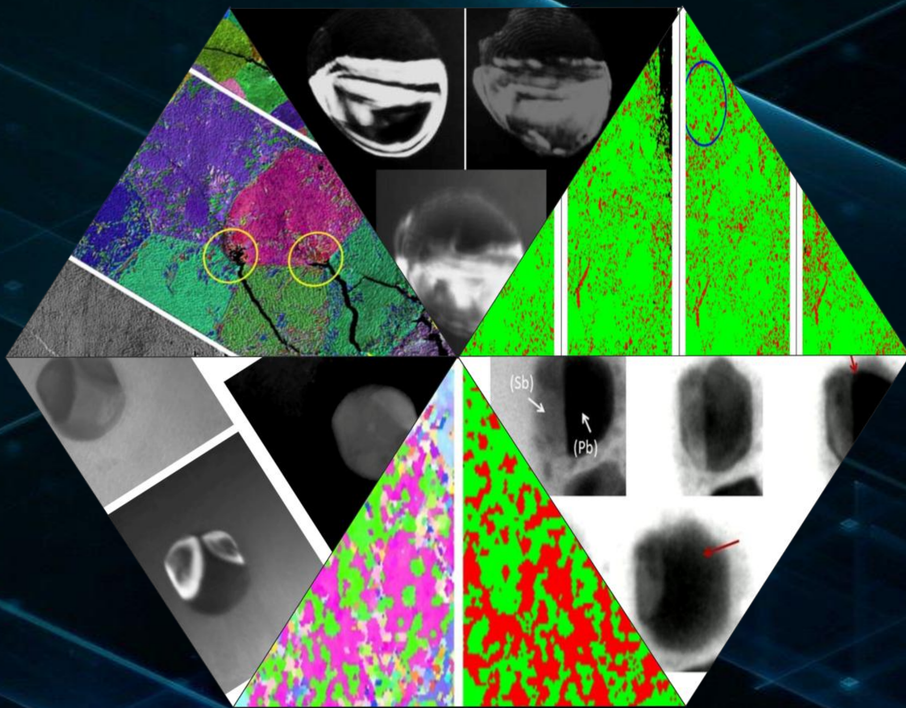
Abstract Booklet



Online In-situ Electron Microscopy -2021

8-9 July, 2021

Organised by

IIT Kanpur in association with IIT Kharagpur

&

Electron Microscopy Society of India (EMSI)

Welcome Preface

In situ Electron Microscopy, also known as *Operando* observation, is an important tool today to investigate materials and biological systems. In general, electron microscopic observations are made under static conditions. The transition from static to dynamic microscopy has evolved over last 30 years, making *in situ* electron microscopy a powerful tool for researchers trying to understand various phenomena with application of wide range of variable; temperature, load, electric field, magnetic field etc to study the effect of these variables on various materials related phenomena. This has allowed understanding the dynamics of these phenomena in details. Hence, *in situ* electron microscopy has opened up the door to new world of electron microscopy with development of new tools, techniques, machinery and observation, leading to gamut of new developments in the last decade.

In order to take a stock of the recent development and future endeavour, Department of Materials Science and Engineering, Indian Institute of Technology Kanpur in association with Department of Metallurgy and Materials Engineering of Indian Institute of Technology Kharagpur, are organizing Online Symposium on *in-situ* Electron Microscopy under the banner of Electron Microscope Society of India (EMSI) from 8-9 July, 2021. Current pandemic has made our life difficult and it is not possible to organize symposium in person and hence, the Online mode has become convenient, useful and in fact successful of disseminating knowledge to the researchers working on this advanced field as well as scholars who would like to take up *in situ* microscopy in their future research.

This symposium spanning over two days with three sessions each day, will encompass 24 speakers across the globe working on various aspects of *in situ* electron microscopy. The speakers are chosen from eminent researchers working in academia, industry and research labs from India and abroad. This is expected to provide the depth and breadth of the field with emphasis on the basic of the technique and applications. In addition, a Panel Discussion is being organized with panellists from academia, funding agencies as well as research labs to channelize our resources garnering momentum in order to set up world class facilities on *in situ* electron microscopy in the country. An e-poster session is also organized to encourage young researchers showcasing their research and interacting with the experts. This is expected to provide young researchers a unique opportunity to learn from the experts and even build network.

We hope that this event will be successful in bringing all involved with *in situ* electron microscopy in India and abroad. We would like to thank IIT Kanpur, IIT Kharagpur, EMSI, speakers, session chairs as well as Industry partners for their generosity and co-operation.

Krishanu Biswas (IIT Kanpur)

Chandra Sekhar Tiwary (IIT Kharagpur)

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Online Symposium on In-situ Electron Microscopy (EMSI-2021)
8-9 July, 2021

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Session I	**Chairperson: R. Tewari, BARC Mumbai**
09:15 – 10:00 AM	- David Muller, USA
10:00 – 10:45 AM	- Partha Ghosal, India
10:45 – 11:30 AM	- Y. Oshima, Japan
11:30 – 12:15 AM	- Abhay Gautam, India
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Session II	**Chairpersons: N.K. Mukhopadhyay, IIT BHU, K. Mondal, IIT Kanpur**
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Valedictory	**07:30 – 07:45 PM**

Online Symposium on In-situ Electron Microscopy (EMSI-2021)
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List of E-Posters Presentation

E-Poster No.	Presenter	Title
EP-01	Debadarshini Samantray	Controlling polarization through defects in Si integrated $BaTiO_3$
EP-02	Dhananjay Kumar Yadav	In-situ Electron Back Scattered Diffraction of SS316L
EP-03	Khushubo Tiwari	Phase Transformation and Influence of Solid-Solution Properties of Ag-Cu Alloy Nanoparticle Embedded in Ni matrix
EP-04	Roopam Jain	In situ tensile deformation of silicon doped $Fe_{50-x}Mn_{30}Co_{10}Cr_{10}Si_x$ high entropy alloy
EP-05	Vivek Kumar Sahu	Micro-mechanism of damage evolution in commercially pure titanium: an in-situ study
EP-06	Sumita Santra	Electron microscopic study on nano sized precipitation during ageing in Alloy 617
EP-07	Rupesh Kumar	Grain Boundary Precipitate Embrittlement in Alloy 617 due to Partial Solution Annealing
EP-08	Sudha Saini	Electrical properties of YSZ/double perovskite composites for solid oxide fuel cell (SOFC) application

Abstracts of Invited Speakers Online Oral Presentations

Phase and Microstructural Characterization using Advanced Techniques: in-situ SEM and TEM-PED

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ABSTRACT: Continuous advances in electron microscopy has led to the development of new advanced techniques which have enabled us to get new information about the microstructure which was not available to us before thereby enabling us to have a much more comprehensive understanding of the material at the microstructural level. And, this understanding is the key to manipulate the material properties according to the functional requirement.

This study attempts to highlight the use of two such new /advanced techniques; i) Precision Electron diffraction in TEM. This is a new technique to image the sample in complete zone axis in diffraction mode to get the right orientation of the crystal and match them with the crystal structure diffraction library to get the precise grains orientation in this small volume of the material. ii) In-situ heating / cooling technique in SEM. The in-situ heating cooling technique in SEM allows us to observe the morphological and structural changes as they happen in a material during exposure to varying temperatures. This enables us to have a sound understanding of the material behavior as function of temperature.

These techniques have been used for morphological and structural characterization of high temperature coating materials which include Pt-aluminide (Pt-Al) and Fe-Cr modified silicide and other examples will be discussed, if we have time.

Pt-aluminide (Pt-Al) coatings – They are applied on the Ni-based superalloy components operating in the hot-sections of gas turbine engines. The microstructure of the coating is comprised of the intermetallic B2-NiAl phase. The coating undergoes dynamic phase transformation from B2→ reversible B2/$L1_{0\ \mathrm{marten}}$ site + γ'-Ni_3Al →γ + γ'-Ni_3Al with thermal exposure. In the present study, we are doing i) in-situ-examination of the effect of temperature on the above phase transformations, ii) identification of local variation of phases/ orientation in the microstructure using PED, & iii) in-situ-examination of the effect of thermal cycling (in high temperature regime) on the structural stability of the coating substrate system.

Fe-Cr modified silicide coatings – They are applied on Nb-base alloys such as C103 for providing oxidation protection at high temperatures. Extensive studies have been done on their ability to withstand high temperature in aerospace applications. However, their behaviour in low temperature regime (RT to sub zero) is equally important. The present study involves in-situ -examination of the effect of cryogenic temperature on the microstructure and phase stability of the silicide coating using the above mentioned techniques.

Mechanical properties of Pt atomic chains measured by in-situ TEM technique

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One- and two-dimensional materials have different bonding states from bulk counterparts, so the bond stiffness is expected to be different for such low dimensional materials. The bond stiffness is important for understanding the mechanical response, but it is difficult to measure because of local information. To solve this problem, we developed an in-situ TEM holder equipped with a quartz resonator as force sensor (Fig. 1a) [1,2]. By using this in-situ TEM holder, we observed platinum monatomic chains at atomic scale simultaneously with measuring the equivalent spring constant based on frequency modulation (FM) method.

A quartz length-extension resonator (LER) was used to measure the stiffness of Pt monatomic chains from its frequency shift. The stiffness of the atomic chain suspended between the edge of LER and the fixed counter base can be measure precisely with very small oscillation amplitude (about 30 pm). The atomic resolution TEM images (Fig. 1b) were captured simultaneously with measuring the conductance and stiffness.

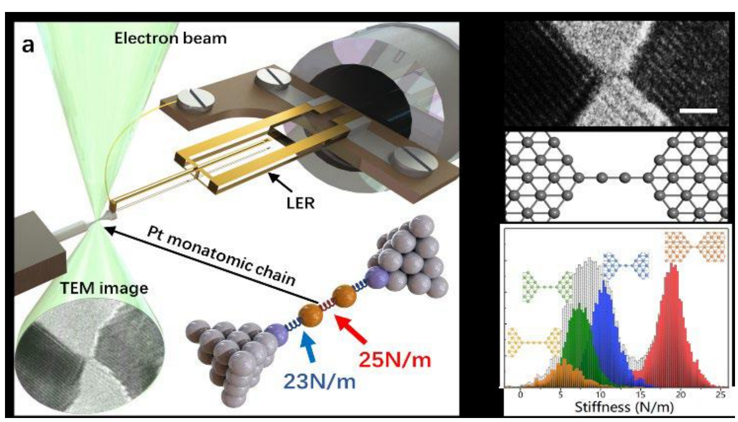

Fig.1. (a) Schematic illustration of the experimental setup. (b) TEM image of at atomic chain with 4 atoms and corresponding atomic configurations of the chain. (c) Stiffness histograms of the Pt monatomic chains.

The stiffness of atomic chains with 2-5 atoms were obtained (Fig. 1c). By subtracting the stiffness of the electrodes supporting the monatomic chain from the measured stiffness, we found that the stiffness of a Pt monatomic chain varied with the number of the constitute atoms in the chain. We investigated the stiffness of about 150 Pt monatomic chains for reproducibility and confirmed that the middle bond stiffness (25N/m) in the chain was slightly higher than that of the bond connect to the suspending tip (23N/m). In addition, the maximum elastic strain of individual bond in the chain was as large as 24% [3].

Fig.1. (a) Schematic illustration of the experimental setup. (b) TEM image of at atomic chain with 4 atoms and corresponding atomic configurations of the chain. (c) Stiffness histograms of the Pt monatomic chains

Our developed method also enabled us to measure the critical shear stress of gold (Au) nanocontact by measuring the energy dissipation. We measured it for Au nanocontact with the axis of the [111] and [110] direction and found that the critical shear stress was 0.94 ± 0.1 GPa, which corresponds to the slip along the [112] direction on the (111) plane [4].

[1] K. Ishizuka et al, Appl. Phys. Express 13 (2021) 025001.
[2] J. Zhang et al, Nanotechnology. 31 (2020) 205706.
[3] J. Zhang et al, Nano letters. 21 (2021) 3922-3928.
[4] J. Liu et al, Appl. Phys. Express 14 (2021) 075006.

Study of interface structure and dynamics using in-situ transmission electron microscopy

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Few techniques offer the resolution and sensitivity required to study the detailed structure and dynamics at a solid-solid interface. In-situ electron microscopy techniques have always been the tool of choice for such studies. Recent development in detector as well as holder technologies have enabled better controlled in-situ experiments with higher spatial and temporal resolution. In this work we present the dynamics of interface fluctuation at higher temperature and possibly due to the influence of the high-energy electron beam. The in-situ transmission electron microscopy images from experiments on different material system will be used to gain insight to the unit process responsible for such interfacial motion and effect of this fluctuation on the structure of the interface.

References:

- JM Howe, A Gautam, K Chatterjee, F Phillipp: Atomic-level dynamic behavior of a diffuse interphase boundary in an Au–Cu alloy, Acta materialia 55 (6), 2159-2171
- Stephen M.Foiles. J.J.Hoyt: Computation of grain boundary stiffness and mobility from boundary fluctuations, Acta Materialia, Volume 54, Issue 12, July 2006, Pages 3351-3357
- A Gautam, C Ophus, F Lançon, P Denes, U Dahmen: Analysis of grain boundary dynamics using event detection and cumulative averaging, Ultramicroscopy 151, 78-84

Exploiting automatic image processing to understand the stability of supported nanoparticles

James P. Horwath[1], Leena Vyas[1], Peter W. Voorhees[2], and **Eric A. Stach**[1, 3]

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The activity and lifetime of heterogeneous catalysts are intimately linked with their structural stability in reactive environments. However, it can be challenging to both understand and predict how reactive environments lead to nanoparticle coarsening via center of mass motion and Ostwald ripening and how evaporation can lead to mass loss. In this work, we develop and exploit advanced data analysis tools to track the temporal evolution of nanoparticles as a function of time, temperature, and reactive environment using transmission electron microscopy. The first portion of the talk will describe our development of a fast and highly accurate image segmentation approach based on deep learning. We will describe how a systematic investigation of dataset preparation, neural network architecture, and accuracy evaluation can lead to a generalizable tool for determining the size and shape of nanoparticles in high pixel resolution bright-field TEM images.[1]. In the second half of the talk, we will show how we exploit this analysis approach to generate rich data regarding the complexities of nanoparticle coarsening, ripening, and evaporation. In particular, we show how Au nanoparticles created through colloidal synthesis approaches [2] undergo a combination of both evaporation and diffusive mass transport. We have developed an analytical model that describes this process and shows how both local and long-range particle interactions through diffusive transport affect the evaporation process. The extensive data of the time evolution of several hundred particles allows us to determine physically reasonable values for the model parameters, quantify the particle size at which the Gibbs-Thompson pressure accelerates the evaporation process, and explore how individual particle interactions deviate from the mean-field model. [3] Recent extension of the approach to evaporated Au nanoparticles and Pt particles in reactive environments will be described.

References:

1. Horwath, J.P., Zakharov, D.N., Megret, R. and Stach, E.A., 2020. Understanding important features of deep learning models for segmentation of high-resolution transmission electron microscopy images. npj Computational Materials, 6(1), pp.1-9.
2. Elbert, K.C., Jishkariani, D., Wu, Y., Lee, J.D., Donnio, B. and Murray, C.B., 2017. Design, self-assembly, and switchable wettability in hydrophobic, hydrophilic, and janus dendritic ligand–gold nanoparticle hybrid materials. Chemistry of Materials, 29(20), pp.8737-8746.

3. Horwath, J.P., Voorhees, P.W. and Stach, E.A., 2021, Quantifying Competitive Degradation Processes in Supported Nanocatalyst Systems, Nano Letters, in press.

In situ TEM – Using state-of-the-art imaging approaches to follow mechanically and thermally induced processes in nanocrystalline metals and metallic glasses

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In situ transmission electron microscopy (TEM) techniques have developed tremendously during the last decade providing the possibility to use the full power of electron microscopy to follow the structural, chemical and morphological changes during reactions and processes, thus providing direct insight into the related mechanisms. In particular, thermally, electrochemically or mechanically induced transformations/reactions have been studied extensively with the advances of MEMS based *in situ* setups for electron microscopy. This has resulted in a significantly improved materials understanding. With this presentation, I will introduce some of our recent results to highlight the possibilities *in situ* TEM provides for understanding processes in nanocrystalline metals and metallic glasses focusing on crystallization, grain growth and segregation/precipitation phenomena. I will illustrate how *in-situ* TEM can be combined with ACOM-STEM, EFTEM or PDF analysis to quantitatively follow different aspects of these processes. As part of this, effects of the electron beam and the environment inside the TEM on *in situ* measurements, both in terms of structure, materials properties and kinetics, will be critically discussed.

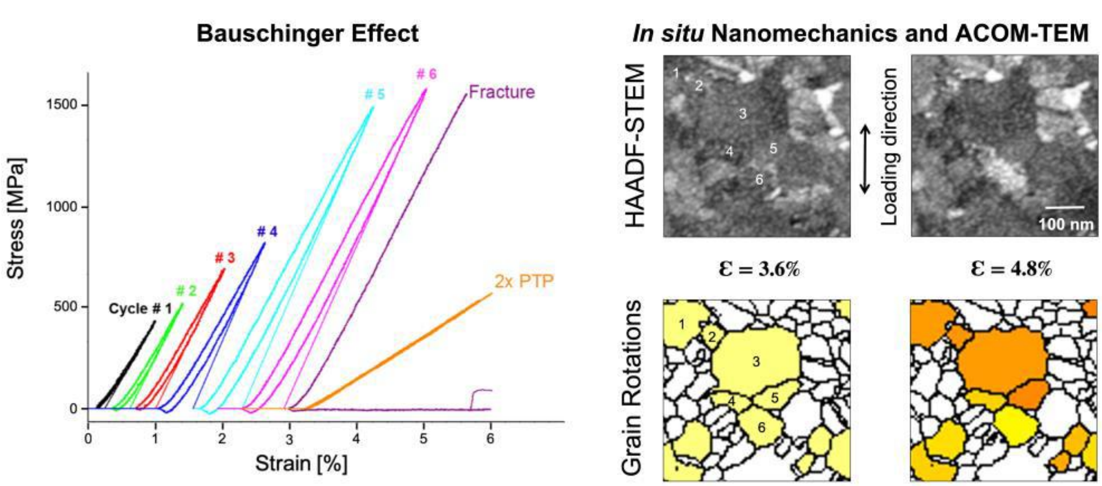

Figure 1: *In-situ* mechanical testing of nanocrystalline Pd: mechanical response indicating a strong Bauschinger effect and successive (partially) reversible grain rotation in response to the mechanical deformation.

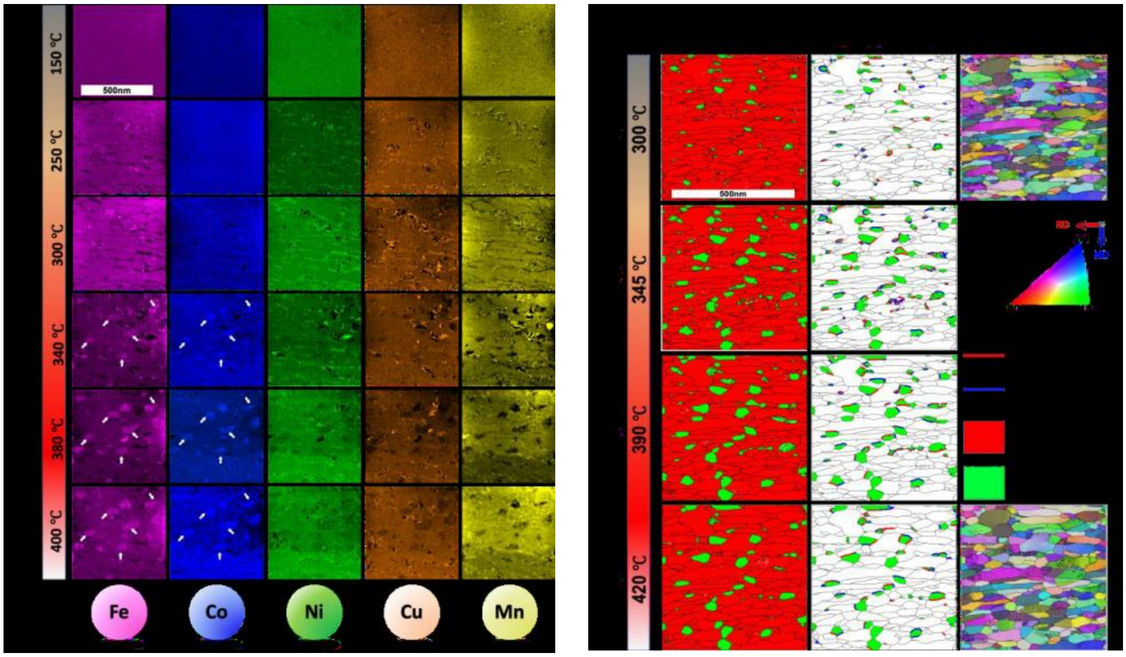

Figure 2: *In-situ* TEM annealing of a HPT-deformed HEA: EFTEM-SI analysis of the complex segregation of precipitation process and ACOM-STEM analysis of the corresponding crystallographic changes.

In-situ **tensile and cyclic testing in scanning electron microscope**

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Seeing is believing! Many developed classical theories in materials science and metallurgy have been proven indirectly. Modern techniques in in-situ characterization can be a way to observe related phenomena experimentally and prove or modify related theories. In addition, in order to develop next-generation high-performance materials, it is essential to gain knowledge about weak-points in the microstructure of conventional materials. In-situ testing is a very powerful tool for this purpose. This presentation aims to shed light on some development in in-situ tensile and cyclic testing combined with digital image correlation, which was used for deeper understanding of the behavior of individual microstructure features in metallic alloys under mechanical loading.

Fracture of Two-Dimensional Materials

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Two-dimensional (2D) materials, such as Graphene, hBN and MoS_2, are promising candidates in a number of advanced functional and structural applications, owing to their exceptional electrical, thermal and mechanical properties. Understanding mechanical properties of 2D materials is critically important for their reliable integration into future electronic, composite and energy storage applications. However, it has been a significant challenge to quantitatively measure mechanical responses of 2D materials, due to technical difficulties in the nanomechanical testing of atomically thin membranes. In this talk, we will report our recent effort to determine the engineering relevant fracture toughness of graphene with pre-existing defects, rather than the intrinsic strength that governs the uniform breaking of atomic bonds in perfect graphene. Our combined experiment and modeling verify the applicability of the classic Griffith theory of brittle fracture to graphene. Strategies on how to improve the fracture resistance in graphene, and the implications of the effects of defects on mechanical properties of other 2D atomic layers will be discussed. More interestingly, stable crack propagation in monolayer 2D *h*-BN is observed and the corresponding crack resistance curve is obtained for the first time in 2D crystals. Inspired by the asymmetric lattice structure of *h*-BN, an intrinsic toughening mechanism without loss of high strength is validated based on theoretical efforts. The crack deflection and branching occur repeatedly due to asymmetric edge elastic properties at the crack tip and edge swapping during crack propagation, which toughens *h*-BN tremendously and enables stable crack propagation not seen in graphene.

Operando observation of reversible oxygen migration and phase transitions in ferroelectric $Hf_{0.5}Zr_{0.5}O_2$ thin films

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Unconventional ferroelectricity, robust at reduced nanoscale sizes, exhibited by hafnia-based thin films presents tremendous opportunities in nanoelectronics. However, the exact nature of polarization switching remains controversial. We investigate epitaxial $Hf_{0.5}Zr_{0.5}O_2$ (HZO) capacitors, interfaced with oxygen conducting metals ($La_{0.67}Sr_{0.33}MnO_3$, LSMO) as electrodes, using atomic resolution electron microscopy while *in situ* electrical biasing (*1*).

We utilize differential phase contrast (DPC) STEM imaging in conjunction with *in situ* biasing, and follow directly interpretable oxygen dynamics at an atomic scale. These are complimented with operando nanobeam x-ray diffraction experiments.

We concretely show that:

a) Oxygen voltammetry is very much intertwined with ferroelectric switching in these devices.
b) HZO acts as a fast conduit for oxygen migration between reactive electrodes (such as LSMO/TiN), rendering ferroelectric switching possible. However, under longer time scales (DC stressing), HZO acts as sink and source of oxygen vacancies resulting in structural phase transitions.
c) Oxygen voltammetry still exists even when one of the electrodes is not reactive (Au, for e.g.). But in this case even in the short-term (milliseconds) HZO acts as a sink or source of oxygen vacancies.

In addition to these, I will also discuss the insights obtained on the mechanical response of HZO during ferroelectric switching, and corresponding "strange" electromechanical coefficients.

Our results unmistakably demonstrate that oxygen voltammetry is very much intertwined with polarization switching in HZO. Time permitting, I will also discuss some results on in-situ heating of these materials again using DPC STEM to identify the ferro-para phase transitions in these unconventional ferroelectrics (*2*).

Reference

1. P. Nukala et al., Reversible oxygen migration and phase transitions in hafnia-based ferroelectric devices, *Science*, 372, pp: 630 (2021).
2. P. Nukala et al., In situ heating studies on temperature-induced phase-transitions in epitaxial $Hf_{0.5}Zr_{0.5}O_2$/ $La_{0.67}$ $Sr_{0.33}MnO_3$ heterostructure, *Appl. Phys. Lett.*, 118, 062901 (2021).

Understanding structure of dispersoids in oxide dispersion strengthens materials

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Oxide dispersion strengthened (ODS) alloys are possibly the most promising next generation high strength structural materials. The authors have been involved for a decade in developing a host of such materials including ODS ferritic and ferritic-martensitic steels and Ni base super alloys using powder metallurgical (PM) processes. In all cases, the dispersoids are basically made of yttria owing to their exceptional high temperature stability. Although bcc phase of yttria is the stable phase at room temperature, our recent results show that it transforms to a metastable monoclinic phase during high energy ball milling, a mandatory PM step to production of ODS materials. It has also been observed that to react with other oxide forming matrix elements selectively during the high temperature PM processing steps to form complex oxides. These fine complex oxide particles impart high temperature creep strength to the material by impeding motion of dislocations. Therefore it is of utmost importance to learn in detail about their structure and chemistry down to atomistic detail. In this presentation, some recent results of probe-aberration-corrected sub-Å resolution TEM imaging of the dispersoids will be discussed. Intriguing aspects of transformation of the ball-mill induced metastable monoclinic yttria back to its cubic phase during in-situ heating experiments in XRD and TEM will also be discussed.

In situ experiments across real and reciprocal space aided by crystal plasticity simulations

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Design and development of new metallic materials and processes for various engineering applications involve understanding the effect of structure and microstructure on their mechanical properties and the underlying deformation and damage micro-mechanisms. In situ or in operando experiments offer a direct evidence of operative micro-mechanisms during thermomechanical treatment that have traditionally been deciphered by post mortem characterization. Different micro-mechanisms of deformation in crystalline metals and alloys leave a trail by altering the structure and/or microstructure and this has been monitored using diffraction and microscopy respectively. Therefore, in situ deformation experiments have focused on carrying out thermo-mechanical testing with simultaneous microscopy and diffraction using visible radiation in optical microscope or electrons in a scanning and transmission electron microscope as well as X-ray, synchrotron or neutron radiation in diffraction experiments.

In situ electron backscatter diffraction experiments provide real time data in the real and reciprocal space to provide in depth information about micro-mechanisms of deformation. The effect of initial texture on anisotropic commercially pure titanium, aerospace grade Ti6Al4V and aluminium magnesium silicon alloy have been investigated to develop fundamental understanding of processes operative for monotonic, reverse and cyclic loading as well as recrystallization. A fundamental understanding of twinning which is an additional shear mechanism in deformation of hexagonal close packed titanium that plays an important role in strain hardening and failure was developed. Similarly the effect of precipitate dislocation interaction in strain hardening response of aluminium magnesium silicon alloy was fully established in monotonic loading and load reversal. In situ EBSD experiments were complimented with synchrotron diffraction and coupled with mean field and full field crystal plasticity simulations to develop a fundamental understanding of deformation and build comprehensive modelling schemes to cut down on further experiments thus establishing the dexterity of high throughput in situ experiments combined with crystal plasticity simulations for rapid process and material development.

Understanding Sample Size Effects on Creep Micromechanics Using *In-Situ* Testing

Syed Idrees Afzal Jalali, Vikram Jayaram and **Praveen Kumar**

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In this talk, the effect of sample size on the steady-state creep properties of materials will be discussed. Flat dog-bone uniaxial tensile specimens of commercial purity Al in the size range of 0.3 to 2 mm, with one side laid with silver particles for tracking deformation as a function of time, were tested at 250 °C inside a scanning electron microscope (SEM). Digital image correlation (DIC) was used to obtain two-dimensional strain fields and creep curves from time lapse images. A softening as high as 10 times in terms of steady-state creep rates was observed with decreasing sample thickness. The softening effect was more pronounced at low stresses. Accordingly, a lower apparent stress exponent of 3.9 was also observed. Further the post-creep dislocation sub-structures revealed an abnormal substructure sizes near to the free surface (surface affected area, SAR) possibility due to the loss of dislocations to the free surface. The substructure size in the sample interior followed the routine bulk behaviour. However, the strain profile from DIC did not show any gradient in strain as a function of distance from free surface. Finally, a microstructure-sensitive iso-strain model based on load shedding between soft SAR and hard interior was formulated to account for the overall increased strain in the strain rate and non-ideal substructure near the free surface. The critical insights into the creep micro-mechanics obtained in this study, therefore, seamlessly unify the power-law creep response at large and small length scales. The same technique of in situ DIC using SEM micrographs can be used to understand the effect of phase and composition on strain distribution in complex alloys.

Diffusion at Nanoscale: An *in-situ* real-time TEM study

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The detailed nanoscale dynamics at various surfaces and interfaces, for example, diffusions, are crucial for fundamental understanding and device applications. *In-situ* (S)TEM is becoming a popular technique to exhibit live performance, functional mechanisms, diffusions and reactions for complicated and nanoscale materials under various conditions, such as annealing, biasing, e-beam irradiation and strain. This technique opens a new era to observe real-time microstructural evolution in the materials and correlate it with their mechanical and electrical properties. This lecture will briefly discuss various in-situ results of different materials starting from thin films to nanomaterials/carbon nanotubes and challenges in preparing electron-transparent samples for such in-situ experiments.

In situ electron microscopy for probing vapor phase growth and phase change induced actuation

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Isn't it seeing in real time make our life easy to find where things are? The same is true for scientific observation of materials through modern electron microscopes. Many of the scientific and technological developments in the area of materials science and nanotechnology are enabled by electron microscopy. Real time observations in transmission electron microscopy (TEM) have enabled direct imaging of growth and mechanical motion in nano and microscale structures, structural phase transitions and interfacial phenomena with atomic scale resolution. In my talk, I will present two examples, namely CNT growth, their self-organization in to dense CNT forest structure and in situ visualization of phase change induced actuation in VO_2 cantilevers. First, I will briefly discuss how *in situ* TEM technique can be used to visualize and quantify actuation in nanostructures that undergo structural transitions by Fresnel contrast imaging of electron transparent cantilevers. Second, I will present aberration corrected environmental TEM observations to answer some of the fundamental questions related to the growth of carbon nanotube forest. The investigated E-TEM method was employed to study CNT growth and provided more details of CNT growth, mechanical interaction between growing CNTs and their dense forest growth. Finally, the recent progress made on the above two aspects will be also presented to correlate the in-situ microscopy findings to practical applications in large scale.

In-situ electron microscopy using MEMS based devices and Graphene liquid cells

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Over the years, Transmission Electron Microscopy (TEM) has been a primary characterization tool to understand the structure-property relationship of most of the materials. In most of the studies, the specimens are investigated post-mortem. There have been several successful attempts to carry out in situ TEM experiments wherein dynamic changes in a specimen are investigated while applying a stimulus like heating, electrical bias, mechanical deformation or exposing to a reactive environment. Owing to the advancements in TEMs and micro-electro-mechanical systems (MEMS), the area of in-situ TEM has progressed extensively over the last decade. Some notable in-situ TEM studies using MEMS devices are applications such as heating, electrical biasing, gas environmental TEM studies as well as liquid cell studies to understand the mechanisms for phenomena in materials science as well as life sciences. Apart from the MEMS devices, there have been recent developments as well as promising applications using graphene as encapsulating membranes for liquid cells. In this talk, a few examples of MEMS based heating devices as well as graphene liquid cells to understand the microstructural evolution in metallic thin films as well as growth of nanoparticles will be discussed. These recent studies using MEMS devices and graphene liquid cells show that graphene liquid cells are the most promising candidates for static-liquid cell experiments, the application of graphene for liquid flow-cells is still in a nascent state. In addition to the mechanisms, some of the issues pertaining to design, fabrication and challenges while carrying out in-situ TEM experiments using MEMS based devices and graphene liquid cells will be highlighted.

Keywords: in-situ TEM, MEMS devices, in-situ corrosion, in-situ heating, phase transformations, graphene liquid cells.

Tracking in-situ morphological changes of nanoparticles in a variable gaseous environment at the atomic scale

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Nanomaterials attract more and more attention over the last few decades thanks to their improved properties and utility in a plethora of scientific areas including catalysis. In catalytic reactions unwanted structural, morphological and compositional changes take place which in turn affect dramatically the catalytic performance. A detailed characterization of these parameters in-situ is of utmost importance if one wants to obtain a better insight concerning the structure-to-property connection.
Transmission Electron Microscopy (TEM) is an ideal technique to investigate materials at both the nanometer and atomic scale and has therefore been widely used in the study of nanomaterials both ex-situ and in-situ. By combining the technique with tomography, a technique which derives three-dimensional (3D) information from two-dimensional (2D) projections, one is able to determine the structure and shape of nanostructures in 3D, even with atomic resolution. Although TEM has often been used to characterize nanoparticle catalysts, unfortunately most observations so far have been performed at room temperature and in ultra-high vacuum, conditions which are completely irrelevant for the use of NPs in real catalytic applications. By using dedicated in-situ holders which became recently commercially available, one can reach higher pressures and temperatures and also introduce liquids in the microscope, and therefore create an environment which is identical to that during actual catalytic reactions.

In my talk, a methodology which was very recently developed in our group to quantify variations of the 3D atomic structure and morphology of nanoparticles under the flow of a selected gas, will be presented.[1] 2D atomic resolution Scanning TEM (STEM) projection images were acquired in an aberration-corrected microscope in different gaseous environments and elevated temperatures using an in-situ gas holder. These images were used as an input for atom counting with the purpose to quantify the 3D shape and structure of the catalyst nanoparticles at different steps of a redox reaction. Prior to their use for quantification, the images were corrected for drifting and other distortions using a novel methodology based on deep convolutional neural networks (CNNs).

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Abstracts of Invited Industry Speakers Online Oral Presentations

In-situ observation in aberration corrected TEM

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Keywords: In-situ, aberration correction, cold FEG, catalyst, electro plating

A various experimental results in In-Situ observations which were combined with aberration corrected transmission electron microscopy (Cs-corrected TEM) are introduced. In this lecture, the features and the performances of newly developed Cs-corrected TEM and a various kind of *In-Situ* specimen holders are introduced in detail.

Owing to the aberration corrected TEM/STEM, atomic resolution observation and analysis are commonly used, today [1]; however, the TEM specimen is kept under high-vacuum condition. It is very important to investigate the structures analysis of materials under practical operating environment; therefore the In-Situ observation technology is becoming very important. Recently, a various kinds of In-Situ specimen holders have been developed; the In-Situ observation can be introduced into the market. And also, JEOL developed Cs-corrected TEM with wide gap objective polepiece for accepting the In-Situ holders and offering higher efficiency X-ray signal collection.

Figure 1 shows an appearance of newly developed Cs-corrected TEM (*JEM- ARM300FII*). The cold field emission gun (cold-FEG) is employed and owing to a small energy spread and a high-coherency of electrons of cold-FEG, a TEM lattice resolution of 60 pm (Cs-corrected with WGP at 300 kV) is performed. And dual type of largest size of EDS detector (158 mm^2, each) can be installed into the wide-gap objective polepiece (WGP); thus, the X-ray collection solid angle becomes very large as 2.21 sr. for analytical capability.

Figure 2 shows an example of In-Situ observation of Ir/CeO_2 catalyst redox reaction obtained at specimen temperature of 600 deg. C. with gas environment holder (Protochips Atmosphere) and ARM300FII, and gases were transferred as H_2 – O_2 – H_2. The Ir/CeO_2 catalyst redox reaction was observed at atomic resolution.

Figure 3 shows another example of In-Situ observation of electrochemical reaction (copper electro plating) obtained with liquid environment holder (Protochips Poseidon) and ARM300FII. The copper plating layer growing in a cupric sulfate solution is observed dynamically. Owing to In-Situ specimen holders and aberration corrected TEM combination, the In-Situ observation is realized.

Fig.1. Appearance of newly developed Cs-corrected TEM (*JEM-ARM300FII*)

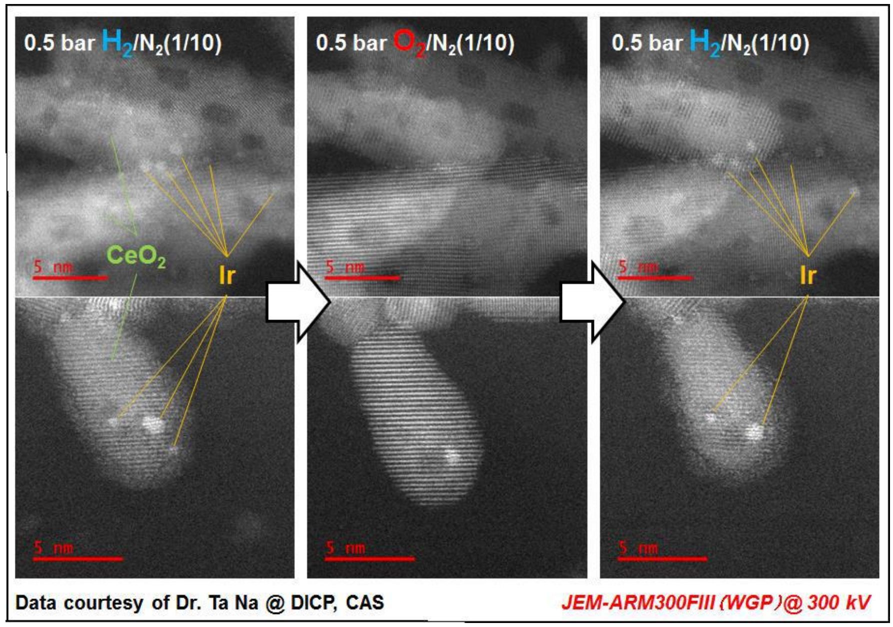

Fig. 2. *Ir/CeO*$_2$ catalyst redox reaction @600deg C. The gases were transferred as $H_2 – O_2 – H_2$ in gas environment holder.

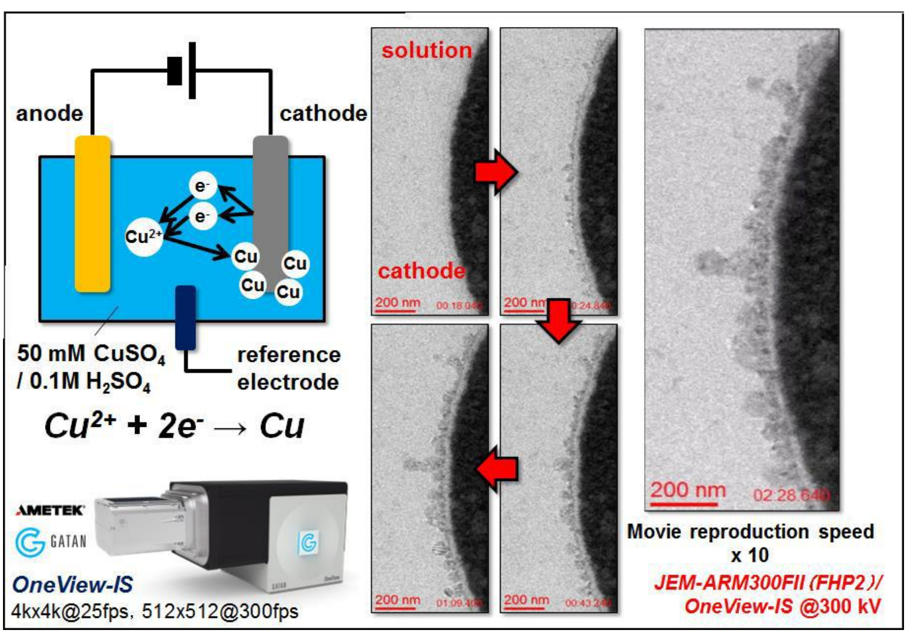

Fig.3. Electrochemical reaction (copper electro plating) the copper plating layer growing in a cupric sulfate solution is observed dynamically with liquid environment holder (Protochips Poseidon) and JEM-ARM300FII.

Detector: *Gatan OneView Insitu.*

Mapping Crystalline Regions During In-Situ Heating: Comparing TEM and 4D STEM

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Often during in-situ TEM experiments, it is important to see and distinguish crystalline regions in a sample. Yet, this can be challenging for several reasons. In a large TEM image, it may be difficult to see at a glance the extent of crystalline regions because neighboring regions are not easily distinguished, or because the large image cannot be displayed at full resolution on the monitor. When using 4D STEM, it is also inherently difficult to see anything as visualization requires a dimensional reduction that can hide important information. In both cases, the difficulty is increased if a series of images or data cubes are acquired as part of an in-situ experiment.

In this work, we demonstrate Python-based processing of both 4D STEM and high-resolution TEM images to map the spatial distribution of crystalline regions. This can be done (with some limitations) during live during acquisition, or it can be applied to previously acquired datasets. While we are processing the 4D STEM data and the HR TEM data in a similar way, there are differences and tradeoffs which make each technique optimal for some different experiments. These will be briefly covered.

Ultimately, the goal for this processing is to perform it live during acquisition to guide decision making at the microscope. This can already be done for TEM imaging at a rate of about 1Ips. Better parallelization of the processing in Python, faster CPUs or GPUs, and other optimizations, will undoubtedly make this faster in the future.

Recent Advancements in In-Situ Nanomechanical Testing

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Understanding the mechanical response and properties of materials at multiple length, time scales, and the test conditions are becoming very important to optimize the performance and develop materials with unique properties. Materials science community has been coming out with new materials with outstanding properties and for applications at normal and extreme conditions. For the underlying research effort, recent instrumentation for structure property correlation has played a critical role. In recent two decades, depth sensing nanoindentation emerged as not only a tool to measure hardness and modulus of materials but other important properties such as viscoelasticity , creep resistance, fracture resistance etc.. at depths as shallow as a few nanometers and temperatures as high as 1000^{o}C. The measurement techniques that were believed not possible a decade ago are becoming possible now with much superior resolutions and accuracies. Besides indentation, today's nano- and micromechanical methods include compression, tension bending, fracture, fatigue and creep tests. This talk will demonstrate this capability of structure property correlation from results on the in-situ nanomechanical testing of various engineering materials using Bruker-Hysitron Nanomechanical Test Instruments. The results will be reported and the physical insight regarding the deformation mechanisms will be discussed. The main focus will be on the instrumentation techniques to improve the research efforts, and develop fundamental understanding of deformation mechanisms of materials

Increasing In Situ Success Through Artificial Intelligence

David Nackashi

Machine vision and Artificial Intelligence technologies are poised to have a significant impact on nearly all areas of electron microscopy. Automating background tasks that are distracting to the microscopist, reducing the skill level required for new users and, most importantly, helping manage the immense amount of data generated in electron microscopy today are just a few of the areas these technologies are expected to impact over the next few years. Protochips AXON software can act as a separate pair of experienced hands, allowing the user to focus on the science of the sample rather than the operation of the instrument. In addition, with new tools like AXON Studio™, users can utilize the vast amount of synchronized metadata to organize and review thousands of images at once, at your desk and on your laptop and more easily create datasets shareable with collaborators. The key is to connect the TEM, imaging detectors and holder systems together utilizing machine vision technology to stabilize images for traditional and *in situ* electron microscopy to elevate the performance, productivity and impact of scientific discovery for TEM.

Using Machine Vision to Drive Nanoscale In Situ Discoveries That Solve Real-World Problems

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In order to fine-tune materials for stronger steels, more efficient catalysts, better batteries, etc., understanding how the environment affects a material's atomic-scale foundation over time is critical. Protochips has created the first-of-its-kind line of machine vision empowered in situ TEM solutions for this purpose. These solutions enable you to quantitatively analyze your material's atomic foundation, in a highly realistic environment within the TEM, while taking advantage of machine vision software to generate higher quality data in less time and unveil trends that would otherwise be hidden. Join us to learn about in situ liquid phase, environmental gas phase, and vacuum heating and electrical characterization inside the TEM that furthers research for catalysts, batteries, corrosion of steel, electronic devices, nanomaterial synthesis and characterization, and more.

Correlative Probe and Electron Microscopy technology using AFM in SEM

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Scanning electron microscopy (SEM) and atomic force microscopy (AFM) are two of the most used, complementary techniques for surface analysis at the nanoscale. Thus, combining them by integrating a compact AFM into SEM brings novel possibilities for true correlative microscopy and advanced multi-modal sample characterization that would be often unfeasible using each imaging modality separately. It is extremely useful in variety of fields, such as Material science, Nanotechnology, Semiconductors, or Life science.

Correlative Probe and Electron Microscopy (CPEM) represents a hardware correlative technology, enabling simultaneous acquisition of SEM and AFM data, and their seamless correlation into one 3D image. The strength lies in combination of AFM modes (3D topography, electrical, mechanical, and magnetic measurements) and SEM capabilities (fast imaging with wide resolution range, chemical analysis, surface modification, etc.). This technique can be applied using LiteScope 2.0, produced by NenoVision, ensuring the data are collected in the same coordinate system and with identical pixel size which results in 3D complex multi-channel sample characterization.

Above mentioned advantages can be demonstrated on correlative in-situ analysis of $LiNiO_2$ cathode material used in rechargeable batteries. Since the powdered cathode material is prone to immediate oxidation upon air exposure, it would represent a very difficult sample for standard AFM and SEM systems and needs to be analyzed by the AFM-in-SEM approach. The SEM combined with EDX technique provided fast navigation of the AFM probe on the sample, information of elemental composition and material contrast. The AFM LiteScope was used to measure the sample topography and conductive mapping to characterize the changes in the cathode after charge/discharge cycling. Lastly, the correlated CPEM image combines AFM topography with SEM material contrast and provides unmistakable data interpretation.

As we can see, the AFM-in-SEM strategy benefits from the complementarity of both techniques alongside significant savings both in time and resources. In-situ analysis together with CPEM technology opens door to completely new possibilities for advanced data correlation and measurements, in many areas of both research and industry.

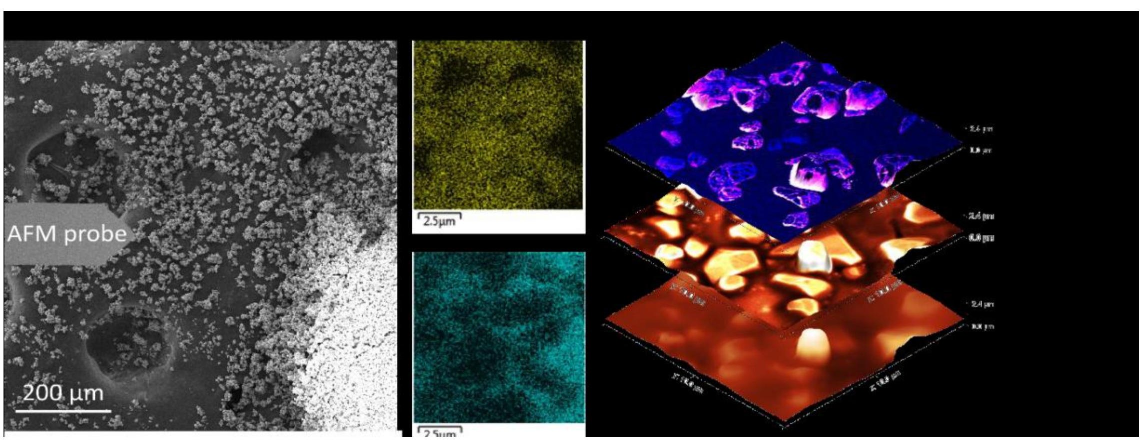

Figure 1: Correlative analysis of $LiNiO_2$ cathode powder: SEM provided fast navigation, elemental analysis (EDX) and material contrast. AFM provided 3D topography and conductivity mapping. The 3D CPEM view merged AFM topography and SEM signal.

In-situ Transmission Electron Microscopy: a MEMS-based route to explore untapped research possibilities at the nanoscale

Dr. Hugo Perez

Abstract: In Situ TEM technology combines the imaging capabilities of transmission electron microscopy with the power and versatility of MEMS devices, in order to observe real-time dynamics at the nanoscale as a function of different stimuli. This is therefore transforming the way we understand things at the atomic scale. In this presentation, we´ll explain the unique architecture of our MEMS devices, which are equipped with different nano-sensors and/or nano-actuators. And how, in combination with our gas and liquid supply systems, they allow you to introduce and control the environment around your sample. We will show the unique benefits of our solutions and specific application examples of high impact research that has been made possible by our technology.

New Developments for In-Situ and Operando TEM Experimentation

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As in-situ transmission electron microscopy (TEM) is becoming more readily available throughout the scientific community, the needs and wants of the community expand as well. This presentation will focus on a series of new in-situ TEM sample holders that expand the horizons of in-situ TEM experimentation. This new suite of electrical, mechanical, and temperature controllable sample holders include: *a liquid-electrochemical TEM holder that can, for the first time, match bulk scale electrochemistry data, a biasing manipulator holder for sample manipulation and site-specific probing, an* air-free transfer holder for protecting air-sensitive samples, a heating and biasing holder that allows heating while measuring electrical properties, and a cryo-biasing holder that allows fine electrical measurements at cryogenic temperatures.

We will present recent works performed using these products in research fields such as electrochemistry, batteries, 2D material fabrication, and low-temperature devices. Most importantly, we will present our liquid electrochemistry holder's ability to match bulk scale electrochemistry data inside a TEM. We will show nano battery setup and charge/discharge behavior in individual nanoelectrodes. We also show an example of our holder heating a MoS_2 sample to 1000°C, resulting in well-segregated MoS_x quantum dot particles. Finally, we will discuss a constant current experiment performed on a nanowire at cryogenic temperatures.

Abstract of E-Poster Presentations

Controlling polarization through defects in Si-integrated $BaTiO_3$

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$BaTiO_3$ (BTO) is a uniaxial ferroelectric with polarization along c-axis of the unit cell. The orientation of c-axis in plane or out of plane of the substrate decides its application as a memory device or electro optic modulator in integrated photonics. In our earlier work [1], we have shown that it is possible to directly grow epitaxial BaTiO3 with precise orientation control. From the room and high temperature XRD analysis we have concluded that the origin of an out of plane orientation in $BaTiO_3$/TiN/Si, is anti-site like defect mediated. Here we further present the structure and chemistry of these Si integrated BTO systems through aberration corrected STEM.

STEM-EDS analysis reveals that our samples are Ba rich. Figure-1 gives a clear picture of alternate Ba (brighter) and Ti (darker) atoms present along [001] zone axis. Antisite positions as conjectured previously, can be identified. Peak pair analysis with optimized conditions to maximize the SNR (kernel size and no of Bragg spots) performed on these images, revealed an average domain size of ~ 3-4 nm, with inhomogenous strain gradients, possibly arising from the antisite defects. Ti displacements were mapped using a home-built algorithm. In this defective sample, while the tetragonality is in the OOP direction, the Ti displacements are not, and exhibit a distribution, which will be discussed in greater detail. This indicates a reduction in symmetry from P4mm of these samples. The microstructural inhomogeneity in composition (contrast), strain as supported by PPA analysis and polarization maps bolster our hypothesis of defect mediated polarization engineering. Inspired by other defective perovskites and fluorites, we are currently investigating the possibility of obtaining giant electromechanical response from these Si integrated materials, and also setting up in situ capabilities to understand the structure-property-defect correlations.

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In-situ Electron Back Scattered Diffraction of SS316L

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In-situ experiments have gained a tremendous use in the field of microscopy and to study the michro-mechanisms of plastic deformation in metallic materials like slip, twinning and phase transformations at different strains. In-situ experiments have completely opened up a new horizon to simultaneously deform a material and also capture it's deformation. In-situ electron backscattered diffraction of SS316L at different strain of 5%, 10%, 20% and 40% is carried out. After deforming it upto 5%, it is found that twin initiation takes place and eventually at 20% twin bundles are formed The post-EBSD analysis using TSL-OIM software includes IPF map, KAM, GROD, etc. KAM is average misorientation angle of a given pixel with all its neighbouring pixel. As the strain increases, KAM also increases. GROD is angle of misorientation of each point in grain with reference orientation selected for the grain. A crystal plasticity fast fourier transformation tool i.e, DAMASK is used to deform SS316L to compare the simulated and experimental data. Till date the micro-mechanism of deformation and their correlation with macroscopic properties was aided by post-mortem characterization using tools like scanning and transmission electron microscopy and electron back scattered diffraction. However, with advent of in situ microscopy, it is possible to obtain information of the operative micro-mechanisms as they happen and use this information to tune the crystal plasticity model. In the present seminar, an overview of in situ experiments aimed towards aiding crystal plasticity simulations to decipher the operative micro-mechanisms of deformation in metallic materials will be presented.

Phase Transformation and Influence of Solid-Solution Properties of Ag-Cu Alloy Nanoparticle Embedded in Ni matrix

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Nanoalloy particles (Ag,Cu) are embedded in Ni matrix were prepared by melt spinning technique, namely, Ni-6wt %(Ag-wt%15Cu) (ACN-HYPO), Ni-6wt %(Ag-wt%28.15Cu) (ACN-EU), and Ni-6wt %(Ag-wt%85Cu) (ACN-HYPE), respectively. In order to understand the effect of matrix on the embedded nanoparticle, extensive DSC (differential scanning calorimetry) and *in-situ* TEM (transmission electron microscopy) has been performed. Detailed electron microscopy study indicates that ACN-HYPO and ACN-EU nanoalloy particle shows a

single phase of (Ag) solid solutions, whereas and ACN-HYPE alloy reveals the bi-phasic alloy nanoparticles. This multiphase nanoparticle consists, (Ag) (face centered cubic) and (Cu) (face centered cubic) solid solution co-exist in the particle at room temperature. DSC results suggest the melting depression in each case. Further, in order to understand the influence of solid-solution properties between Ag, Cu, and Ni, cyclic heating and cooling experiments have been performed by using DSC. In ACN-HYPE alloy, we do not observe any changes during melting and cooling events. Although, in other alloys, composition showed that the area of exothermic peak of (Ag) solid solution increase as the number of cycles were carried. *In-situ* TEM investigation has been carried out for ACN-HYPE to understand the melting kinetics of bi-phasic nanoparticle. The detailed investigation reveals the formation of single phase (Ag) nanoparticle prior to the melting while heating. In order to justify our experimental results, we have done confirmatory experimental and theoretical calculations on the phase stability of Ag-Cu-Ni at the nanoscale.

Keywords: Silver-Copper-Nickel, Nanoalloys, Nanophase diagram, Solid-Solution.

In situ tensile deformation of silicon doped $Fe_{50-x}Mn_{30}Co_{10}Cr_{10}Si_x$ high entropy alloy

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Novel alloy design strategy of complex concentrated alloys, popularly known as high entropy alloys, have emerged as the potential alternative to the conventional strategies which have broadened the regime of mechanical properties under various loading conditions. Recently, focus of alloy design has shifted towards metastability to impart twinning (TWIP) and phase transformation (TRIP) induced plasticity effects by engineering the stacking fault energy through alloying. Two phase $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ TRIP alloy has been the representative alloy for further improvements via thermomechanical treatment as well as by doping with interstitial alloying elements like carbon, nitrogen and silicon. Due to balanced thermodynamic stability of the gamma and epsilon phases, deformation behaviour of such alloys can be altered between TWIP and TRIP by doping. We studied the effect of 0.2 % silicon doping in the representative $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ alloy which showed single phase microstructure with gamma phase after annealing and water quenching compared to undoped alloy that showed two phase FCC + HCP microstructure with dominant TRIP effect during deformation . In situ EBSD was employed to study the evolution of microstructure on tensile deformation. Interestingly, the TRIP behaviour of alloy gets supressed rather bundles of nano twins were observed using electron channelling contrast imaging. Nearly eighty percent tensile ductility with sustained work hardening was observed. Further the deformation behaviour was simulated using mean field viscoplastic self-consistent simulations and full field fast Fourier transformation simulation strategies. Grains

with <111> parallel to tensile axis deform by slip and twinning whereas grains with <100> parallel to tensile axis primarily deformed by slip only. Excellent correlation between experimental and simulated stress strain response, texture evolution, and intragranular misorientation was achieved. A mechanistic perspective on the role of silicon on the gamma phase stability and imparting higher twinning induced strain hardening to achieve extraordinary uniform strain and tensile strength will be discussed.

Micro-mechanism of damage evolution in commercially pure titanium: an in-situ study

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Ductile damage is characterized by significant plastic deformation accompanied with void nucleation, growth, and coalescence at the microscale contributing to final fracture. Ductile fracture in polycrystalline metallic materials is initiated by decrease in strain hardening ability due to limited slip activity which is dependent on intrinsic parameters like grain size and extrinsic parameters like temperature and rate of loading. Unlike metals and alloys with FCC and BCC structure which are well studied in literature, ductile fracture of HCP metals like zirconium and titanium that have inherent anisotropy due to the hexagonal crystal structure and deform by different modes of slip and twinning systems is not well reported. In the present investigation, a miniature tensile specimen of commercially pure titanium (CP-Ti) was deformed till the ultimate tensile strength using a universal testing machine. Thereafter, the deformed sample was prepared by electro polishing for in-situ electron backscatter diffraction (EBSD). Secondary electron imaging and EBSD were performed in a FESEM with an in situ tensile stage at different locations in the neck of the sample for displacement of 0.0, 0.15 and 0.44 mm. A detailed correlative SEM and EBSD analysis based on Schmid factor of different slip/twinning systems, elastic mismatch and strain evolution in the microstructure indicated the synergy between local stress state and defects like twin boundaries and regions of high lattice curvature in the microstructure on ductile damage in titanium.

Electron microscopic study on nano sized precipitation during ageing in Alloy 617

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The efficiency of conventional power plants can be improved by increasing the steam inlet temperature in gas turbines. Advanced Ultra Super Critical (AUSC) technology is being developed in India to improve the efficiency of conventional power plants with inlet steam inlet temperature to turbines more than 700 °C- 760 °C [1].
Alloy 617 is solid solution strengthened alloy of nickel, chromium cobalt and molybdenum having good corrosion resistance, high temperature strength, creep resistance and high temperature oxidation resistance[2, 3]. Hence, Alloy 617 is considered as candidate material for high temperature application [4]. Chromium present in the alloy is responsible for high temperature oxidation resistance and carbide formation with molybdenum to improve the creep properties. Ti and Al are added to form secondary ordered phase precipitates γ' within the matrix. This phase is finely distributed and responsible for high temperature strength. The formation and evolution of γ' is studied as a function of ageing temperature. Solution annealed tube free from precipitates was aged at 700, 750 and 800 °C for 100 h in air followed by furnace cooling. Nano sized γ' precipitates measuring 6-13 nm were observed to form uniformly at 700 °C which was supported by increase in hardness. SAED (selected area electron diffraction) patterns were indexed and identified as $Ni_3(Al,Ti)$ type γ'. At 750 °C ageing temperature, the γ' particles coarsened and the average particle size increased to 20 nm. With further increase of temperature the particles are observed to grow more in size and the average particle size became 35 nm at 800 °C. Enrichment in Al & Ti content in the γ' with temperature was found through STEM-EDS. In addition, formation of fine intragranular carbides is observed from 750 °C. These intergranular carbides formed at elevated temperature are likely to be responsible for high creep resistance. The orientation relationship between these carbides, matrix and γ' was also identified from the SAED patterns.

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Grain Boundary Precipitate Embrittlement in Alloy 617 due to Partial Solution Annealing

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High temperature Nickel based alloy 617 is considered as a candidate material mainly for the boilers in the AUSC thermal power plants due to its combination of creep strength and good workability and weld ability [1]. Alloy 617 tubes are being used with aim of increasing the thermal efficiency of power plant by increasing the operating temperature [2].

The tubes are manufactured in the multiple steps of hot deformation followed by cold working. The cold deformation is followed by solution annealing at high temperature. This heat treatment involved heating and soaking at elevated temperature of 1190°C and water quench. During manufacturing process, some tubes got cracked while undergoing intermediate cold pilgering. Those tube samples were subjected to failure analysis for root cause identification. No significant difference in Vickers Hardness values were observed in annealed mother tubes of cracked tube and tubes without cracks. Fractography using Scanning Electron Microscope revealed inter-crystalline crack propagation confirming inter-granular mode of fracture. Micro-structural characterization showed re-crystallized grains with formation of very thin layer of fine plate type second phase particles (SPP) along the grain boundary. Elemental analysis using EDS spot acquisition confirmed precipitates enriched with Ni, Cr and C compared to matrix. As a result it was suspected absence of complete solutionising in these tubes. The cracked portion of the tubes was cut and the tubes were re-annealed. No grain boundary enrichment was observed after re-annealing of mother tubes and no cracks were encountered during pilgering of re-annealed tubes. Hence it is confirmed that grain boundary enrichment resulted in embrittlement of grain boundary and subsequent cracking during cold working. The failure analysis also concluded that though proper solution annealing was imposed, slight delay in quenching resulted in formation of such precipitation and subsequent embrittlement. As the microstructure developed during hot extrusion significantly affects the subsequent processing, microstructure, other mechanical and metallurgical properties, micro-structural characterization of hot extruded blank of Alloy 617 was carried out using optical microscopy and electron microscopy techniques. Equiaxed grain structure was observed in the longitudinal section with some grains showing serrated grain boundaries (SGBs). This type of structure is special to Ni based alloys and is formed by precipitation of second phase precipitates (SPPs) along grain boundaries during slow cooling and mostly affecting fracture toughness, fatigue properties and alloy brittleness [3].

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Electrical properties of YSZ/double perovskite composites for solid oxide fuel cell (SOFC) application

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Mixed ionic electronic conductors (MIEC) showed promising next-generation electrode material for solid oxide fuel cells (SOFC). MIEC composites were designed by mixing two different materials with compositions (1:1 wt.% ratio) that possess ionic as well as electronic conductivity. In the current work, we have synthesized the STC/3YSZ and STC/8YSZ composite by using the solid state reaction route. The phase constitution of STC/YSZ composites was confirmed by using XRD. The morphological was carried out via FESEM. These composite systems displayed dense microstructure with little porosity, and subsequently, EDS analysis was performed to confirm the elemental distribution. The electrical properties of the STC/YSZ composite system were computed using electrochemical impedance spectroscopy. Impedance spectra showed the diffused semicircle revealing grain and grain boundary polarisation resistance.

Further, we were calculated typical parameters like modulus, admittance, and activation energy. Almond-West (AW) power law was illustrated the transport phenomenon of conductivity for STC/YSZ composites. The time-temperature superposition principle (TTSP) was described the conductivity relaxation and also suggested the ionic conduction mechanism is temperature-dependent. The estimated value of the dielectric constant was high at the low frequency region, which also meant electrode polarisation and charge transport.

Keywords: SOFC, MIEC, STC (Sr_2TiCoO_6), Impedance spectroscopy, AC conductivity, Relaxation, TTSP, Polarization.

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