

Nicholas Blanchard



Bertrand De MOORTELE

décédé, le 2 janvier 2015, à l'âge de 42 ans

Revue historique du développement du FIB : Source d'ions à métal liquide (LMIS)

Nicholas Blanchard

Retour aux sources : de la pluie à la microélectronique

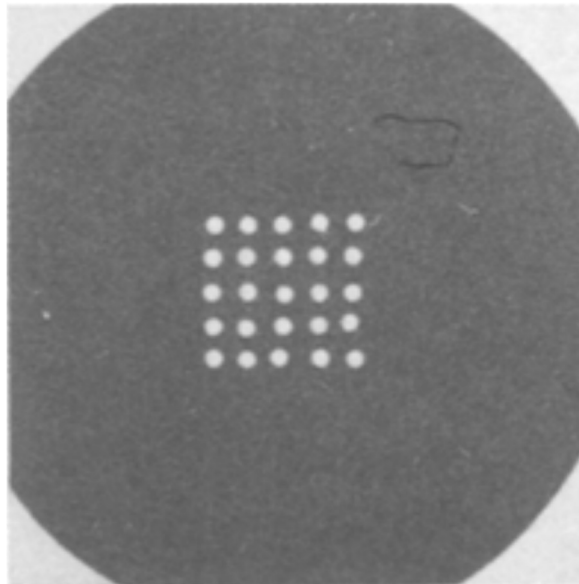
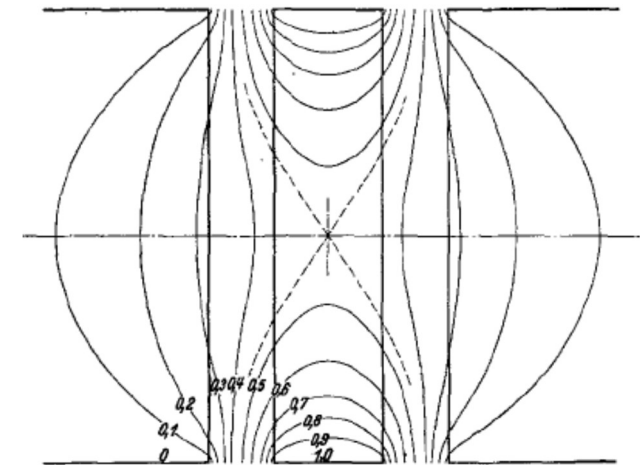
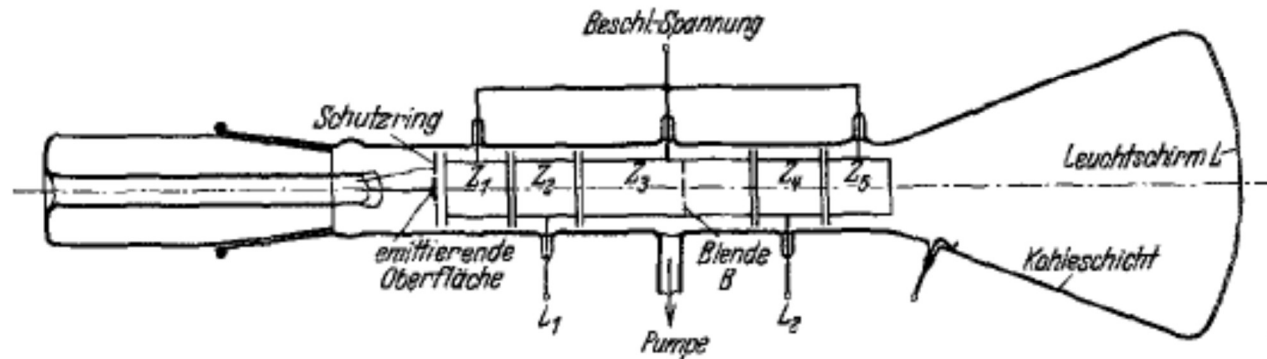
Nicholas Blanchard

Very early days...

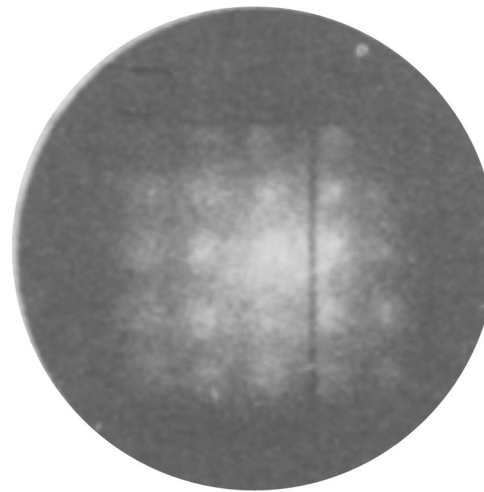
Ionenoptische Abbildungen mit elektrischen Linsen.

Von **Jørgen Koch** und **Wilhelm Walcher** in Berlin-Charlottenburg.

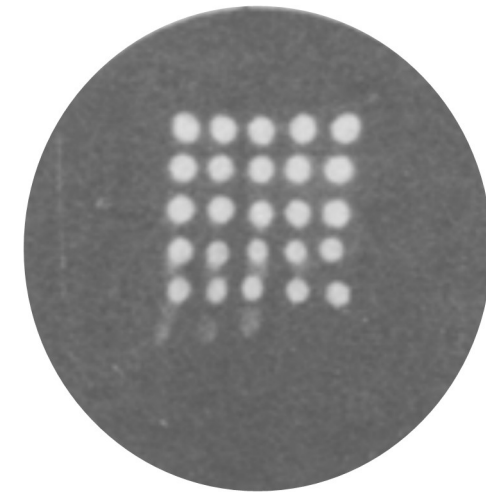
Mit 8 Abbildungen. (Eingegangen am 19. Juli 1935.)



25 hole aperture



Potassium Ion image

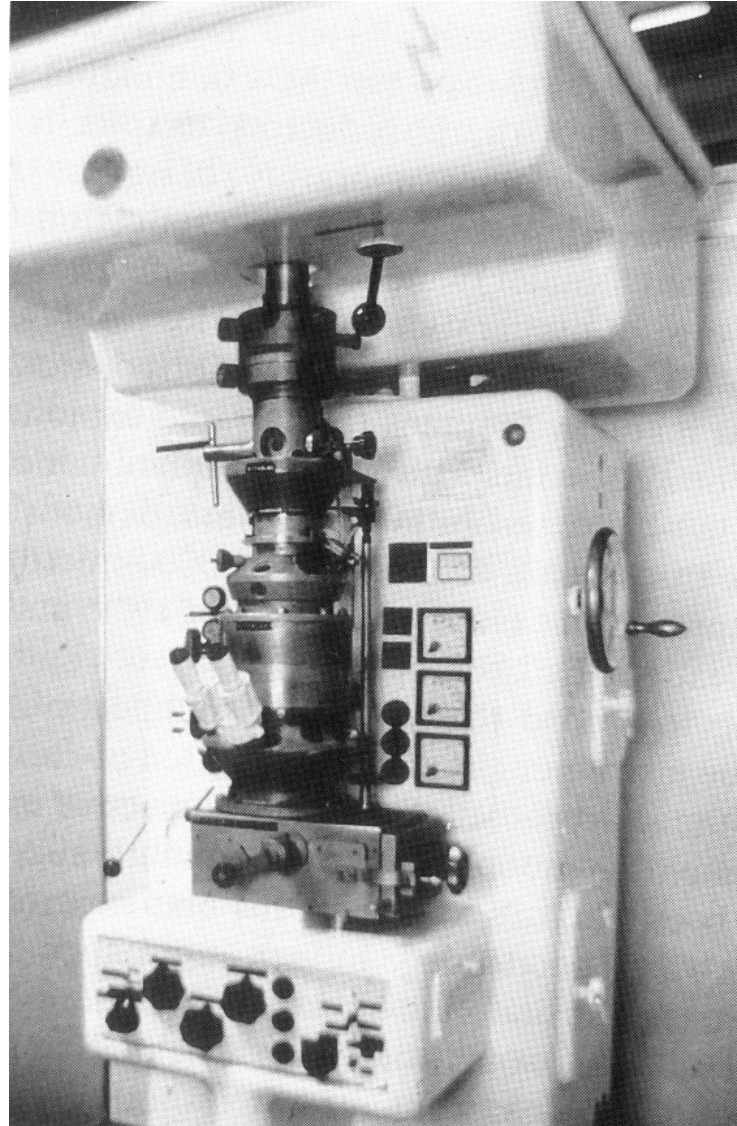


Electron image

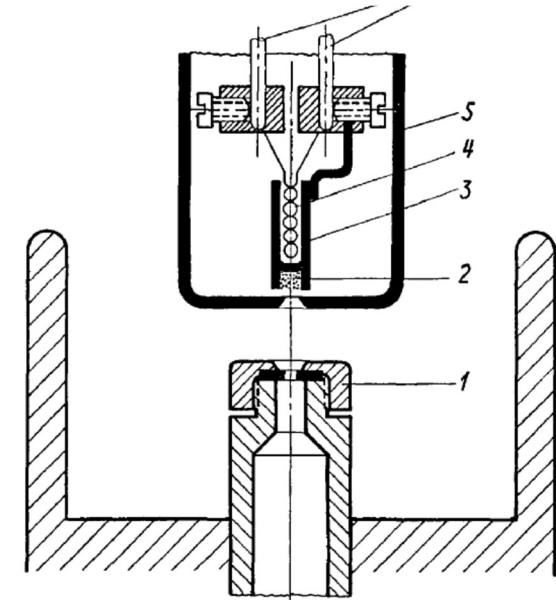
CTEM to CTIM



Hans Boersch
(1909 – 1986)



60 kV Siemens Übermikroskop



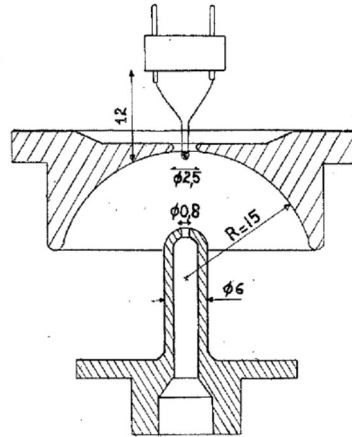
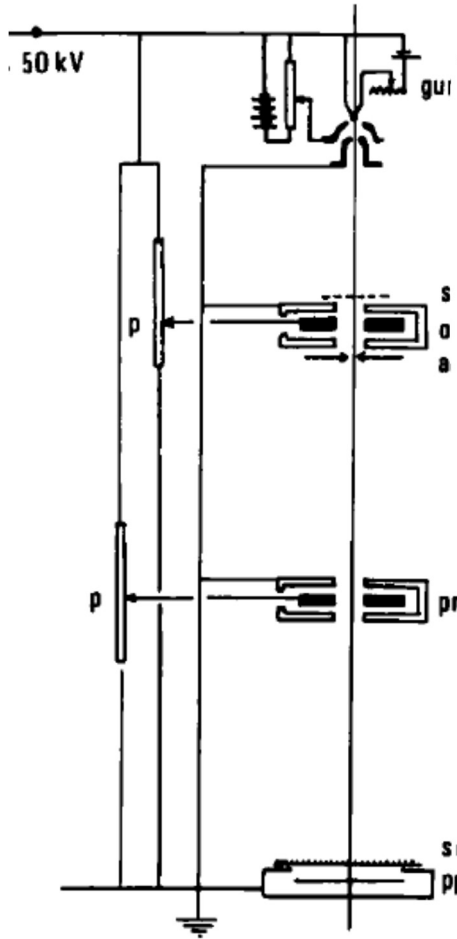
Lithium Ion Source



MgO crystals imaged with Li⁺

CTIM in France

Lithium Microscope based on a two-lens CSF microscope (Pierre Grivet) was built by **Maurice Gauzit** at the Laboratoire de Radioélectricité, Sorbonne/ENS



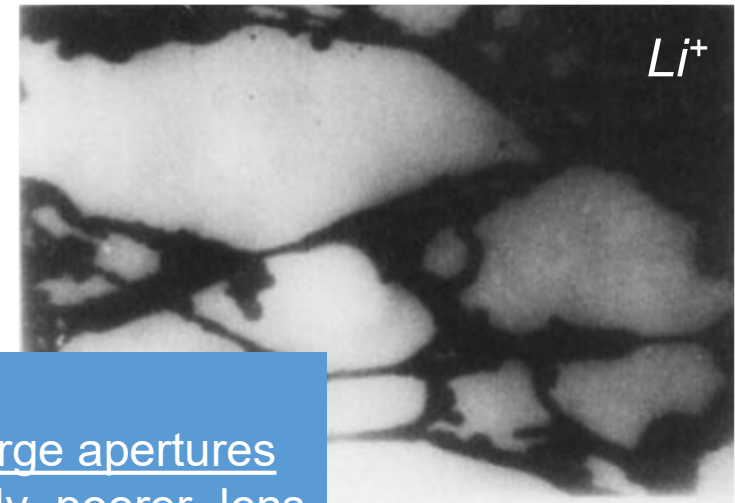
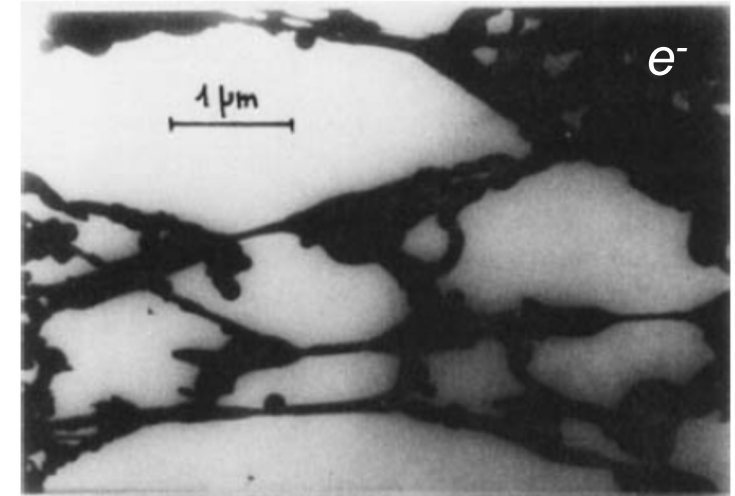
Small sphere of glassy β -eucryptite (Li_2O)

Observed “milling” of fine structures and **ion beam-induced deposition of carbon** (1 nm/min) due to the poor vacuum (10^5 torr)

Problems

- Low brightness ion source, so large apertures
- Electrostatic lens have intrinsically poorer lens properties compared to magnetic lenses
- Short IMF of ions therefore chromatic aberration

ZnO crystals

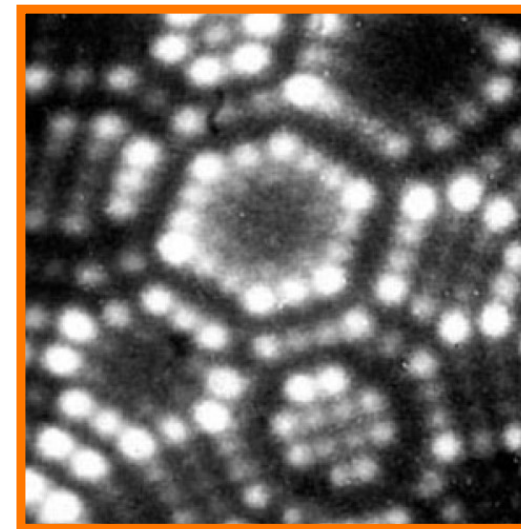


Atoms, ja, atoms...



Erwin Müller

After working on field emission in Berlin for Siemens he invented the **Field Ion Microscope (FIM)** also in Berlin before moving to the Pennsylvania State University, USA in 1951.



October 11, 1955

Erwin Müller, and his graduate student **Kanwar Bahadur**, used their FIM to resolve clearly the individual atoms in a piece of crystalline wire. For mankind this was a landmark achievement, the first convincing direct visual evidence that the atomic notion of Democritus was indeed true.



- 1911: Born June 13 in Berlin, Germany.
- 1936: Dr.-Ing from Gustav Ludwig Hertz.
- 1937: Invented the Field Emission Microscope.
- 1941: Discovered Field Desorption.
- 1950: Habilitation, Technical University Berlin
- 1951: Invented the Field Ion Microscope.
- 1952: Joined the Penn State faculty.
- 1956: First observation of individual atoms.
- 1967: Invented the Atom-Probe.
- 1975: National Academy of Science.
- 1975: National Academy of Engineering.
- 1975: National Medal of Science⁺.
- 1977: Died, May 17 of a heart attack.

Recollections of Erwin Müller's laboratory: the development of FIM (1951-1956),
Allan J. Melmed, Applied Surface Science, **94/95** (1996) 17-25

But which atoms?

14th

FIELD EMISSION SYMPOSIUM

June 26-30, 1967

National Bureau of Standards
Gaithersburg, Maryland

and

Georgetown University
Washington, D. C.

ERWIN W. MÜLLER

1975 Dr. honoris causa, Claude-Bernard University of Lyon
Honorary member, Indian Vacuum Society
Elected member, National Academy of Engineering
Elected member, National Academy of Sciences

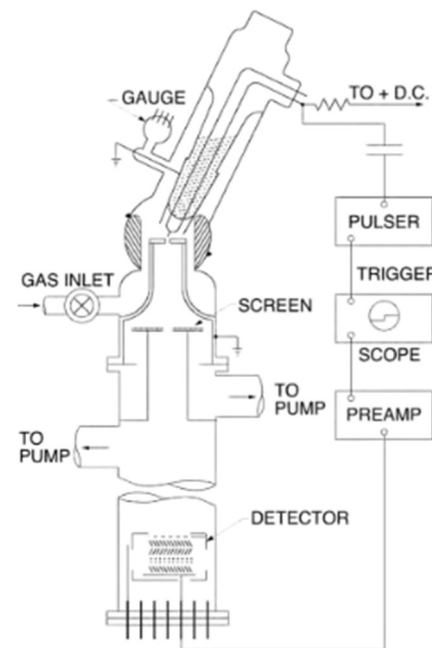
The Atom-Probe Field Ion Microscope

Erwin W. Müller and John Panitz

Physics Department, The Pennsylvania State University

University Park, Pa.

A serious limitation of the field ion microscope has been its inability to identify individually imaged atoms. A newly conceived Atom-Probe FIM, consisting of a combination probe hole FIM and mass spectrometer having single particle



Scanning Transmission Ion Microscope (STIM)

Scanning Transmission Ion Microscope with a Field Ion Source

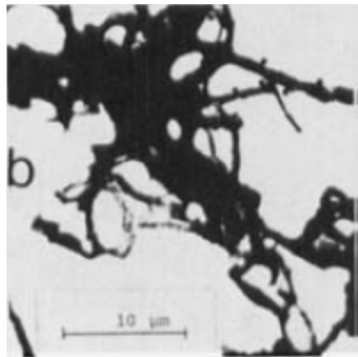
(ion optics/field ionization/microradiography)

W. H. ESCOVITZ, T. R. FOX, AND R. LEVI-SETTI

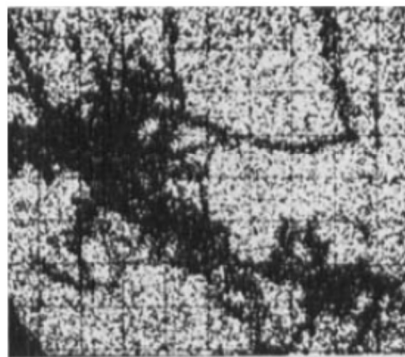
The Enrico Fermi Institute and Department of Physics, The University of Chicago, Chicago, Illinois 60637

Communicated by Albert V. Crewe, February 24, 1975

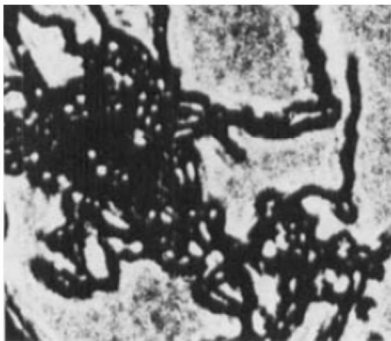
TEM



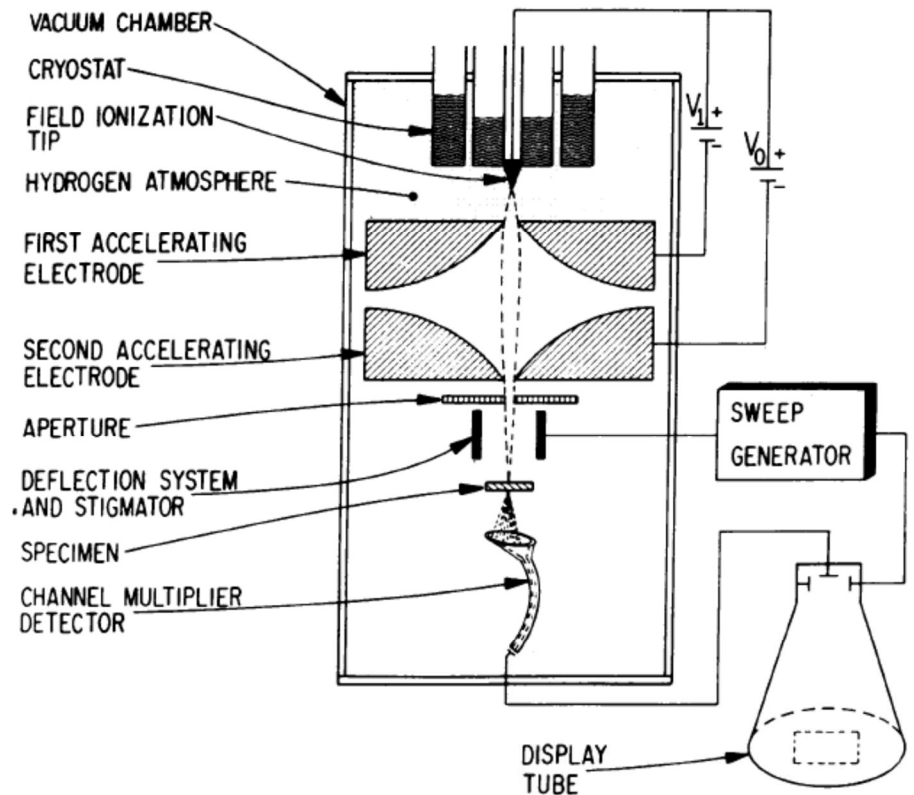
STIM



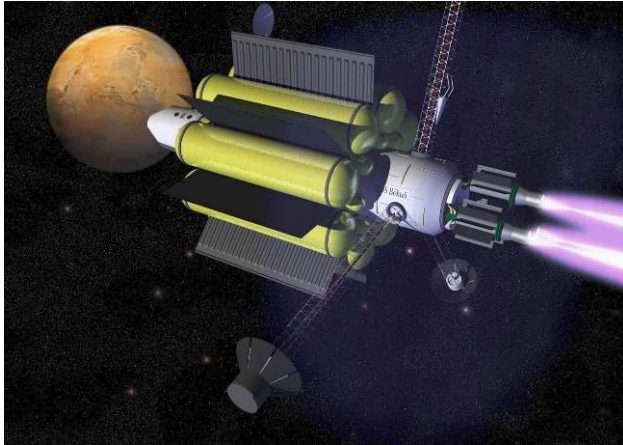
OM



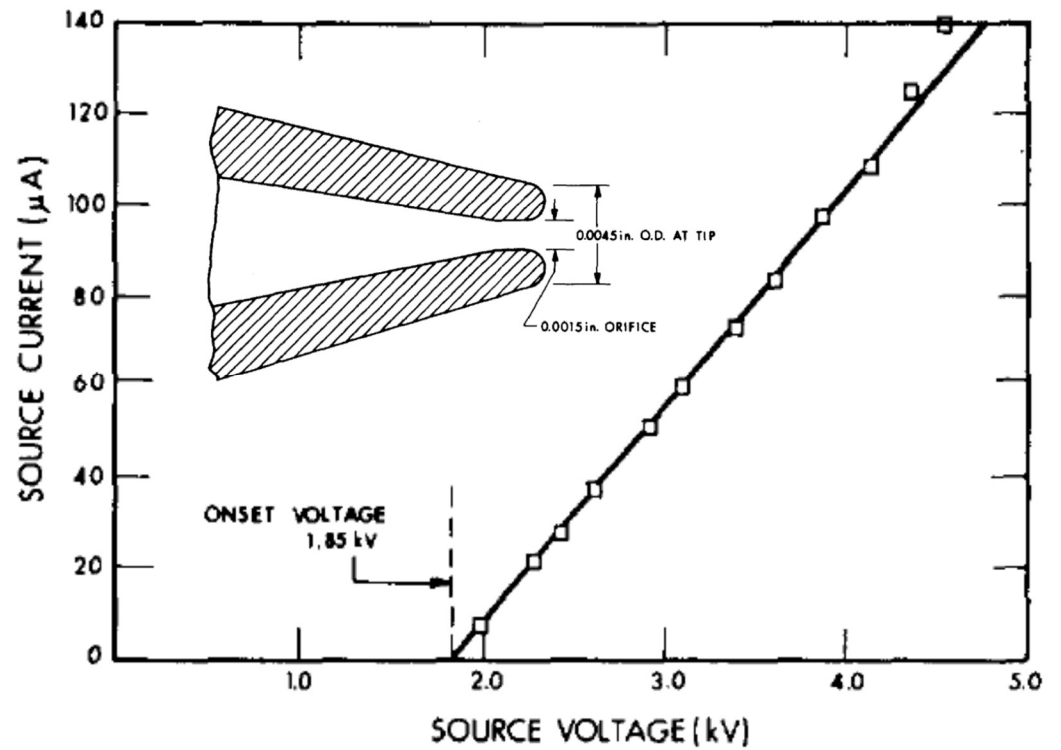
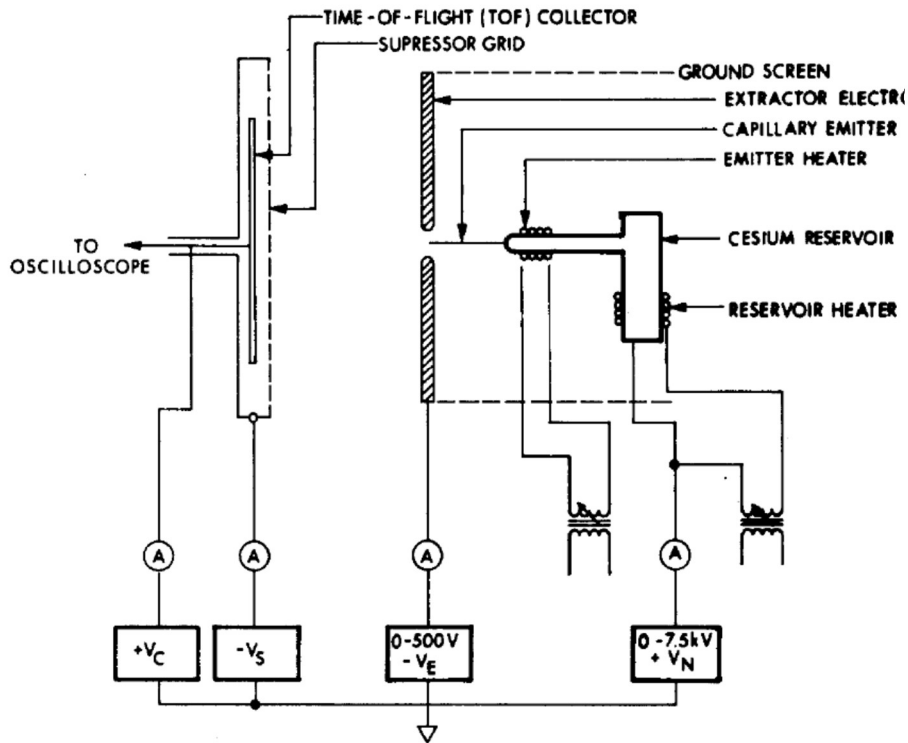
Biological sample:
Microfibrils



Space Propulsion



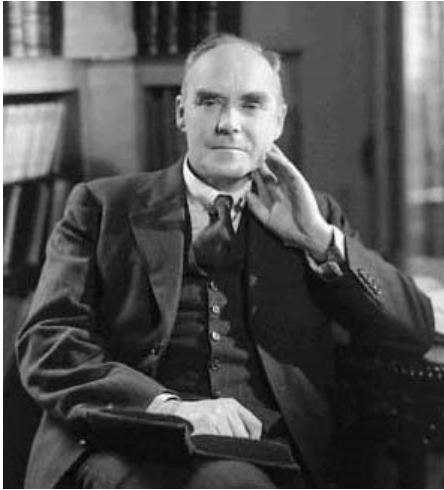
John F Mahoney and Julius Perel developed a Cs EHD source at Electro-Optical Systems (Xerox subsidiary), Pasadena, California USA during the 1960's. The work was partly funded by the Air Force Aero Propulsion Laboratory



*Electrohydrodynamic Ion Source, J.F. Mahoney et al, J. Appl. Phys. **40** (1969)*

5101-5106

My tailor is rich....

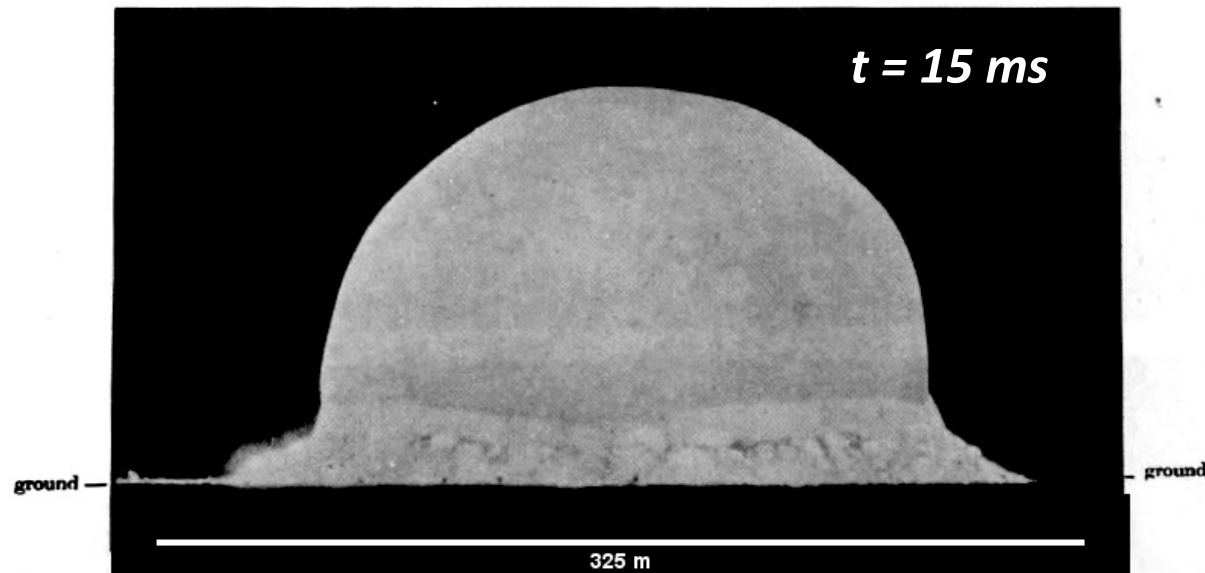


Sir Geoffrey TAYLOR
(1886 – 1975)

Taylor was the grandson of George Boole, the inventor of Boolean logic.

He was sent to the United States as part of the British delegation to the Manhattan project between 1944 and 1945. In 1944 he also received his knighthood.

His overriding interest in the movement of air and water, and by extension his studies of the movement of unicellular marine creatures and the weather, were related to his lifelong love of sailing.



Photograph by J. E. Mack of the first atomic explosion in New Mexico

The Formation of a Blast Wave by a Very Intense Explosion. II. The Atomic Explosion of 1945,
G. Taylor, Proc. R. Soc. Lond. A **201**, (1950) 175-186

Taylor's mathematical cone

"The disintegration of drops in strong electric fields is believed to play an important part in the formation of thunderstorms"



"It is shown theoretically that a conical interface between two fluids can exist in equilibrium in an electric field, but only when the cone has a semi-vertical angle 49.3°"

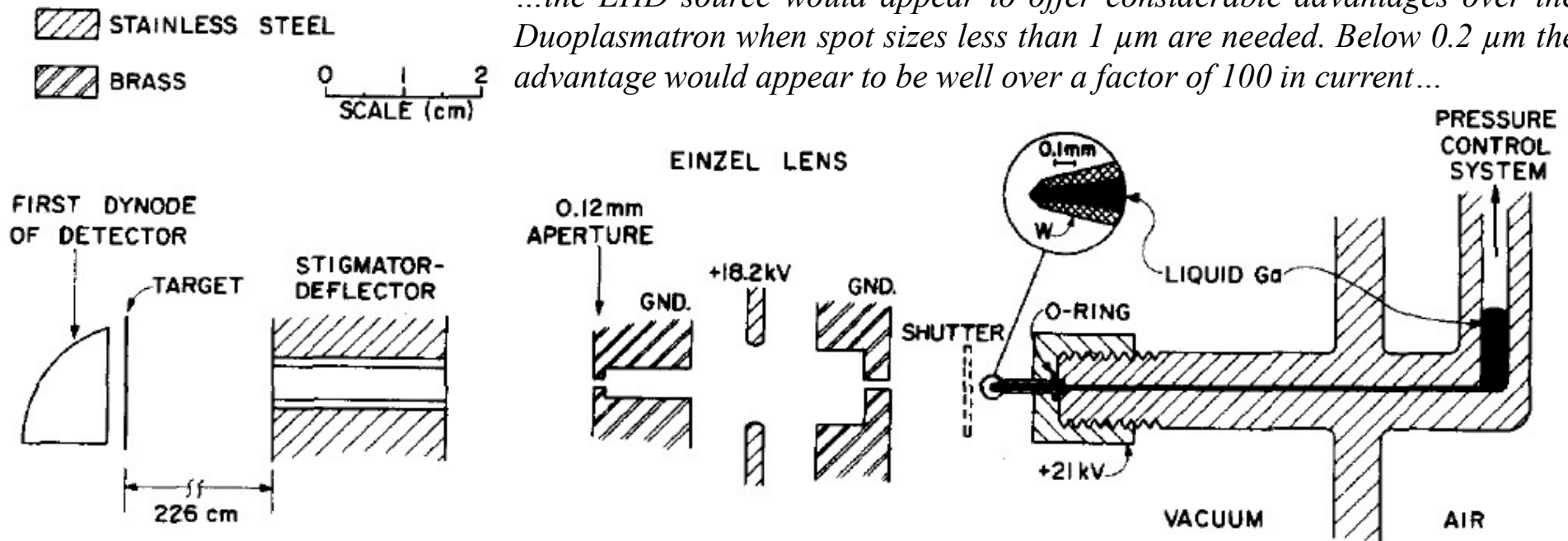


Oil/water interface

In conclusion I express my thanks to **Professor C. W. Oatley** for lending apparatus and to Mr A. D. MacEwan for help in the design of the apparatus and in carrying out the experiments.

Ion Microprobe (SIMS)

Victor E. Krohn and G.R. Ringo working at the Argonne National Laboratory, Argonne, Illinois, USA developed a Ga EHD source for use in their ion microprobe (SIMS), however they mentioned micro-machining, micro-implantation as additional applications

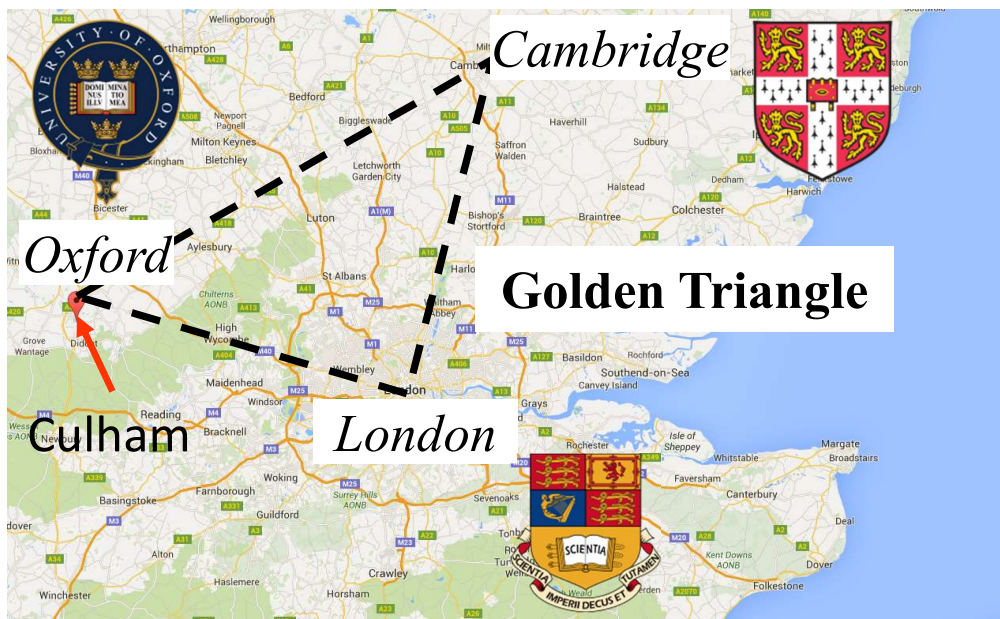


...the EHD source would appear to offer considerable advantages over the Duoplasmatron when spot sizes less than 1 μm are needed. Below 0.2 μm the advantage would appear to be well over a factor of 100 in current...

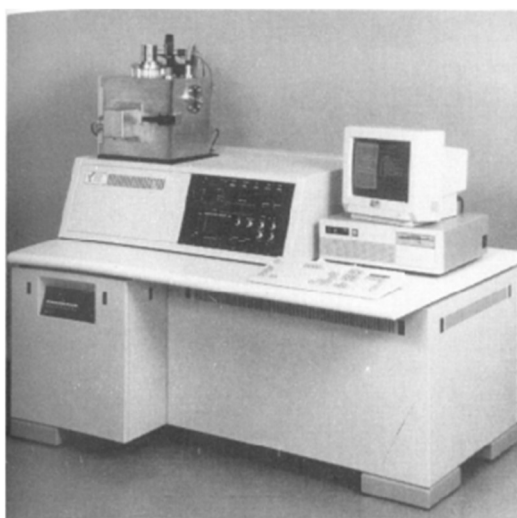
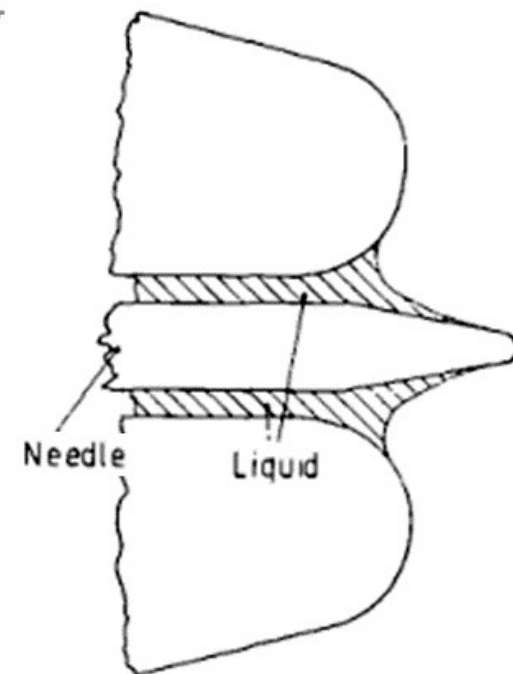
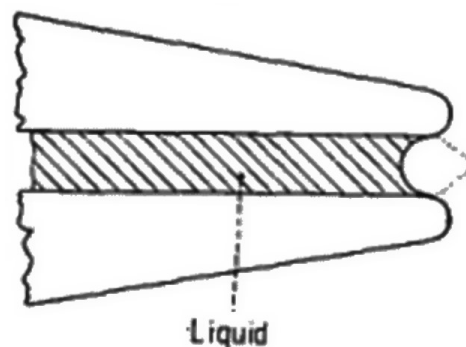
In the conclusion they wrote:

...More radical changes should also be considered. For example, the very sharp tungsten needles used by Clampitt and Jefferies [Second Workshop on Electric Propulsion and its Space Application, Toulouse, France, 1972] with the liquid metal, applied externally, migrating to the tip in the form of a film...

UKAEA, Culham



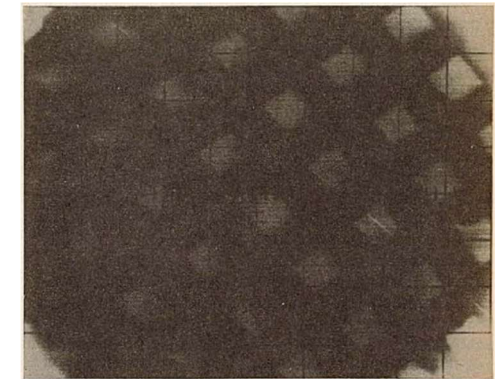
Roy Clampitt in the UK, introduced the idea of using a tungsten needle wetted by the liquid metal to anchor the field emission, rather than trying to form a Taylor cone in the liquid metal itself.



Miniature Ion Source for analytical instruments, R. Clampitt and D.K. Jefferies, *Nuc. Inst. Meth.* **149** (1978) 739-742

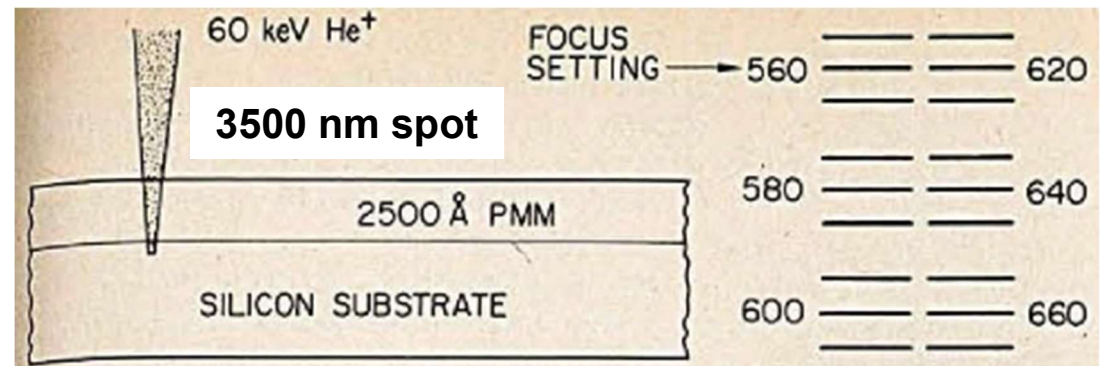
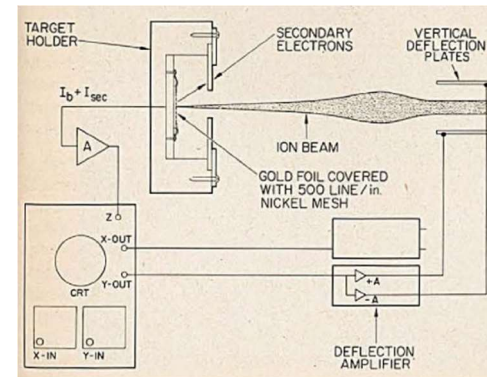
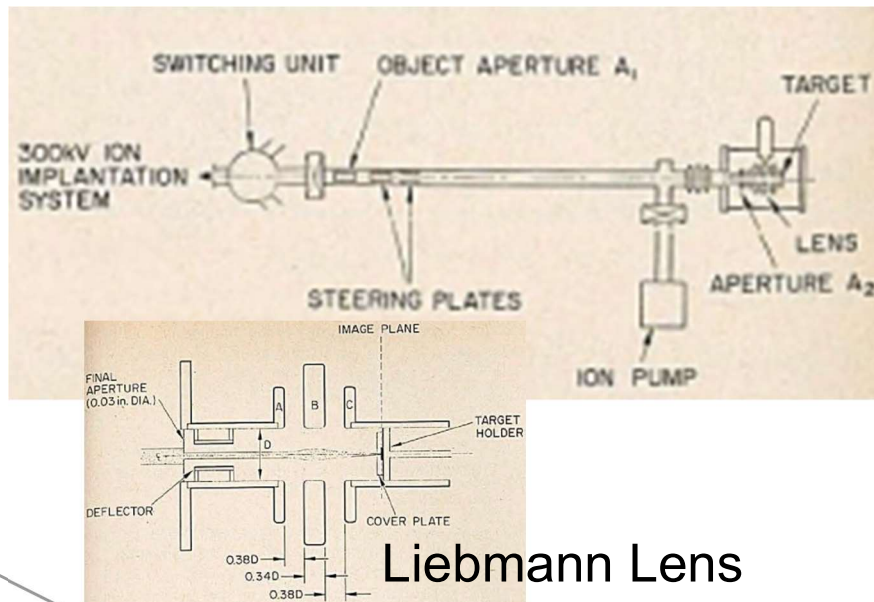
Characteristics of a Ga liquid metal field emission ion source, P.D. Prewett and D.K. Jefferies, *J. Phys. D: Appl. Phys.* **13** (1980) 1747-1755

Malibu, California



SE image of a Ni grid with scanned primary ions

Maskless doping of semiconductors

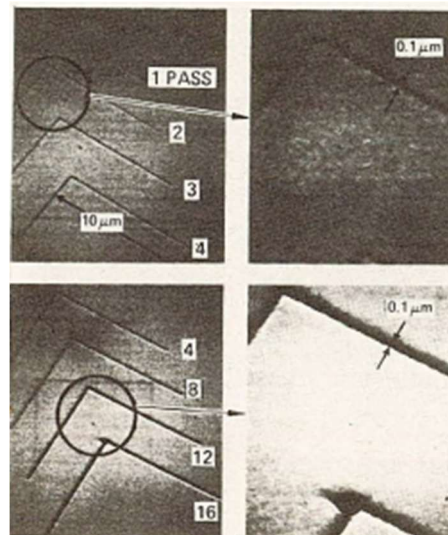
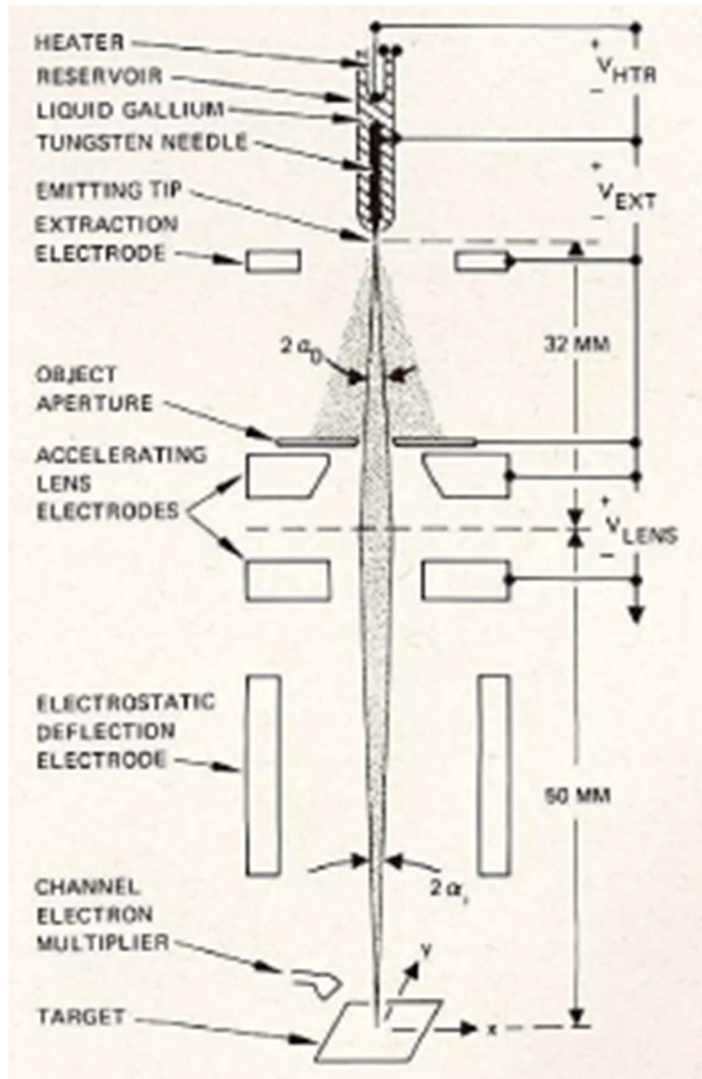


Focused ion beams in microfabrication, Robert L. Seliger and W. P. Fleming, J. Appl. Phys. **45** (1974) 1416

- Ga LMIS (Culham design)
- Single-gap accelerating lens
- Post lens deflector

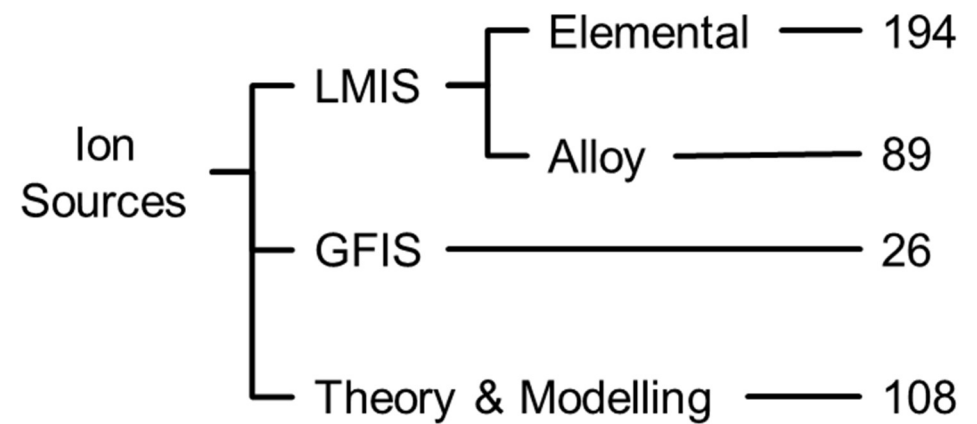
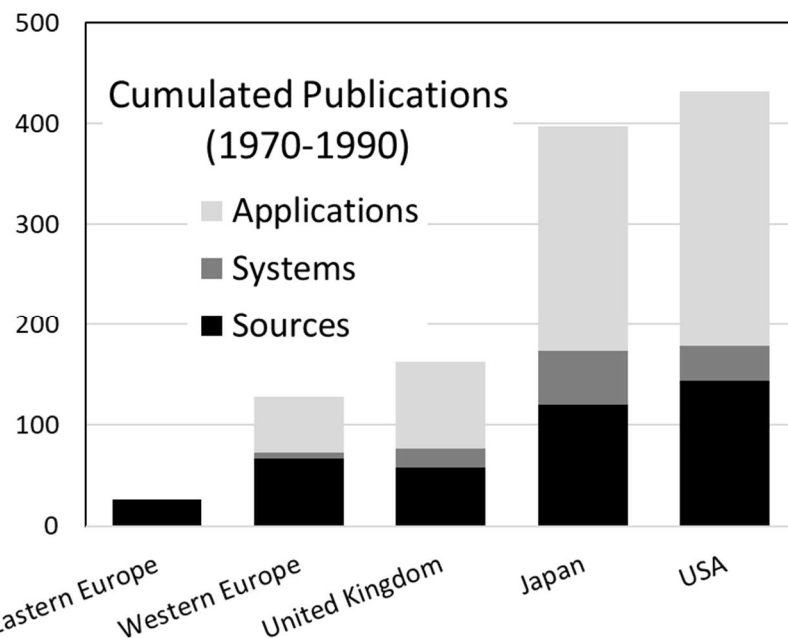
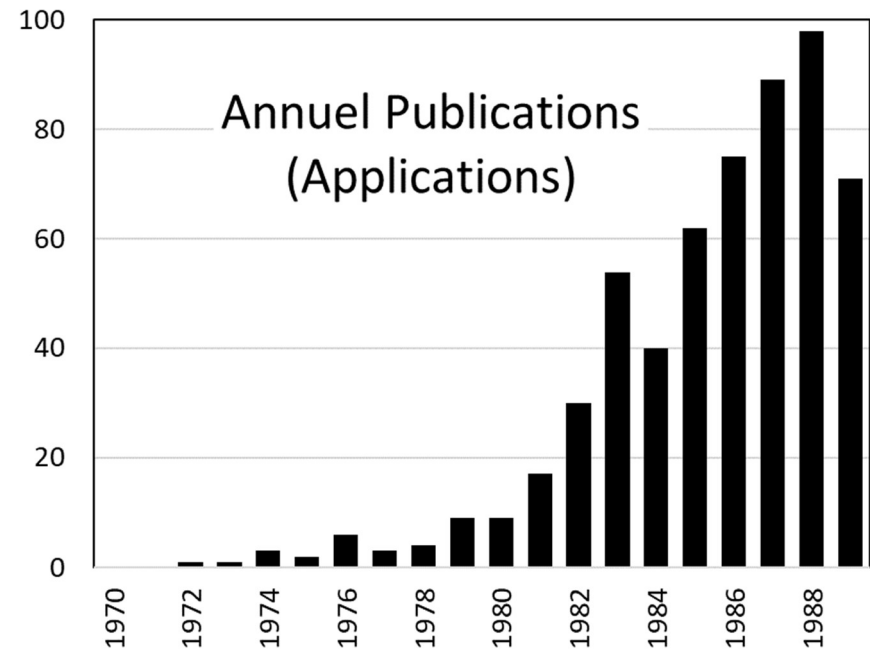
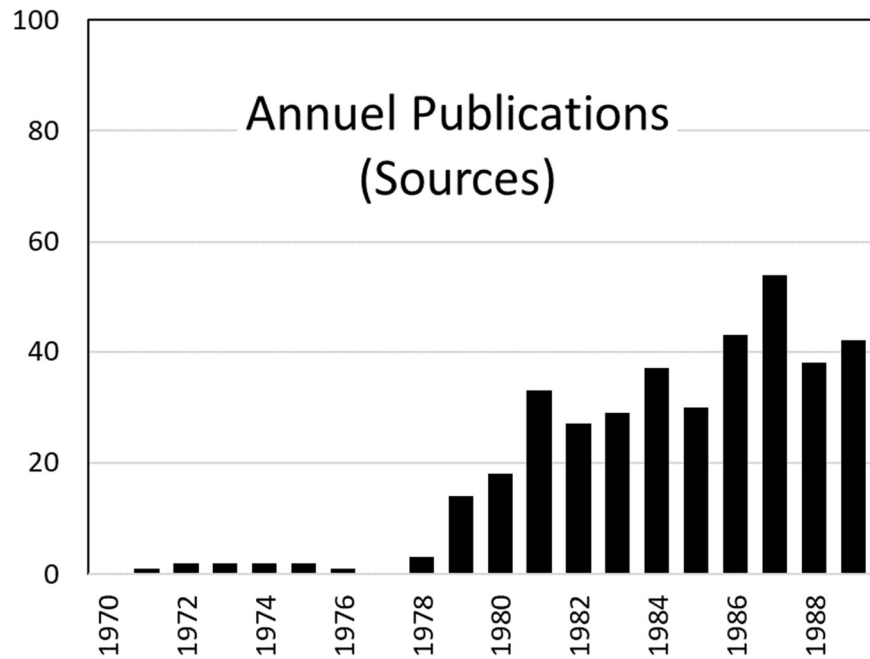
Primary Ga ion energy: 57 keV, I_{emission} : 10 μA

Probe Diameter	Probe Current
100 nm	120 pA
500 nm	3 nA

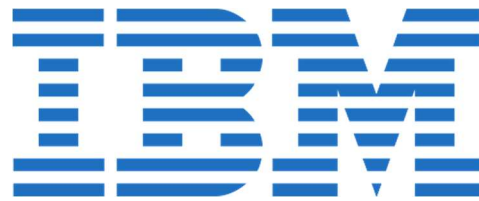
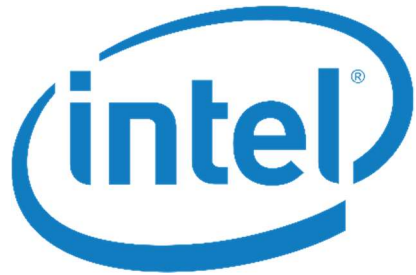


Lines etched into a 40 nm thick Au layer on silicon (SEM micrographs)

Tipping point...



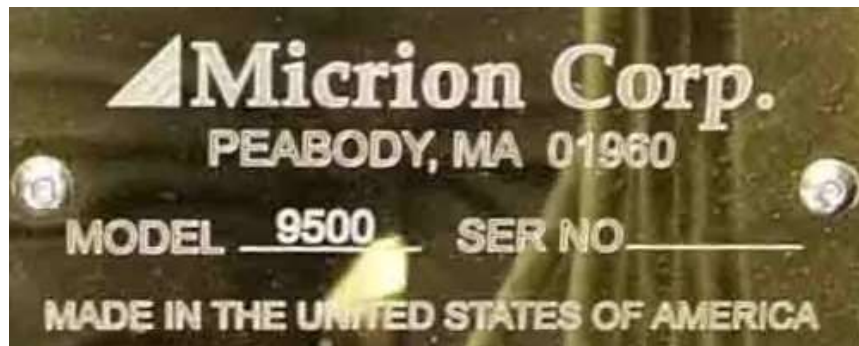
Semiconductor industry



MOTOROLA

- 1984, **IBM** started work on mask repair
- 1985, **Motorola** worked on maskless implantation
- 1985, **Hitachi** pioneered integrated circuit cross-sectioning
- 1990's Crow et al at **Intel** pursued the use of FIB in device modification and TEM sample preparation

HITACHI
Inspire the Next



1985

FIB for commercial use in mask repair
and IC production failure analysis

In **1986**, the number of FIB fabrication systems totalled about **35 (25 of them in Japan)**; in **1998**, manufacturers of FIB systems estimated the number of such systems to be between **520 to 870**.

Manufacturers producing FIB instruments at the end of the 1990's:
FAI, FEI/Philips, Hitachi, JEOL,
Metron/Seiko, Micrion, NANO-FAB, and
Schlumberger.



Oregon Graduate Center

1963 - 2001

Private, postgraduate-only
research university in
Beaverton-Hillsboro, Oregon,
U.S.A



\$2M grant from
the Tektronix
Foundation



THEN



NOW

Lynwood W. Swanson, a professor of Applied Physics and Electrical Engineering at OGC, co-founded the Field Electron and Ion Company in 1971, as an extension of his research in **focused ion beams (FIB)** and **liquid metal ion sources (LMIS)**. His PhD student **Jon Orloff** joined the board of FEI in 1978.

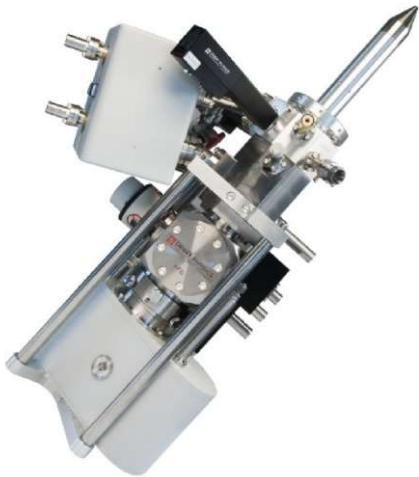


FEI™

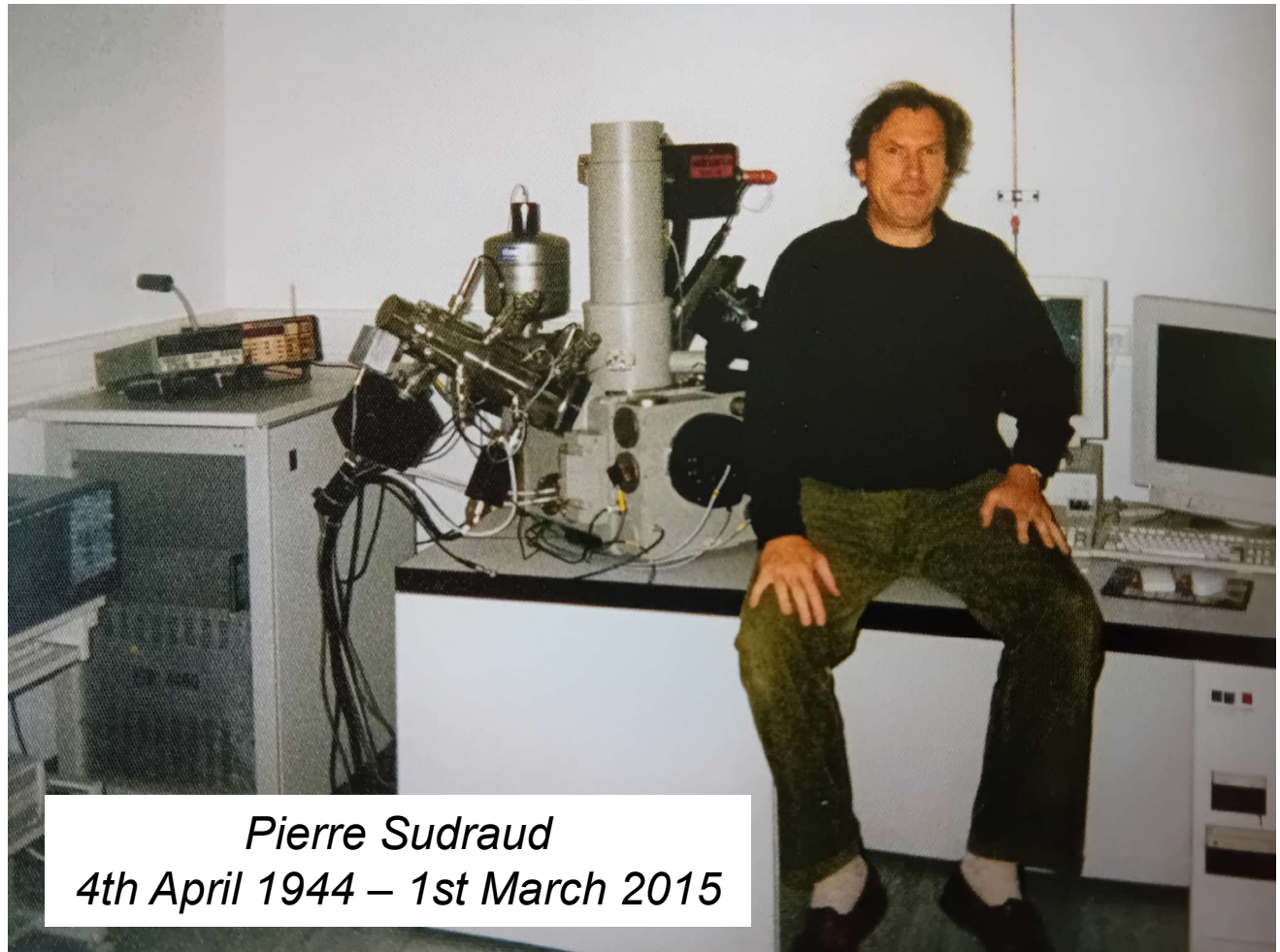


La Ferme du Moulon
Chemin du Moulon
91190 Gif / Yvette, France
Tel: (1) 60 19 09 13

*High Technology Consulting,
R&D, Diffusion of Scientific Instruments for
Microscopy, Microanalysis and Microfabrication*



1989 : Company founded by **Pierre Sudraud**, from Orsay-Paris University



Pierre Sudraud
4th April 1944 – 1st March 2015

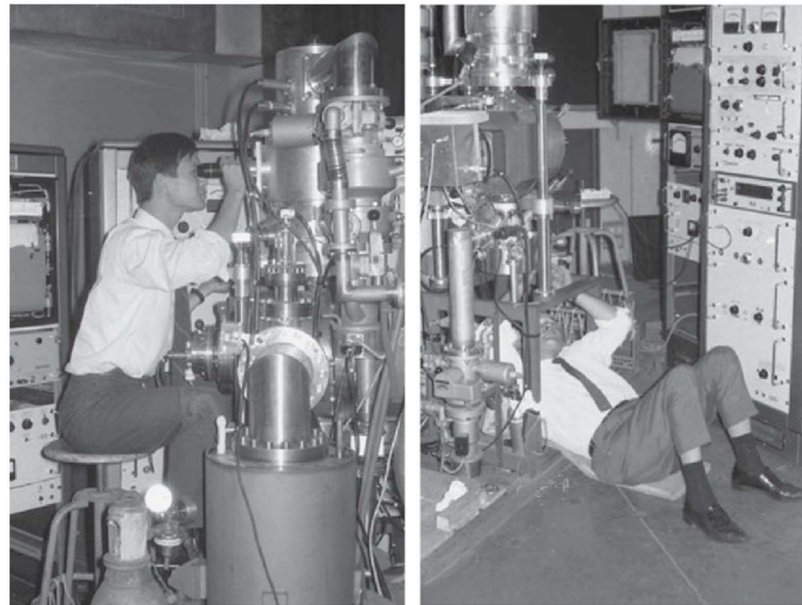
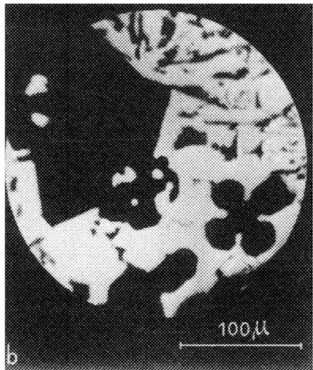
1990: World's first FIB/SEM developed for IBM

1997: Relocated to Fuveau, Bouches-du-Rhône

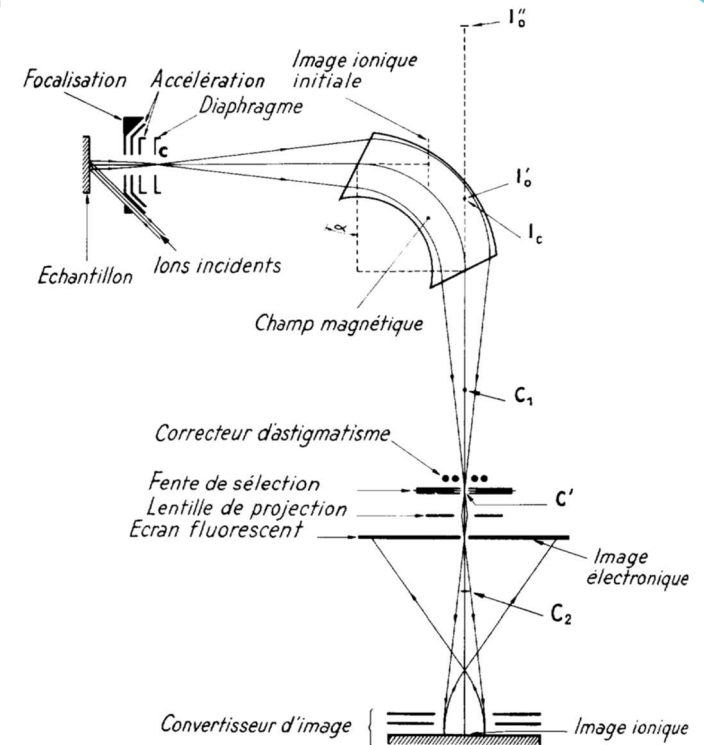
Castaing's legacy



Raymond Castaing
(1921 – 1998)



Cameca replica of Castaing's ion microscope (1965)



1962 Slodzian & Castaing
SIMS

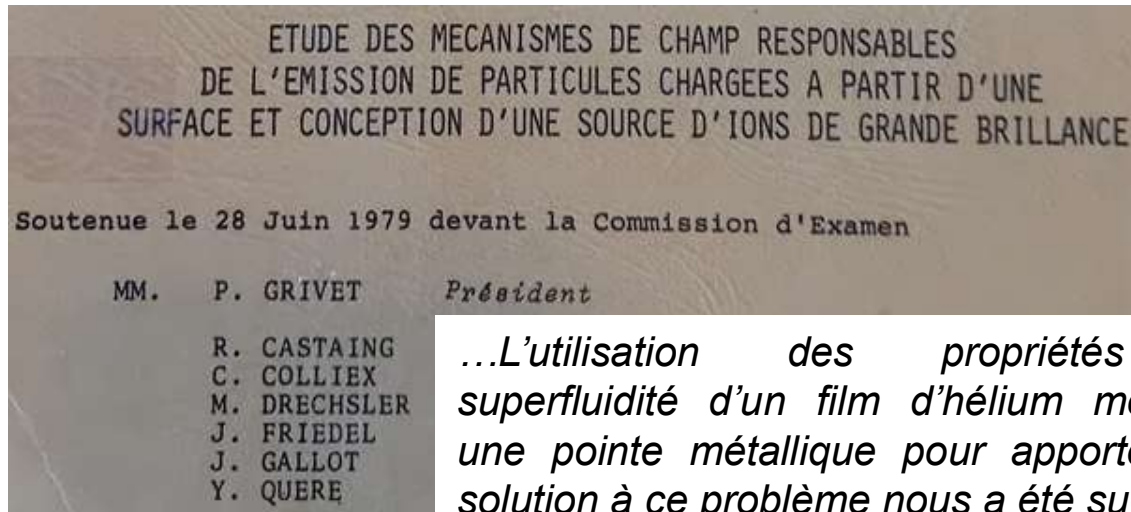
R Castaing and G Slodzian, *Microanalyse par émission ionique secondaire*, *J. Microscopie*, **1** (1962) 395-410

we go?" Sarfati asked. In February 1995, contacts were made with Peter Sudraud, who had just left the University of Orsay and founded Orsay-Physics. But eventually, relations between Sarfati and Orsay-Physics became strained after a contract on a liquid cesium source for SIMS did not yield the expected results.



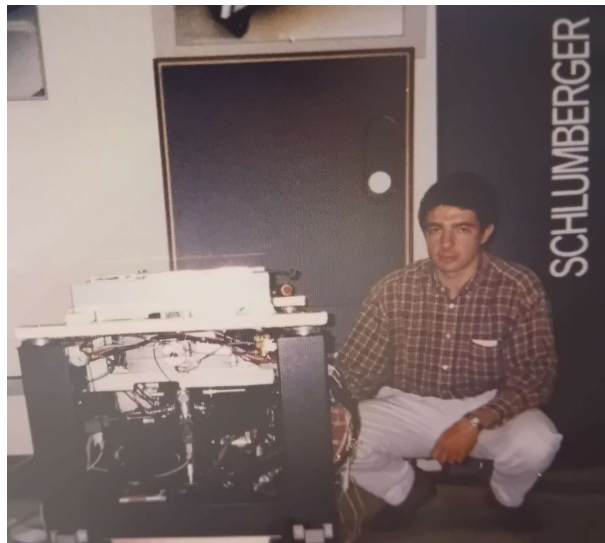
First steps...

Pierre Sudraud, Thèse d'Etat, Université Paris-Sud, Orsay, 1979.

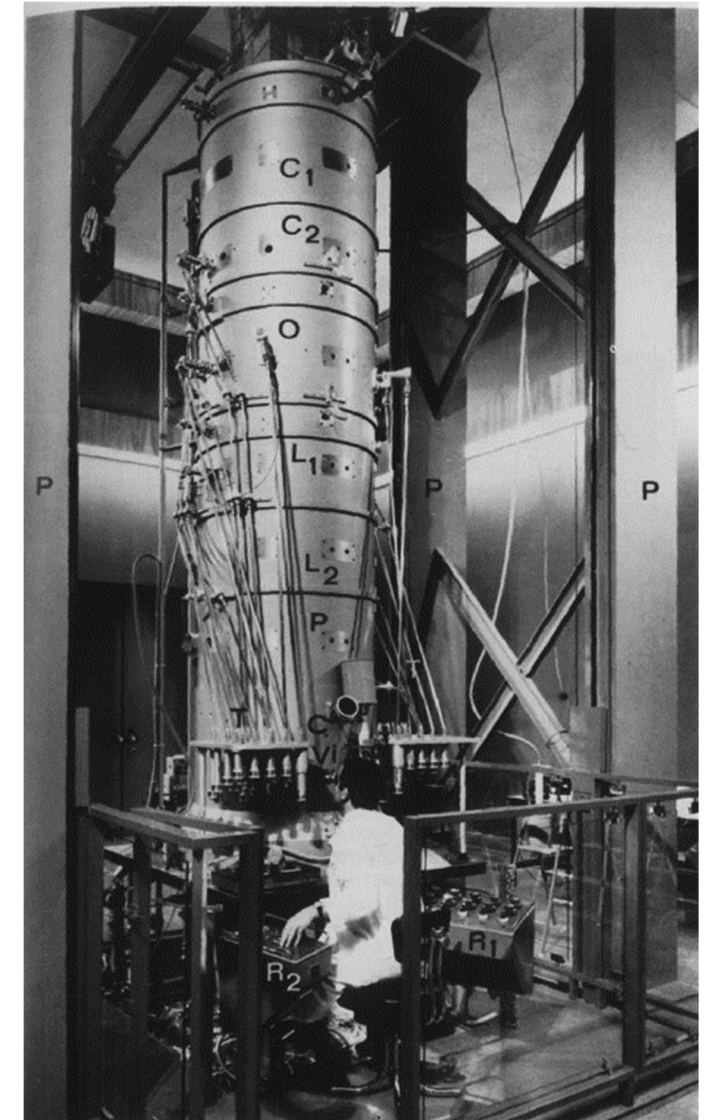
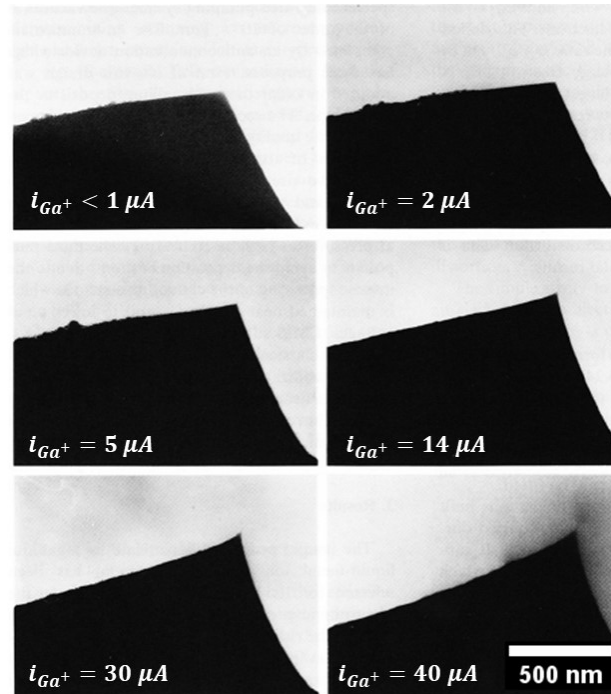


MET 3 MeV, Toulouse

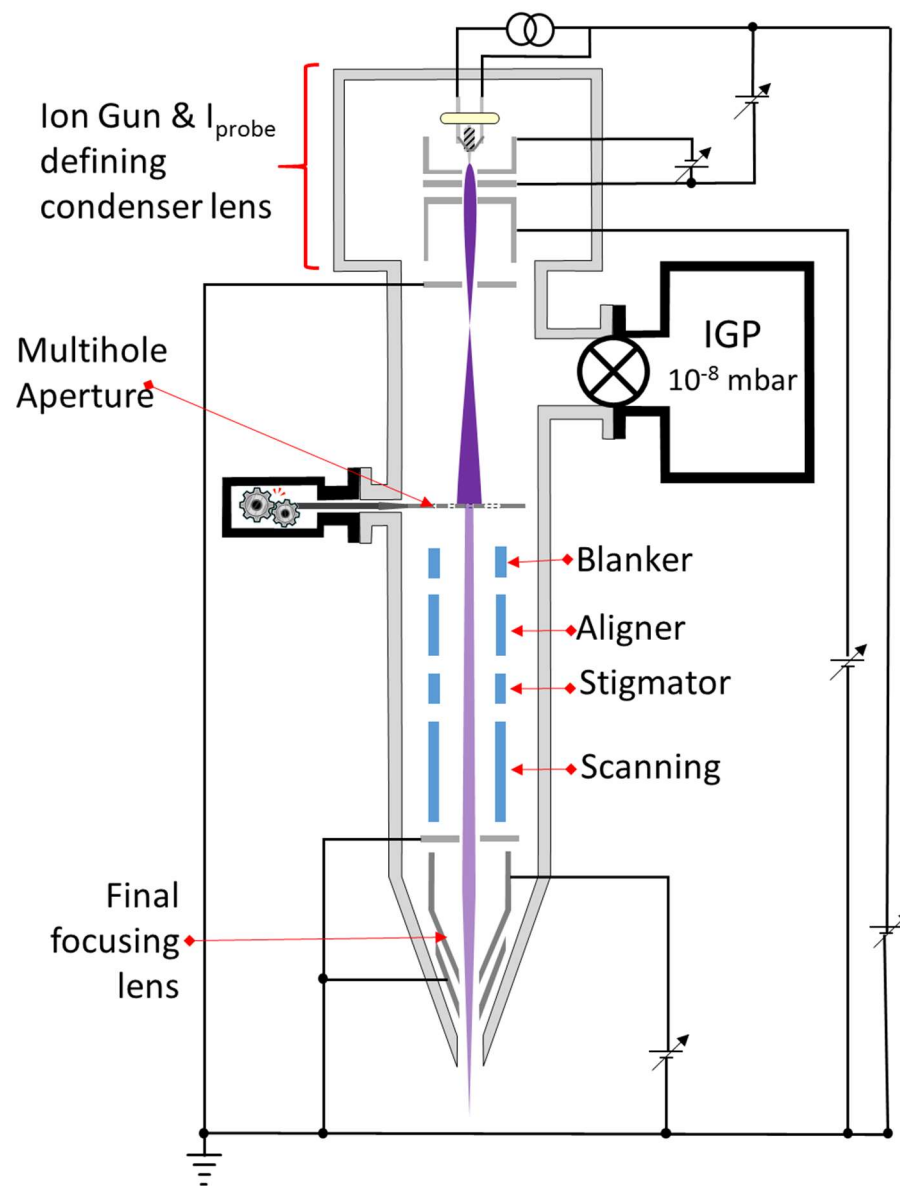
...L'utilisation des propriétés de superfluidité d'un film d'hélium mouillant une pointe métallique pour apporter une solution à ce problème nous a été suggérée par **R. CASTAING** au début de cette étude...



Antoine Corbin, Phd 1995



In situ HV-TEM observation of an EHD ion source, G Benassayag, P Sudraud, B Jouffrey, Ultramicrosc. **16** (1985) 1-8



« L'histoire, c'est la rencontre d'une volonté et d'un événement. »

Charles de Gaulle

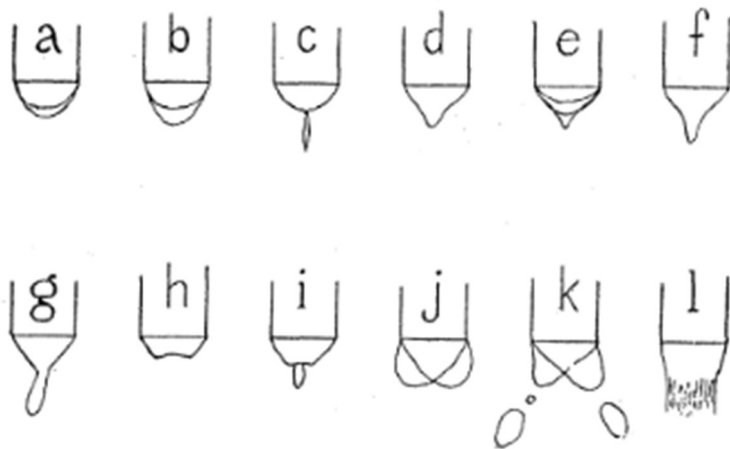
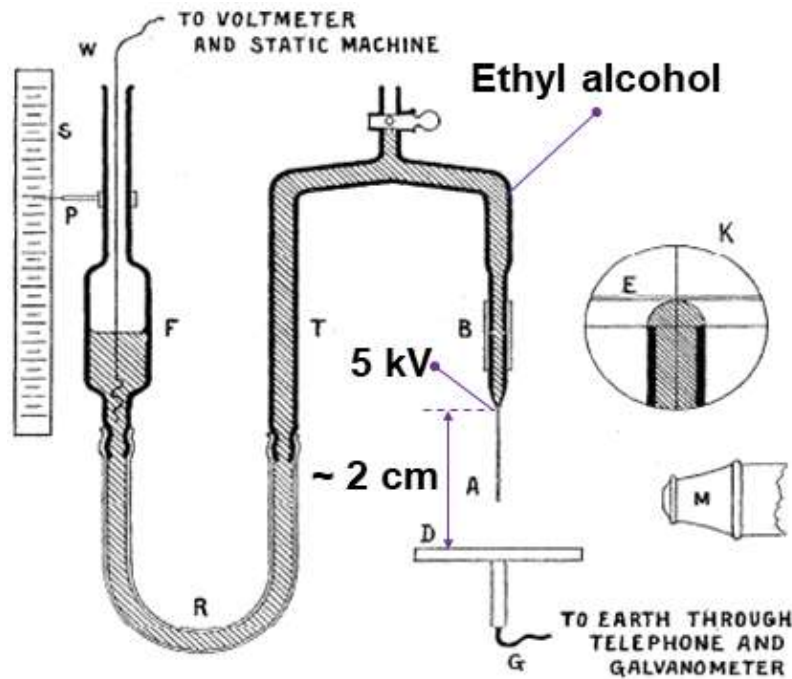
"I believe that the more you know about the past, the better you are prepared for the future."

Theodore Roosevelt

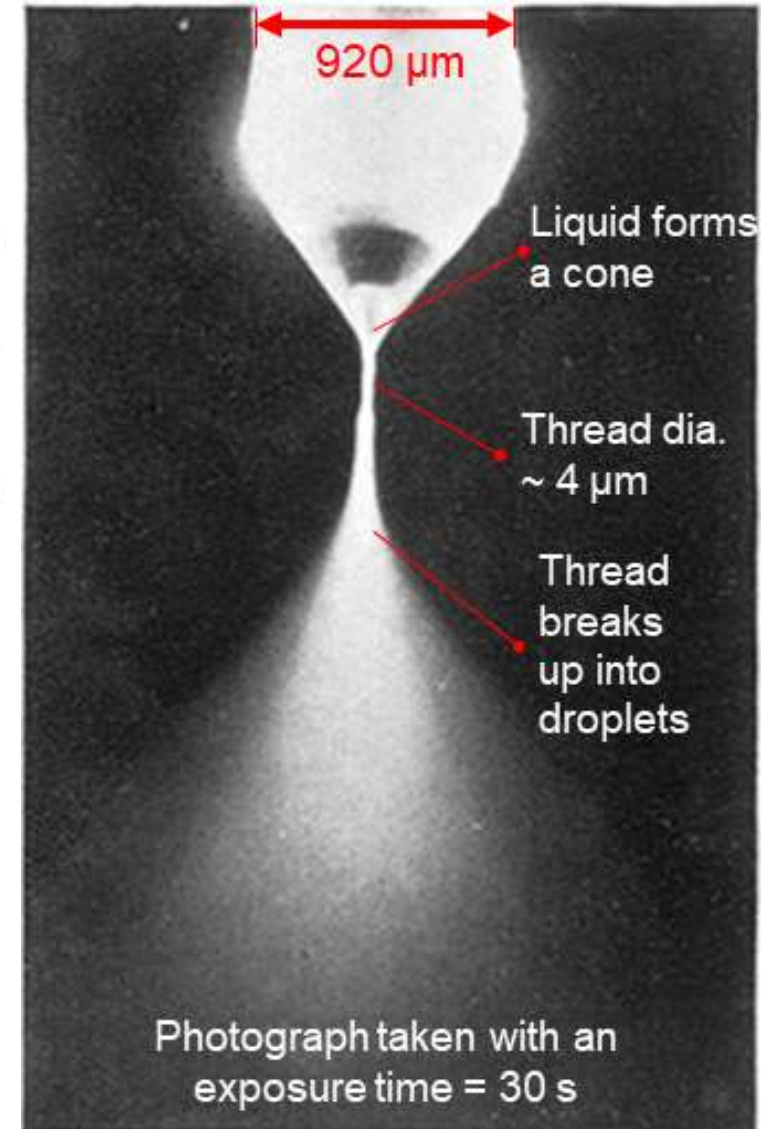
Electrosprays



John ZELNY
(1872 – 1951)
American physicist



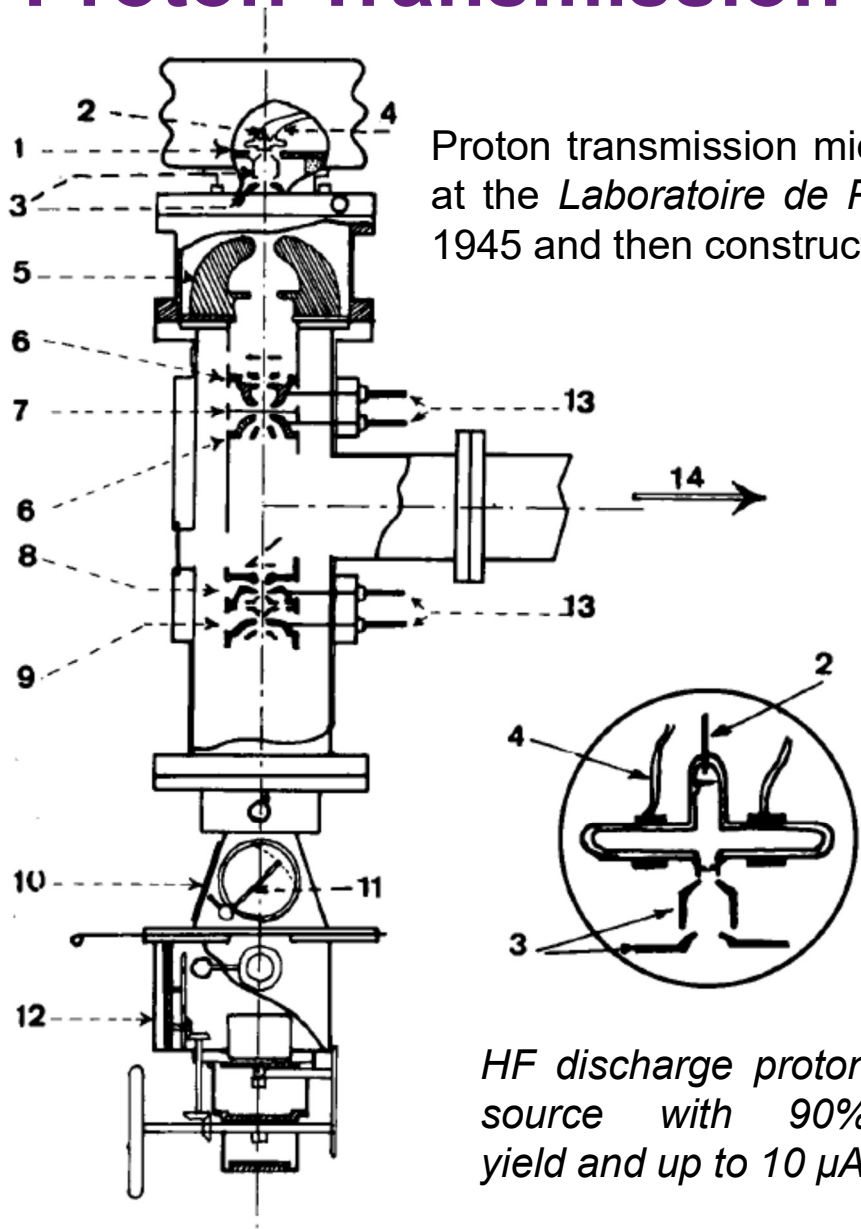
The Electrical Discharge from Liquid Points, and a Hydrostatic Method of Measuring the Electric Intensity at Their Surfaces,
J. Zeleny, *Physical Review*, **3** (1914) 69–91



Photograph taken with an exposure time = 30 s

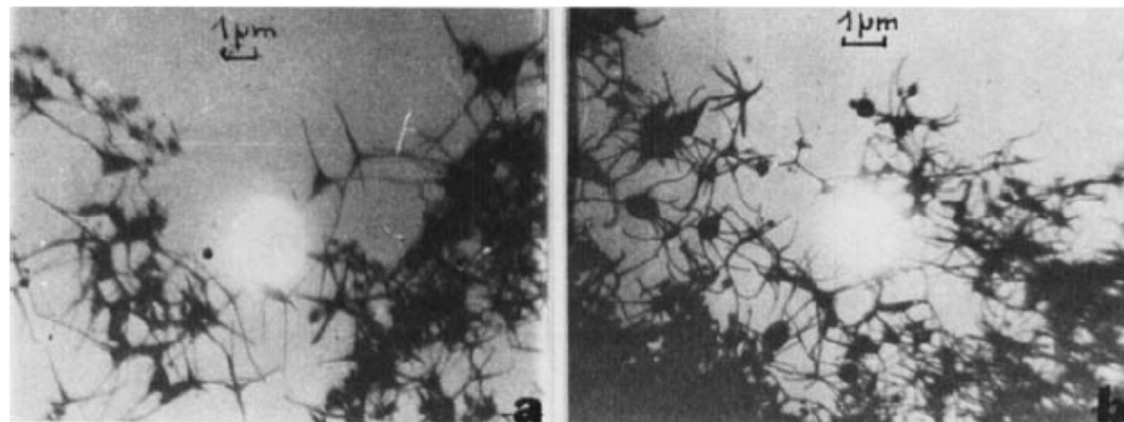
Instability of Electrified Liquid Surfaces,
J. Zeleny, *Physical Review*, **10** (1917) 1–6

Proton Transmission Microscope



Proton transmission microscope proposed by **Claude Magnan** and **Paul Chanson** at the *Laboratoire de Physique Atomique et Moléculaire du Collège de France* in 1945 and then constructed 3 instruments between 1950 and 1958. Resolution 5 nm

HF discharge proton source with 90% yield and up to 10 μ A



ZnO crystals after 3 hours of proton exposure

LE PROTON DÉTRÔNERA-T-IL L'ÉLECTRON en microscopie corpusculaire ?

Par C.-G. BOSSIÈRE
Publié le 01 décembre 1951

Le Monde

Problems

- Low brightness ion source, so large apertures
- Electrostatic lens have intrinsically poorer lens properties compared to magnetic lenses
- Short IMF of ions therefore chromatic aberration

5, 4, 3, 2, 1... Lift-out



Dr Lucille Giannuzzi



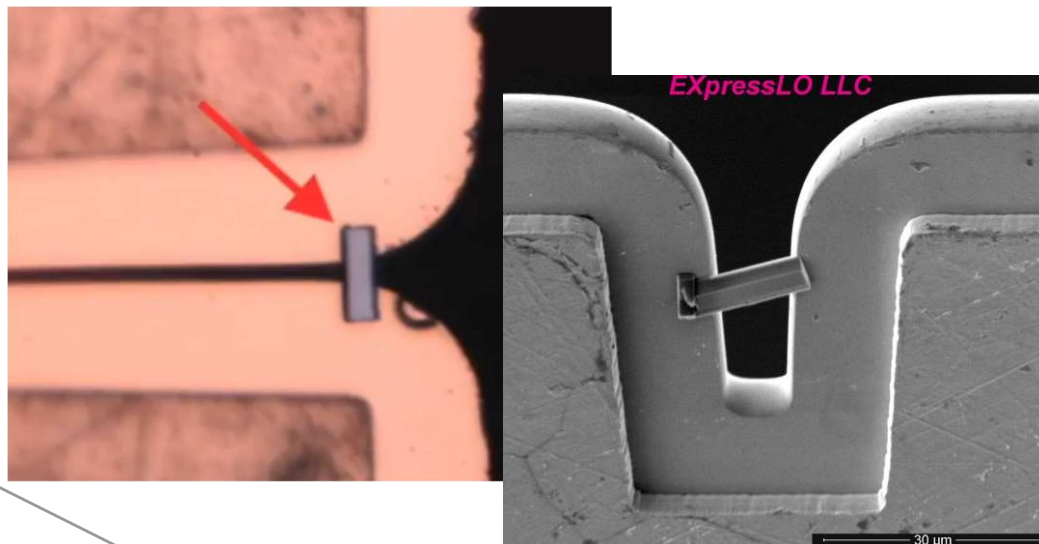
Introduction to Focused Ion Beams

Instrumentation, Theory,
Techniques and Practice

Edited by
Lucille A. Giannuzzi
Fred A. Stevie



 Springer

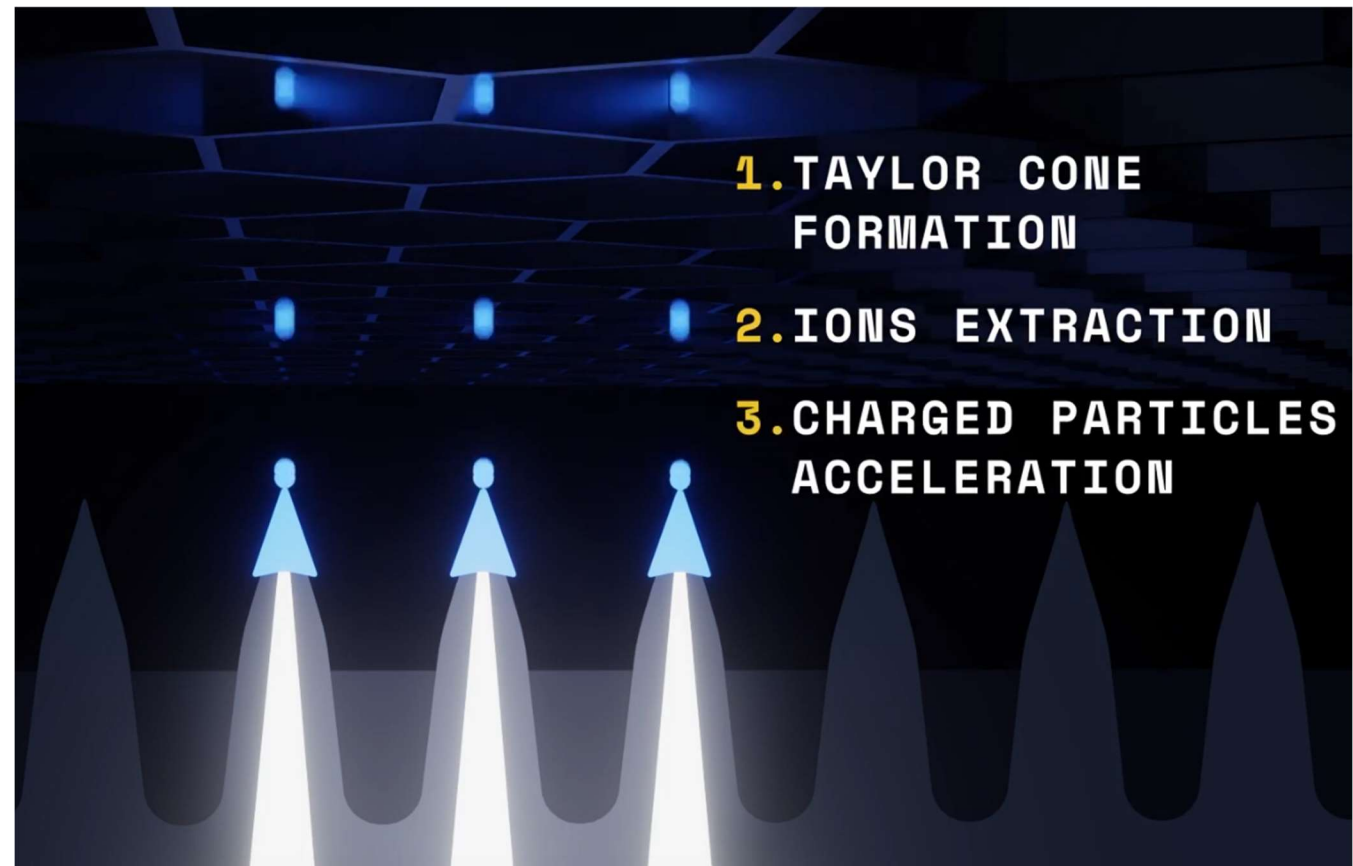
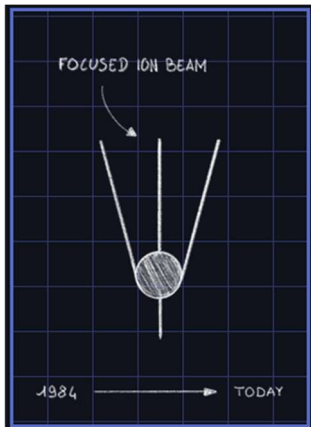


A review of focused ion beam milling techniques for TEM specimen preparation, L.A. Giannuzia & F.A. Stevie, *Micron* **30** (1999) 197–204

Médaille de l'innovation 2023 du CNRS : Jacques Gierak récompensé



©Frédérique Plas / CNRS Images



<https://ion-x.space/technology/>