LA-UR-24-25592
Approved for public release; distribution is unlimited.

Title: History of Scanning Electron Microscopy (SEM)

Author(s): Sickafus, Kurt Edward

Intended for: presentation at LANL internal summer workshop

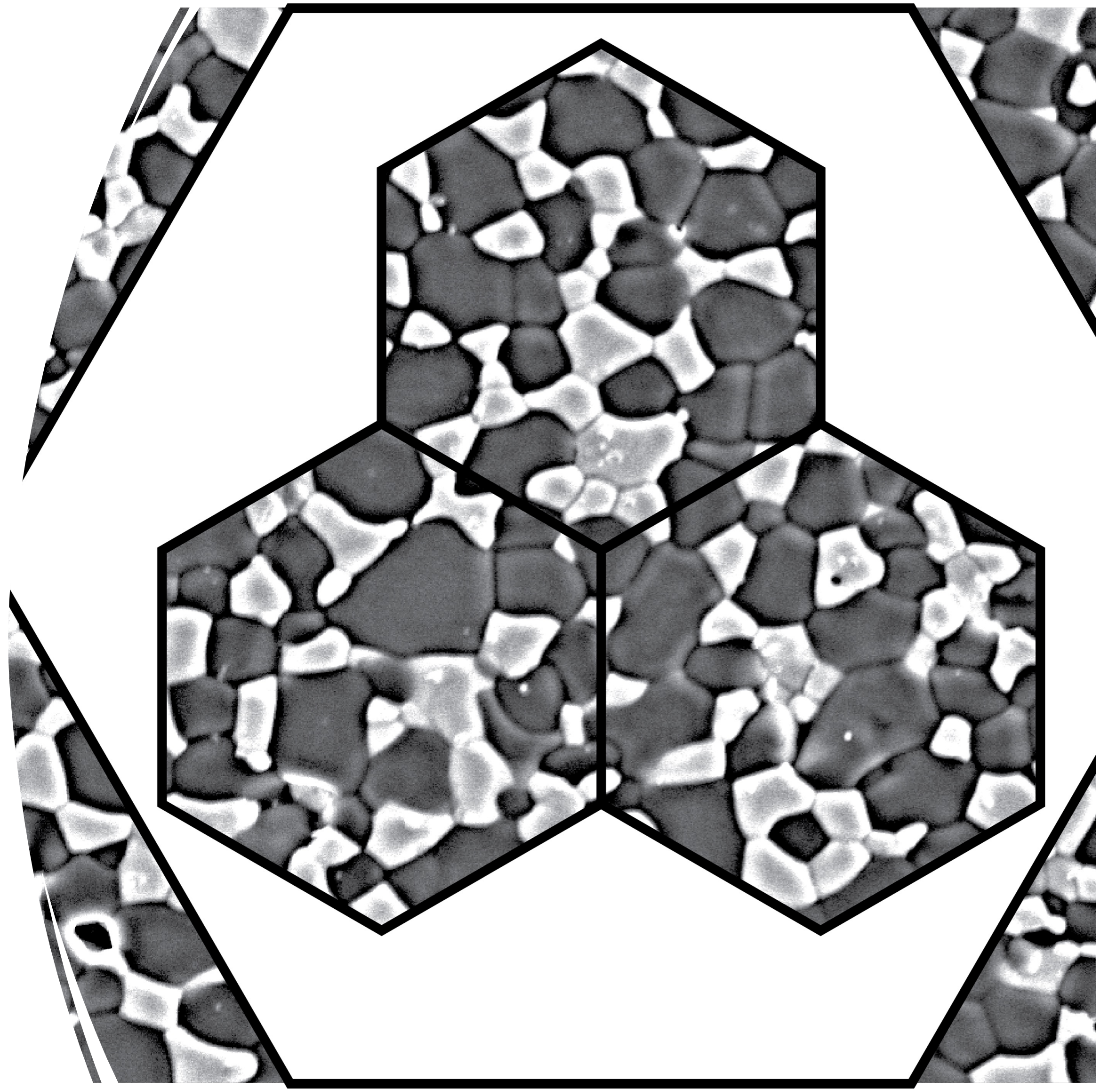
Issued: 2024-06-07

Los Alamos NATIONAL LABORATORY

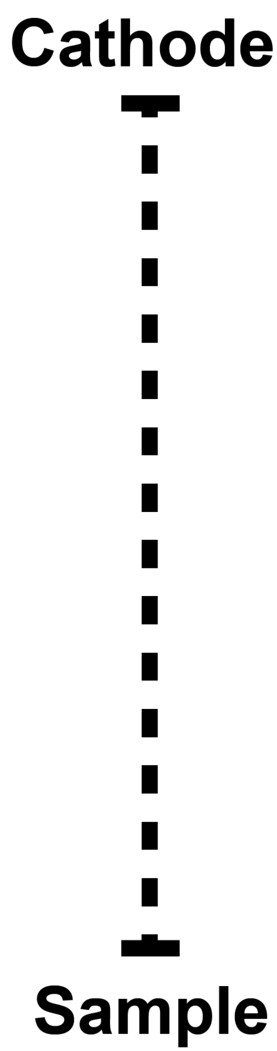
History of Scanning Electron Microscopy (SEM)

Kurt Sickafus, MST-8

June 3, 2024



Grand Canyon Pioneer 50-mi. Endurance Ride,
October 12, 2004



Scanning Electron Microscopy

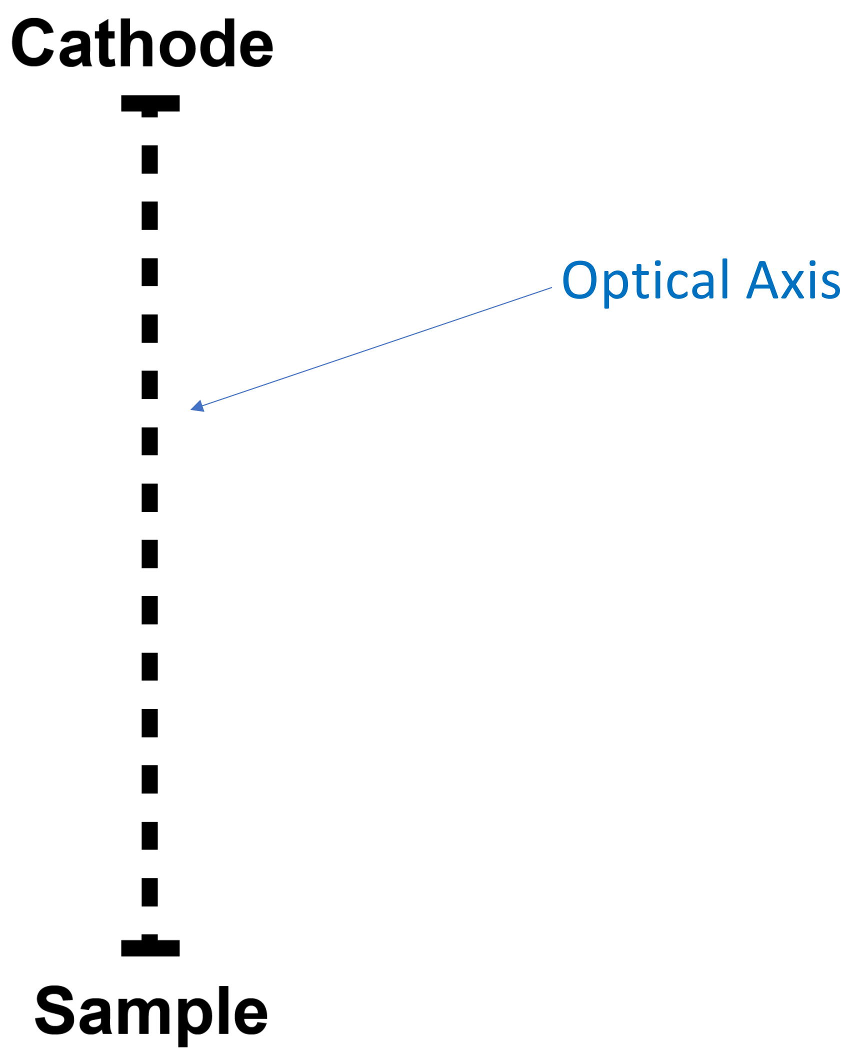
C. W. OATLEY, W. C. NIXON, AND R. F. W. PEASE

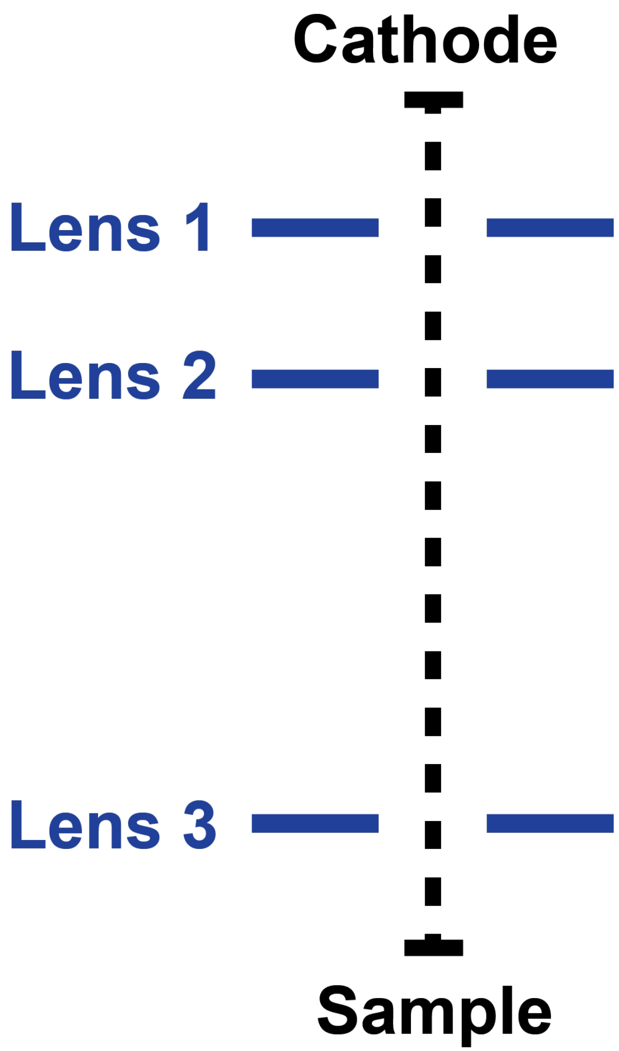
Engineering Department, Cambridge University, Cambridge, England

1965

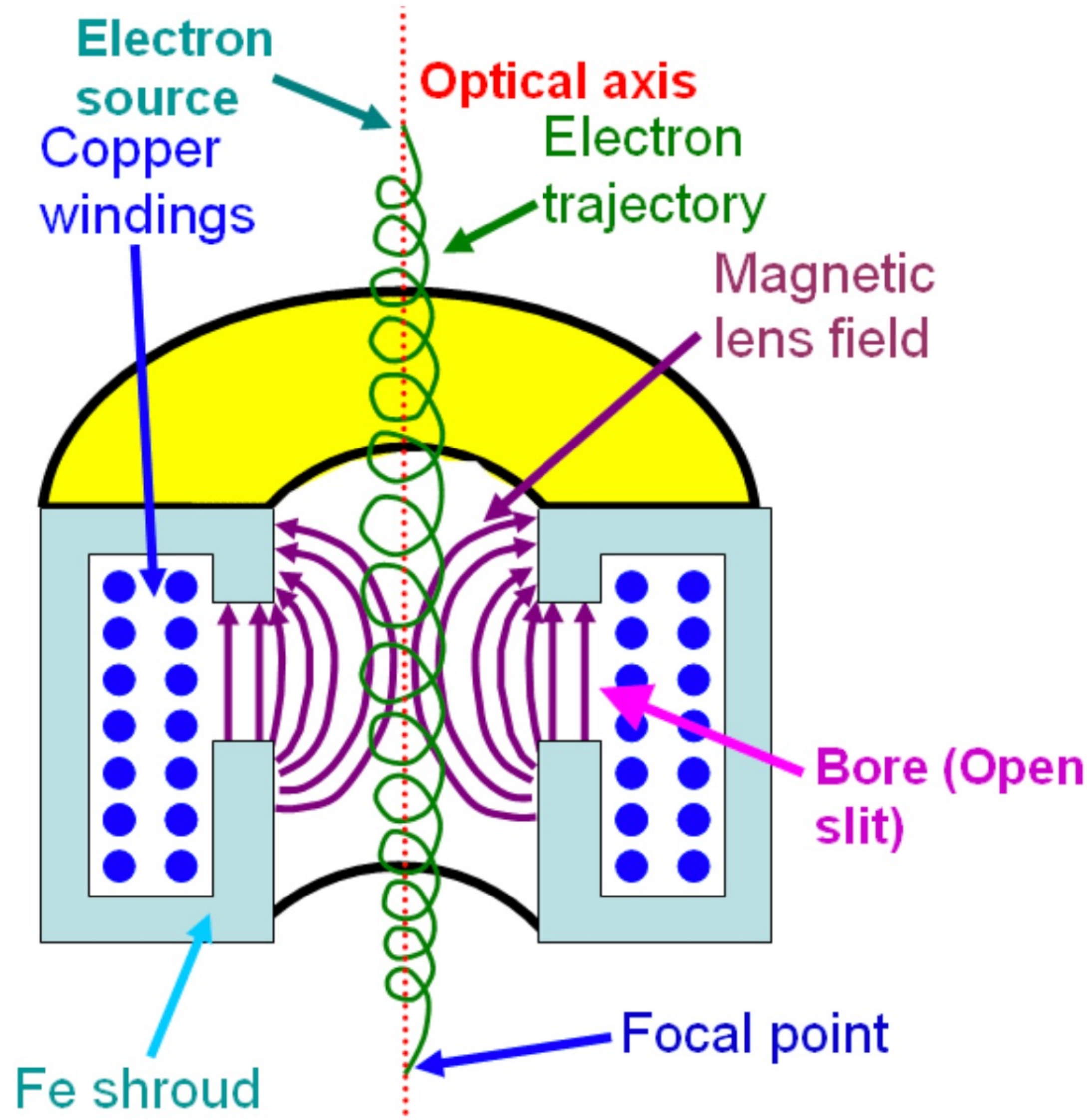
Figure 1 – page 182

C. W. Oatley, W. C. Nixon, and R. F. W. Pease, (1965): "Scanning electron microscopy," Adv. Electron. Electron Phys. **21**, 181–247.

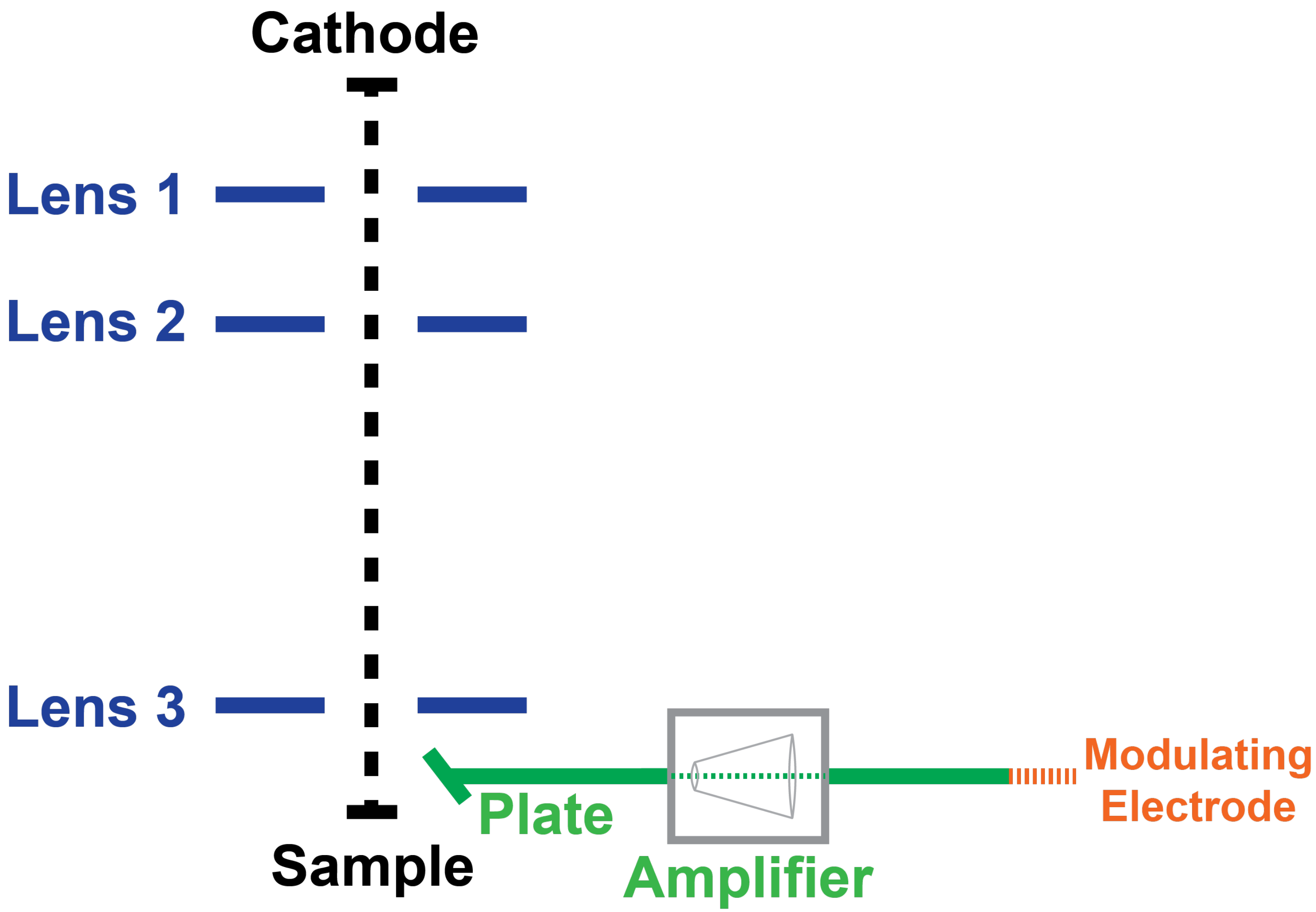

Cathode
Optical Axis
Sample

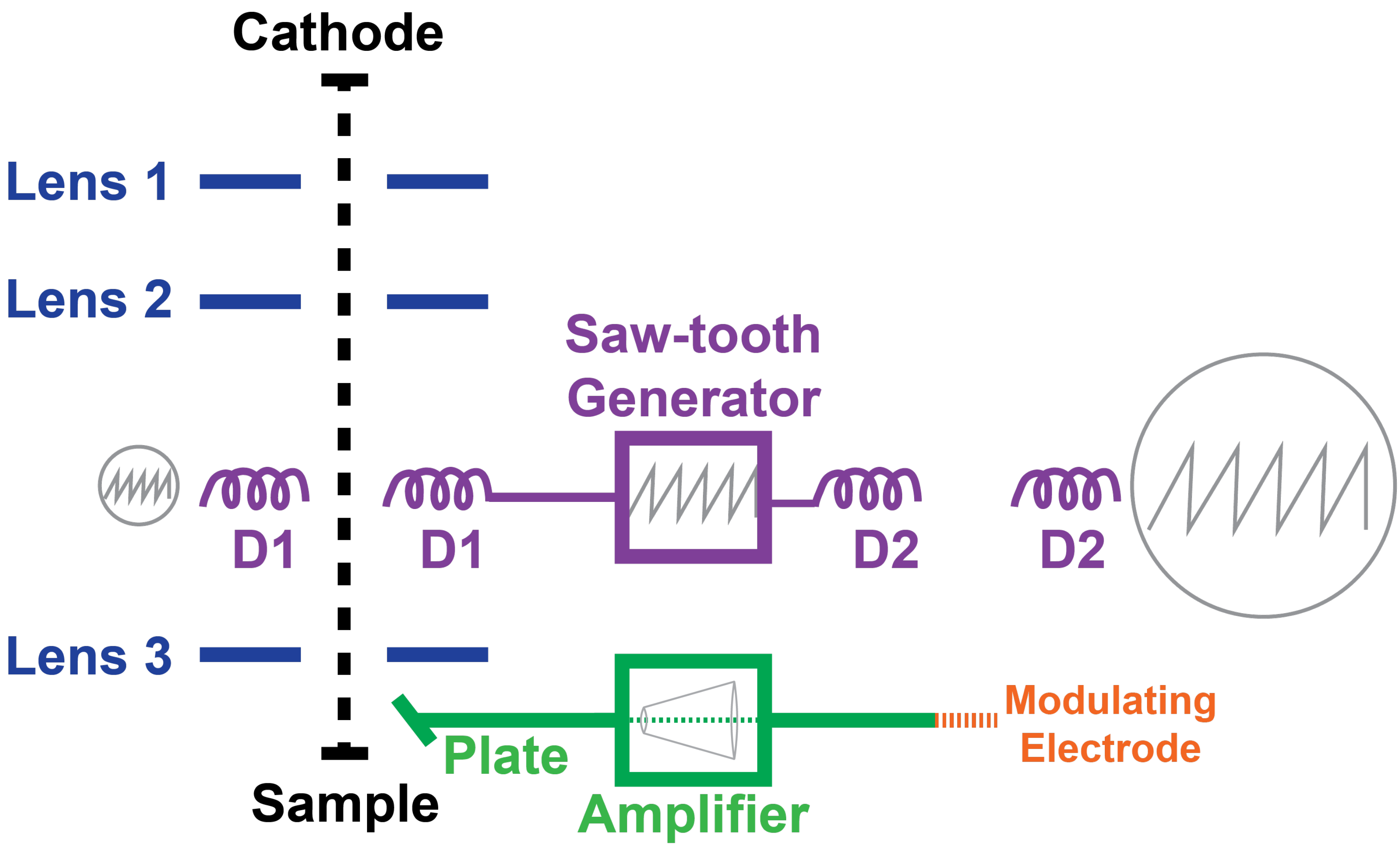

Cathode
Lens 1
Lens 2
Lens 3
Sample

An electromagnetic lens has a *focusing* effect on electrons

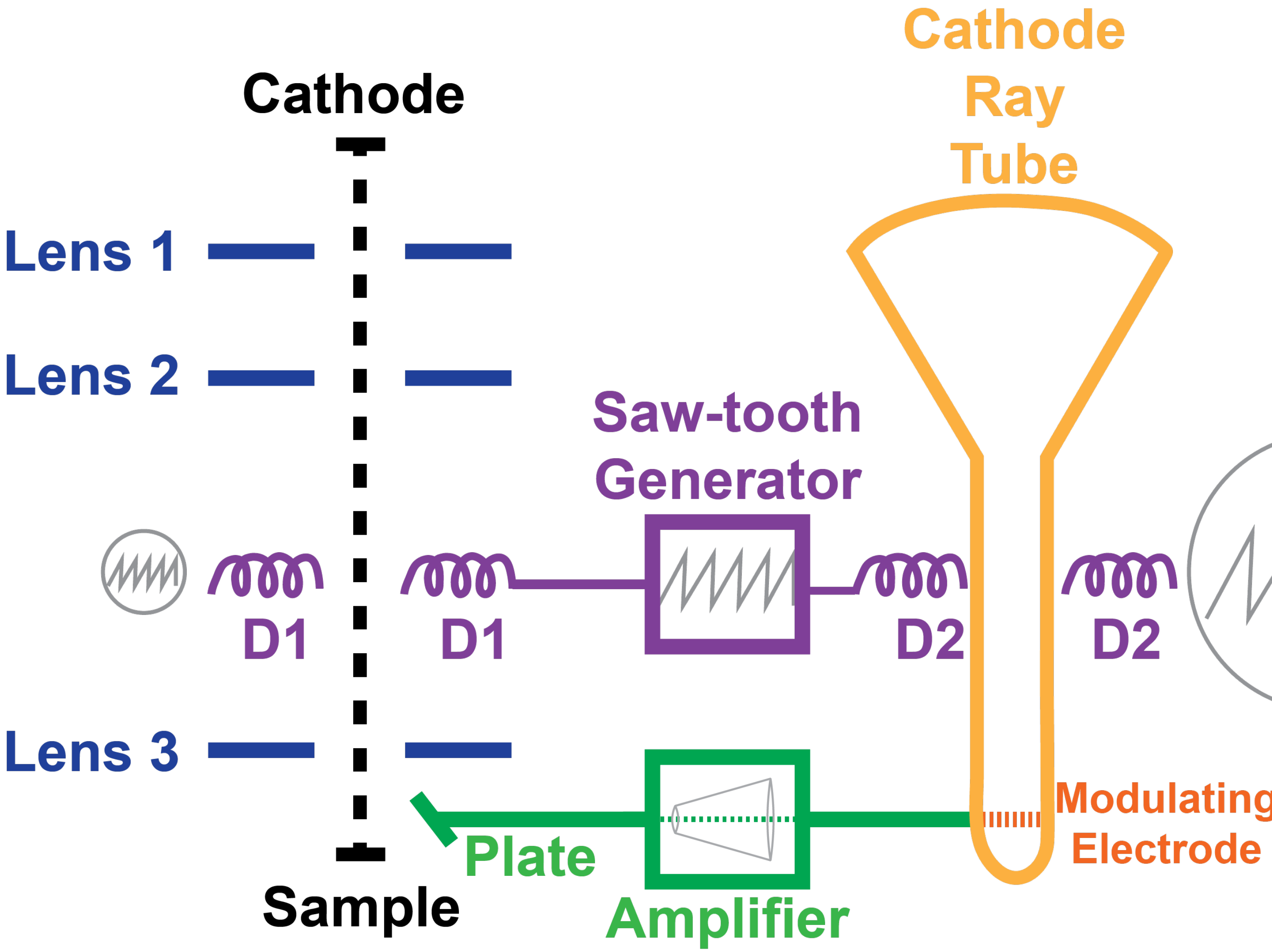


https://www.globalsino.com/EM/page3756.html

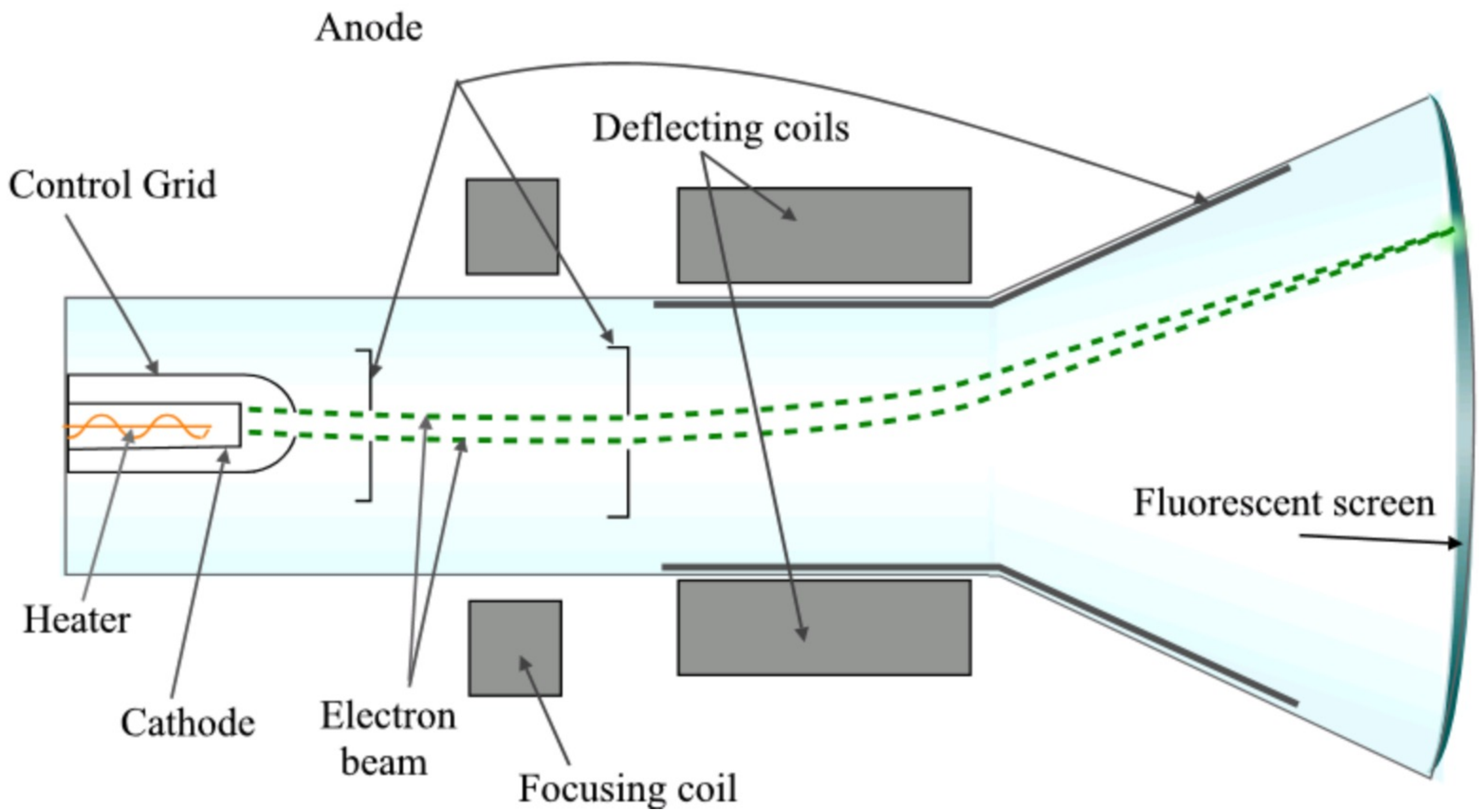

Cathode
Lens 1
Lens 2
Lens 3
Plate
Sample
Amplifier
Modulating
Electrode



Cathode
Lens 1
Lens 2
Saw-tooth
Generator
D1
D1
D2
D2
Lens 3
Modulating
Electrode
Plate
Sample
Amplifier


Cathode
Cathode
Ray
Tube
Lens 1
Lens 2
Saw-tooth
Generator
D1
D1
D2
D2
Lens 3
Modulating
Electrode
Plate
Sample
Amplifier

The Cathode Ray Tube (CRT)



https://en.wikipedia.org/wiki/Cathode-ray_tube#/media/File:Cathode_ray_Tube.PNG

1858-1876 - Discovery of Cathode Rays: What we now call electrons

The term cathode ray (named by Eugen Goldstein) was used to describe electron beams when they were first discovered, before it was understood that what was emitted from the cathode in a vacuum tube was a beam of electrons.

Cathode rays were discovered by Julius Plücker and Johann Wilhelm Hittorf. Hittorf observed that some unknown rays were emitted from the cathode (negative electrode) which could cast shadows on the glowing wall of the tube, indicating the rays were traveling in straight lines.

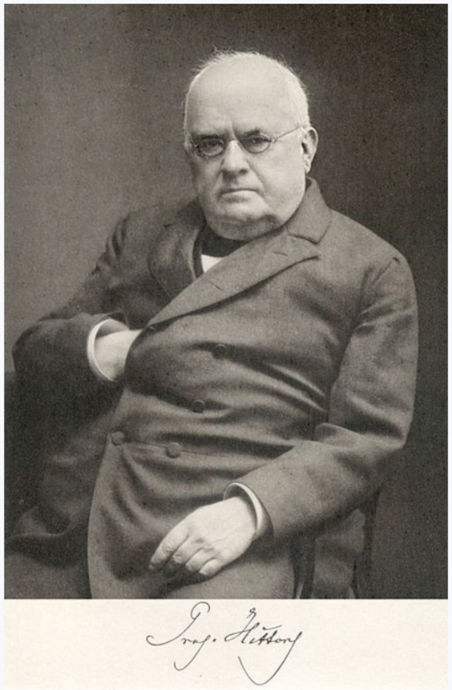
https://en.wikipedia.org/wiki/Cathode-ray_tube#:~:text=The%20term%20cathode%20ray%20was,was%20a%20beam%20of%20electrons.

Julius Plücker

Julius Plücker

https://en.wikipedia.org/wiki/Julius_Pl%C3%BCcker

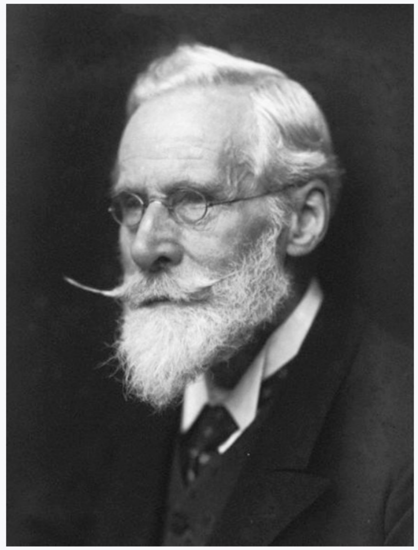
Johann Wilhelm Hittorf



https://en.wikipedia.org/wiki/Johann_Wilhelm_Hittorf

Discoveries that cathode rays can be deflected (bent) by electric and magnetic fields

Sir
William Crookes
OM FRS

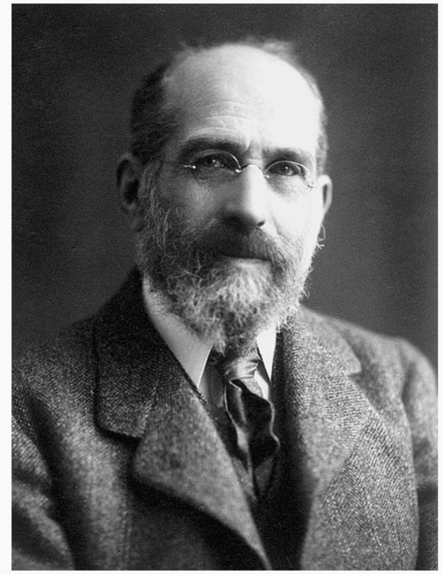


Sir William Crookes in 1906

1879

English physicist and chemist William Crookes investigated cathode rays in 1879 and found that they were bent by a magnetic field; the direction of deflection suggested that they were negatively charged particles.

Sir Arthur Schuster
FRS FRSE



1890

Arthur Schuster demonstrated that cathode rays could be deflected by electric fields.

1897
Sir J. J. Thomson
Credited with the Discovery of the Electron

J. J. Thomson found that the cathode rays can be deflected by an electric field. By balancing the effect of a magnetic field on a cathode-ray beam with an electric field, Thomson was able to show that cathode rays are actually composed of particles.

Sir J. J. Thomson
OM FRS

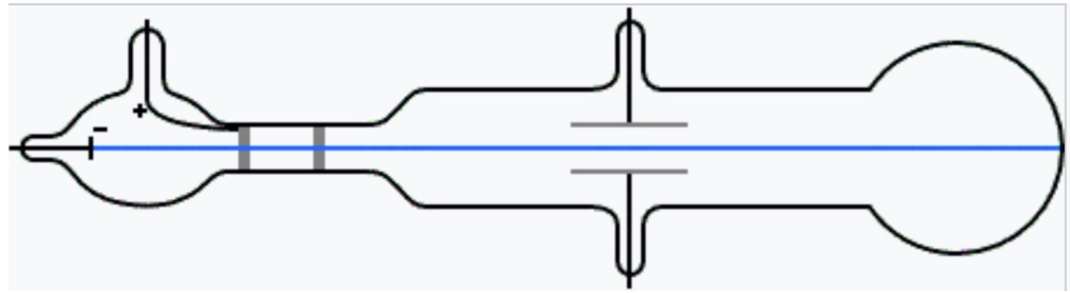
Thomson in 1915

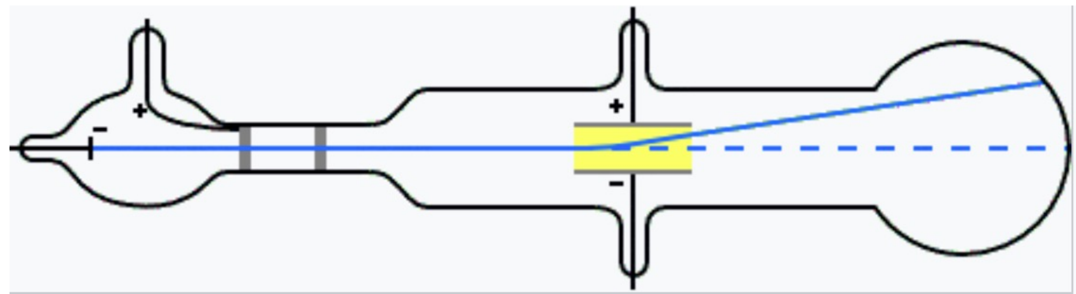
Thomson showed that cathode rays were composed of previously unknown negatively charged particles (now called electrons), which he calculated must have bodies much smaller than atoms and a very large charge-to-mass ratio.

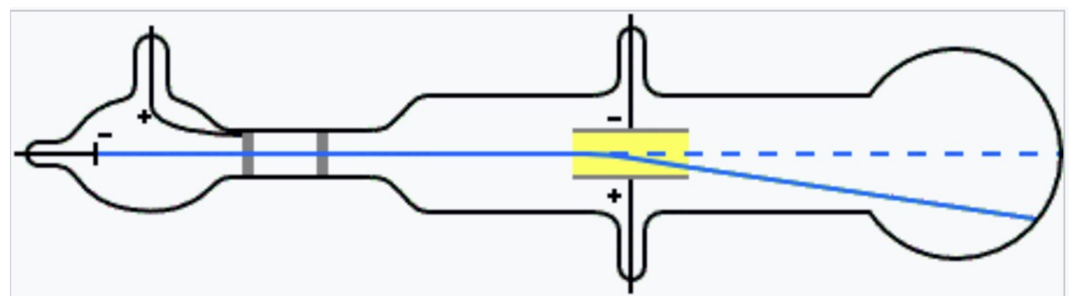
https://chemed.chem.purdue.edu/genchem/history/thomson.html#:~:text=In%20the%20SI%20system%2C%20charge,108%20coulomb%20per%20gram.

https://en.wikipedia.org/wiki/J._J._Thomson

Thomson constructed a Crookes tube with a better vacuum. At the start of the tube was the cathode from which the rays projected. The rays were sharpened to a beam by two metal slits – the first of these slits doubled as the anode, the second was connected to the earth. The beam then passed between two parallel aluminum plates, which produced an electric field between them when they were connected to a battery. The end of the tube was a large sphere where the beam would impact on the glass, created a glowing patch. Thomson pasted a scale to the surface of this sphere to measure the deflection of the beam. Any electron beam would collide with some residual gas atoms within the Crookes tube, thereby ionizing them and producing electrons and ions in the tube (space charge); in previous experiments this space charge electrically screened the externally applied electric field. However, in Thomson's Crookes tube, the density of residual atoms was so low that the space charge from the electrons and ions was insufficient to electrically screen the externally applied electric field, which permitted Thomson to successfully observe electrical deflection.

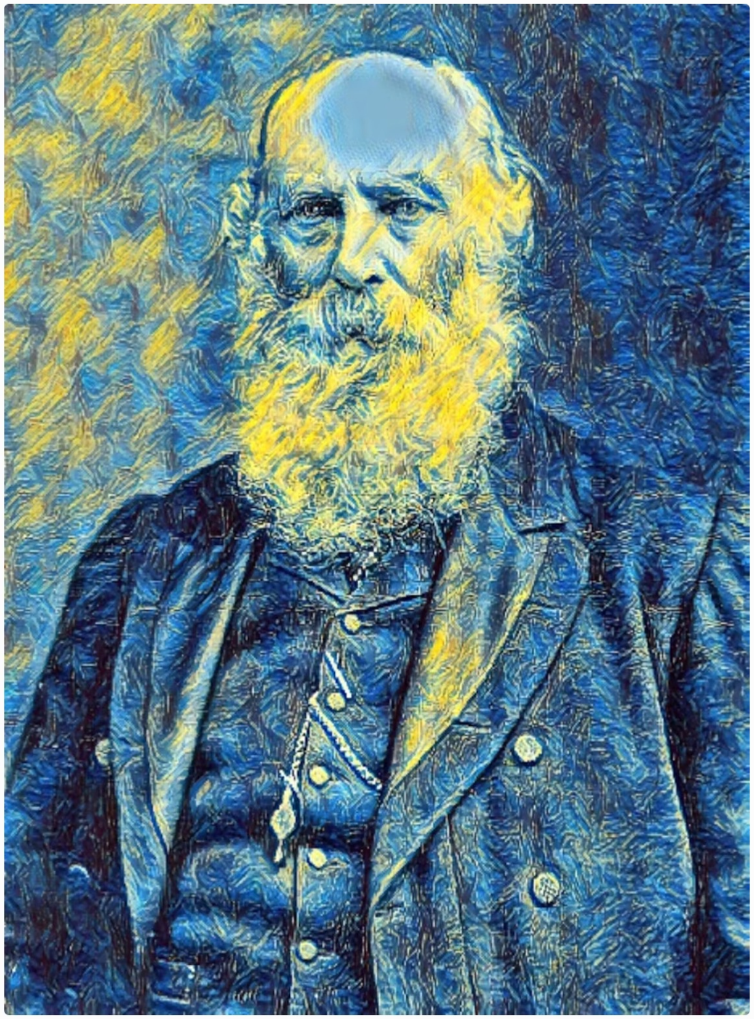






1891
Irish physicist George Johnstone Stoney

names the first ”subatomic particles" electrons



George Johnstone Stoney (1826–1911)

https://www.irishstewpodcast.com/blog/george-johnstone-stoney-physicist-1826-1911/

THE

SCIENTIFIC TRANSACTIONS

OF THE

ROYAL DUBLIN SOCIETY.

VOLUME IV.—SERIES II.

DUBLIN:

PUBLISHED BY THE ROYAL DUBLIN SOCIETY.

PRINTED AT THE UNIVERSITY PRESS, BY PONSONBY AND WELDRICK,

PRINTERS TO THE SOCIETY.

1888–1892.

CHAPTER III.

THE PROBLEM TREATED FROM THE STANDPOINT OF THE ELECTRO-MAGNETIC THEORY OF LIGHT.

A charge of this amount is associated in the chemical atom with each bond. There may accordingly be several such charges in one chemical atom, and there appear to be at least two in each atom. These charges, which it will be convenient to call *electrons*, cannot be removed from the atom; but they become disguised when atoms chemically unite. If an electron be lodged at the point P of the molecule, which undergoes the motion described in the last chapter, the revolution of this charge will cause an electro-magnetic undulation in the surrounding æther. The

“When in 1897 an English physicist, J.J. Thomson, discovered that cathode rays were beams of negatively-charged particles, Fitzgerald immediately realized these particles were Stoney’s electrons. Today, Thomson is credited with discovering the electron (though for years he persisted in calling them **corpuscles**) and Stoney is credited with inventing the concept and name.”

Stoney, G. J. (1881). "On the Physical Units of Nature" . *Phil.Mag.* Vol. 5, no. 11. pp. 381–390.
(https://www.biodiversitylibrary.org/item/120838#page/394/mode/2up)

Scanning Corpuscle Microscopy (SCM)

The Discovery of the Wave Nature of Electrons

1924
de Broglie's
Hypothesis
(in his Ph.D. thesis!!)

Louis de Broglie

Broglie in 1929

1929
de Broglie's
Nobel Prize

https://en.wikipedia.org/wiki/Louis_de_Broglie

Wavelength of Electron vs. Electron Energy

$$\lambda_{electron} = \sqrt{\frac{150}{E(\mathrm{eV})}} \quad [\text{Å}]$$

Note! This is a non-relativistic expression, useful for low-energy electrons (up to a few keV), but not correct for high energy electrons (a few hundred keV, as in a transmission electron microscope).

Useful exercise: Derive the corresponding relativistic formula.

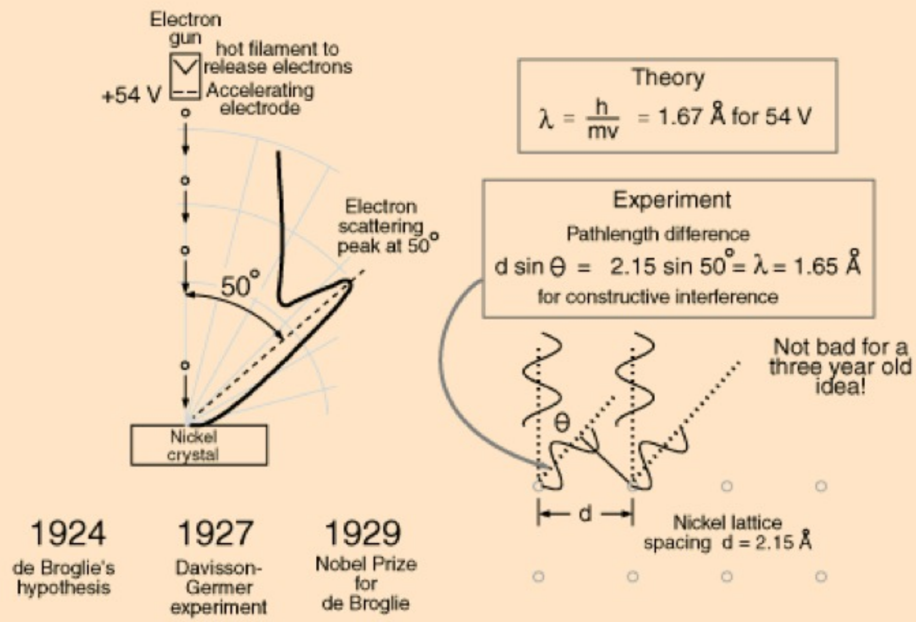
Wavelength of Electron vs. Electron Energy

Low Energy Electron Diffraction (LEED) : E = 150 eV

$$\lambda_{electron} = \sqrt{\frac{150}{150}} = 1\ [\text{Å}]$$

Discovery of the wave nature of the electron

Davisson-Germer Experiment



This experiment demonstrated the wave nature of the electron, confirming the earlier hypothesis of deBroglie. Putting wave-particle duality on a firm experimental footing, it represented a major step forward in the development of quantum mechanics. The Bragg law for diffraction had been applied to x-ray diffraction, but this was the first application to particle waves.

Wavelength of Electron vs. Electron Energy

Transmission Electron Microscopy (TEM) : E = 300 keV

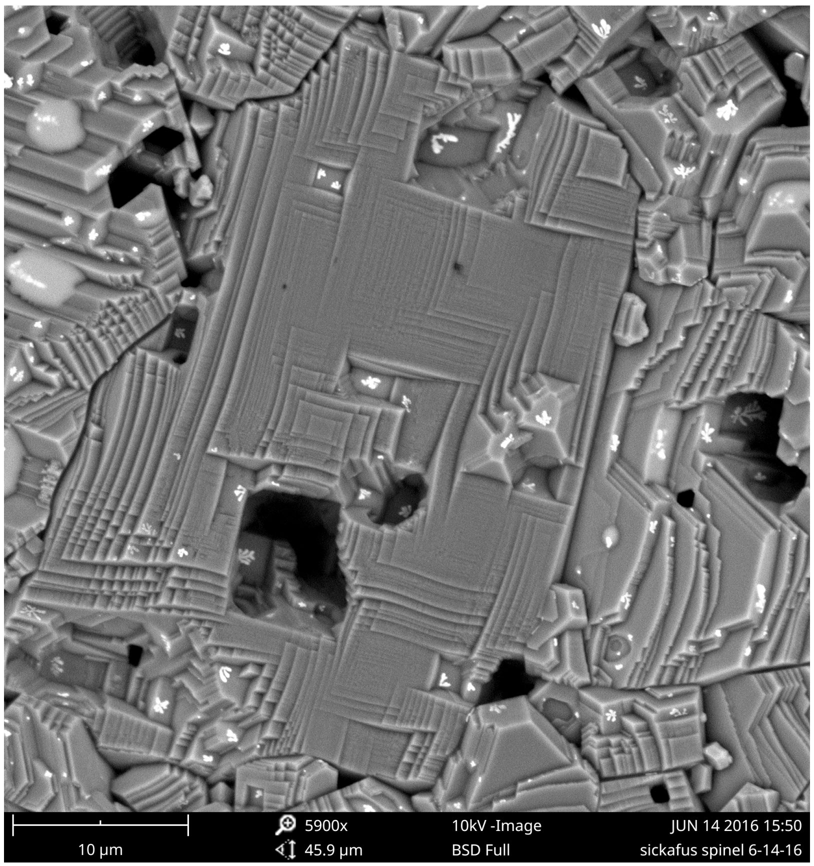
$$\lambda_{electron} = \sqrt{\frac{150}{300{,}000}}$$

$$= \sqrt{0.0005} = 0.0224 \ [\text{Å}]$$

This is an approximation – not relativistically correct

Serial vs. Parallel image acquisition

Serial acquisition



Parallel acquisition

Alexander Bain

The Real Father of Televisior

Ivan S Ruc

Alexander Bain photographed in 1876 on behalf of the Society of Telegraph Engineers, the forerunner of the Institution of Electrical Engineers, which merged with the Institution of Incorporated Engineers in 2004 to form the Institution of Engineering and Technology. *IET photograph.*

SCOTTISH LOCAL HISTORY

ISSUE 83 - SUMMER 2012

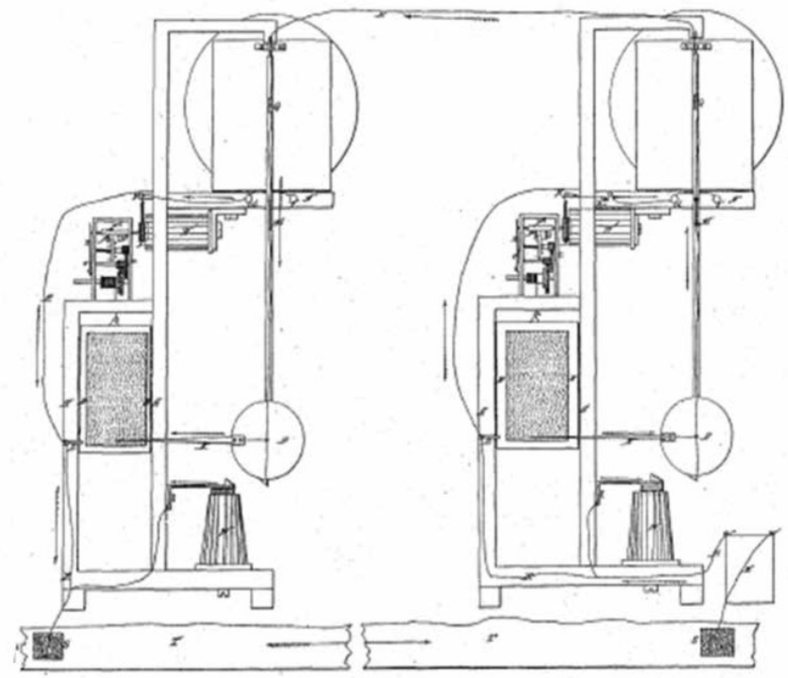


Fig.5: Bain's facsimile telegraph of 1843. The document to be sent comprises metal typeface characters in the left hand rectangular frame, while the printed copy will be on a sheet of chemically treated paper in the right hand rectangular frame. Each of the two pendulums has a stylus protruding from it that makes electrical contact with the raised surfaces in the original and makes a corresponding mark on the paper via electrical printing as in the chemical telegraph. The synchronised swinging pendulums scan and print lines respectively, while the original and the printed copy are steadily lowered after each swing. In this way every point on the original is transferred to a printed point on the paper. Illustration from US Patent No.5957: A Bain, *Improvement in copying surfaces by electricity,* December 1848.

The first “true” Scanning Electron Microscope was demonstrated by Manfred von Ardenne in 1938

Manfred von Ardenne

Ardenne (1907-97) in 1930

https://en.wikipedia.org/wiki/Manfred_von_Ardenne

von Ardenne – scanned electron image at 23 kV (1938)

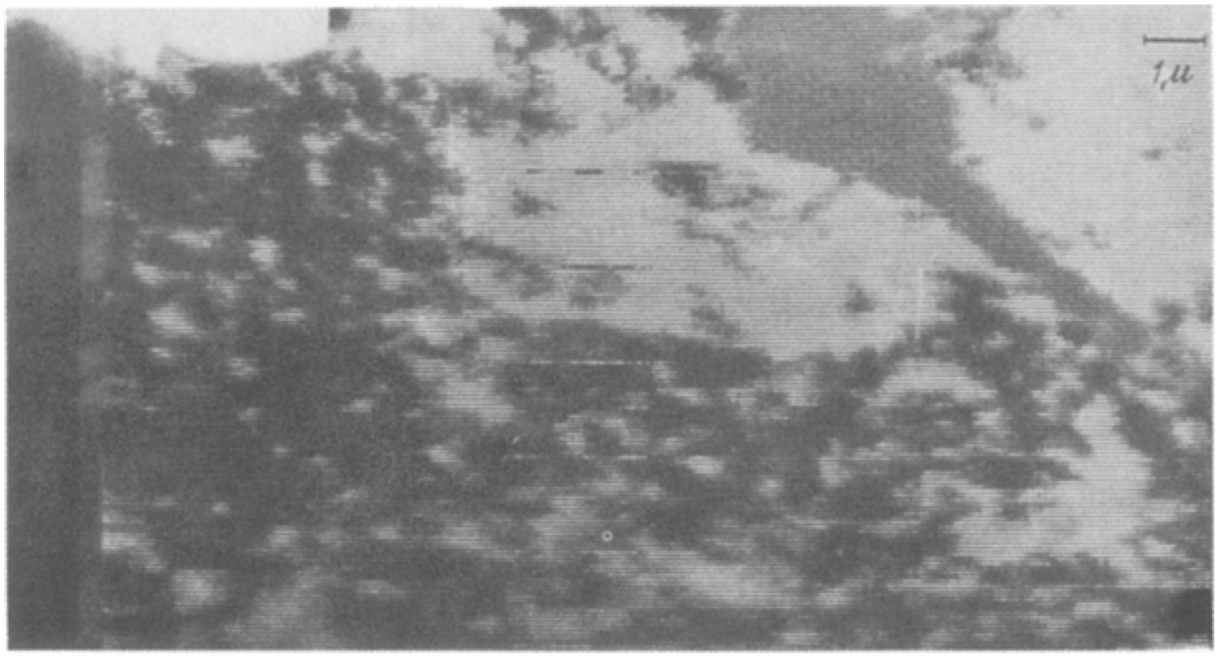


Fig. 13 A. Rastermikrobild von Zn O-Kristallen.
Bilddaten: Spannung 23 kV. $f = 1$ mm (Öffnungsverhältnis $3 \cdot 10^{-2}$ mm), Registrierzeit 20 Min. Vergrößerung des Originals 8000. Auflösungsvermögen in Zeilenrichtung $\approx 4 \cdot 10^{-5}$ mm. Kollodiumträgerfolie $3 \cdot 10^{-5}$ mm stark. Helligkeitsmodulation durch Absorption.

M. von Ardenne, *Z. Physik* **109**, 553 (1938).

Dennis McMullan

University of Cambridge

"The scanning electron microscope has a prehistory, in Germany and the USA, but the instrument we know today began life in Cambridge in 1948, when Charles Oatley gave "Construction of a scanning electron microscope" to Dennis McMullan as his PhD project."

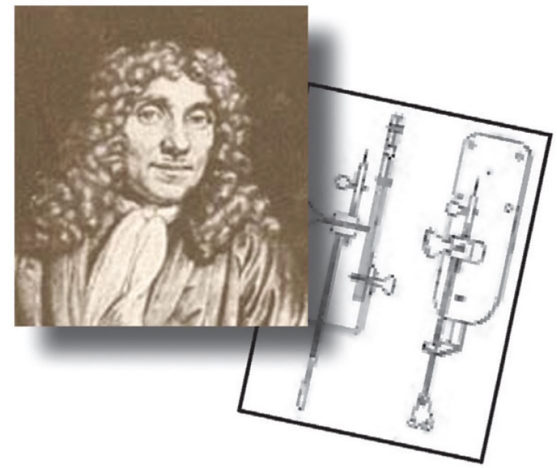
Scanning Electron Microscope Pioneers Dennis McMullan (1923–2015) and Ken Smith (1928–2020): SEM, Whisky, Chocolate, and Fast Cars

Dennis McMullan. Photo: Finn Johannessen.

doi:10.1017/S155192952000111X

MicroscopyPioneers

Scanning Electron Microscope Pioneers Dennis McMullan (1923–2015) and Ken Smith (1928–2020): SEM, Whisky, Chocolate, and Fast Cars



Peter Hawkes*
CEMES-CNRS, B.P. 94347, F-31055 Toulouse cedex, France
Editor: Cameron Varano, The Pennsylvania State University, 201 Old Main, University Park, PA 16802

*peter.hawkes@cemes.fr

Microscopy TODAY doi:10.1017/S155192952000111X www.microscopy-today.com • 2020 July

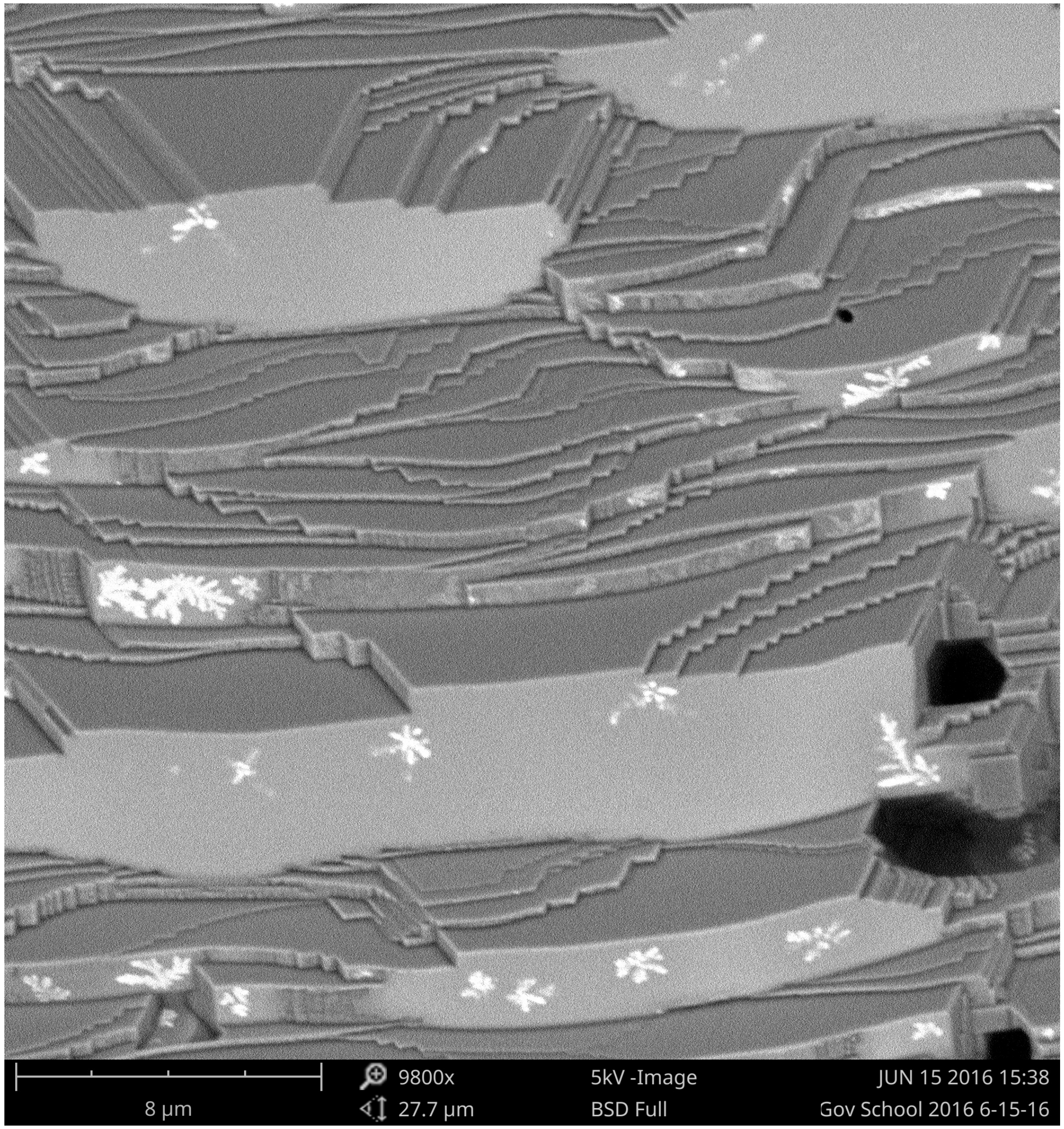
"Surely Dennis must be an inspiration to us all for living to 91 years old on a diet heavily influenced by whisky and chocolate!"

(quote attributed to Dennis's niece)

That first instrument contained all the essential features of the sophisticated instruments we know today. In particular, he designed and built a stabilized power supply and CRT display with a nonlinear video amplifier and introduced double-deflection, which is now standard.

Dennis McMullan. Photo: Finn Johannessen.

doi:10.1017/S155192952000111X



2016
Sickafus
SEM image of the surface of a sintered sample of $MgAl_2O_4$ spinel