by Ernest Granson

Invasion of the micro machines

Welcome to the microscopic world of material processing and manufacturing

The world of manufacturing is increasingly probing into the nano, micro and meso scale to produce more efficient and better-performing equipment, parts, materials and compounds.

Some of those experimental processing and manufacturing techniques, not to mention the resulting products, can challenge our very concepts of industrial composition and fabrication.

In the past half decade or so, the scientific community has been trying to harness that non-linear innovation to penetrate a new level of microscopic environment known as the mesoscale.

Just as the engineers used nature's own blueprint to design the self-constructing robot, researchers around the world are trying to study and modify molecular behaviour at the meso scale, which then will affect structure and properties at the macro-scale.

This phenomenon has been deemed important, and promising, enough that several years ago, the U.S. Department of Energy (DOE) commissioned a report prepared by a subcommittee of the DOE's Basic Energy Sciences Advisory.

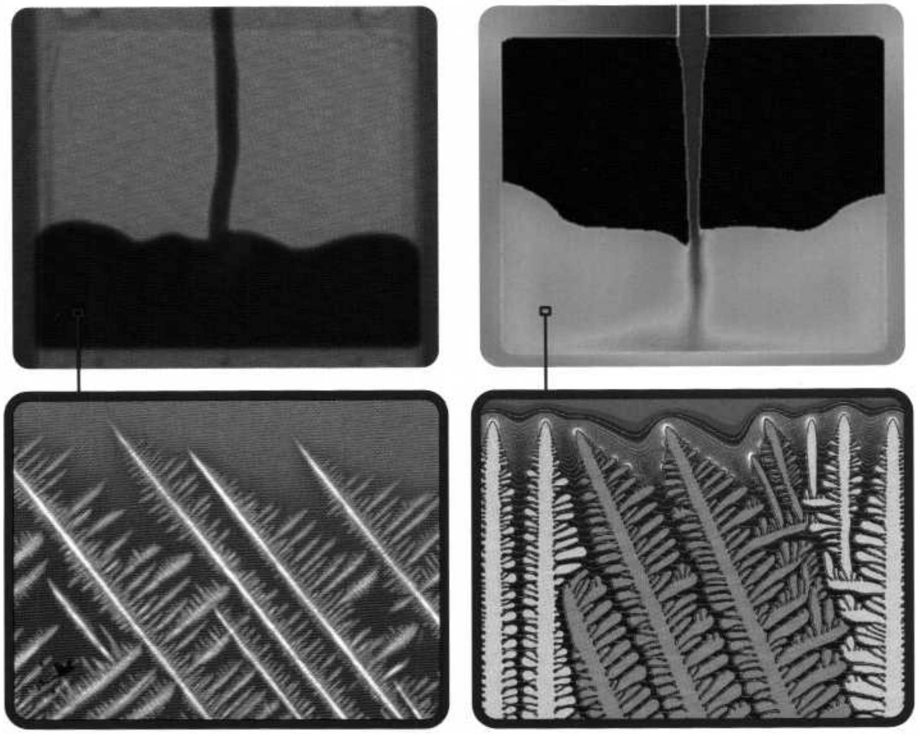
Entitled *From Quanta to the Continuum: Opportunities for Mesoscale Science*, the report said "it's the mesoscale where classical, quantum and nanoscale science meet. It has become clear that in many important areas the functionality that is critical to macroscopic behavior begins to manifest itself not at the atomic or nanoscale, but at the mesoscale, where defects, interfaces and non-equilibrium structures are the norm

"The ability to predict and control mesoscale phenomena and architectures is essential if atomic and molecular knowledge is to blossom into a next generation of technology opportunities, societal benefits and scientific advances."

It's those defects, interfaces and structures that researchers Amy Clarke and Cindy Bolme are targeting.

Clarke and Bolme are scientists at Los Alamos National Laboratory (LANL) engaged in research on the micro and meso scale that could, one day, influence how materials are processed and fabricated.

Some 70 years ago, LANL served as the nucleus (so to speak) of the famed Manhattan Project, which led to the development of the first atomic bomb in Los Alamos, New Mexico. Here at the LANL, an enormous amount of research takes place, much of it affiliated with national security, but also research into leading-edge scientific and engineering technologies.



An example proton image of a tin-bismuth alloy during casting mold filling is shown on the top left, while an example simulation is shown on the top right, providing information about the fluid dynamics and thermal exchanges during processing. At the micro-scale, directional solidification of a tin bismuth alloy is shown on the bottom left and an example phase-field simulation is shown on the bottom right, revealing patterned (dendritic) microstructural growth and local chemistry variations. The bottom images are not specifically related to the top images, but schematically highlight the different scales of interest. *Illustrations courtesy Los Alamos National Laboratory.*

Using proton radiography to create images on the micro scale, Clarke, who is a research and development scientist in LANL's Metallurgy Group, examines metal alloy solidification to understand how the microstructure of the molten metal alloys can be manipulated as they solidify.

The knowledge gained from this research could be applied to metal casting in various industries — for instance, the automotive sector, where it might be used to manufacture components such as aluminum engine blocks and other parts.

Clarke — who holds a PH.D from the Colorado School of Mines and is a recipient of the Presidential Early Career Award for Scientists and Engineers and also of the Minerals, Metals & Materials Society Young Leaders Professional Development Award — feels the experiments she and her team are carrying out could revolutionize the process of metal casting, according to the team's 2013 paper, *Proton Radiography Peers into Metal Solidification,* as published online in *Scientific Reports*. >>

<< The paper points out that understanding the link between processing and structure is important because structure profoundly affects the properties of engineering materials.

"The mesoscale, at which we're experimenting is where you need to account for factors in the microstructure such as defects, grains and texture that develop during the solidification process as we manipulate that microstructure," says Clarke.

"Those factors are then included in the model that we generate. These changes that we make in the meso-scale directly affect the material at the macro-scale. Meso-scale is where the structure and the crystallinity matter because they predict the material's properties."

The significance of Clarke's team's research is not only that the results of material manipulation are observable in real-time, but the use of proton radiography also allows for the process parameter changes to be controlled as the solidification is actually taking place.

"Historically, metals are cut up and polished to see the structure and to infer how processing influences the evolution. We can now peer into a metal during processing without destroying it using proton radiography," researchers say in their paper.

"This will also enable process-aware manufacturing studies of materials, reducing the time for process development and the time from discovery to deployment through the elimination of trial and error, which are critical for advanced manufacturing initiatives."

While Clarke's home base is at the Los Alamos facility, much of her research takes place at Argonne National Laboratory's Advanced Photon Source (APS) at the University of Chicago. The APS produces ultra-bright, high-energy storage ring-generated X-ray beams for research into most scientific disciplines.

Cindy Bolme, too, is conducting materials research in the mesoscale using a different type of technology to observe changes in the microstructure.

Bolme says she uses primarily uses a free-electron laser that is an ultra-brilliant source of X-rays. In fact, the equipment she uses is considered the most powerful X-ray source in the world. It's called the Linear Coherent Light Source (LCLS) at the Stanford Linear Accelerator Center (SLAC) National Accelerator Laboratory, housed and operated at Stanford University for the DOE.

One of Bolme's recent experiments using the LCLS involves permeating her material samples with the extremely powerful pulses of X-ray laser light as the sample is being manipulated — in this instance, by shock wave.

"We can now peer into a metal during processing without destroying it using proton radiography"

Diffraction of the X-rays during that action is captured on the X–ray detector, creating images of the microscopic structures and properties, as well as measurements that can be analyzed. According to the SLAC, this X-ray process actually freezes the motions of atoms and molecules, allowing the images to be linked into stop-motion movies.

"This not only allows us to explore the role of chemistry as the structure evolves though measurements such as temperature feedback, but it gives us a lot of control over the way that structure evolves," Bolme says.

The fact that both Clarke and Bolme must travel to other research facilities is one reason why there is now a proposal to establish a new, high-level facility at LANL.

The future facility is called MaRIE for Matter-Radiation Interactions in Extremes. The multi-purpose, billion-dollar materials research complex and user facility would include an electron accelerator, an X-ray free-electron laser, a diagnostic hall and a materials fabrication facility.

Canadian perspective

In Canada, there is also considerable research and development taking place in the area of nano/micro/meso scale.

In 2010, the National Research and Engineering Research Council of Canada (NSERC) announced funding for the Canadian Network for Research and Innovation in Machining Technology (CANRIMT) to develop the world's most advanced virtual machining technology for macro and micro-machining operations.

Canadian university researchers, as well as corporate partners, have been collaborating as they test the simulation models on industrial machines and develop a new five-axis meso-milling machine for the medical and electronics manufacturing industries.

Yusuf Altintas, co-ordinator of Mechatronics Option at the University of British Columbia, is the scientific director and principal investigator for CANRIMT.

UBC's Manufacturing Automation Laboratory (MAL), founded and led by Altintas, is considered one of the leading machining research and development facilities in the world having developed advanced machining process simulation and process optimization software that is used across the globe.

The University of Calgary is also a member of CANRIMT with a number of faculty members contributing to the research effort. As a professor of mechanical and manufacturing at the Shulich School of Engineering, Simon Park has pursued research into areas such as mechanics and dynamics of micro and nano metal-cutting operations, micro injection molding, nano-composites and virtual simulation of machining, among many others.

Some of that research takes place within the engineering school's Micro Engineering Dynamics and Automation Lab (MEDAL). The lab's main goal is to dramatically advance existing fabrication technologies through rapid, cost-effective fabrication of three-dimensional nano-, micro- and meso-scale components using a variety of engineering materials such as metallic alloys and polymers.

The lab integrates both subtractive processes (micro machining using tiny micro tools where the diameter is slightly bigger than thickness of hair) and additive processes (injection molding used to manufacture the smallest components to bottle caps to entire car parts). The result provides the productivity, flexibility and accuracy to develop complex 3-D micro-scale components.

Assistant professor Seonghwan Kim, also at the Schulich School of Engineering and principal investigator at the school's Nano/Micro-Sensors and Sensing Systems (NMSSS) Lab, is concentrating on research at mainly the nano-/micro-scale levels.

Kim, who has also completed research at the Oak Ridge National Laboratory in Tennessee and at the University of Alberta, was recently awarded a research grant by the Canada Foundation for Innovation to develop high-performance coating materials for oil and gas pipelines.

He is also looking at better understanding the physical and chemical processes occurring at micro and nanoscale interfaces to improve bitumen recovery and the overall energy efficiency of oilsands processing. >>

<< To do this, Kim is developing a one-of-a kind, multi-functional scanning probe microscope (SPM) system to enable non-invasive, high-resolution characterization of nano-materials, nano-composites and micro-/nano-porous oilsands.

Kim says this system will identify nano-scale subsurface features (nano-particles, nano-fibers, nano-platelets) buried deep inside samples by acquiring ultrasonic holograms generated by the coupling between the oscillations of a sample and a probing cantilever.

A probing cantilever is a tiny tip mounted on a bendable strip placed closely to a sample being measured, but doesn't actually touch the surface of the sample. As the tip scans over the surface, attractive and repulsive atomic forces bend the strip. The bending is measured and provides a graphic depiction of the surface of the sample in atomic scale.

Kim says a micro-cantilever is an essential part of SPM.

"It has a probing tip which can be regarded as a 'nano-finger,' enabling us to feel the surface rather than see," he says.

With 10 years of extensive experience with SPM and its instrumentation, Kim is well aware of its capabilities.

"An SPM is one of the most versatile tools in the arsenal of nano-scale metrology," he says. "Compared with conventional optical microscopes, the nano-scale scanning tip of the SPM dramatically improves spatial resolutions.

"Considering the current limitations on non-invasive, high-resolution physical and chemical materials characterization methods, the integration of deep-subsurface imaging, hyperspectral imaging and nano-thermal analysis capabilities into a single SPM unit will enable multi-modal characterization of nano-materials, nano-composites, nano-electronics and even such micro/nano-porous materials as oilsands, which have heterogeneous interfaces."

Kim points out there are no commercially available SPMs that provide this multi-functionality, so his proposed one-of-a-kind SPM system will be developed by retrofitting a commercially available SPM system with a radio frequency lock-in amplifier and a piezo-electric crystal on the sample stage.

"These modifications will allow the sample to be oscillated at ultrasonic frequencies, which are required for deep-subsurface imaging of novel polymeric nano-composites," he says.

In addition to the deep-subsurface imaging technique, Kim will also implement a high-resolution, non-contact infrared spectroscopic imaging technique. It works photoacoustically by exciting a sample with a powerful IR light source such as a broadly tunable quantum cascade laser (QCL).

"We then carefully monitor the coupling between the photoacoustic waves from a sample surface and a probing cantilever tip at a certain offset distance in the order of one nano-meter," he explains.

Kim says use this deep subsurface technique will allow researchers to more widely explore fundamental structure-property relationships of coating material, which have been limited by the use of conventional microscopy systems.

"Non-contact infrared spectroscopy technique can also be used to investigate the surface properties of novel polymeric nano-composites that show hydrophobicity (repelling water), as well as oleophobicity (repelling oil)," says Kim.

"An SPM is one of the most versatile tools in the arsenal of nano-scale metrology"

"This knowledge will be applied for a science-based surface modification process for polymeric nano-composites with a specific aim of making high-performance liners for pipelines that can reduce hydrodynamic drag and prevent internal corrosion."

In addition, the nano-thermal analysis capability of the SPM system will be used to measure localized transition temperature, thermal conductivity and thermal expansion coefficients of novel polymeric nano-composites.

Kim says these thermal properties are important parameters of polymeric nano-composite coatings on metallic pipeline substrates.

"Environmental or operational temperature fluctuations of pipelines may induce delamination of the coating layer because of the large mismatch between thermal expansion coefficients of polymeric nano-composites and that of metallic pipeline," he says.

"Delamination between the coating layer and the pipeline substrate could weaken the performance of the polymeric nano-composite coating."

Kim believes that with the knowledge gained from the SPM system, advanced, high-performance pipeline coating materials could be designed and manufactured that would reduce delamination and erosion/corrosion, ultimately preventing oil/gas leakage from the coated pipeline.

The SPM system will also help researchers understand the fundamental processes occurring at the interfaces between liquids (bitumen, water) and solids (sand, clay, and other minerals) in the oilsands, with and without, the presence of additives and surfactants. This knowledge is crucial for the process of manipulating the interfacial properties of oil sands.

"Control of these interfacial interactions would help to develop improved, environmentally friendly and cost-effective oilsands processing techniques," says Kim.

Earlier studies have investigated interactions between bitumen-bitumen, bitumen-water, and bitumen-air using variety of techniques to understand the mechanisms of interactions involved.

In recent years, with the development of advanced characterization instruments such as the atomic force microscope (AFM), a member of the SPM family, various attempts have been made to quantify the interaction forces between bitumen-silica, bitumen-clays and bitumen-minerals to explore the essence and nature of interactions between the various components in a bitumen extraction system.

Kim notes they were successful, to some extent, in generating averaged force profiles under various processing conditions. However, since real fines in oilsands ore are more complex than the single clay or mineral used in these studies, the role of real fines in oilsands processing needs to be further investigated, says Kim.

"In addition, interfacial forces between bitumen and heterogeneous micro/nano-porous oilsands haven't been investigated so far because of the current limitation of conventional AFM techniques," he says.

"The key is to be able to differentiate the surface species at the nanometer length scale and to determine how they are structured."

With its capability to identify material properties non-invasively in nano-scale resolution under changing conditions, the SPM is not limited to the above applications, Kim points out.

The facility will have broad applications in the development of unexplored nano-materials, nano-composites, nano-electronics and nano-medicine for energy, environment, information technology and health industries.

Ernest Granson is a Calgary-based writer and editor, and a contributor to PROCESSWest.