

In Focus – our new FESEM

Our newly acquired FEI Verios Field Emission Scanning Electron Microscope (FESEM) has provided ANFF ACT Node with several unique imaging and analytical capabilities. These include:

- High resolution secondary electron imaging (0.7nm@30kV, 1nm@1kV)
- Coating free imaging of charging samples
- Backscatter electron imaging (lossless, low angle and high angle)
- Scanning transmission electron microscopy imaging (BF, DF and HAADF)
- Cathodoluminescence imaging, spectroscopy and mapping
- EDX spectroscopy and mapping
- Gigapixel imaging.

The Verios has two detectors for imaging using secondary electrons (SE), the Evaheart-Thornly detector (ETD) and the through lens detector (TLD). In the column there are also two backscatter (BS) detectors, the middle detector (MD) and the in-column detector (ICD), which allow the collection of BS images without the need to insert a detector. This enables BS images to be collected with short working distances and on tilted samples. An insertable concentric backscatter detector (CBS) is also available for large area backscatter imaging or when the BS signal is weak. A scanning transmission electron microscopy (STEM) detector is also available on the Verios which is able to collect bright-field (BF), dark-field (DF) and high-angle annular dark-field (HAADF) images simultaneously. The STEM detector can be used in conjunction with EDX for the collection of high resolution X-ray maps. Figure 1 shows images of mesoporous SiO_2 with NaYF_4 cores collected with the STEM detector and TLD detector simultaneously.

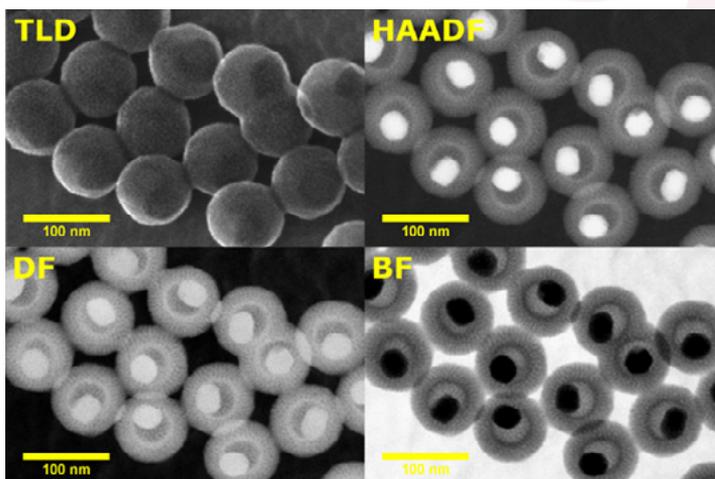


Figure 1: STEM images of mesoporous SiO_2 with NaYF_4 cores. Images were collected at 30kV with a beam current of 6pA using the TLD, DF, BF and HAADF detectors simultaneously.

Using FEI Maps software, zoomable gigapixel images can be created through the stitching of images, allowing for samples to be imaged in high resolution over large areas. FEI Maps automates the collection

process which enables the collection of gigapixel images using a range of detectors, beam conditions and sample areas. Figure 2 shows an image of shale collected with the CBS detector using FEI Maps. This image was constructed from 192 images covering a total area of $500 \times 300 \mu\text{m}$. The zoomed insert shows the level of detail in the original map.

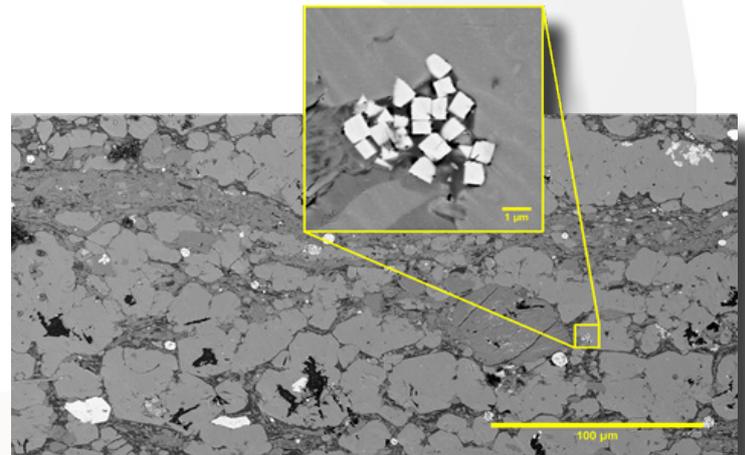


Figure 2: Large area stitched image of shale collected using the CBS detector. The image was collected using FEI Maps and is constructed from 192 images.

In insulating samples charging occurs when the injected charge, from the electron beam, is greater than the emitted charge, from SE and BS electrons. The Verios FESEM has several features for assisting in the imaging of charging samples. The imaging quality is maintained at low-voltages (500V to 1kV) which, for most samples, will balance the emitted and injected charge. In conjunction with its low-voltage imaging capabilities, the Verios has a beam-deceleration (BD) mode which applies a decelerating voltage between the sample and polepiece (50V to 4000V). The effects of BD mode are twofold:

- The decelerating bias allows for the landing energy of the electron beam to be further reduced down to 30V whilst maintaining the imaging resolution.
- The decelerating voltage assists in the extraction of SE allowing the sample to reach a charge balance.

Figure 3 (overleaf) shows an image collected from an insulating plastic film showing the micro-pores generated after the film has been stressed. The image was collected using BD mode with a landing voltage of 1kV, decelerating voltage of 1kV and a beam current of 13pA.

The Verios is also equipped with a Gatan MonoCL4 system which allows for the collection of cathodoluminescence (CL). CL is the light emitted from a sample under the excitation of the electron beam. The MonoCL4 system is able to collect images, spectra and full spectral maps and has detectors that can cover the wavelength range from 200nm to 2000nm. Figure 4 shows a SE image and a RGB CL image of quartz that has been fractured and regrown hydrothermally. The blue regions show the original crystal grains while the orange regions show where the crystal has regrown. If this tool has capabilities that can assist your research please contact us. Training is available, or you can take advantage of using our skilled process engineers to help you achieve your goals.

In Focus – our new FESEM (continued)

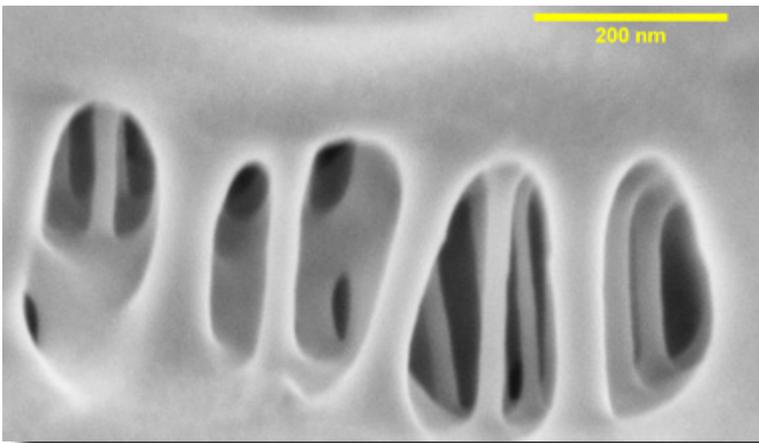
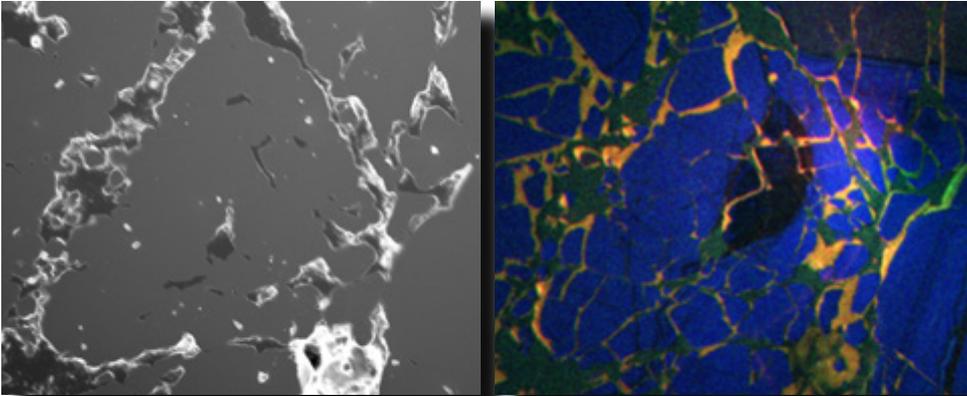


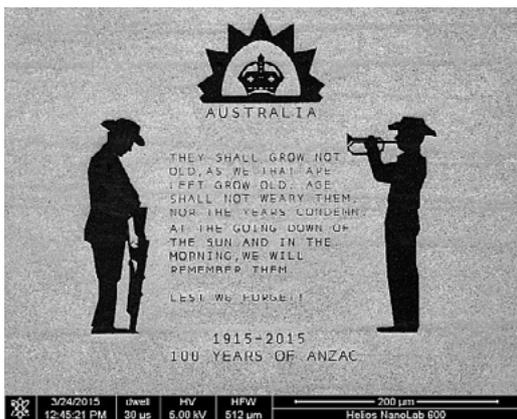
Figure 3 (above): Image of an insulating plastic film showing the pores generated after deformation. The image was collected with a landing voltage of 1kV, decelerating voltage of 1kV and a beam current of 13pA.

Figure 4 (below): Secondary electron (left) and RGB CL (right) images of regrown quartz. In the CL image the blue colour shows the quartz grains while the yellow shows where the quartz crystals have started to reform. (Figure 4 images courtesy of Kathryn Hayward, RSES ANU.)



Story and images by Mark Lockrey, Microanalysis Research Officer/SEM Process Engineer, ANFF ACT Node

A tiny tribute for the ANZAC Day centenary



Our newsletter cycle meant we needed to wait until well after ANZAC Day to get this 'little' story out - but thought it should be shared.

This unique tribute to the ANZACs was made using the nanotechnology tools at the ACT Node of the Australian National Fabrication Facility ANFF. Atoms were fired into gold to create the tiny picture - the soldiers are thinner than a human hair and the bugle is the size of a red blood cell.

The ANZAC pattern was fabricated by using Focused Ion Beam (FIB) system at the ANFF by Dr Evgeny Mironov, a recently

graduated visiting PhD student from ADFA, to commemorate the 100 years of ANZAC (1915-2015).

The structure was milled by accelerated gallium ions, which bombarded a 300 nanometer thick golden film deposited on top of the quartz substrate. The pattern was created by vectoring the original image into 900 individual elements and uploading their coordinates to the specially designed patterning script. The structure has the dimensions of 375 by 375 micrometers with the smallest features less than one micrometer in size.



Welcome to our winter issue! It's strange how things work out sometimes. Our new

FESEM, which was installed earlier this year, is the main subject of our newsletter this issue. While there has been a lot of interest in this new tool we thought it important to highlight the capabilities now that we have had it running for a few months and bring these to the attention of our Node users. We had planned this article a few weeks ago. Now for the strange part. We have just begun our 5th Annual User Survey and - without giving too much away - a suggestion from one of the responses already received for our newsletter was that we run a story each issue on one of our tools (new and old) highlighting the capabilities etc. Maybe we can get into fortune telling as well! We will try and highlight a tool each issue from now on - that will keep us going for years!

On the subject of our survey, this will run for the month of June and an invitation to participate has been circulated to all users that have accessed our facilities at the ACT Node in the past 12 months. If you have received the email we encourage you to send in your responses as it genuinely assists us in delivering better services to you all. A lot of people don't add comments (I personally find it hard at times to add relevant comments in surveys - so don't feel too bad), but given the ANFF is looking to the future one thing you could suggest is a 'wish list' of tools/capabilities that could assist you in your future research. Further incentives to participate is the prize that include eight hours of free access time, a 7" Android tablet and an ANFF polo shirt for one lucky participant. We also welcome input from people receiving the newsletter and/or use our website - although the access time might not be of use to you, but don't let that put you off. You can access the on-line survey form here:

http://www.supersaas.co.uk/form/ANFF_ACT_Node/Feedback_Form

Cheers, JK

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