, Canada

Nanocomposite film based on chitosan and nanocrystal cellulose (CNCs) was developed as matrices for incorporation of essential oils as antimicrobial compounds. The addition of CNCs in chitosan based-film has permitted to reinforced the physico-chemical properties of the films. An optimal concentration of 5% (w/w) NNCs improved by 26% the tensile strength (TS) and decreased by 27% the water vapor permeability of the films. A three factor central composite design (CNCs concentration ; microfluidization pressure ; number of cycle of microfluidization) with five levels was designed to optimize the microfluidization process. Microfluidization has permitted to reduce the CNC-chitosan aggregates and improved the mechanical properties of the nanocomposite films by 43%. Two antifungal formulations based on tea tree or on mint in combination with thyme essential oils (Eos) under nano emulsion were developed. When encapsulation under nanoscale, the size of the drop of the antimicrobial formulation was reduced from 219 to 71 nm. The encapsulation efficiency improved from 37 to 83% and the antifungal efficiency was improved from 32 for 3 days to 81% for over one month showing, that the doses required to ensure the biological activity was reduced significantly. The film also showed an effectiveness and slow release of EOs during storage. Combination of active films with γ -irradiation (750 Gy) was synergistic and caused 4 log UFC/gr reduction of fungi for more than 8 weeks of storage. These results showed that the bioactive films and γ -irradiation combination has commercial potentiality to extend shelf life of rice products.

9:20am NM-WeM1-5 Tuning Spin Interactions of Magnetic Molecules on Au(111) by Atomic Adsorbates, *Min Hui Chang*, Korea University, Republic of Korea; *Y. Chang*, Korea Advanced Institute of Science and Technology, Republic of Korea; *N. Kim*, *Y. Kim*, Korea Advanced Institute of Science and Technology, Republic of Korea; *S. Kahng*, Korea University, Republic of Korea

Sensing and tuning spin interactions of magnetic molecules have been actively studied due to possible applications in molecular spintronic and quantum computer. On metallic surfaces, exchange interactions between molecular spins and spins of conduction electrons of substrates have been detected as Kondo resonances at Fermi level. It has been demonstrated that Kondo resonances can be tuned by small molecule bindings, but not by atomic adsorbates. Here, we demonstrate that the Kondo resonances of Co-porphyrin on Au(111) can be tuned by various magnetic atomic adsorbates and be detected using scanning tunneling microscopy and spectroscopy (STM and STS). We observed several adsorbate-induced complexes in STM images, and proposed their atomic structures based on density functional theory calculation results. Our STS results were explained with the redistribution of unpaired spins of Co-porphyrin by atomic adsorbates. Our study shows the spin states and interactions of metallo-porphyrin can be tuned by magnetic atomic adsorbates.

9:40am NM-WeM1-6 Critical Utilization of Scan Probe Microscopy (Spm) Methods for 2d Materials Research, *Jason Tresback*, *J. Deng*, Harvard University

Atomic Force Microscopy (AFM) is a powerful, and critical technology to characterize a diversity of nanomaterials across many interdisciplinary research fields. The use of AFM is rapidly evolving into a myriad of different modes for highly localized (<30nm) property measurement techniques in addition to roughness, and topography with sub-nm spatial resolution. In order to support the momentum of quantum materials research, advanced modes of Scan Probe Microscopy (SPM) techniques are being used to measure electrical, magnetic, and optical properties of new materials as well as Scan Probe Lithography (SP-L) methods for device fabrication. A highly active area of quantum materials research is the characterization and device fabrication of 2d materials (Graphene and TMDs). The goal of this talk is to provide an overview of how SPM is applied for the advancement of 2d materials research, utilizing Kelvin Force (KPFM), Electrostatic Force (EFM), Piezo Force (PFM), and Scattering Near Field Microscopy (s-SNOM) and other advanced modes. The main focus will be on the exquisite, direct imaging of Moire lattice patterns in twisted bi-layer (t-BL) materials by SPM technologies. Each of the imaging modes used to demonstrate direct visualization and angle measurements of t-BL will be discussed. Furthermore, the impact of a closed cell environmental chamber with Ar/N₂ gas flow for each of these modes will be demonstrated. Some

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examples of direct writing and patterning of 2d structures using a lithography mode for device fabrication will also be presented.

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