Confessions of a Former Microscopist: What Really Drives Progress in Science and Technology

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ABSTRACT

For more than eight decades, electron microscopy and materials science have developed in parallel: advances in one have inevitably called for, or relied upon, advances in the other. In this presentation we will review the historical synergy between the tool and the problems that it has been used to solve, focusing on events that seem to have had profound impacts. We will see that it has predominantly been the capabilities of the tool that have determined the direction of the science and that the science, in turn, has determined the direction of materials technology.

We are entering an era in which technology more and more frequently calls for specific scientific input and data, rather than being driven by available theories and information. Information is increasingly being *pulled* out of science by technology, rather than being *pushed* into technology by science: we will show that this often allows for significant acceleration in the development and deployment of new materials.

The talk will conclude with a few examples of how all this might affect electron microscopy, and some ideas of what capabilities will be of the greatest value.

Atomic-Scale Analytical Tomography: Taking the Best from (S)TEM and Atom Probe

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ABSTRACT

Current efforts are being directed toward achieving billion atom tomography with high spatial fidelity and 100% detection of the atoms in real space. This capability can be called atomic-scale tomography (AST). Our plan is to

couple this with complete analytical characterization at the atomic scale in a single instrument which would be called atomic-scale analytical tomography (ASAT). ASAT will thus require coupling atom probe tomography with (scanning) transmission electron microscopy. Once these large data sets become available, computational materials engineering can be integrated into the characterization process. Atomic-scale structure may thus be coupled to atomic-scale properties and structure-properties relationships at the atomic-scale may be studied, predicted, and observed.

The instrumental developments needed to reach AST and ASAT include: trajectory corrections for precise atom placement and detecting 100% of the atoms without ambiguity in identity. Specimen holders for 0.2 nm STEM imaging of needles at 20K under high electric field will be very challenging. The conceptual and tactical challenges to reaching these goals will be outlined and the future benefits will be discussed.

Collaborators: Dierk Raabe and Baptiste Gault of the Max Planck Institut fur Eisenforschungs in Dusseldorf; Rafal Dunin-Borkowski and Joachim Meyer of Forschungszentrum Jlich; Christoph Koch and Wouter van den Broek of Humboldt Universitt zu Berlin, Robert McDermott and Joseph Suttle of the University of Wisconsin; Simon Ringer of the University of Sydney; and Brian Gorman and David Dierks of Colorado School of Mines.

Transmission Electron Microscopy for Microelectronics Reliability

Brendan Foran
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ABSTRACT

The current status of electron microscopy supporting microelectronics and optoelectronics reliability science will be discussed with examples from The Aerospace Corporation’s internally funded research and development programs. Challenges will be discussed from the perspective of needs for materials characterization and metrology as they are relevant to device reliability. Results will be presented to highlight developing knowledge and capabilities for next generation materials and devices, and plans to address metrology challenges going forward.

Soft Matter / Biomaterials

Validation of CryoEM Atomic Resolution Structure

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ABSTRACT

Electron cryo-microscopy is a rapidly growing field for macromolecular structure determination at atomic resolution. We establish a computational protocol to construct a *de novo* model from a cryoEM density map, with associated metadata, and accurately establish a model that accords with the experimental map densities. This model is validated using multiple metrics and is annotated with the atom positions and their uncertainty. I will discuss the validation procedures that are being practiced in the field for atomic resolution cryoEM structures.

Individual-Particle Electron Tomography (IPET): an approach to study flexible soft-/bio-molecular structure and dynamics

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ABSTRACT

Soft-/bio-molecules have the unique ability to function specifically and efficiently, which is attained through its three-dimensional (3D) structures and flexibility, as well as necessary conformational changes. However, structural study on soft-/bio-molecules that have large-scale flexibility, dynamics, and heterogeneity is challenging by current techniques, including X-ray crystallography, nuclear magnetic resonance (NMR) spectrum, small angle scattering (SAXS) and electron microscopy (EM) single-particle reconstruction. A fundamental approach to study the structure of flexible soft-/bio-molecules should be based on the signal from each individual molecule itself instead of averaging from different protein molecules. EM provide a novel tool to image each individual molecule at atomic resolution level; while electron tomography (ET) provide an approach to image a targeted molecule from a series of tilt angles.

Although the signal obtained from an individual molecule has been believed for decades to be too weak to achieve any 3D structure with a meaningful resolution, we recently re-investigated this possibility carefully and proposed an individual-particle electron tomography (IPET) approach with a ‘‘focused electron tomography reconstruction’’ (FETR) algorithm to improve the 3D structure resolution via decreasing the reconstructing image size with an iterative refinement process. IPET does not require a pre-given initial model, class averaging of multiple molecules or an extended ordered lattice, but can provide near one nanometer resolution 3D structure from an individual protein molecule. Through the structure determination of each individual molecule, the comparison of these molecular structures provides a new opportunity to reveal the dynamic character and equilibrium fluctuation of soft-/bio-molecules, such as DNA-nanogold and antibody.

Morphology Characterization and Directing Self-Assembly in Nanostructured Soft Materials

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ABSTRACT

Self-assembly of block copolymers and small molecule mesogens gives rise to a rich phase behavior as a function of temperature, composition, and other relevant parameters. The ability to precisely control their chemical functionality combined with the readily tunable characteristic length scales (~1 to 100 nm) of their self-assembled mesophases position these systems as a versatile and attractive class of materials for compelling applications ranging from membranes for size and chemo-selective transport, to lithography. As a result there is intense interest in elucidating the physical processes relevant for directing self-assembly in these materials, with a goal of exploiting such fundamental understanding to create useful materials or devices. This presentation discusses recent advances in directed self-assembly of soft nanostructured materials and emerging methods for generating highly ordered and heterostructured systems. In particular, we will focus on the use of magnetic fields, physical confinement, and electrospray deposition as scalable approaches for

directed self-assembly of soft nanostructured materials. Challenges in the characterization of these systems using electron microscopy, will be discussed and applications as membranes and as pattern transfer templates will be highlighted.

Computing:

Improving the Resolution, Acquisition Time and Quality of Scanning Microscopy Images Through Computational Rather Than Hardware Means

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ABSTRACT

After fifty years of significant advances in hardware development that have included brighter and smaller electron sources as well as dramatically improved electron optics and signal detectors, electron probes as small as five angstroms are available at beam energies routinely used in scanning electron microscopy. Going the next step through the use of beam energy filters and aberration correctors is very complex and expensive. However, it may not be necessary to go to smaller probes to obtain the highest level of resolution if effective methods are implemented to determine the spatial distributions of electrons in a focused beam. This is because such information can then be combined with regularized deconvolution methods to obtain resolutions of less than the probe size while simultaneously reducing noise by more effectively by using more of the collected signal. Our research has shown that this is clearly possible and as an added benefit images of a desired level of resolution can be collected more rapidly. Noise reduction is extremely important with the rapid growth of three dimensional imaging particularly in the characterization of microelectronic devices and complex biological structures even when the highest level of resolution is not required. In these cases hundred, thousands or even a much greater number of images must be collected rapidly and somehow combined to reveal interconnectivity without noise limitations due to short acquisition times. To accomplish these objectives totally new algorithms and computing platforms will be needed as well as a symbiotic relationship between experts in microscopy and diverse fields of image processing including astronomy, surveillance and medical imaging.

Additive Manufacturing:

Electron-microscopy studies of additively manufactured 17-4PH stainless steel

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ABSTRACT

Traditional manufacturing technologies such as castings or forgings can draw from decades to centuries of practical experience. This experience base not only comes essentially for free today in the form of published literature, but also constitutes the basis for modeling and simulation efforts. Only very limited technical data exists by comparison for additive manufacturing. The limited experience base impacts additive manufacturing development in two ways: on one hand it elevates the need for modeling and simulations to avoid costly experiments, but at the same time the limited practical experience raises the risk for models and simulation codes to miss important processing aspects. Electron microscopy plays a key role in overcoming some of the barriers to transform additive manufacturing into a validated manufacturing technology. Defect and microstructure identification, for example, help validate microstructure simulation codes. But microscopy studies also reveal features that point toward new or unknown phenomena in additive manufacturing. Using samples made from 17-4PH powder with a 3DSystems ProX300 machine, some of these previously unrecognized features will be highlighted along with examples of processing-microstructure linkages. These include inclusions at length-scales from the ten nanometer to the tens of micrometer scale, pores and un-melted regions, and internal surface oxide layers, but also microstructure reflections of laser beam settings. The current observations of defects across length-scales and also at processing- and geometry-specific sites reveal a need for electron microscopy to tie in with other analysis techniques, for example, with X-ray computer tomography. This need arises mainly because additive manufacturing allows for designs with internal features, but also because of part-to-part variations. These

variations require efficient protocols to compare parts across length scales. The new demands for electron microscopy and related characterization techniques from additive manufacturing should draw the attention from funding agencies. The impact of novel analysis approaches would benefit the manufacturing community at large.

Powder-Bed Additive Manufacturing Modeling and Measurement Challenges

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GE Global Research

ABSTRACT

Various models are being developed around the world for modeling the powder bed additive manufacturing process. These models include multi-physics codes for modeling laser-particle interaction, diffusion codes for microstructural prediction at micron-scale and continuum mechanics codes for distortion and residual stress predictions at meter length scale. Validation of these models is a challenge now due to difficulties in measuring output responses from the materials and machines during the build process. Furthermore, certain inputs required in the models such as materials properties, e.g. temperature dependent viscosity and surface tension and thermal and stress boundary conditions are difficult to measure or determine.

The focus of our talk will be on measurements that are needed to fully validate the different simulation models. These range from in-situ monitoring to post-processing sensors for metallic alloys. Special attention will be paid to: particle size and its distribution; melt-pool size and depth; microstructure and the formation of transient phases; defects such as cracks, lack of fusion and porosities; distortion and residual stresses; material constitutive models; and the impact the repeated thermal cycling may have on grain nucleation models and residual stresses.

The role of microscopy on ICME Methods development during the DARPA OM Program

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ABSTRACT

Honeywell has been active under the DARPA OM program, Contract # HR0011-12-C-0037, in the development of Integrated Computational Materials Engineering (ICME) modeling and simulation tools for the additive manufacturing process of metals and has demonstrated the extreme conditions that take place during this process. The material is heated by the laser to the boiling point and then cools well below the solidification point in a couple of milliseconds. Some of the challenges in the development, validation and verification of the ICME tools consists of the collection of data needed to calibrate the models and to compare the model predictions with experiments for validation and verification. In the case of the material models information on the microstructure is needed such as precipitate sizes, and the location and distribution of the precipitates or solutes in order to predict the strength of the material, for example. In other cases one may need the dislocation density and location of the dislocation pile-ups. In the case of residual stress models one may need to understand the distribution of the residual stresses at the sublevel of the melt pool size. The goal of this presentation is to highlight the needs that exist to develop microscopy techniques to acquire such measurements in an accurate and efficient manner.