



# 2<sup>nd</sup> international HeFIB conference on Helium and emerging Focused Ion Beams

Dresden, June  $11^{\rm th} – 13^{\rm th},\,2018$ 



# $2^{\mathrm{nd}}$ international HeFIB

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### Orientation

#### Hotel Innside Dresden

Salzgasse 4 01067 Dresden

#### **Conference dinner in the Restaurant Luisenhof** Bergbahnstraße 8 01324 Dresden





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Zeitbedarf ( Stunden:Minuten				00:39			00:25			00:28			00:25

#### TRAM connections after conference dinnner

# Contributions

1	Monday					
	1.1	Graphene nano-electro- mechanical (GNEM) devices functionalized by using helium ion				
		beam	2			
	1.2	Anderson localization of graphene by sub-				
		nm diameter helium ion beam irradiation	4			
	1.3	In-situ measurement of electron conduc-				
		tion modulation in graphene by helium				
		ion beam irradiation	6			
	1.4	He-Ion Microscopy Combined with Mi-				
		cromanipulators for Pick and Place, Elec-				
		trical and Mechanical Characterization,				
		and Other Tasks	8			
	1.5	Helium and neon ion microscopy and milling				
		for microbiological applications	10			
	1.6	FIB/SEM Processing of Biological Samples	12			
	1.7	Correlative Microscopy with Light, Elec-				
		trons and Ions in Environmental Micro-				
		biology	14			
	1.8	Comparison of image fusion techniques				
		for ion beam based correlative microscopy	16			
	1.9	Multimodal Imaging for Physical and Chem-				
		ical Surface Characterization using a Com-				
		bined Helium Ion Microscope and Sec-				
		ondary Ion Mass Spectrometry Platform	18			

2

1.10	ColdFIB – a new FIB column with a laser	
	cooled source	20
1.11	New non - Gallium FIB Alternatives for	
	Nanofabrication	22
1.12	Ion Sources for Focused Ion Beam Appli-	
	cations	24
1.13	Source Shot Noise Mitigation in Helium	
	Ion and Focused Ion Beam Microscopy .	26
1.14	The Xe plasma FIB - the other extreme	30
1.15	Revolutionary Field Emission Thruster De-	
	velopments for Nano-satellites at TU Dres-	
	den and its correlative LMIS-applications	
	at terrestrial Implanters	32
-		24
lue	sday	34
2.1	Thermal Effects from Light Ion Beams in	~ .
	Thin Films	34
2.2	FIB milling strategies for the mitigation	
	of beam-induced artifacts in polymer thin	~ ~
	films: He vs. Ga	36
2.3	Metallography of Thin Films with Focused	
	Ion Beams	38
2.4	Compositional analysis and in-situ exper-	
	iments in the HIM	40
2.5	SIMS performed on the Helium Ion Mi-	
	croscope: a unique tool for highest spatial	
	resolution imaging and correlative microscop	oy 42
2.6	Transmission He Ion Microscopy	44
2.7	Towards observing diffraction in a HIM .	46
2.8	Multi-Modal Characterization with Sec-	
	ondary Ion Mass Spectrometry on ZEISS	
	ORION NanoFab	48

62

2.9	Characterising Carbon-based materials in the HIM: probing the invisible with sec- ondary electron spectroscopy	50
2.10	Understanding Focused Ion Beam Sput-	
	EnvizION Monte Carlo Simulation	52
2.11	Helium Ion Based Lithography of Advanced	
	Resists	54
2.12	Reduced proximity and improved depth of field by using resist-based lithography	-
	with helium ions	56
2.13	Advanced FIB nanopatterning employing a high precision sample stage	58
2.14	Fabrication of Plasmonic Nanostructures	
	by He+ and Ga+ Milling	60

#### 3 Wednesday

3.1	A revolutionary quantum computer de- vice fabricated from implanted ordered	60
	arrays of single donor atoms in silicon	62
3.2	Binary Collision Computer Simulation of	
	FIB Induced Erosion and Atomic Mixing	
	in Nanostructures	64
3.3	Site-controlled Si Nanodot Formation for	
	a RT-SET via Ion Beam Mixing and Phase	
	Separation	66
3.4	Latest development for failure analysis –	
	When ions meet chemistry	68
3.5	Cs ion coldbeam suitability for circuit edit	
	and other nanomachining applications .	70
	Ŭ •••	

	3.6	Nanoscale defect engineering in ferroelec-	
		tric thin films by focused ion beam mi-	
	0.7	croscopy	72
	3.7	Modification of MoS2 with Helium Ion	
		Beam: Fabrication of Gate-Tunable Mem-	74
	<b>n</b> 0	ristor	14
	3.8	Application of Gas Field Ion Source to	70
	2.0	Patterning Nanoscale Magnetic Structures	10
	3.9	Writing Magnetic Domains with a He-	70
		num ion Microscope	18
4	Post	ter Presentations	80
	4.1	Nanoscale Engineering of Metal Nanopar-	
		ticles in Dielectric Films by Energetic Ions	
		for Some Novel Applications	80
	4.2	FIB Nanopatterning of Metal Films on	
		PMMA Substrates: Non-Sputtering Mode	82
	4.3	Helium Ion and Optical Microscopy of	
		Spider Silk	84
	4.4	On the preparation of dechlorinating bac-	
		teria for scanning electron microscopy	86
	4.5	Scanning Transmission Helium Ion Mi-	
		croscopy on 1nm Thick Carbon Nanomem-	
		branes	88
	4.6	Molecular dynamics simulations of He ion	
		channeling in Gold nanoclusters	90
	4.7	Fundamental focus beam-solid interactions	
		and applications for rapid prototyping .	92
	4.8	Helium Ion Microscopy for the imaging of	
		biological samples	94
	4.9	HIM-SIMS: a powerful tool for analysis	
		of energy and micro-electronic materials	96

4.10	Scanning Transmission Ion Detection in	
	the Helium Ion Microscope	98
4.11	A rubidium focused ion beam instrument	100
4.12	Quantification of HIM imaging resolution	102
4.13	Interaction of an Energetic Ar Molecular	
	Cluster Beam with Graphene	104
4.14	Cleaning of Pt deposits with an in-situ	
	low-energy Ar ion beam	106
4.15	Nanofabrication using He ions - from 2D	
	materials to nanometer sized gaps	108
4.16	Magneto-transport measurements in para-	
	and ferromagnetic Fe60Al40 wires	110
4.17	Correlative In-Situ Analysis of Helium Ion	
	treated Graphene Membranes with AF-	
	$SEM^{TM}$	112
4.18	Nano-patterning of $\beta$ -Ga <sub>2</sub> O <sub>3</sub> nanostruc-	
	tures with He and Ne focused ion beams	114
4.19	Atomic force microscopy of HIM milled	
	be vels of thin films and nanostructures $% \left( {{{\left( {{{{{{{}}}}} \right)}}}_{i}}} \right)$ .	116

# 1 Monday

# 2 Tuesday

# 3 Wednesday

# 4 Posters

# 1 Monday

# 1.1 Graphene nano-electromechanical (GNEM) devices functionalized by using helium ion beam

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Monday 10:40 - 11:05 (+3)

We first show single-nanometer suspended graphene nanoribbons (GNRs) milled with a Helium ion microscope (HIM). The 6 nm GNRs are patterned, and the electrical conduction is measured as function of temperature. We discuss the milling results and electrical characterization along with their impact on the performance of the graphene nanoelectromechanical (GNEM) singlemolecular sensor. We then report on fabrication of largearea graphene nanomesh (GNM) by patterning the 2D array of pores with a diameter < 4 nm with the neck of down to 10 nm. Electrical transport measurements reveal an effective energy gap opening of up to ~50 meV. The impact of the GNM structure is discussed on the formation of THz phonon bandgaps for thermal engineering applications. The authors acknowledge T. Iijima for the usage of the HIM at AIST SCR Station.

This research was supported through KAKENHI 25220904, 16K13650, 16K18090 from JSPS and COI program of the Japan Science Technology Agency.

# 1.2 Anderson localization of graphene by sub-nm diameter helium ion beam irradiation

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Monday 11:10 - 11:25 (+3)

Irradiation of helium ions to a single-layer graphene (SLG) by HIM controllably generates defect distributions, which create a charge carrier scattering source within the SLG, and it was applied to tune electrical properties [1]. In this work, metal-insulator transition in SLGs on Si/SiO2 sub. induced by Anderson localization [2] was characterized. The transition was investigated using SCM with increase of ion dose. Results showed that a defect density of more than  $\sim 1.2\%$  induced Anderson localization. The localization length was investigated by comparison of ion irradiated and formed insulator regions, and it was estimated several dozen nanometers, no fewer than 10 nm and no more than 25 nm(3,4). The HIM at AIST SCR station was used for this work.

[1] S. Nakaharai, S. Ogawa *et al.* ACS Nano 7, 5694 (2013)

[2] A. Lherbier *et al.* Phys. Rev. B 86, 075402 (2012)

[3] Y. Naitou and S. Ogawa, Appl. Phys. Lett. 106, 033103 (2015)
[4] Y. Naitou and S. Ogawa, Appl. Phys. Lett. 108,

171605 (2016)

# 1.3 In-situ measurement of electron conduction modulation in graphene by helium ion beam irradiation

Shu Nakaharai<sup>1\*</sup>, Shinichi Ogawa<sup>2</sup>, Elisseos Verveniotis<sup>1</sup>, Yuji Okawa<sup>1</sup>, Masakazu Masakazu<sup>1</sup>, Christian Joachim<sup>1</sup>

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Monday 11:30 - 11:45 (+3)

We report real-time detection of modulation of electric conduction in graphene by helium ion beam irradiation, which revealed that the conductance decays exponentially as the length of irradiated region increases. For this measurement, we have developed an *in-situ* measurement system of current-voltage characteristics in a helium ion microscope. Helium ion beam irradiation can generates defects in graphene with an extremely high spatial resolution of a nano meter scale without using resist, and also, the small size of the ions enables to generate atomic size defects. These point defects work as scattering centers for the two-dimensional electron system, and consequently, they can induce interference of electron waves. It is discussed that the exponential dependence of conductance on the length of the irradiated region is induced by the localization of electrons. We would like to thank T. Iijima and Y. Morita for the usage of the AIST SCR HIM.

# 1.4 He-Ion Microscopy Combined with Micromanipulators for Pick and Place, Electrical and Mechanical Characterization, and Other Tasks

Andrew Jonathan Smith<sup>1\*</sup>, Klaus Schock<sup>1</sup>, Andreas Rummel<sup>1</sup>, Matthias Kemmler<sup>1</sup>, Stephan Kleindiek<sup>1</sup>

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Monday 11:50 - 12:05 (+3)

He-Ion microscopes are experiencing ever-increasing popularity due to their unique imaging capabilities - especially for samples/materials that are sensitive to incident beams with high energies. While the platforms already offer a wide range of analytical options such as EDX as well as sample modification using other ionbeams (e.g. Ga), adding a set of micromanipulators yields additional possibilities in regard to arranging particles or assembling small devices *in-situ*. In addition, materials can be characterized using force measurements or through electrical probing experiments. This work will give an overview of how micromanipulators can be integrated into He-Ion microscopes and some application examples of their use.



Figure 1: A set of four micromanipulators mounted on the He-FIB tilt stage

# 1.5 Helium and neon ion microscopy and milling for microbiological applications

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Monday 14:00 - 14:25 (+3)

Imaging of microbial interactions has until now been based on well-established electron microscopy methods. In this talk I review our recent drive to study microbiological samples using a helium ion microscopy (HIM). The main focus will be given on bacterial colonies and interactions between bacteria and their viruses, bacteriophages, which we imaged *in-situ* on agar plates [1]. Other recent biological applications will also be briefly discussed. In biological imaging, HIM has advantages over traditional scanning electron microscopy with its sub-nanometer resolution, increased surface sensitivity, and the possibility to image nonconductive samples. Furthermore, by controlling the He beam dose or by using heavier Ne ions, the HIM instrument provides the possibility to mill out material in the samples, allowing for subsurface imaging and *in-situ* sectioning.



Figure 2: HIM micrograph of individual T4bacteriophages attached onto e. coli bacterial cell surfaces, some with contracted tails indicating genome injection.

[1] M. Leppänen, L.-R. Sundberg, E. Laanto, G. Almeida,
P. Papponen and I. J. Maasilta, Advanced Biosystems 1, 1700070 (2017)

### 1.6 FIB/SEM Processing of Biological Samples

Annalena Wolff<sup>1\*</sup>, Nico Klingner<sup>2</sup>, William Thompson<sup>3</sup>, Yinghong Zhou<sup>4,6</sup>, Jinying Lin<sup>5,6</sup>, Yong Peng<sup>7</sup>, John Ramshaw<sup>7,8</sup>, Yin Xiao<sup>4,6</sup>

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Monday 14:30 - 14:45 (+3)

The FIB/SEM has become the "go to" tool in the materials sciences and semiconductor industry, however, it is not yet fully established in the biological sciences. This is predominantly due to the heat-induced damage from the ion beam when processing soft materials including biological samples. This study focuses on the underlying ion-solid interactions and the effect of processing parameters on heating induced by ion beams. The interactions of gallium ions in skin were simulated using Monte Carlo methods (SRIM) and the ion beam induced temperature increases are estimated using using Fourier's law of conductive heat transfer. The theoretical results are compared to experimental results from FIB processing of collagen. Collagen was chosen as a suitable test sample as it loses its fibrillary structure when denaturated by heat, permitting damage to easily be recognized. The results show that heat damage avoided when using the proposed heat reducing approach.

### 1.7 Correlative Microscopy with Light, Electrons and Ions in Environmental Microbiology

Matthias Schmidt<sup>1\*</sup>, Nedal Said<sup>1</sup>, Jairo Moreno<sup>1</sup>, Hryhoriy Stryhanyuk<sup>1</sup>, Niculina Musat<sup>1</sup>, Hans Richnow<sup>1</sup>

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Monday 14:50 - 15:05 (+3)

The ProVIS - Centre for Chemical Microscopy is specialized on the visualization of biogeochemical processes on cellular level by means of correlative microscopy. It comprises of equipment for sample preparation, different light microscopes, electron (SEM) and helium ion microscopes (HIM) as well as imaging secondary ion massspectrometers. Correlative work-flows that range from preparation of the sample via chemical imaging to digital image processing have been established. An example of an algal biofilm that was micro-imaged correlatively by light, electrons and ions is presented. Pre-condition for correlative chemical microscopy is an appropriate preparation of the sample such that morphology and chemical composition are preserved at the same time. For that, we have developed protocols for the preparation of bacteria that compromise between integrity of the cells and preservation of their chemical composition.



Figure 3: E.coli imaged by He-ion microscopy

The differently prepared samples were imaged by HIM.

### 1.8 Comparison of image fusion techniques for ion beam based correlative microscopy

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Monday 15:10 - 15:25 (+3)

The combination of different imaging techniques to better understand the properties of a sample is known as correlative microscopy (CM). In this contribution, we discuss two approaches for image fusion in the context of combining the inherently lower-resolution chemical images obtained using secondary ion mass spectrometry (SIMS) with the high-resolution ultrastructural images obtained using electron or ion microscopies. We show that the intensity-hue-saturation fusion method often applied for EM-sharpening can result in serious image artifacts, especially when different contrast mechanisms interplay. We therefore introduce and demonstrate Laplacian pyramid fusion as a powerful and more robust alternative method for image fusion. Both physical and technical aspects of correlative image overlay and fusion specific to SIMS-based CM are discussed in



Figure 4: Image fusion in the EM/SIMS context. Top left: HR-SEM of a stained mouse brain tissue sample; bottom left: SIMS map of the 15N-enrichment after an isotope uptake experiment. Right: Result of Laplace pyramid image fusion of EM and SIMS data. This fused image allows for reliable identification of cellular features while retaining the spectroscopic information of the SIMS data set.

detail alongside the advantages, limitations, and the potential artifacts.

[1] Vollnhals et al., Anal. Chem. 89 (2017), 10702.

# 1.9 Multimodal Imaging for Physical and Chemical Surface Characterization using a Combined Helium Ion Microscope and Secondary Ion Mass Spectrometry Platform

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Monday 15:30 - 15:45 (+3)

The goal of multimodal imaging is to transcend the existing analytical capabilities for nanometer scale spatially resolved material characterization at interfaces through a unique merger of advanced microscopy, mass spectrometry and optical spectroscopy. Combining helium ion microscopy (HIM) and secondary ion mass spectrometry (SIMS) onto one platform has been demonstrated as a method for high resolution spot sampling and imaging of substrates. To advance this approach and to expand its capabilities I will present our results of multimodal chemical imaging using this technique on test substrates and show application of this approach for the multimodal analysis of perovskite (HOIPs) materials. I will discuss the performance metrics of the multimodal imaging system on conductive and non-conductive materials and discuss our results on understanding the chemical nature of ferroelastics twin domains in methylammonium lead triiodide (MAPbI3) perovskite using HIM-SIMS.

# 1.10 ColdFIB – a new FIB column with a laser cooled source

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Monday 16:20 - 16:45 (+3)

In these last years, different ionic sources, have been developed to go beyond the Gallium FIB ultimate resolution. Indeed, to improve FIB resolution performances, two development axes can be considered: optimize the optics, but we can't expect an important gain, or introduce new kind of ion sources. The goal of courses of these new sources is to bring specifications which will help to fulfil a wide field of FIB applications. Among this application, it appears clearly that low energy performances become very important for FIB users and in this case the energy dispersion of the source is the main limiting factor. Furthermore, a source which has low energy dispersion will offer better resolution at higher energy (30 kV). In this objective we are developing a new source, based on a laser cooled cesium beam, that offer a really low divergence and energy dispersion and a two steps ionisation (Rydberg excitation + field ionisation) able to keep, as best as possible, this properties.



Figure 5: Carbon graphite: Example of images obtains with ColdFIB at 30 keV, with a different field of view, respectively 10 and 1  $\mu$ m.

### 1.11 New non - Gallium FIB Alternatives for Nanofabrication

Paul Mazarov<sup>1\*</sup>, Lothar Bischoff<sup>2</sup>, Wolfgang Pilz<sup>2</sup>, Sven Bauerdick<sup>1</sup>, Lars Bruchhaus<sup>1</sup>, Michael Kahl<sup>1</sup>, Ralf Jede<sup>1</sup>

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Monday 16:50 - 17:05 (+3)

Nanofabrication requirements for FIB technologies are specifically demanding in terms of patterning resolution, stability and the support of new processing techniques. Moreover the type of ion defines the nature of the interaction mechanism with the sample and thus has significant consequences on the resulting nanostructures [1, 2]. Therefore, we have extended the technology towards the stable delivery of multiple ion species selectable into a nanometerscale focused ion beam by employing a liquid metal alloy ion source (LMAIS) [3]. This provides single and multiple charged species of different masses, resulting in significantly different interaction mechanisms. We present the main aspects for improvements of LMAIS long time stability and usability and report on recent investigation of new alloys (sources).

- [1] L. Bruchhaus, et al. APR 4 (2017) 011302.
- [2] S. Bauerdick, et al. JVST B 31 (2013) 06F404.
- [3] L. Bischoff, et al. APR 3 (2016) 021101.

### 1.12 Ion Sources for Focused Ion Beam Applications

Lothar Bischoff<sup>1\*</sup>, Paul Mazarov<sup>2</sup>, Wolfgang Pilz<sup>1</sup>, Jacques Gierak<sup>3</sup>

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Monday 17:10 - 17:25 (+3)

A most important element of a FIB is the ion source. Main points are spot size, ion current, stability and ion species itself. At present half of periodic table can be used in FIBs to modify locally materials properties. The Liquid Metal (Alloy) Ion Source [1] is most popular with resolution of a few nm at pA current. Ionic Liquid Ion Sources emit an- or cations from compounds [2]. For volume removing ECR or plasma sources can help with Xe ions up to 2  $\mu$ A [3]. Presently Gas Field Ion Source initiated the Helium Ion Microscope [4]. The spot of 0.5 nm opens new prospects in imaging and nano-engineering. Another approach are magneto-optical trap ion sources will be compared.

[1] L. Bischoff et al. APR 3 (2016) 021101.

[2] A.N. Zorzos and P. Lozano, JVST B 26 (2008) 2097.
[3] A. Delobbe *et al.* Microsc. Microanal. 20 (2014) 298.

- [4] G. Hlawacek et al. JVST B 32 (2014) 020801.
- [5] B. Knuffman et al. AIP Conf. Proc. 1395 (2011) 85.

# 1.13 Source Shot Noise Mitigation in Helium Ion and Focused Ion Beam Microscopy

Minxu Peng<sup>1</sup>, John Murray-Bruce<sup>1\*</sup>, Karl K. Berggren<sup>2</sup>, Vivek Goyal<sup>1</sup>

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2. Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, Cambridge

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Monday 17:30 - 17:45 (+3)

State-of-the-art techniques for imaging samples with high resolution demand the use of microscopes. Recent demonstrations using Helium ion microscopy (HIM) can produce images with sub-nanometer resolution [1]. However, these technologies cause damage to samples: which is a fundamental limit to imaging with focused beams [2-4]. Indeed, sample damage can be controlled by reducing imaging doses. Consequently, studies analyzing damage and safe dose have appeared [5, 6]. Herein, we provide theoretical justifications for multiple low-dose measurements being more informative than a single one maintaining the same total dose. Next, we present a novel data processing strategy for these multiple lowdose measurements and demonstrate a lowered reconstruction mean-squared error (MSE). Our findings are verified in simulation using a sample with mean secondary electron yield ranging from 2 to 8 (as suggested by Notte *et al.* [7]), and obtain an MSE reduction by a factor of 2.4 (see Figure).

M. S. Joens, C. Huynh, J. M. Kasuboski, D. Ferranti,
 Y. J. Sigal, F. Zeitvogel, M. Obst, C. J. Burkhardt, K.
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 J. A. J. Fitzpatrick, Sci. Rep. 3, 3514 (2013).

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Figure 6: Simulated HIM experiment for a sample with mean secondary electron yield in [2,8]: (a) Ground truth image with secondary electron yield rescaled from 2 to 8. (b) Conventional HIM image with MSE of 0.5934. (c) Maximum likelihood estimation with MSE of 0.5513. (d) Our work without dose reduction with MSE of 0.2297. These results have not utilized spatial regularization.

# 1.14 The Xe plasma FIB - the other extreme

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Monday 17:50 - 18:05 (+3)

The Ga+ FIB has been around for many years and its beam has been optimized in two directions: higher resolution and lower kV. With the development of the many new applications, the limitations of Ga also became apparent: embedding of the beam particle in the sample material, the residual damage to the sample top surface and practical limitations in mill-sizes of the sample. The search for higher milling speeds, results in the application of laser ablation or in the search of a different ion source, such as a plasma based Xe+ beam. The source has a high-enough brightness to still allow a small beam diameter at low current, while at high current the 1 µA limit can easily be exceeded. In this paper the main characteristics of the plasma FIB will be discussed and its own application space will be addressed. Related techniques such as ion beam deposition and etching appear to have different characteristics compared to Ga+.



Figure 7: Site specific cross-sections for scratch testing and adhesion in paint coatings.

# 1.15 Revolutionary Field Emission Thruster Developments for Nano-satellites at TU Dresden and its correlative LMIS-applications at terrestrial Implanters.

Philipp Laufer<sup>1\*</sup>, Daniel Bock<sup>1</sup>, Wolfgang Pilz<sup>2</sup>, Lothar Bischoff<sup>2</sup>, Martin Tajmar<sup>1</sup>

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Monday 18:10 - 18:25 (+3)

Propulsion systems for Nano-satellites are of strong interest for educational purposes as well as research and industrial space missions which can be accomplished by highly miniaturized field emission electric propulsion thrusters. Since the momentum of the emitted ions in the  $\mu$ A-range generates a  $\mu$ N-thrust, an electron source is needed to prevent a spacecraft charging. This paper will present an overview of the NanoFEEP-system as well as the further development towards terrestrial applications of the high-current liquid metal alloy ion source (LMAIS) for terrestrial needs at single-end ion beam systems like an ion implanters. The developed ion beam injector module with different types of LMIS and different metals (Ga, GaBi and In) for continuous operation is presented and characterized. With the suited ion optics (Einzel-lens), the LMIS module provides a nearly parallel ion beam with a diameter of a few mm and with ion currents in the  $\mu$ A-range and high polyatomic ion currents.

# 2 Tuesday

#### 2.1 Thermal Effects from Light Ion Beams in Thin Films

Deying Xia<sup>1</sup>, Brett Lewis<sup>1</sup>, John Notte<sup>1\*</sup>

1. Carl Zeiss Microscopy \* john.notte.65@gmail.com

Tuesday 08:30 - 08:55 (+3)

Light ion beams such as helium and lithium offer unique advantages compared to their heavier cousins, gallium and xenon. One of the fundamental differences arises from the stopping power which is dominated by electron interactions for light ions with energies above 5 keV. For thin films the advantages are more significant because light ions will often pass right through the material before nuclear stopping power can begin to dominate, or implantation effects can arise. The thermal analysis is correspondingly different since the distribution of heat is quite different for light versus heavy ions. In this presentation, the thermal boundary value problem is analyzed for geometries that are reasonable approximations to practical situations. The results suggest temperature effects are quite manageable in most geometries and most materials. Strategies for thermal management will be provided.



Figure 1: SRIM simulation of typical beam interaction for light ions (He) versus heavy ions (Ga) for an unsupported polymer film of 200 nm thickness. Field of view is 1 µm.

# 2.2 FIB milling strategies for the mitigation of beam-induced artifacts in polymer thin films: He vs. Ga

Frances Allen<sup>1\*</sup>

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Tuesday 09:00 - 09:15 (+3)

Focused ion beam (FIB) milling using the gallium liquid metal ion source is a well-established technique for siteselective micromachining. Key application areas include cross-sectional analysis, circuit edit and device prototyping. In certain cases, however, the implantation of gallium ions into the specimen and associated damage pose serious limitations. Samples of low thermal conductivity are also particularly susceptible to localized beam-heating effects. A constrained geometry such as a thin film can exacerbate the situation. I will present recent work investigating focused gallium and helium ion milling of polymer thin films using a Zeiss ORION NanoFab (the helium ion beam is generated by the specialized gas field-ionization source). The milled specimens are analyzed at high resolution by Transmission Electron Microscopy, including elemental mapping. The advantages of helium ion milling for the mitigation of beam-induced artifacts will be discussed.

#### 2.3 Metallography of Thin Films with Focused Ion Beams

Serguei Chiriaev<sup>1\*</sup>, Vadzim Adashkevich<sup>1</sup>, Jacek Fiutowski<sup>1</sup>, Elzbieta Sobolewska<sup>1</sup>, Jacques Chevallier<sup>2</sup>, Horst-Günter Rubahn<sup>1</sup>

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Tuesday 09:20 - 09:35 (+3)

In the presented work we explore a capability of focused ion beams (FIB) for the metallography studies, which is based on the differences in FIB sputtering rate at material and structural inhomogeneities. We focus on the visualisation of the thin film grain structure by FIB sputtering in the case where the rate of material removal is controlled by ion-channelling effects. We show that widely used assessments of the inner film structure with atomic force microscopy (AFM) is often ludicrous, simply because of AFM images do not reflect the true grain structure. On the contrary, FIB-etching combined with HIM or SEM has appeared to be a very convenient tool for a comparative express analysis of thin films in the cross-beam instruments. Reflecting local grain arrangements this technique is complementary to the "averaging" metallography performed with X-ray diffraction. The etched patterns can be further imaged with AFM to obtain maps of the surface etching rate for statistical analysis.

#### 2.4 Compositional analysis and in-situ experiments in the HIM

Nico Klingner<sup>1\*</sup>, René Heller<sup>1</sup>, Gregor Hlawacek<sup>1</sup>, Johannes von Borany<sup>1</sup>, Eduardo Serralta<sup>1</sup>, Stefan Facsko<sup>1</sup>

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Tuesday 09:40 - 09:55 (+3)

The HIM is well known for its imaging with spot sizes below 0.5 nm, its nano-fabrication capabilities, the small energy spread of less than 1 eV and the extremely high brightness. However, it still suffers from the lack of instruments for *in-situ* studies as well as capabilities for a well integrated material analysis.

In the first part a plug and socket system for sample holders will be shown with up to six freely customizable high-voltage electrical connections Additionally time-of-flight spectrometry has been implemented for compositional analysis [1]. New results, drawbacks and derive conclusions for the practical use of time-of-flight SIMS will be presented [2]. Our setup delivers a mass resolution delta m < 0.3 u (for m/q < 80 u) and a lateral resolution of 8 nm.

[1] N. Klingner, R. Heller, G. Hlawacek, J. von Borany, J.A. Notte, J. Huang, S. Facsko. Ultramicroscopy 162 (2016), pp 91-97



Figure 2: (a) SIMS+ image of a rock salt crystal on SiO2. (b) SIMS+ image of the Ne ion beam milled letters HZDR. The contrast arises as the NaCl has been partially removed by the Ne milling.

[2] N. Klingner, R. Heller, G. Hlawacek, S. Facsko, J. von Borany (2018), submitted

# 2.5 SIMS performed on the Helium Ion Microscope: a unique tool for highest spatial resolution imaging and correlative microscopy

Tom Wirtz<sup>1\*</sup>, Jean-Nicolas Audinot<sup>1</sup>

1. Advanced Instrumentation for Ion Nano-Analytics (AINA), MRT Department, Luxembourg Institute of Science and Technology (LIST)

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Tuesday 10:00 - 10:15 (+3)

In 2015, we first presented a SIMS system which we specifically developed for the Zeiss ORION NanoFab HIM. This SIMS system is based on (i) specifically designed secondary ion extraction optics, (ii) a compact floating double focusing magnetic sector mass spectrometer allowing operation in the DC mode at full transmission (and hence avoiding performance degrading duty cycles like in TOF systems) and (iii) a specific detection system allowing the detection of several masses in parallel. We have demonstrated that our instrument is capable of producing (i) mass spectra with high mass resolution, (ii) very local depth profiles and (iii) elemental SIMS maps with lateral resolutions down to 12 nm.Here, we will review the instrument performance and present a number of examples taken from various fields of mate-



Figure 3: Analysis of Ti nanoparticles deposited on InP substrate. a) SE image (20 keV He beam, 1024x1024).
b) and c) SIMS images of Na (blue), Ti (green) and In (red) (20 keV Ne beam, 2 pA, 2 ms/pixel, 512x512).
d) Combined SIMS and SE imaging according to the methodology presented in [6]. Scale bar 500 nm.

rials science and life science to show the powerful analytical capabilities and correlative microscopy possibilities enabled by the integrated HIM-SIMS instrument.

#### 2.6 Transmission He Ion Microscopy

Karen L. Kavanagh<sup>1\*</sup>

1. Dept of Physics, Simon Fraser University, Burnaby, BC, Canada \*kavanagh@sfu.ca

Tuesday 10:50 - 11:15 (+3)

A camera for imaging transmitted ions has been installed 20 cm below the sample in a He ion microscope (HIM, Zeiss Nanofab). [1] This talk will describe the properties of the camera, measurements of transmission scattering patterns, and the detection of planar channeling, and beam steering, for crystalline Si (001) nanomembranes (50 nm). [2] Experimental results were compared to simulations using Large-scale Atomic/Molecular Massively Parallel Simulator which supported our understanding. Compared to TEM, THIM at 35 keV requires thinner samples if significant energy loss is to be avoided. The potential for diffraction contrast will be discussed.

[1] K. L. Kavanagh, C. Herrmann, and J. A. Notte, JVST B 35, 06G902 (2017).

[2] Wang, Huang, Herrmann, Scott, Schiettekatte, Kavanagh, submitted to JVSTB.



Figure 4: Example of channeling through a 25 nm Si nano-membrane. HIM secondary electron image with transmision images from areas indicated. Grey regions better aligned, more channeling. White areas higher SE intensity less channeling.

#### 2.7 Towards observing diffraction in a HIM

Christoph Herrmann<sup>1\*</sup>, Karen L. Kavanagh<sup>1</sup>

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Tuesday 11:20 - 11:35 (+3)

A HIM (Zeiss Orion Nanofab) was modified to observe transmitted He+ ions by adding a camera at the bottom of the chamber. This camera consists of a commercially available square array of Si p-i-n diodes with 256x256 pixels each 55x55  $\mu$ m<sup>2</sup> square. The distance between the camera and the sample is 20 cm, and hence first order diffraction spots are expected to be found at normal operating voltages (25-35 kV) between 35-45 µm apart, which is below the size of one pixel. Yet higher order diffraction spots should be observable, as their spacing increases above the pixel size (e.g. 70-85 µm for n=2). We will show efforts obtaining these pattern from different thin films, e.g. silicon, graphene, gold, and black phosphorus.



Figure 5: Transmission HIM images taken (left) without a sample and the beam focussed on the camera resulting in just one pixel registering counts, and (right) with a thin Au sample in place showing strong scattering. Dark areas with no counts are due to dead pixels.

# 2.8 Multi-Modal Characterization with Secondary Ion Mass Spectrometry on ZEISS ORION NanoFab

Jennifer Braggin<sup>1\*</sup>, Fouzia Khanom<sup>1</sup>

Carl Zeiss Microscopy, LLC
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Tuesday 11:40 - 11:55 (+3)

Understanding the structure, processing, properties and performance of materials requires many characterization techniques. Several analytical capabilities have been paired with microscopy techniques to best understand materials at the nanoscale. One particular technique, secondary ion mass spectrometry (SIMS), is particularly interesting when combined with microscopy techniques. SIMS is a powerful surface analysis technique that provides trace element identification, isotope differentiation, shallow depth profiling and high sensitivity. When combined with high resolution imaging, researchers can better understand their materials. To enhance characterization efforts, a SIMS detector has been developed for ZEISS ORION NanoFab. In this presentation we will show the combination of high resolution secondary electron imaging and SIMS elemental mapping, including standard measurement samples and applications examples from various fields.



Figure 6: ZEISS ORION NanoFab SIMS generated elemental mapping distribution of Al (a) and Cu (b) on CuAl alloy sample. Image (c) represents a composite image. Field of view is 225µm.

# 2.9 Characterising Carbon-based materials in the HIM: probing the invisible with secondary electron spectroscopy

Kerry Abrams<sup>1\*</sup>, Robert Masters<sup>1</sup>, Nicola Stehling<sup>1</sup>, Isabella Douterello<sup>1</sup>, Maurizio Dapor<sup>2</sup>, Hongzhou Zhang<sup>3</sup>, Cornelia Rodenburg<sup>1</sup>

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Tuesday 12:00 - 12:15 (+3)

The HIM has already shown strong potential in the characterisation of organic or biological materials. However, damage from He ion implantation is an issue and the existing tools for characterising material properties in the HIM (based upon SIMS or backscattered ions) are ill-suited to these carbon-based materials. Here, we use SE Spectroscopy in a Low voltage SEM to characterise several organic materials and biofilms to provide a baseline for SE Spectroscopy in HIM. We will then compare and demonstrate how energy spectroscopy of secondary electrons (SEs) in the HIM can offer novel insights in to carbon-based materials. Additionally, by combining experimental SE spectra with state-of-the-art Monte Carlo modelling techniques, we offer an insight in to the electronic and material properties that can be probed with SE spectroscopy.

# 2.10 Understanding Focused Ion Beam Sputtering and Gas-Assisted Etching via the EnvizION Monte Carlo Simulation

Philip Rack<sup>1,2+</sup>, Kyle Mahady<sup>3\*</sup>, Shida Tan<sup>4</sup>, Yuval Greenzweig<sup>5</sup>, Amir Raveh<sup>5</sup>

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Tuesday 14:00 - 14:25 (+3)

We overview the attributes of our EnvizION Monte Carlo Simulation. At its core, EnvizION consists of ion-solid interactions based on SRIM/TRIM, but has a dynamic voxelized substrate matrix to simulate focused ion beam processing. Recently we have added a secondary electron routine to emulate imaging and end-point detection during nanoscale processing. Additionally, we have added a routine to model adsorbed precursor gas, and ion- and electron-induced chemical reactions to emulate gas-assisted focused ion beam etching. In this talk, we describe EnvizION simulations of FIB etching of SiO2 using a XeF2 precursor. We first compare pure SiO2 sputtering results and then study the effect of gasassisted etching on the resolution of etched nanoscale vias, and the influence of ion species such as Ne+ and Ga+, to characterize the underlying limitations on etching resolution. Simulations are compared against experimental results, for validation and to understand experimentally observed features.

#### 2.11 Helium Ion Based Lithography of Advanced Resists

Mengjun Li<sup>1</sup>, Viacheslav Manichev<sup>1</sup>, Fangzhou Yu<sup>2</sup>, Danielle Hutchison<sup>3</sup>, May Nyman<sup>3</sup>, Torgny Gustafsson<sup>4</sup>, Leonard C. Feldman<sup>4\*</sup>, Eric L. Garfunkel<sup>5</sup>

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Tuesday 14:30 - 14:45 (+3)

Extreme ultraviolet (EUV) lithography is expected to replace current photolithographic methods, providing improved resolution due to the smaller wavelength. Tin is a particularly strong absorber for EUV photons (~92 eV). One of the candidate organo-tin compounds,  $\beta$ -NaSn13, has been studied using helium ion beam lithography (HIBL). High aspect ratio (15:1) and dense line patterns (20 half pitch) have been achieved with no defects. Studies on various substrates indicate that the high Z substrates can help improve the pattern performance at low doses. We compare these results to the early simulations of Ishitani and co-workers (1) to address a number of fundamental questions concerning the ultimate imaging and exposure capabilities of the HIM. The work also demonstrates the use of a HIM as an effective tool for exploring the ultimate capabilities of new candidate resists.

Inal *et al*, J. of Elec. Mic. 56, 163 (2007);
 Ohya *et al.*, NIMB, 267, 584 (2009).

# 2.12 Reduced proximity and improved depth of field by using resist-based lithography with helium ions

Ranveig Flatabø<sup>1\*</sup>, Akshay Agarwal<sup>2</sup>, Richard Hobbs<sup>3</sup>, Martin M. Greve<sup>4</sup>, Bodil Holst<sup>4</sup>, Karl K. Berggren<sup>5</sup>

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Tuesday 14:50 - 15:05 (+3)

Helium ion beam lithography (HIL) offers reduced proximity effects, higher resist sensitivity and potentially higher resolution compared to an electron beam of similar energy. Furthermore, the small angular spread of the helium ion beam gives rise to a large depth of field. This should enable patterning on tilted and curved surfaces without the need of any adjustments (e.g. laser-auto focus). So far, most work on HIL has been focused on reaching single-digit nanometer resolution, and has thus been concentrated on exposures over small areas. Here we explore two new areas of application. Firstly, we investigate HIL's capabilities in fabricating precise highdensity gratings on large planar surfaces (100  $\mu$ m x 100  $\mu$ m, with pitch down to 35 nm). Secondly, we exploit the large depth of field by making the first HIL patterns on tilted surfaces (sample stage tilted 45 degrees). We demonstrate a depth of field greater than 100  $\mu$ m for a resolution of about 20 nm.

### 2.13 Advanced FIB nanopatterning employing a high precision sample stage

Sven Bauerdick<br/>1\*, Paul Mazarov<sup>1</sup>, Achim Nadzeyka<sup>1</sup>, Michael Kahl<sup>1</sup>

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Tuesday 15:10 - 15:25 (+3)

Focused ion beam systems and combined FIB-SEM microscopes are used for an increasing number of applications in nanopatterning and rapid prototyping. The nanofabrication requirements for FIB instrumentation are more demanding in terms of stability and accuracy, which applies in particular also to the sample positioning. With a stage technology based on conventional motors and piezo actuators in combination with laser interferometer based position measurements, a mechanical accuracy on the nm level is possible with a large travel range like 100 mm. We investigated, optimized and tested milling approaches for pattern (write field) stitching as well as for truly continuous patterning based on precise stage movement while milling or exposing with the ion beam. Here we report on the capabilities and accuracy as well as benefits for nanopatterning applications.

59

### 2.14 Fabrication of Plasmonic Nanostructures by He+ and Ga+ Milling

Michael Westphal<sup>1\*</sup>, Sven Stephan<sup>2</sup>, Vladimir Smirnov<sup>2</sup>, Daniel Emmrich<sup>1</sup>, Henning Vieker<sup>1</sup>, Heiko Kollmann<sup>2</sup>, André Beyer<sup>1</sup>, Martin Silies<sup>2</sup>, Armin Gölzhäuser<sup>1</sup>

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Tuesday 15:30 - 15:45 (+3)

Plasmonic nanostructures are essential for controlling and directing light on the nanoscale. While fabrication techniques like standard electron beam lithography (EBL) methods or focused ion beam (FIB) milling with Ga+ ions are approaching their limit in the 10nm-regime, ion beam milling with He+ ions is capable of milling features below 6 nm [1]. We will show a combined approach using a Ga+ FIB for milling large features and employing the fine resolution of the helium ion microscope (HIM) for milling small features. We will discuss different patterning strategies to optimize the writing speed and minimize substrate swelling. In addition, the problem of quantifying the sizes of milled gaps will be addressed and an automated, reproducible approach for measuring the size of written features will be demonstrated.
[1] H. Kollmann *et al.*, Nano Letters. 14, 4778–4784 (2014).

## 3 Wednesday

### 3.1 A revolutionary quantum computer device fabricated from implanted ordered arrays of single donor atoms in silicon

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Wednesday 08:30 - 08:55 (+3)

We have fabricated nanoscale devices which exploit the quantum degrees of freedom of single 31P atoms in 28Si with nuclear spin coherence times above 30 s. These devices bridge the foundations of modern information technology based on silicon into the future of ultrascaled devices where quantum mechanics offers new functionalities for sensing, information storage, information processing and secure data transmission guaranteed by the laws of Physics. We have developed a deterministic ion implantation technique that employs charge transients induced by shallow ion implantation, within 20 nanometers of the surface, which is compatible with a prototype scanned nanostencil, machined with a Ga focused ion beam and backfilled with Pt, to allow the fabrication of large scale arrays. This presentation reports progress on the application of an upgraded system with sub-20 nm resolution in the near term towards the goal of a 10 qubit device within 5 years.

### 3.2 Binary Collision Computer Simulation of FIB Induced Erosion and Atomic Mixing in Nanostructures

Wolfhard Möller<br/>1\*, Karl-Heinz Heinig<sup>1</sup>, Xiaomo Xu<sup>1</sup>, Thomas Prüfer<sup>1</sup>

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Wednesday 09:00 - 09:15 (+3)

Collisional computer simulation based on the binarycollision approximation (BCA) has been widely applied to describe effects of ion irradiation on flat solid surfaces, such as ion implantation, surface sputtering, ioninduced damage and atomic mixing. Recent extensions allow fully three-dimensional simulations of irradiated nanostructures both in static and dynamic mode, the latter addressing the shaping of nanostructures under high-fluence ion irradiation as well as compositional modifications.

The contribution will briefly describe the TRI3DYN code [1], and present results on sputter erosion by He and Ne FIB irradiation resulting in hole formation at flat surfaces and shaping of nanostructures such as nanospheres and nanopillars. Further, the atomic mixing in a Si/SiO2/Si trilayer system will be addressed for



Figure 1: Atomic mixing and surface erosion in a Si (20 nm) / SiO2 (7 nm) / Si layer stack after irradiation with 30.000 Ne ions at an energy of 25 keV, a Gaussian beam profile of 5 nm FWHM, and a scan area of 3x3 nm<sup>2</sup>, as obtained from TRI3DYN 3D dynamic computer simulation. The graph shows the local composition as quantified by the color code, averaged over a central slice of 1.33 nm thickness.

both a semi-infinite flat structure and a stacked nanopillar.

[1] W. Möller, Nucl. Instrum. Meth. Phys. Res. B322(2014)23

### 3.3 Site-controlled Si Nanodot Formation for a RT-SET via Ion Beam Mixing and Phase Separation

Xiaomo Xu<sup>1\*</sup>, Thomas Prüfer<sup>1</sup>, Daniel Wolf<sup>1</sup>, René Hübner<sup>1</sup>, Lothar Bischoff<sup>1</sup>, Hans-Jürgen Engelmann<sup>1</sup>, Ahmed Gharbi<sup>2</sup>, Karl-Heinz Heinig<sup>1</sup>, Gregor Hlawacek<sup>1</sup>, Johannes von Borany<sup>1</sup>

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Wednesday 09:20 - 09:35 (+3)

CMOS-compatible formation of Si nanodots (NDs) as Coulomb islands is a prerequisite for an RT Single Electron Transistor operation. In this work, Si NDs are formed via ion beam mixing and thermally stimulated phase separation. Broad-beam Si+ and Ne+ beams followed by a rapid thermal annealing treatment were utilized to create a layer of NDs and visualized by Energy-Filtered Transmission Electron Microscopy (EFTEM). The conditions for ND formation are optimized based on an extensive survey of the parameter space. The work is guided by TRIDYN simulations during the ion beam mixing and 3D Kinetic Monte-Carlo simulation for the phase separation during the thermal treatment. To tailor towards a single Si ND, the focused Ne+ beam from



Figure 2: (a) EFTEM shows 17eV Si plasmon-loss signal, compared to (b) modulated dot signal the formation of single Si ND via HIM Ne+ implantation is confirmed (c)

the Helium Ion Microscope (HIM) is utilized to create patterns of NDs in planar layer stacks. The formation of site-controlled single NDs with a diameter of 2.2 nm is confirmed by comparing the EFTEM Si plasmon-loss intensity with simulated intensity.

### 3.4 Latest development for failure analysis – When ions meet chemistry

Gregory Goupil<sup>1</sup>, Tomas Hrncir<sup>2</sup>, Sharang Sharang<sup>2</sup>, Pascal Gounet<sup>3</sup>, Anne Delobbe<sup>1\*</sup>

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Wednesday 09:40 - 09:55 (+3)

Many trials have been attempted to use a Focused Ion Beam (FIB) to remove homogeneously different metal/ insulator layers. But ion beam etching alone is not able to achieve planar surface on interconnect technologies. The milling rate of different materials is too unequal and their architecture is too tricky to reach deep layers with the minimum roughness on the sample. To overcome these artifacts, a solution is to control FIB milling rates of all the different as-constitutive element of the SC's surface by adding a specific gas near the area of interest during operation.



Figure 3: Delayering attempted without gas (left) and same process using new chemistry (right)

### 3.5 Cs ion coldbeam suitability for circuit edit and other nanomachining applications

Yuval Greenzweig<sup>1\*</sup>, Roy Hallstein<sup>2</sup>, Yariv Drezner<sup>1</sup>, Minh Ly<sup>2</sup>, Richard Livengood<sup>2</sup>, Shida Tan<sup>2</sup>, Amir Raveh<sup>1</sup>

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Wednesday 10:00 - 10:15 (+3)

Charged particle beams for semi-conductor applications have been challenged by device density doubling every two years during the last 2-3 decades. Circuit Edit, TEM lamella and Atom Probe Tomography sample preparation, have been long enabled by Ga LMIS technology. Recently, however, Circuit Edit has been noticeably losing ground on currently developed microprocessors. We have been seeking new focused ion beam candidates among several emerging ion beam technologies. We report herein our testing of the Low Temperature Ion Source (LoTIS) at zeroK Nanotech – a Cs cold ion source attached to an old FIB platform. We report on image resolution, beam profile, Cs contamination levels in common substrates, lack of invasiveness of Cs to 14nm Intel transistors, minimum micro-trench sizes, SE vield from Al and Si substrates, material properties of FIB deposited dielectric and metal. We comment on the suitability of Cs for Circuit Edit and other applications.

### 3.6 Nanoscale defect engineering in ferroelectric thin films by focused ion beam microscopy

Leo McGilly<sup>1\*</sup>, Petr Yudin<sup>2</sup>, Ludwig Feigl<sup>3</sup>, Cosmin Sandu<sup>4</sup>, Dragan Damjanovic<sup>4</sup>, Alexander Tagantsev<sup>4</sup>, Nava Setter<sup>4</sup>

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Wednesday 10:50 - 11:15 (+3)

For materials where crystallinity is essential, such as ferroelectrics, defect type and concentration can vastly influence properties and are often used to optimize device performance. This work shows a method to effectively control the density and position, on the nanoscale, of defect regions in thin films of Pb(Zr,Ti)O3 via lowdose Ga+ focused ion beam microscopy (FIB) which allows for engineering of the domain nucleation sites, coercive fields and rate of switching with high spatial precision [1].Furthermore, armed with this knowledge, domain walls can be pinned at defect regions indefinitely for low applied voltages or confined to predetermined propagation channels like a waveguide [2]. These results can be used as a tool to control properties useful for fundamental ferroelectrics research as well as leading a way towards domain wall nanoelectronics.

L. J. McGilly *et al.* Adv. Func. Mater. 27, 15 (2017)
L. J. McGilly *et al.* Appl. Phys. Lett. 111, 022901 (2017)

### 3.7 Modification of MoS2 with Helium Ion Beam: Fabrication of Gate-Tunable Memristor

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Wednesday 11:40 - 11:55 (+3)

A focused helium ion beam can modify the electrical and optical characteristics of MoS2 through the introduction of defects as well as tune the crystal structure and stoichiometry, due to the preferential removal of sulfur, with unprecedented spatial resolution. Here we fabricate FET devices which behave as memristors, in which sulfur vacancies, introduced with well-defined geometry, act as mobile dopants and migrate under applied field, modulating the resistance. These devices can be reliably switched between high and low resistance states. Furthermore, owing to the 2D nature of these devices, these states can be tuned by the application of a back gate which is not possible for higher dimensional memristive systems. The mechanism of the resistive switching behavior is explored through further analysis techniques used to probe the irradiated region, including Raman mapping, atomic force microscopy, and transmission electron microscopy.



Figure 4: Left: Schematic of fabricated device. Right: Typical IV sweep showing switching from high to low resistance states.

### 3.8 Application of Gas Field Ion Source to Patterning Nanoscale Magnetic Structures

Rantej Bali<sup>1\*</sup>, Alexander Schmeink<sup>1</sup>, Gregor Hlawacek<sup>1</sup>, Jürgen Lindner<sup>1</sup>, Jürgen Faßbender<sup>1</sup>

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Wednesday 12:00 - 12:15 (+3)

Magnetic nanostructures are necessary components in a variety of sensors and devices, and in prototypes of spin-transport and spin-wave devices. In this contribution, we describe the application of the gas field ion source (GFIS), to the nanoscale modification of magnetic properties in alloy thin films. Materials in which the saturation magnetization (Ms) can be drastically enhanced via small changes to the atomic arrangement are ideal for the GFIS approach. In B2 alloys such as Fe60Al40, Fe50Rh50 and Fe60V40, light noble gas ionirradiation leads to the formation of anti-site defects, which increases the Fe – Fe nearest neighbor interactions and generates an associated increase of the Ms. These B2 alloys can be used as non-ferromagnetic templates, on to which the highly focused ion-beam acts as a magnetic writing stylus. We examine the conditions necessary and experimentally achievable limits for producing magnetic nanostructures using GFIS.

### 3.9 Writing Magnetic Domains with a Helium Ion Microscope

Daniel Emmrich<sup>1\*</sup>, Alexander Gaul<sup>2</sup>, Dennis Holzinger<sup>2</sup>, Farzaneh Karimian<sup>3</sup>, Matic Klug<sup>3</sup>, Jeffrey McCord<sup>3</sup>, André Beyer<sup>1</sup>, Arno Ehresmann<sup>2</sup>, Armin Gölzhäuser<sup>1</sup>

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Wednesday 12:20 - 12:35 (+3)

Two-dimensional ion bombardment induced magnetic patterning (IBMP) [1] is demonstrated with a helium ion microscope to create magnetic domains in an exchange biased thin film system. Such a system consists of a thin ferromagnetic layer coupled to an underlying antiferromagnet. Low dose helium ion irradiation at an energy of 15 keV in an external magnetic field leads to a new remanent magnetization direction, determined by the external magnetic field. By subsequently patterning the sample in differently orientated external magnetic fields, complex magnetic domain patterns such as chiral structures can be written. Based on magnetic force microscopy and optical Kerr microscopy, we will discuss the achievable resolution as well as the shapes of different artificial magnetic domains.

 A. Gaul *et al.*, Journal of Applied Physics 120, 33902 (2016).

### **4** Poster Presentations

### 4.1 Nanoscale Engineering of Metal Nanoparticles in Dielectric Films by Energetic Ions for Some Novel Applications

Santanu Ghosh<sup>1\*</sup>, Debalaya Sarker<sup>1</sup>, Saswata Bhattacharya<sup>1</sup>, Pankaj Srivastava<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

The interaction between energetic ions and a solid has been recognized as one of the important physical process to generate and engineer nanostructure in materials. The energy deposited to the electron-ic subsystem of a material known as elec-tronic energy loss gets coupled with the lattice via a complex thermodynamical process within the framework of 'Thermal spike mechanism', is one of the important process in the evolution



Figure 1: Shape engineering of FeCo nanoparticles embedded in SiO2 matrix and Colossal rise in tunneling current

and modification of nanostructures. In this presentation after giving a brief description of this mechanism, the fol-lowing topics will be discussed: (i) Role of thermal spike in inelastic sputtering process, (iii) shape modification of metallic nano-partiles (NPs) embedded in insulating media. The influence such modification of the nanostructures on electronic and and magnetic properties of these films will be discussed in detail.

### 4.2 FIB Nanopatterning of Metal Films on PMMA Substrates: Non-Sputtering Mode

Luciana Tavares<sup>1\*</sup>, Vadzim Adashkevich<sup>1</sup>, Serguei Chiriaev<sup>1</sup>, Horst-Günter Rubahn<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

Nanofabrication with focused ion beams (FIB) is a widely used technology for tailoring of e.g. optical and plasmonic elements [1]. The technology is essentially based on material removal by ion sputtering (ion milling) or ion-beam assisted chemical etching [1]. In addition, FIBs can decompose polymer materials, which results in material shrinkage in the irradiated areas [2]. In this work, we demonstrate that this mechanism can be used for nanopatterning thin metal films deposited on PMMA resist spin coated on silicon substrates. For this purpose, the samples were irradiated with He+ FIB in a Zeiss Nanofab HIM to form patterns. We show that this technique is capable of forming continuous planar metal patterns with a dynamic depth range of 100 nm and low film damage. Benefits, limitation and possible applications of this technique are outlined.

[1] G. Hlawacek et. al., Helium Ion Microscopy, 2016,



Figure 2: Atomic force microscope image of a microstructure fabricated in Au-Pd/PMMA system with a Helium FIB in a Zeiss Nanofab HIM.

Switzerland. [2] L. Sawyer et. al., Polymer Microscopy, 2008, Springer New York.

### 4.3 Helium Ion and Optical Microscopy of Spider Silk

Irina Iachina<sup>1</sup>, Jacek Fiutowski<sup>2\*</sup>, Serguei Chiriaev<sup>2</sup>, Horst-Günter Rubahn<sup>2</sup>, Jonathan Brewer<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

Spider silk has many properties that may be of industrial use as the tensile strength of spider silk is comparable to alloy steel and Kevlar, but has the advantage of being spun at room temperature. The aim of this project is to characterize Major Ampullate silk (MAS) and Minor Ampullate silk (MiS) spider silk fibers from the orb web weaving spider Nephila Madagascariensis by determining the biochemical, nano- and microscopic structures within the silk and couple these to the macroscopic properties. Using Coherent Anti-Stokes Raman Scattering (CARS) and fluorescence microscopy the lipids and proteins of the fiber were analyzed and visualized revealing the overall structure. He Ion Microscopy was applied to etch away the most outer lipid layers and to visualize the inner protein arrangements with no sample preparation. It was found for the first time without cryofreezing that the protein core consists of fibrils arranged parallel to each other and to the long axis of the fiber.

### 4.4 On the preparation of dechlorinating bacteria for scanning electron microscopy

Nedal Said<sup>1\*</sup>, Matthias Schmidt<sup>1</sup>, Ivonne Nijenhuis<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

Chlorinated organic compounds are widespread contaminants in groundwater and soil. The bacterial strains Sulfurospirillum multivorans, Hyphomicrobium GJ, Dehalococcoides mccartyi BTF08 and Desulfitobacterium PCE1 are capable of organic dechlorination and can degrade such contaminants. Despite sharing dechlorinating properties their cell membranes differ significantly: Desulfitobacterium PCE1 for instance is gram-positive and Dehalococcoides mccartyi BTF08 exhibits a delicate protein-rich cell membrane. High resolution and surface sensitivity make HIM an ideal tool to study morphology and membranes of these organisms. However, this is only possible if the cells are well prepared. For that we developed protocols for the preparation of these bacterial strains. The protocols involve aldehyde-fixation, filtration, post-fixation and dehydration. A novel H2O2 post-fixation technique as well as ionic-liquid preparation (Golding *et al.*, Sci. Rep. 2016) are presented.



Figure 3: Helium-ion micrograph of Desulfitobacterium PCE1

### 4.5 Scanning Transmission Helium Ion Microscopy on 1nm Thick Carbon Nanomembranes

Daniel Emmrich<sup>1</sup>, Annalena Wolff<sup>2</sup>, André Beyer<sup>1\*</sup>, Armin Gölzhäuser<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

The Helium Ion Microscope (HIM) offers different detection schemes. The most important one is secondary electron (SE) imaging which enables an edge resolution of 0.3 nm [1]. Recording the transmission ion signal is less common, but it attracts growing attention. Here we show a dark field transmission ion imaging study on 1 nm thick Carbon Nanomembranes (CNMs) by using a SE conversion plate. CNMs are made of self-assembled monolayers that are cross-linked by low energy electrons resulting in 1 nm thick carbon membranes with tunable conductivity [2]. By imaging the same sample site with different acceptance angles, we are able to compare the measured dark field transmission ion signal with simulated scattering angle distributions from SRIM and we will discuss the applicability of those simulations on ultimate thin membranes.

[1] G. Hlawacek, A. Gölzhäuser (Eds.), Springer Intl.,

Switzerland 2016.

[2] A. Turchanin, A. Gölzhäuser, Adv. Mater 2016, 28, 6075.

### 4.6 Molecular dynamics simulations of He ion channeling in Gold nanoclusters

Sadegh Ghaderzadeh<sup>1\*</sup>, Gregor Hlawacek<sup>1</sup>, Arkady Krasheninnikov<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

Ion channeling is a well-known effect in ion irradiation processes, which is a result of ion moving between the rows of atoms. It drastically affects the ion distribution, ion energy-loss and consequently the damage production in the target. Therefore one could derive the ion-channeling pattern out of the energy-loss behavior of ion-target interaction.

Ion channeling effect is studied for a few pure element crystals and also for some compounds in a systematic way [1]. In this work, we focus on nano-structures which are of major importance, due to their high surface-tovolume ratio. Our results, for different gold cluster sizes, show that ion-channeling occurs not only in the principal low-index, but also in other directions in between. The strengths of different channels are specified, and their correlations with sputtering-yield and damage production is discussed. [1] Nordlund, K., and G. Hobler. 'Dependence of ion channeling on relative atomic number in compounds.' NIMB 2017.

### 4.7 Fundamental focus beam-solid interactions and applications for rapid prototyping

Michael Stanford<sup>1</sup>, Kyle Mahady<sup>1\*</sup>, Pushpa Pudasaini<sup>1</sup>, Jason Fowlkes<sup>2</sup>, Philip Rack<sup>2</sup>

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Monday 18:30 - 19:55 (Poster session)

Fundamental focused beam-solid interactions will be discussed to evaluate the potential for focused ion and photon beams as a rapid prototyping tools. Combining the benefits of multiple beams (i.e. ion and photon) for processing techniques, can enable expanded nanopatterning capabilities. Several novel methods are discussed for direct-write nanoscale processing, such as laser assisted focused ion beam induced deposition and laser assisted focused ion beam induced etching. Studies will also be discussed in which focused beams were used for rapid prototyping materials. First, focused He+ irradiation was used as an athermal activation for amorphous IGZO. Focused ion beam induced etching was utilized as a top-down method to create 2D transition metal dichalcogenide nanoribbons with < 10 nm resolution. A focused He+ beam was also used to induce defects within single layer and multilayer WSe2, which results in tunable transport properties.

# 4.8 Helium Ion Microscopy for the imaging of biological samples

Till Leißner<sup>1</sup>, Luciana Tavares<sup>1\*</sup>,

Linnea Rebecka Gustafsson<sup>2</sup>, Horst-Günter Rubahn<sup>1</sup>, Jakob Kjelstrup-Hansen<sup>1</sup>, Niels Marcussen<sup>2</sup>

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Monday 18:30 - 19:55 (Poster session)

Imaging of biological samples benefits from several unique capabilities of the Helium ion microscope, e.g. charge compensation, large depth of focus, and sequential milling and imaging obtained by combining the different ion sources for investigation of different tissue layers. In this work we investigate the influence of the sample preparation procedures of biological samples on the resulting imaging quality. We tested various fixatives and embedding procedures and investigated the impact of critical point drying. We find that using glutaraldehyde as a fixative combined with critical point drying of the sample preserve the tissue of shrinking and allows imaging the sample with low-current Helium beams at high resolution.

### 4.9 HIM-SIMS: a powerful tool for analysis of energy and micro-electronic materials

Jean-Nicolas Audinot<sup>1\*</sup>, Florian Vollnhals<sup>1</sup>, Paul Gratia<sup>1</sup>, Santhana Eswara<sup>1</sup>, Patrick Philipp<sup>1</sup>, Tom Wirtz<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

Owing in particular to its excellent sensitivity, Secondary Ion Mass Spectrometry (SIMS) constitutes an extremely powerful technique of surfaces analysis. Therefore, we have specifically developed a high-performance SIMS system for the Zeiss NanoFab Helium Ion Microscope, allowing us to get chemical information with both highest sensitivity and highest spatial resolution. Furthermore, we developed specific methodologies based on the HIM-SIMS system opening the way for *in-situ* correlative imaging combining high resolution secondary electron (SE) images with elemental and isotopic ratio maps from SIMS. This approach allows SE images of exactly the same zone analyzed with SIMS to be acquired easily and rapidly, followed by a fusion between the SE and SIMS data sets. Here, we will take advantage of the instrument's powerful analytical capabilities and correl-
ative microscopy possibilities to characterize energy materials (battery materials, solar cells) and micro-electronics devices

## 4.10 Scanning Transmission Ion Detection in the Helium Ion Microscope

Eduardo Serralta<sup>1\*</sup>, Nico Klingner<sup>1</sup>, Sadegh Ghaderzadeh<sup>1</sup>, Gregor Hlawacek<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

The helium ion microscope has already proven its value for high-resolution imaging, composition analysis, nanofabrication, and material modification. However, imaging in transmission mode remains not fully explored. Mass-thickness contrast has been studied using a conversion plate below the specimen and collecting secondary electrons with an ET detector. Changing from bright to dark field regime was demonstrated using an annular microchannel plate and changing the acceptance angle by adjusting the distance between the sensor and the sample. Channeling and diffraction phenomena provide information about the crystal structure and can be recorded by a position-sensitive detector. In this report, we present our approach to explore this imaging mode, the challenges and main figures of merit. Our test setup with a position-sensitive detector will be shown, and simulations of the contrast mechanism will be presented.

## 4.11 A rubidium focused ion beam instrument

Edgar Vredenbregt<sup>1\*</sup>, Gijs ten Haaf<sup>1</sup>, Steinar Wouters<sup>1</sup>, Peter Mutsaers<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

Focused Ion Beams are important tools for the semiconductor industry. Essential applications are editing circuits and repairing masks in the development phase, and failure analysis during wafer processing. As a result of the reduction of feature sizes in semiconductor circuits, FIBs also face higher demands in terms of resolution and reduced damage. Here a FIB instrument that can overcome these limitations is presented. The essential innovation is the use of a new type of ion source based on photo-ionization of a laser-intensified and extremely cold atomic rubidium beam. The performance of the source was characterized by studying deliverable current, brightness and energy spread. The source was then mounted on a commercial FIB system and first ion microscopy and milling experiments were performed.



Figure 4: Rb+ FIB installed at CQT Labs

# 4.12 Quantification of HIM imaging resolution

Kai Arstila<sup>1\*</sup>, Timo Sajavaara<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

An excellent imaging resolution is one of the main advantages in using HIM. However, it is quite common that the resolution has been determined quantitatively only during the tool acceptance tests. In this work we present a set of samples, imaging procedures and software to determine HIM resolution values during the daily imaging work. A single sample stub used in this work includes three different samples: a TEM grid with graphene for determining conventional 25% - 75% intensity level resolution, a sample with 100-nm tin spheres on carbon allowing for beam alignment and stigmator settings, and a sample with 5 - 20 nm gold islands on silicon for fine tuning the image quality and determining imaging resolution in daily work. Open source software has been developed to determine quantitative resolution values from images of graphene and gold samples. This software can be installed in the HIM computer to allow for immediate determination of the resolution during the imaging work.

## 4.13 Interaction of an Energetic Ar Molecular Cluster Beam with Graphene

Songkil Kim<sup>1\*</sup>, Anton Ievlev<sup>1</sup>, Jacek Jakowski<sup>1</sup>, Ivan Vlassiouk<sup>1</sup>, Matthew Burch<sup>1</sup>, Alex Belianinov<sup>1</sup>, Bobby Sumpter<sup>1</sup>, Stephen Jesse<sup>1</sup>, Olga Ovchinnikova<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

Manipulation of low-D nanomaterials provides intriguing opportunities to design new functional materials as well as to develop next-generation device applications. In this study, we investigated the effect of Argon molecular cluster beam irradiation on both defect formation and removal of organic contaminants on graphene. An Argon cluster beam was generated using the Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) combined with Atomic Force Microscopy (AFM). A systematic study has been conducted to provide in-depth understanding about defect formation of graphene by synergistic theoretical and experimental approaches. Raman spectra clearly indicate that suspended graphene is more susceptible to Ar cluster beam irradiation than supported graphene on a SiO2/Si substrate under the same irradiation conditions. The underlying mechanisms for the experimentally observed phenomena are demonstrated by theoretical analysis using the firstprinciples molecular dynamics calculations.

## 4.14 Cleaning of Pt deposits with an in-situ low-energy Ar ion beam

Paul Alkemade<sup>1\*</sup>, Boyd Verdoorn<sup>1</sup>, Hans Mulders<sup>2</sup>

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Monday 18:30 - 19:55 (Poster session)

Electron and Ion Beam Induced Deposition (E/IBID) are techniques for growing 3D objects on substrate surfaces. The movements of the focused electron or ion beam across the surface, covered by adsorbed molecules, determine the shape of the growing E/IBID object. Because of scattering of the primary beam and of emission of secondary electrons every deposit is surrounded by a halo, which could thwart the functioning of the grown object, e.g. causing a short between deposited Pt lines. In this work we use an *in-situ* low-energy Ar ion beam for Beam Induced Polishing and Sputtering (BIPS) of a PtC IBID-deposit on Si. The source of ions is a stream of Ar gas, ionized by the electron beam. The Pt, Ga, C and Si EDX signals are measured as a function of distance to the deposit, both before and after Ar ion beam polish or sputter cleaning. The results show that BIPS can be used for *in-situ* pre- or postprocessing of surfaces and deposits in a FIBSEM instrument.



Figure 5: Illustration of the ionization process and the attraction of positively charged ions towards the surface.[1]

[1] D. van Leuken and J.J.L. Mulders, in preparation.

## 4.15 Nanofabrication using He ions - from 2D materials to nanometer sized gaps

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Monday 18:30 - 19:55 (Poster session)

The Helmholtz-Zentrum Berlin runs the Corelab facility 'Correlative Spectroscopy and Microscopy' which is specialized to the needs of external users. It offers a variety of scanning electron, transmission electron and ion beam microscopes as well as scanning probe and optical microscopes with a multitude of experimental addons. Many of the available techniques can be performed simultaneously, or correlated for the same sample volume under specified conditions. It serves as a highly specialized unit for the characterization and structural modification of complex 3D materials composites with geometric features at the nanoscale. The He and Ne ion microscopy being part of the Corelab and provides the possibility of high resolution imaging of organic and non-conductive samples as well as of sub 10 nm patterning. We will present recent examples of collaborative work including the patterning of 2D materials, high aspect ratio FIB cutting for plasmonics and imaging of polymer nanofibers.

#### 4.16 Magneto-transport measurements in para- and ferromagnetic Fe60Al40 wires

Alexander Schmeink<sup>1\*</sup>, Vico Liersch<sup>1</sup>, Sebastian Wintz<sup>1,2</sup>, Jonathan Ehrler<sup>1</sup>, Roman Böttger<sup>1</sup>, Gregor Hlawacek<sup>1</sup>, Kay Potzger<sup>1</sup>, Jürgen Lindner<sup>1</sup>, Jürgen Faßbender<sup>1</sup>, Artur Erbe<sup>1</sup>, Rantej Bali<sup>1</sup>

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Monday 18:30 - 19:55 (Poster session)

A number of B2 alloys like Fe60Al40, Fe65V35, and Fe50Rh50 can be gradually transformed from the ordered B2 to the disordered A2 crystal structure by use of ion irradiation. This transition is adjustable via the applied ion fluence and can be reversed through vacuum annealing. The highly focused beam of a gas field ion source (GFIS) can be used to irradiate locally and therefore induce disorder in selected regions of such a material.We use a focused Ne+ ion beam of ~2 nm spot-size to irradiate wires made of Fe60Al40, which is paramagnetic in the B2 and ferromagnetic in the A2 state. By performing *in-situ* resistivity measurements during the irradiation process we show that the B2 -> A2 transition results in a change in conductive properties.



Figure 6: Fe60Al40 wire with gold contact lines, contacted with tungsten probes. The region of Ne+ irradiation is marked.

## 4.17 Correlative In-Situ Analysis of Helium Ion treated Graphene Membranes with AFSEM<sup>™</sup>

Stefan Hummel<sup>1\*</sup>, Pinar Frank<sup>1</sup>, Michael Leitner<sup>1</sup>, Frances Allen<sup>2</sup>, Mehdi Balooch<sup>2</sup>, Christian H. Schwalb<sup>1</sup>, Peter Hosemann<sup>2</sup>

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Monday 18:30 - 19:55 (Poster session)

Tailoring the mechanical and electrical properties of Graphene and other two dimensional (2D) materials is crucial for almost all current and future applications regarding those materials. The most prominent technique for creation of high-resolution patterned structures is Focused Ion beam employing Helium Ions. Although this technique is well established, monitoring the effects of Helium treatment on 2D materials is still difficult, since investigation directly inside the Ion Microscope is, up to now, highly invasive due to the interaction of high energetic particles with the sample. Here, we will show recent results of nano-mechanical and -electrical measurements employing our *in-situ*, non-invasive, correlative AFSEM<sup>TM</sup> technology. Few-layered Graphene membranes have been patterned inside the Orion Nanofab at University of Berkeley and been investigated with AFM and conductive AFM. With our novel AFSEM<sup>TM</sup> scanner, dedicated for the seamless integration into the Zeiss Orion Nanofab, we show, for the first time, correlative AFM-SEM studies on He-FIB treated suspended Graphene membranes and hence, demonstrate the strength of *in-situ* AFM experiments inside Helium Ion Microscopes (HIMs). This paves the way for applying the full strength of AFM inside HIMs by adding the complementary analysis capabilities of real 3D topography with sub-nanometer resolution, electrical, magnetic and thermal characterization of nanostructures, as well as determination of nano-mechanical properties directly inside the HIM.

## 4.18 Nano-patterning of β-Ga<sub>2</sub>O<sub>3</sub> nanostructures with He and Ne focused ion beams

Julien Barrat<sup>1</sup>, Jürgen Albert<sup>1</sup>, Holger Kropf<sup>1</sup>, Klaus Schwarzburg<sup>1</sup>, Zbigniew Galazka<sup>2</sup>, Sebastian Schmitt<sup>1</sup>, Catherine Dubourdieu<sup>1,3\*</sup>

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Monday 18:30 - 19:55 (Poster session)

We report the fabrication of  $\beta$ -Ga2O3 nanostructures with neon and helium focused-ion beam milling. The starting materials were (100)  $\beta$ -Ga2O3 single crystals. Under the prolonged irradiation of helium ions, a blistering effect of the target material occurred. This effect will be discussed as a function of the irradiation dose and of the incident energy. Ne ion beam was used to fabricate sub-micrometer nanostructures. In order to reduce the critical dimensions down to the sub-100 nm range, a patterning strategy was developed by combining subsequently Ne+ and He+ beams. High-aspect ratio nanopillars were obtained, with a radius down to ~65 nm and a reduced taper. The topography of the structures was analyzed *in-situ* and with atomic force microscopy. The compositional and structural changes



Figure 7: This nanopillar was produced by first milling with a Ne+ beam with alternating direction (out-wards/inwards), and then by polishing with a He+ beam directed only inwards.

of the etched surface, studied by Raman spectroscopy, will be discussed.

## 4.19 Atomic force microscopy of HIM milled bevels of thin films and nanostructures

Niklas Valjakka<sup>1</sup>, Spyridon Korkos<sup>1\*</sup>, Kai Arstila<sup>1+</sup>

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Monday 18:30 - 19:55 (Poster session)

Milling in helium ion microscopy (HIM) allows for studying thin films by creating a low angle bevel on the sample surface. This kind of a shallow bevel gives a magnified image of the in-depth structure of a thin film, thus allowing to study even very thin films. However, these bevels are flat, which limits the direct HIM imaging to depend on the material contrast only. Modern atomic for microscopy (AFM) tools include imaging modes, which depend on the e.g. mechanical or electrical properties of the sample surfaces instead of the topography. Actually, often these imaging modes work better if the surface is flat and the topography does not disturb the specific imaging mode. In this work we present the first results of using so called PeakForce tapping mode of an AFM tool to determine the structures of thin film on bevelled samples. Bevels were created by HIM milling and scanned with an AFM and the generated force curves were used to study the thin film structures.

#### PART 4. POSTER PRESENTATIONS

Abrams, Kerry, 50 Adashkevich, Vadzim, 38, 82 Agarwal, Akshay, 56 Albert, Jürgen, 114 Alkemade, Paul, 106 Allen, Frances, 36 Allen, Francis, 112 Arstila, Kai, 102, 116 Audinot, Jean-Nicolas, 16, 42, 96 Böttger, Roman, 110 Bali, Rantej, 76, 110 Balooch, Mehdi, 112 Barrat, Julien, 114 Bauerdick, Sven, 22, 58 Belianinov, Alex, 18, 104 Berggren, Karl K., 26, 56 Beyer, André, 60, 78, 88 Bhattacharya, Saswata, 80 Bischoff, Lothar, 22, 24, 32, 66 Bock, Daniel, 32 Braggin, Jennifer, 48 Brewer, Jonathan, 84 Bruchhaus, Lars, 22 Burch, Matthew, 18, 104

Chevallier, Jacques, 38 Chiriaev, Serguei, 38, 82, 84 Comparat, Daniel, 20 Damjanovic, Dragan, 72 Dapor, Maurizio, 50 Delobbe, Anne, 20, 68 Douterello, Isabella, 50 Drezner, Yariv, 70 Dubourdieu, Catherine, 114 Ehresmann, Arno, 78 Ehrler, Jonathan, 110 Emmrich, Daniel, 60, 78, 88 Engelmann, Hans-Jürgen, 66 Erbe, Artur, 110 Eswara, Santhana, 16, 96 Faßbender, Jürgen, 76, 110 Facsko, Stefan, 40 Feigl, Ludwig, 72 Feldman, Leonard C., 54 Fiutowski, Jacek, 38, 84 Flatabø, Ranveig, 56 Fowlkes, Jason, 92 Frank, Pinar, 112 Gölzhäuser, Armin, 60, 78, 88 Galazka, Zbigniew, 114 Garfunkel, Eric L., 54 Gaul, Alexander, 78 Ghaderzadeh, Sadegh, 90, 98 Gharbi, Ahmed, 66

Ghosh, Santanu, 80 Gierak, Jacques, 24 Gounet, Pascal, 68 Goupil, Gregory, 68 Goval, Vivek, 26 Gratia, Paul, 96 Greenzweig, Yuval, 52, 70 Greve, Martin M., 56 Gustafsson, Torgny, 54 Höflich, Katja, 108 Hübner, René, 66 Hallstein, Roy, 70 Heinig, Karl-Heinz, 64, 66 Heller, René, 40 Herrmann, Christoph, 46 Hlawacek, Gregor, 40, 66, 76, 90, 98, 110 Hobbs, Richard, 56 Holst, Bodil, 56 Holzinger, Dennis, 78 Hosemann, Peter, 112 Houel, Arnaud, 20 Hrncir, Tomas, 68 Hummel, Stefan, 112 Hutchison, Danielle, 54 Iachina, Irina, 84 Ievlev, Anton, 18, 104 Jadwiszczak, Jakub, 74 Jakowski, Jacek, 104 Jamieson, David, 62 Jede, Ralf, 22

Jesse, Stephen, 104 Joachim, Christian, 6 Kahl, Michael, 22, 58 Karimian, Farzaneh, 78 Kavanagh, Karen L., 44, 46 Keane, Darragh, 74 Kemmler, Matthias, 8 Khanom, Fouzia, 48 Kim, Songkil, 18, 104 Kjelstrup-Hansen, Jakob, 94 Kleindiek, Stephan, 8 Klingner, Nico, 12, 40, 98 Klug, Matic, 78 Kollmann, Heiko, 60 Korkos, Spyridon, 116 Krasheninnikov, Arkady, 90 Kropf, Holger, 114 Laufer, Philipp, 32 Leißner, Till, 94 Leitner, Michael, 112 Lewis, Brett, 34 Li, Mengjun, 54 Liersch, Vico, 110 Lin, Jinying, 12 Lindner, Jürgen, 76, 110 Liu, Yongtao, 18 Livengood, Richard, 70 Ly, Minh, 70

Möller, Wolfhard, 64 Maasilta, Ilari, 10

Mahady, Kyle, 52, 92 Manichev, Viacheslav, 54 Marcussen, Niels, 94 Masakazu, Masakazu, 6 Masters, Robert, 50 Mazarov, Paul, 22, 24, 58 McCord, Jeffrey, 78 McGilly, Leo, 72 Mizuta, Hiroshi, 2 Moreno, Jairo, 14 Mulders, Hans, 30, 106 Murray-Bruce, John, 26 Muruganathan, Manoharan, 2 Musat, Niculina, 14 Mutsaers, Peter, 100 Nadzevka, Achim, 58 Naitou, Yuichi, 4 Nakaharai, Shu, 6 Nijenhuis, Ivonne, 86 Notte, John, 34 Nyman, May, 54 Ogawa, Shinichi, 2, 4, 6 Okawa, Yuji, 6 Ovchinnikova, Olga, 18, 104 Peng, Minxu, 26 Peng, Yong, 12 Philipp, Patrick, 96 Pilz, Wolfgang, 22, 24, 32 Potzger, Kay, 110 Prüfer, Thomas, 64, 66

Pudasaini, Pushpa, 92 Rack, Philip, 52, 92 Ramshaw, John, 12 Raveh, Amir, 52, 70 Rebecka Gustafsson, Linnea, 94 Reveillard, Morgan, 20 Richnow, Hans, 14 Rodenburg, Cornelia, 50 Rubahn, Horst-Günter, 38, 82, 84, 94 Rummel, Andreas, 8

Said, Nedal, 14, 86 Sajavaara, Timo, 102 Sandu, Cosmin, 72 Sarker, Debalaya, 80 Schmeink, Alexander, 76, 110 Schmidt, Marek, 2 Schmidt, Matthias, 14, 86 Schmitt, Sebastian, 114 Schock, Klaus, 8 Schwalb, Christian H., 112 Schwarzburg, Klaus, 114 Serralta, Eduardo, 40, 98 Setter, Nava, 72 Sharang, Sharang, 68 Silies, Martin, 60 Smirnov, Vladimir, 60 Smith, Andrew Jonathan, 8 Sobolewska, Elzbieta, 38 Srivastava, Pankaj, 80 Stanford, Michael, 92

Stehling, Nicola, 50 Stephan, Sven, 60 Stryhanyuk, Hryhoriy, 14 Sumpter, Bobby, 18, 104 Tagantsev, Alexander, 72 Tajmar, Martin, 32 Tan, Shida, 52, 70 Tavares, Luciana, 82, 94 ten Haaf, Gijs, 100 Thompson, William, 12 Valjakka, Niklas, 116 Verdoorn, Boyd, 106 Verveniotis, Elisseos, 6 Vieker, Henning, 60 Viteau, Matthieu, 20 Vlassiouk, Ivan, 104 Vollnhals, Florian, 16, 96 von Borany, Johannes, 40, 66 Vredenbregt, Edgar, 100 Westphal, Michael, 60 Wintz, Sebastian, 110 Wirtz, Tom, 16, 42, 96 Wolf, Daniel, 66 Wolff, Annalena, 12, 88 Wouters, Steinar, 100 Xia, Deying, 34 Xiao, Yin, 12 Xu, Xiaomo, 64, 66 Yu, Fangzhou, 54

Yudin, Petr<br/>,72

Zhang, Hongzhou, 50, 74 Zhou, Yinghong, 12