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Focussed beams for use in EBSD

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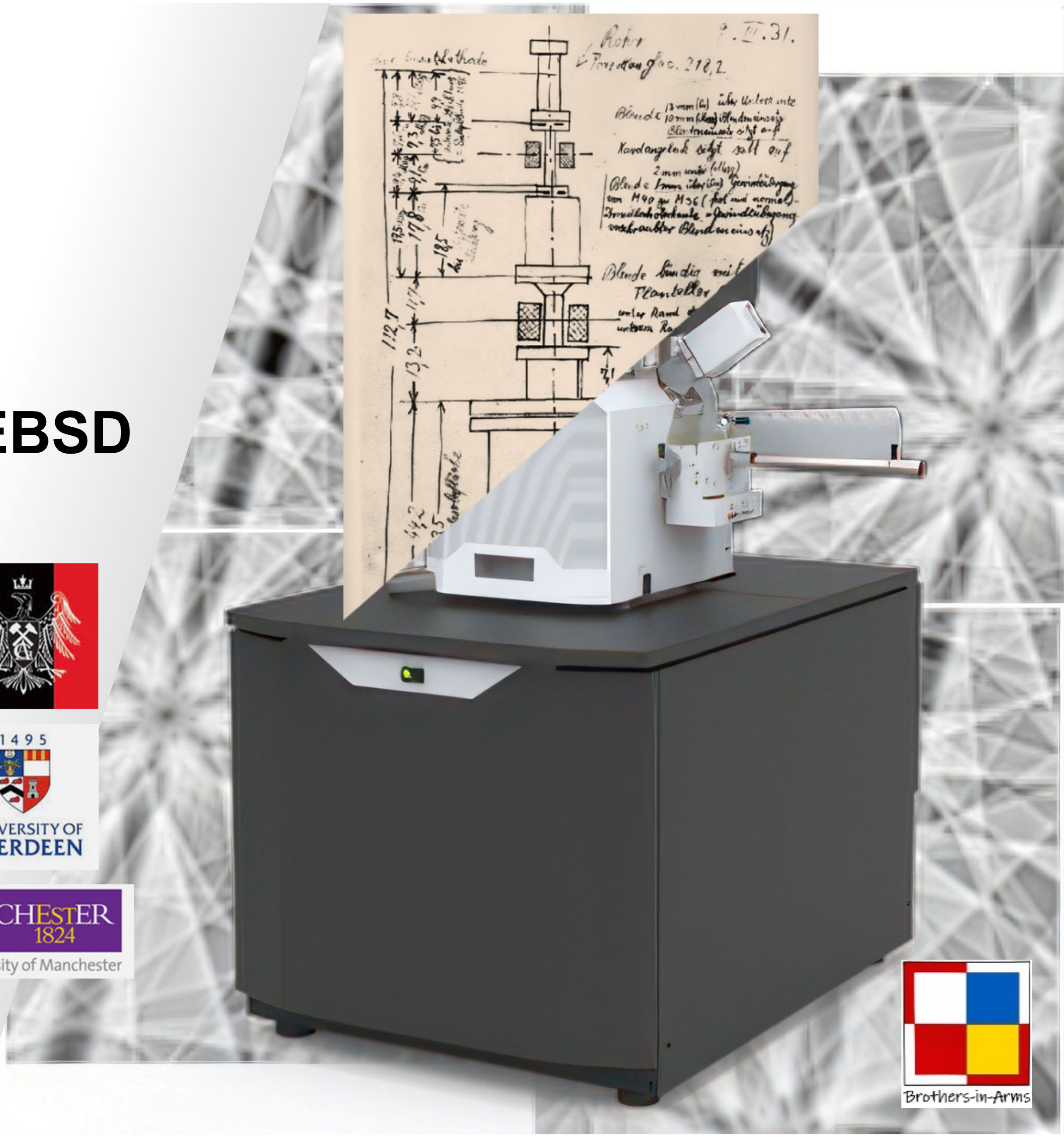
EMAS European Microbeam Analysis Society

17th European Workshop on MODERN DEVELOPMENTS AND APPLICATIONS IN MICROBEAM ANALYSIS
7-11 May 2023, Kraków, Poland

1495 UNIVERSITY OF ABERDEEN

MANCHESTER 1824 The University of Manchester



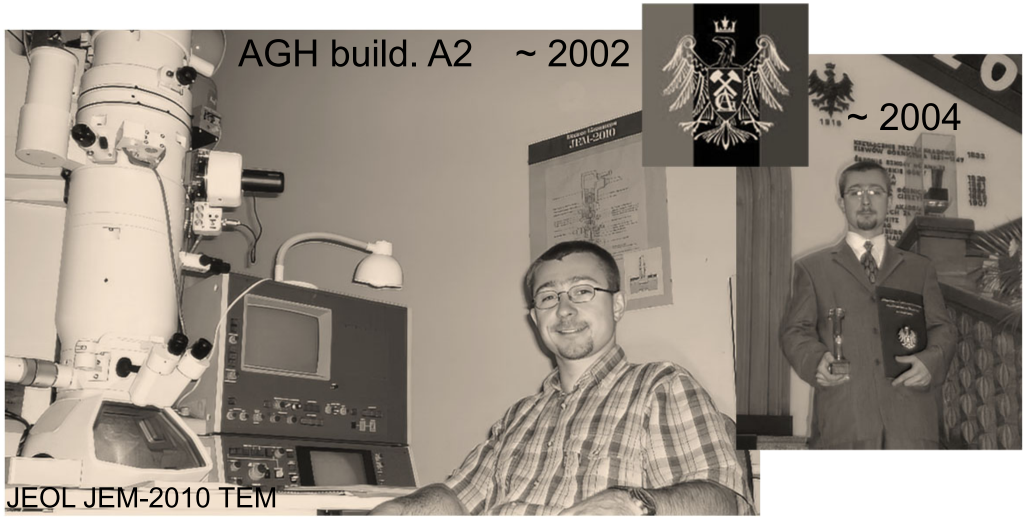
Brothers-in-Arms

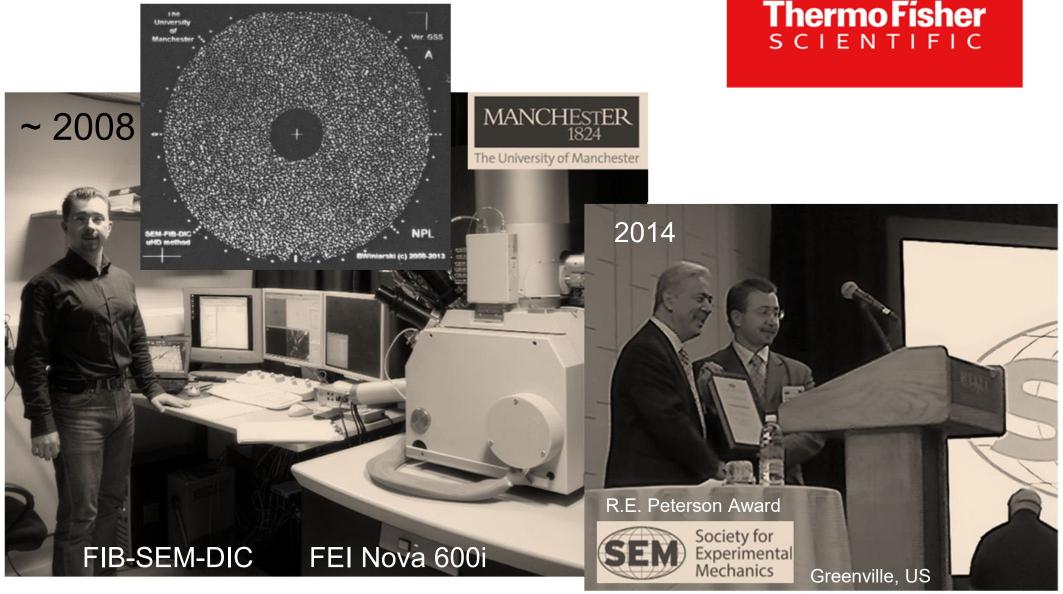
The world leader in serving science

Outline

- Short introduction to focussed beams
- Historical brief on development of focussed beams
- Focussed beams & EBSD method
- Current and future directions

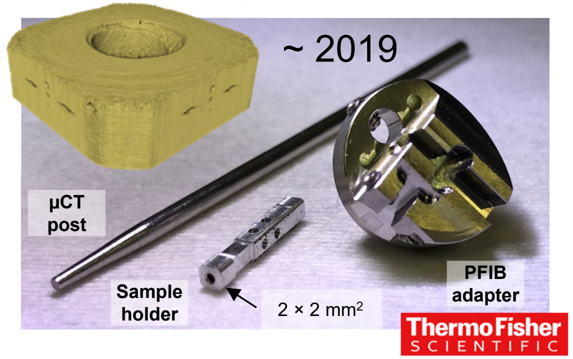
My passion for Electron Microscopy



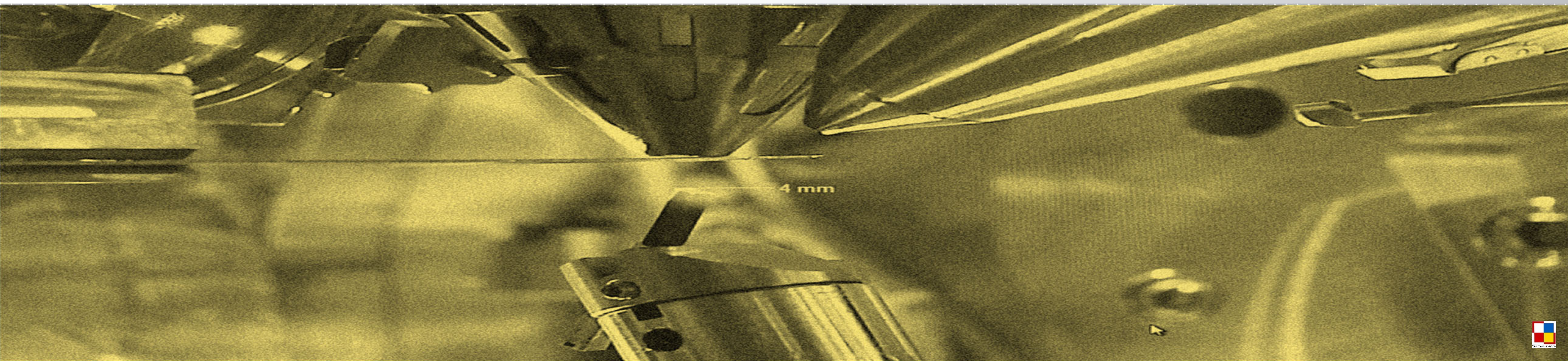


My Correlative Electron Microscopy journey

mCT-SDB cross-platform correlative holder kit



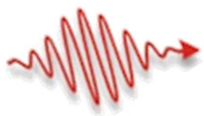
Short introduction to focussed beams



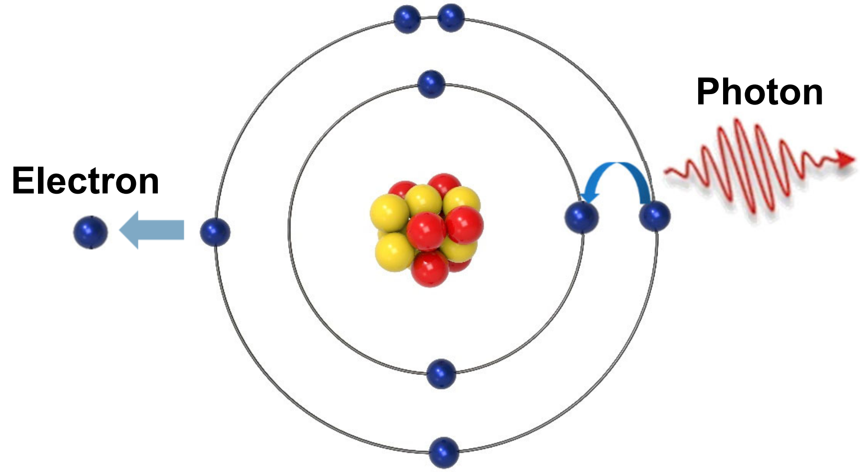
Focussed beams ?

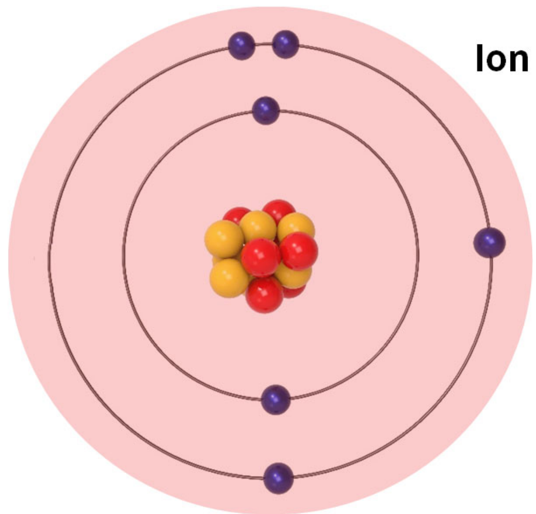
Focused beams are complex and compact systems that **emit**, **accelerate**, **deflect** and **raster** a narrow stream of:

Electrons e^-

Photons  fs pulses: 515 nm, 1030 nm

Ions He^+ Ne^+ Xe^+ Ar^+ O^+ N^+ Ga^+ + other





Focussed beams - schematics

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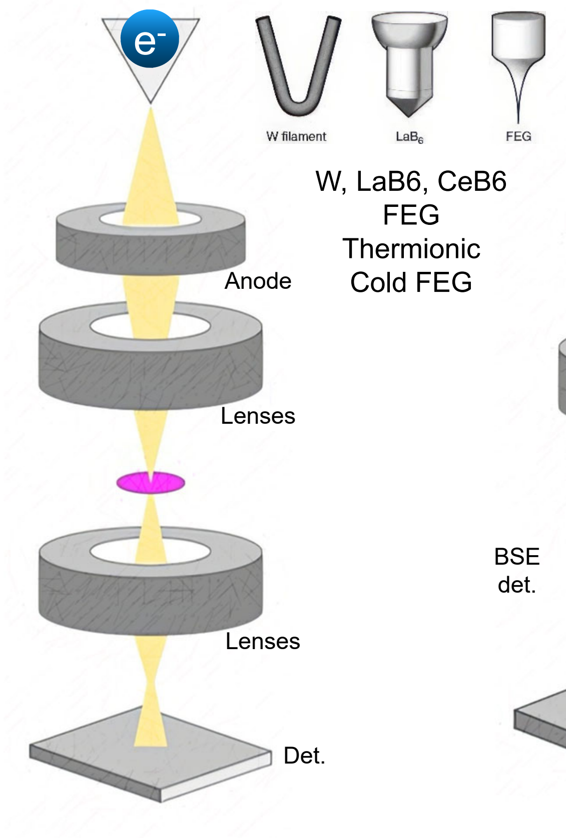
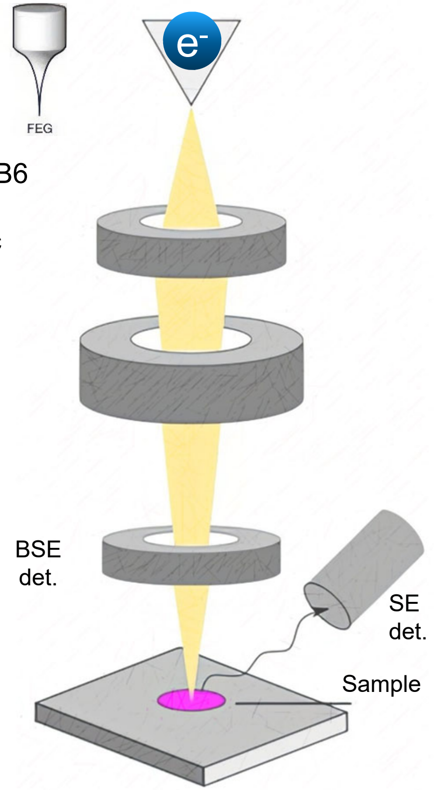
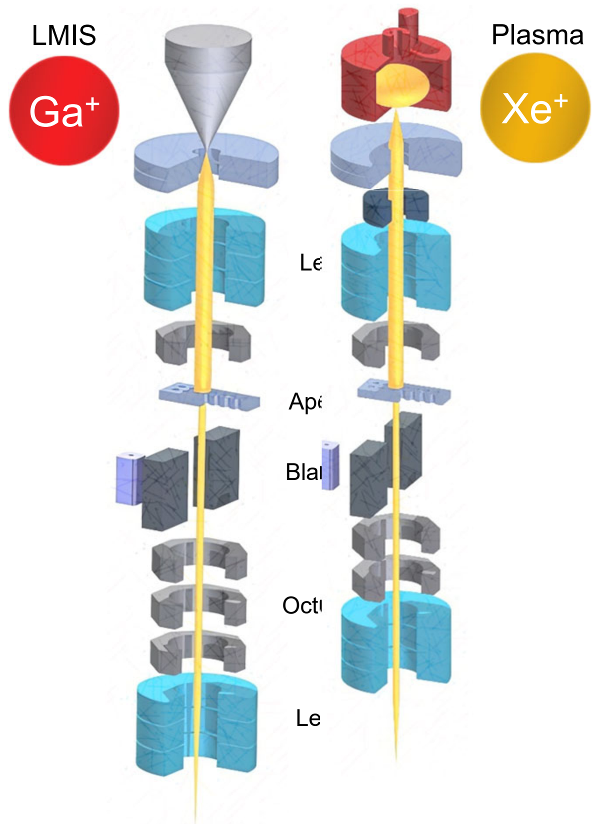
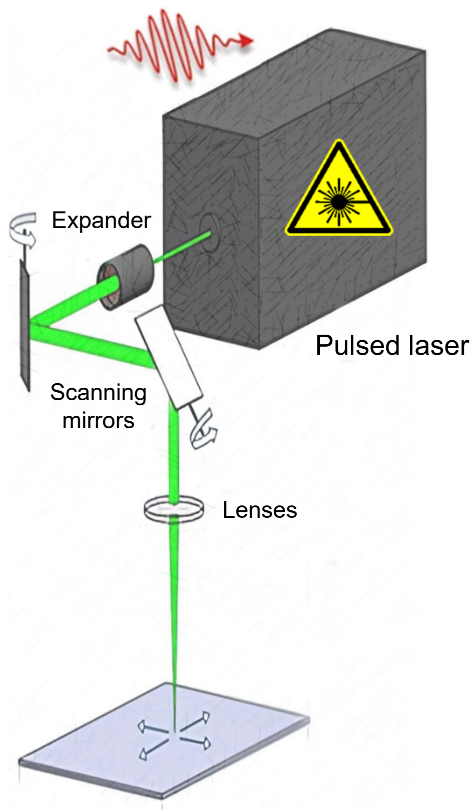
TEM | **SEM** | **FIB** | **fs Laser**





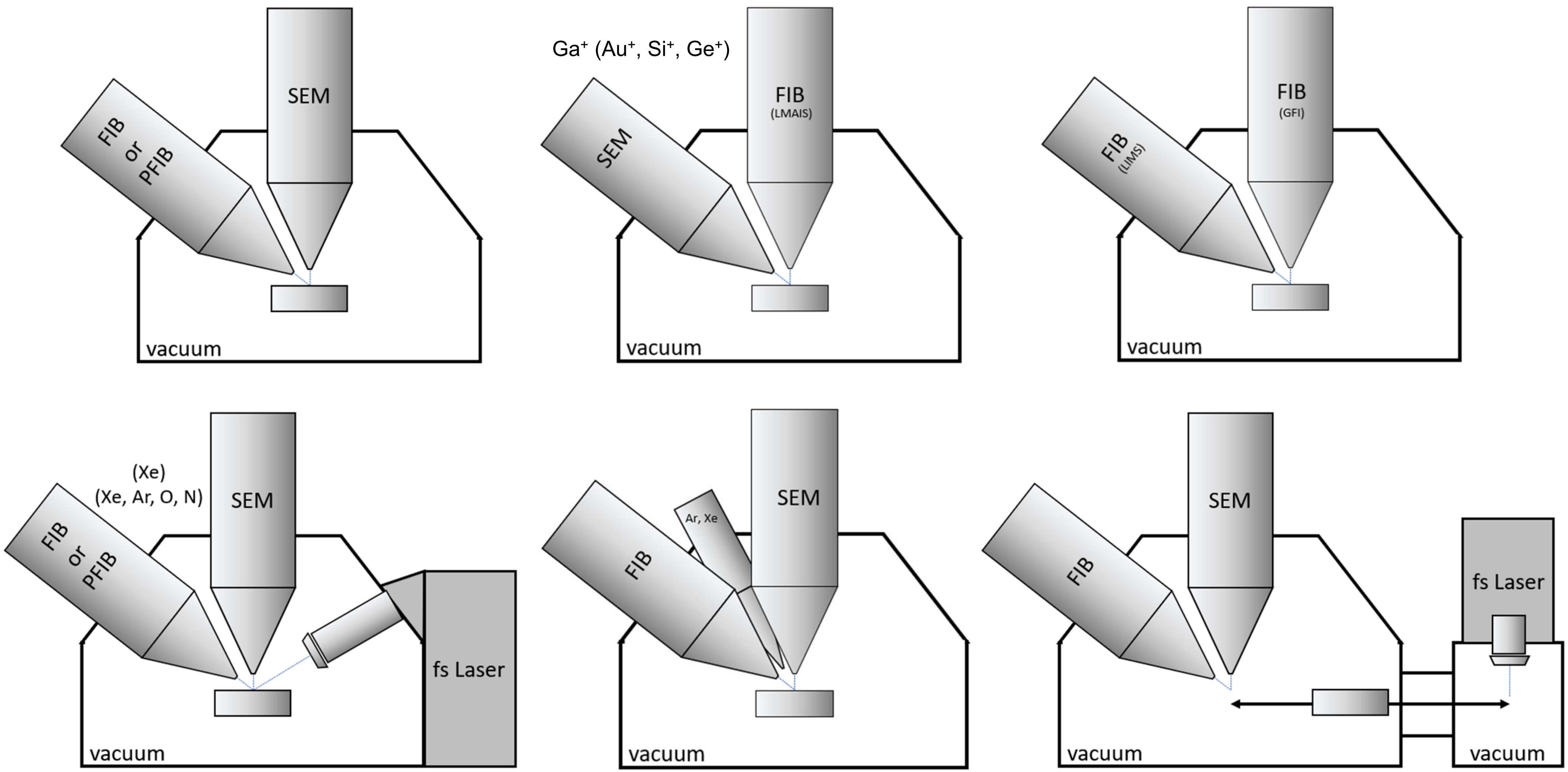




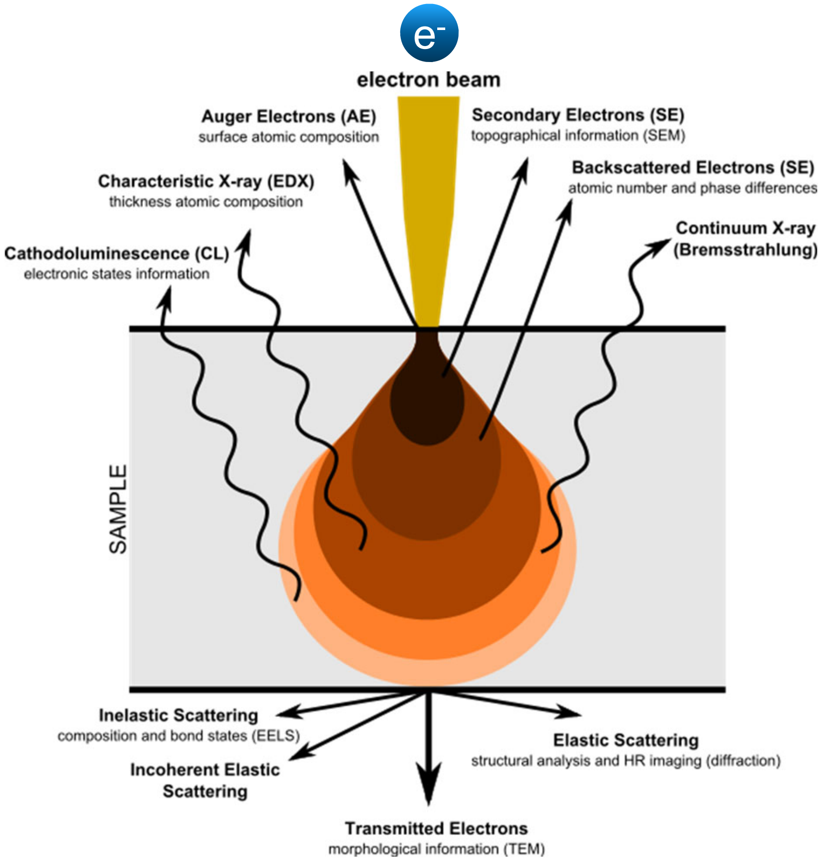
<< 1 nm | **< 1 nm** | **< 6 nm** | **< 30 nm** | **< 20,000 nm**

Other ion sources: GFI, LMAIS, LOTIS, MOTIS

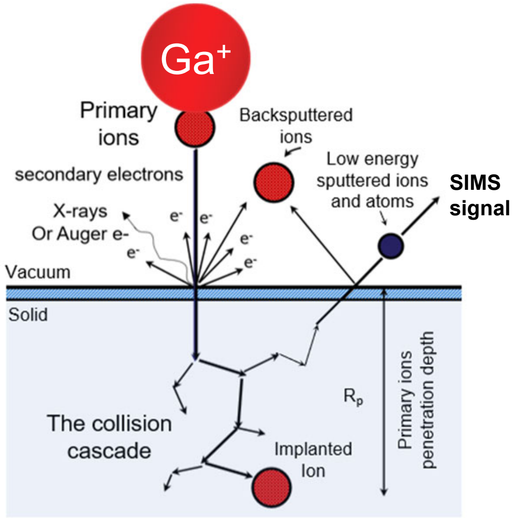
Focussed beams - schematics

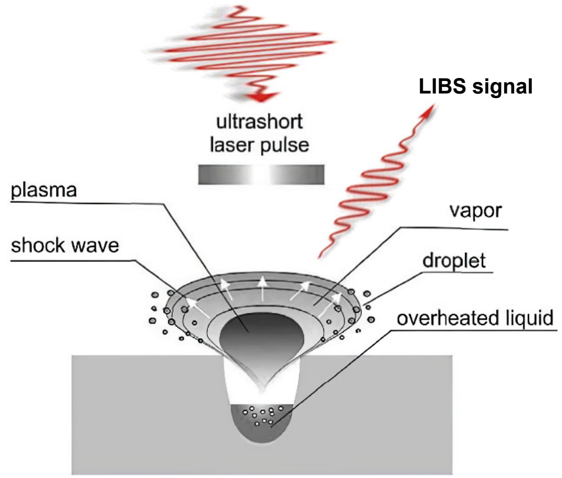


Focussed beams - applications



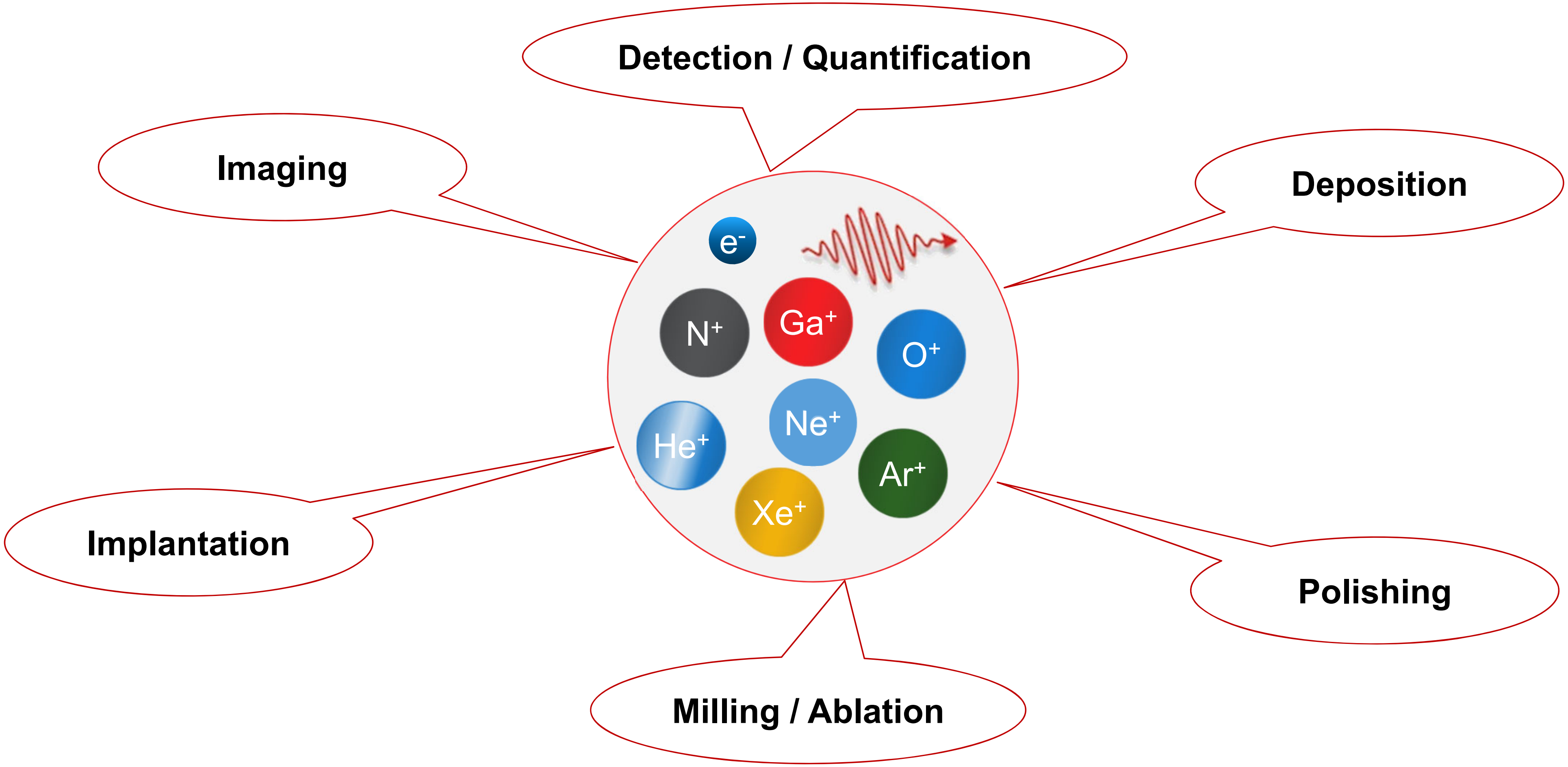
https://commons.wikimedia.org/wiki/File:Electron_Interaction_with_Matter.svg.





Focussed beams - applications

Currently these **beams are typically used for** six purposes:



Surface preparation methods for EBSD

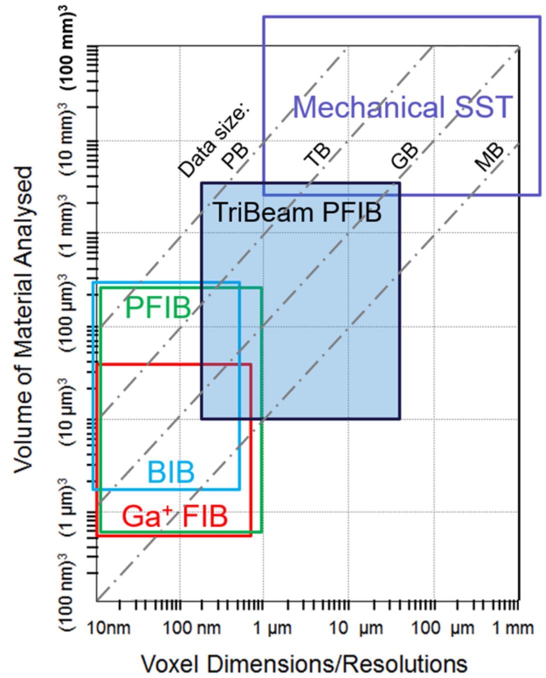
Key material factors for 2D and 3D EBSD data collection:

- Crystalline solids
- Pristine Surface finish (roughness → few nm)
- Intact crystal structure during material removal
- No surface contaminations

	Ga FIB	Xe PFIB	Laser PFIB	Xe PFIB SM*	Broad IB	Mech. Polish.
Slice thickness [nm]	5 ... 50	25 ... 500	**250 ... 10,000**	1 ... 1000	10 ... 1000	**200 ... 10,000**
Slice rate [µm^3/s]	**20**	**400**	**40,000**	400	33	**100,000 ... 500,000**
Max sample size [um]	50 x 50	400 x 400	**2000 x 2000**	800 x 800	300 x 300	50,000 x 50,000
Damage depth [nm]	**3 ... 22**	**2.5 ... 13**	**20 ... 50**	2.5 ... 13	< 30	**35 ... 60**
Si amorphisation						

×20 ×100

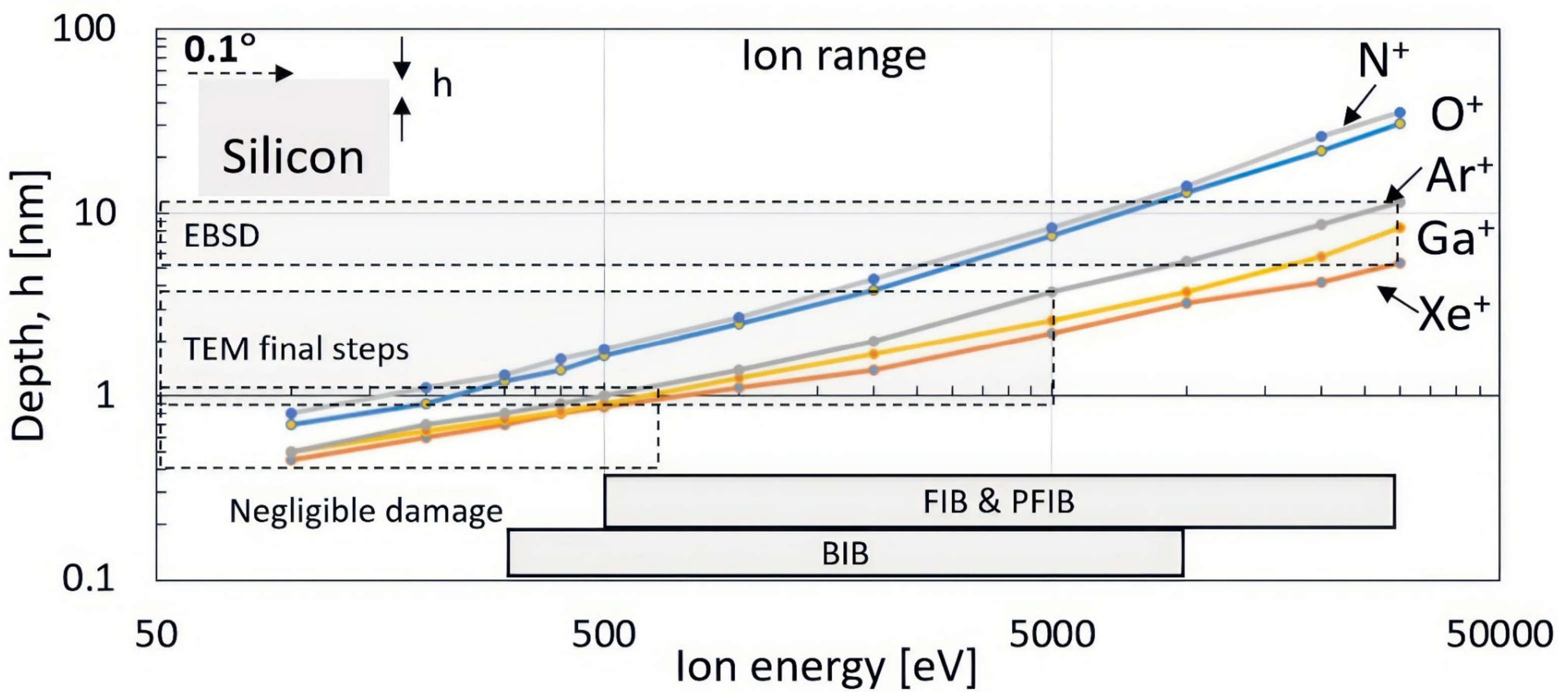
* - Spin Milling



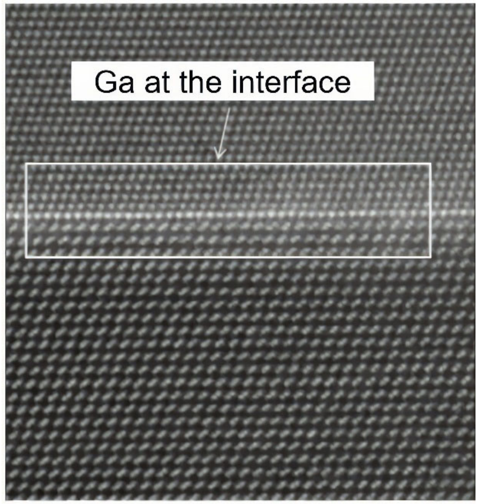
Based on: *M.P. Echlin, et al. Current Opinion in Solid State & Materials Science 24 (2020) 100817*

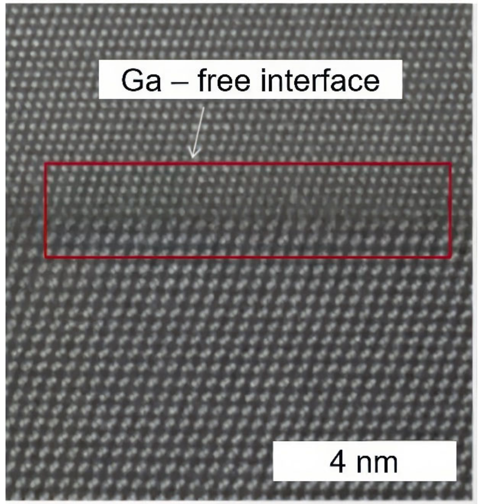
Surface preparation methods for EBSD

SRIM simulation of ions implantation ranges for energies 100 eV to 30 keV; 0.1 deg glancing angle of ions.



Jiao, C., von Lear, B. *Low Energy 500 eV Focused Argon Ion Beam Provided by Multi-Ions Species Plasma FIB for Material Science Sample Preparations*. in *2021 Microscopy and Microanalysis Conference*. **2021**. Virtual Meeting: Cambridge Press.

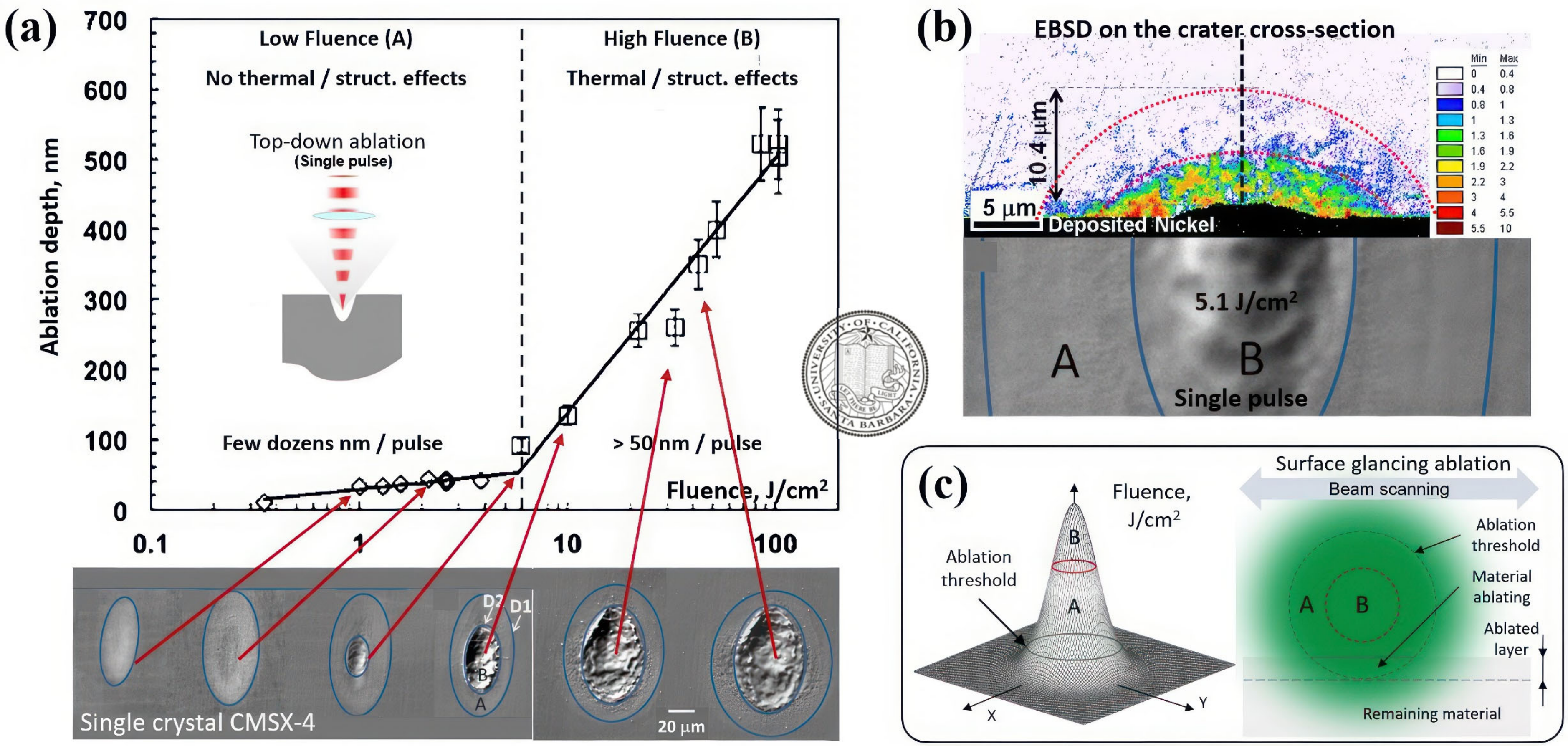




Titan 300, STEM HAADF Z-contrast;
Al/Si interface; Ga FIB vs Xe PFIB.

Surface preparation methods for EBSD

Femtosecond laser interaction with matter

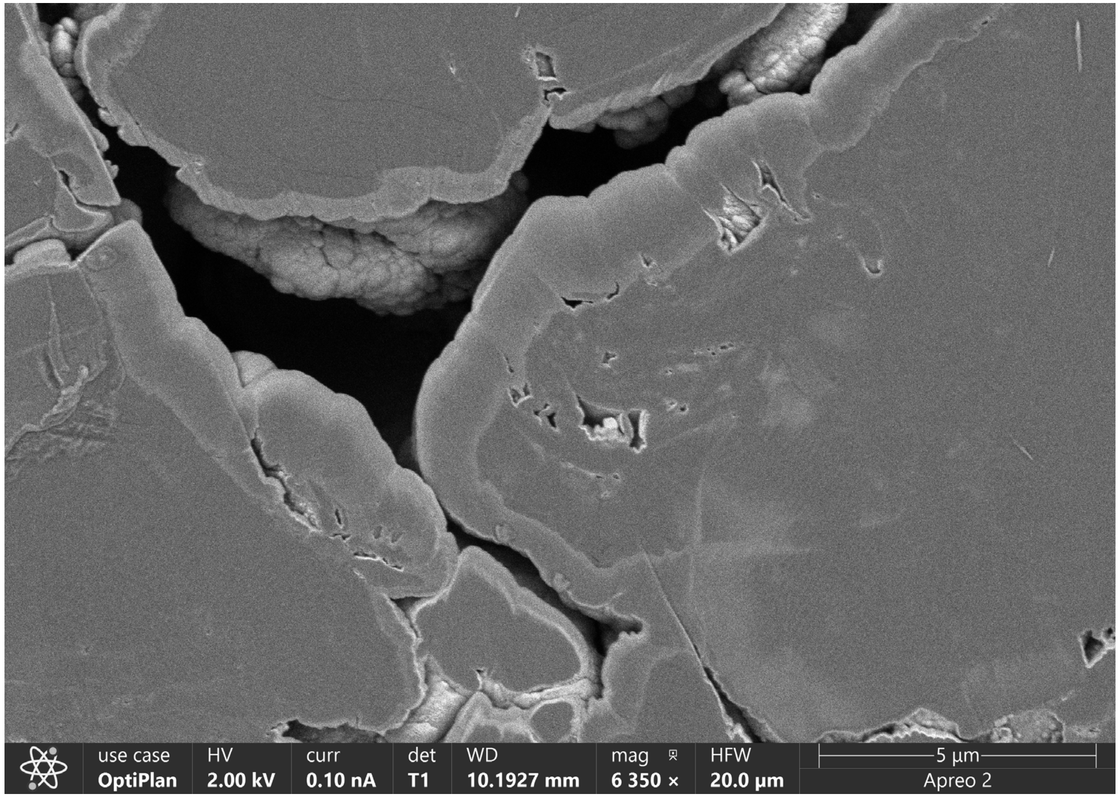


Kumar, A. and T.M. Pollock, *Mapping of femtosecond laser-induced collateral damage by electron backscatter diffraction.* Journal of Applied Physics, 2011. **110**(8).

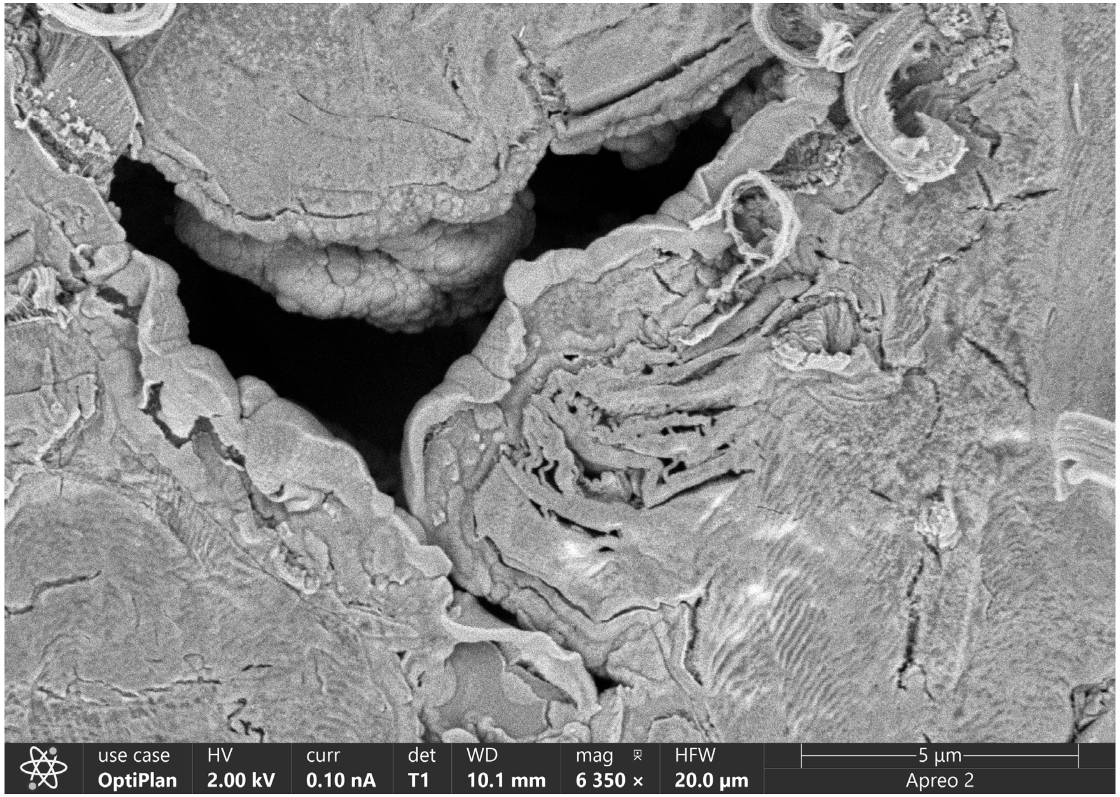
Echlin, M.P., et al., *Materials response to glancing incidence femtosecond laser ablation.* Acta Materialia, 2017. **124**: p. 37-46.

Surface preparation methods for EBSD

Surface contamination visible after exposing the anode to air



Lithiated carbon anode – CleanConnect transfer

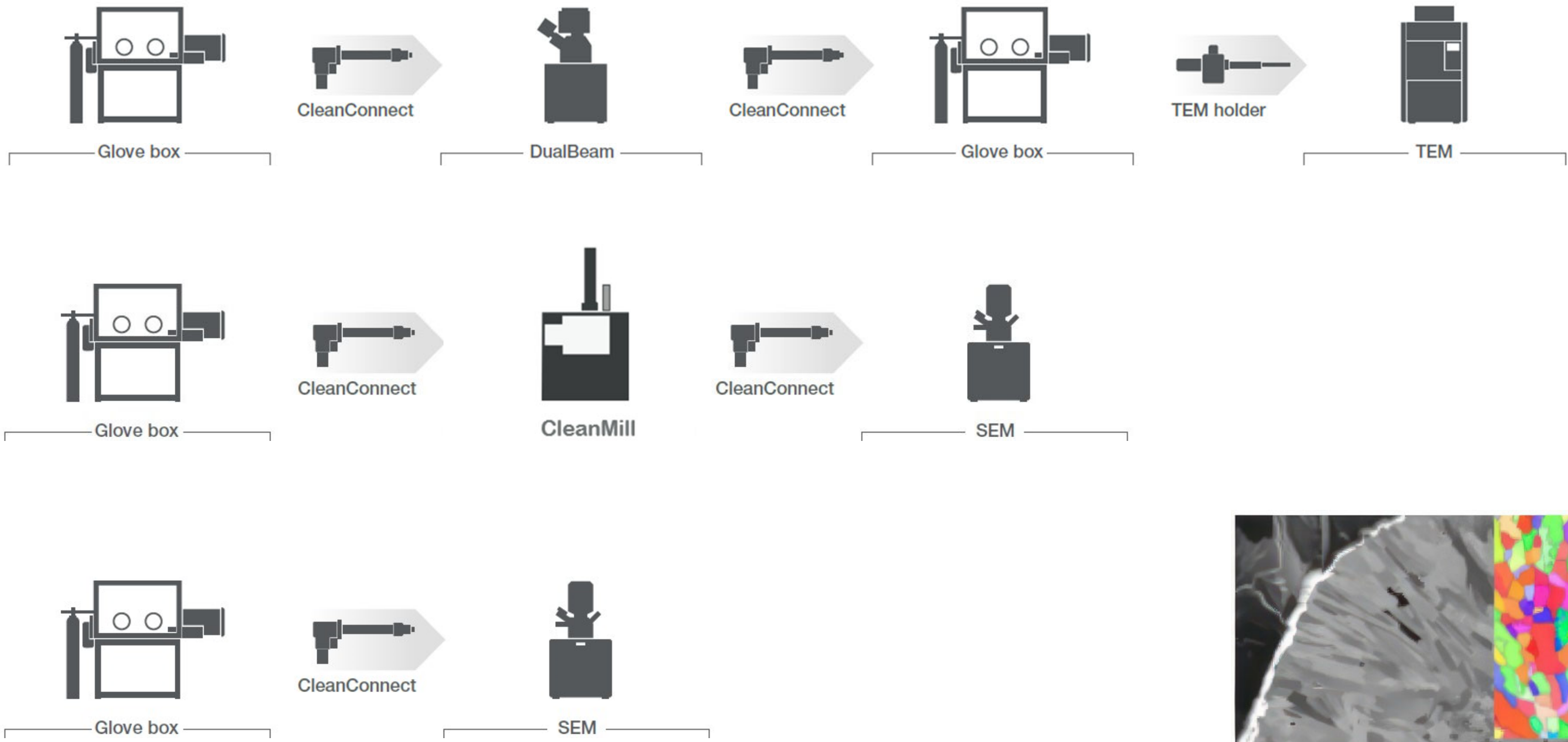


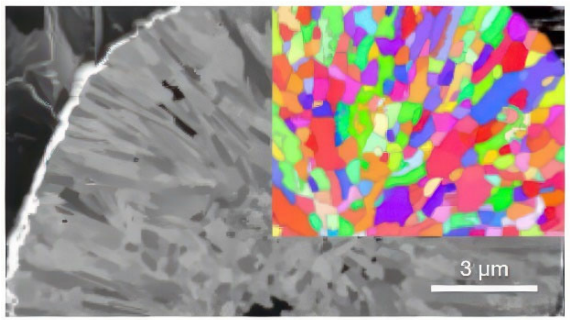
Lithiated carbon anode – exposed to air (~1 min)

CleanMill: 10 kV, 3.5 mA, 1.5° tilt, 40° oscillation, 1 h

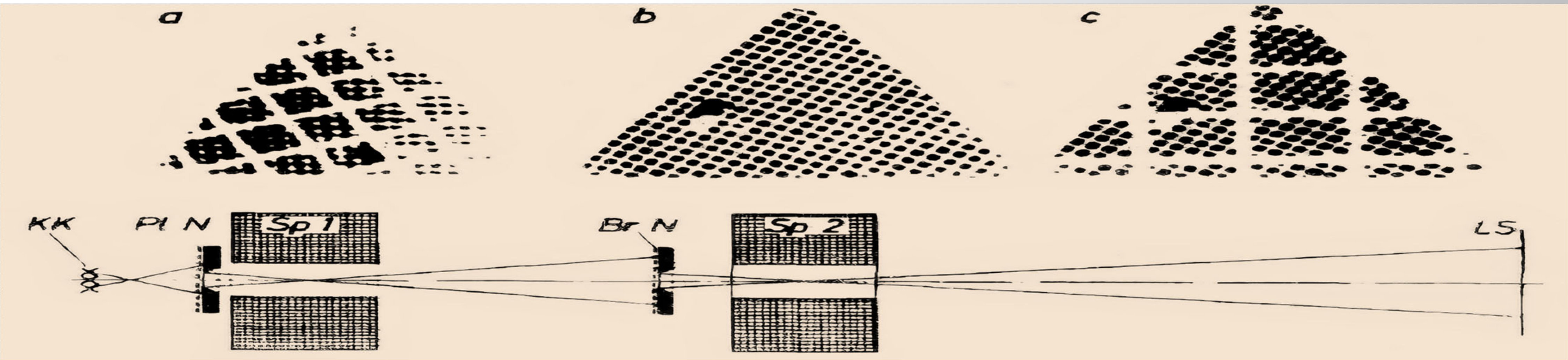
Images courtesy: Peter Priecel, Thermo Fisher Scientific

Surface preparation methods for EBSD





Historical brief on development of focussed beams



Historical brief on development of focussed beams

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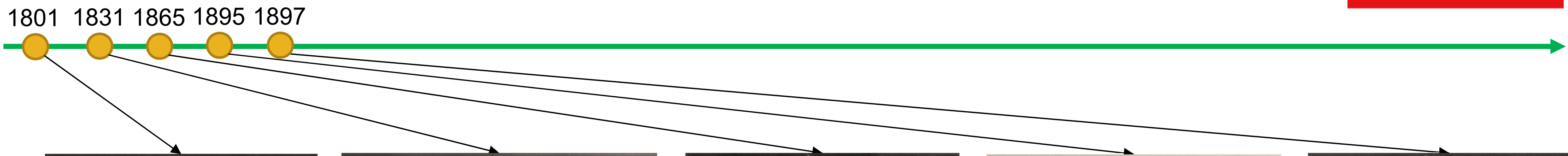
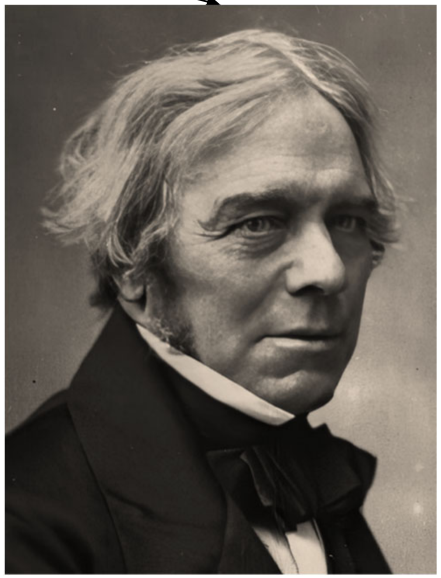


Thomas Young (1801)

Experimental proof of dual nature of light



Michael Faraday (1831)

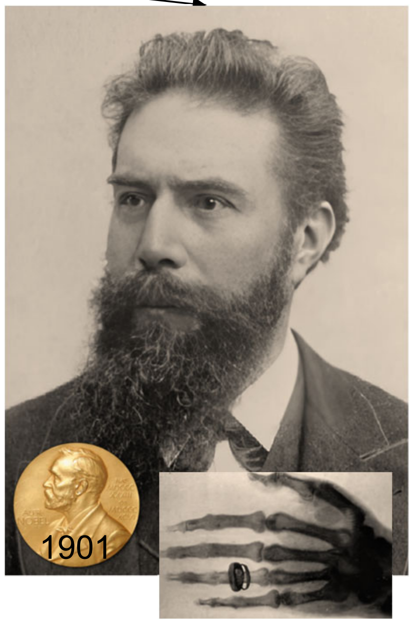
Electromagnetic induction

Joseph Henry 1832

James C. Maxwell (1865)

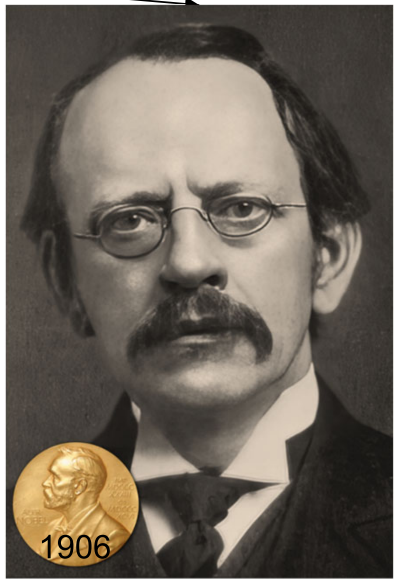
“A Dynamical Theory of the Electromagnetic Field”

J. W. Hittorf and J. Plücker, *Philos. Trans. R. Soc. London*, 1865, **155**, 1.



Wilhelm C. Röntgen (1895)

Discovery of X-Rays



Joseph J. Thomson (1897)

Discovery of electron

J. J. Thomson, *Phil. Mat. Ser.*, 1927, **74**, 1128.

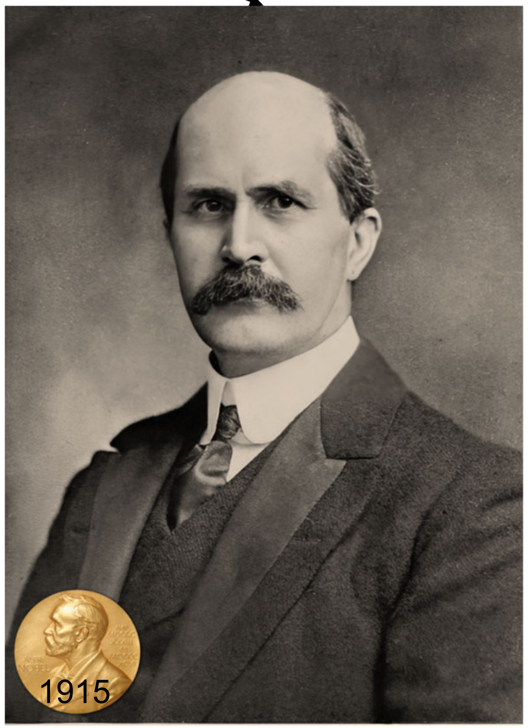
Historical brief on development of focussed beams

1801 1831 1865 1895 1897 1912

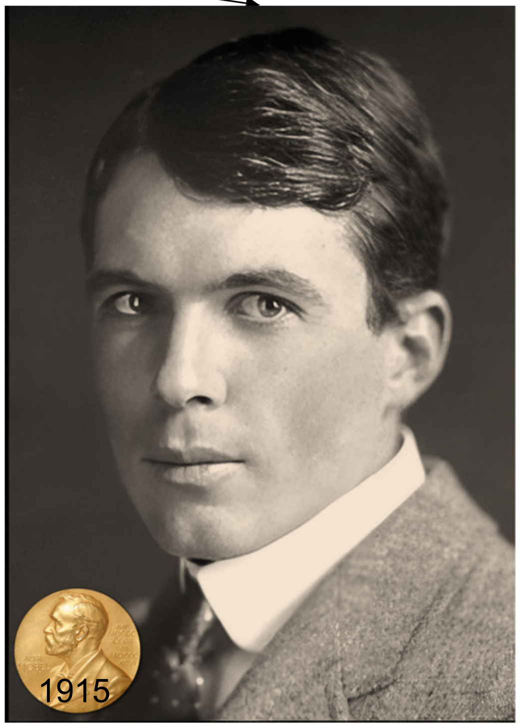
...schr. XVIII, 1917. Einstein, Zur Quantentheorie der Strahlung.

Max von Laue (1912)

The diffraction of X-rays by crystals



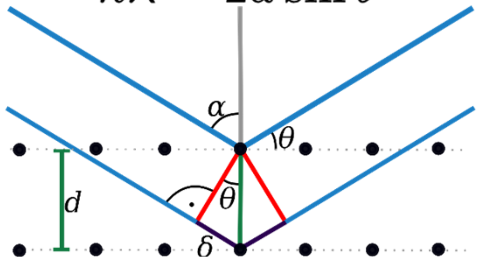
William Henry Bragg (1912)



William Lawrence Bragg (1912)

The law of diffraction

$$n\lambda = 2d\sin\theta$$



X-ray spectrometer

Historical brief on development of focussed beams

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Wave–particle duality

Experimentally proven

L. de Broglie (1925)

Hans Busch (1926)

Cylindrical electro-magnetic lens

C. J. Davisson & L. Germer (1927)

G. P. Thomson & A. Reid

890 NATURE [JUNE 18, 1927

Letters to the Editor.

[The Editor does not hold himself responsible for opinions expressed by his correspondents. Neither can he undertake to return, nor to correspond with the writers of, rejected manuscripts intended for this or any other part of NATURE. No notice is taken of anonymous communications.]

Diffraction of Cathode Rays by a Thin Film.

IF a fine beam of homogeneous cathode rays is sent nearly normally through a thin celluloid film (of the order 3×10^{-6} cm. thick) and then received on a photographic plate 10 cm. away and parallel to the film, we find that the central spot formed by the undeflected rays is surrounded by rings, recalling in appearance the haloes formed by mist round the sun. A photograph so obtained is reproduced (Fig. 1). If the density of the plate is measured by a photometer at a number of points along a radius, and the intensity of the rays at these points found by using the characteristic blackening curve of the plate (see *Phil. Mag.*, vol. 1, p. 963, 1926), the rings appear as h[...] intensity-distance curves. In this way r[...]

FIG. 1.

detected which may not be obvious to d[...] tion. With rays of about 13,000 volt[...] have been found inside the obvious [...] have been found of a fourth ring in [...] graphs, but not more than three have [...] on any one exposure. This is probably [...] limited range of intensity within which [...] measurements are feasible.

The size of the rings decreases with incre[...] of the rays, the radius of any given ring b[...] inversely proportional to the velocity, [...] rings are rather wide the measurements [...] are not very accurate. The energy of [...] measured by their electrostatic defle[...] from 3900 volts to 16,500 volts. Th[...] sharpest at the higher energies and were i[...] able at about 2500 volts. In one pho[...] radii of the rings were approximately [...] mm. for an energy of 13,800 volts.

It is natural to regard this phenomen[...] to the effect found by Dymond (NATU[...] 1926, p. 336) for the scattering of electron[...] though the angles are of course much sm[...] found. This would be due partly to the [...] of the rays giving them a smaller [...]

Using the formula $\lambda = h/mv$ the wave-length in the above-quoted case would be $\lambda = 1{\cdot}0 \times 10^{-9}$ cm. It is quite possible that there are other rings inside or outside those observed at present, and no opinion is advanced as to whether the diffracting systems are atoms or molecules. The disappearance of the rays at low speeds is probably due to the increased total amount of scattering which occurs. In all, about fifteen plates have been taken showing the effect, including some using a slit, instead of a pin hole, to limit the beam of rays. It is hoped to make further experiments with rays of greater energy and to obtain more accurate measurements of the size of the rings.

G. P. THOMSON.
A. REID.

University of Aberdeen,
May 24.

British Settlement in the Dominions Overseas.

HAVING read the interesting criticism in NATURE of May 14 of the annual report of the Oversea Settlement Committee, may I, as a member of that [...]

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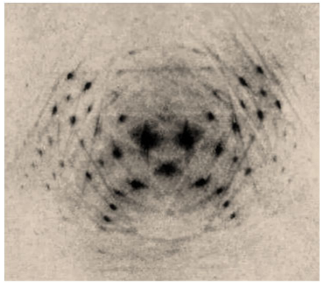
Historical brief on development of focussed beams

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928

Shoji Nishikawa and Seishi Kikuchi (1928)

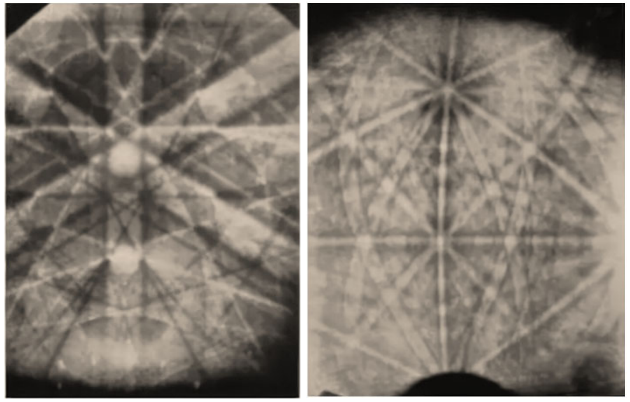
X-Ray "Kikuchi" pattern discovered

Walter Kossel



Kikuchi pattern from mica

Kikuchi pattern from calcite



20 keV

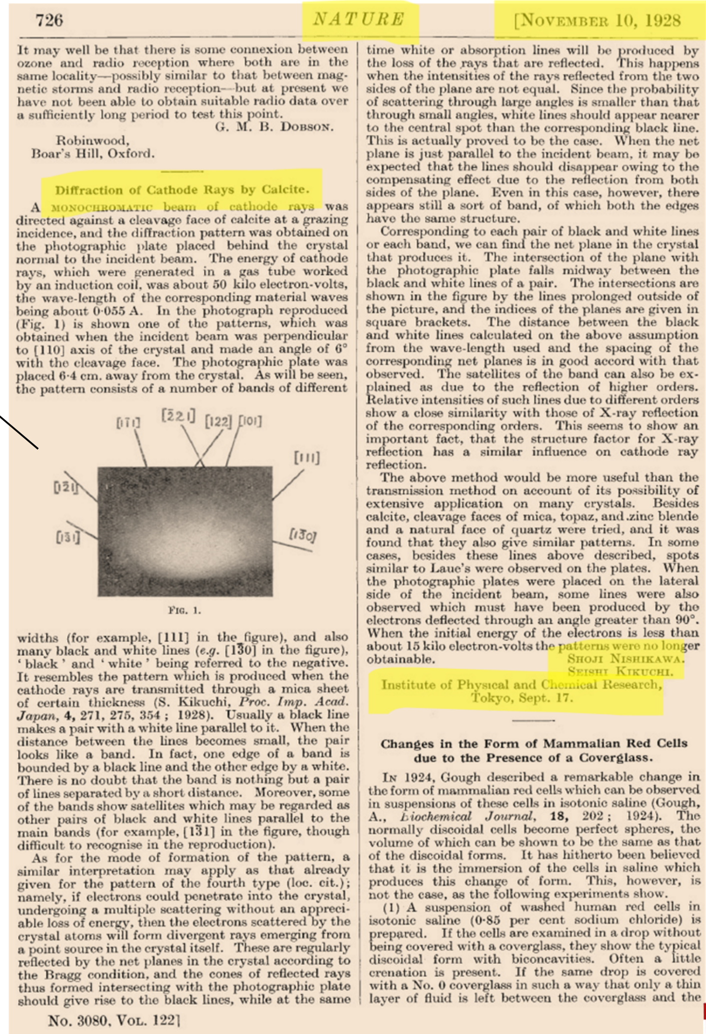
Iron

H. Boersch (1937)

H. Boersch, About bands in electron diffraction, Phys. Z. 38 (1937) 1000

History of EBSD by Prof. R. Schwarzer

http://ebsd.info/



726 NATURE [November 10, 1928

It may well be that there is some connexion between ozone and radio reception where both are in the same locality—possibly similar to that between magnetic storms and radio reception—but at present we have not been able to obtain suitable radio data over a sufficiently long period to test this point.

G. M. B. Dobson.

Robinwood,
Boar's Hill, Oxford.

Diffraction of Cathode Rays by Calcite.

A monochromatic beam of cathode rays was directed against a cleavage face of calcite at a grazing incidence, and the diffraction pattern was obtained on the photographic plate placed behind the crystal normal to the incident beam. The energy of cathode rays, which were generated in a gas tube worked by an induction coil, was about 50 kilo electron-volts, the wave-length of the corresponding material waves being about 0·055 A. In the photograph reproduced (Fig. 1) is shown one of the patterns, which was obtained when the incident beam was perpendicular to [110] axis of the crystal and made an angle of 6° with the cleavage face. The photographic plate was placed 6·4 cm. away from the crystal. As will be seen, the pattern consists of a number of bands of different

Fig. 1.

widths (for example, [111] in the figure), and also many black and white lines (*e.g.* [$1\bar{3}0$] in the figure), 'black' and 'white' being referred to the negative. It resembles the pattern which is produced when the cathode rays are transmitted through a mica sheet of certain thickness (S. Kikuchi, *Proc. Imp. Acad. Japan*, **4**, 271, 275, 354; 1928). Usually a black line makes a pair with a white line parallel to it. When the distance between the lines becomes small, the pair looks like a band. In fact, one edge of a band is bounded by a black line and the other edge by a white. There is no doubt that the band is nothing but a pair of lines separated by a short distance. Moreover, some of the bands show satellites which may be regarded as other pairs of black and white lines parallel to the main bands (for example, [$1\bar{3}1$] in the figure, though difficult to recognise in the reproduction).

As for the mode of formation of the pattern, a similar interpretation may apply as that already given for the pattern of the fourth type (loc. cit.); namely, if electrons could penetrate into the crystal, undergoing a multiple scattering without an appreciable loss of energy, then the electrons scattered by the crystal atoms will form divergent rays emerging from a point source in the crystal itself. These are regularly reflected by the net planes in the crystal according to the Bragg condition, and the cones of reflected rays thus formed intersecting with the photographic plate should give rise to the black lines, while at the same time white or absorption lines will be produced by the loss of the rays that are reflected. This happens when the intensities of the rays reflected from the two sides of the plane are not equal. Since the probability of scattering through large angles is smaller than that through small angles, white lines should appear nearer to the central spot than the corresponding black line. This is actually proved to be the case. When the net plane is just parallel to the incident beam, it may be expected that the lines should disappear owing to the compensating effect due to the reflection from both sides of the plane. Even in this case, however, there appears still a sort of band, of which both the edges have the same structure.

Corresponding to each pair of black and white lines or each band, we can find the net plane in the crystal that produces it. The intersection of the plane with the photographic plate falls midway between the black and white lines of a pair. The intersections are shown in the figure by the lines prolonged outside of the picture, and the indices of the planes are given in square brackets. The distance between the black and white lines calculated on the above assumption from the wave-length used and the spacing of the corresponding net planes is in good accord with that observed. The satellites of the band can also be explained as due to the reflection of higher orders. Relative intensities of such lines due to different orders show a close similarity with those of X-ray reflection of the corresponding orders. This seems to show an important fact, that the structure factor for X-ray reflection has a similar influence on cathode ray reflection.

The above method would be more useful than the transmission method on account of its possibility of extensive application on many crystals. Besides calcite, cleavage faces of mica, topaz, and zinc blende and a natural face of quartz were tried, and it was found that they also give similar patterns. In some cases, besides these lines above described, spots similar to Laue's were observed on the plates. When the photographic plates were placed on the lateral side of the incident beam, some lines were also observed which must have been produced by the electrons deflected through an angle greater than 90°. When the initial energy of the electrons is less than about 15 kilo electron-volts the patterns were no longer obtainable.

Shoji Nishikawa.
Seishi Kikuchi.

Institute of Physical and Chemical Research,
Tokyo, Sept. 17.

Changes in the Form of Mammalian Red Cells due to the Presence of a Coverglass.

In 1924, Gough described a remarkable change in the form of mammalian red cells which can be observed in suspensions of these cells in isotonic saline (Gough, A., *Biochemical Journal*, **18**, 202; 1924). The normally discoidal cells become perfect spheres, the volume of which can be shown to be the same as that of the discoidal forms. It has hitherto been believed that it is the immersion of the cells in saline which produces this change of form. This, however, is not the case, as the following experiments show.

(1) A suspension of washed human red cells in isotonic saline (0·85 per cent sodium chloride) is prepared. If the cells are examined in a drop without being covered with a coverglass, they show the typical discoidal form with biconcavities. Often a little crenation is present. If the same drop is covered with a No. 0 coverglass in such a way that only a thin layer of fluid is left between the coverglass and the

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Historical brief on development of focussed beams

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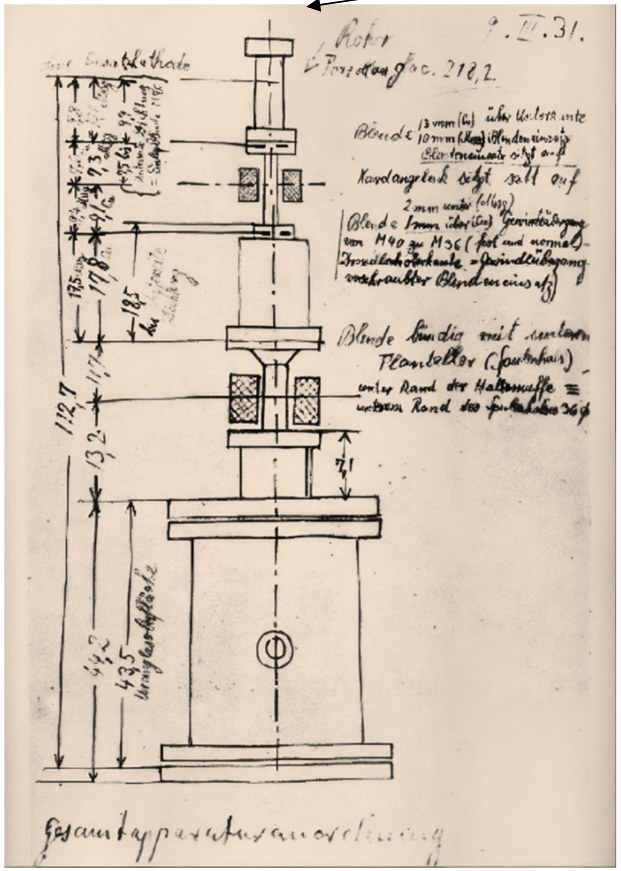
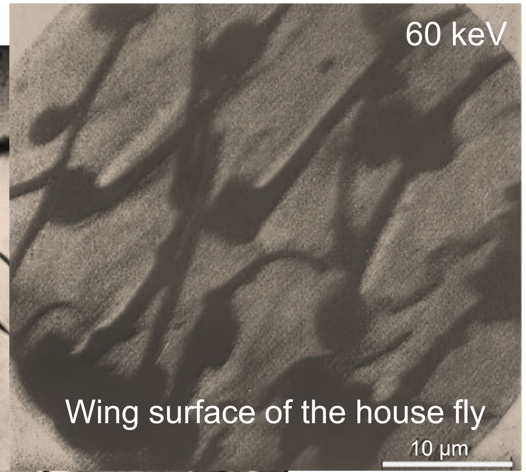


Electron microscope sketched by Ernst Ruska (9 III 1931)



Ernst Ruska and Max Knoll with the first functioning electron microscope (1933)

Historical brief on development of focussed beams

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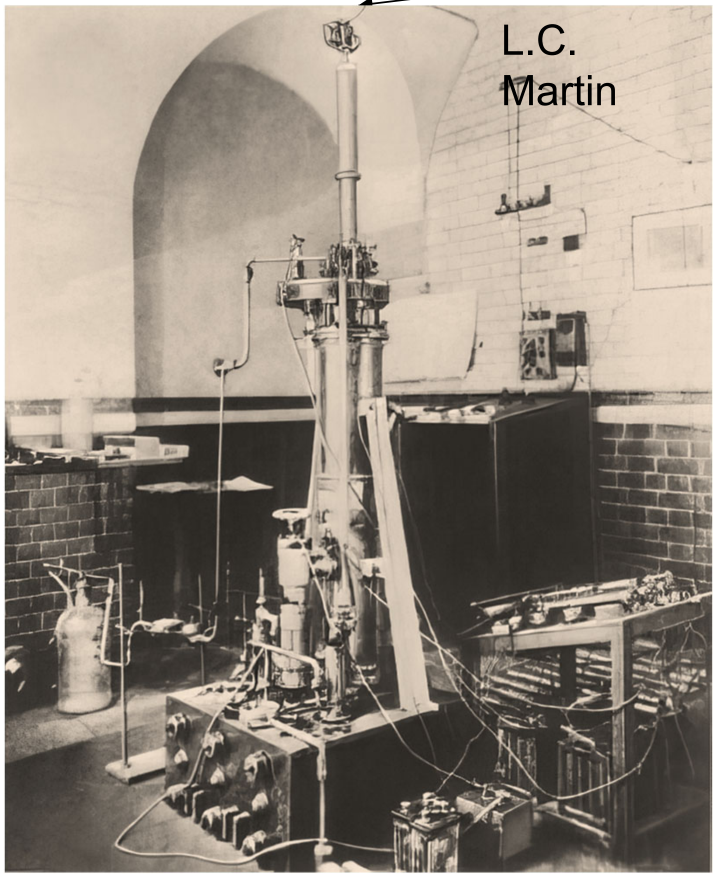
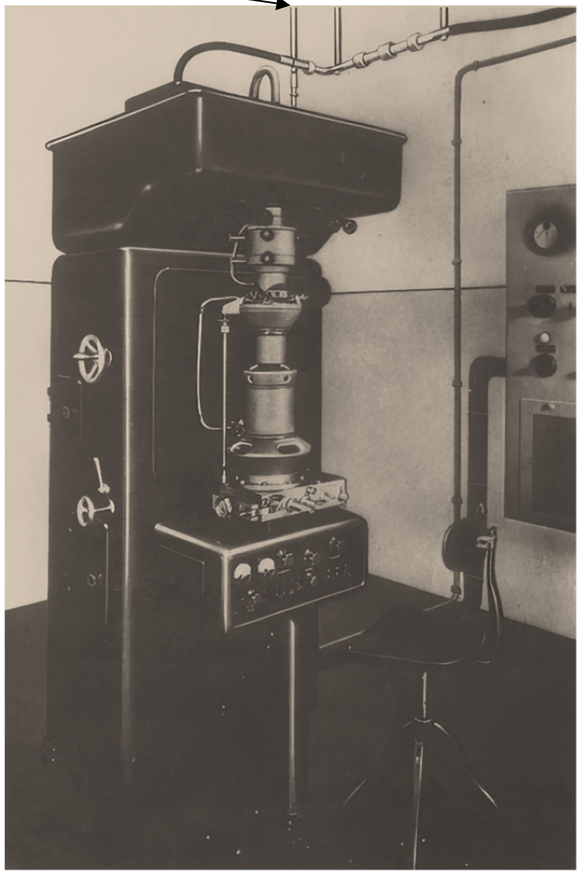
1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1939



EM1 - the first commercial EM in Imperial College London

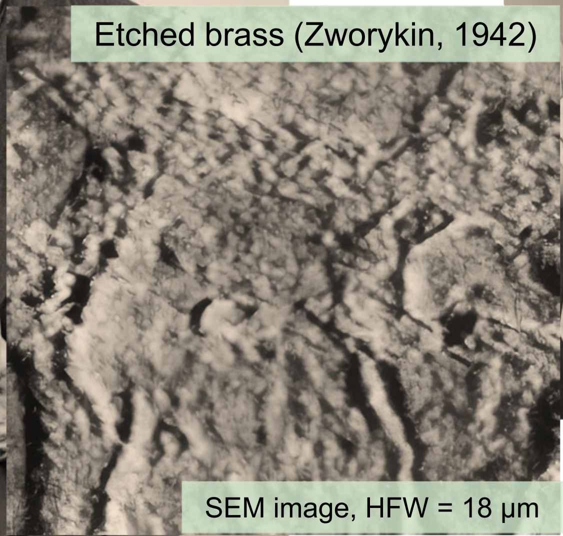


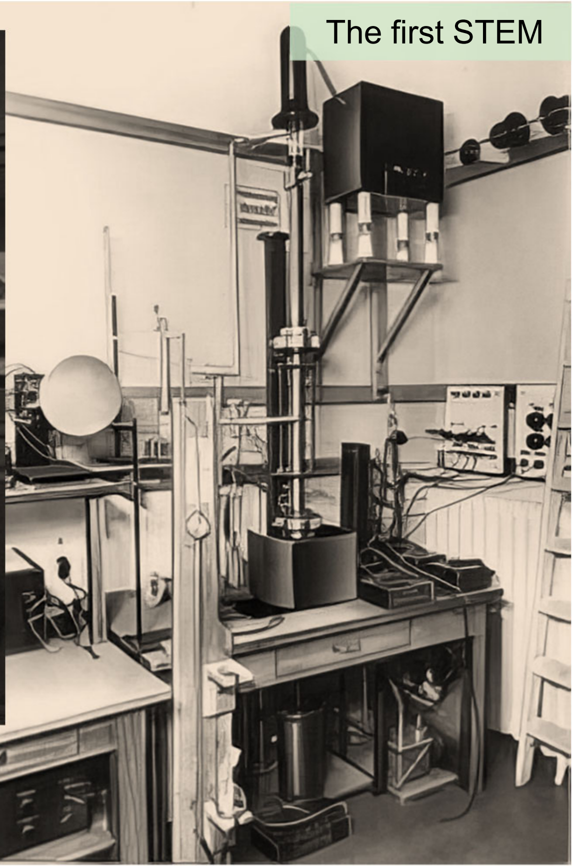
The first regularly produced EM by Siemens & Halske, Berlin

Historical brief on development of focussed beams e⁻

ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939





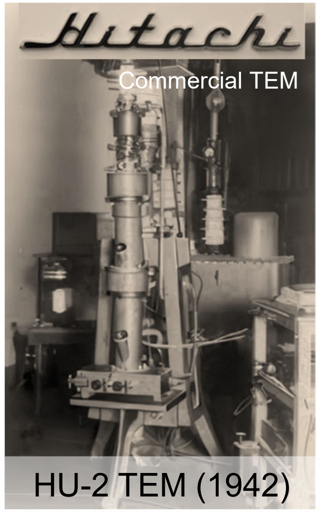
Vladimir Zworykin (~ 1937) RCA

Manfred von Ardenne (~ 1937), Berlin

Historical brief on development of focussed beams

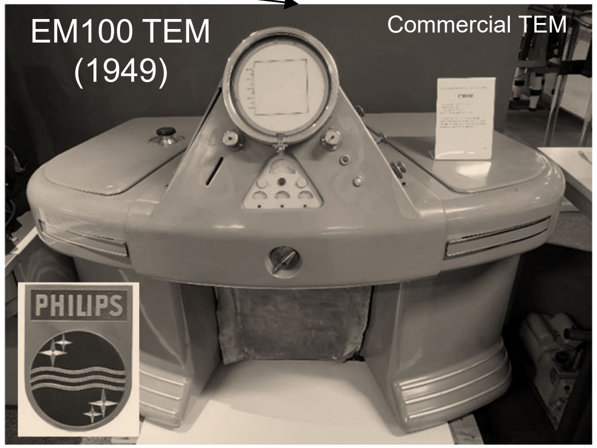
1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949

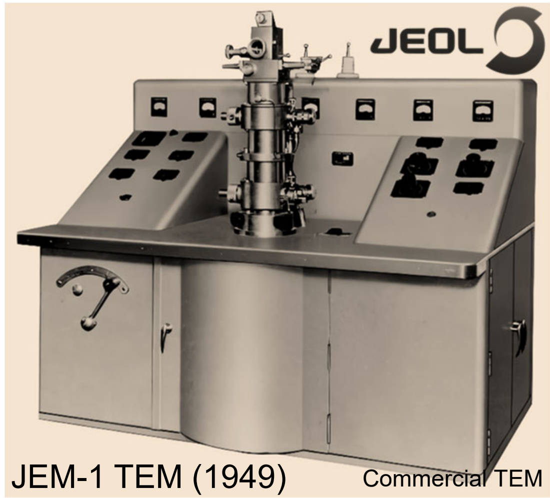
Albert Prebus and James Hillier (~ 1940) RCA



HU-2 TEM (1942)

Tesla BS241 (1951)





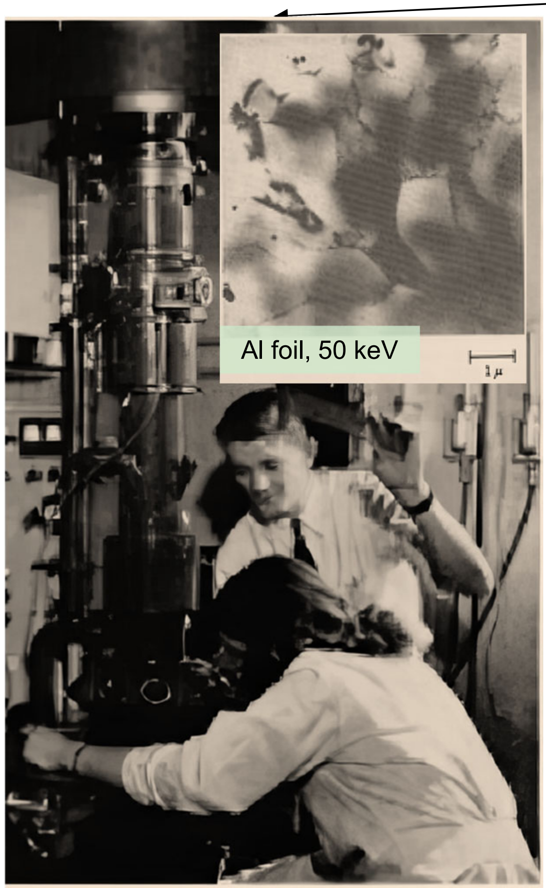
JEM-1 TEM (1949)

Historical brief on development of focussed beams

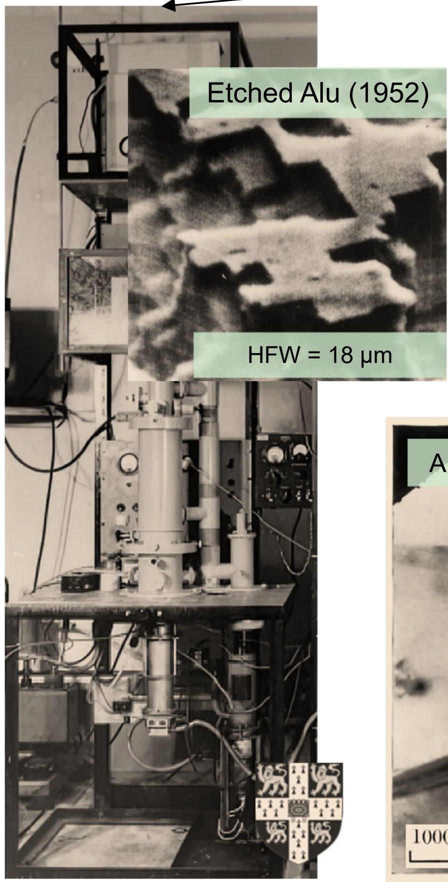
e⁻

ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956

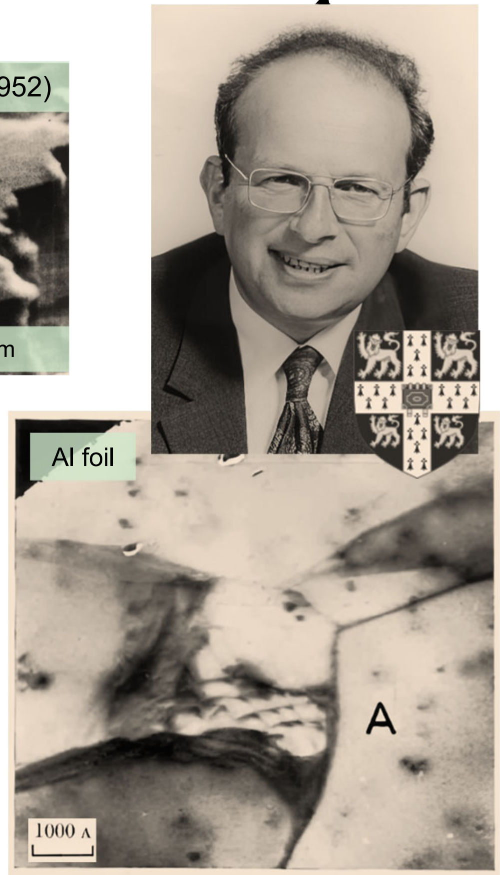


Robert D. Heidenreich (~ 1949)



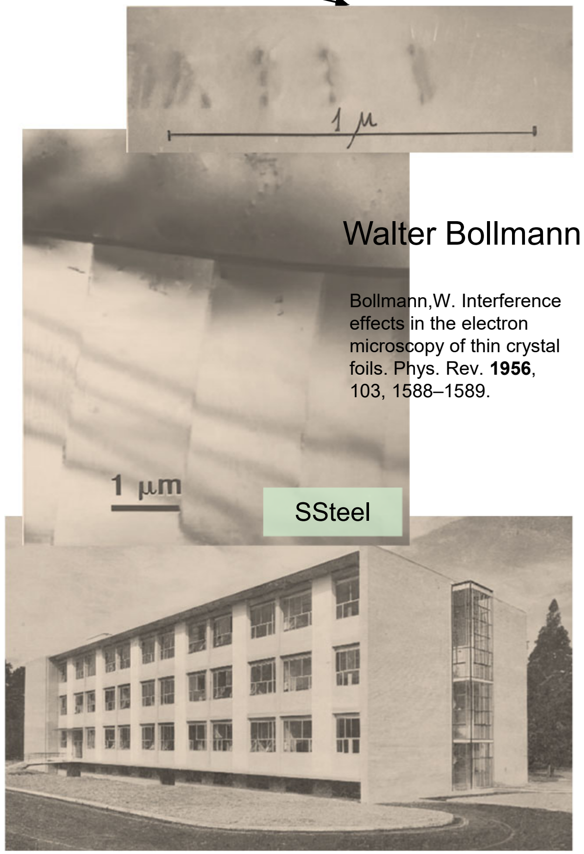
David McMullan (1951)

In 1951 first FEG -SEM



Peter B. Hirsch (1956)

Hirsch, P.B.; Horne, R.W.; Whelan, M.J. Direct observations of the arrangement and motion of dislocations in aluminium. Philos. Mag. **1956**, 1, 677–684.



Walter Bollmann

Bollmann,W. Interference effects in the electron microscopy of thin crystal foils. Phys. Rev. **1956**, 103, 1588–1589.

Battelle Lab., Geneva (1956)

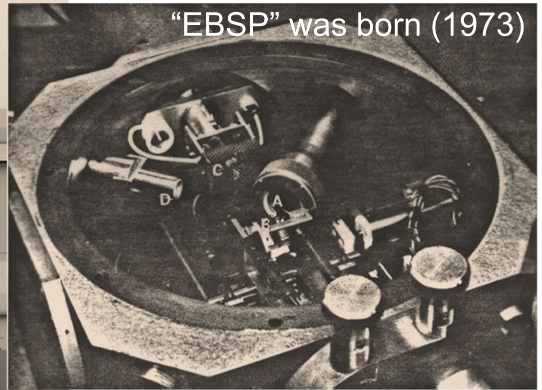
Historical brief on development of focussed beams e⁻

Thermo Fisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

Stereoscan MK1, the first commercial SEM (1965)

J. A. Venables and C. J. Harland



J. A. Venables and C. J. Harland (1973) „Electron Back Scattering Patterns –A New Technique for Obtaining Crystallographic Information in the Scanning Electron Microscope", *Philosophical Magazine*, **2**, 1193-1200.

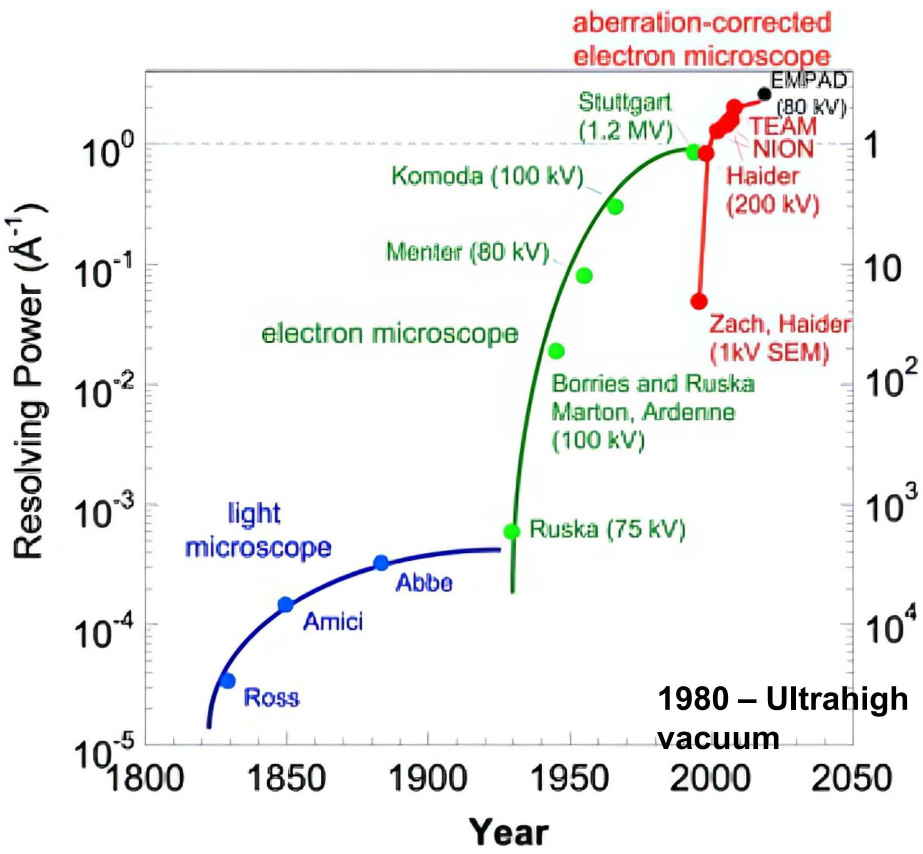
University of Chicago

The first practical STEM microscope (1969)

Historical brief on development of focussed beams e⁻

ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969



1980 – Ultrahigh vacuum

J. Liu, Advances and Applications of Atomic-Resolution Scanning Transmission Electron Microscopy. **Microscopy and Microanalysis** (**2021**), 27, 943–995

IX. BIBLIOGRAPHY OF SCANNING ELECTRON MICROSCOPY, CUED, 1951–

Compiled by

K. C. A. SMITH, B. C. BRETON, N. H. M. CALDWELL, D. McMULLAN

This bibliography constitutes a comprehensive record relating to work undertaken in the Cambridge University Engineering Department in connection with the scanning electron microscope and allied subjects.

1951
Sander, K. F. An automatic electron trajectory tracer and contributions to the design of an electrostatic electron microscope. Ph.D. Dissertation, University of Cambridge (1951)

1952
McMullan, D. Investigations relating to the design of electron microscopes. Ph.D. Dissertation, University of Cambridge (1952)

1953
McMullan, D. An improved scanning electron microscope for opaque specimens. *Proc. Inst. Electr. Engrs.* **100**, Part II, 245-259 (1953)

⋮

2008
Caldwell, N.H.M., Bui, M.T., Naran, J., Holburn, D.M. and Breton, B.C'Do-it-yourself web-based diagnosis for the scanning electron microscope' in Microscopy and Microanalysis, 14 (2), 894-895CD. (2008)

2009
Caldwell, N.H.M., Martin, G.C., Mitchell, A.L., Holburn, D.M., and Breton, B.C. 'Virtual SEM (VSEM) - Ongoing Development of Teaching Resources' in Microscopy and Microanalysis, 15 (2), (in press) (2009)
Caldwell, N.H.M., McMahon, C.A., Darlington, M.J., Heisig, P., Holburn, D.M. and Clarkson, P.J. 'Applying Engineering Information Management Principles to Microscopy' in Microscopy and Microanalysis, 15 (2), (in press) (2009).

http://www-g.eng.cam.ac.uk/oatley/biblio.html

Historical brief on development of focussed beams e⁻

ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969 Today


Thermo Scientific Spectra Ultra (S)TEM


JEOL ARM300 GrandARM™2


Thermo Scientific Apreo 2


Zeiss GeminiSEM 560


Tescan Magna


Hitachi HF5000

Tescan Tensor


JEOL JSM-IT800


Hitachi SU8600

Historical brief on development of focussed beams

ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

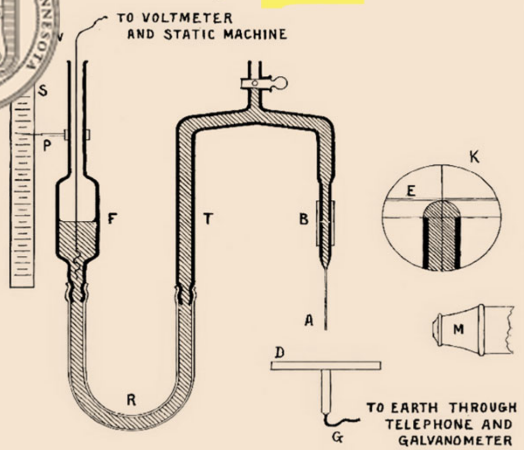
1914 1979

The liquid metal ion source (LMIS)

THE PHYSICAL REVIEW. 1914

THE ELECTRICAL DISCHARGE FROM LIQUID POINTS, AND A HYDROSTATIC METHOD OF MEASURING THE ELECTRIC INTENSITY AT THEIR SURFACES.[1]

BY JOHN ZELENY.



LIQUID METAL DROPLETS FOR HEAVY PARTICLE PROPULSION[1]

Victor E. Krohn, Jr.[2]

Ramo-Wooldridge a Division of Thompson Ramo Wooldridge, Inc. Canoga Park, California

Krohn VE (1961). Progress in Astro. and Rocketry. Vol. *5*. pp. 73-80.

FIB technology based on LMIS

Ion source of high brightness using liquid metal*

V. E. Krohn and G. R. Ringo *Appl. Phys. Lett.* 27, 479–481 (1975)

Argonne NATIONAL LABORATORY

Argonne National Laboratory, Argonne, Illinois 60439
(Received 23 July 1975)

The beam from an EHD ion source using liquid gallium has been shown to have a brightness of 0.9×10^5 A cm^{-2}sr^{-1} at 21 kV and an energy spread of 12 eV at 10-μA total current. The effective source diameter was 0.2 μm as imaged by a 2-cm-long Einzel lens with a 0.12-mm aperture. Cesium was significantly poorer and mercury much poorer than gallium.

Surface Science 70 (1978) 392–402

ORSAY PHYSICS

CONTRIBUTION OF FIELD EFFECTS TO THE ACHIEVEMENT OF HIGHER BRIGHTNESS ION SOURCES *

P. SUDRAUD, J. VAN DE WALLE, C. COLLIEX and R. CASTAING

Laboratoire de Physique du Solide associé au CNRS, Bâtiment 510, Université Paris-Sud, 91405 Orsay, France

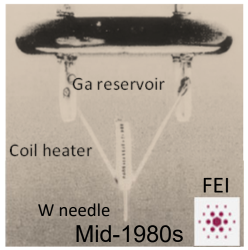
Current FIB manufacturers

ThermoFisher SCIENTIFIC, ZEISS, TESCAN, Raith, JEOL, HITACHI Inspire the Next, DCG Systems, ULVAC ULVAC-PHI, INC., APPLIED MATERIALS, SII

Other: liquid metal alloy ion sources (LMAIS), low temperature ion sources (LOTIS), and magneto optical trap ion sources (MOTIS)

G.L.R. Mair (1980)

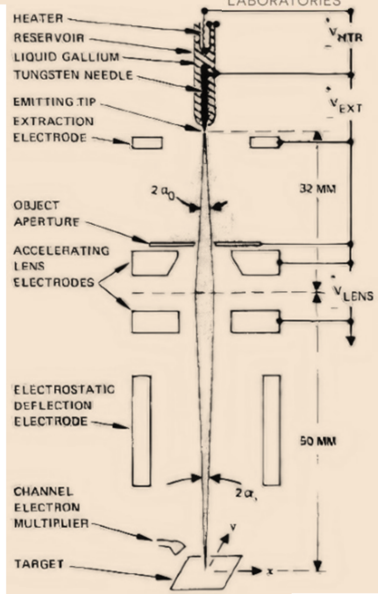
OGC, L. Swanson (1980)



A high-intensity scanning ion probe with submicrometer spot size

R. L. Seliger, J. W. Ward, V. Wang, and R. L. Kubena

HRL LABORATORIES



Appl. Phys. Lett. 34, 310–312 (1979)

Historical brief on development of focussed beams

ThermoFisher SCIENTIFIC

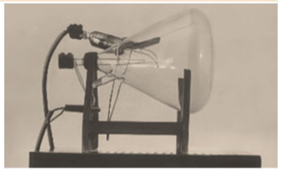
1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

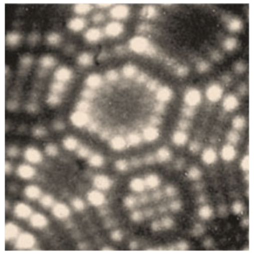
1914 1951 1979

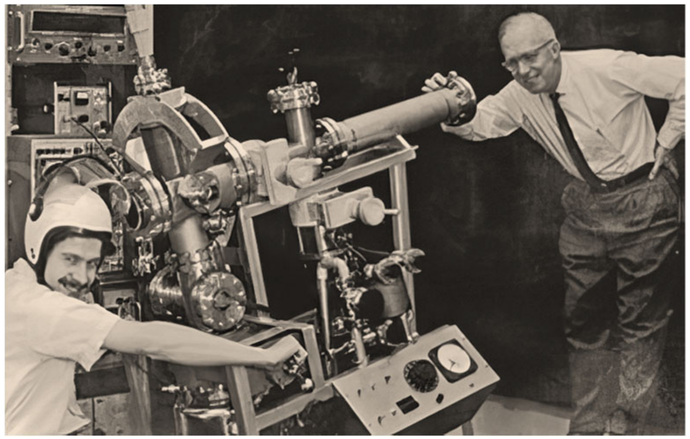
Gas Field Ionization (GFI)

Ervin Müller developed GFI in the **1950s** at **Penn State Uni** *ionize and acc*elerate **He+**.

Müller EW. 1951. Das Feldionenmikroskop. Zeitschrift für Physik 131:136.



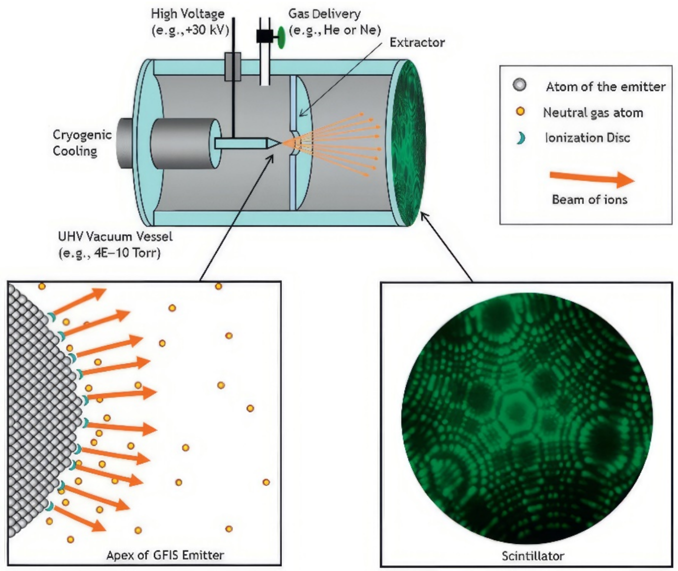




John Panitz (left), Erwin Muller (right), and the 3D atom probe (center).

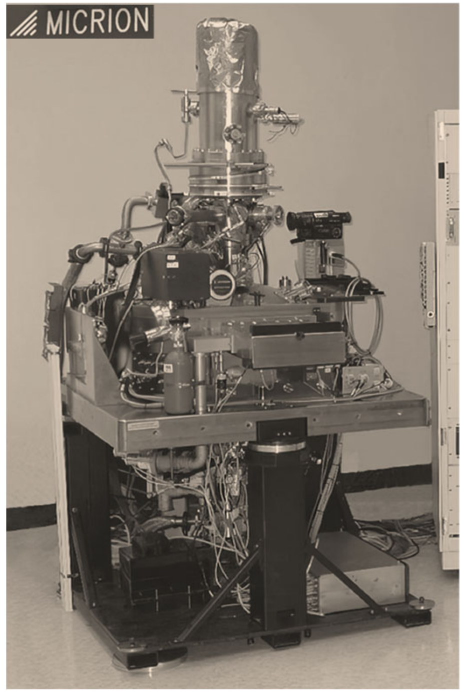
FIB technology based on GFI

Micrion Co. in mid-**1990s** and **FEI Co.** in early **2000s.** Advanced Lithography Ion Sources **ALIS** (FEI spinoff) about **2005**, and since **2006 Zeiss** HIM microscope.



The gas field ion source produces an ion emission pattern that corresponds to the atomic arrangement at the apex of the emitter

DOI: 10.1557/mrs.2014.53



The early Micrion GFIS research platform

Historical brief on development of focussed beams

ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

1914 1947 1951 1961 1979

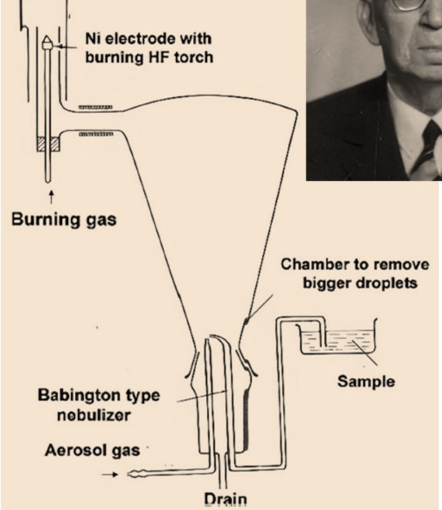
Thermal Plasma Source

G.I. Babat (1947)

G. I. Babat, *J. Inst. Electr. Eng.*, 1947, **94**, 27.

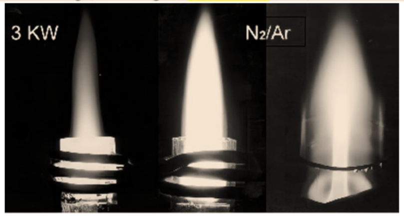
Eugen Bădărău (1956)

E. Bâdârâu, M. Giurgea, G. H. Giurgea and A. T. H. Truția, *Spectrochim. Acta*, 1956, **11**, 441.



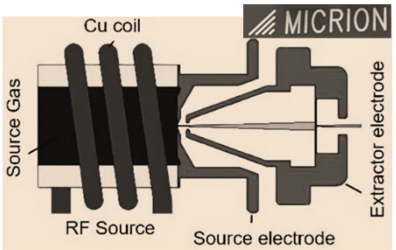
The inductively coupled plasma (ICP)

S. Greenfield, I. L. Jones and C. T. Berry, *Analyst*, 1964, **89**, 713.



Plasma FIB technology based on ICP

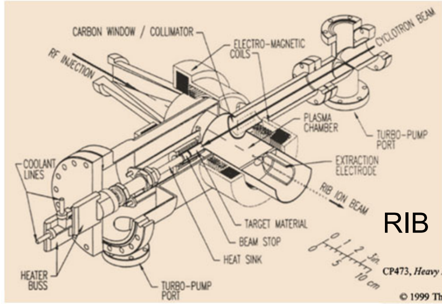
Micrion Co., USA in mid-**1980s** developed ICP source for Ion Beam Deposition (IBD) system (removing and deposition of materials) for semiconductor applications.



In 1999 FEI Co. acquires **Micrion Co.** and continues developed ICP source for **FEI EM portfolio**.

The electron cyclotron resonance (ECR)

Richard Geller at **CEA** in **1960s** developed ECR. **Louis-Jacques M. Celona** further improved ECR in the **1970s** at **CEA** and in the **1980s** at **ORNL**.

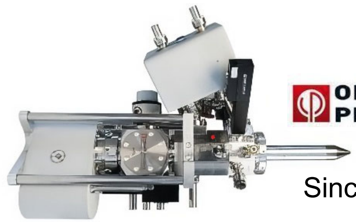


CP473, *Heavy Ion Accelerator Technology: Eighth International Conference*, edited by Kenneth W. Shepard © 1999 The American Institute of Physics 1-56396-806-1/99/$15.00

Plasma FIB technology based on ECR

A. Edmonds, A. Schenkel, and C.E. Wickersham developed ECR in the **1990s** at **LBNL** and **CIMAP** to ionize and accelerate **Xe+** and **Ar+**.

CIMAP BERKELEY LAB



ORSAY PHYSICS

Since ~ 1998

Historical brief on development of focussed beams

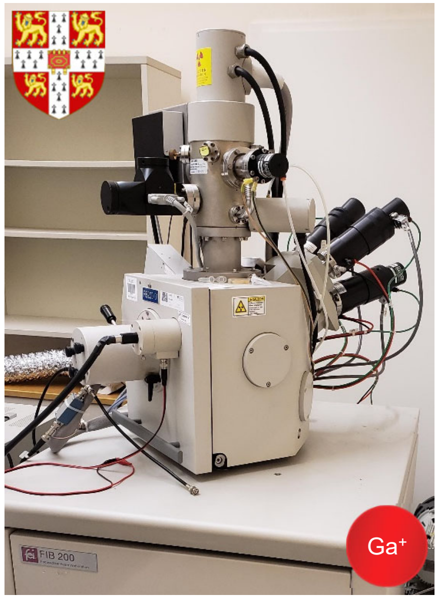
Thermo Fisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

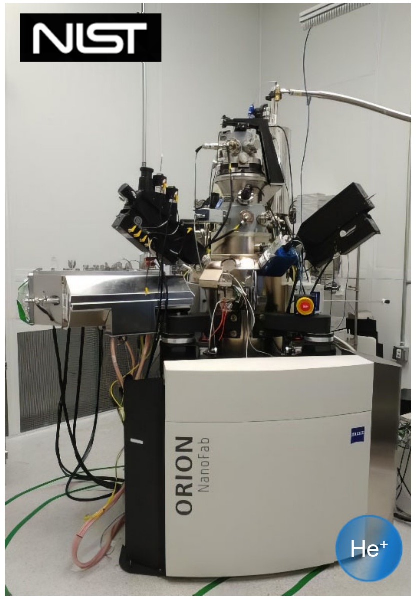
1914 1917 1947 1951 1960 1961 1989 1997 2004 2012

FEI FIB (Ga^+) workstation (1989)

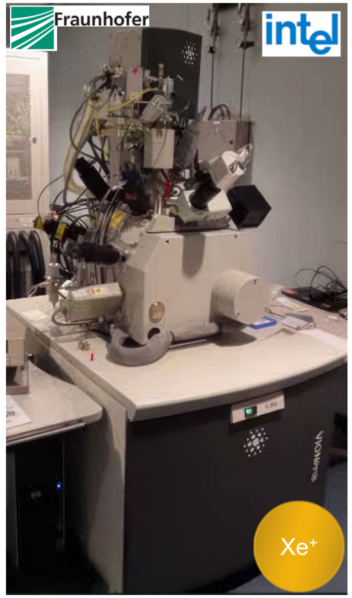
Micrion 9500 FIB (Ga^+) workstation (1994)



FEI FIB 200 (Ga^+) workstation (1995)

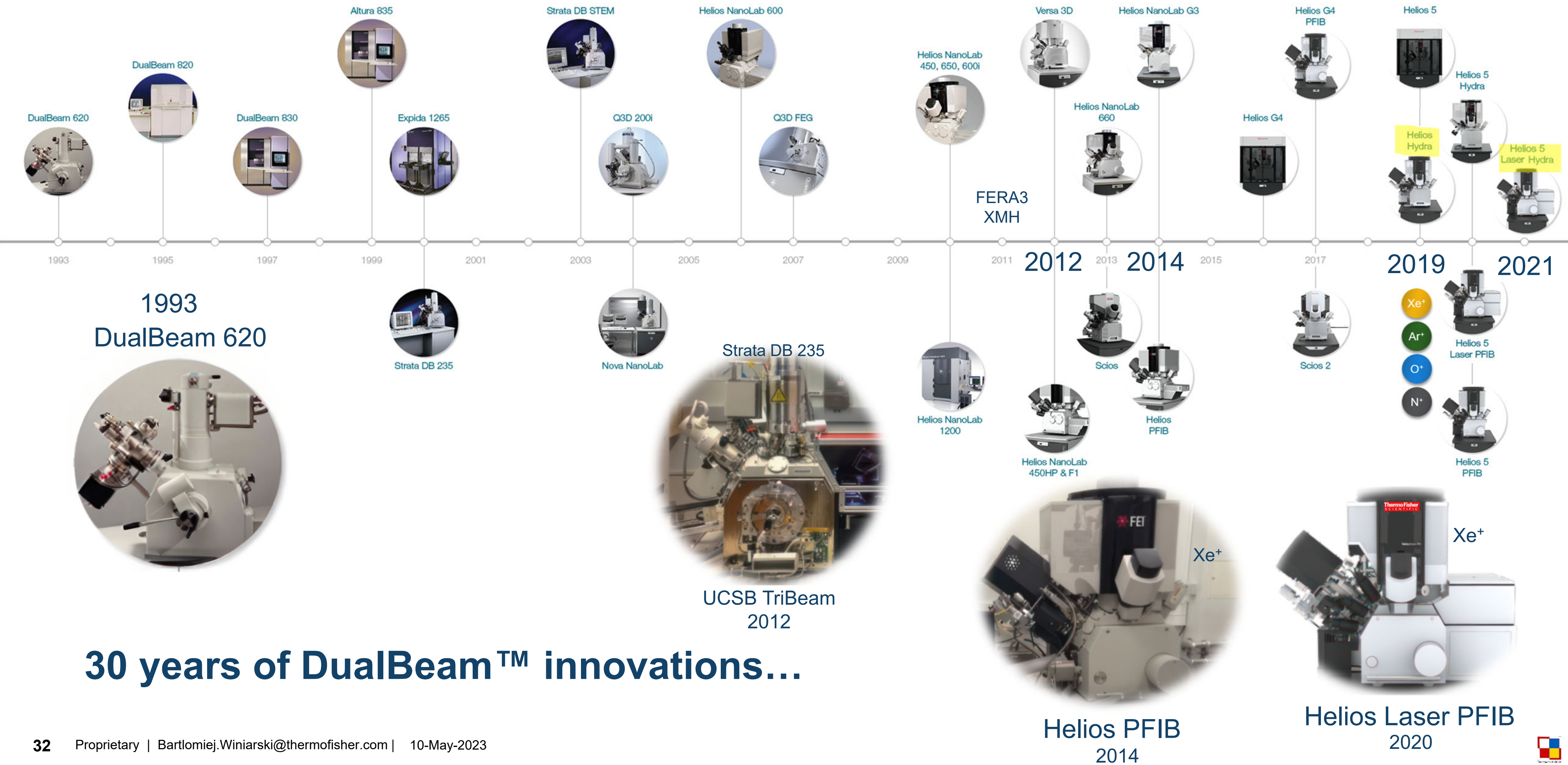


Zeiss Orion (He^+) workstation (2008)



FEI Vion (Xe^+) workstation (~2011)

Historical brief on development of focussed beams



30 years of DualBeam™ innovations…

Historical brief on development of focussed beams

ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1914 1917 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1947 1949 1951 1951 1956 1960 1961 1965 1969 1985 1989 1997 2004 2012

Today

Thermo Scientific Helios

Zeiss Crossbeam 550

Tescan Solaris

Thermo Scientific Helios Hydra

Tescan Solaris X

Hitachi Ethos NX5000

JEOL JIB-4700F

Raith Velion

Historical brief on development of focussed beams

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

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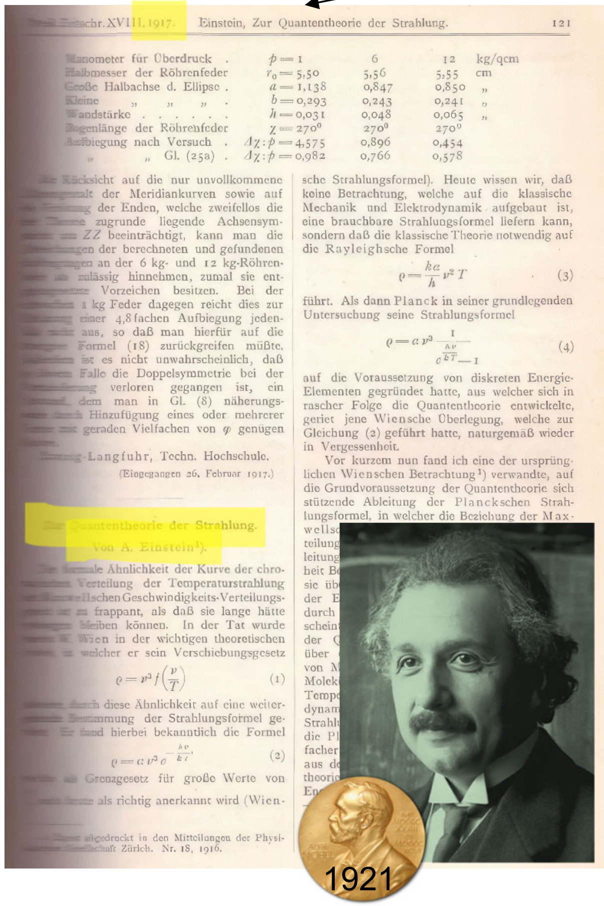
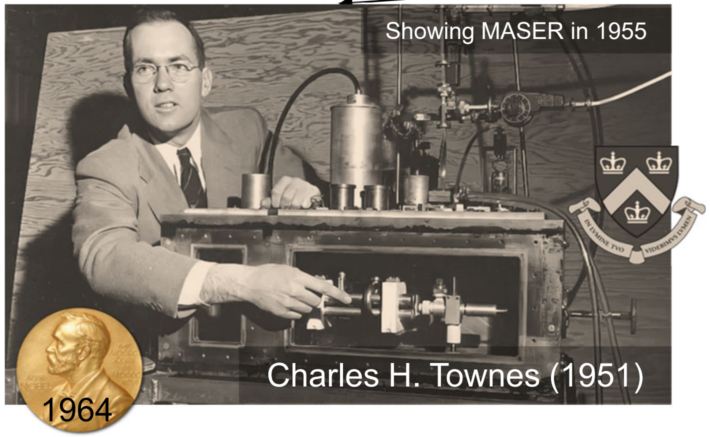
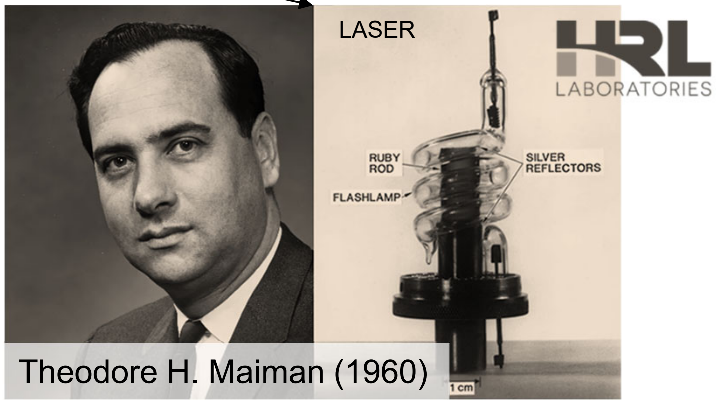
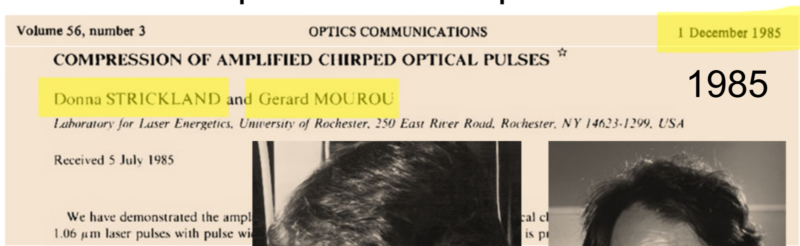






Laser spectroscopy

Chirped Pulse Amplification



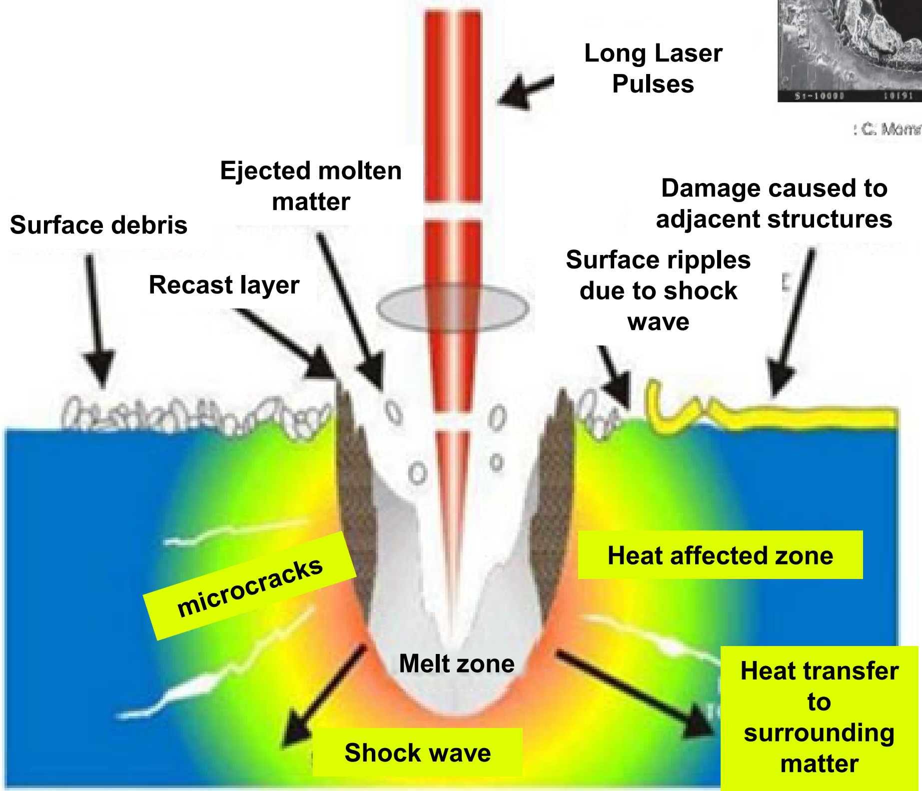
D. Strickland
G. Mourou

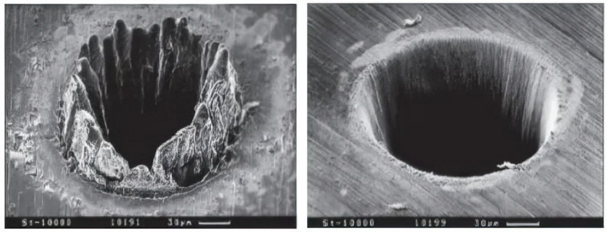
Historical brief on development of focussed beams

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

1914 1917 1947 1951 1960 1961 1985

LPL

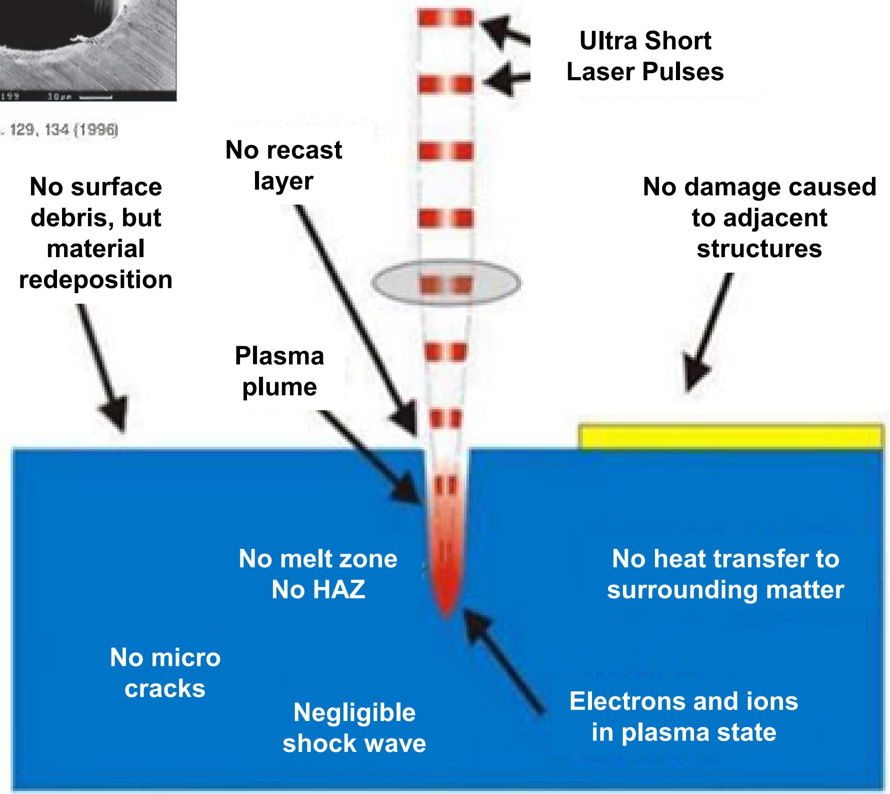




: C. Momma, A. Tunnermann et al., Opt. Commun. 129, 134 (1996)

Based on: Hamad A.H. High Energy and Short Pulse Lasers, IntechOpen, 2016

USPL



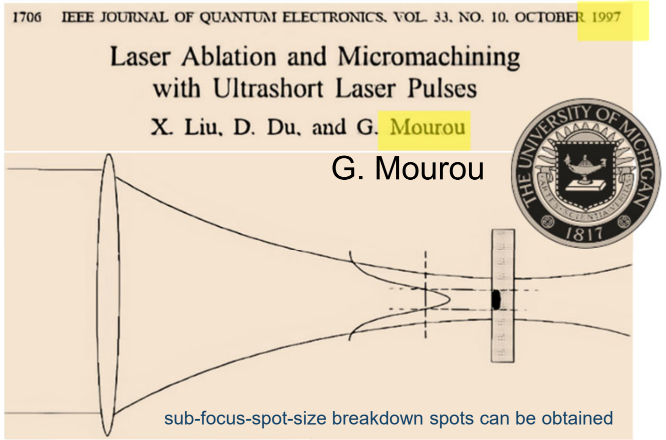
Historical brief on development of focussed beams

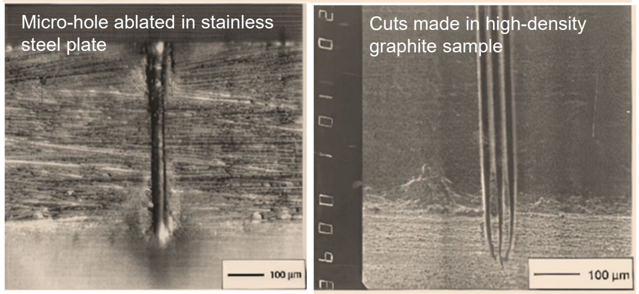
G. Mourou, T.M. Pollock

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

1914
1917 1947 1951 1960 1961 1985 1997 2004 2012

1997





Ti:sapphire laser pulses of 60 fs, wavelength at 790 nm

Laser used: J. V. Rudd, G. Korn, S. Kane, J. Squier, G. Mourou, and P. Bado, "Chirped-pulse amplification of 55-fs pulses at a 1-kHz repetition rate in a Ti:Al_2O_3 regenerative amplifier," *Opt. Lett.*, vol. 18, pp. 2044–2046, 1993.

G. Mourou, T.M. Pollock (2004)

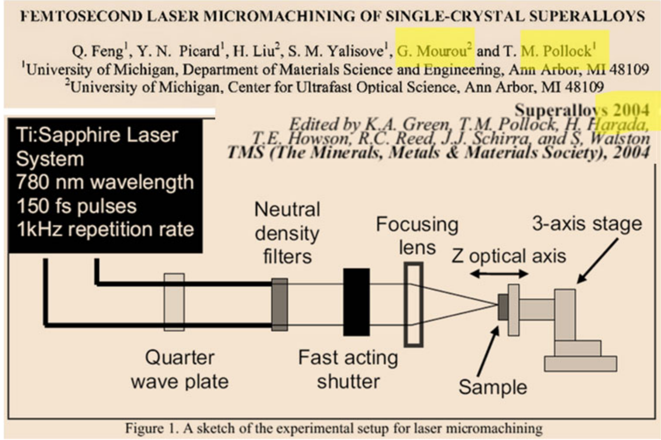
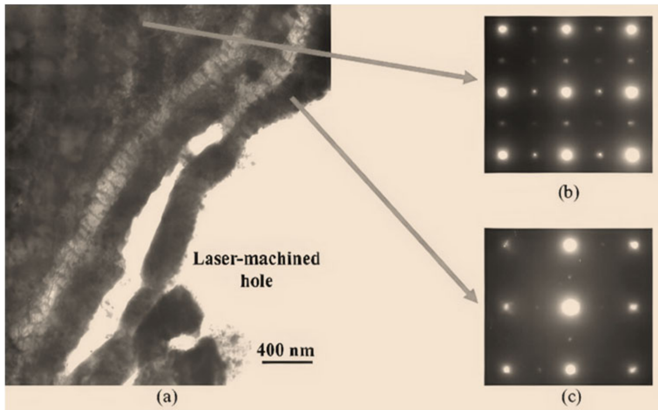
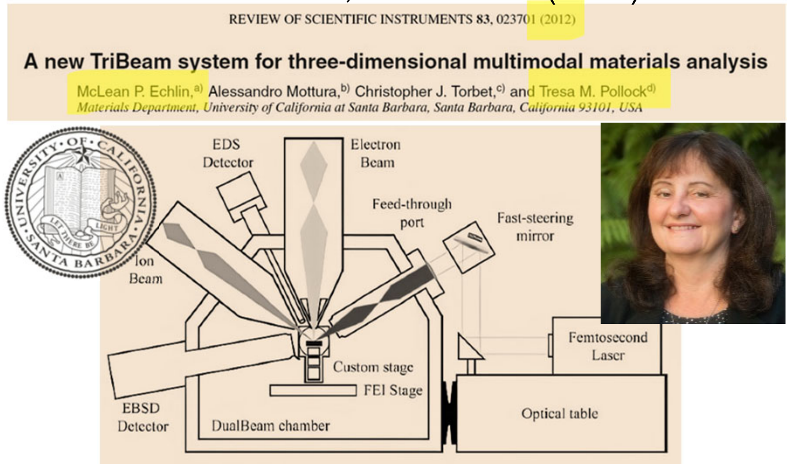


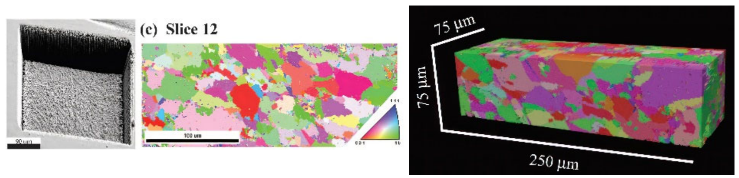
Figure 1. A sketch of the experimental setup for laser micromachining



Single crystal alloy MK-4. (a) bright-field TEM, (b) SAD of non-ablated area, (c) SAD of ablated area

McLean Echlin, T.M. Pollock (2012)





TriBeam femtosecond laser serial section 3D reconstruction of polycrystalline nickel.

Historical brief on development of focussed beams

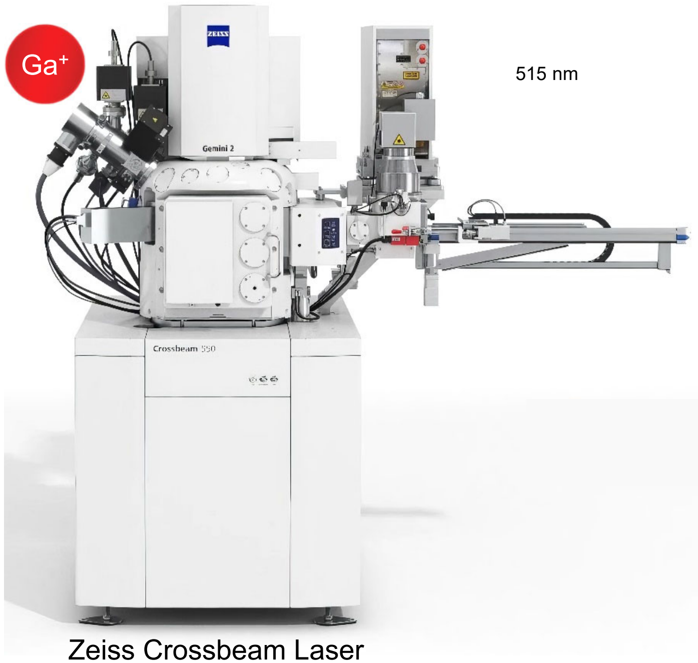
ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

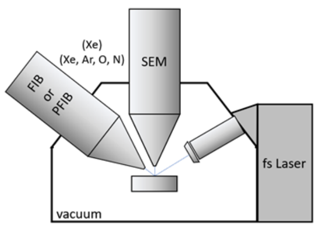
1914 1917 1947 1951 1960 1961 1985 1997 2004 2012 Today

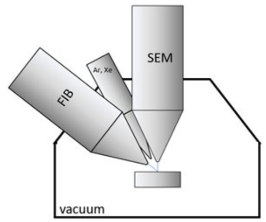
Thermo Scientific Helios Laser Hydra

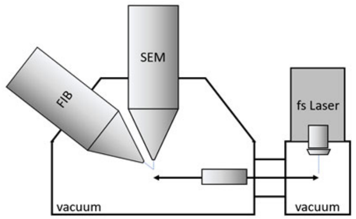
Hitachi Ethos NX5000



Zeiss Crossbeam Laser







Focussed beams & EBSD method

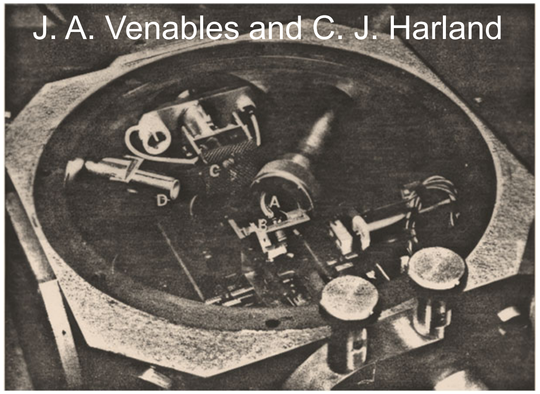
Focussed beams & EBSD method

ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

1914
1917 1947 1951 1960 1961 1985 1997 2004 2012

“EBSP” in SEM was born (1973)



J. A. Venables and C. J. Harland (1973), *Philosophical Magazine*, **2**, 1193-1200.

Stereoscan MK1

J. A. Venables

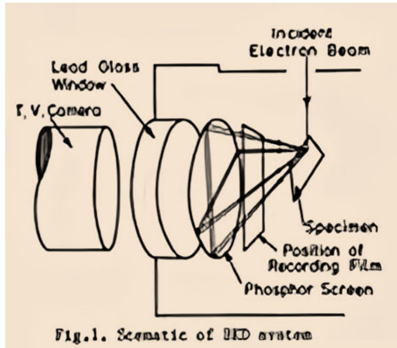
“EBSP” in SEM recoded on photo film (1984)

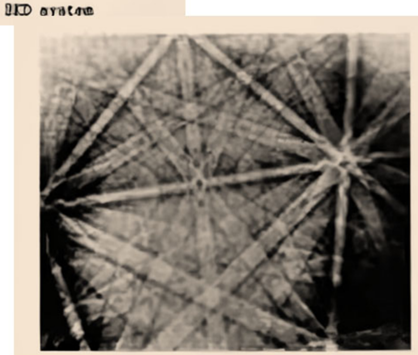
David J. Dingley (1984) „Diffraction From Sub-Micron Areas Using Electron Backscattering In A Scanning Electron Microscope”, *Scanning Electron Microscopy*, **11**, 569-575

David J. Dingley at ICOTOM18 (2017): https://www.youtube.com/watch?v=dLCHGGXdMTo

“EBSP” in SEM recoded on TV camera (1987)

D.J. Dingley, M. Longdon, J. Wienbren and J. Alderman (1987) „On-line Analysis of Electron Backscatter Diffraction Patterns, Texture Analysis of Polysilicon”, *Scanning Electron Microscopy*, **11**, 451-456.





1st commercial system for SEM

Focussed beams & EBSD method

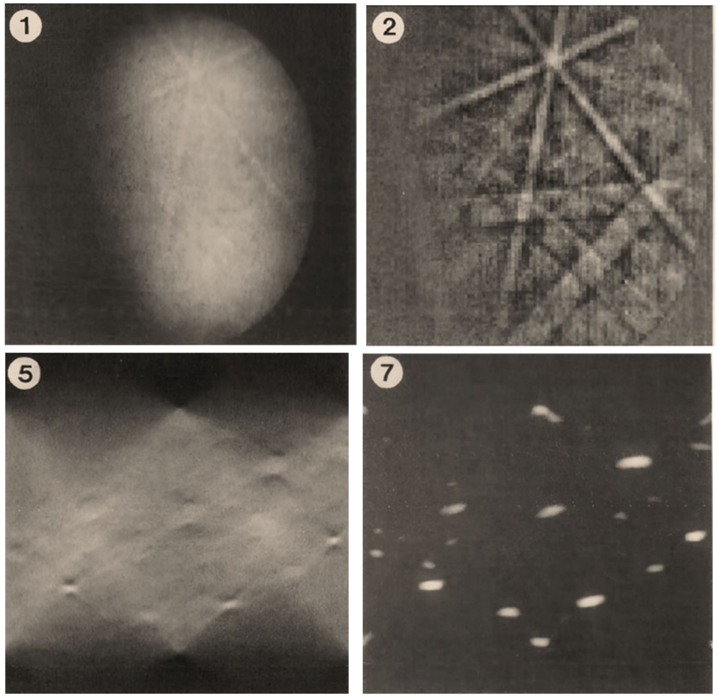
ThermoFisher SCIENTIFIC

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

1914 1917 1947 1951 1960 1961 1985 1997 2004 2012

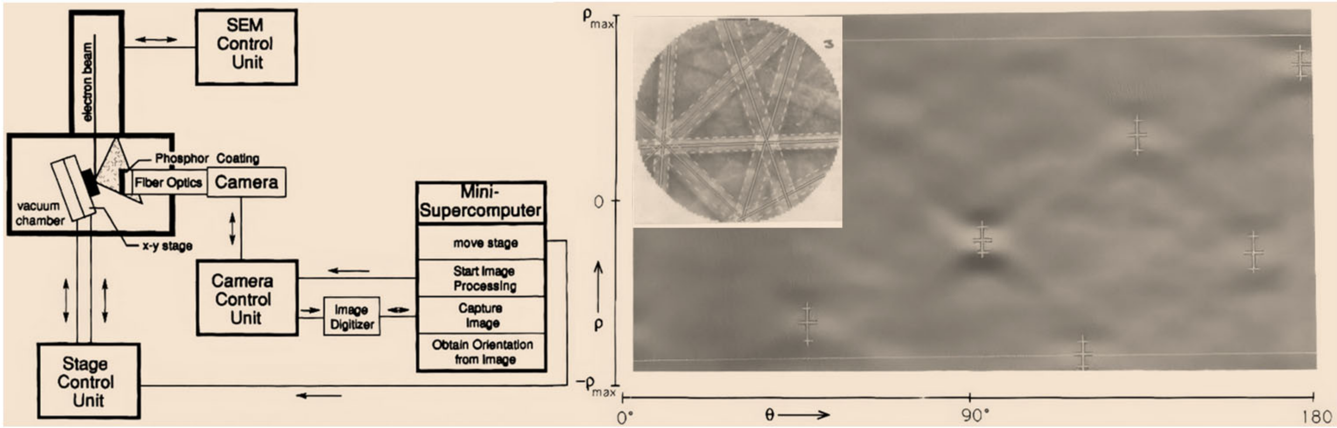
Hough transformed used (1992)

N. C. Krieger-Lassen, **K. Conradsen and D. Juul-Jensen (1992)** „Image Processing Procedures for Analysis of Electron Back Scattering Patterns", Scanning Microscopy, 6, 115-121.



Full automation of EBSP processing (1993)

K. Kunze, S. I. Wright, B. L. Adams, and D. J. Dingley, (1993) „Advances in Automatic EBSP Single Orientation Measurements", *Textures Microstructures,*20, 41-54.



"OIM"

Phase identifications (1986), CCD-based EBSD detector (1993)

D. J. Dingley and K. Baba-Kishi (1986) „Use of Electron Backscatter Diffraction Patterns for Determination of Crystal Symmetry Elements", *Scanning Electron Microscopy*, **II**, 383-391.

D. J. Dingley, R. Mackenzie and K. Baba-Kishi (1989) „Application of Backscatter Kikuchi Diffraction for Phase Identification and Crystal Orientation Measurements in Materials", *Microbeam Analysis*, ed. P.E. Russell, San Francisco Press, 435-436.

J. R. Michael and R. P. Goehner (1993) „Crystallographic Phase Identification in the Scanning Electron Microscope: Backscattered Electron Kikuchi Patterns Imaged with a CCD-Based Detector", MSA Bulletin, 23, 168-175.

N.H. Schmidt (1990): "HKL Channel" commercial system

CMOS-based detector in 2017

Focussed beams & EBSD method

ThermoFisher SCIENTIFIC

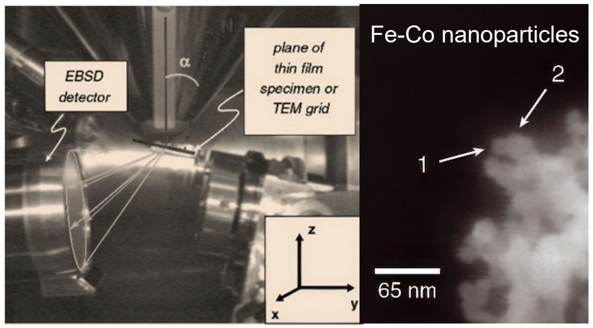
1801 1831 1865 1895 1897 1912 1914 1917 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1947 1949 1951 1951 1956 1960 1961 1965 1969 1985 1997 2004 2012

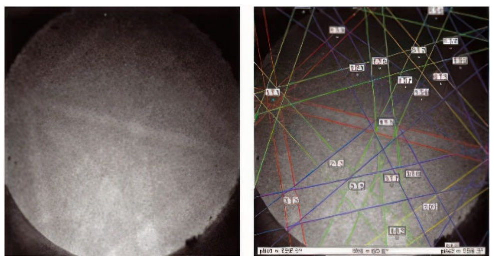
t-EBSD (TKD) in SEM was born (2012)

Journal of Microscopy, Vol. 245, Pt 3 2012, pp. 245–251
Received 11 July 2011; accepted 9 October 2011

Transmission EBSD from 10 nm domains in a scanning electron microscope

R.R. KELLER & R.H. GEISS
Materials Reliability Division, National Institute of Standards and Technology, Boulder, CO 80305, U.S.A.





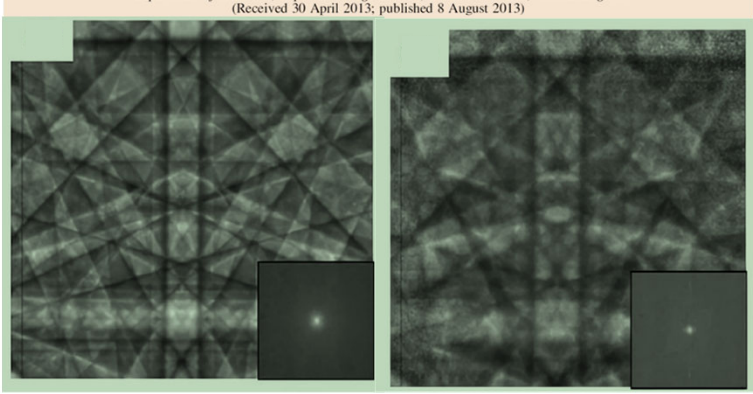
Direct detection of EBSP (2013)

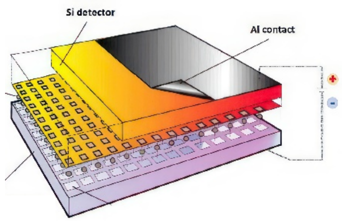
Angus J. Wilkinson

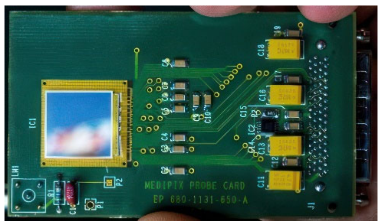
PRL 111, 065506 (2013) PHYSICAL REVIEW LETTERS week ending 9 AUGUST 2013

Direct Detection of Electron Backscatter Diffraction Patterns

Angus J. Wilkinson,[1] Grigore Moldovan,[1,*] T. Benjamin Britton,[1,3] Angus Bewick,[2] Robert Clough,[1] and Angus I. Kirkland[1]
[1]*Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH, United Kingdom*
[2]*Oxford Instruments, Halifax Road, High Wycombe HP12 3SE, United Kingdom*
[3]*Department of Materials, Imperial College London, London SW7 2AZ, United Kingdom*
(Received 30 April 2013; published 8 August 2013)

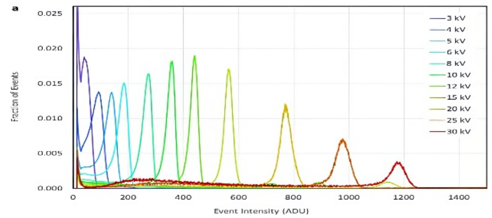






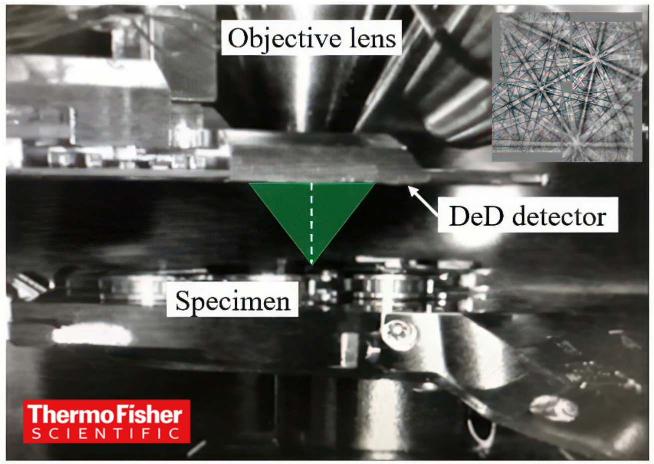
Energy-filtering DeD EBSD/TKD (2020)

Bammes, B. and **R. Bilhorn**, A New Energy-Filtering EBSD/TKD Direct Detector. Microscopy and Microanalysis, **2020**. 26(2): p. 1186.

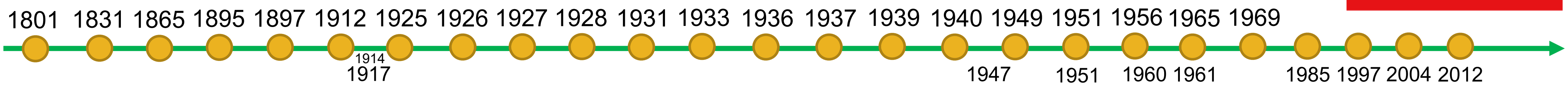


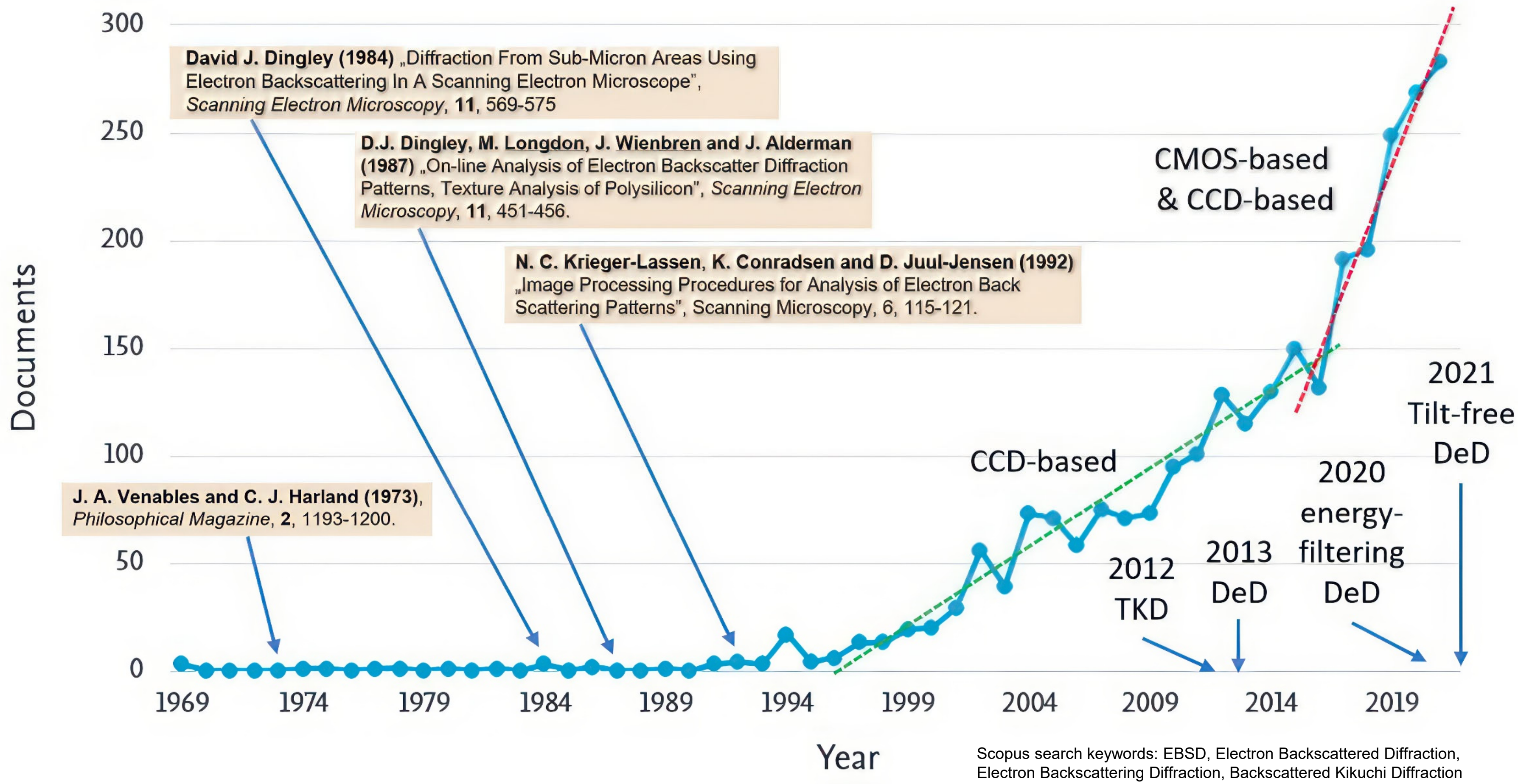
Tilt-free DeD EBSD (2021)

Marshall, A.L., et al., The EBSD spatial resolution of a Timepix-based detector in a tilt-free geometry. Ultramicroscopy, **2021**. 226: p. 113294.



Focussed beams & EBSD method

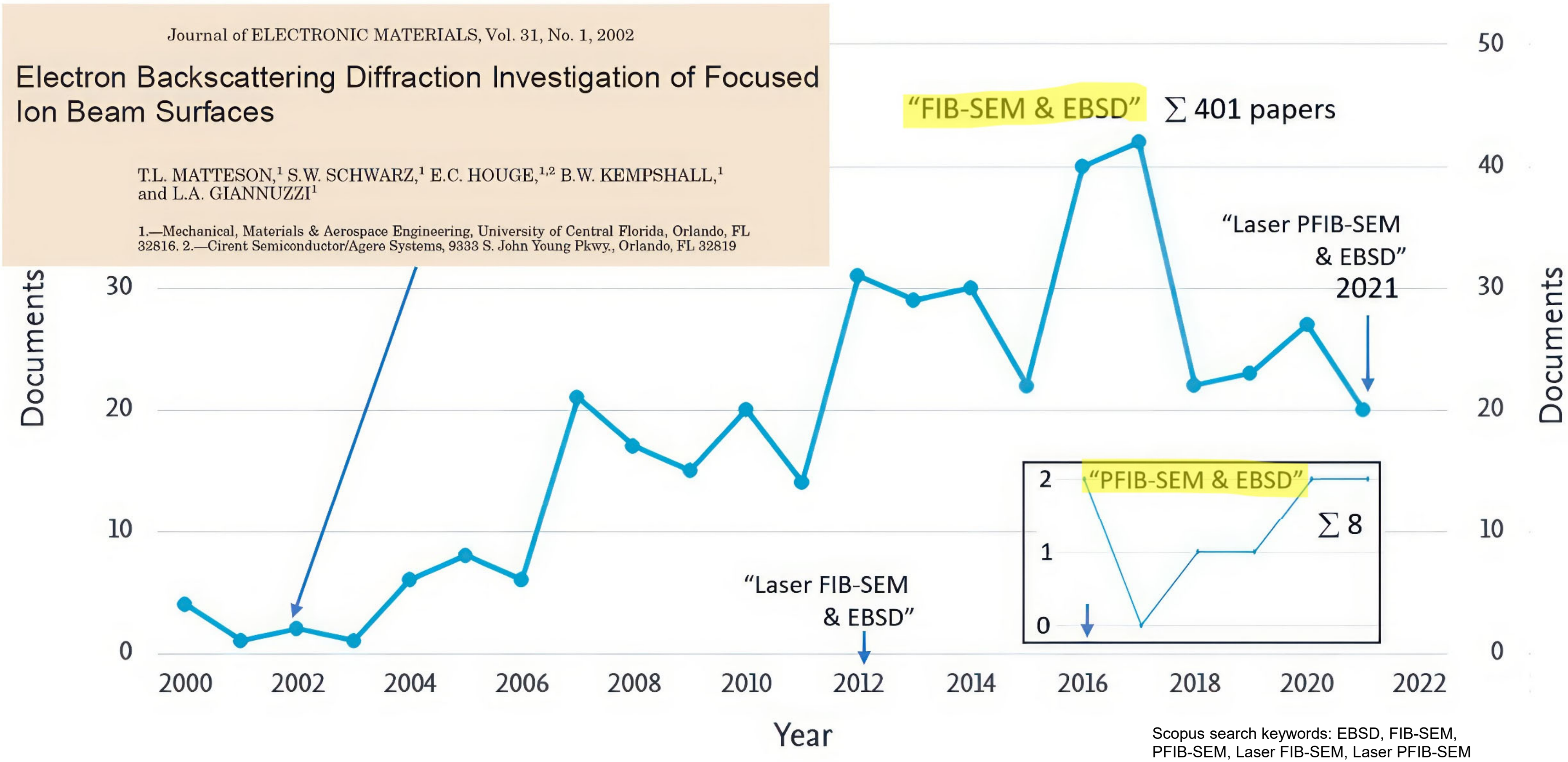




Scopus search keywords: EBSD, Electron Backscattered Diffraction, Electron Backscattering Diffraction, Backscattered Kikuchi Diffraction

Focussed beams & EBSD method

1801 1831 1865 1895 1897 1912 1914 1917 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1947 1949 1951 1951 1956 1960 1961 1965 1969 1985 1997 2004 2012

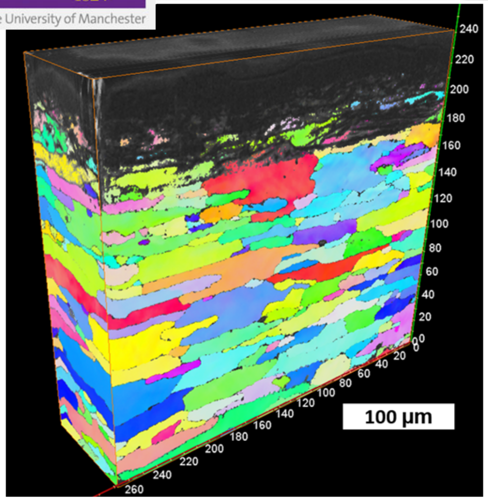


Scopus search keywords: EBSD, FIB-SEM, PFIB-SEM, Laser FIB-SEM, Laser PFIB-SEM

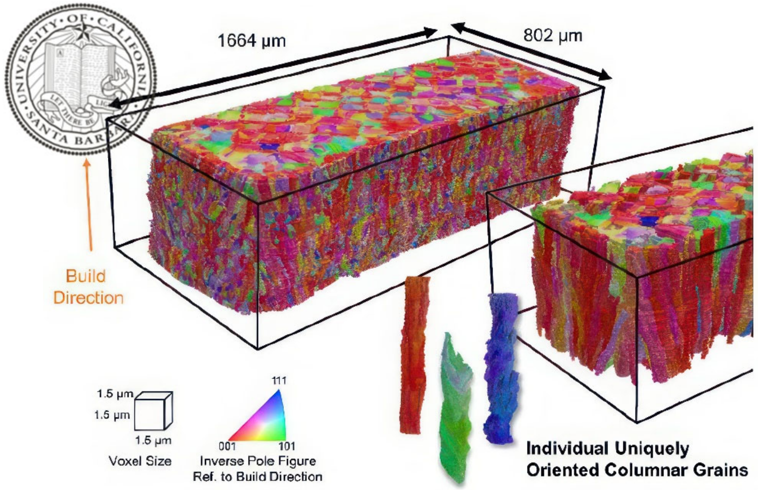
Focussed beams & EBSD method

1801 1831 1865 1895 1897 1912 1925 1926 1927 1928 1931 1933 1936 1937 1939 1940 1949 1951 1956 1965 1969

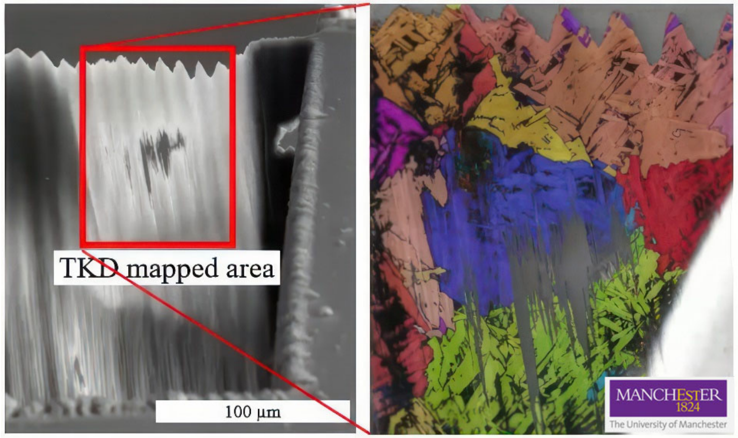
1914
1917 1947 1951 1960 1961 1985 1997 2004 2012



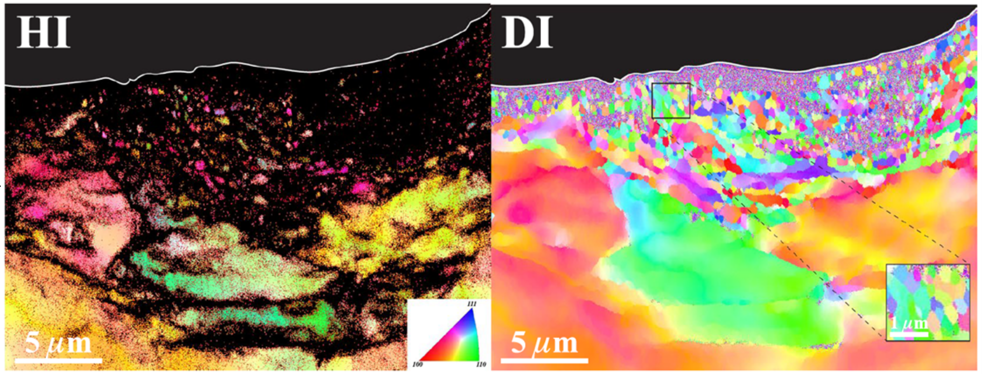
Winiarski, B., P.J. Withers, and T.L. Burnett. *Xe+ Plasma FIB Milling and Lift-out Approach for Site- Specific Preparation of Large Volume Blocks for 3D-EBSD.* in ***2016*** *Microscopy and Microanalysis Conference*. 2016. Columbus, OH: Cambridge Press.



Echlin, M.L.P., Polonsky A.T., Lamb J., Geurts. R. Randolph S.J. Botman A., Pollock T.M. , *Recent Developments in Femtosecond Laser-Enabled TriBeam Systems.* JOM, **2021**. **73**(12): p. 4258-4269.

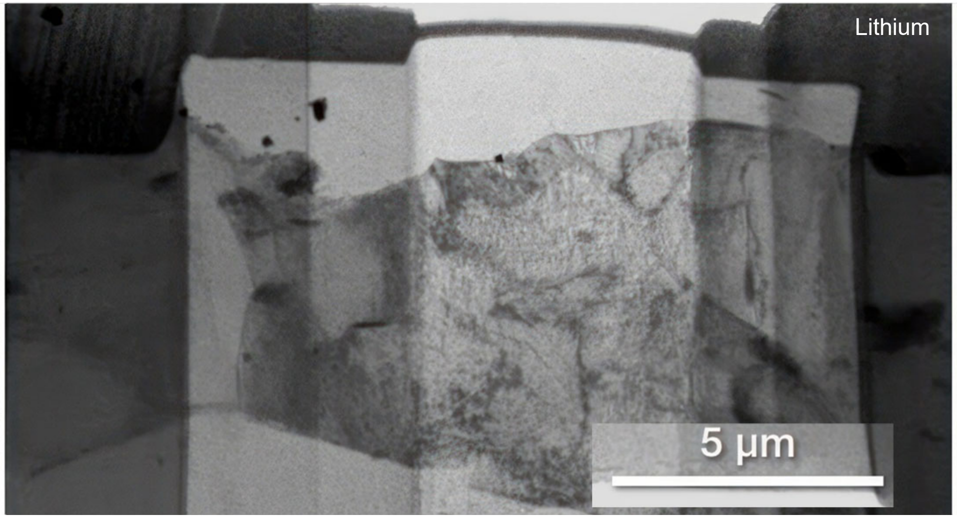


Laser PFIB-SEM prepared TKD lamella of tensile tested Ti64 alloys, Image courtesy: **Dr Ali Gholinia**, The University of Manchester.



S. Singh, Y. Guo, B. Winiarski, T.L. Burnett, P.J. Withers & M. De Graef. *High resolution low kV EBSD of heavily deformed and nanocrystalline Aluminium by dictionary-based indexing.* Scientific Reports 8, (**2018**), 10991

Current and future directions



Current and further directions

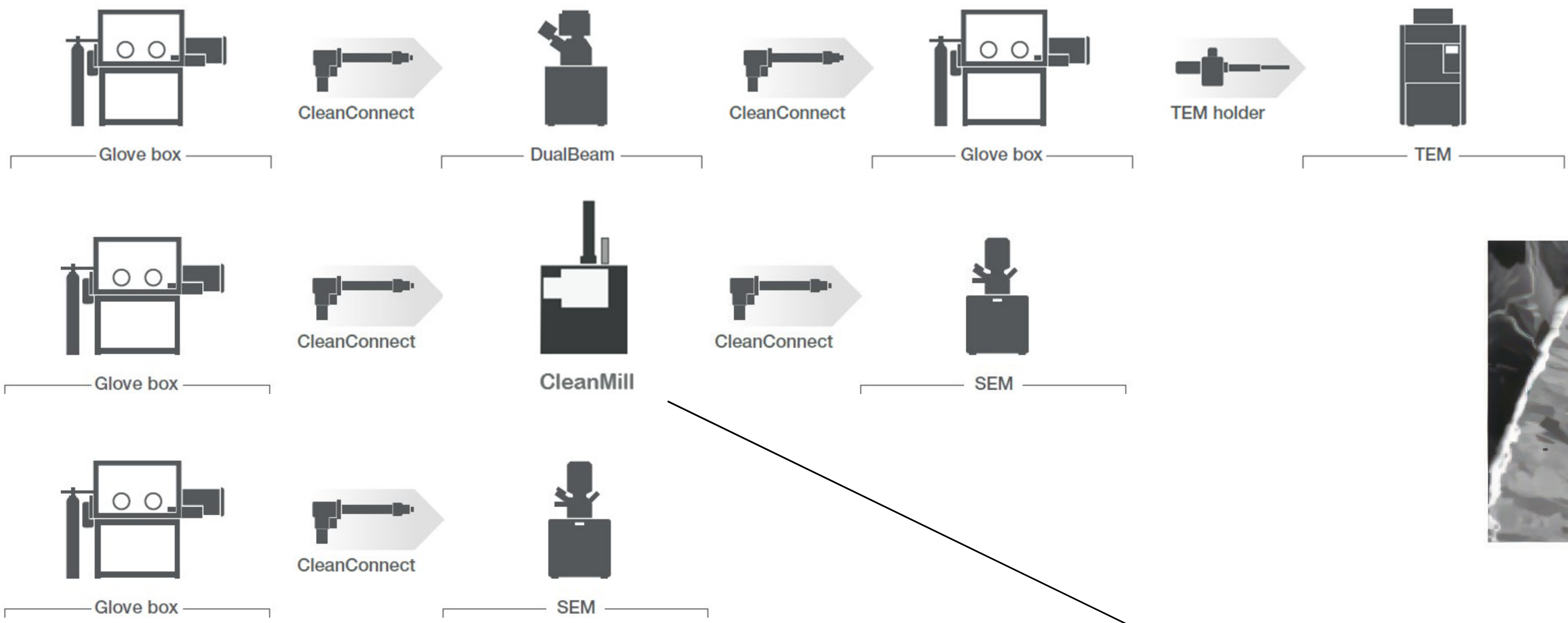
(the list is not exhaustive)

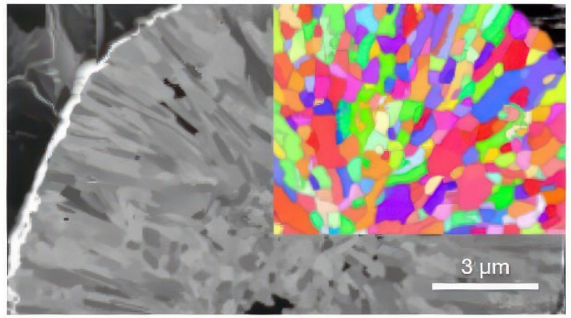
- DeD EBSD/TKD
- DeD energy filtering EBSD/TKD
- Increasing the speed of pattern collection
  - faster more sensitive hardware
  - use of sparse pixels of DeD detectors
- iCHORD & eCHORD
- Smart sparse sampling
- Use of ML and AI in EM, e.g. live image processing, microscope auto alignments, etc.
- Full integration of detectors (e.g. ChemiSEM) and techniques (e.g. ECCI on Scios and Helios) with microscopes
- Python API to access functionalities of microscopes
- User friendly workflows and automations
- Correlative microscopy (CM) with more imaging modalities (XPS, LIBS, EPMA, APT, Raman, etc.) and tuned workflows
- EM and CM with full inert gas workflow (Glove box – CT – SDB – TEM)
- Increased demand of cryo-EM in materials science
- etc.

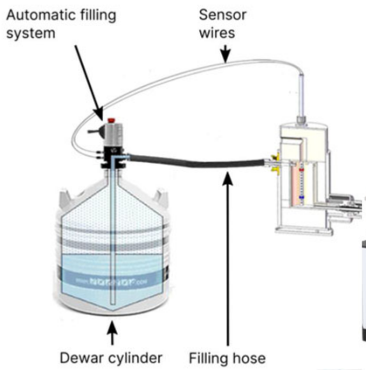
Monday PM | March 20, 2023 TMS2023
2:00 PM
On the Feasibility of Back-scattered or Ion-induced Secondary Electron Imaging to Determine Grain Orientations: *Marc De Graef*[1]; [1]Carnegie Mellon University

Ballistic channelling contrast reference patterns

Surface preparation methods for EBSD

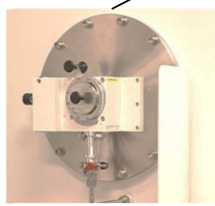






Clean Mill

Up to 16keV



https://www.youtube.com/watch?v=x_Ed6ilrzS4