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MICROSCOPIA ELETRÔNICA

ANDRÉ LUIZ PINTO

CBPF

Roteiro

- Introdução
- Fundamentos
  - Fontes de elétrons
  - Lentes de elétrons
  - Interação elétron-matéria
- Microscópio Eletrônico de Varredura
- Microscópio Eletrônico de Transmissão
- Aplicações à Nanotecnologia
- Comentários Finais
  - LabNano

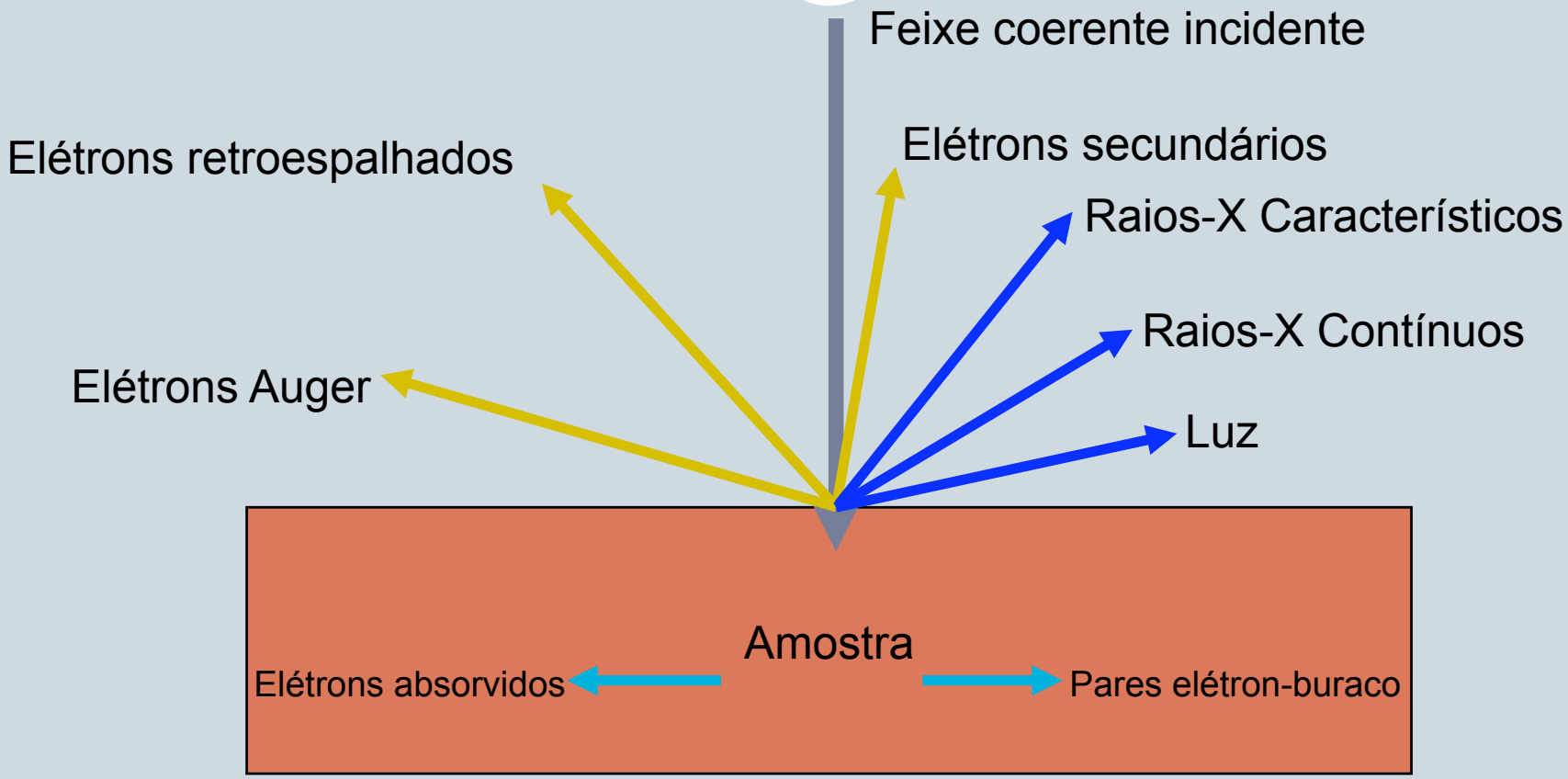
Opções para visualizar a microestrutura

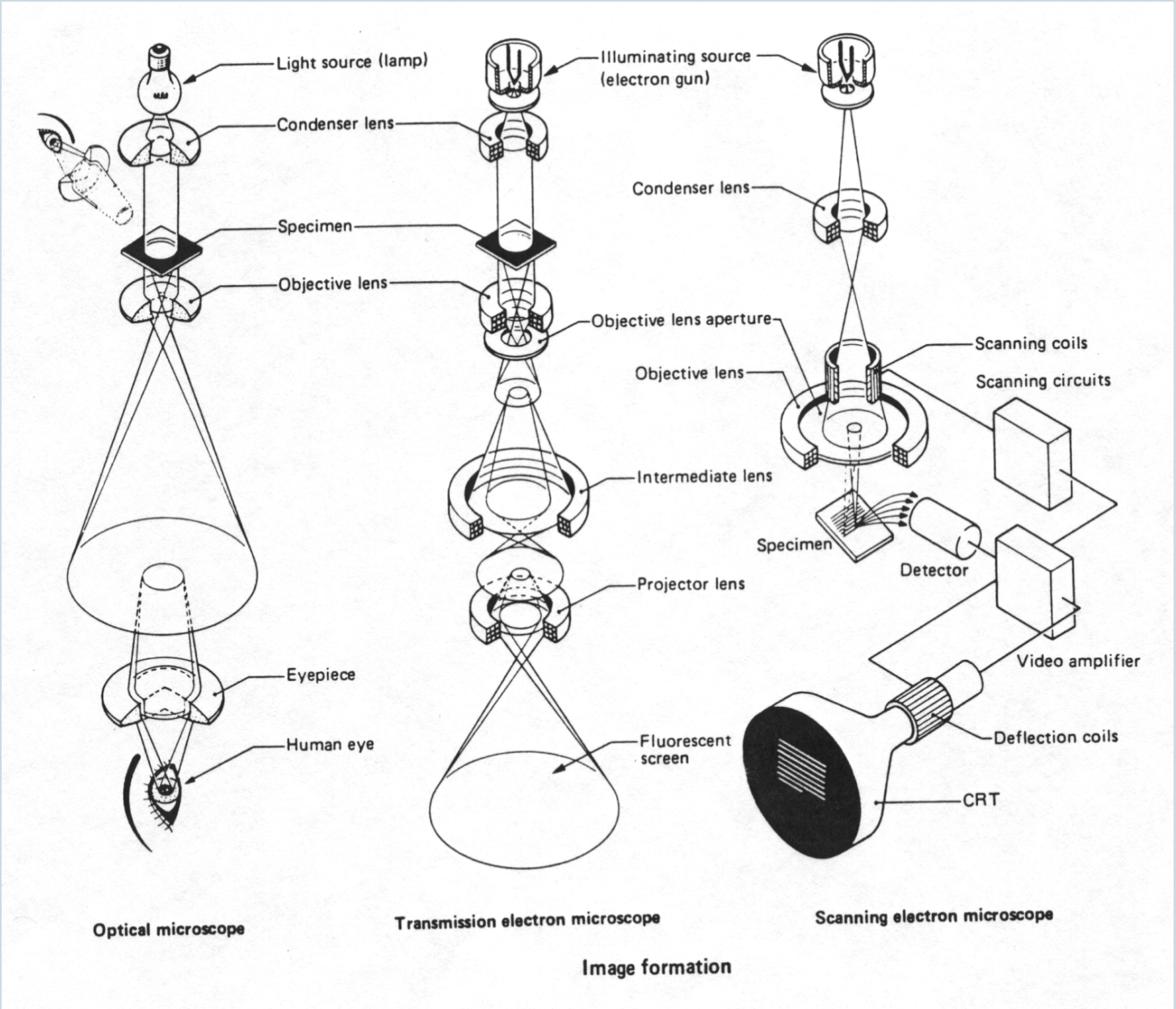
- Aumento x Resolução (lateral)



- Olho humano – 0,1 mm
- Microscopia Ótica – 0,5 μm
- Microscópio Eletrônico de Varredura (MEV) – 1 - 4 ηm
- Microscópio Eletrônico de Transmissão (MET) – 0,8 - 1,4 Å
- Microscópio de Ponta de Prova (SPM) – 0,3 Å

Interação Elétron-Amostra




Light source (lamp)
Condenser lens
Specimen
Objective lens
Eyepiece
Human eye
Optical microscope
Illuminating source (electron gun)
Condenser lens
Objective lens aperture
Objective lens
Intermediate lens
Projector lens
Fluorescent screen
Transmission electron microscope
Scanning coils
Scanning circuits
Specimen
Detector
Video amplifier
Deflection coils
CRT
Scanning electron microscope
Image formation

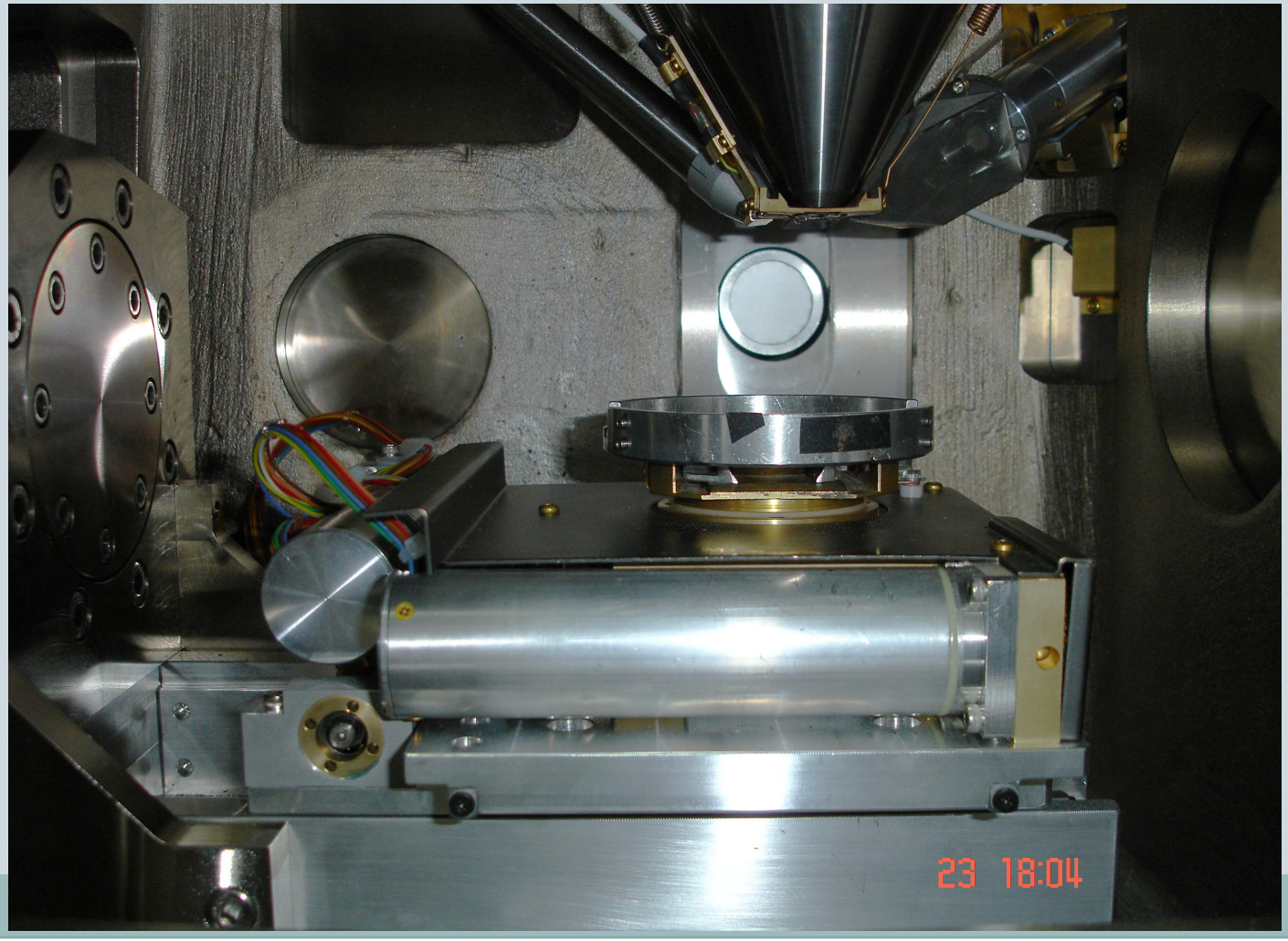
Jeol

Microscopia Eletrônica de Varredura



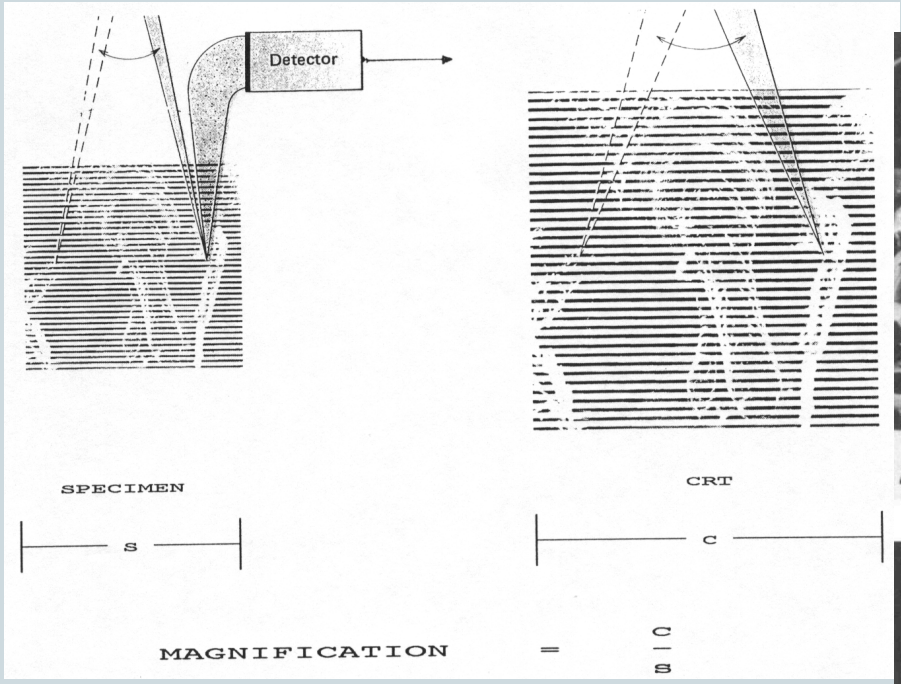
Jeol

Câmara

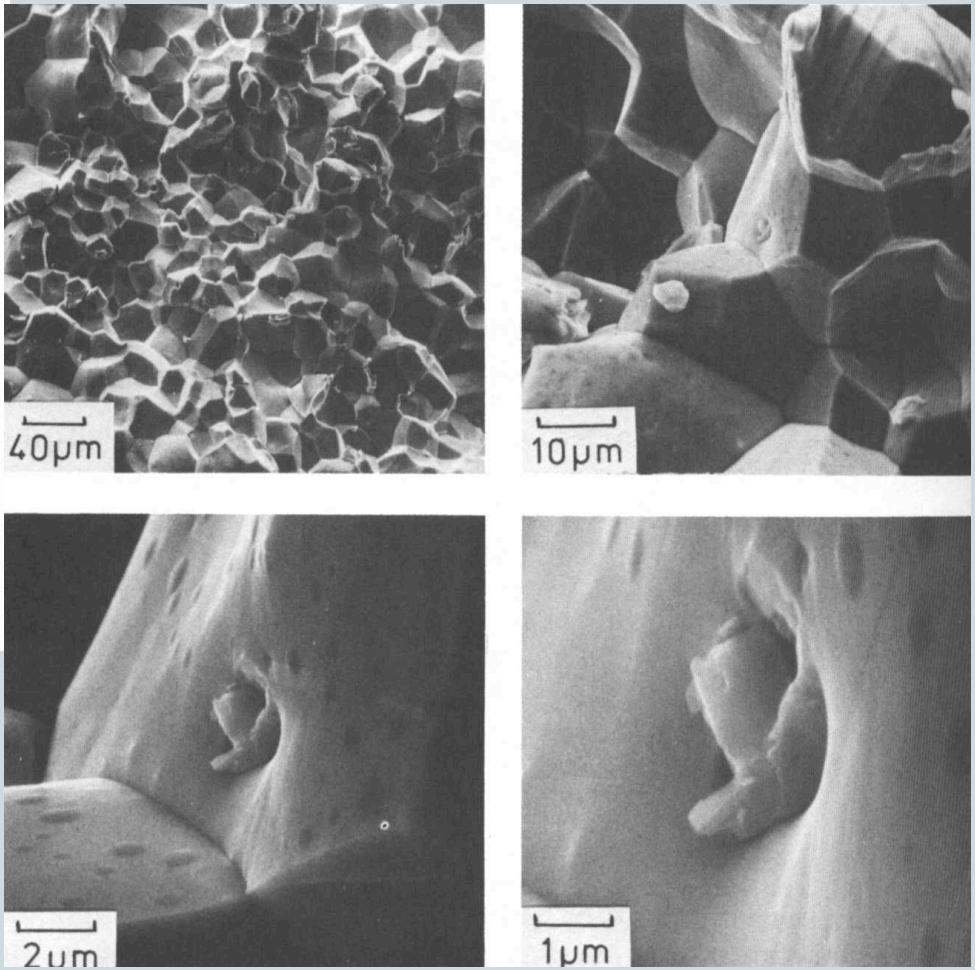


Microscopia Eletrônica de Varredura

Jeol

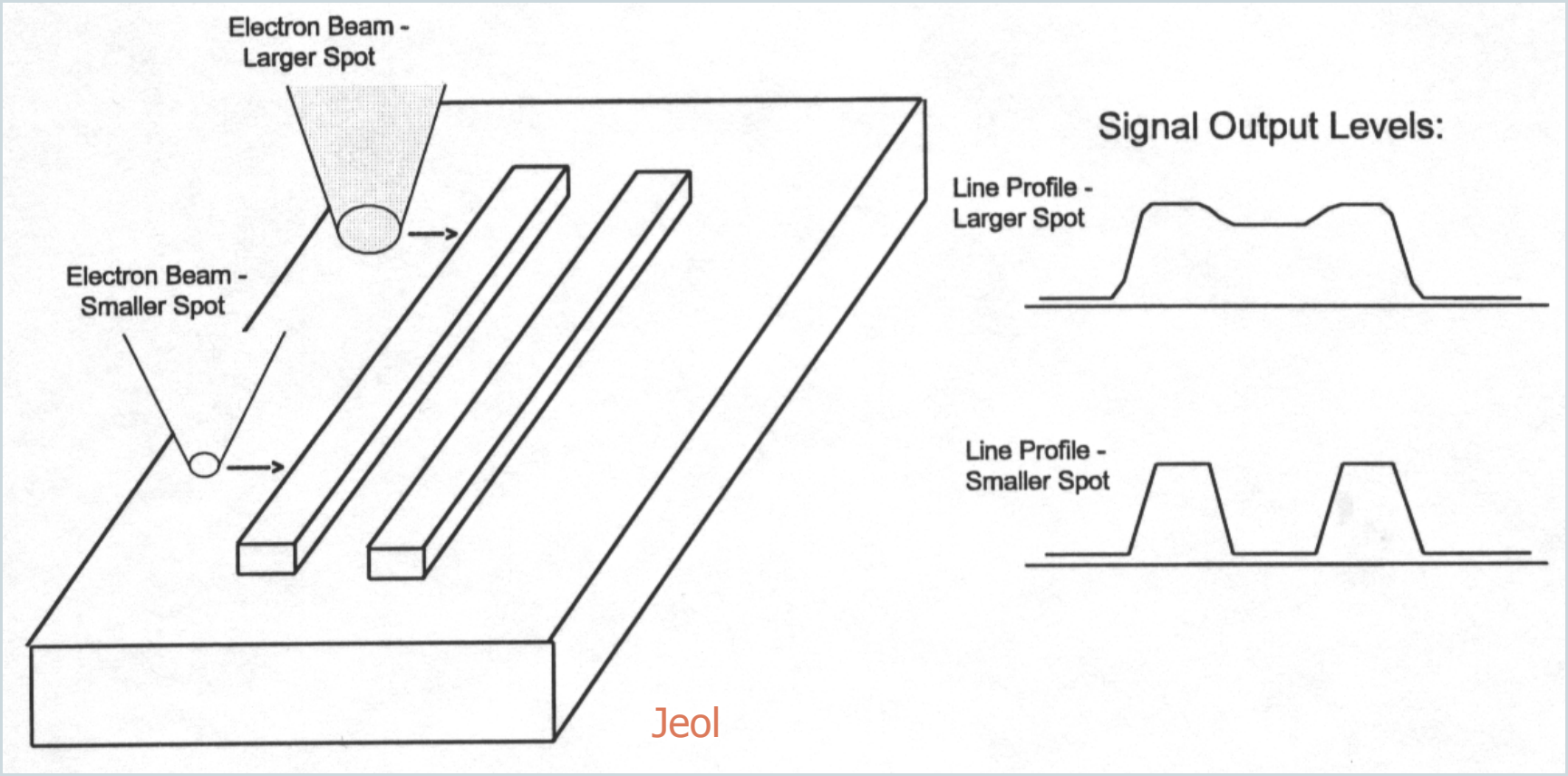


Goldstein et alli, 2003

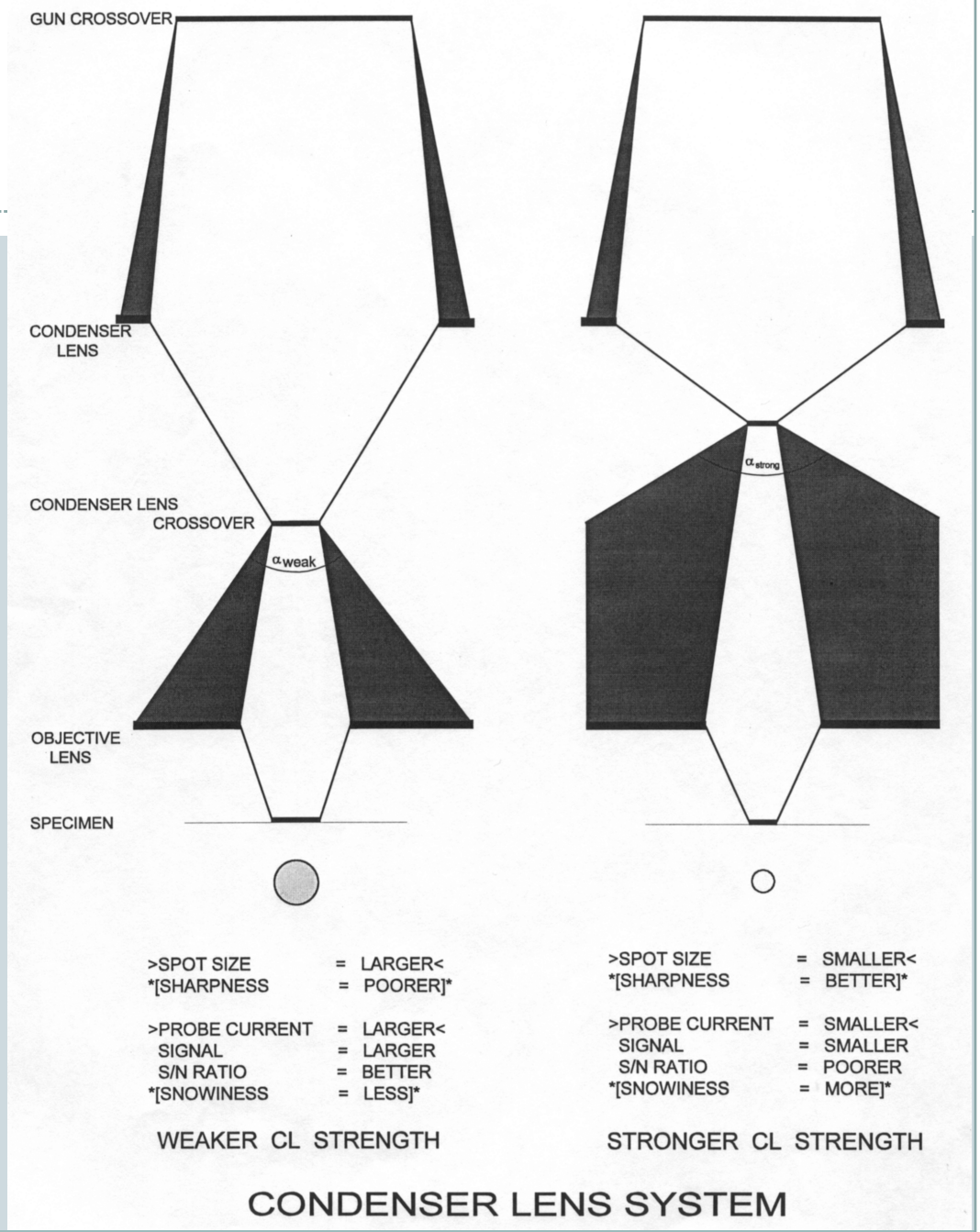


No MEV, o aumento é dado pela relação entre as dimensões varridas na amostra e as dimensões da tela.

“Spot Size” e Resolução

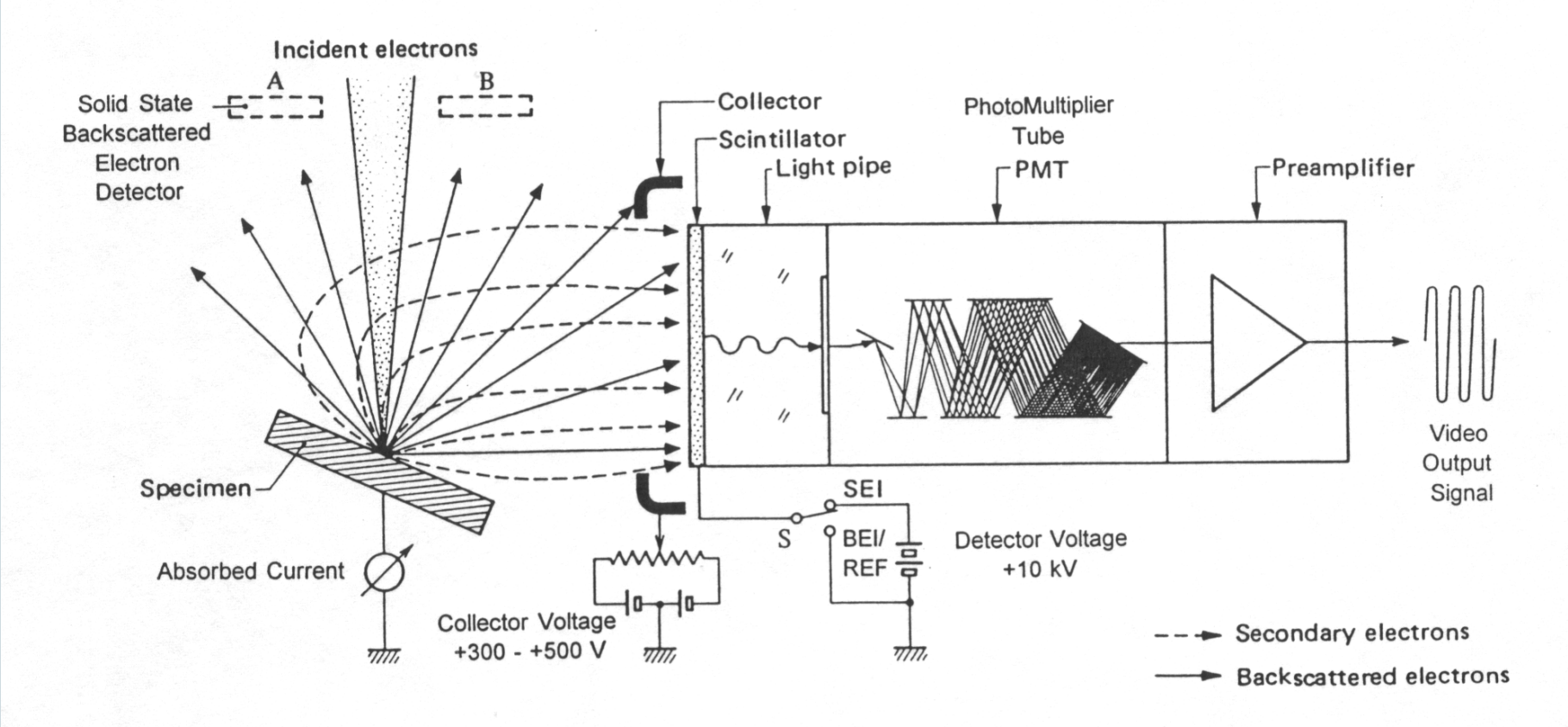


“Spot Size” e Qualidade



Jeol

Detectores de Elétrons Secundários

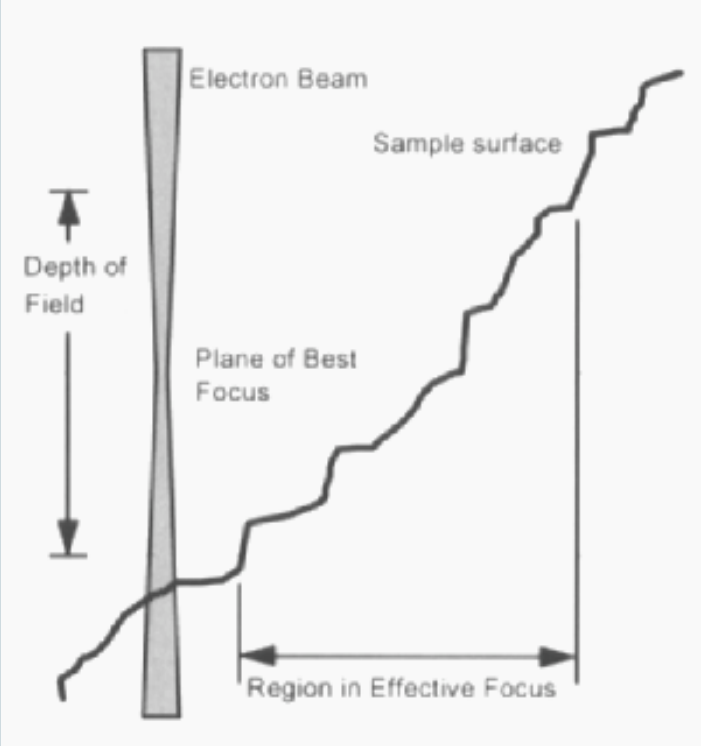


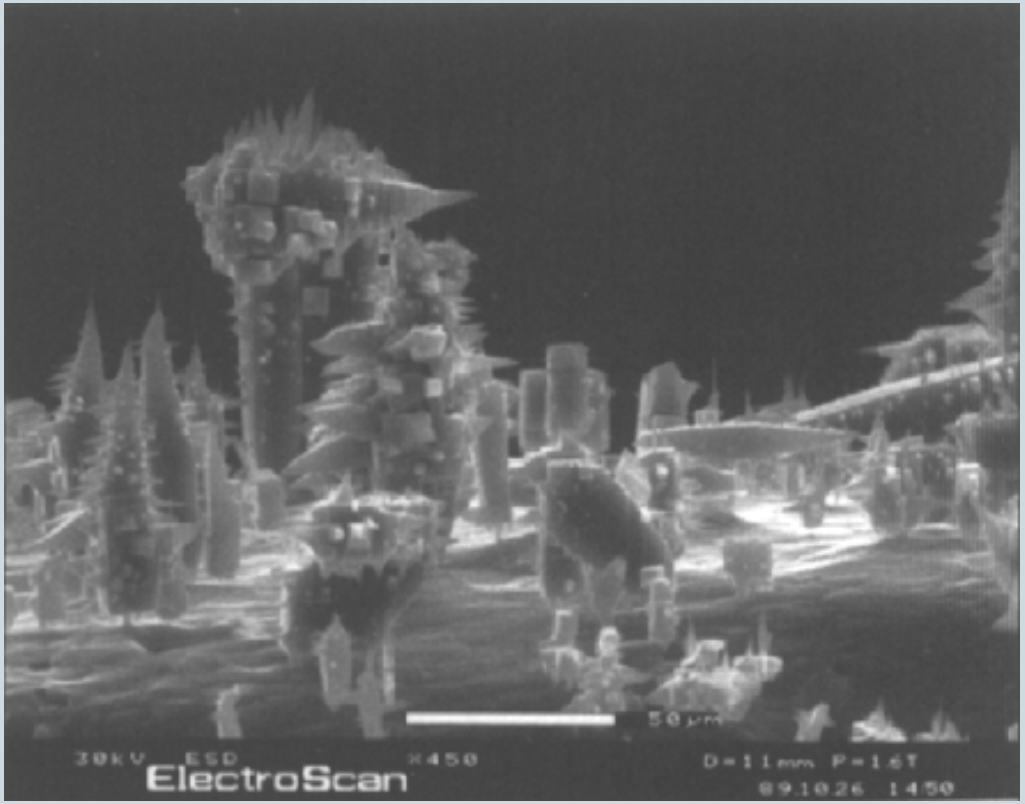
- Detetor de Everhart-Thornley

Detectores de Elétrons Secundários

- Cintilador utilizado é um YAG (yttrium-aluminium garnet) dopado com Ce, para obter menor tempo de decaimento (ηs ao invés dos μs do ZnS)
- Ganho da fotomultiplicadora de 10^n, dependendo de qtos n dinodos estão presentes
- Ganhos de 10^8 são comuns, logo o nível de ruído é baixo
- Velocidade de resposta compatível com as taxas de imagem de TV
- Bx eficiência energética (1 e^- de 100kV gera 4.000 fótons)

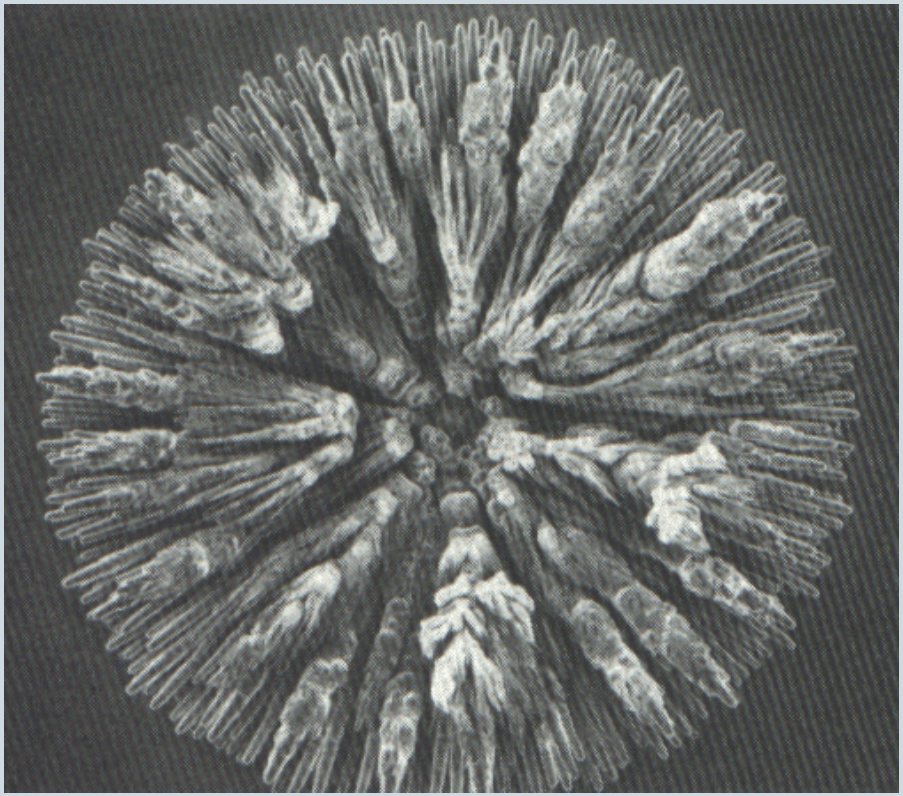
Elétrons Secundários – Topografia



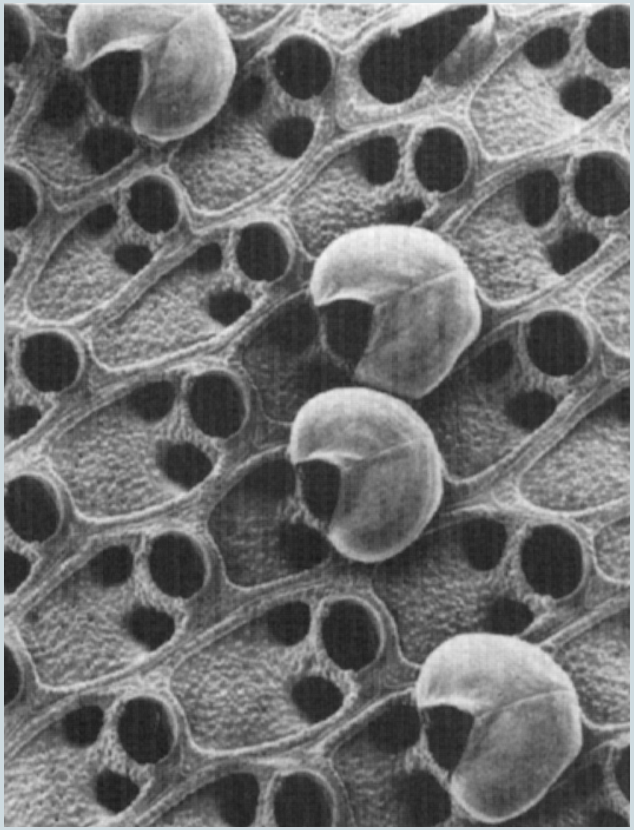


- Profundidade de Campo >> Resolução

Elétrons Secundários – Topografia

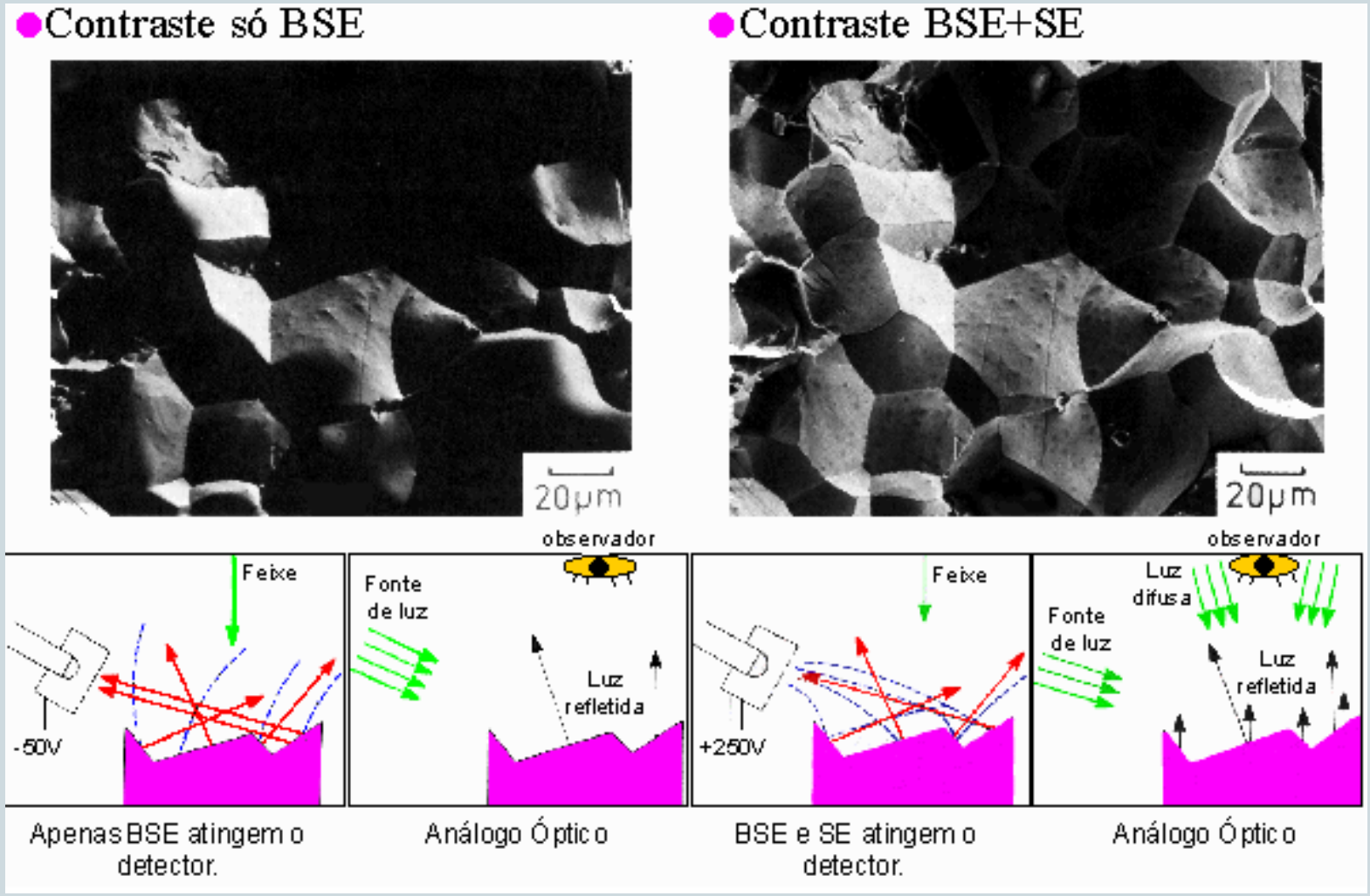


Gota de Au aplicada por “jet spray Electroplating”.

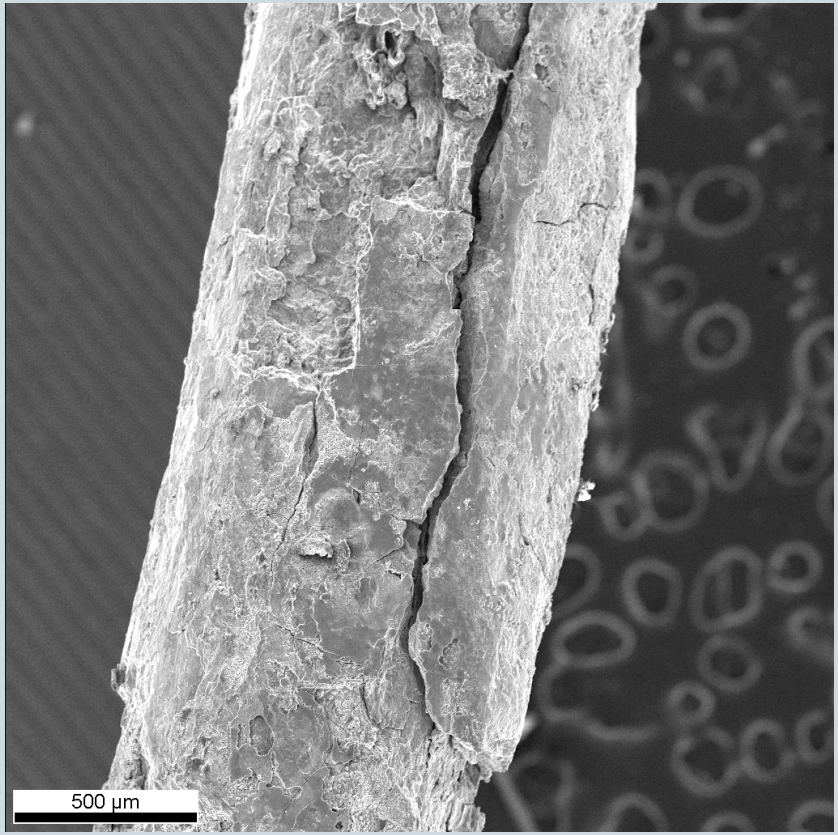


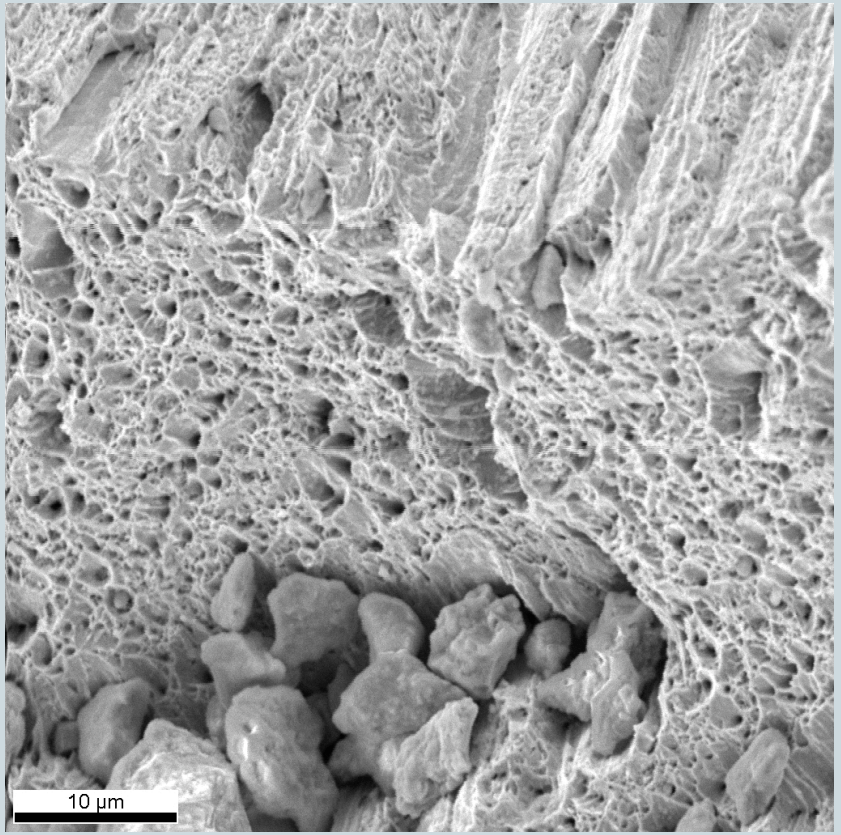
Thalamoporella stapifera.

Elétrons Secundários – Topografia



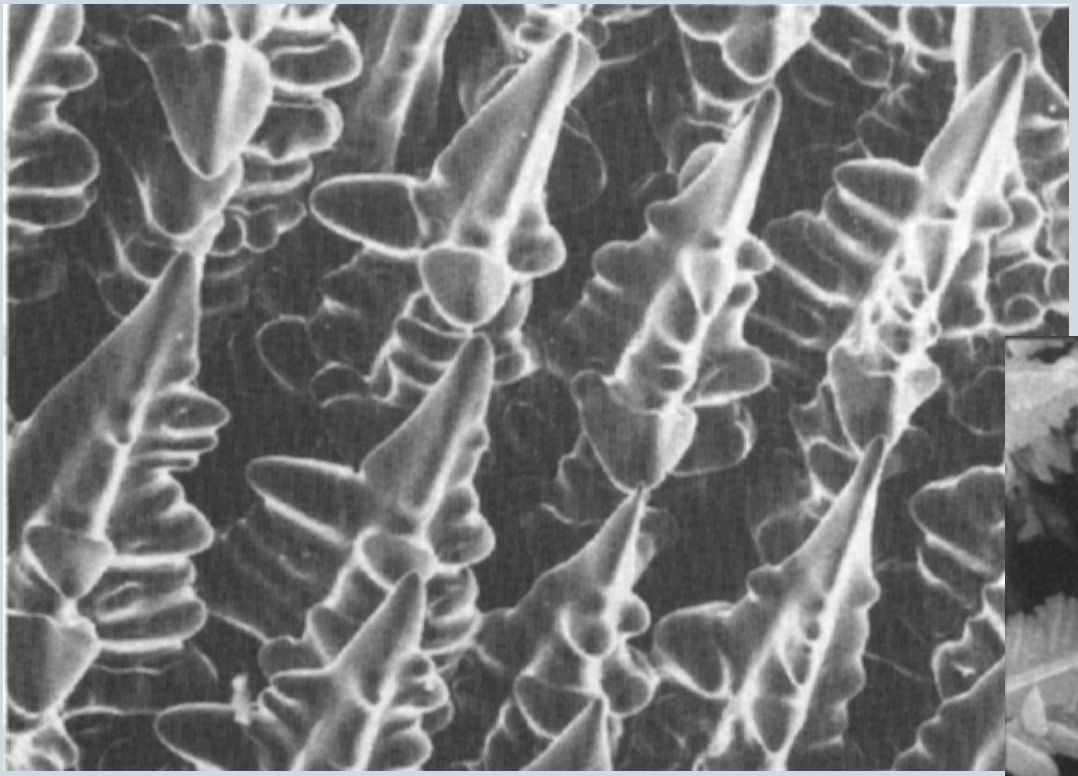
Elétrons Secundários – Topografia



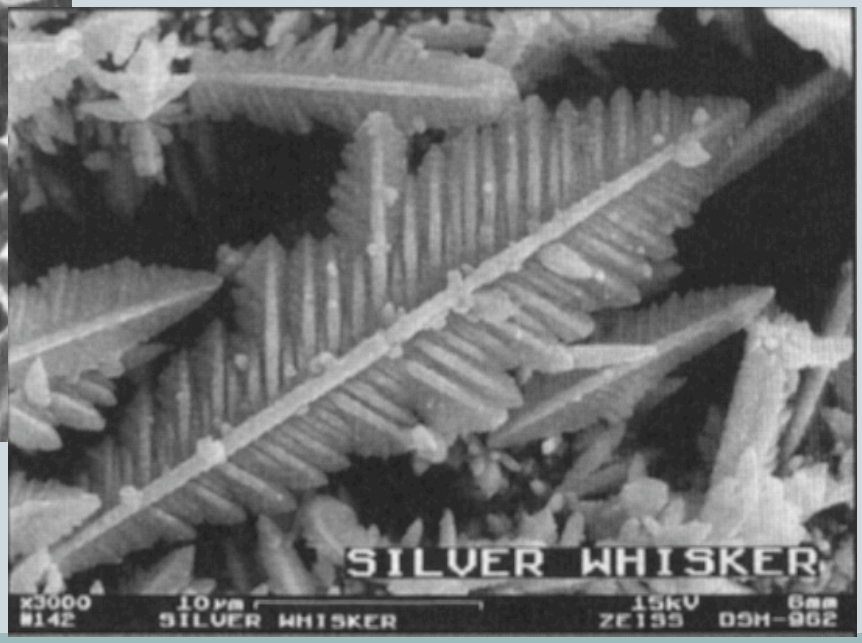


- Análise de falha em cabo de aço.

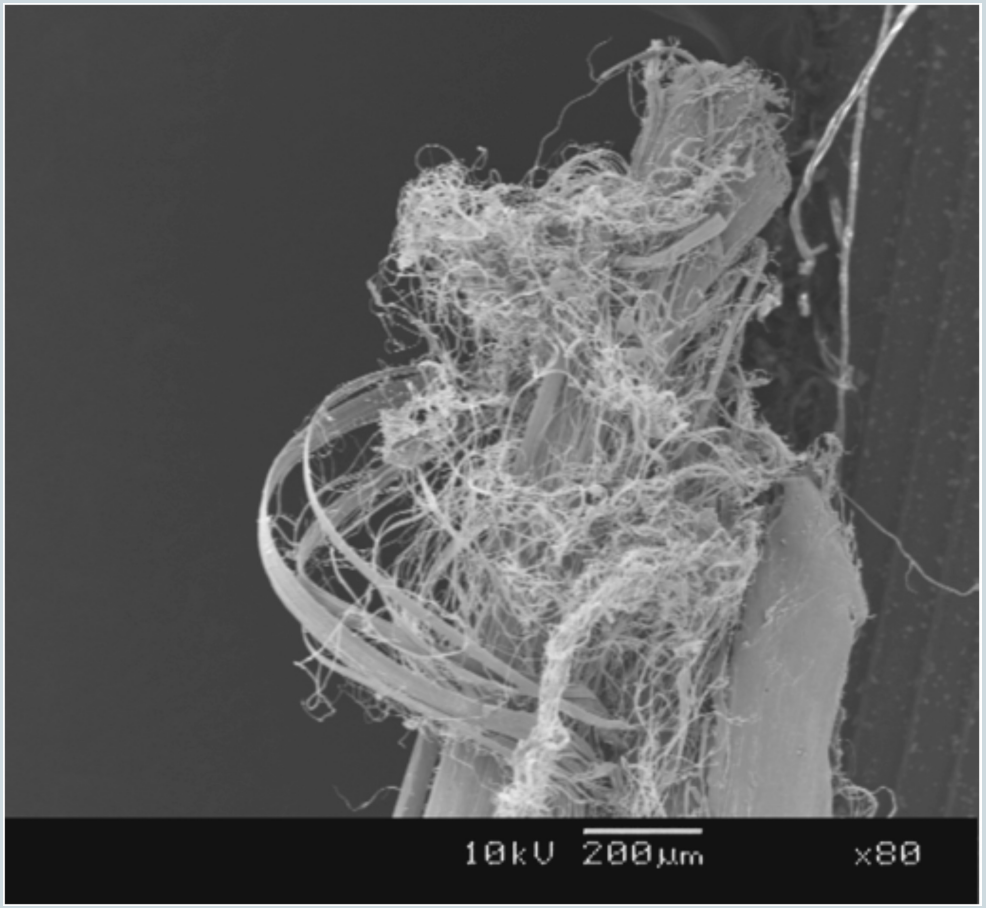
Elétrons Secundários – Topografia



Dendritas em um vazio de fundição da superliga 718.

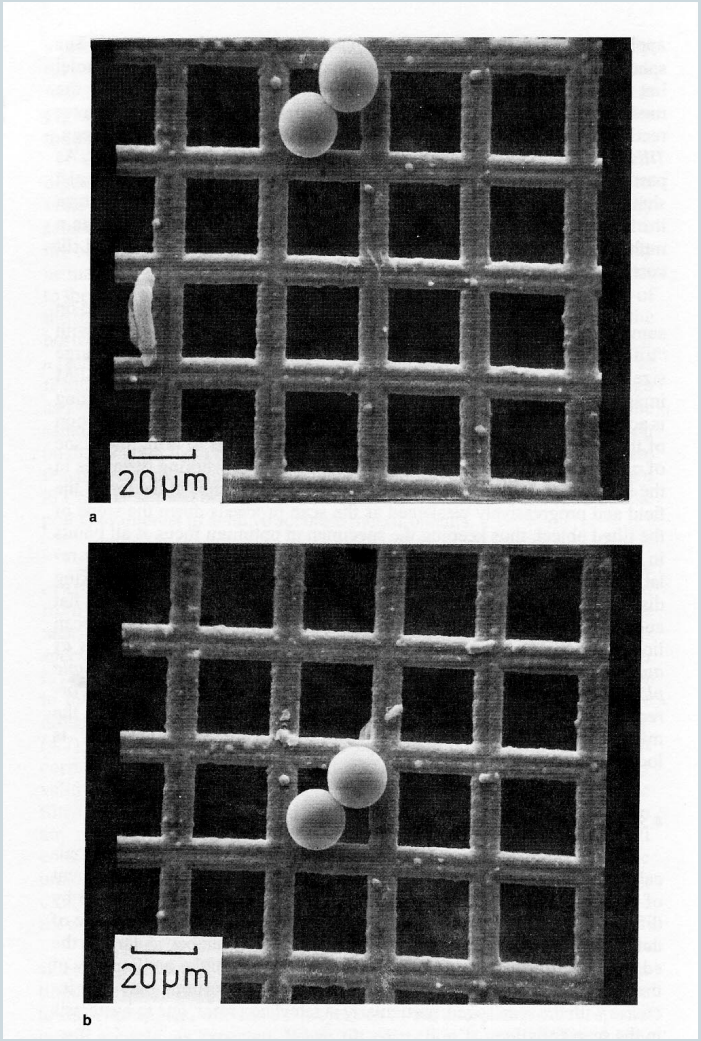


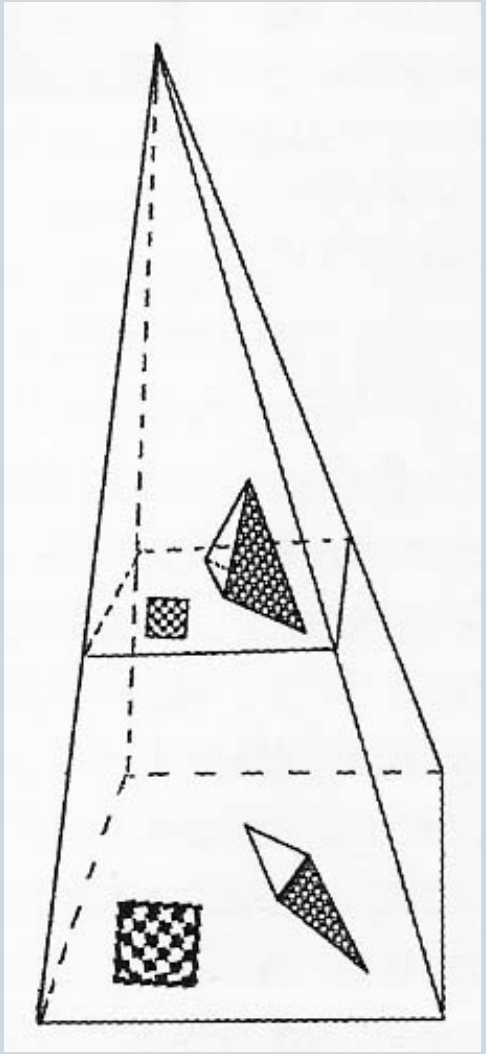
Elétrons Secundários – Topografia



Placa de Armodon (UHMW-PP) submetida a Impacto balístico.

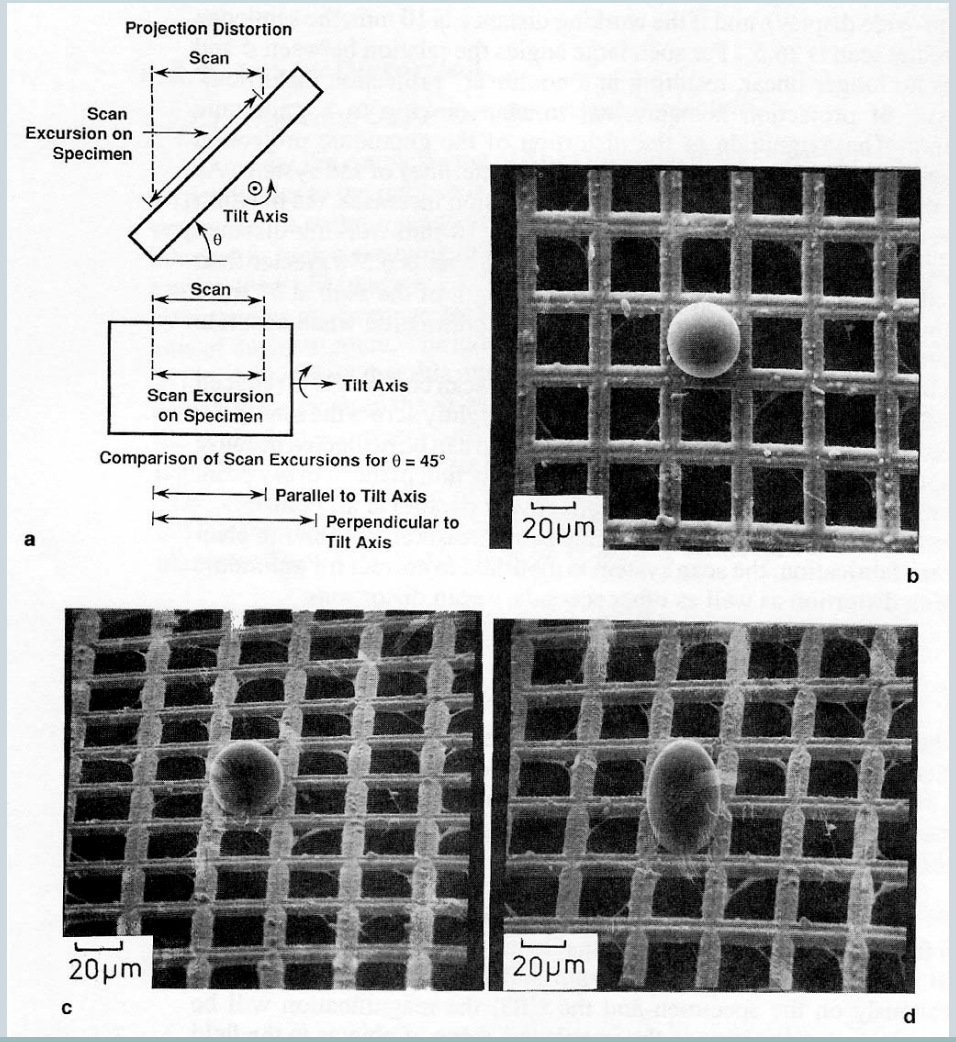
Perspectiva Cônica





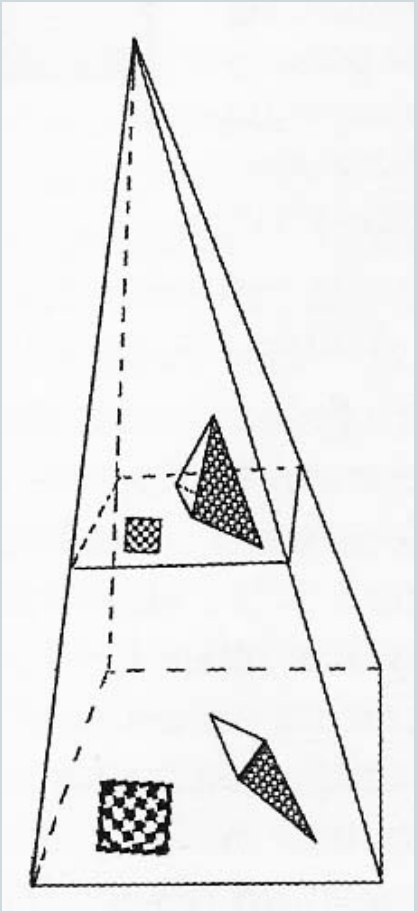
Goldstein

Perspectiva Cônica



55º

0º

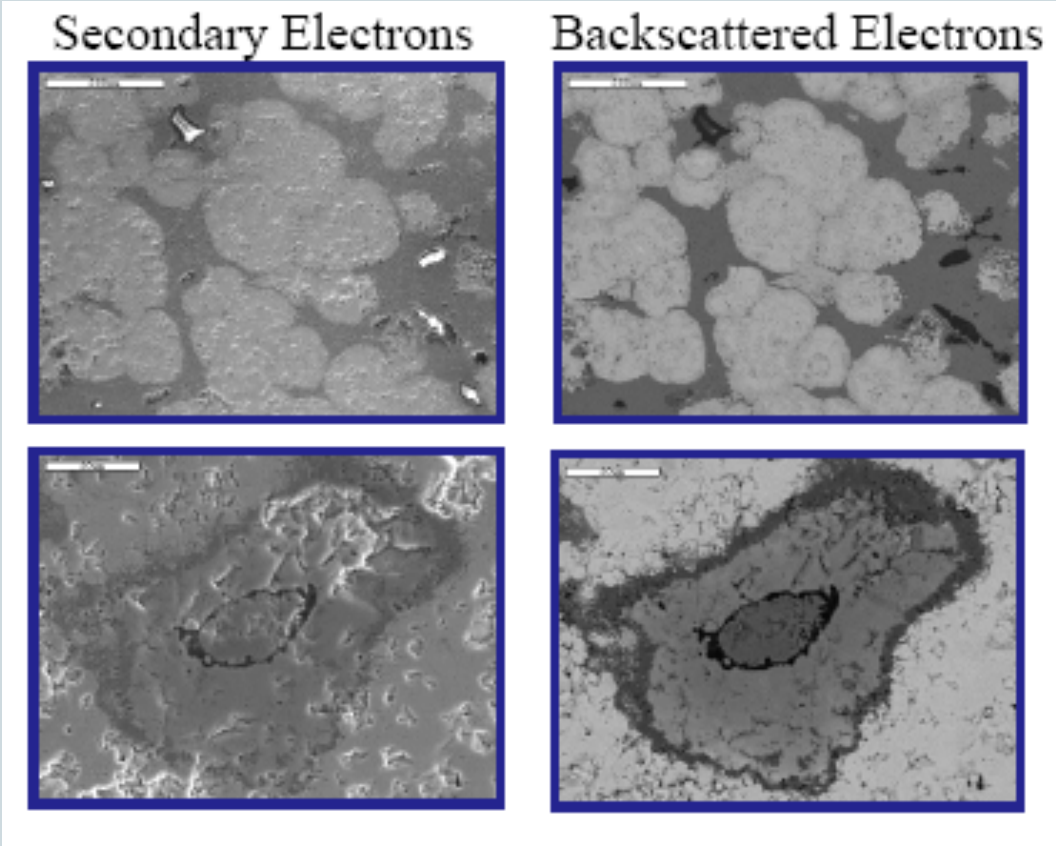


Goldstein

55º com correção

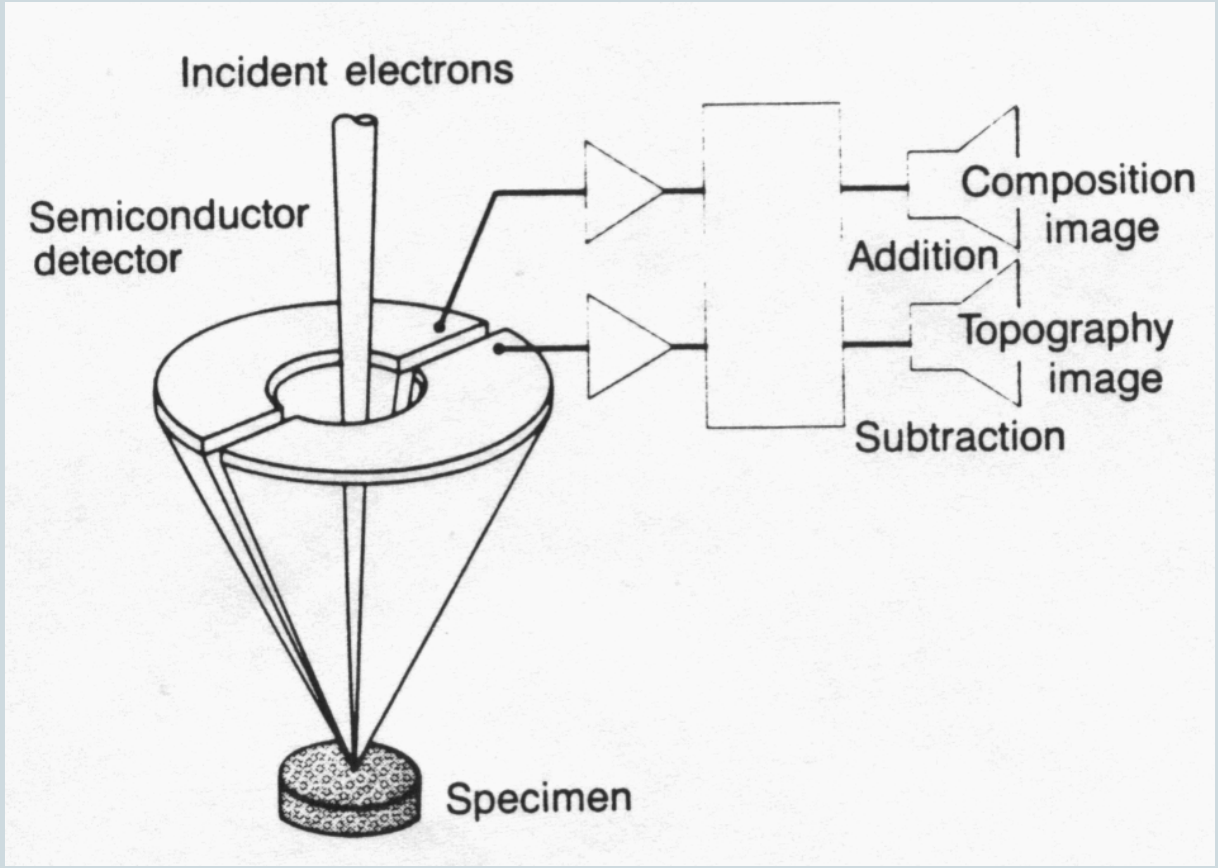
Imagem de Elétrons Retroespalhados

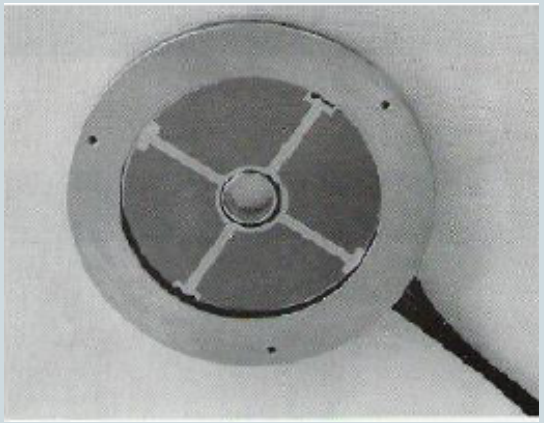
- Contraste de Z



Goldstein

Detector de e_{BSE} de Estado Sólido





- Detector de Estado Sólido

Detector de e_{BSE} de Estado Sólido

- Sensíveis apenas a elétrons de alta energia
- Fáceis de fabricar e baratos
- Podem ser fabricados em qualquer forma plana
- Alta eficiência energética (1 e^- de 100kV gera 28.000 pares)
- Grande ruído de fundo

Detector de e_{BSE} de Estado Sólido

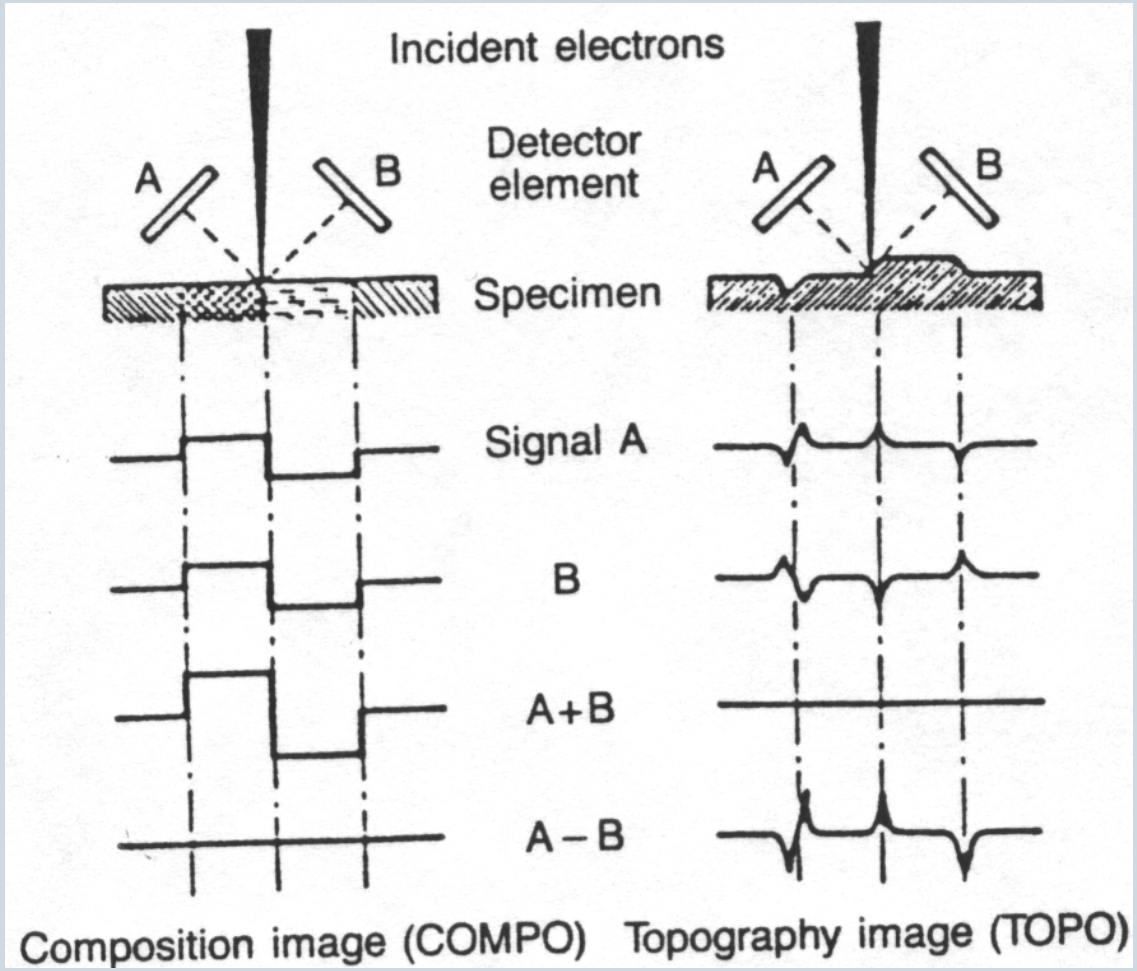
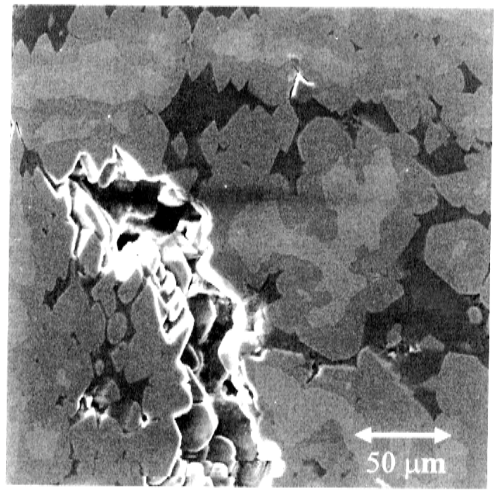


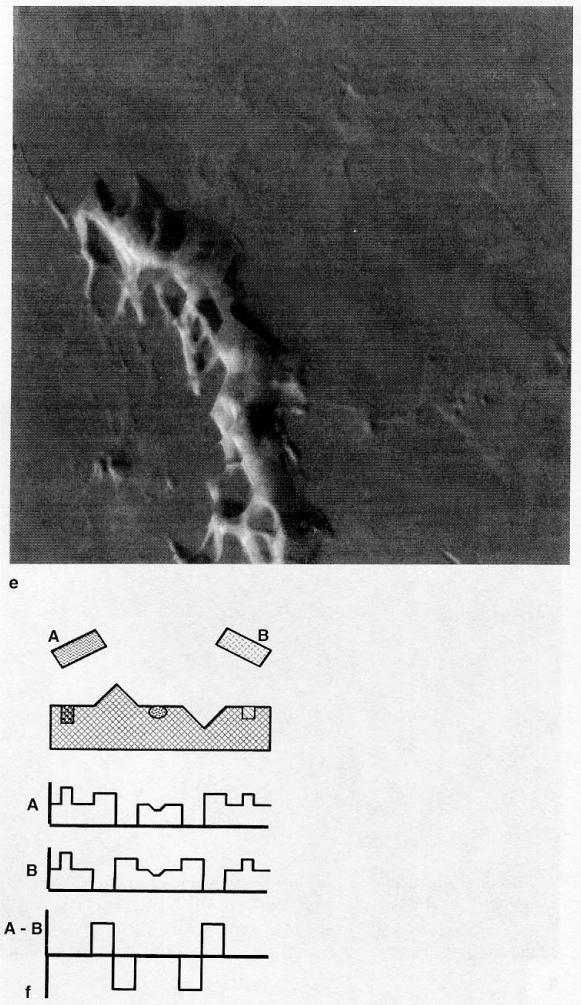
Imagem de e_{BSE}



E-T

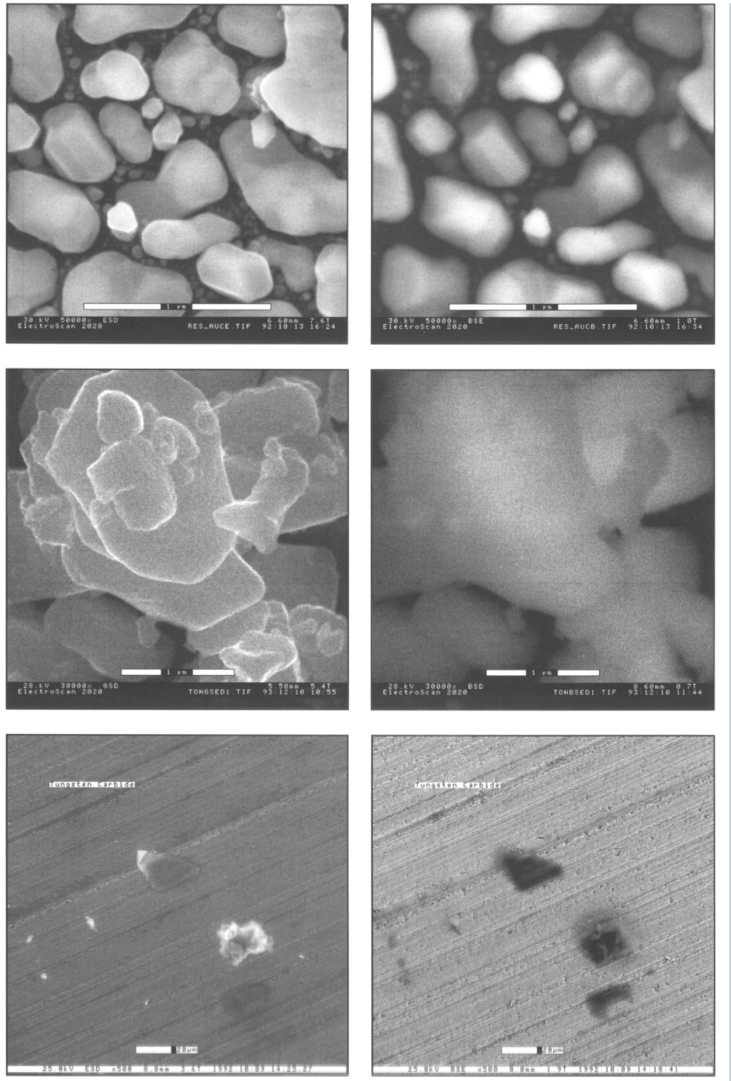
BSE Topo

BSE Compo

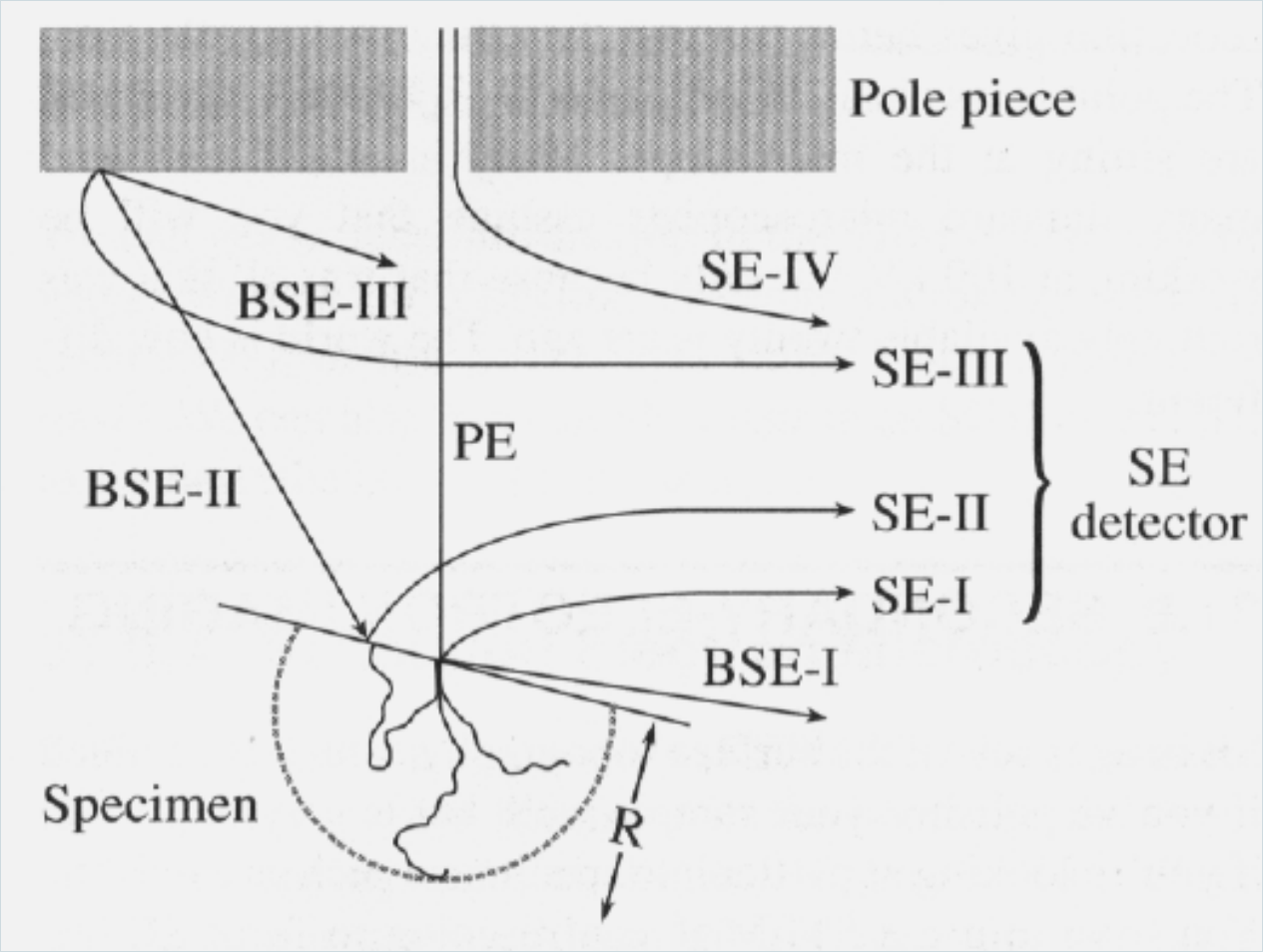


Secundários x Retroespalhados

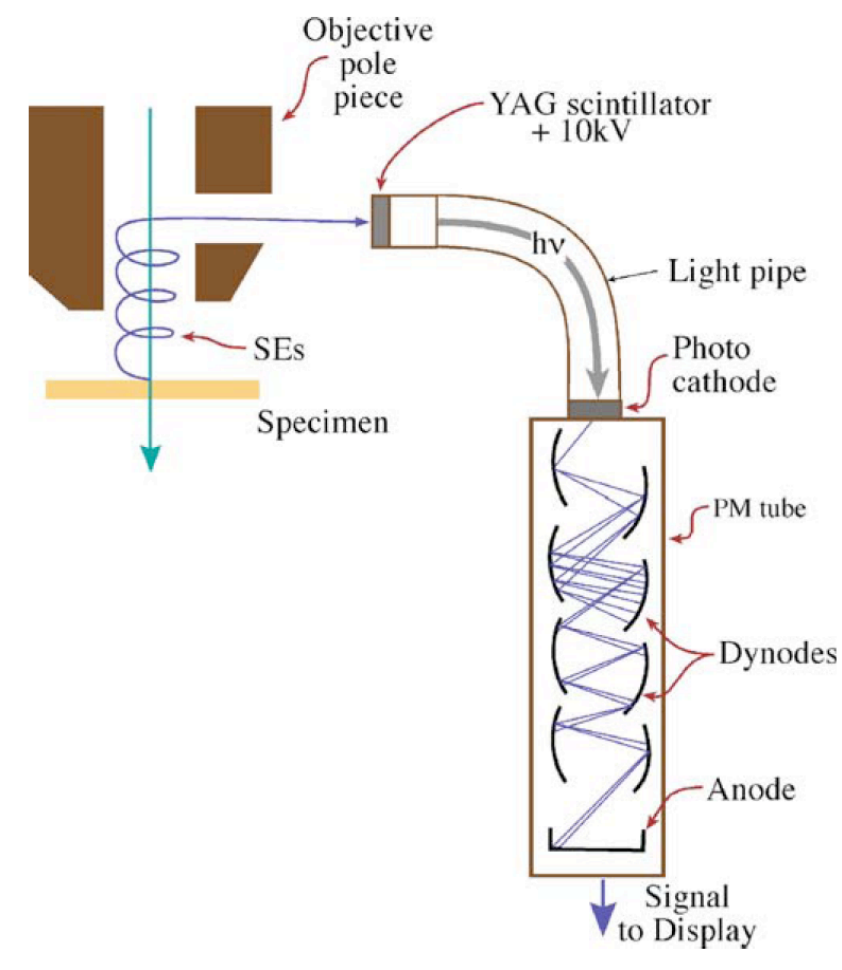
- Topografia com baixa resolução
- Contraste de Z



Elétrons Secundários
Os vários sinais

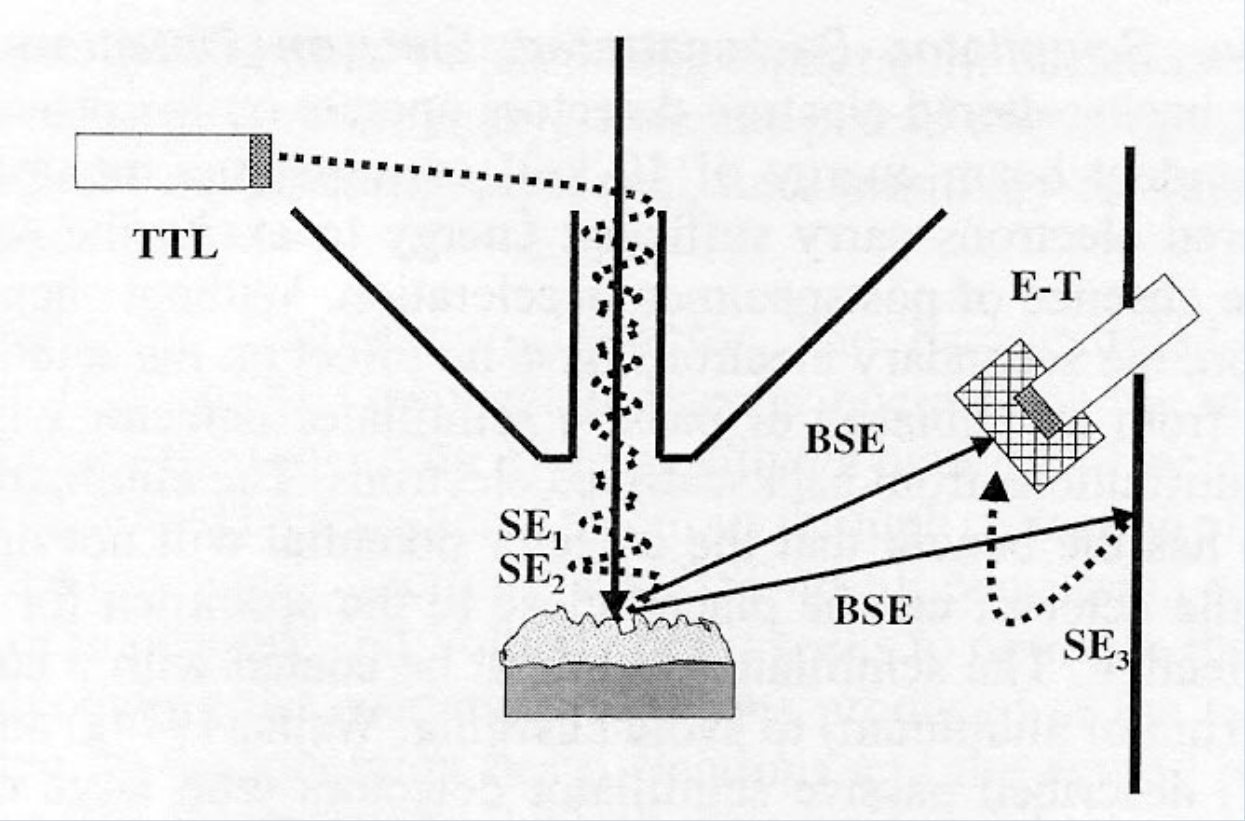


Detectores de Elétrons Secundários



Williams e Carter

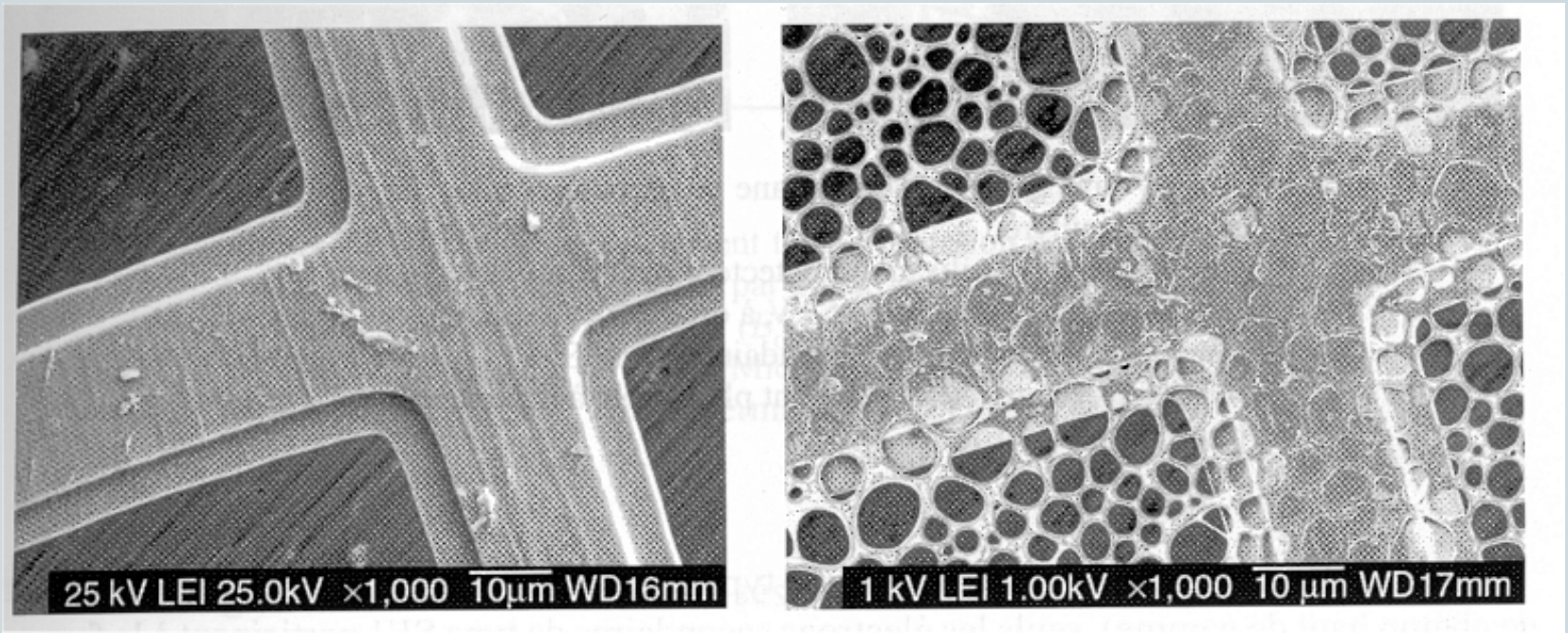
Detectores de Elétrons Secundários



Uma visão comparativa dos vários sinais

SE + BSE

SE + BSE



E_0 influence on the SE image

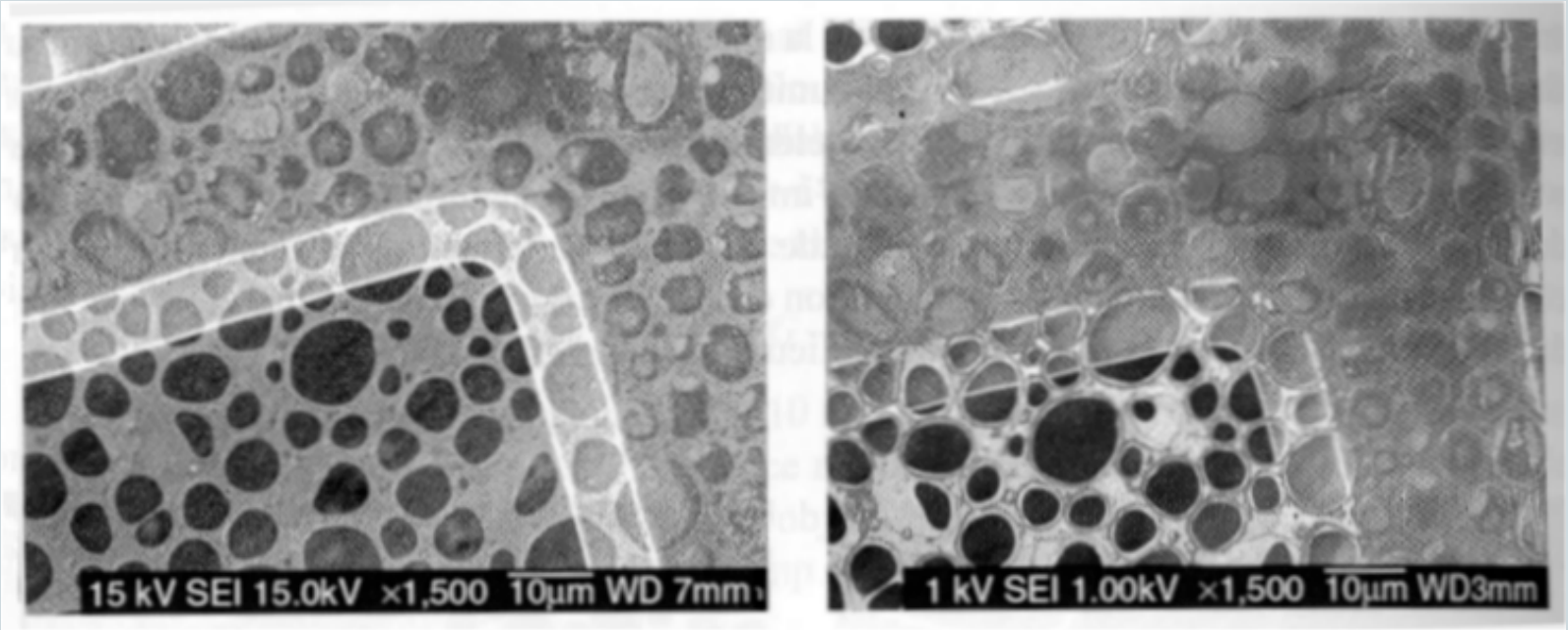
FEI

lateral detector

Uma visão comparativa dos vários sinais

SE

SE

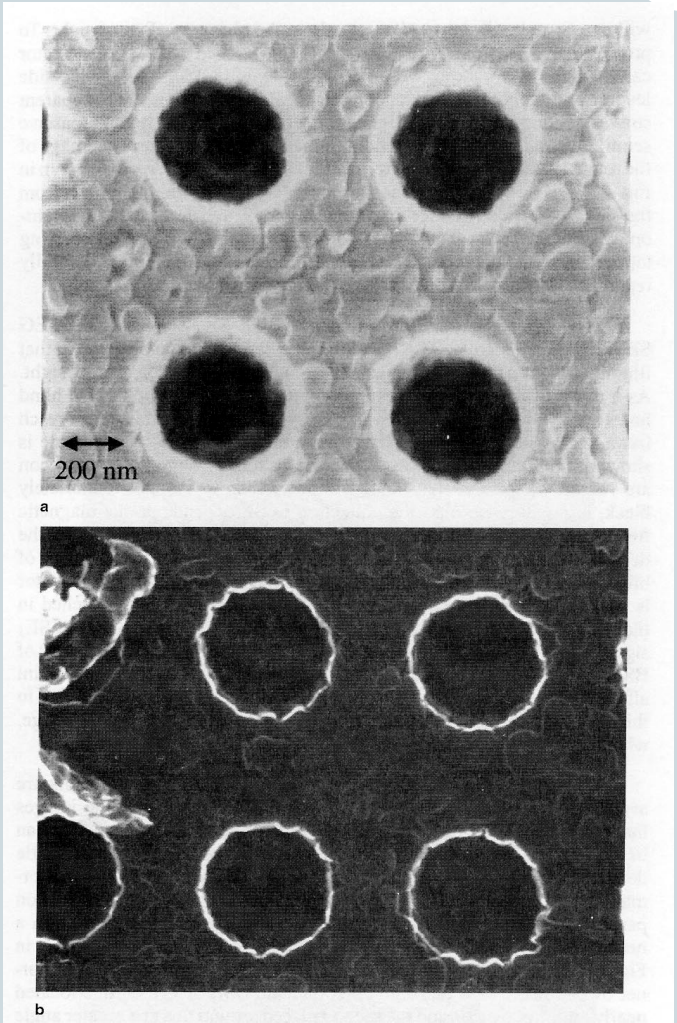


FEI

E_0 influence on the SE image

In-lens detector

Uma visão comparativa dos vários sinais



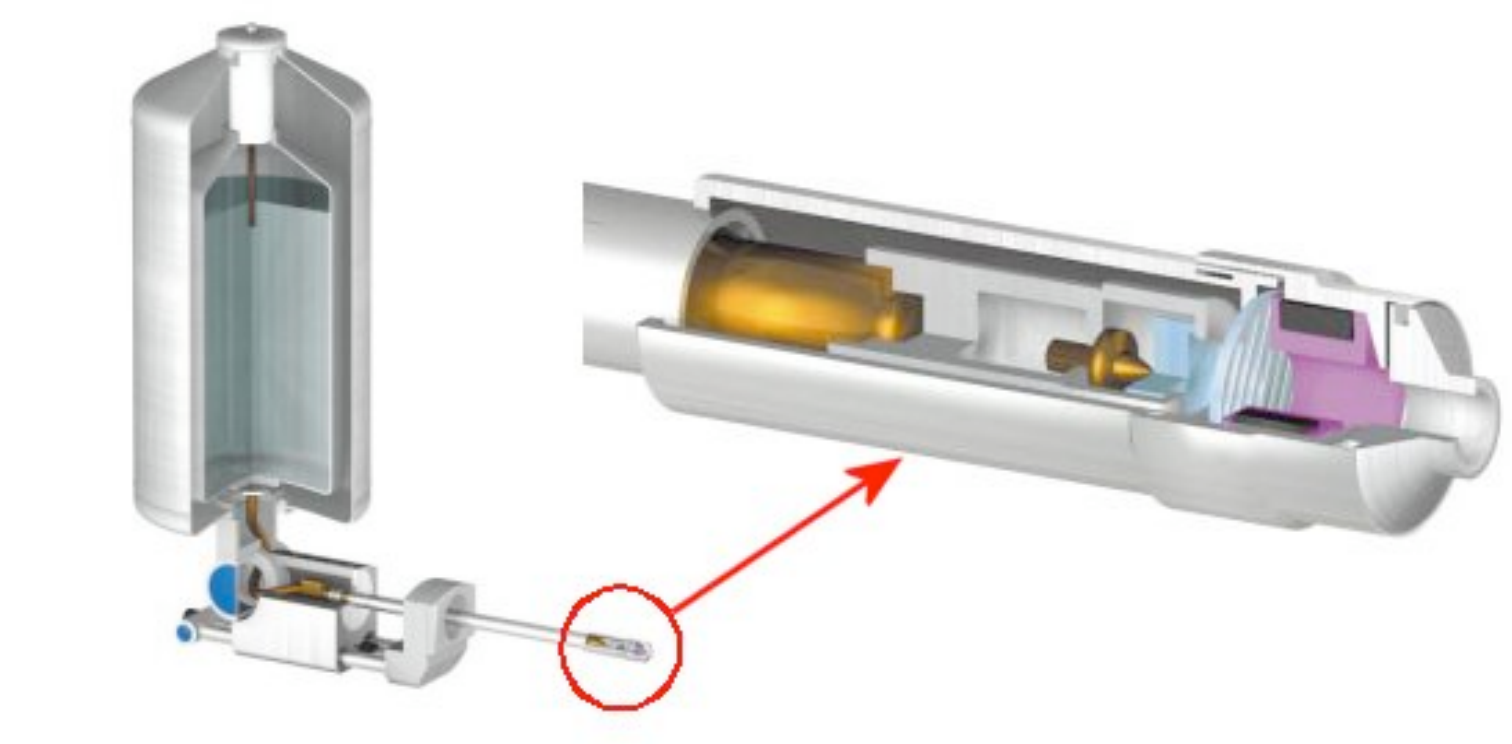
E-T lateral

E-T in lens

FEI

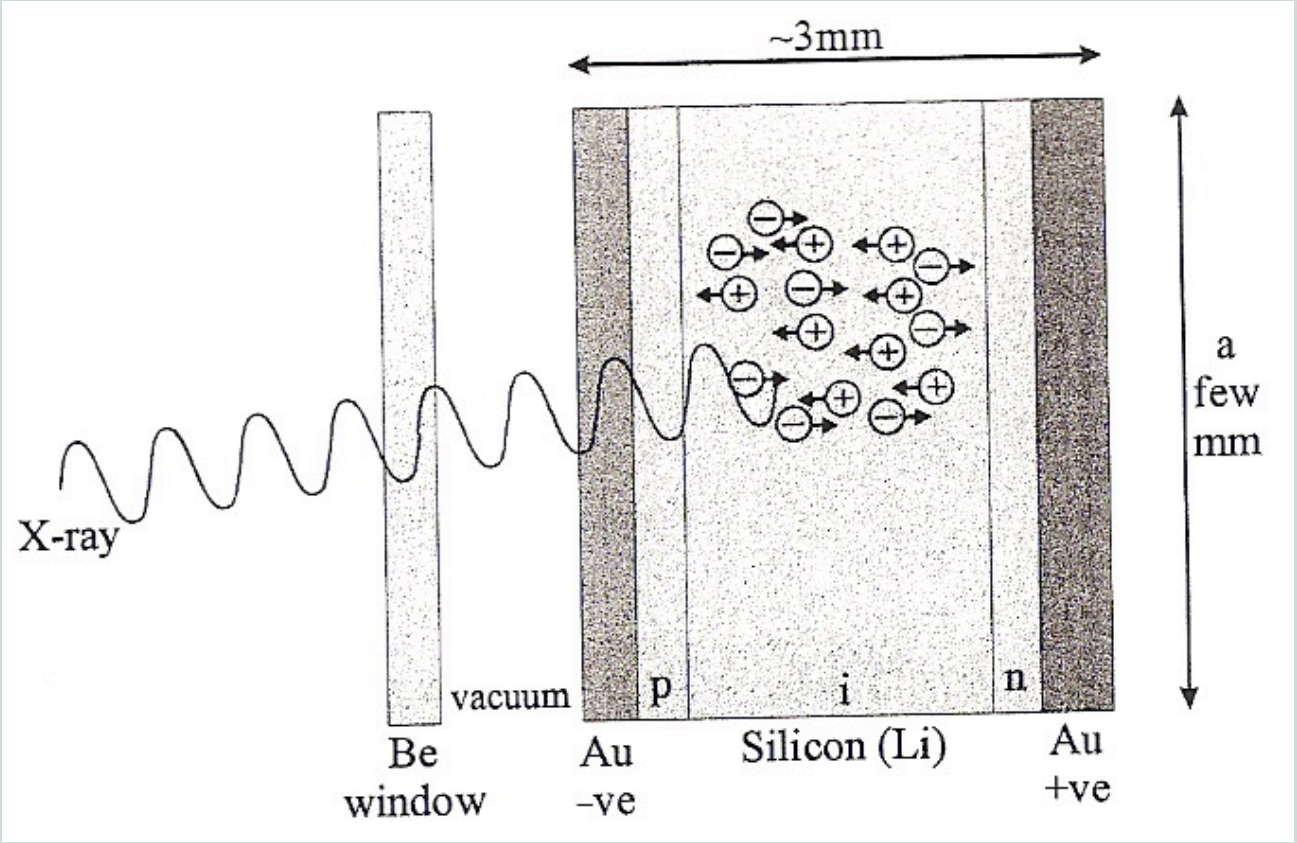
EDS – “Energy Dispersive Spectroscopy”

- Detector de Raios X



Brukers

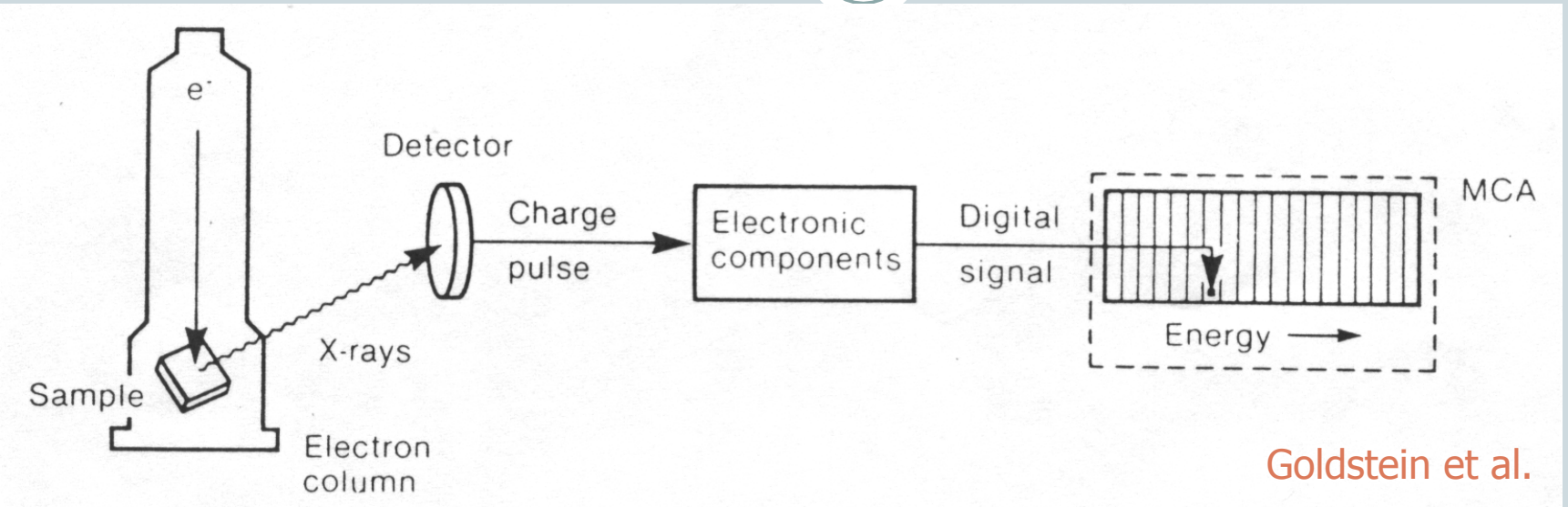
Detector de Raios X (SiLi)



Goodhew et all

Cada par elétron-buraco consome 3,8 eV

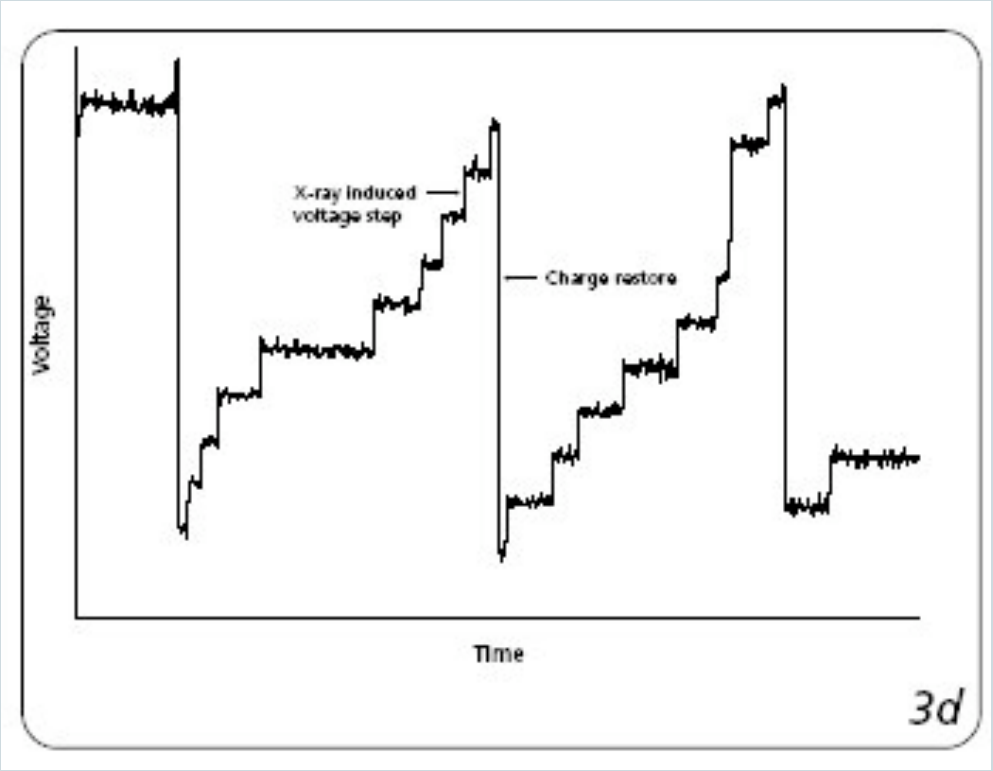
Processamento do Sinal



- Cada fóton gera um pulso de voltagem
- Os pulsos gerados são amplificados e digitalizados num intervalo de tempo, τ (10-50 μs), ou constante temporal
- Os pulsos são separados em diferentes canais com resolução energética de 20 eV

Processamento do Sinal

- Um menor τ implica em maior número de contagens por segundo (cps), mas com menor resolução energética

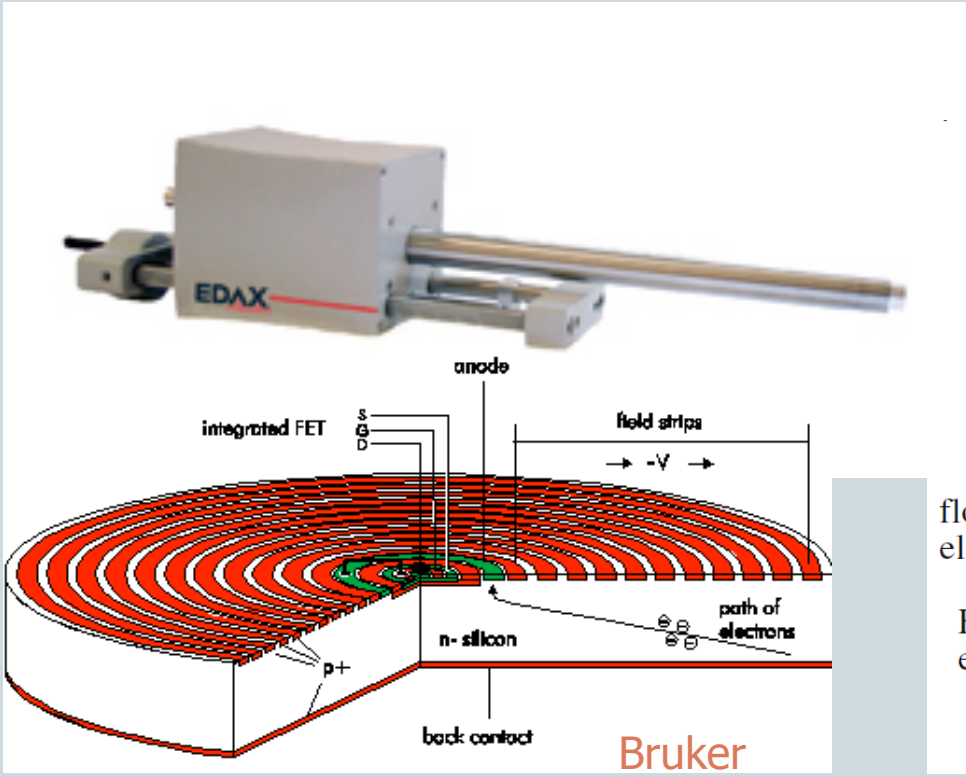


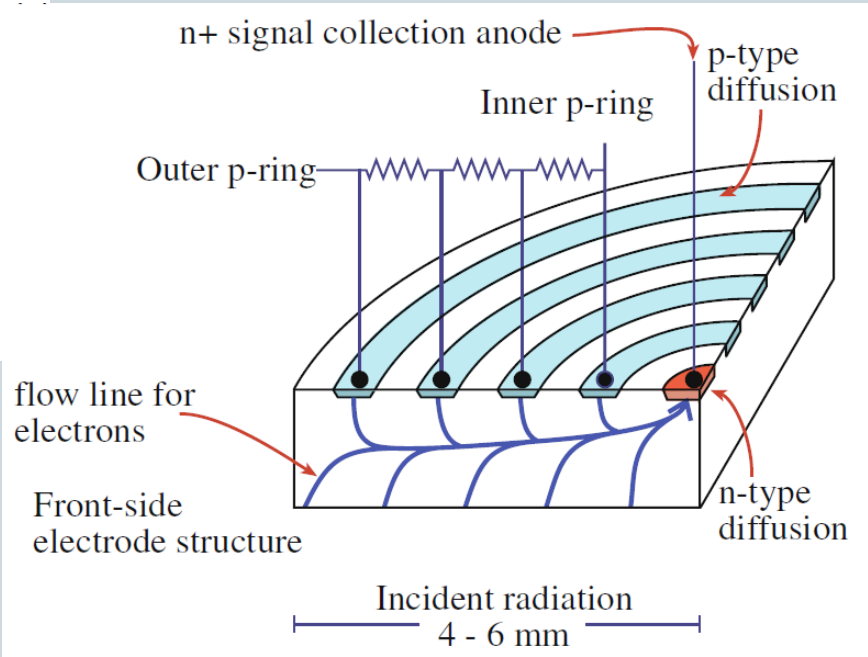
$$TempoMorto\% = \frac{(TempoTotal - TempoVivo)}{TempoTotal} x100\%$$

Goldstein et al.

Silicon Drift Detector (SDD)

- Refriado por Efeito Peltier
- Resolução ~ 145 eV
- Maiores taxas de contagem (~100.000 cps)





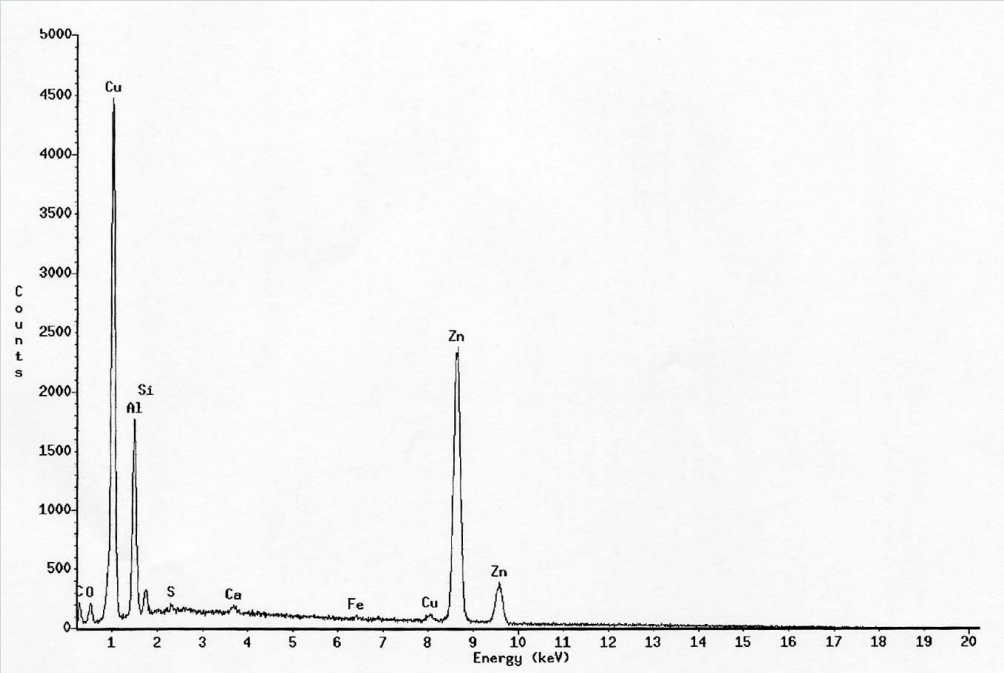
Si(Li) x SDD

- Resolução energética ~ 148 eV
- Melhor resolução energética ~ 127 eV
- 5-10.000 cps
- -196ºC (N_2 Liq)

- Resolução energética ~ 140 eV
- Melhor resolução energética ~ 123 eV
- 1.000-100.000 cps
- -25ºC (Termoelétrico)

Espectro de EDS

- Permite a análise qualitativa da composição química
- Permite a realização de análise de composição atômica semi-quantitativa
- Permite a realização de mapeamentos para localização de elementos químicos



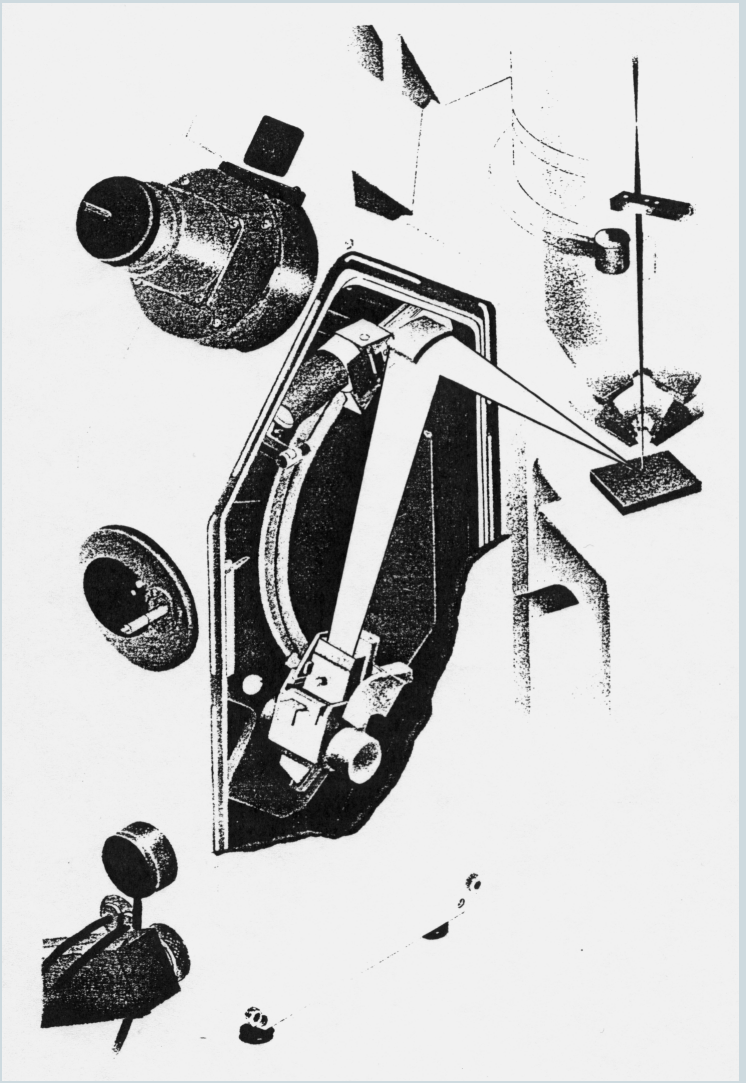
WDS – “Wavelength Dispersive Spectroscopy”

- Neste caso, a energia do fóton de R-X é avaliada através do seu comprimento de onda.

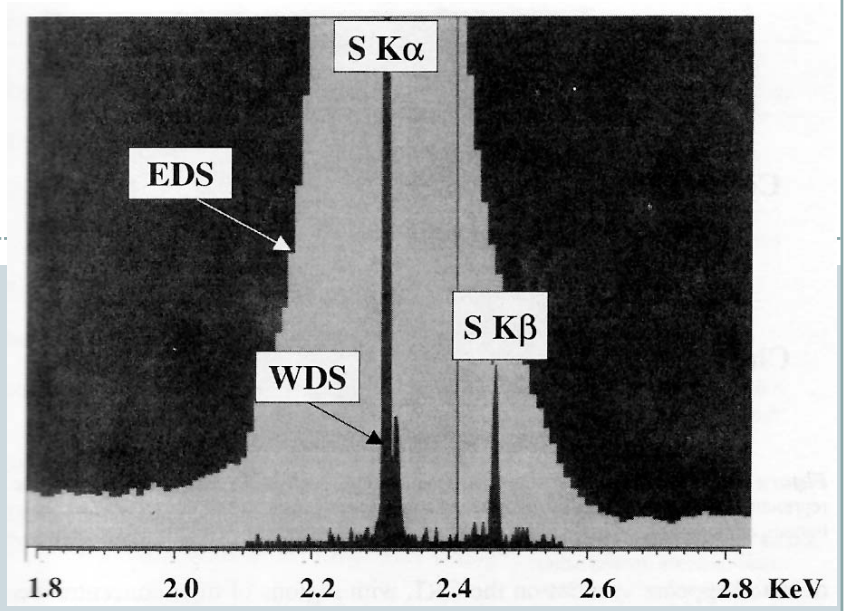
$$E = \frac{h}{\lambda}$$

- O comprimento de onda é avaliado através da difração por um monocristal com ângulo de posicionamento variável.
- Lei de Bragg

$$n\lambda = 2d\,\mathrm{sen}\,\theta$$



Comparação EDS x WDS



EDS

- Rápida análise qualitativa
- Fácil de usar
- Barato
- Baixa resolução energética (150 eV)
- Alto limite de detectabilidade (1%)
- Baixa detectabilidade de elementos leves

WDS

- Análise qualitativa lenta
- Exige muitos cuidados com alinhamento
- Caro
- Alta resolução energética (5 eV)
- Baixo limite de detectabilidade (0,01 %)
- Excelente detectabilidade de elementos leves

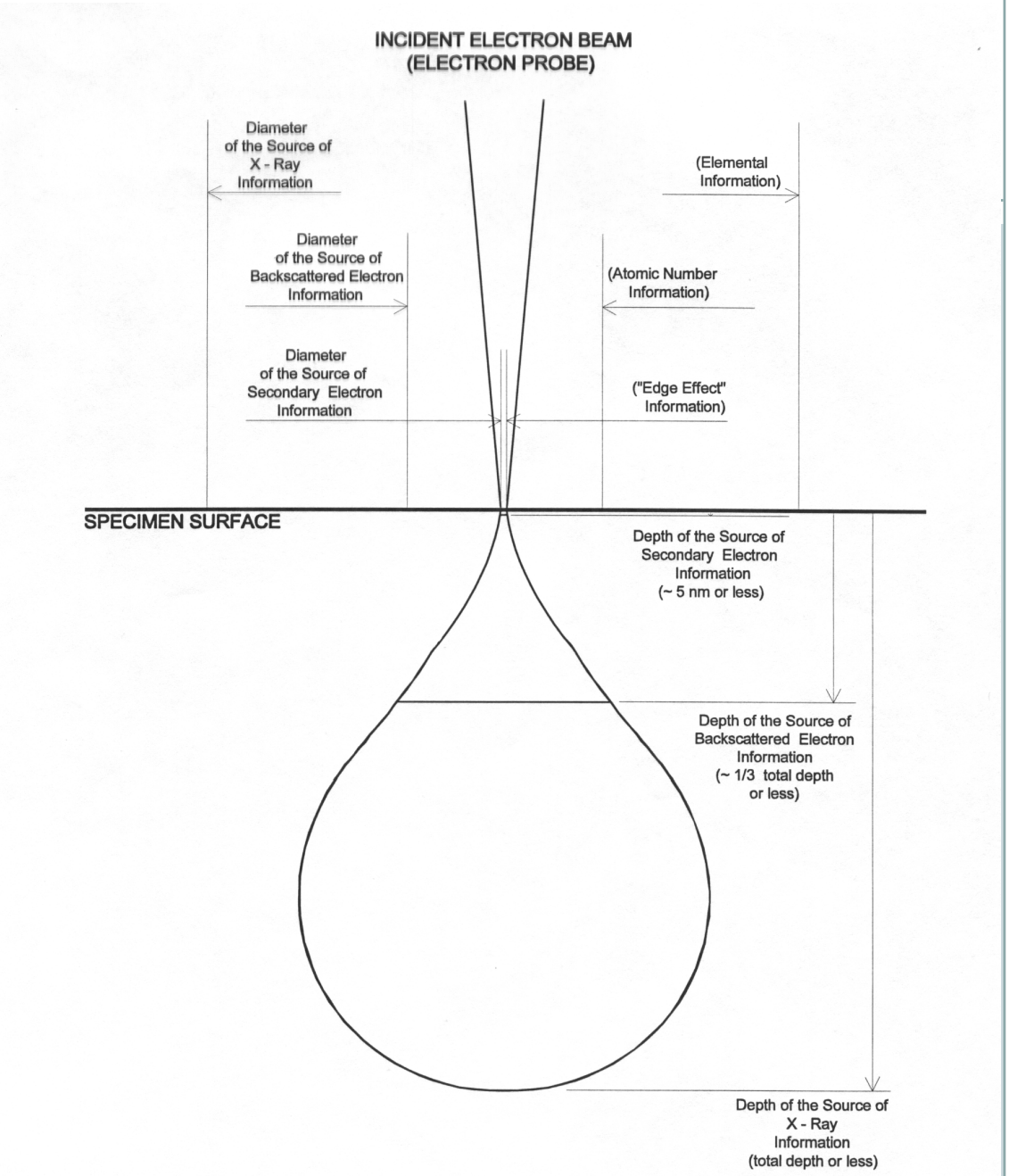
Resolução e origem

- e^- Secundários

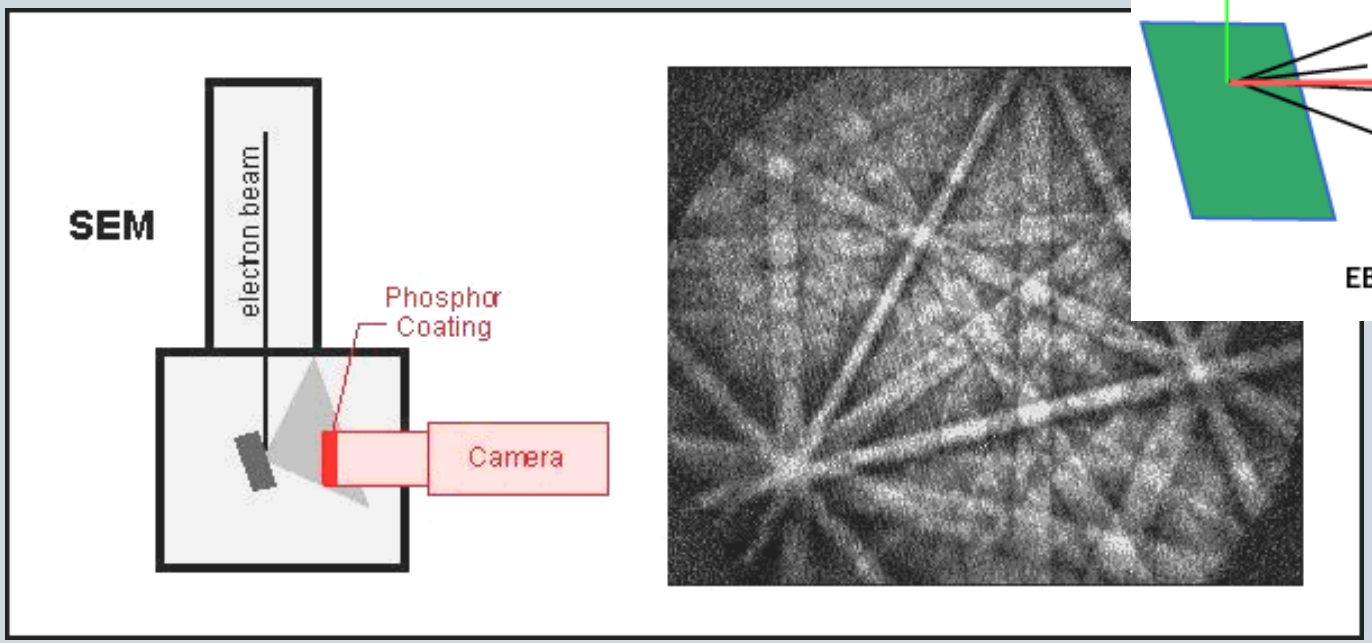
  ~ 1 ηm
- e^- Retroespalhados

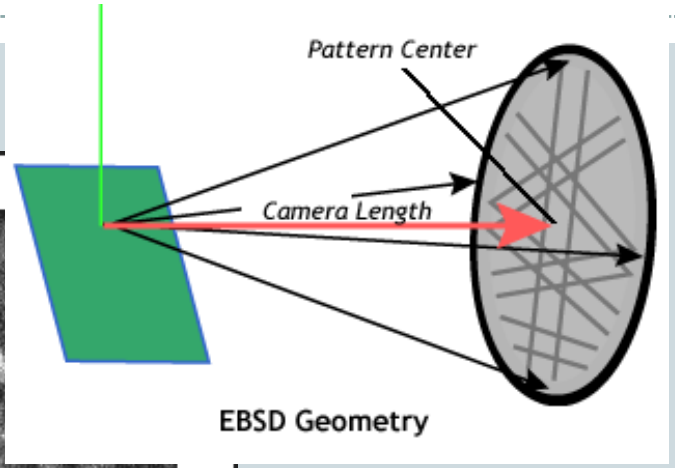
  ~ 0,1 μm
- EDS

  ~ 1 μm



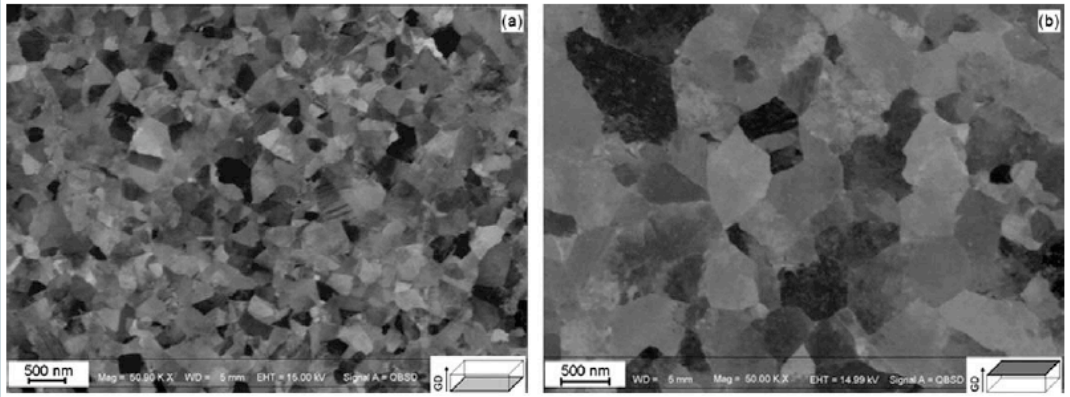
EBSD

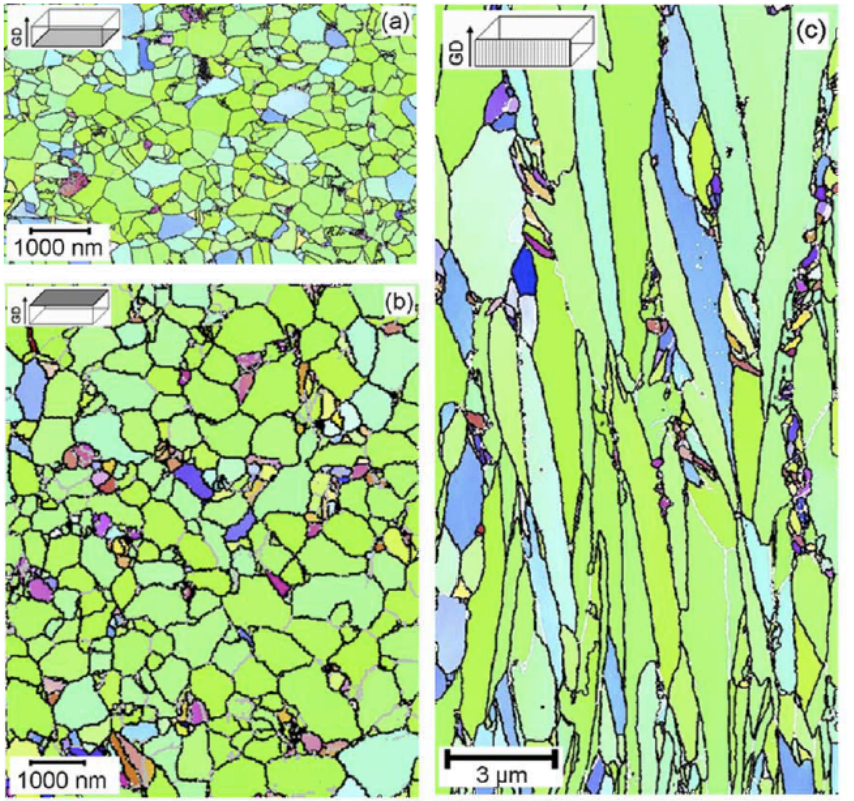


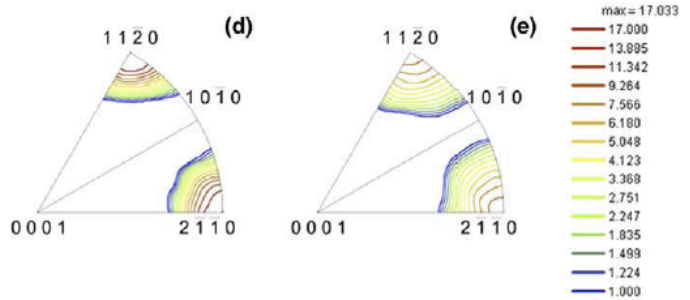


- A largura das bandas está diretamente relacionada ao espaçamento interplanar.
- O ângulo entre as bandas corresponde ao ângulo entre os planos cristalinos.
- A posição das bandas está ligada à posição dos planos.

NiCo Nanoestruturado Eletrodepositado



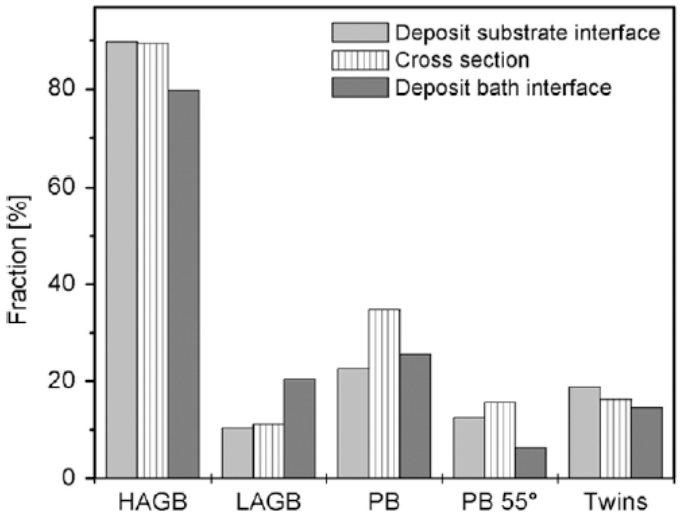


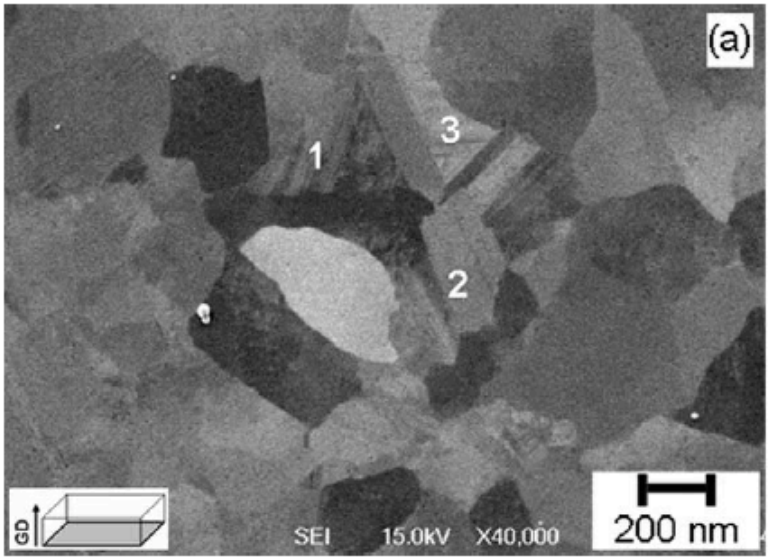


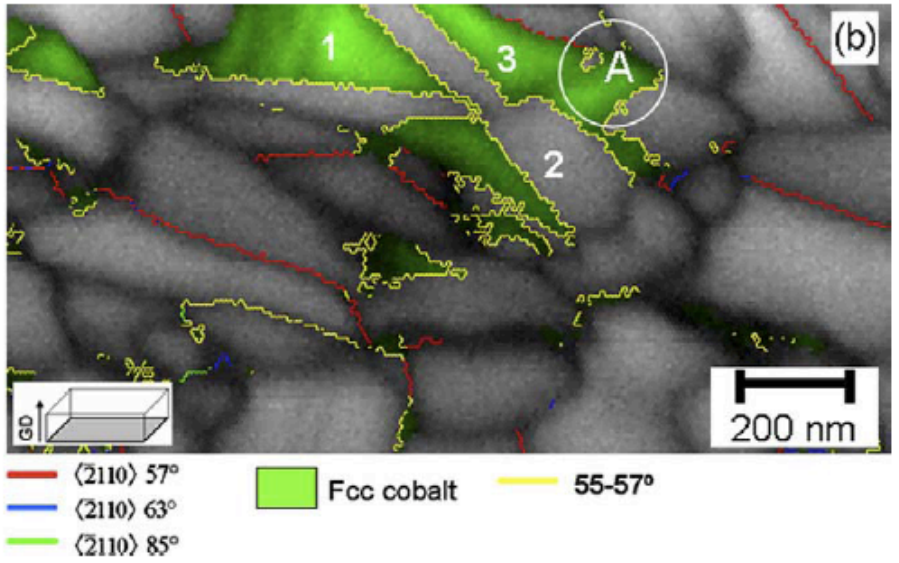
Bastos et all 2006

NiCo Nanoestruturado Eletrodepositado

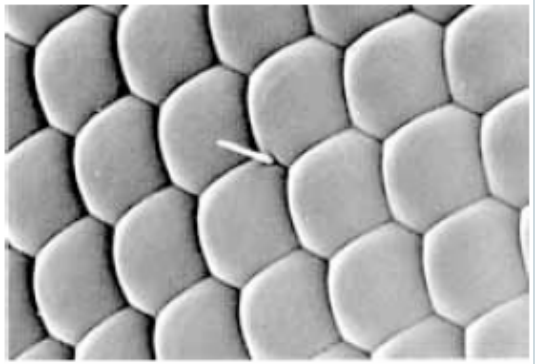
Bastos et all 2006



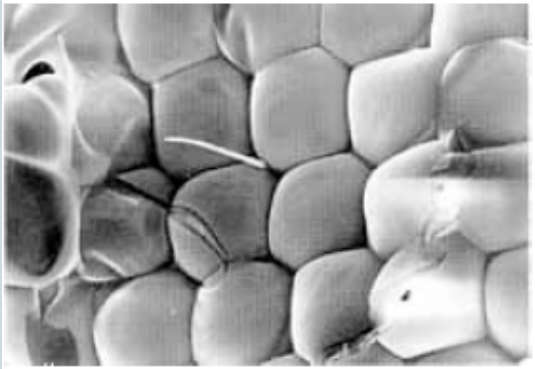




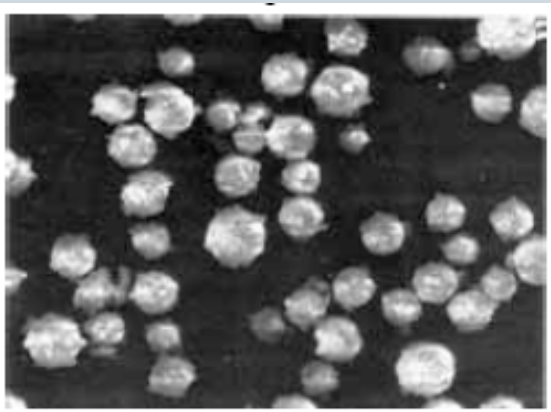
Danos



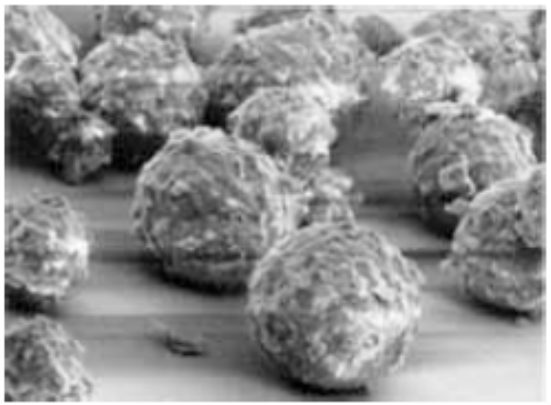
(a) Undamaged specimen



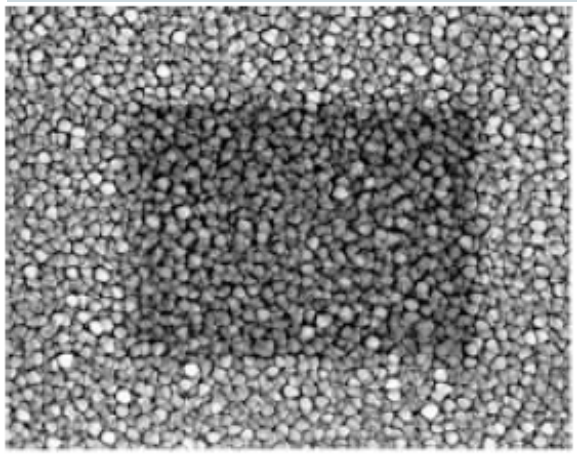
(b) Damaged specimen



(c) 15 kV Tilt angle: 0° x720

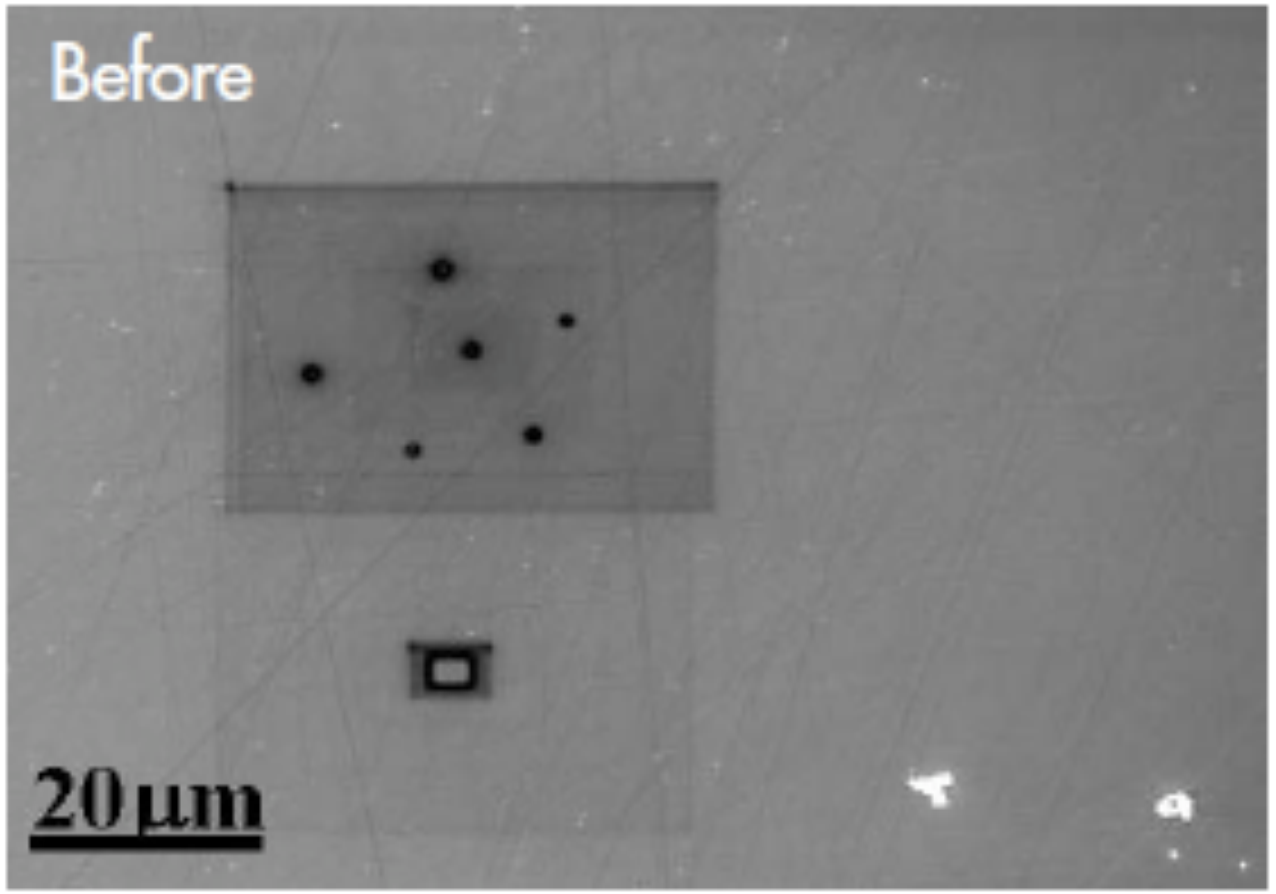


(d) 5 kV Tilt angle: 45° x1,400



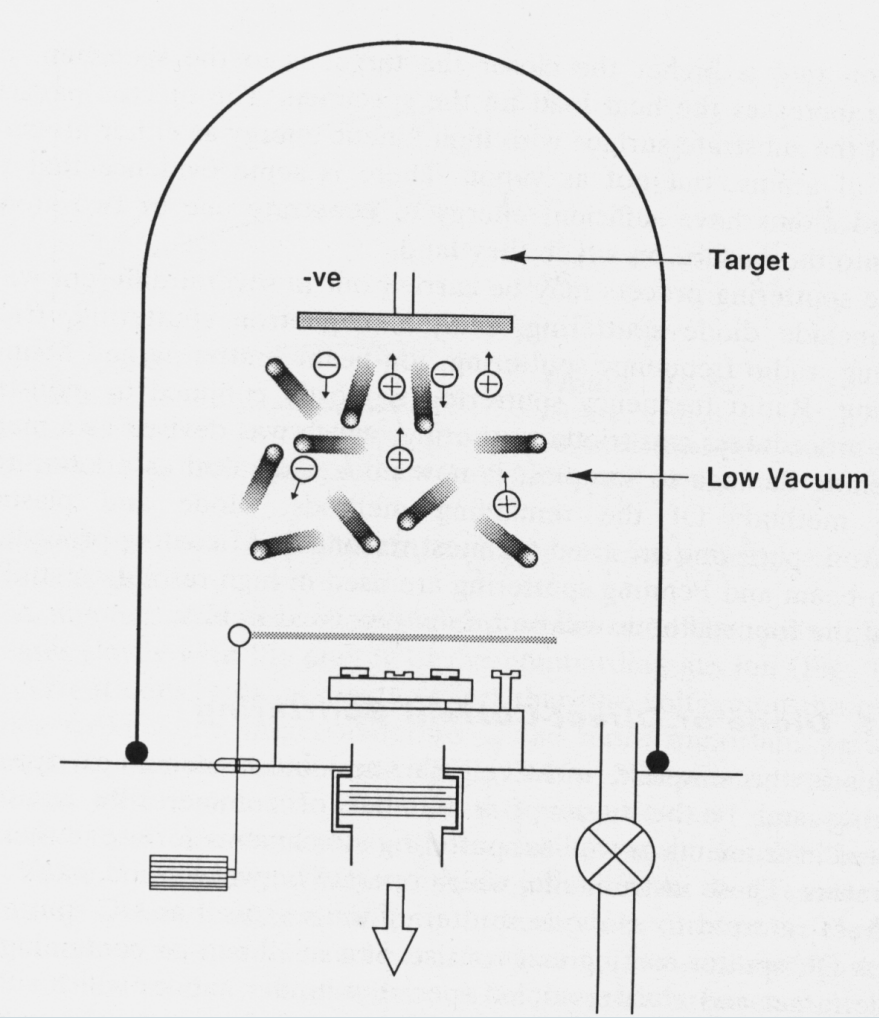
5 kV x18,000

Danos

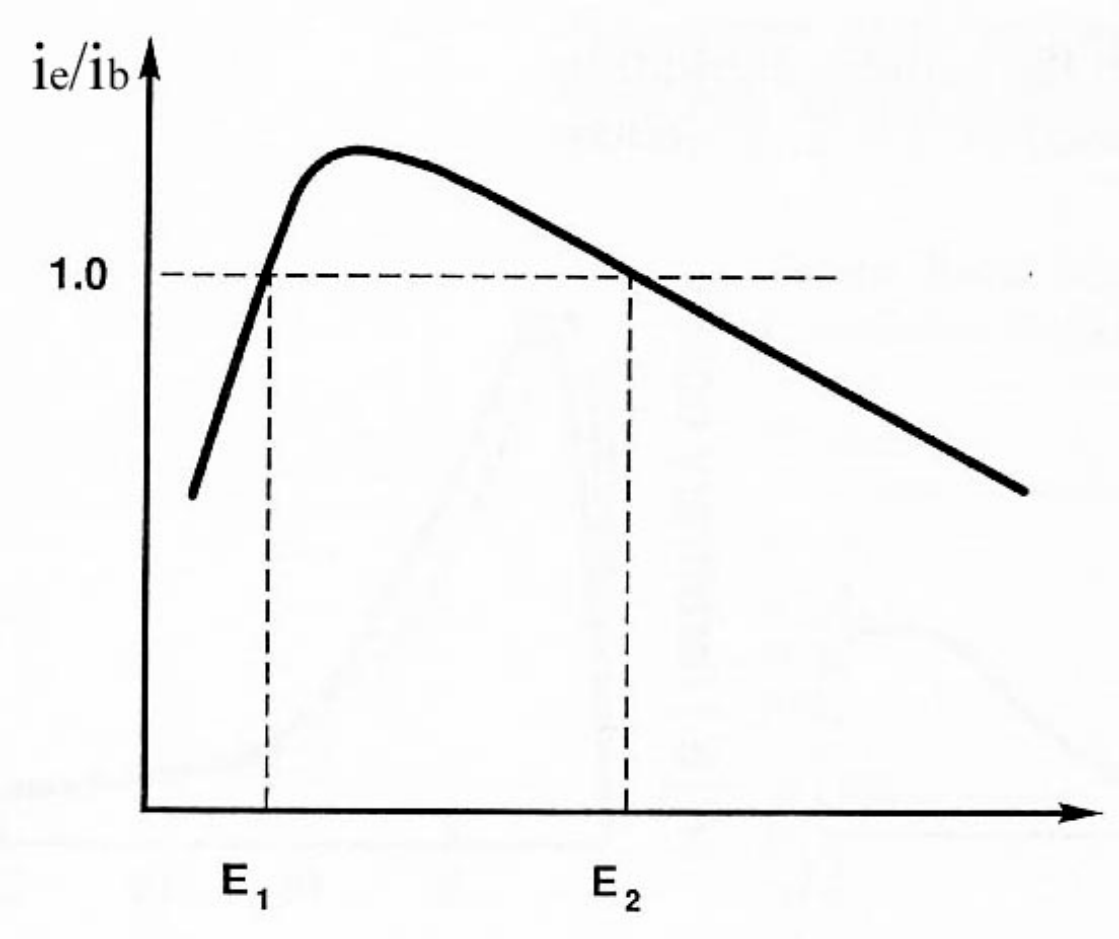


Deposição de Camada Condutora

- A deposição de Au, , Ag, Pt, Ni e Cr por "sputtering" proporciona boa resolução, mas pode representar um problema em EDS e em amostras sensíveis.
- A deposição de C por evaporação costuma resolver este problema.

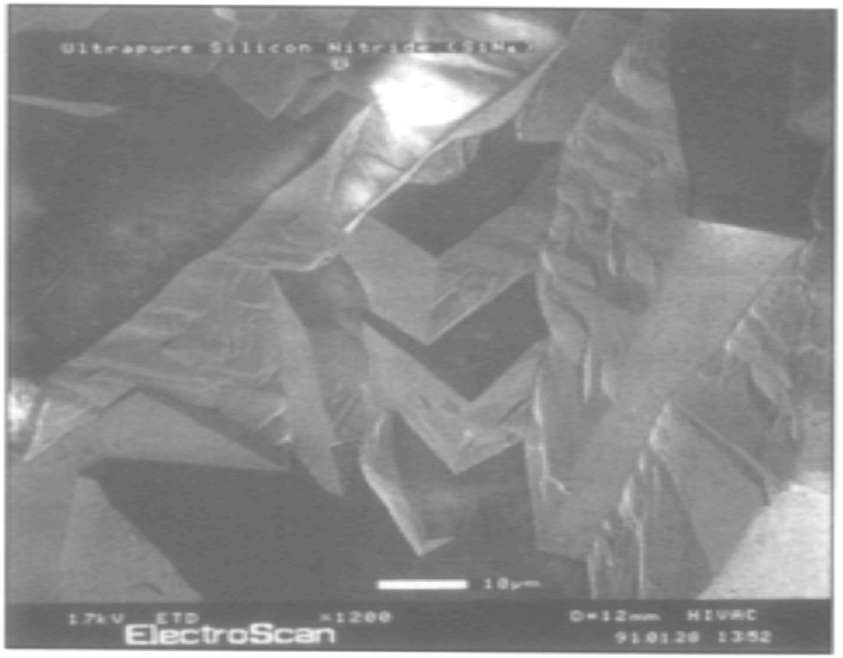


Observação em Baixa Voltagem



Observação em Baixa Voltagem

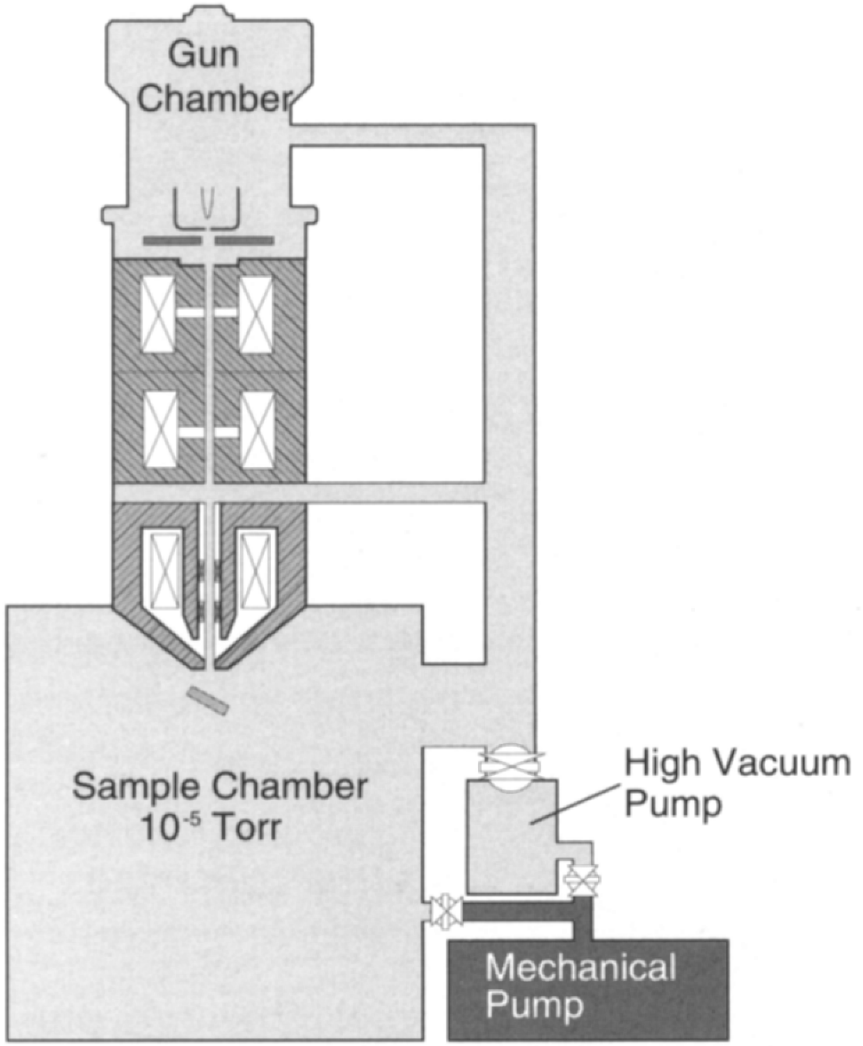




- Todo material possui uma voltagem onde ocorre o equilíbrio de cargas, em geral entre 0,5 kV e 5 kV.

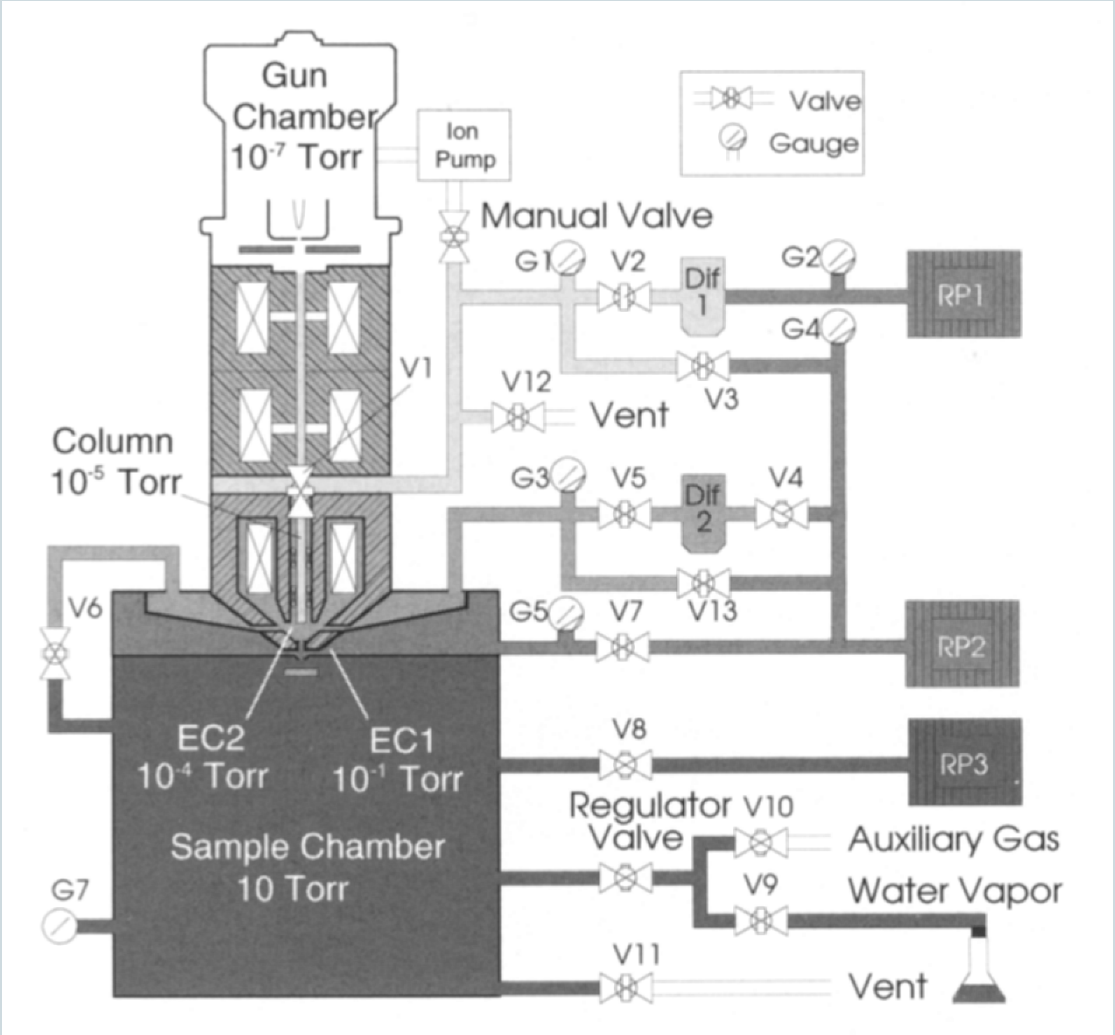
Observação em Alto Vácuo

- Canhão de elétrons, coluna e câmara permanecem à pressão de vácuo em torno de 10^{-6} Torr



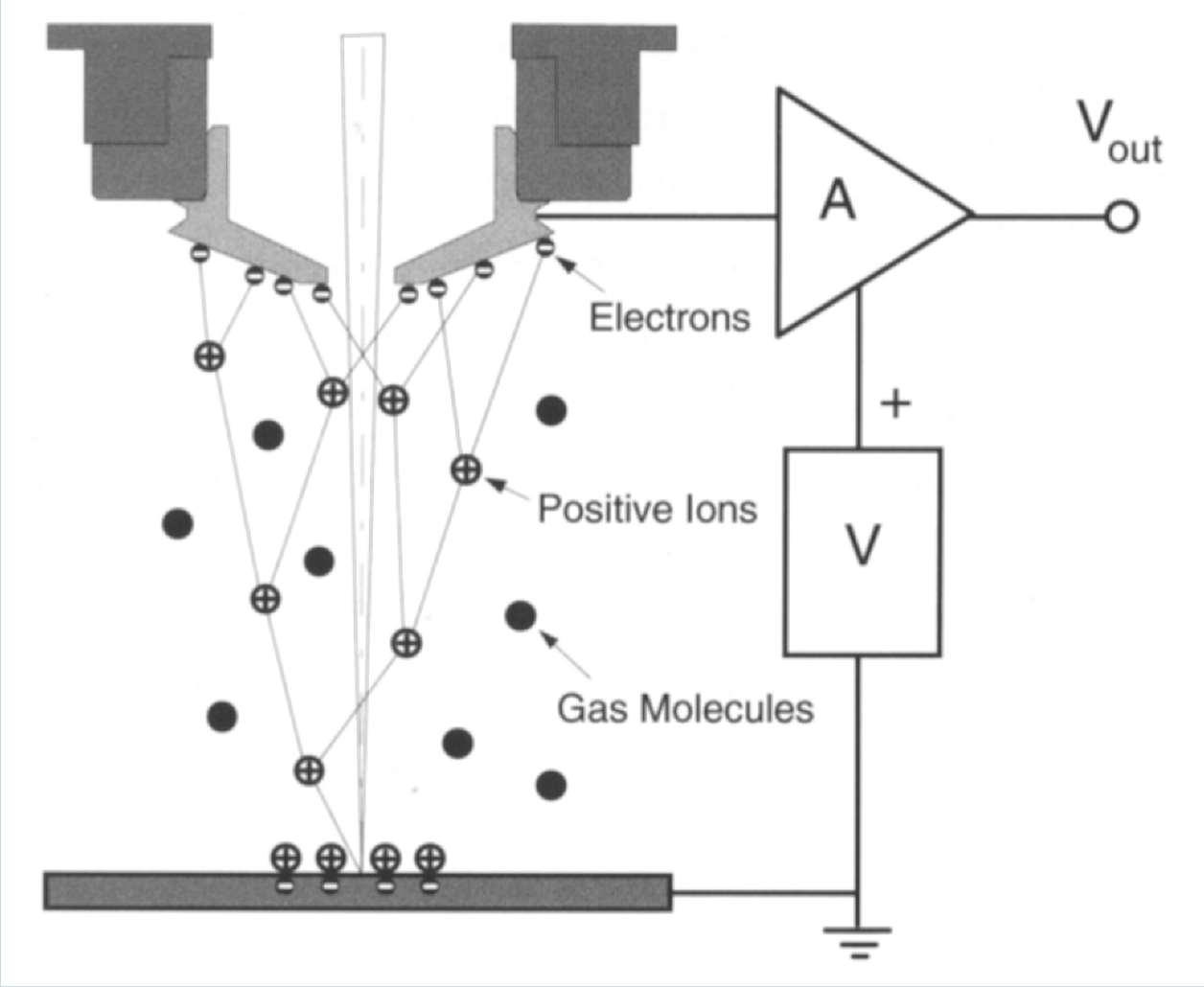
Observação em Baixo Vácuo

- Detector de Everhart-Thornley precisa ser desligado.
- A única imagem disponível é de elétrons retroespalhados.

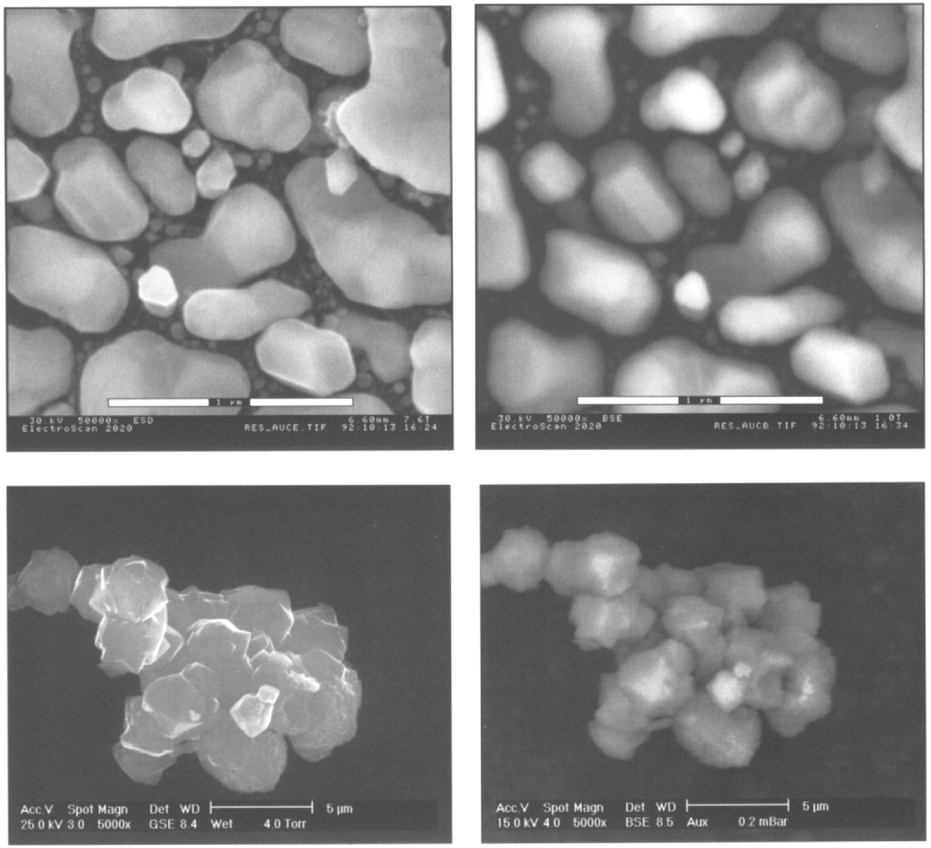


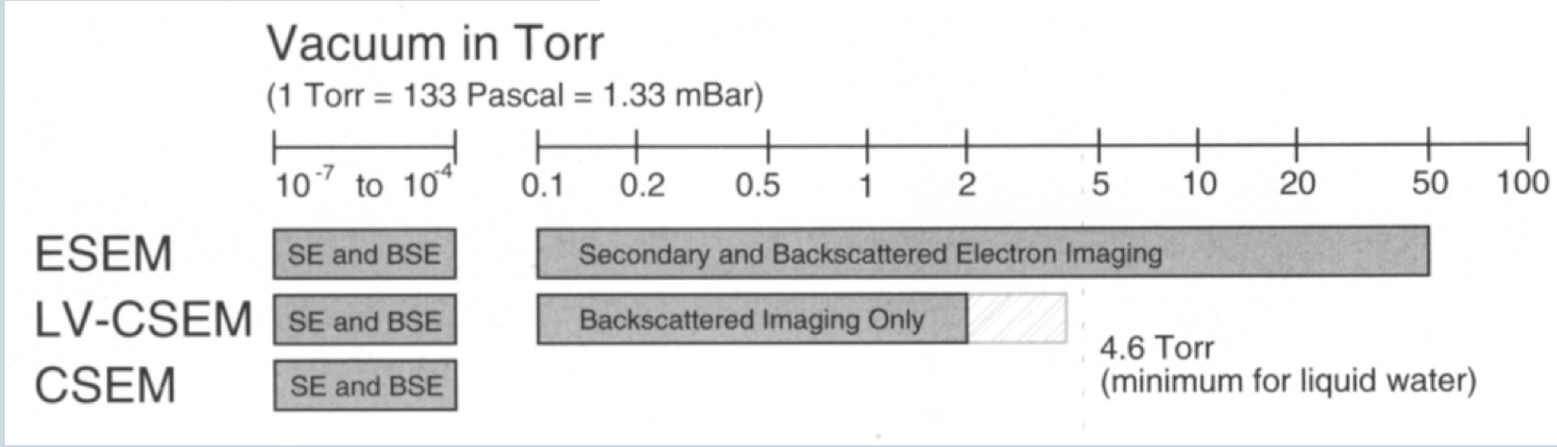
MEV Ambiental

- Novos detectores permitem a observação da imagem de elétrons secundários em baixo vácuo.

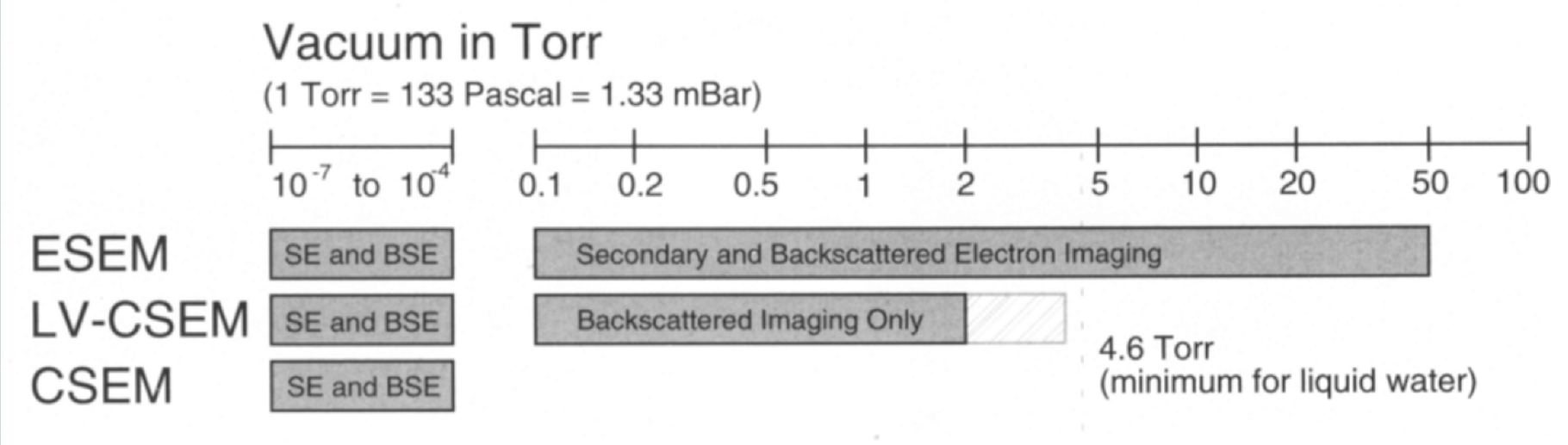


Ambiental x Baixo Vácuo

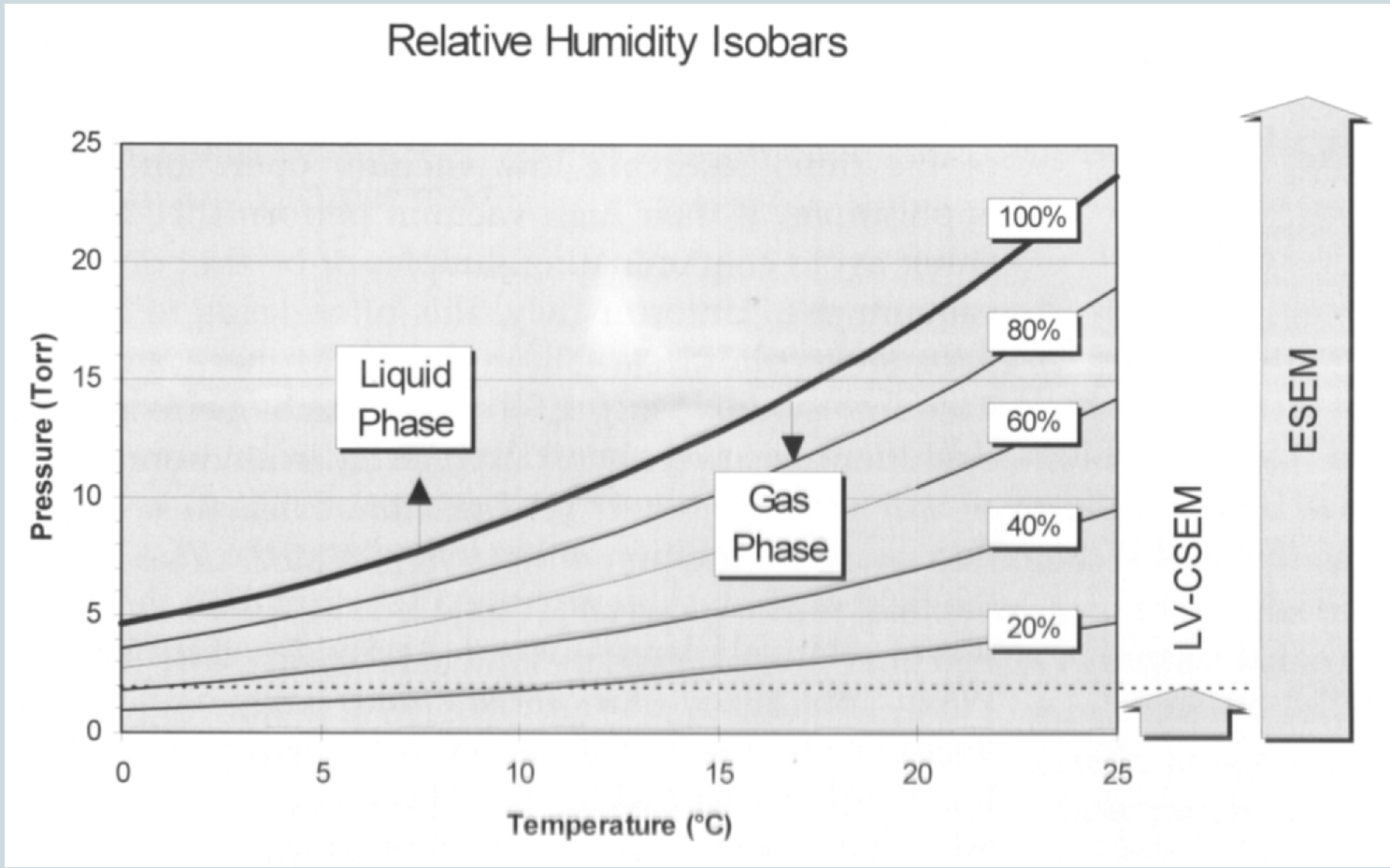




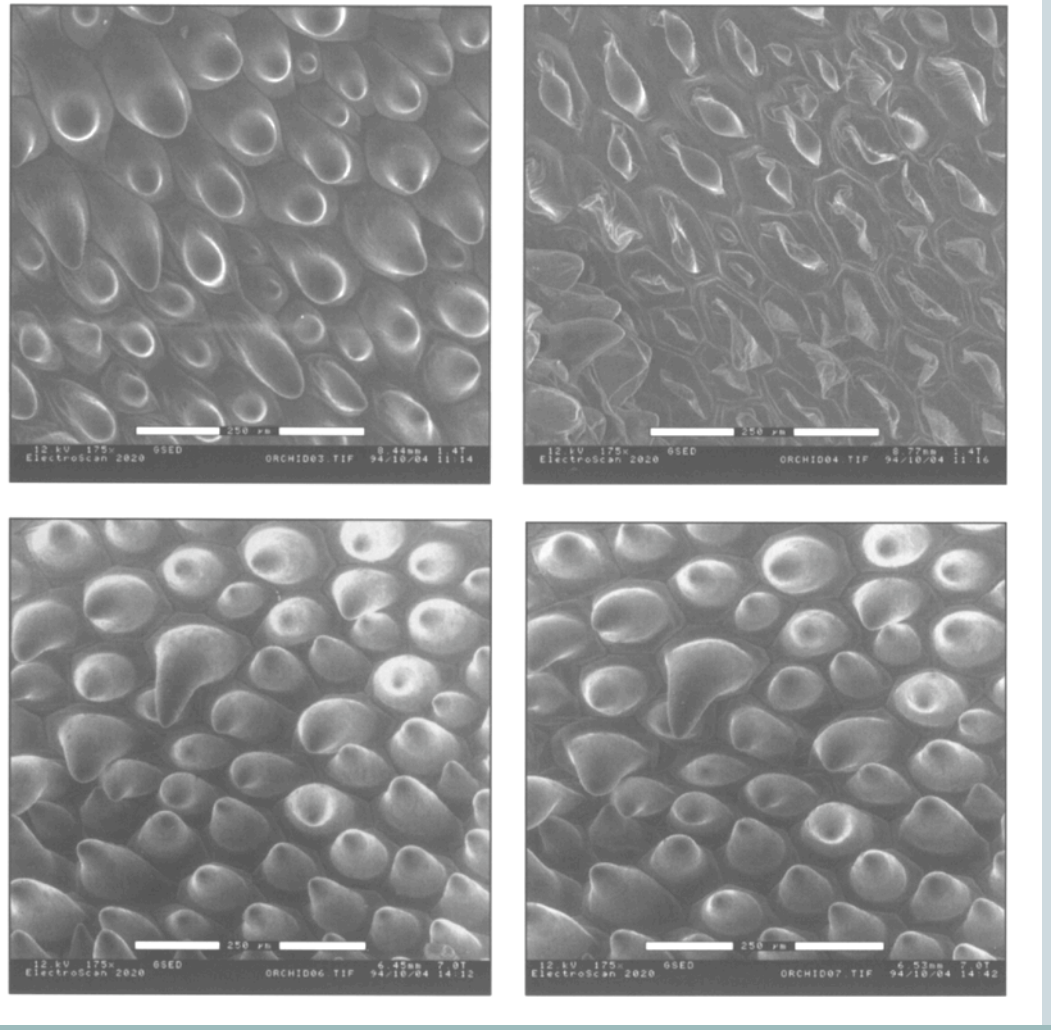
Ambiental x Baixo Vácuo



Ambiental x Baixo Vácuo



Ambiental x Baixo Vácuo



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- Apresentação Gatan
- Site EDAX (www.edax.com)
- Site www.x-raymicroanalysis.com

- Obrigado pela atenção

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