Testing with a difference

Dr Kalin Dragnevski from the Laboratory for In-situ Microscopy and Analysis is taking testing into uncharted territory, reports Rebecca Pool

Dr Kalin Dragnevski carries out mechanical testing with a difference. From stainless steel and titanium alloys to volcanic sand and the tendons in rats tails, he and colleagues at the Laboratory for *In-situ* Microscopy and Analysis, University of Oxford, analyse myriad materials using an array of instruments.

As Dragnevski puts it: "We started out looking at your classic solid mechanics, but a longer-term vision was to go beyond this and really expand our knowledge and understanding of materials."

"We have just finished imaging and scanning some 17th-century printing plates created by the famous botanist Robert Morison," he adds. "To do this in an Engineering department is pretty crazy."

Dragnevski set up the Laboratory for *In-situ* Microscopy and Analysis (LIMA) in 2012, to investigate structure-property-composition relationships in materials systems at different length scales. From word go, he has been developing *in-situ* methods to measure the mechanical properties of materials systems, initially within an Environmental Scanning Electron microscope. His work has flourished.

"It really started out as a very small operation with just me, but has expanded naturally," explains Dragnevski. "People wanted to collaborate on research projects but also use LIMA as a service provision; and in this way we have become a self-sufficient unit with the Engineering Science Department at Oxford."

LIMA now provides a wealth of expertise, equipment, as well as training, so users can perform experiments with, as Dragnevski says, 'flexibility and freedom'. Equipment includes a Carl Zeiss EVO variable pressure SEM, an INCA X-Act silicon drift detector X-ray system from Oxford Instruments, an EDAX Hikari EBSD System and a Peltier stage from Deben UK. The laboratory also has a Atomic Force Microscope, an Alicona Infinite 3D profilometer, as well as tensile compression stages from Deben UK, with heating and cooling capabilities.

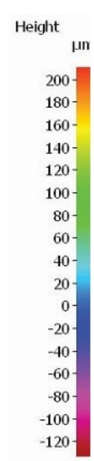
Collaborations are wide and varied with, for example, numerous Rolls-Royce projects relying on the profilometer for swift, 3D profile and surface roughness measurements of alloys. Meanwhile, researchers from Carl Zeiss Germany, have also worked with LIMA on the use of mechanical stages, *in situ*.

"In another recent collaboration, engineers from the Transport Research Laboratory would bring over cut-offs of pot-holes to study," highlights Dragnevski. "What usually starts off as a small job typically evolves into a much longer collaboration."

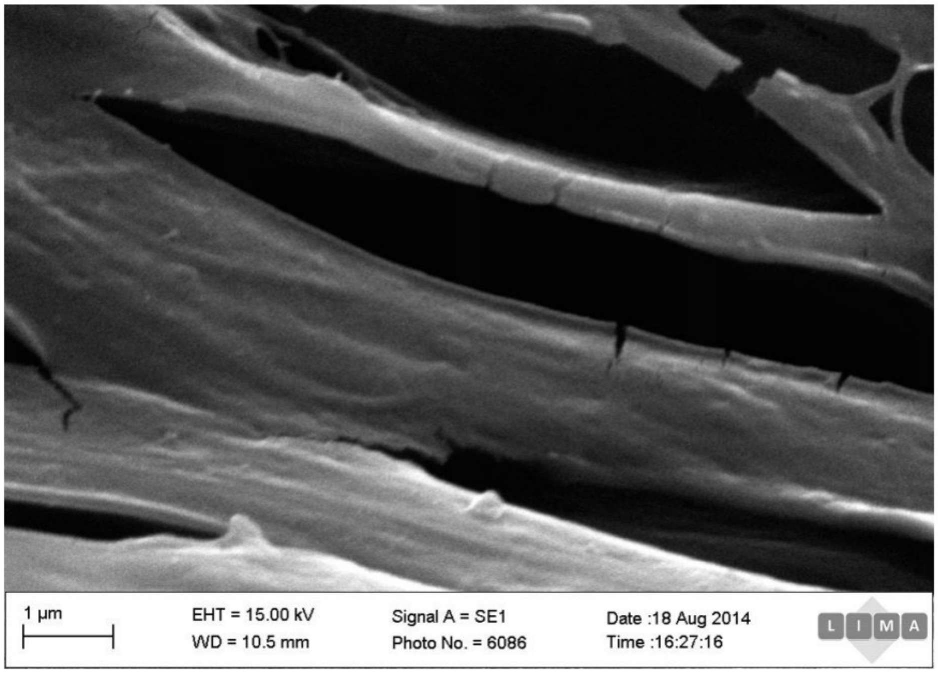
"With our microscopes, mechanical stages as well as the heating and cooling devices, we perform experiments *in-situ* so we can really understand how the structure-property-composition [relationship] evolves as a test progresses," he adds. "I believe this is unique to us."

RESEARCH HIGHLIGHTS

Deben UK has been a long-standing LIMA partner, with its tensile compression stages widely used on the ESEM, as well as the AFM and profilometer. In recent research, LIMA and Deben researchers used the



SWEETCORN *from 17th century copper printing plate: When botanist, Robert Morison, created* Praeludia Botanica *in 1669 – including a huge collection of plants and herbs – he engraved a series of plant structures onto copper printing plates, which until a few years ago were thought to be lost. Now found, Dragnevski and colleagues used the Alicona profilometer to image the plates.*



ENVIRONMENTAL SEM *image of a tendon from a rat tail strained to 10%*

LIMA *is home to several stages manufactured by Deben UK*

ESEM with an enhanced cool-stage – operating from -50°C to +50°C – to study how PMMA latex films form and also to image freeze-dried structures at high resolution.

As Dragnevski points out, extending the temperature range of the cool-stage in effect converted the SEM chamber into a freeze-drying facility. In this way, the structure and features of the PMMA film could be preserved at different stages of formation, and imaged at high resolution without conductive coatings and at lower chamber gas pressures. Using AFM, the researchers also obtained additional high resolution images of the dried films and could measure how drying rates affected adhesive properties.

"A typical cooling stage would operate from –25°C to +50°C but we really needed to take this lower, so worked with Deben to develop this new capability," says Dragnevski.

"We also used this stage to look at the impact of transportation conditions, including temperature, on the mechanical strength of chocolate," he adds. "This was fun as once you've imaged your samples, you can eat them."

Dragnevski was also recently involved in devising an in-situ method based on sound measurements to study how volcanic sand from Mount Etna cracks. The experimental set-up included Deben's 5 kN micromechanical testing stage, as well as the Alicona profilometer and a desktop microphone.

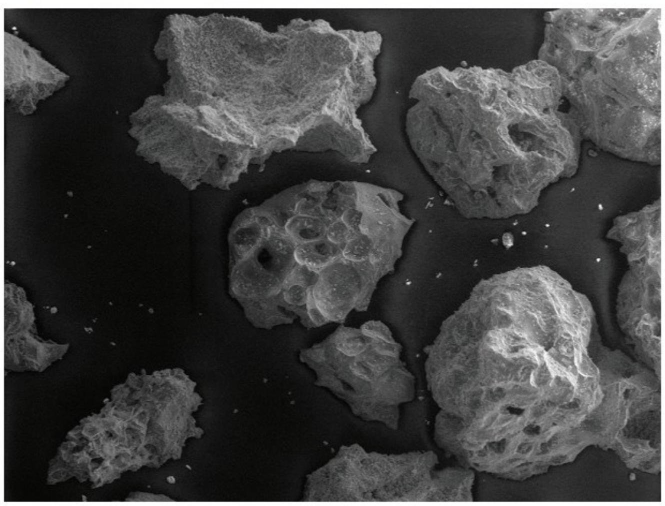
Grains of sand were confined within a steel container covered with thick glass, which was inserted into the tensile-compression stage for mechanical testing. During uniaxial compression tests, images were acquired with the profilometer while sound was recorded with the microphone, allowing Dragnevski and colleagues to analyse the failure mechanics of single grains of sand. The researchers now hope to use the method to study how crack initiate and propagate in concrete, metals, ceramics and composites.

"Again we slightly redesigned the Deben stage to do this and were able to link this with both optical and also electron microscopy, as well as mechanical testing," says Dragnevski.

Indeed, adapting or redesigning equipment according to experimental and materials needs lies at the very heart of what LIMA does. As Dragnevski highlights: "A lot of time and effort is spent on perfecting an experiment in order to get reliable data… it's all about really understanding a sample and an individual piece of equipment, be it a mechanical or cooling stage."

And for Dragnevski and LIMA colleagues, equipment design and development only gets more exciting. Right now, and as part of an ongoing UK Research and Innovation project with Deben and Oxford University researchers are set to deliver a new generation of mechanical heating stage. When used in an *in-situ* SEM set-up, the researchers will be able to scrutinise grain growth in materials systems including novel alloys, lightweight ceramic and metal matrix composites, at high temperatures and under extreme environments.

"There are still many things that aren't understood about grain growth at these high temperatures



– for example we recently observed abnormal growth in steel heated up to the austenitic region," says Dragnevski. "We are really pushing the boundaries of mechanical testing here and can observe what happens in real-time, *in-situ*, in a certain phase, and not post-mortem."

But what about the rat tail tendons? Clearly Dragnevski is pushing the boundaries of mechanical testing in more ways than one. Having assisted researchers from the University of Oxford's musculoskeletal arm – the Botnar Research Centre – to assess the mechanical properties of these rodent tendons, Dragnevski has now moved onto the human meniscus.

This tissue is crucial to load transmission, stability and energy dissipation in the knee joint but today's implants can't yet mimic its important biomechanic properties. However, Olga Barrera from Oxford Brookes and the University of Palermo, Italy, has turned to LIMA to build on mechanical tests to characterise the meniscus.

"We are still at a very early stage with this work and in terms of health and safety, it's going to be interesting to bring a human meniscus into our Engineering department," says Dragnevski. "But with the microscopes and mechanical stages we have absolutely the right set of tools to enable this study."

Looking to the future, the LIMA Facility Manager is also excited about his facility's growing Thermomechanical Laboratory which offers dynamic mechanical analysis, differential scanning calorimetry and rheometry to characterise polymers, pharmaceuticals, food stuffs and more. Mechanical testing, including tensile compression testing at higher loads, is soon to be launched as is digital image correlation. And as Dragnevski says, the entire laboratory is 'starting to take shape'.

"This is all about growing our entire facility and sustaining our expanding activities," he adds. "You know, some people think that experimental research really isn't exciting, but actually it is."

TESTING TIMES, *top, Dr Kalin Dragnevski from LIMA*

VOLCANIC SAND, *above, from Mount Etna: Dragnveski and colleagues devised a novel in-situ sound-based method to study how sand particles fracture*

ELECTRON BACKSCATTER *diffraction image of carbon steel at 850°C: LIMA is developing an in-situ SEM set-up to study grain growth in materials systems at high temperatures*