Multi-User Facilities in LNNano: Electron and Scanning Probe Microscopies, Micro/Nanofabrication

Fernando Galembeck

Director, LNNano/CNPEM

FAPESP
1st Workshop on Multi-User-Equipment and Facilities
04/06/2014

CNPEM

- Non-governamental, non-profit organization (organização social), operates the Synchrotron, Biosciences, Bioenergy (CTBE) and Nanotechnology National laboratories in Campinas, SP.
- Ruled by legislation applicable to private organizations
  - Procurement done following specific rules ("Regimento de Compras"), not subject to Law 8.666.
  - Employment is ruled by CLT.
- Institutional Development Plan completed in 2013, effective from 2014.
  - Directors report to the Board.
  - Career tracks: Researchers, Specialists, Technicians, Management.

CNPEM Mission

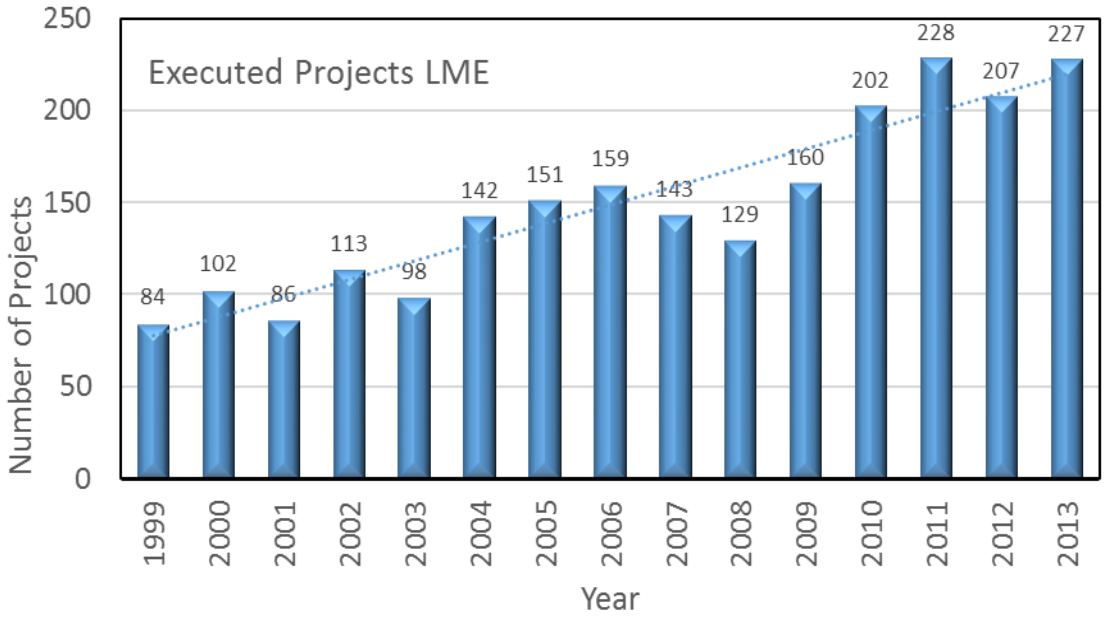
- Operation of national laboratories: support to academic and R&D users
  - In house R&D
- Support to innovative companies
  - Training and education

Funds for Electron Microscopy

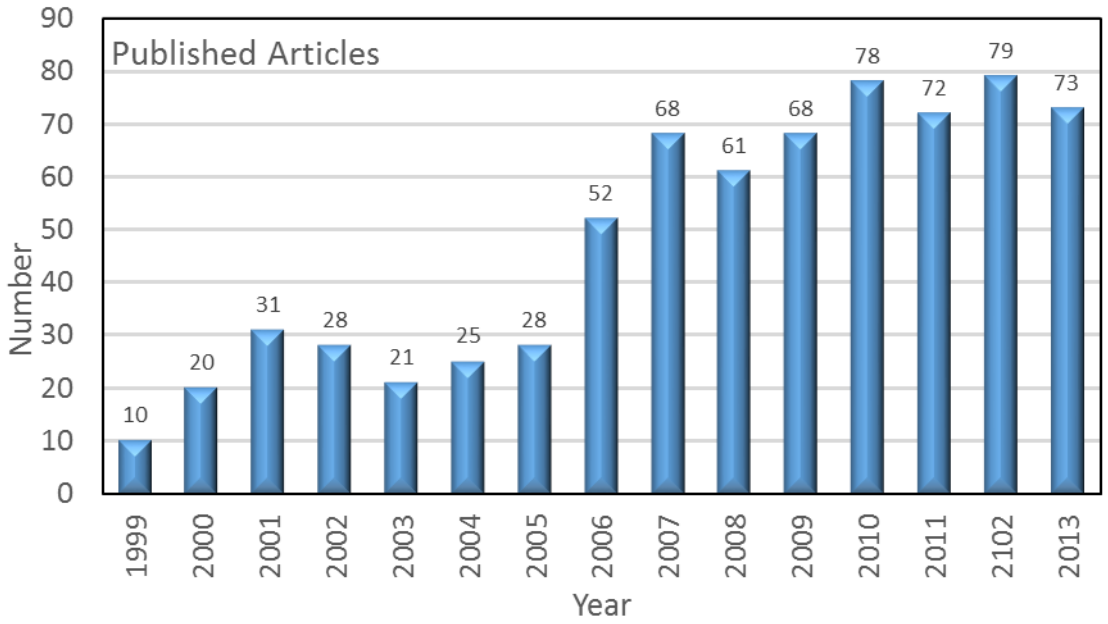
- Multi-User Fapesp Project (Proc. 96/4241-5)
  - later expanded through another multi-user Project (Proc. 2002/04151-9).
- No funds from the 2009 Fapesp call or later.
- Equipment renewed with federal funds
  - Two SEM instruments allowing diferent configurations and experiments: FINEP (01.10.0561.00), 2011.
  - FIB (Dual-beam) instrument, 2014.
  - Annual service contracts for preventive maintenance (ca. R$ 360K .2013).
  - Spare parts, consumables, training.

LME- Electron Microscopy Facility for Materials and Life Sciences

15 years working as an open, multi-user facility



Total: 2004 projects



714 published papers

- 88,548 hours of microscopy provided
- 1072 users trained
- 5 technicians trained for other centers
- 21 user companies (under fees)

The microscope agenda is saturated
Sessions booked *ca.* 4 weeks in advance

- In situ experiments (temperature, mechanical, electrochemical, gas...)
- Atomic resolution
- Diffraction
- Spectroscopy (EELS, EDX)
- Tomography
- Cryomicroscopy
- Single-particle (protein structure)
- Holography

LME- Electron Microscopy Facility for Material and Life Science

Staff - Materials Science

Dr. Jefferson Bettini, CNPEM researcher, > 15 years experience*
Dr. Erico Teixeira Neto, CNPEM researcher, > 8 years experience
Dr. Carlos Ospina, CNPEM researcher, > 7 years experience
Dr. Naga Vishnu, arriving from Ireland in July 2014, > 5 years experience
Sidnei Araujo, CNPEM technician, > 15 years experience*
Fabiano Montoro, CNPEM technician, > 3 years experience*
Other experts in LNNano: Antonio Ramirez, Cristiane Souza, Fernando Galembeck
* Participated from advanced training activities in 2013-2014.

Staff - Life Sciences

Professor Marin Van Heel, CNPq CsF Fellow, > 40 years experience (Imperial College, University of Leyden Cryomicroscopy Center), supported by CsF project
Dr. Rodrigo Portugal, CNPEM researcher > 10 years experience
Dr. Alexandre Cassago,CNPEM researcher, > 2 years experience

Education

V Theoretical-Practical Transmission Electronic Microscopy School (Jan 2014)
315 applicants, 85 participants. http://lnnano.cnpem.br/temschool/

LME- Electron Microscopy Facility for Material and Life Science

Developing Advanced Expertise at LME/LNNano: planned events

Human resources - Materials Science

Dr. Jefferson Bettini – July 2015 – Oxford/UK – Prof. Angus Kirkland
Double corrected microscope – Amorphous materials - **Spectroscopy**

Dr. Erico Teixeira Neto – July 2016 – Lyngby/Demark – Prof. Thomas W. Hansen
Environmental Transmission Microscope - Catalytic nanoparticles – **In-Situ**

Dr. Carlos Ospina – December 2014 UCLA – Prof. Jianwei (John) Miao
Corrected Microscope – Core-Shell Nanoparticles – **Tomography**

Dr. Naga Vishnu – hired from July 2014
December 2015 - Professor Rafal Dunin – Julich/Germany
Double corrected Microscope - Semiconductors Defects – **Holography**

To be selected – **Advanced Electron Diffraction**

Human resources - Life Sciences

Dr. Alexandre Cassago – July 2016 – Leyden – Prof. Marin van Heel
Macromolecular 3D reconstruction – **Single Particle Analysis**

To be selected – **Cryo-tomography**

LME- Electron Microscopy Facility for Materials and Life Sciences

TEM JEM 3010 - 15 years
EDS Capability, Resolution 1.7 Å

TEM JEM 2100F – 6 years
STEM, EDS and EELS
Resolution 1.9 Å

Shared between Materials and Life Sciences

TEM JEOL JEM 2100 – 6 years
STEM, EDS
Resolution 2.4 Å

SEM Inspect 50F – 2 years
STEM and EDS
Resolution 10 Å

SEM Quanta 650F – 2 years
EDS and EBSD
Resolution 10 Å

Dual Beam Helios 660 – 1 month
EDS3D and EBSD3D
Nanolitography, Ion and Electron Beams, 6 Å res.

Other equipment

- JEOL LV SEM, largely used for service to companies and less demanding users.
- JEOL FESEM: 13 years old, currently used as a platform for instrumental development (from Fapesp).
- Equipment for sample preparation (cutting, polishing, ion-milling, ultramicrotomy, sputtering, evaporation...)

Physical infra-structure

- Double-walled building.
- Heavy concrete instrument beds.
- Air-conditioning through smooth dispersant tissue, no wind, strict temperature control.
- Sound absorbent inner wall lining.
- Problems as of 2011:
  - Eletromagnetic noise, solved by making two interventions using active noise suppressors (2011 and 2014).
  - Full dependence from grid power supply: budgeted for 2014 using funds from Finep.

Open, transparent user access

CNPEM
Centro Nacional de Pesquisa em Energia e Materiais

Informe seu login e acesse o Portal de Usuários.

Você não possui login, informe um e-mail e clique no botão acessar.

E-mail

Senha

Esqueci minha senha

Acessar

Portal de Usuários

O Portal de Usuários do CNPEM realiza a gestão das propostas de pesquisas submetidas pela comunidade de pesquisadores do Brasil e do exterior para uso das instalações científicas do LNLS, CTBE, LNBio e LNNano.

Todos os pesquisadores interessados em utilizar os equipamentos dos laboratórios do CNPEM devem utilizar o Portal de Usuários para submeter suas propostas de pesquisa e projetos científicos à aprovação dos laboratórios.

O Portal de Usuários permite ao pesquisador acompanhar, via Web e em tempo real, o status da sua proposta de pesquisa, garantindo a transparência de todo o processo de avaliação.

Ajuda

É recomendado desabilitar bloqueadores de pop-ups.

Serviços de Apoio ao Usuário (SAU)
Telefone: 55 019 3512-1021/ 1025
E-mail: sau@cnpem.br

Comitê de Usuários do LNLS
comite.usuarios@lnls.br

Gestão do desenvolvimento tecnológico:
Grupo de Tecnologia de Informação e Comunicação - TIC

Recomendado

LNLS CTBE LNBIO LNNANO

Attention to users

- Available time, support to users and user output assessed every three months by the CNPEM Board and every six months by a committee nominated by MCTI (Comissão de Avaliação do Contrato de Gestão).
- Complaints and suggestions can be sent to the Electron Microscopy Facility Manager, to the Director of LNNano, to CNPEM General Director and to the CNPEM ombudsman.
- Most recent complaint received in 2011.
  - A user sent a message to the Scientific Director of Fapesp suggesting that São Paulo state researchers should use at least 50% of TEM equipment time. Then, he was informed that São Paulo state researchers (not including CNPEM personnel) were using 80% TEM time.

LME- Electron Microscopy Facility for Material and Life Science

LME Assessment from the LNNano International Advisory Board

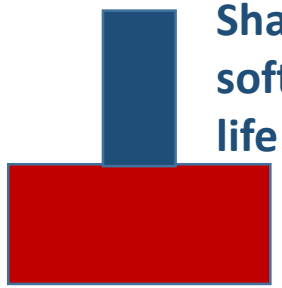
Adalberto Fazzio – IFUSP, USP (Brazil)
Brian Vincent – University of Bristol (Inglaterra)
Daniel Lacks – Case Western Reserve University, Cleveland OH (EUA).
Ponisseril Somasundaran – Columbia University, New York (EUA).
Markus Antonietti – Max Planck Institut, Golm (Alemanha).
Oswaldo Luiz Alves – IQ, Unicamp (Brazil)

This group has established a truly world-class facility. In addition to more standard instruments, this group operates a cryo-TEM, which opens up the exploration of biomaterials and other soft-materials. We note that electron microscopy is rather expensive -both to purchase as well as to maintain and operate. For this reason, it is most efficient to concentrate resources to develop one strong facility in Brazil rather than several weaker ones. In this context, the LNNano group is poised to take a leadership role throughout Brazil and South America. One way to help achieve this goal may be to create satellite locations with remote instrument access, which are nearer to other users but are run by LNNano staff".

LME- Electron Microscopy Facility for Materials and Life Sciences

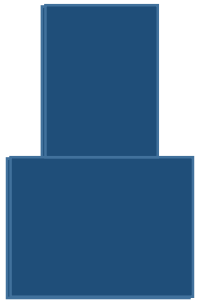
Acquisitions under negotiation in 2014

Double Corrected TEM - Holography STEM, EDS and EELS, In-situ Tomography, Resolution 1.0 Å



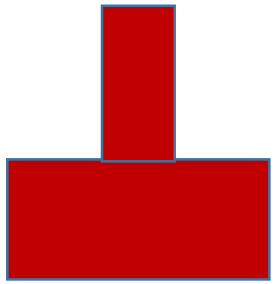
Shared between soft materials and life sciences

TEM 120 kV
EDS - Cryo
Resolution 3.2 Å

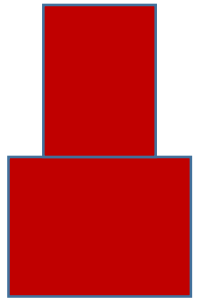


Dual Beam
Dedicated to sample preparation
Resolution 10 Å

~ 2 years



TEM 200-300 kV – Energy Filter, Phase Plate, Direct Detector, Resolution ~ 2 Å

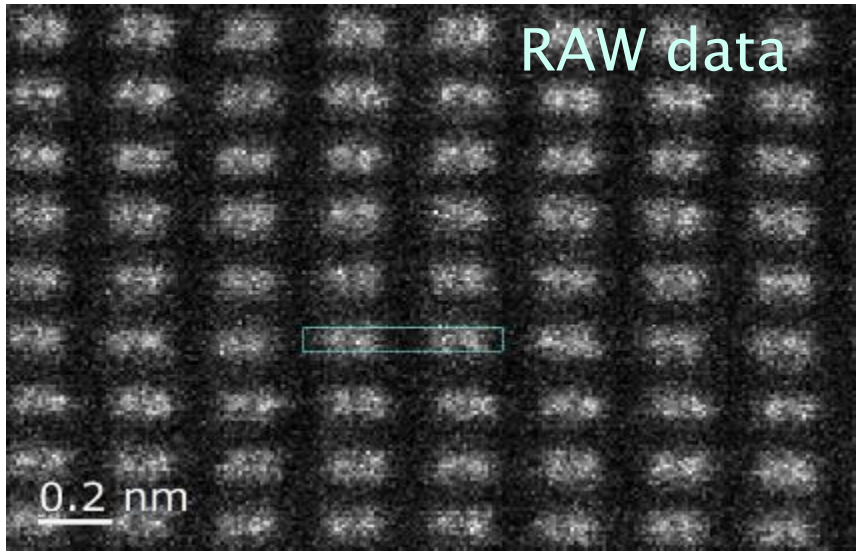


Dual Beam – 3D Reconstruction
Dedicated to Life Sciences
Resolution 10 Å

Currently there are three corrected instruments in Brazil, none in São Paulo state.

LME- Electron Microscopy Facility for Material and Life Science

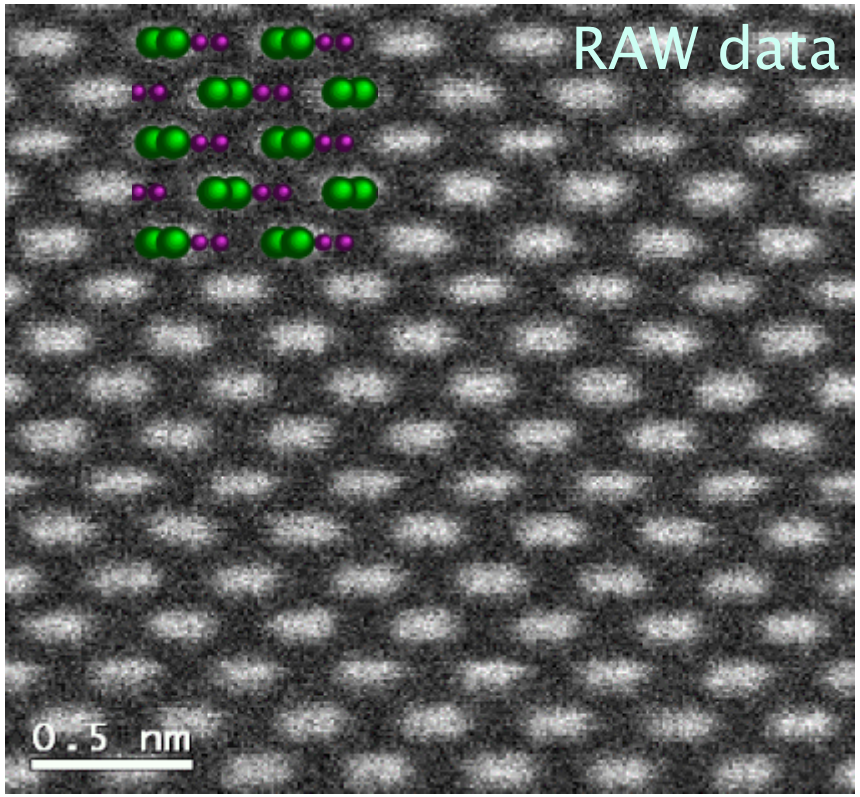
Why a corrected electron microscope?



Si(112) 78 pm dumbbell image

Resolution

GaN

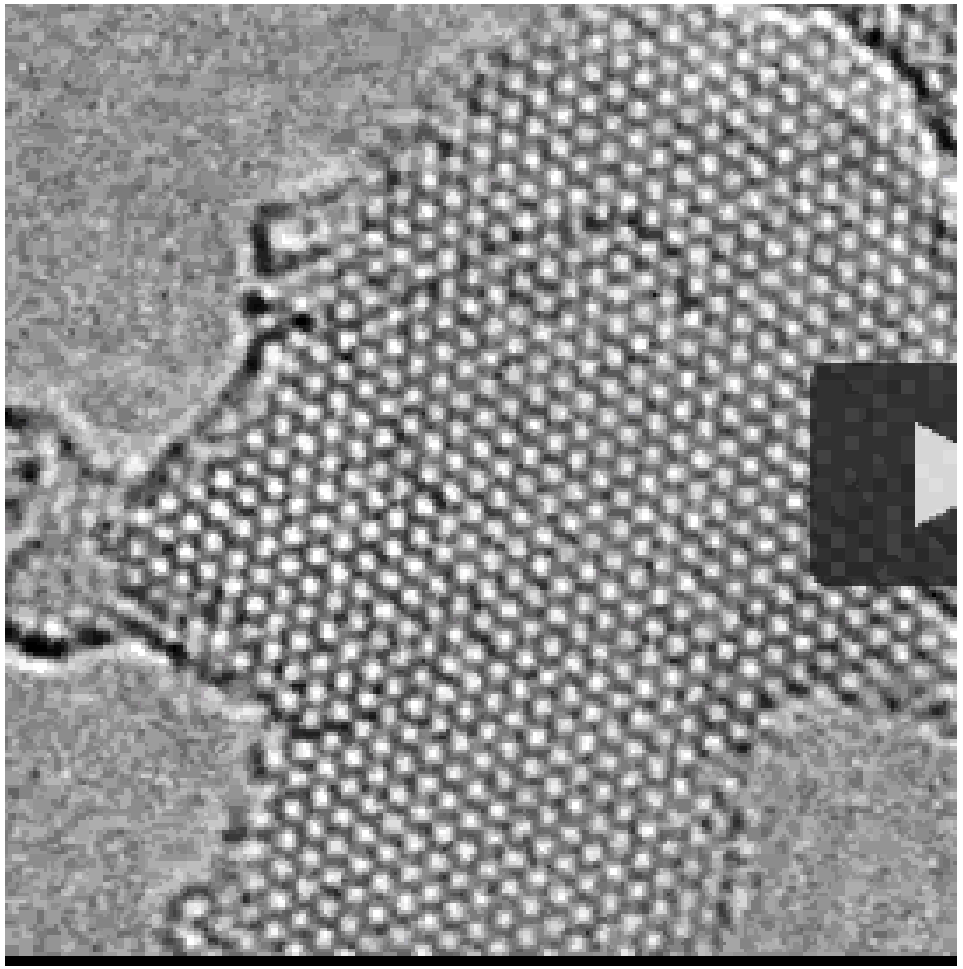


GaN [211] 63 pm HAADF at 200 kV

Resolution bellow **1.5 Å** can be reached automatically.
Atomic resolution (bellow **1 Å**) can be achieved in STEM and TEM.

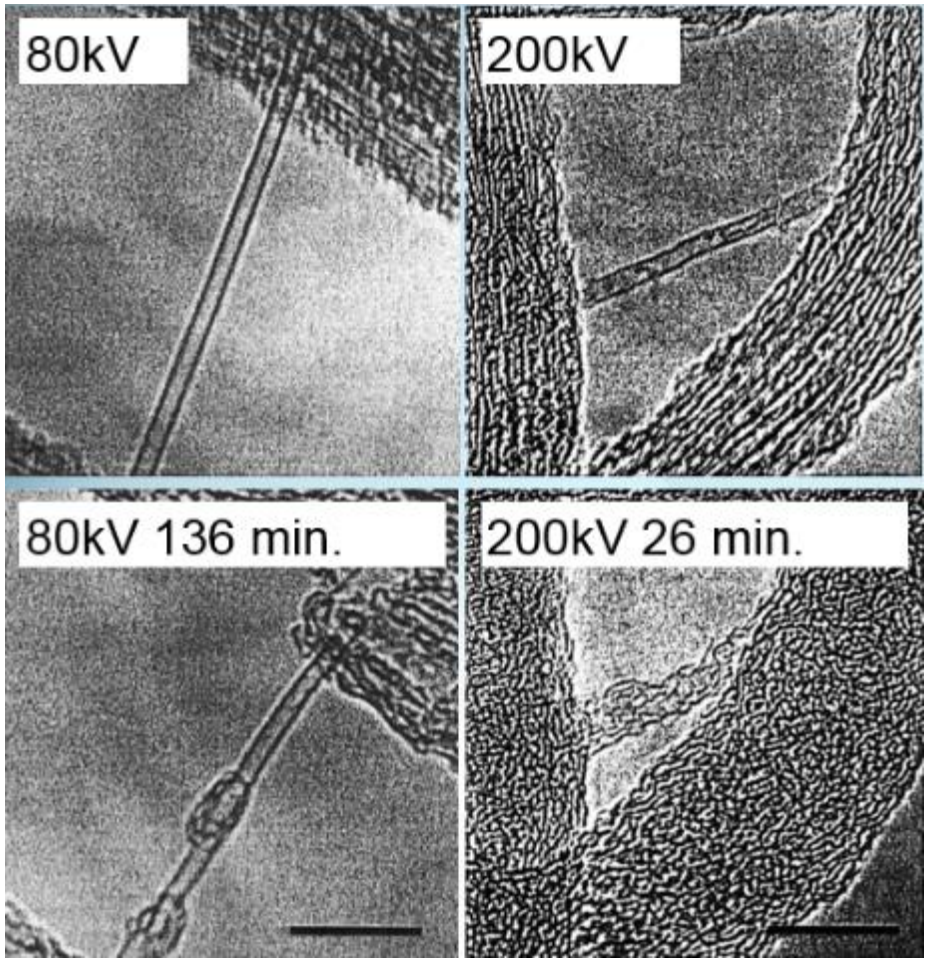
LME- Electron Microscopy Facility for Material and Life Science

Why a corrected electron microscope?



Transformation of a graphene sheet into a fullerene.
Video credit: Andrey Chuvilin, et al.

Light Materials

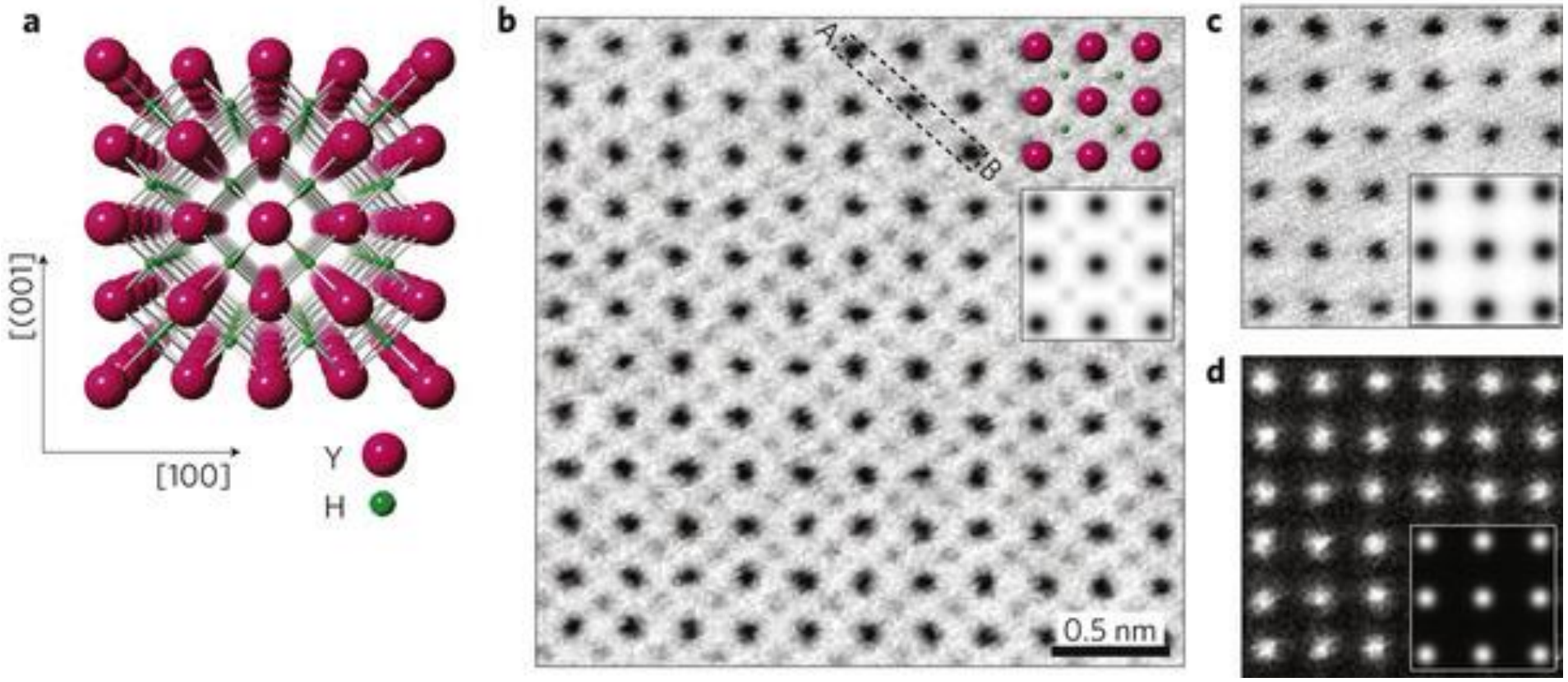


Knock-on damage is reduced at low acceleration voltage.

LME- Electron Microscopy Facility for Material and Life Science

Why a corrected electron microscope?

Light Materials

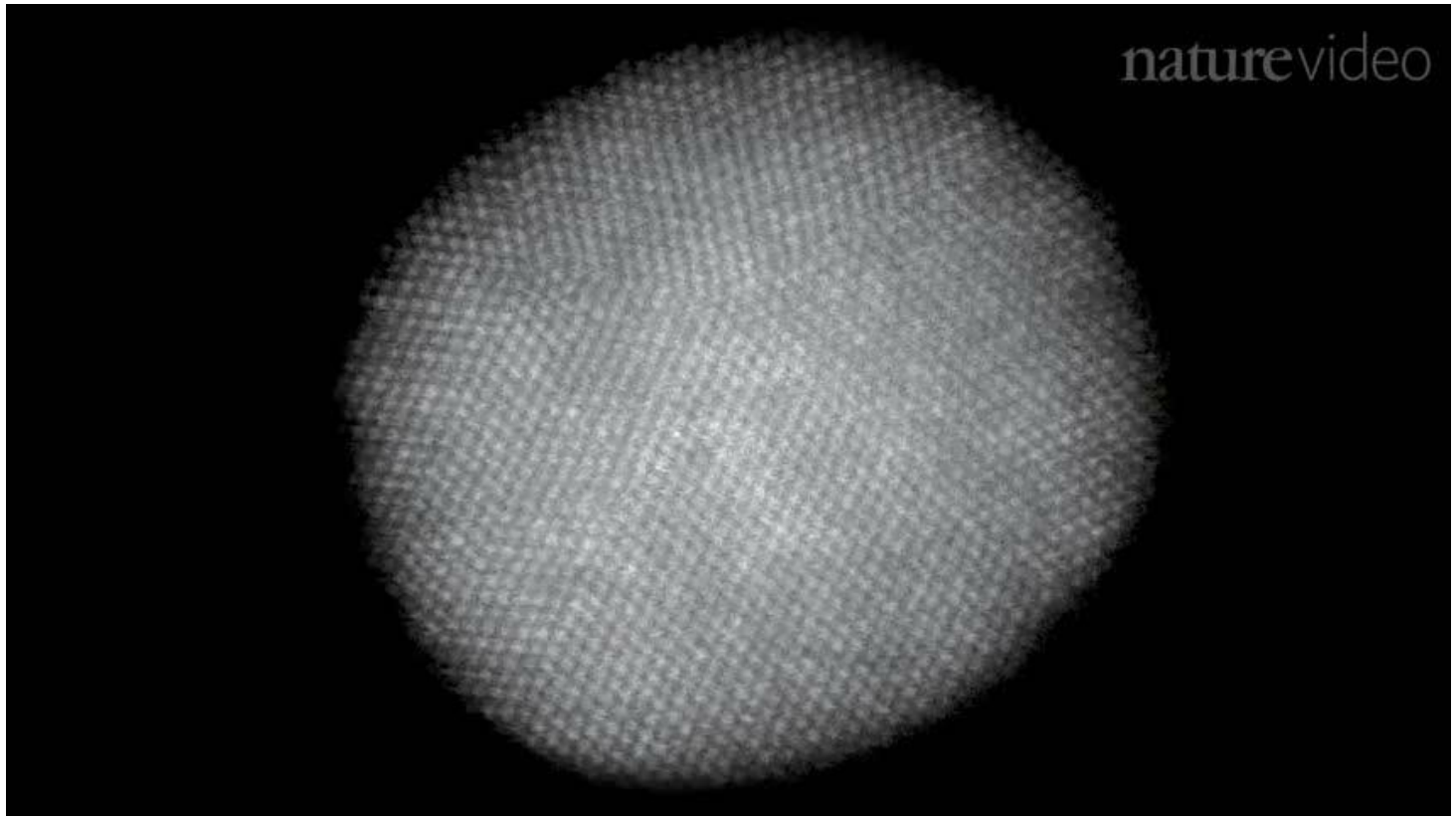


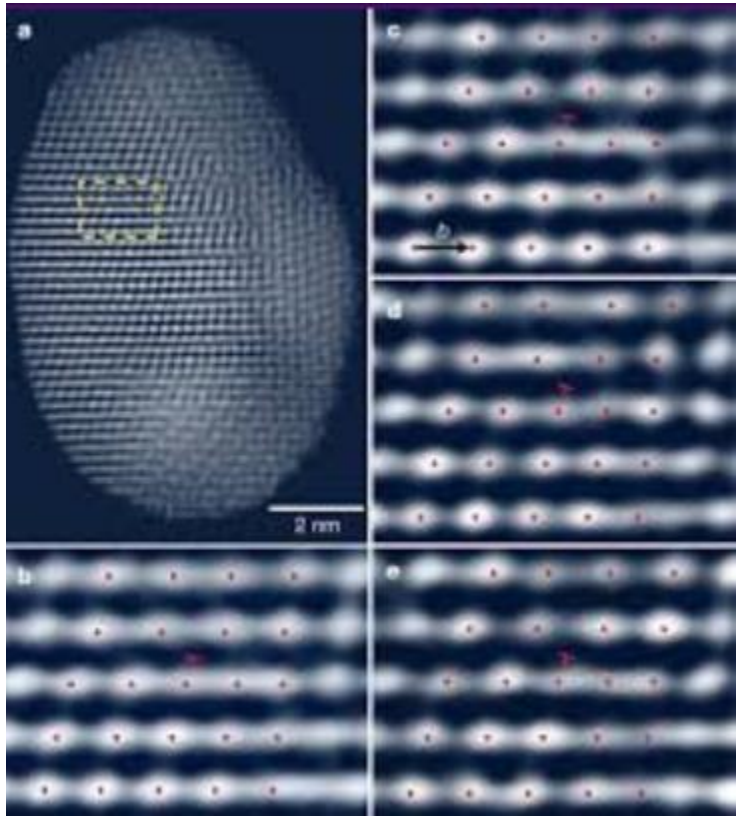
Direct imaging of hydrogen-atom columns in a crystal by annular bright-field electron microscopy
Ryo Ishikawa, Eiji Okunishi, Hidetaka Sawada, Yukihito Kondo, Fumio Hosokawa & Eiji Abe
Nature Materials 10, 278–281 (2011) doi:10.1038/nmat2957

LME- Electron Microscopy Facility for Material and Life Science

Why a corrected electron microscope?

High Resolution Tomography



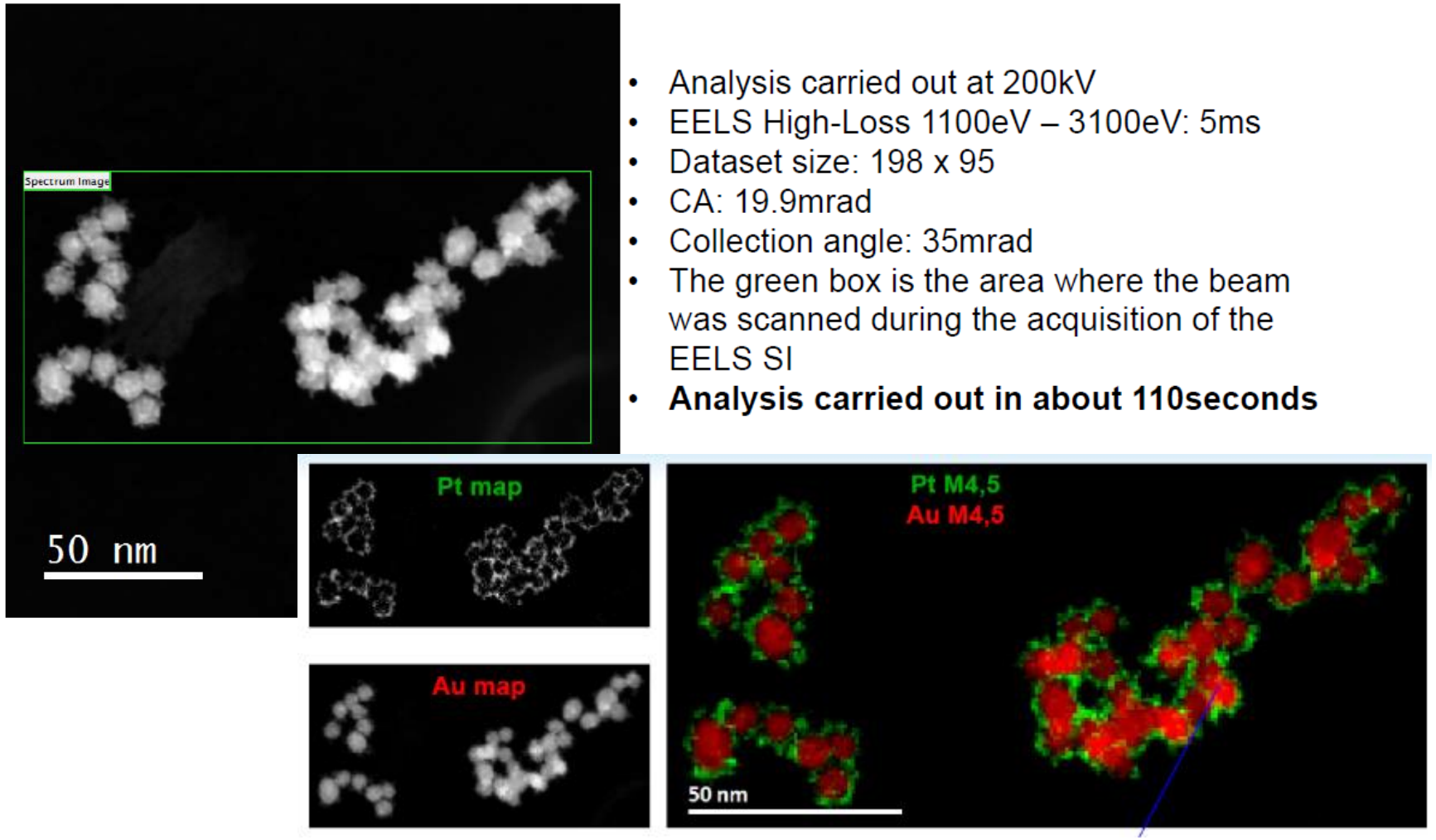


Chen, C. C. , Zhu, C., White, E. R., Chiu, C. - Y., Scott, M. C., Regan, B. C., Marks, L. D., Huang , Y. & Miao, J. Three - dimensional imaging of dislocations in a nanoparticle at atomic resolution. **Nature** 496 , 74 – 77 (2013).

LME- Electron Microscopy Facility for Material and Life Science

Why a corrected electron microscope?

Data throughput



- Analysis carried out at 200kV
- EELS High-Loss 1100eV – 3100eV: 5ms
- Dataset size: 198 x 95
- CA: 19.9mrad
- Collection angle: 35mrad
- The green box is the area where the beam was scanned during the acquisition of the EELS SI
- **Analysis carried out in about 110seconds**

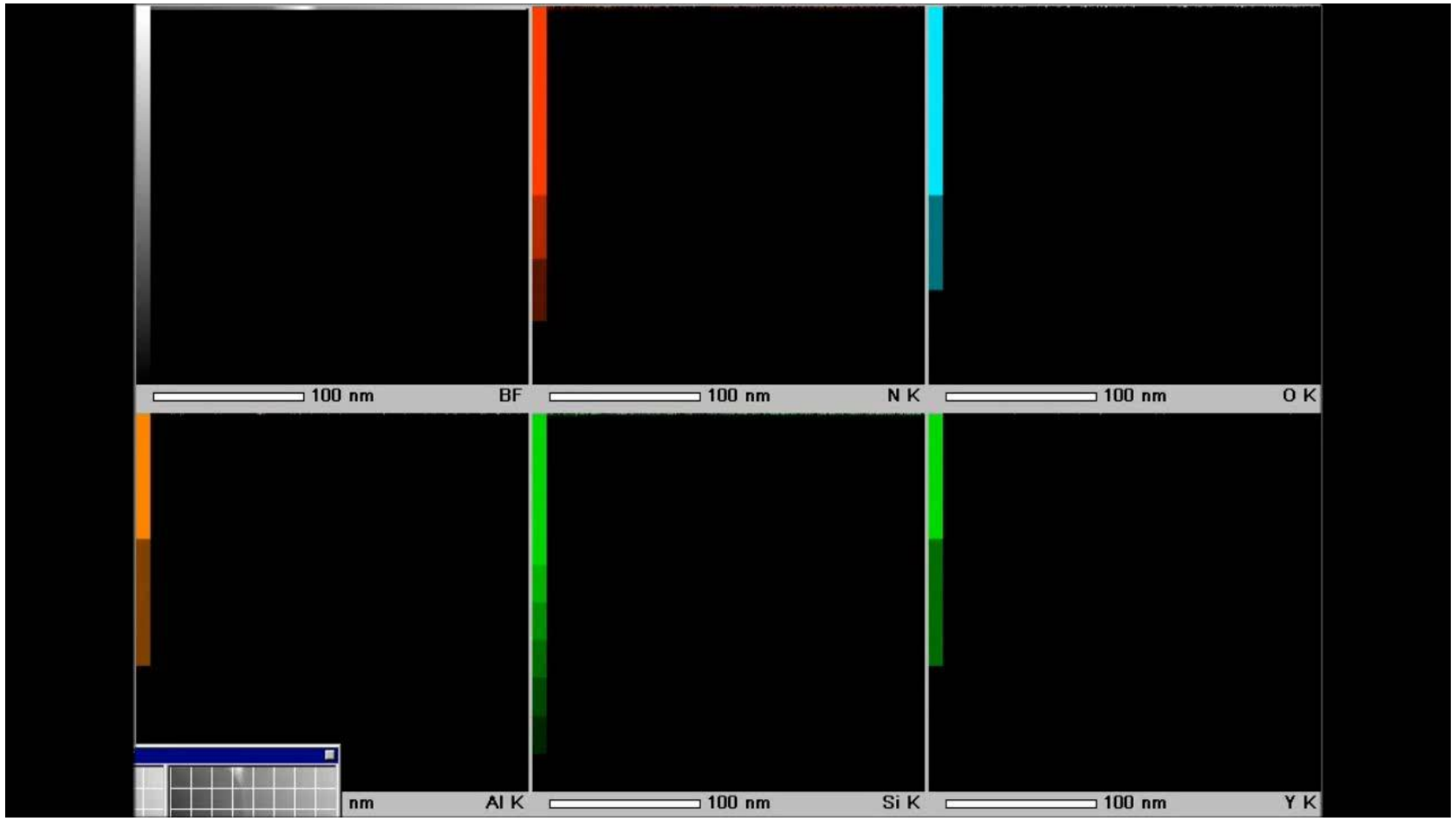
Corrected microscope reaches 6X higher current at the same probe size. New detectors are 6X more efficient than our current ones.

Twice as many users can attended

EELS STEM-SI Acquisition, R. Twesten, P. Longo, A. Aitouchen and P.J. Thomas, Microscopy and Microanalysis

LME- Electron Microscopy Facility for Material and Life Science

Why a corrected electron microscope? **High data throughput**



Faster results

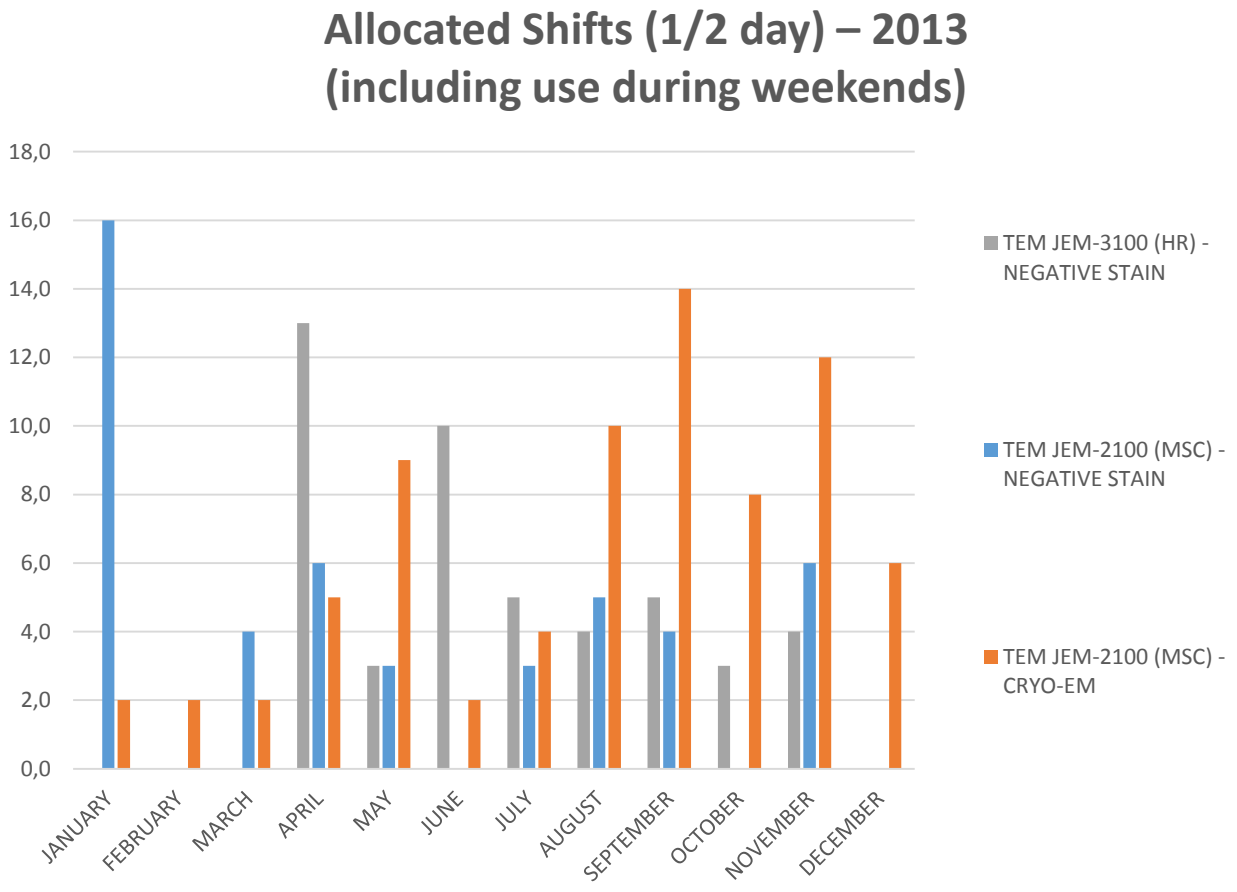
256x256 pixel

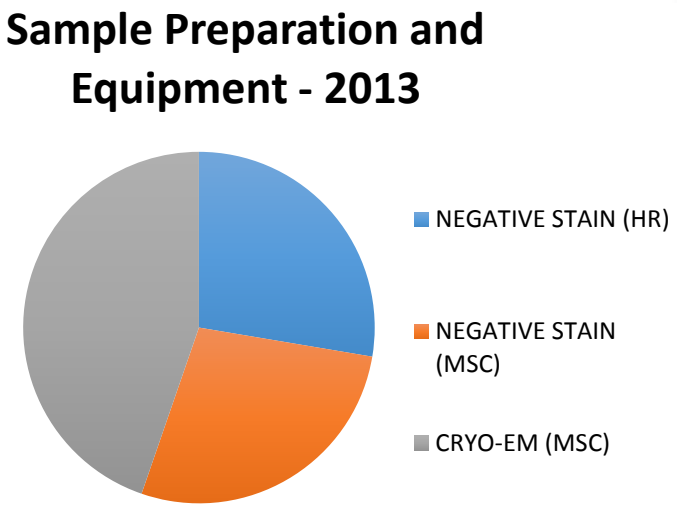
Real time display

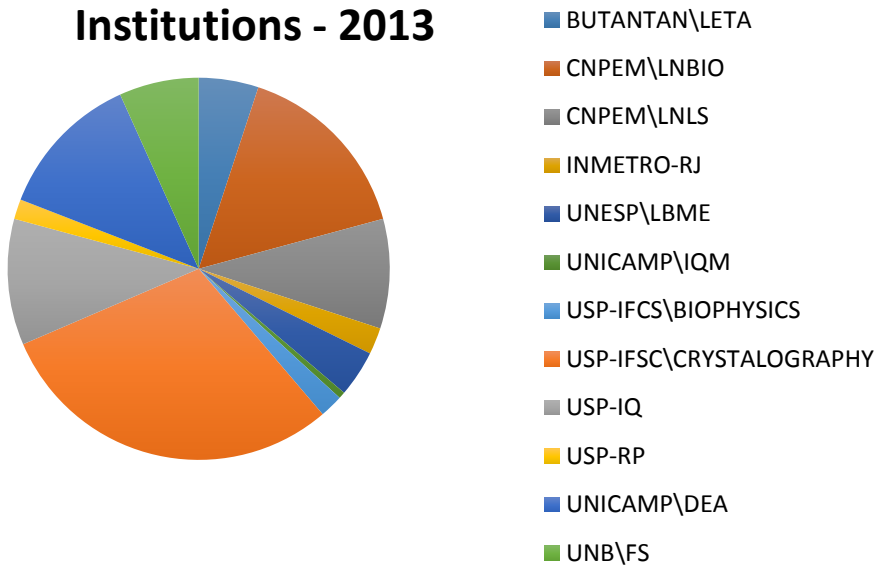
High-Speed, EDS STEM-SI Acquisition: Nitrogen, Oxygen, Aluminum, Silicon and Ytrium

LME- Electron Microscopy Facility for Material and Life Science

Why a electron microscopy facility for Life Sciences?



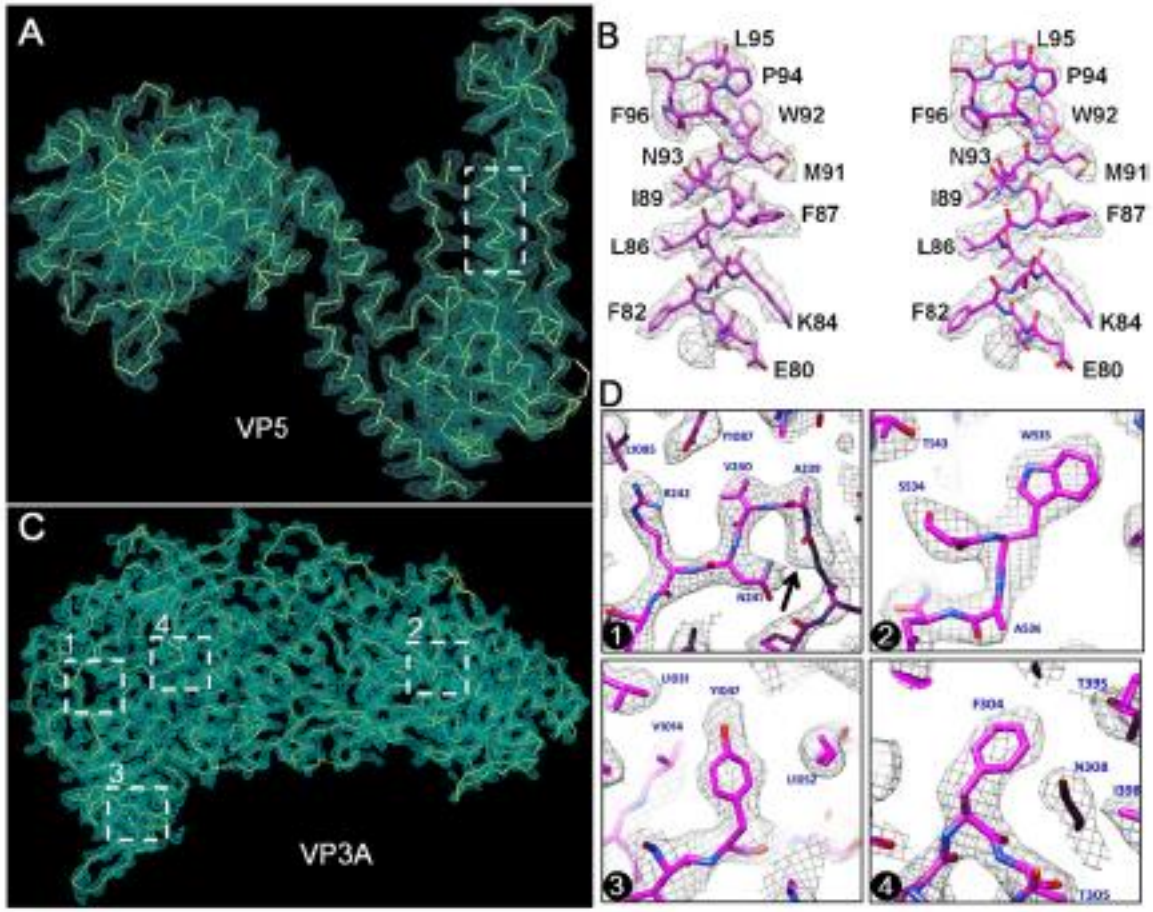


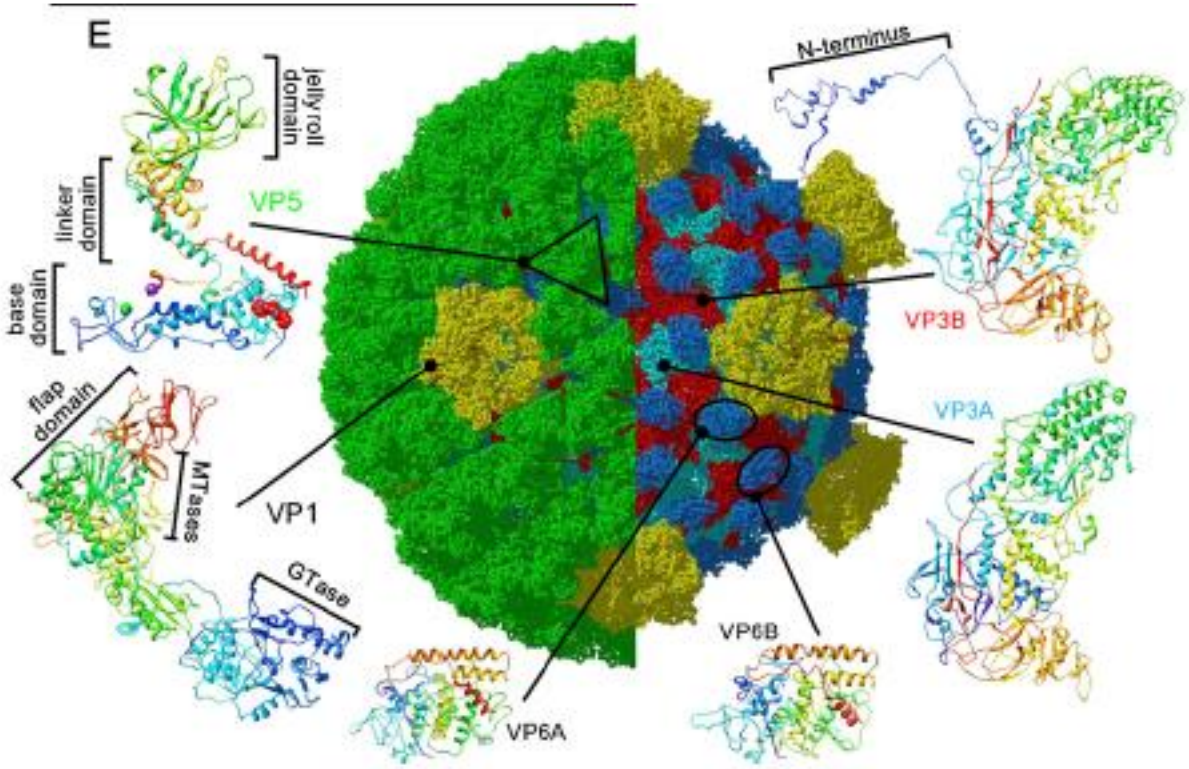


LME- Electron Microscopy Facility for Material and Life Science

Why a high end cryo electron microscope?

3D structure of macromolecules at near atomic resolution





3.3 Å Cryo-EM structure of a nonenveloped virus reveals a primary mechanism for cell entry

Reference

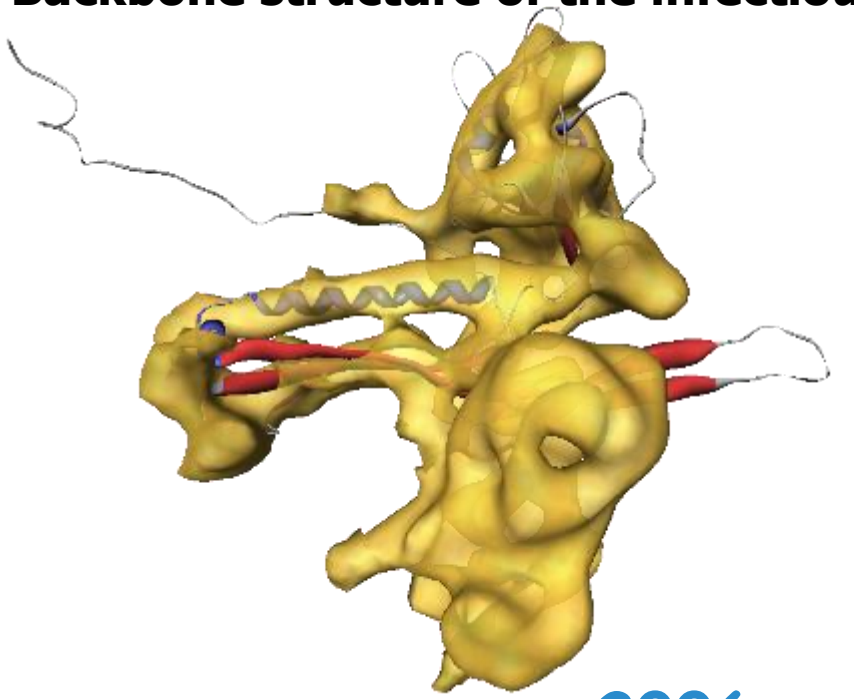
Zhang X., et al., (2010) *Cell*, 141:472-482.

LME- Electron Microscopy Facility for Material and Life Science

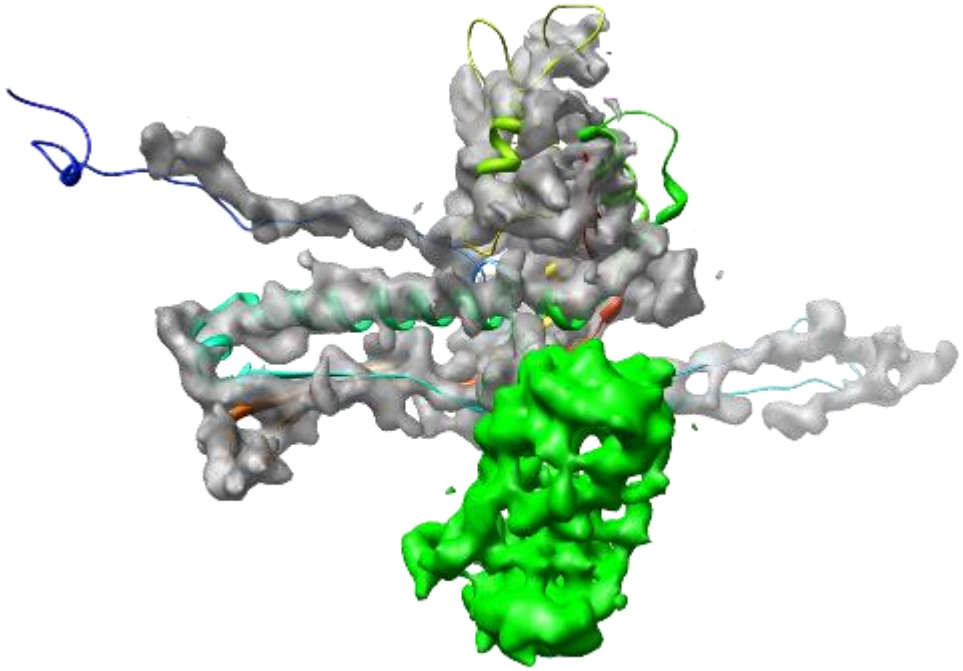
Why a high end cryo electron microscope?

3D structure of macromolecules at near atomic resolution

Backbone structure of the infectious ε15 virus capsid



9.5Å 2006 (Nature)



4.5Å 2008 (Nature)

Reference

Jiang et al., (2008), *Nature*, 451:1130. Backbone structure of the infectious ε15 virus capsid revealed by electron cryomicroscopy

LNNano
Laboratório Nacional
de Nanotecnologia

Surface Science Laboratory – LCS
http://lnnano.cnpem.br/laboratories/mta/

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Ministério da
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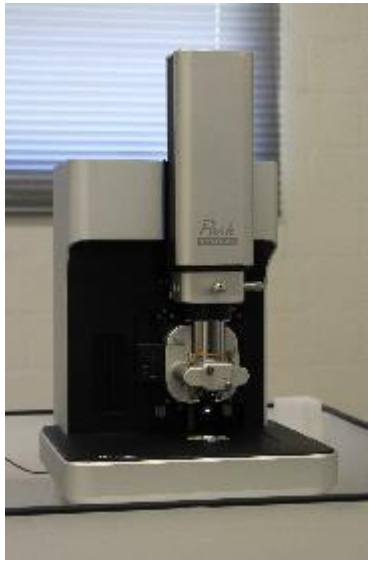
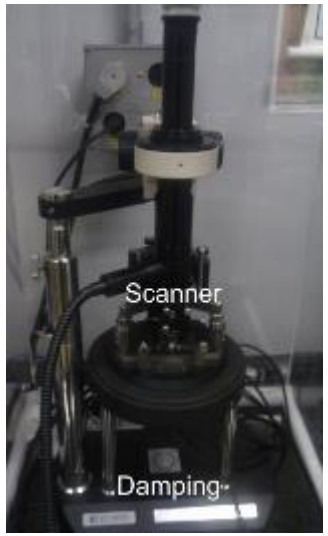
Scanning Probe Instruments



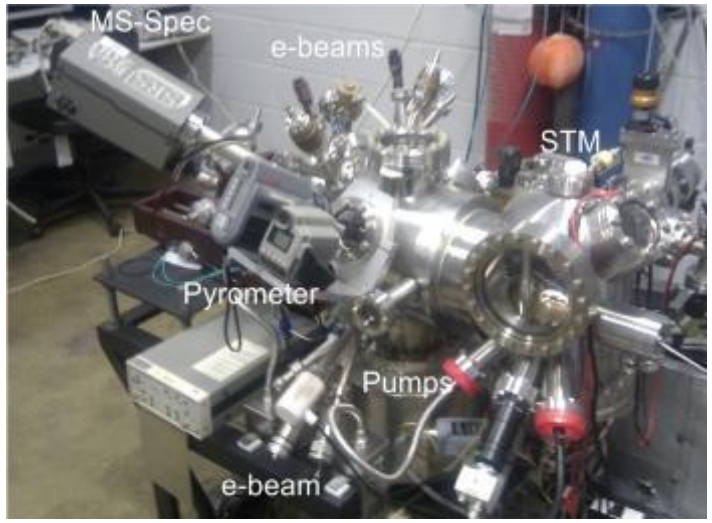
PARK – NX 10
Out. de 2013

DIGITAL – Nanoscope IIIa

NANOSURF - FlexAFM



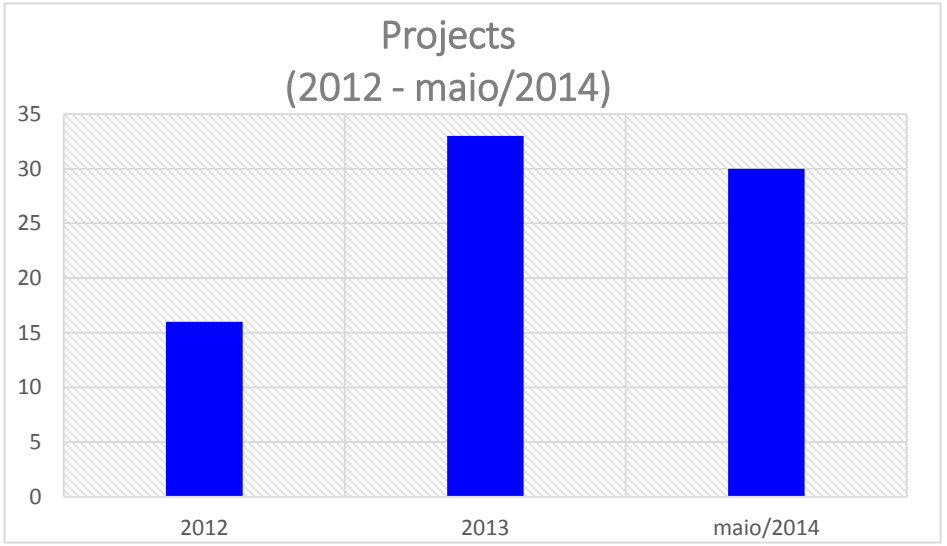
NT-MDT – Solver Pro

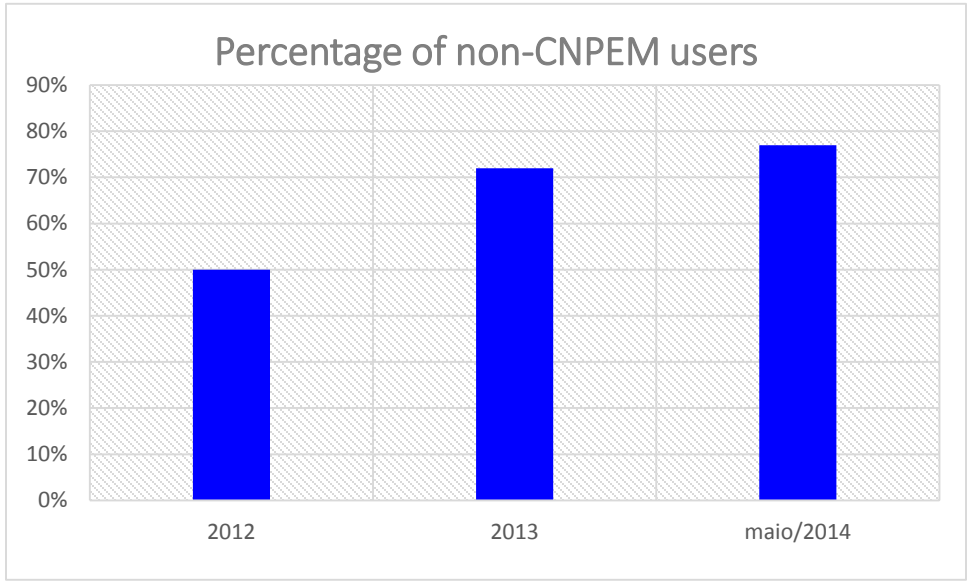


OMICRON – UHV-STM

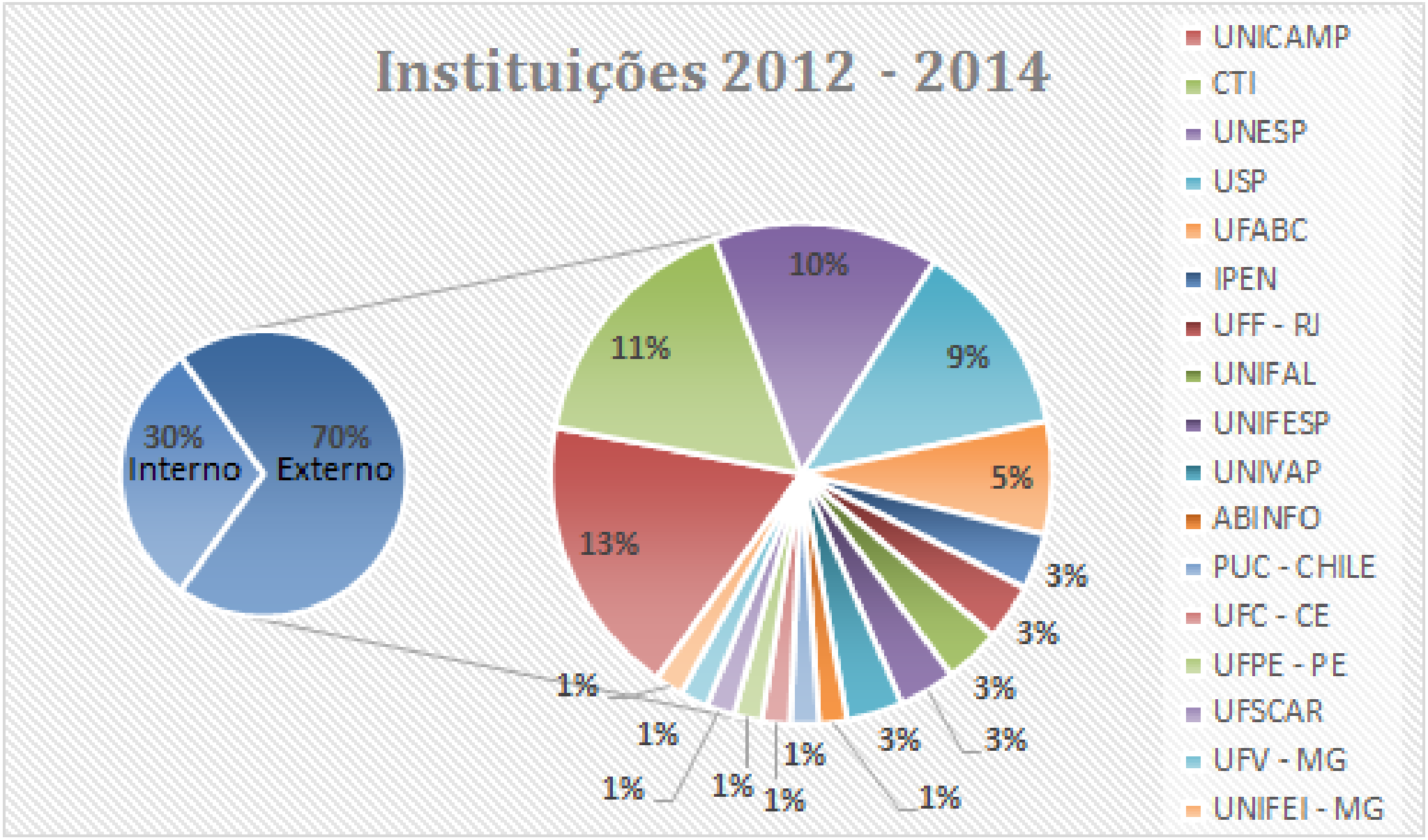
Less expensive than electron microscopes but can only be fully exploited by a highly experienced team.

Users: 2012 – May 2014

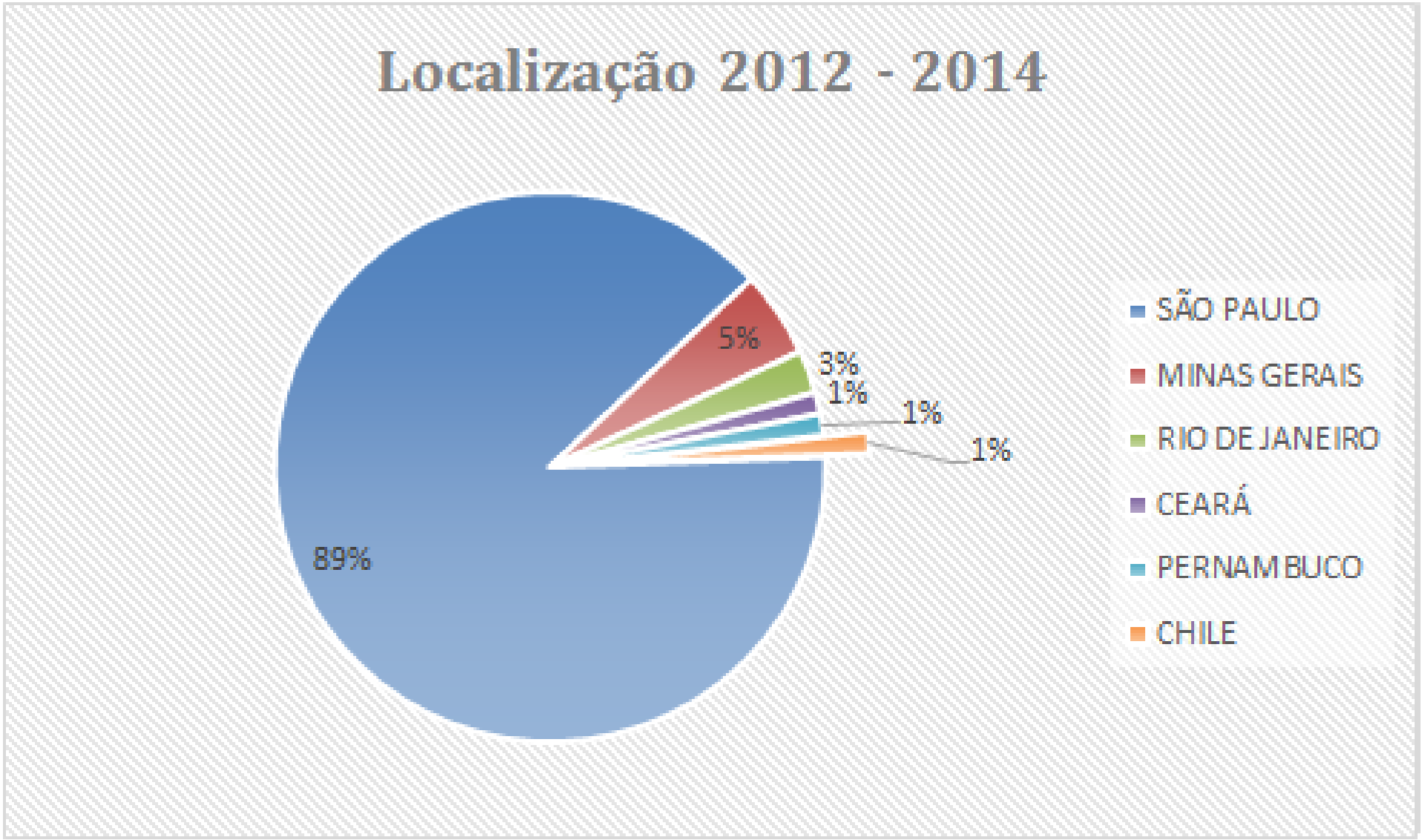




User affiliation



User location



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Laboratório Nacional
de Nanotecnologia

Micro/Nanofabrication – LMF
http://lnnano.cnpem.br/laboratories/lmf/

CNPEM
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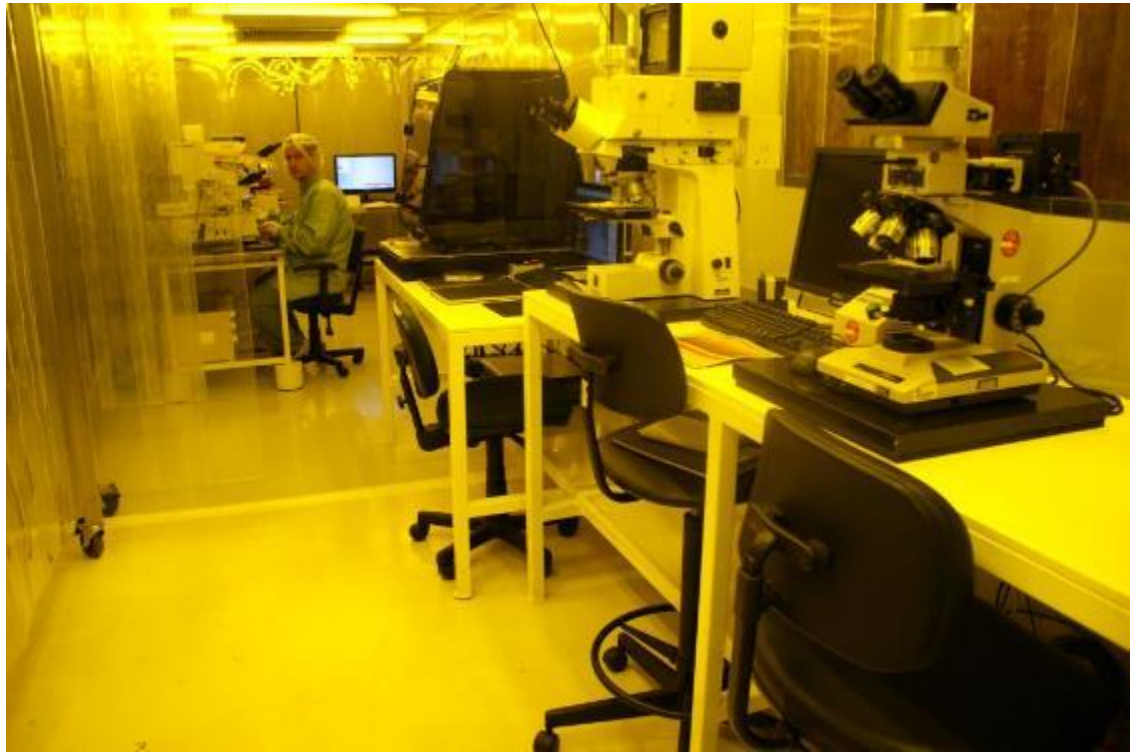
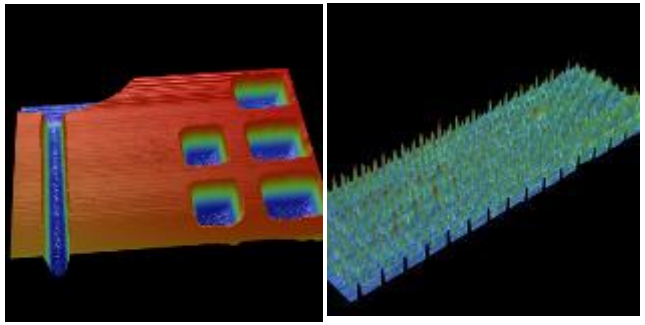
Ministério da
Ciência, Tecnologia
e Inovação

GOVERNO FEDERAL
BRASIL
PAÍS RICO É PAÍS SEM POBREZA

LMF – Clean Room

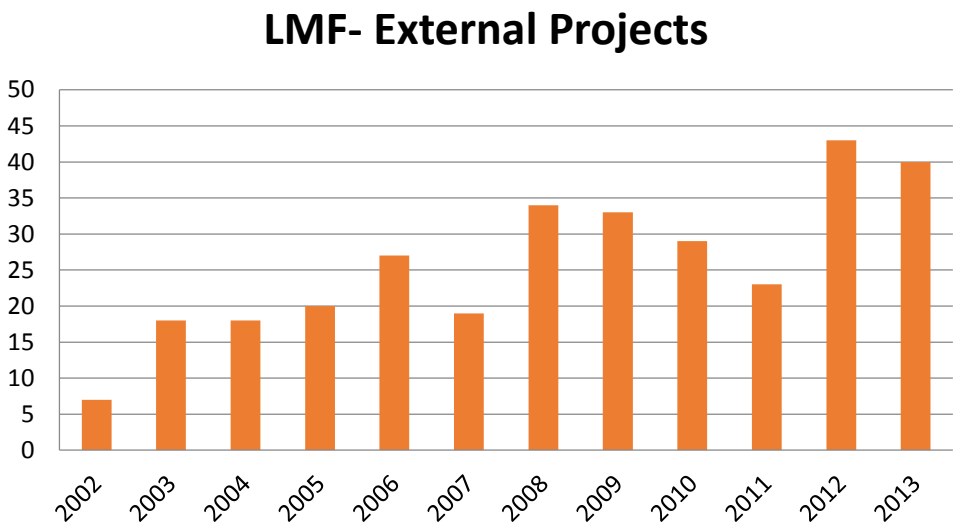


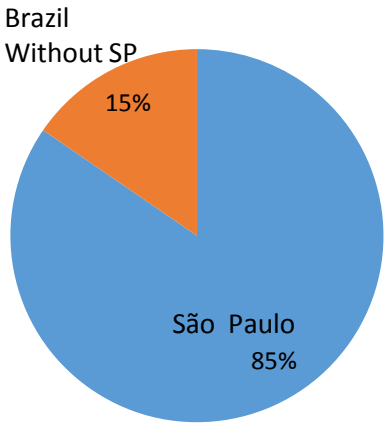


ISO 7 - 20m^2

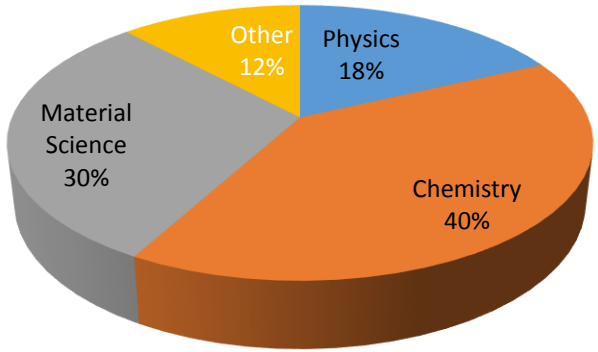
2 Karl Suss photoallignment units
Laser Pattern Generator(3µm)
Profilometer Dektak 150

User data



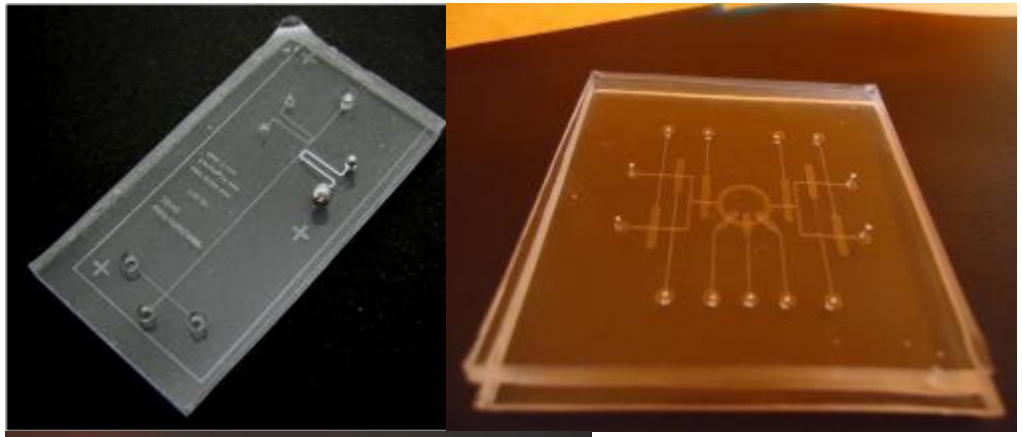


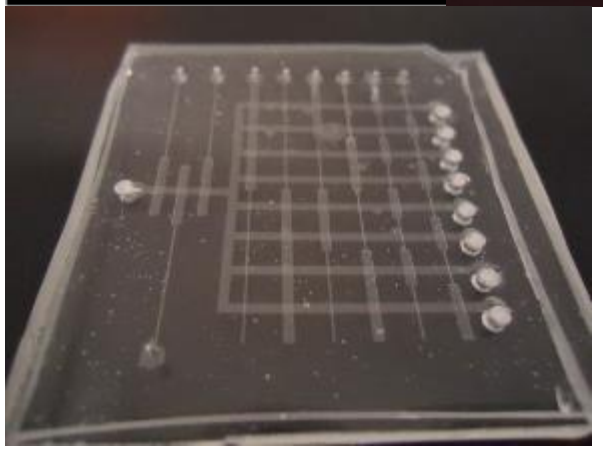
Year	Articles	Int. Conferences	Master Thesis	PhD thesis
2010	12	9	3	2
2011	10	4	2	1
2012	17	19	2	1
2013	23	18	6	3



Microfluidics platform

PDMS





Papel

Output

Water/etanol sensor

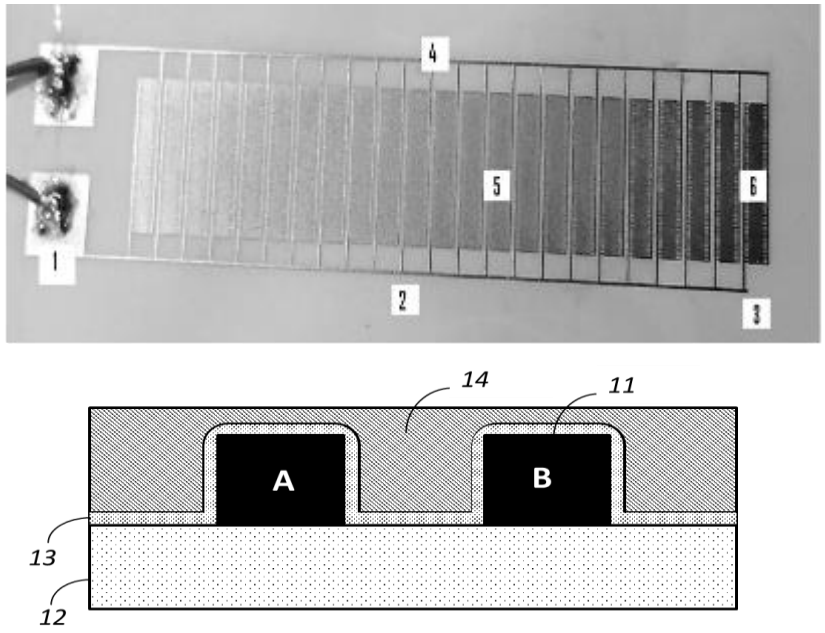


Figura 06

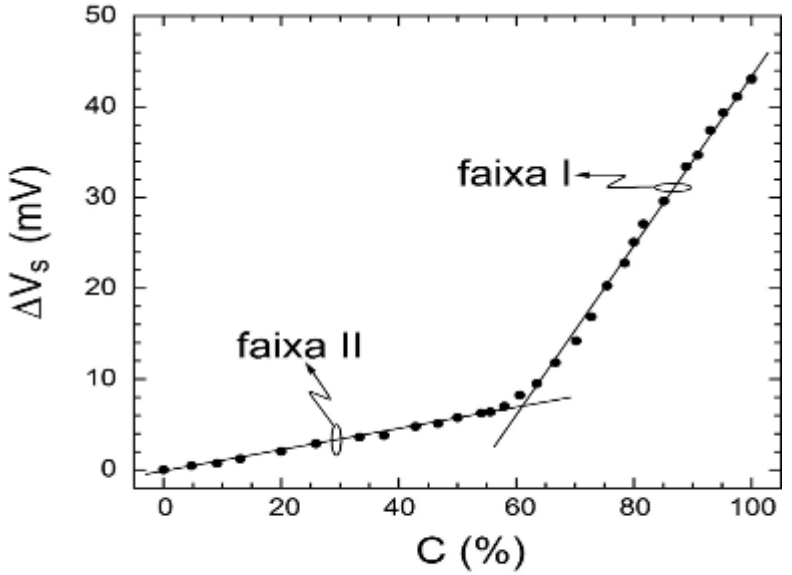
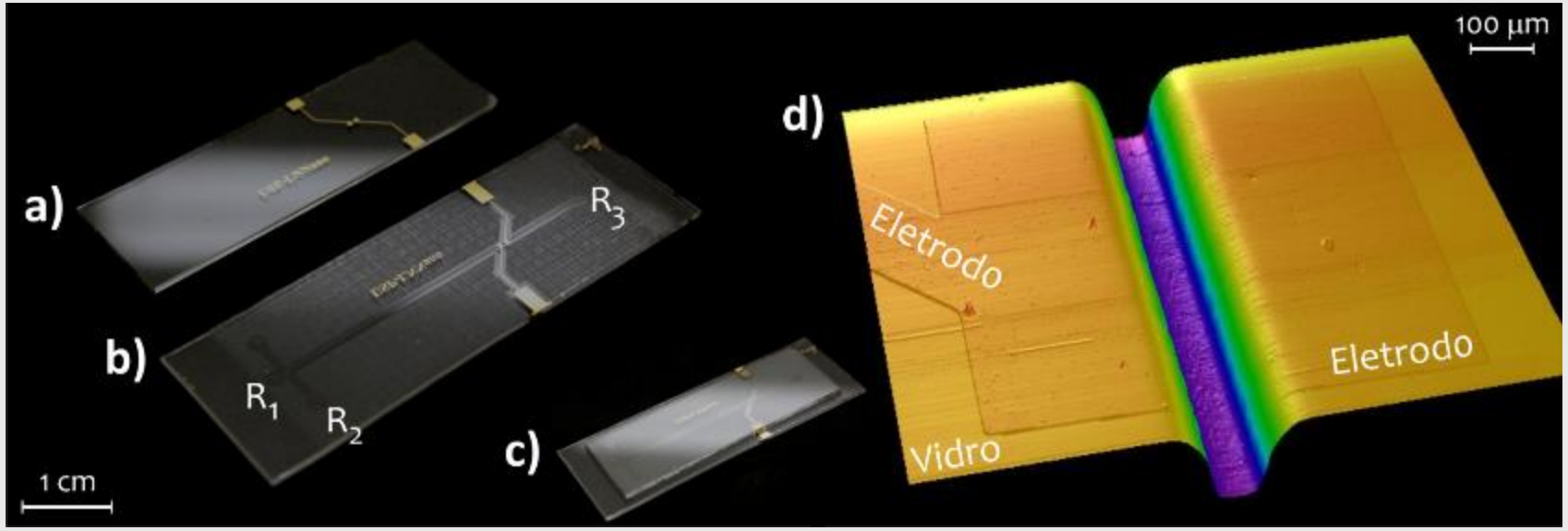


Figura 07

Patent Appl.: BR10 2014 0097457

Output

Concentric electrodes for C^4D applications

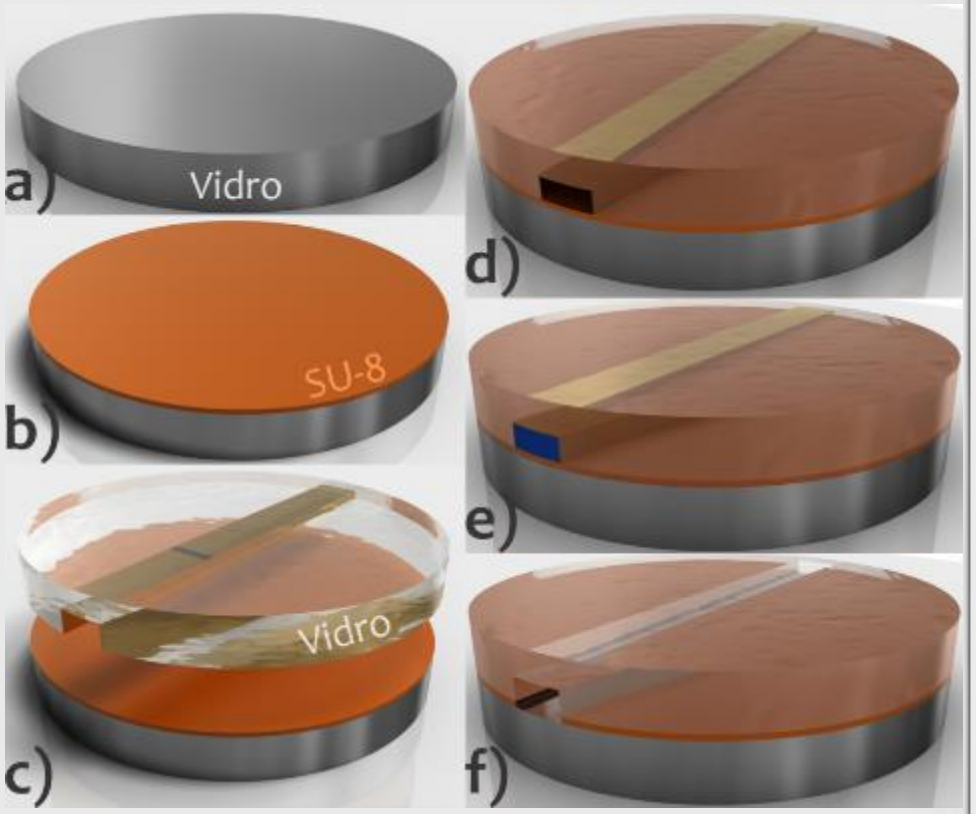


- ✓ Detection limit is four orders of magnitude than using conventional planar electrodes.
- ✓ Patent (USP/SC & CNPEM), paper in *Chemical Communications*.

Output

Sacrificial adhesive sealing for glass microchips

- ✓ **Simples, fast process** without using higher temperatures or pressure. Does not require clean room facilities.
- ✓ **Strong adhesion.** Microchannels support p> 4 Mpa.
- ✓ **P**atent Appl. (CNPEM e USP) **BR 102014012630-9**



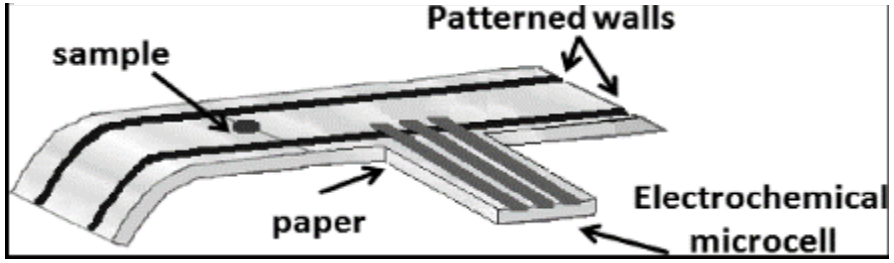
Output

Electrochemical Detection in a Paper-Based Separation Device
UNICAMP and LMF/LNNANO

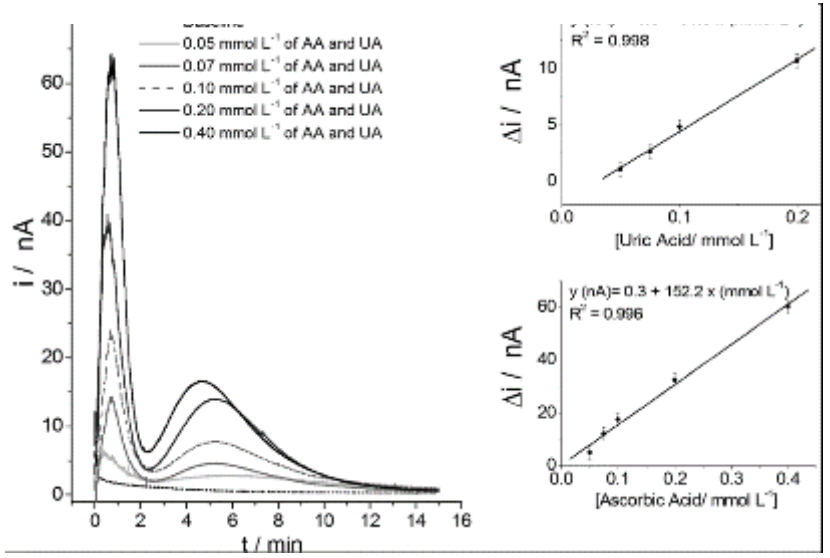
Analysis of salicylate concentrations in spiked blood samples.

Blood samples	Initial salicylate concentration/mmol L^{-1}	Added salicylate concentration/mmol L^{-1}	Biosensor recovery/%	Experimental salicylate concentration[a]/mmol L^{-1}	
				Biosensor	Standard method
1	0.00	0.10	101.9	0.10 ± 0.01	-
2	0.00	0.25	109.1	0.28 ± 0.04	0.26 ± 0.06
3	0.00	0.51	100.6	0.51 ± 0.08	0.51 ± 0.05
4	0.00	1.01	119.3	1.21 ± 0.05	-
5	0.00	2.02	99.4	2.01 ± 0.06	2.02 ± 0.04

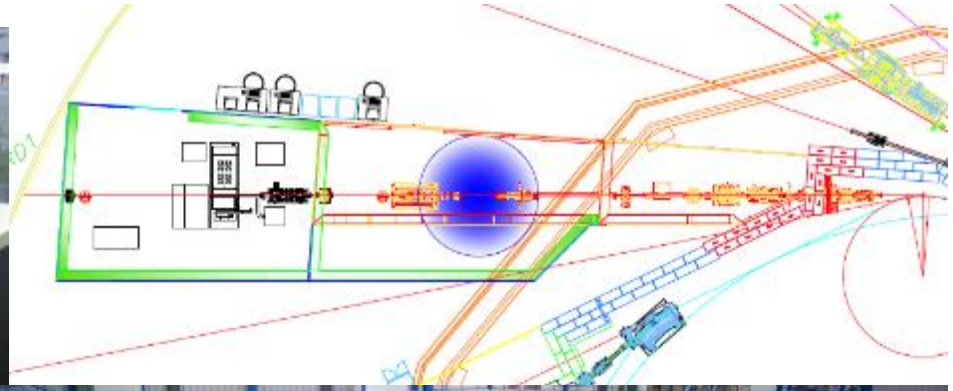
[a] Mean ± standard deviation (n= 3).



Patent Appl. Unicamp-CNPEM
PI 1011116-6 A2



Parallel Diffraction and Thermo-Mechanical Simulation



First Class User Community

Brazil, EUA, Germany, Japan, Argentina, Peru,.....

Funded by Petrobras - FINEP

XTMS Users

Year/ Sem.	Institution Submitting	Origin Countries	Submitted projects	% Approval	Beam time [h]	Beam time for Brazilian Groups	Phase Transformation studies
2013/1	OSU- UNT-UNICAMP; OSU-UT-ORNL, ITA, CNEA	USA, Brazil, Argentina	4	100%	552	35%	Ti-As, SSs, DSSs, Ni-BAs, Steels, SMAs
2013/2	UT-ORNL, ITA, UFC, BAM, LNNano, USP	USA, Brazil, Germany	7	86%	960	58%	SSs, Ni-BAs, Maraging Steels, Adv. low distortion steels, DSSs, Super-Martensitic SSs.
2014/1	UT-ORNL, ITA, UFC, IBARAKI U., LNNano, USP, US-NAVY, UNICAMP, BAM, PETROBRAS	USA, Brazil, Japan, Germany	13	62%	984	59%	SSs, Ni-BAs, Maraging Steels, Ultra-high strength steels, Super-Martensitic SSs, Trip Steels, 9Ni Steels
2014/2	UT-ORNL, ITA, UFC, LNNano, USP, UNILIBoa, ORNL, U. BUENOS AIRES, PETROBRAS	USA, Brazil, Portugal, Argentina	13	46%	936	62%	SSs, Ni-BAs, Ultra-high strength steels, Trip Steels, 9Ni Steels, Pipeline steels.
Total		**6**	**37**	**65%**	**3432**	**55%**	

Ti-As: Ti-alloys; SSs: Stainless steels; DSSs: Duplex Stainless Steels;
Ni-Bas: Ni-based alloys; SMAs: Shape Memory Alloys

In-situ Measurements - XTMS

- Fundamental studies
  - Solid state phase transformations
  - Solidification
  - Solid-gas reactions (oxidation, etc…)
- Study strain/stress effect on phase transformations and vice versa
- Expedite new materials development
- Materials and processes optimization

LMF

- **O Laboratorio de Microfabricação (LMF) do LNNano/CNPEM é um laboratório aberto, multidisciplinar dedicado ao desenvolvimento de dispositivos e processos em escala micro.**

  • fabricação de dispositivos semicondutores
  • sistemas microeletromecânicos (MEMS) (atuadores, engrenagens)
  • microfluídica (lab on a chip, soft litography)
  • sensores ópticos
  • sensores químicos (colorímetros, separação, detecção – amperométrico e voltamétrico)
  • Sensores de deslocamento (MEMS acelerômetros)
  • Dispositivos RF (filtros THz, SAW, RFID)

LMF

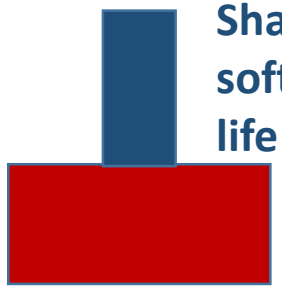
- **O LMF oferece uma ampla capacidade de processamento.**
-

  • Processos de fotolitografia (UV e laser patern generator)
  • Processos de corrosão por via úmida ou seca (metais, semicondutores, dieletricos e polímeros)
  • Physical Vapor Deposition (evaporação por feixe de elétrons e por sputtering DC/RF)
  • Chemical Vapor Deposition (PECVD de óxidos e nitretos)
  • Recozimento e cura (fornos térmicos e de tratamento rápido)
  • Inspeção (microscópios ópticos e imagem 3D)
  • Metrologia em filmes finos (medidores de espessura, elipsometria e resistividade)

LME- Electron Microscopy Facility for Material and Life Science

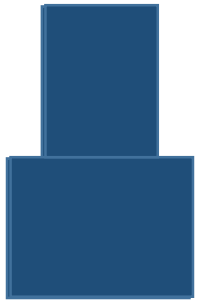
Acquisitions under negotiation in 2014

Double Corrected TEM - Holography STEM, EDS and EELS, In-situ Tomography, Resolution 1.0 Å



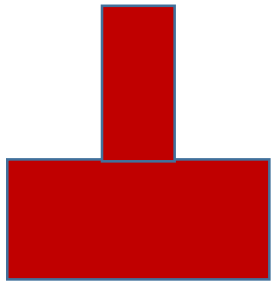
Shared between soft materials and life sciences

TEM 120 kV
EDS - Cryo
Resolution 3.2 Å

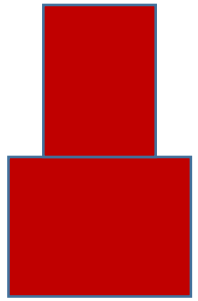


Dual Beam
Dedicated to sample preparation
Resolution 10 Å

~ 2 years



TEM 200-300 kV – Energy Filter, Phase Plate, Direct Detector, Resolution ~ 2 Å



Dual Beam – 3D Reconstruction
Dedicated to Life Sciences
Resolution 10 Å

Currently there are three corrected instruments in Brazil, none in São Paulo state.

LME- Electron Microscopy Facility for Material and Life Science

Outlook – 3 years

Materials Science

Double Corrected TEM - Holo
STEM, EDS and EELS, In-situ
Tomography, Resolution 1.0 Å

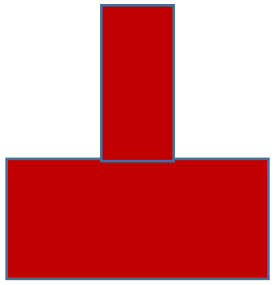
TEM JEOL JEM 2100
STEM, EDS
Resolution 2.4 Å

Dual Beam
Sample Preparation
Resolution 10 Å

SEM Inspect 50F
STEM and EDS
Resolution 10 Å

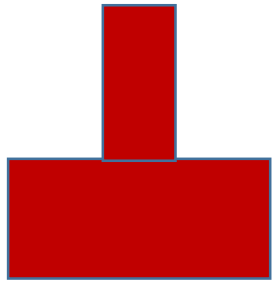
SEM Quanta 650F
EDS and EBSD
Resolution 10 Å

Dual Beam Helios 660
EDS3D and EBSD3D
Nanofabrication
Resolution 6Å

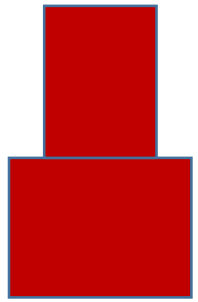


TEM 200-300 kV
Energy Filter, Phase
Plate, Direct Detector,
Resolution ~ 2 Å

TEM JEM 2100F
Phase plate
Resolution 2.4 Å



TEM 120 kV
EDS - Cryo
Resolution 3.2 Å



Dual Beam
3D Reconstruction
Resolution 10 Å

Life Sciences