



Research and Development Laboratory Nanores

An offer for
Welds, sinters etc.

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ABOUT NANORES



Nanores is a hi-tech, independent research and development laboratory, set to provide the highest quality service and improve standards of cooperation between science and business. Through the use of state of the art equipment and by creating a team of specialists in various fields (physics, mathematics, chemistry, materials science), we are able to efficiently identify needs and provide the best solutions for our partners.

We are specialized in analysis and modification of structure of hard materials, both conductive and non-conductive. Our laboratory is equipped with electron and ion Dual Beam microscopes (SEM/Xe-PFIB, SEM/Ga-FIB), and Atomic Forces Microscope (AFM) with multiple advanced 2D and 3D imaging modalities. We offer unique ability of surface and volume imaging and analysis in nanometric scale including the identification of the atomic composition. Beforementioned services allow to reveal manufacturing micro and nano defects along with verification of their causes, supporting production optimization processes. We provide services for the production and design of micro and nano prototypes of photonic, mechanical, electronic and other structures.

OUR EQUIPMENT



1. DualBeam SEM/Xe-PFIB system (1st in Poland, 2nd in Europe) FEI Helios G4 PFIB CXe
2. DualBeam SEM/Ga-FIB system FEI Helios NanoLab 600i
3. AFM Nanosurf FLEX Axiom
4. EDS detector Bruker XFlash 630 mini
5. High vacuum coater Quorum Technologies Q150T E
6. Plasma Cleaner PDC-32G-2
7. Ultrasonic Bath Sonic 2
8. Stereoscopic Microscope Motic Z-171-TLED

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OUR OFFER



1. Capabilities of Ga-FIB and Xe-PFIB microscopes
2. Analysis of large cross-sections made with Xe-PFFIB technology
3. Automated determination of particle size based on SEM images
4. Phase contrast sinters analysis

1. Capabilities of Ga-FIB and Xe-PFIB microscopes

Xenon plasma focused beam technology enables performing new research not possible with other testing methods. Together with an ultra-high resolution electron microscope (SEM/Xe-PFIB FEI Helios G4 PFIB CXe Microscope) and a fast EDS detector by Bruker, it is the only such analytical system in Europe. The energy of focused ion beam enables selective removal and modification of a specimen in a nano-scale. Microscopes in the Research and Development Laboratory of Nanores enable ultra-high resolution imaging of the surface of specimens. At the same time, it is possible to create elemental maps, as well as point and linear analyses with EDS detectors. This technology enables creating cross-sections, 3D reconstructions and performing quick, maskless prototyping processes in nano- and microscale. Electron microscopy, as opposed to optical microscopy, is characterised by exceptionally large depth of field and enables extremely high magnification (up to 1 000 000 times). It is possible to obtain a substantial amount of information on material's phases and to show the material contrast of a specimen depending on the configuration of the electron microscope detectors. SEM microscopes enable specifying the characteristics of a wide spectrum of conductive and non-conductive materials without modifying them. They also enable fast production of micro-scale prototypes of spatial structures. The following are the main advantage of using a Xenon beam (Xe-PFIB):

- 50 times faster operation in comparison with the Gallium technology (Ga-FIB),
- lack of ion implantation in specimens during polishing processes and ion etching,
- thinner amorphous layer developed during ion microscope operation.

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Comparison of technical parameters of dual beam microscopes owned by Nanores.

	SEM/Xe-PFIB	SEM/Ga-FIB
Accelerating voltage	SEM: 350 V - 30 kV Xe-PFIB: 2 kV - 30 kV	SEM: 350 V - 30 kV Ga-FIB: 500 V - 30 kV
Resolution	SEM: <1 nm Xe-PFIB: <25 nm	SEM: <1 nm Ga-FIB: 2.5 nm
Specimen limitations	diameter: 150 mm with full rotation of the specimen admissible height: 100 mm admissible weight: 500 g	
Detectors	ETD (Everhart-Thornley Detector) TLD (secondary electron detector designed by FEI) ICE (semiconductor secondary electron detector designed by FEI)	ETD (Everhart-Thornley Detector) TLD (secondary electron detector designed by FEI)
	EDS (Bruker XFlash 630 mini)	—
Lamellas	thickness below 100 nm	
	compatibility with materials based on aluminium and gallium	—
3D	three-dimensional reconstruction of a specimen based on SEM (volume of approx. 1 000 000 μm^3)	—
Milling	xenon implantation	gallium implantation

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2. Analysis of large cross-sections made with Xe-PFIB technology

The strength properties of sinters depend on many factors, one of them is porosity, which significantly determines their hardness, brittleness and, as a consequence, reliability. A useful tool in the design and optimization of sintered microstructures is an electron microscope with an ion cannon due to its ultra-high imaging resolution and the ability to make cross-sections of sinters. Unfortunately, the analysis of large cross-sections using a conventional SEM/Ga-FIB microscope is a very time-consuming process due to the long-time cross-section completion. However, the use of technology using the xenon plasma beam significantly shortens the time of cross-section realization. Below are some examples comparing the work of both microscopes: SEM/Ga-FIB and SEM/Xe-PFIB.

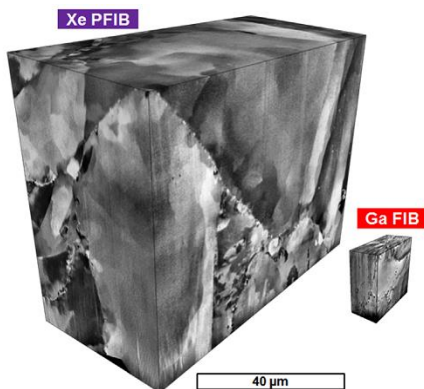


Fig. 1 3D reconstructions of the removed fragment of stainless steel made using Xe-PFIB-and Ga-FIB, the duration of both processes was the same. [Source: FEI.com]

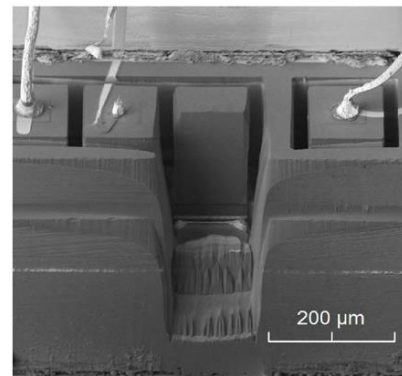
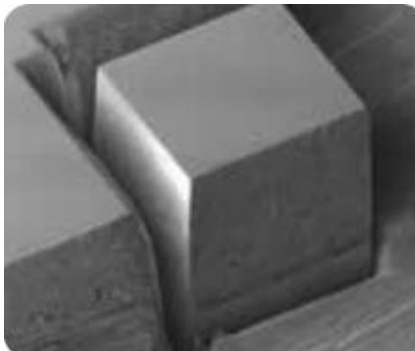
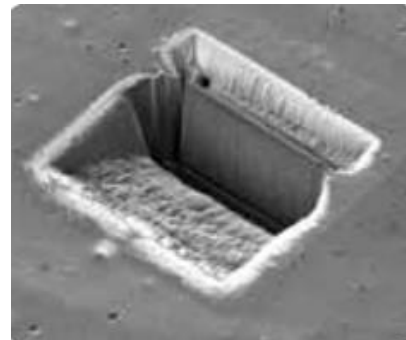


Fig. 2 Cross section of the MEMS sample. Work time: Ga-FIB 40 hours, Xe-PFIB 1 hour 20 minutes. [Source: FEI.com]



Working time of Xe-PFIB: 2 h
Working time of Ga-FIB: 48 h



Working time of Xe-PFIB: 1,5 h
Working time of Ga-FIB: 36 h

Fig. 3 Comparison of operation efficiency of Xe-PFIB and Ga-FIB microscopes during micro-milling. [Source: FEI.com]

Local micro-sections made using Xe-PFIB technology can reach depth of hundreds of micrometres. This is especially important when analyzing sub-millimeter objects. Figure 4 shows examples of deep micro-sections.

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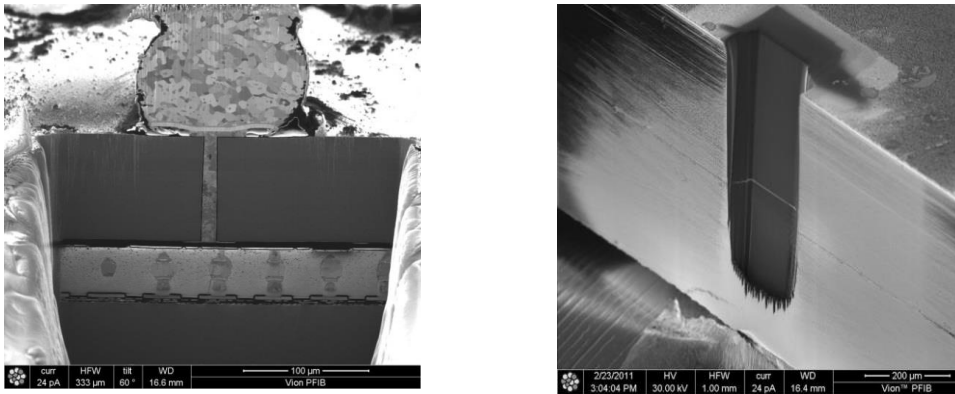


Fig. 4 Local micro-sections made using Xe-PFIB – milling depth above 100 μm . [Source: FEI.com]

3. Automated determination of particle size based on SEM images

Imaging materials using an electron microscope is a short process, but the subsequent interpretation of SEM images requires time, knowledge and experience. In this case, FEI "iFast" software is very helpful. It is a software consisting of two parts. The first one is called "iFast Developer's Kit" and is designed to create, modify and manage scripts that automate various functions and processes of the FEI DualBeam microscopes. The second part is called "iFast Runner" and is used to run previously prepared scripts. The use of this software is unlimited, thanks to this it is possible to automate such processes as determining the size of particles, grains or pores based on SEM images.

4. Phase contrast sinters analysis

The sinters can be made of one, several, and many different materials. Proper determination of component proportions and adjustment of sintering process parameters allows control of strength properties of sinters. In many cases, it turns out that even the minimum difference in the proportion of components has a significant effect on the parameters of the final sinter. Electron microscopes equipped with an ion cannon allow to visualize subtle differences in the composition of the material. The information about phase contrast, which indicates the material differentiation, is based on the detection of signals from the material tested. The areas of the sample with different material composition react differently with the ion beam bombarding the surface. Therefore, the signal coming from different regions received by the detectors also differs, which is clearly visible on the generated images from the ion microscope. Examples of images showing phase contrast imaged with an ionic microscope are shown in Figure 5.

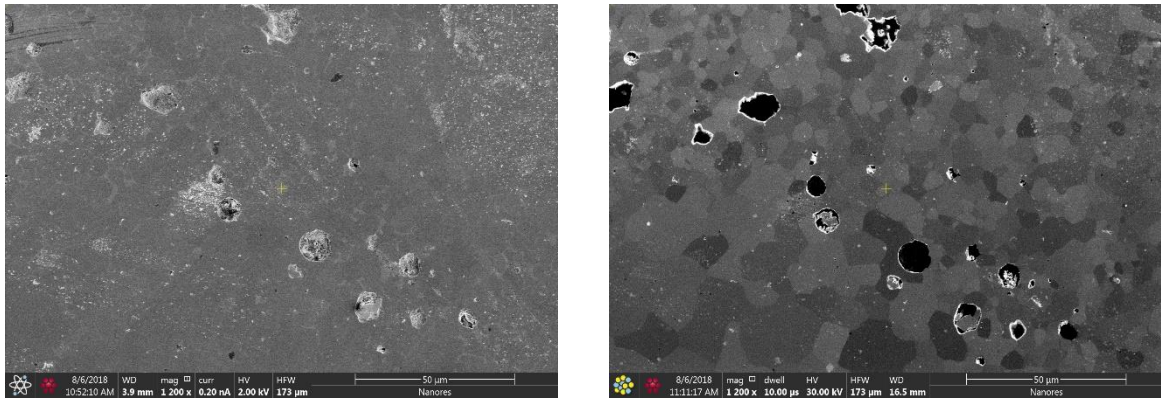


Fig. 5 SEM image (left) and ion microscope image (right) of a sample of the titanium composite doped with several elements. The SEM image shows the topography well, and the image from the ion microscope shows the phase contrast resulting from a different content of elements in different areas.

An ideal complement to material research is the EDS analysis, which provides information on the elemental composition of the material under study. The example is presented below.

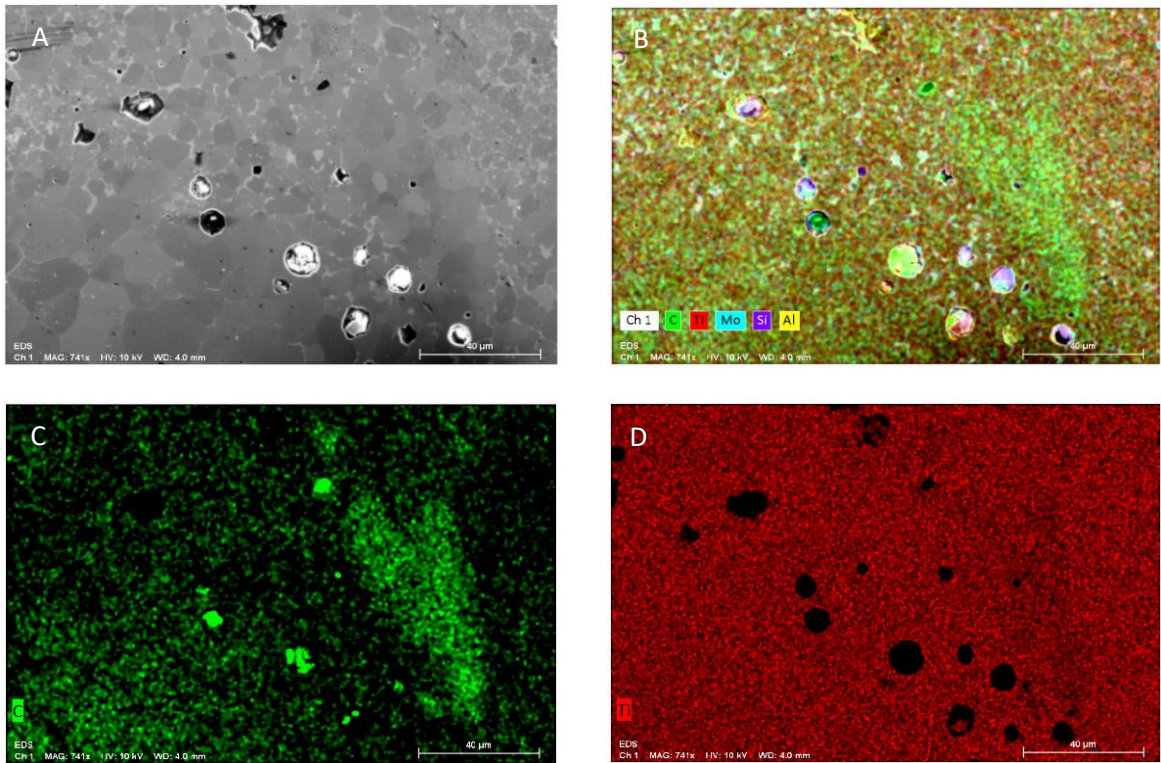


Fig. 6 (A) Ion microscope image of the analyzed area, (B) the elemental map of all detected elements made using the EDS detector, the elemental map separately for carbon (C) and titanium (D).

Thank you for reading our offer. Please contact us should you have any questions.

Nanores Team

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Politechnika Wrocławska



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