

Microscopia

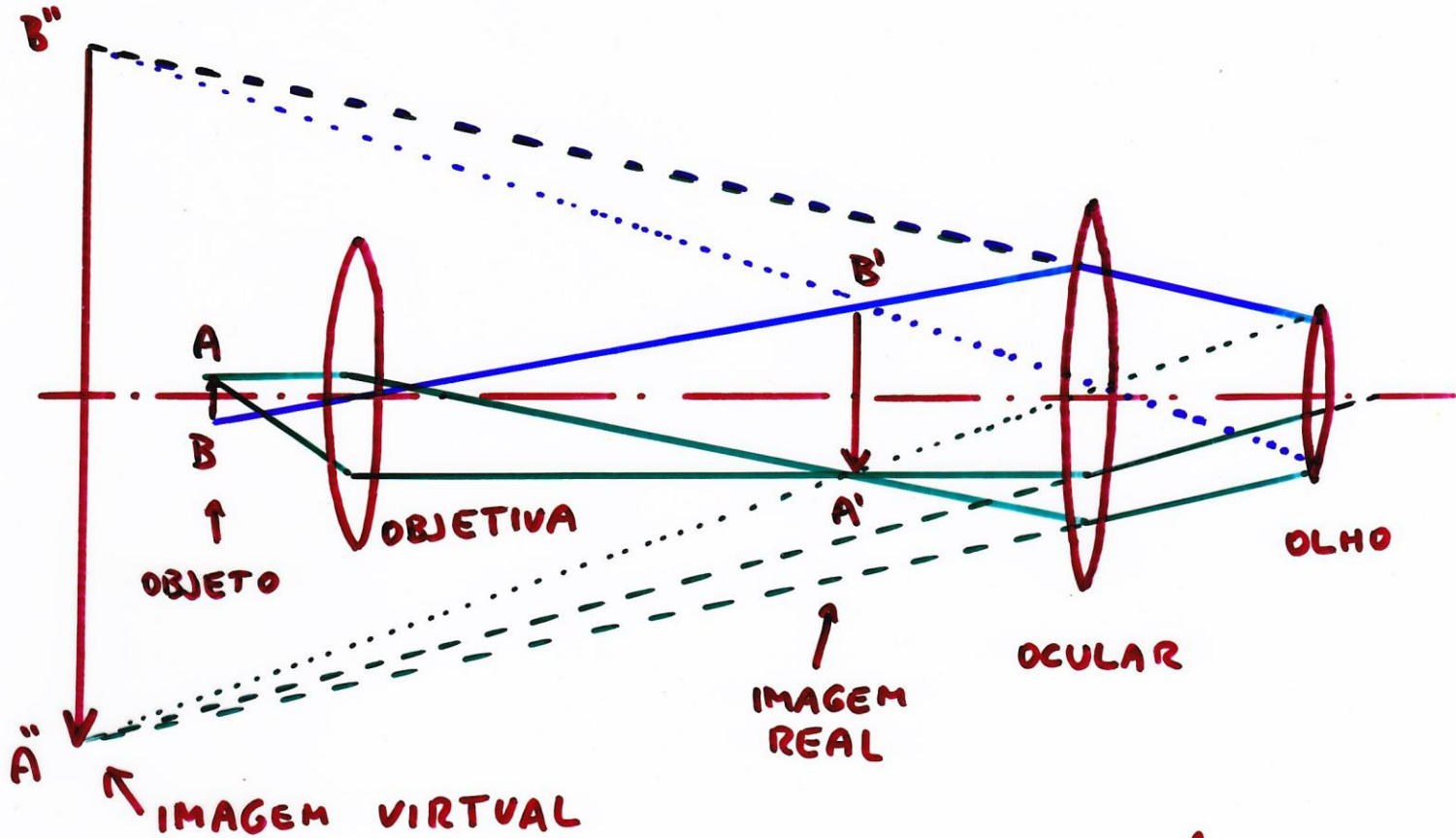
Material de apoio

CABENS

outubro de 2011

A.C.Neiva

4. MICROSCÓPIO ÓPTICO (LUZ VISÍVEL)

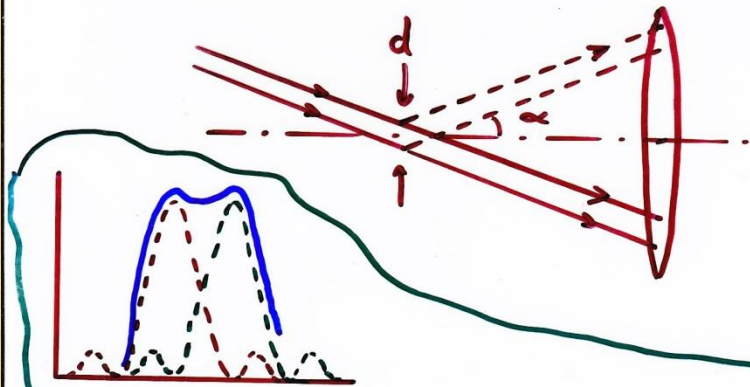


Aumento total

$$M = M_{ob} \cdot M_{oc}$$

objetiva ocular
↓ ↓

LIMITE DE RESOLUÇÃO DA OBJETIVA



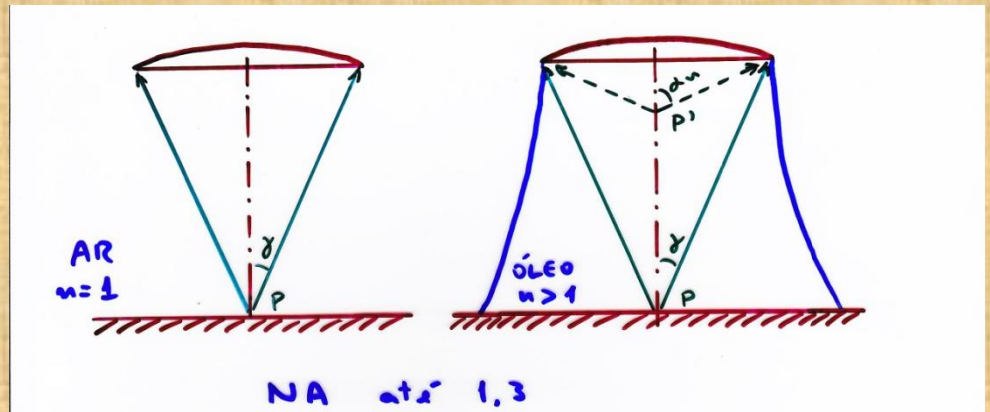
Abbe'

$$d_{min} = \frac{\lambda}{2 n \sin \alpha}$$

$$n \sin \alpha = AN$$

"abertura numérica"

Rayleigh $\rightarrow d_{min} = \frac{1.22 \lambda}{2 n \sin \alpha}$



AUMENTO ÓTIMO

↳ Olho → distingue $0^{\circ}1' \cong \sim 100 \mu\text{m}$

Aumento ótimo → 2 a 4 vezes maior

Exemplos: ($\lambda = 580 \text{ nm}$, amarela)

a) Objetiva de $10\times$, $NA = 0,15$

∴ Resolução = $1935 \text{ nm} \cong 1,935 \mu\text{m}$

Mínimo aumento $\cong 100 / 1,935 \cong 52,5\times$

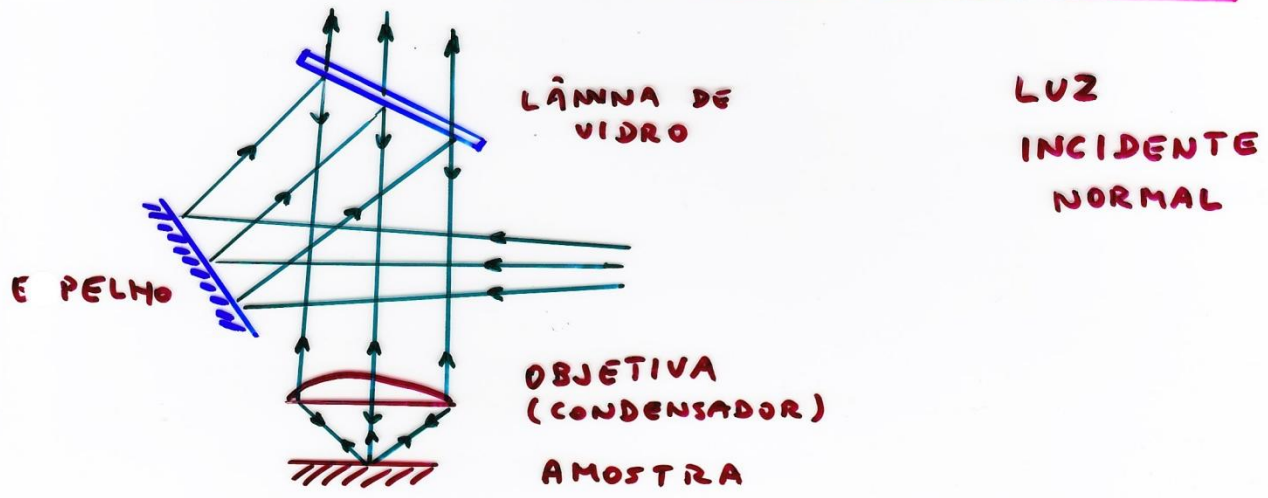
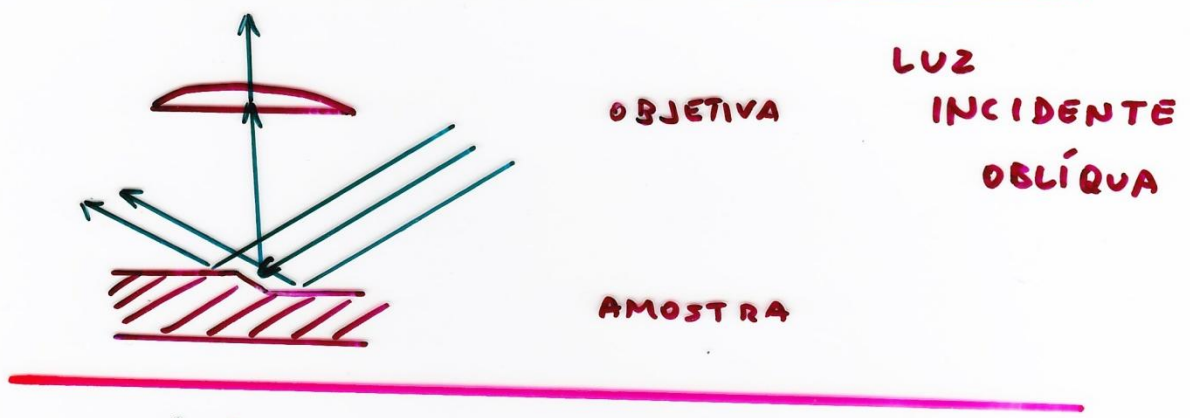
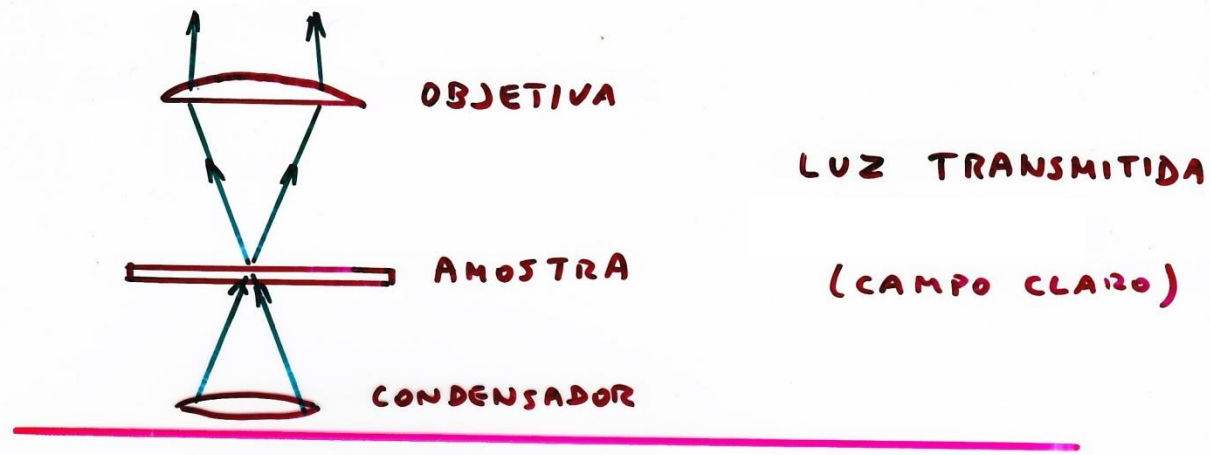
Aumento ótimo = 100 a 200 \times

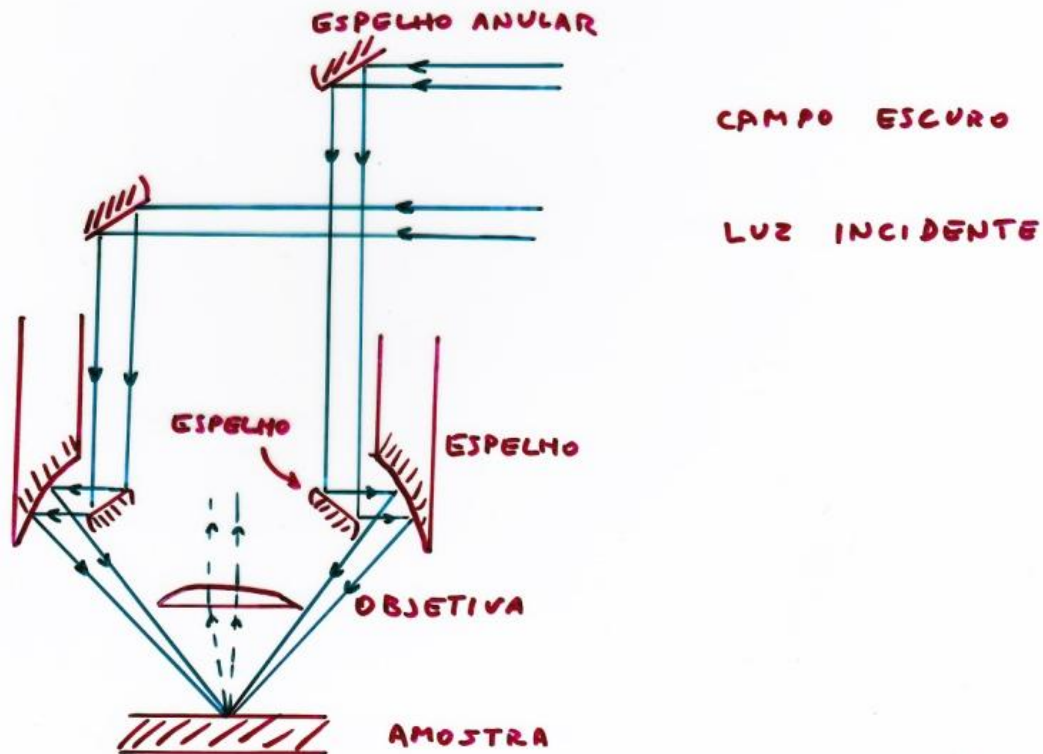
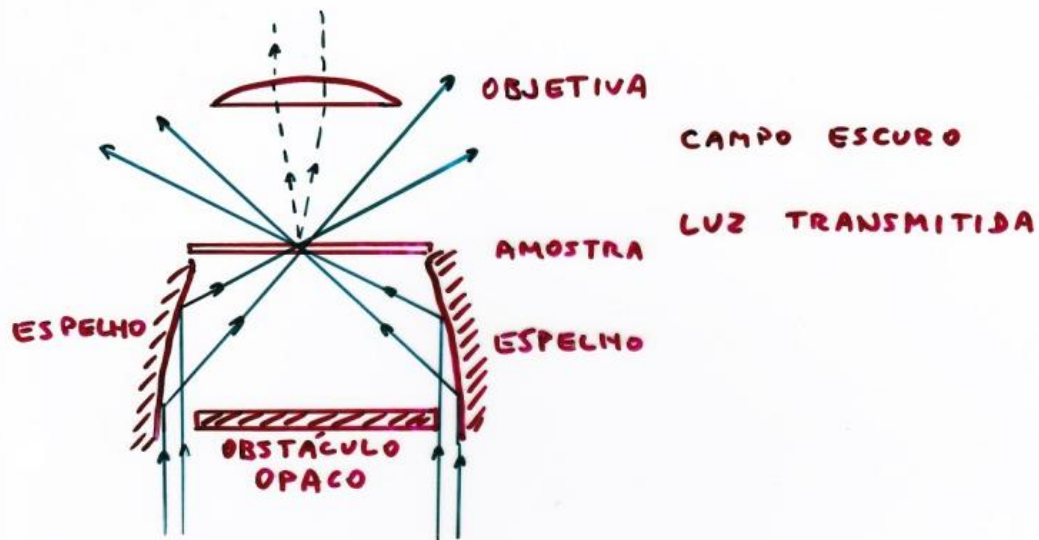
b) Objetiva de $95\times$, $NA = 1,3$

∴ Resolução = 223 nm

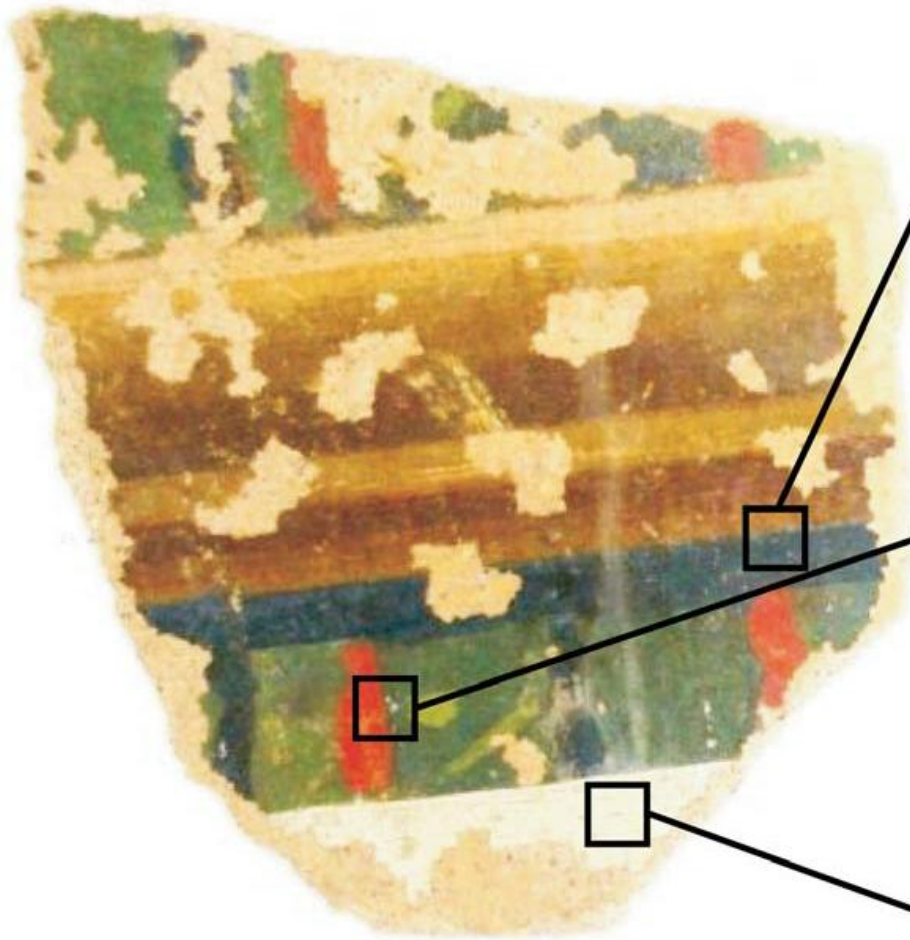
Mínimo aumento $\cong 100 / 0,223 = 460\times$

Aumento ótimo = 1000 a 2000

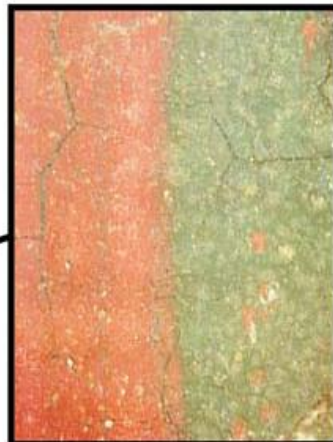




P1-1



5X

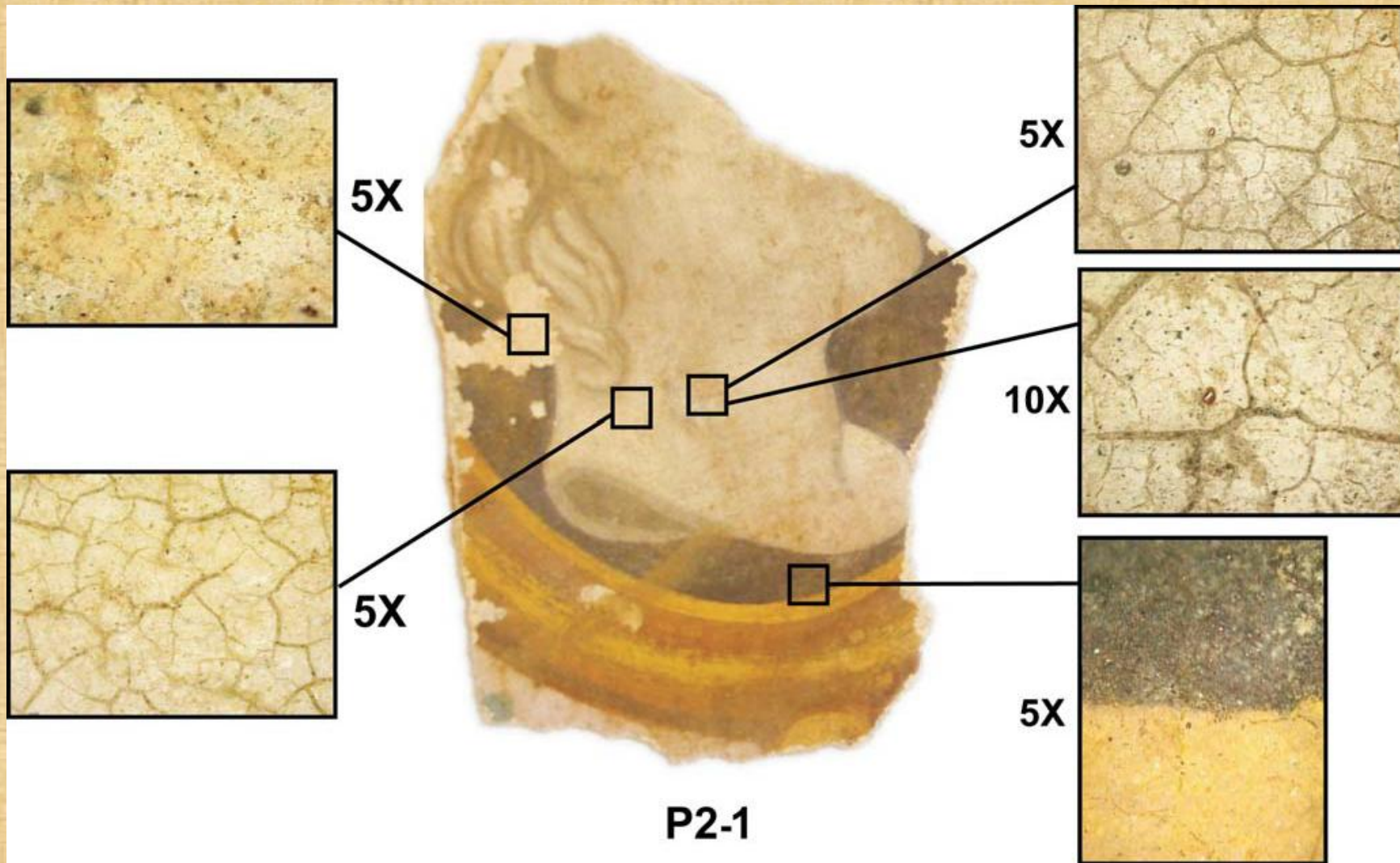


5X

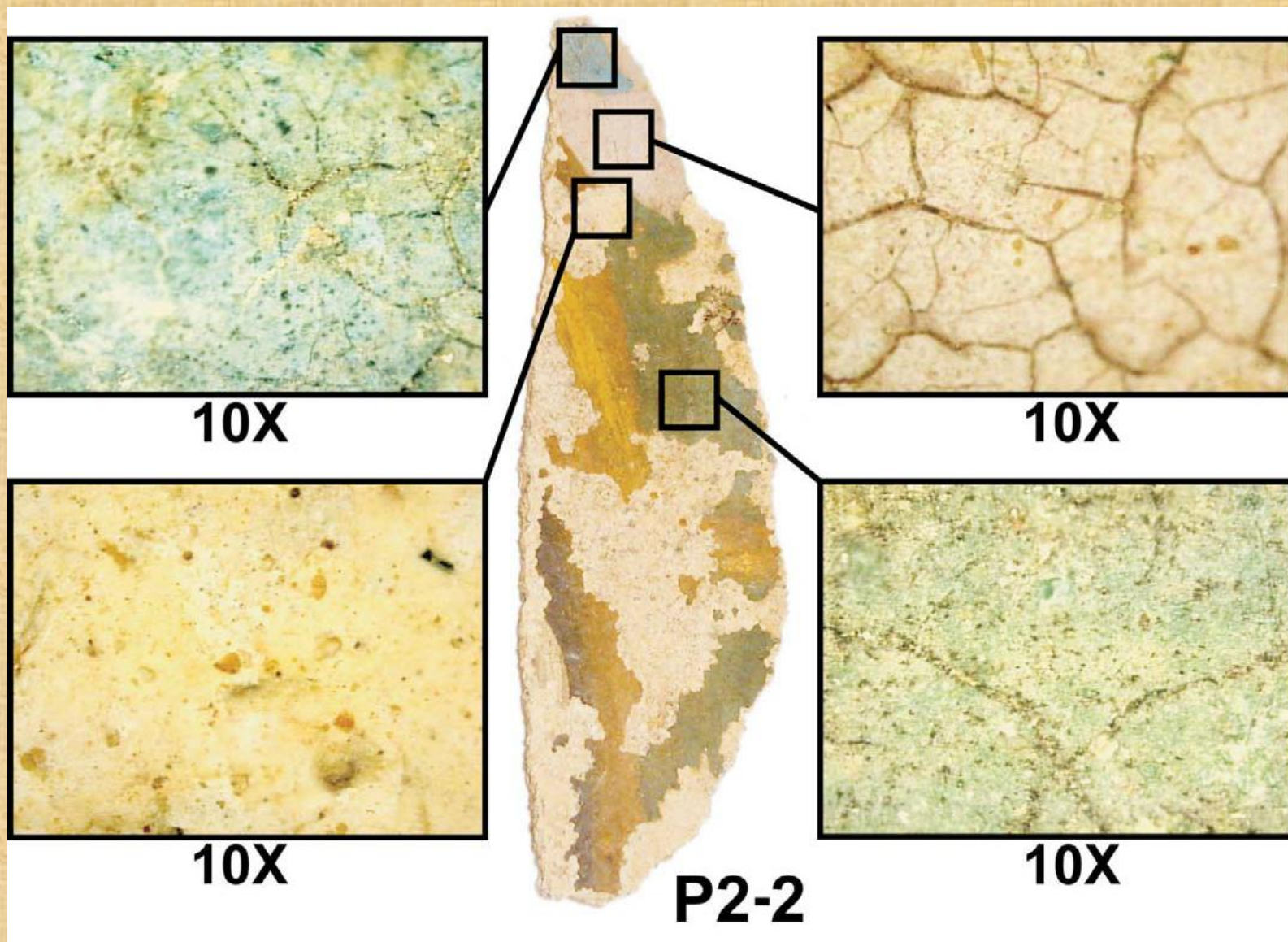
10X



Neiva e Tirello



Neiva e Tirello



Neiva e Tirello

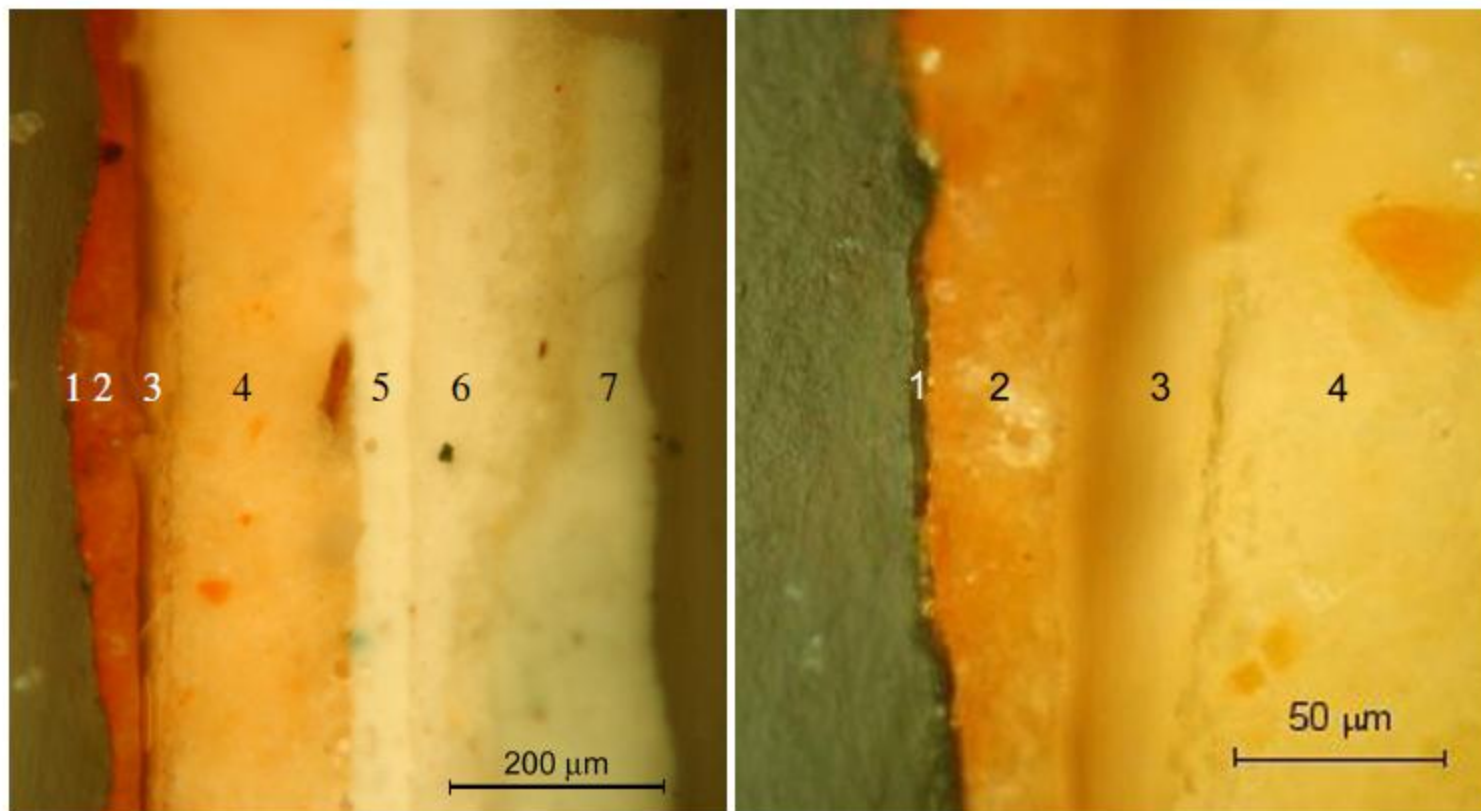
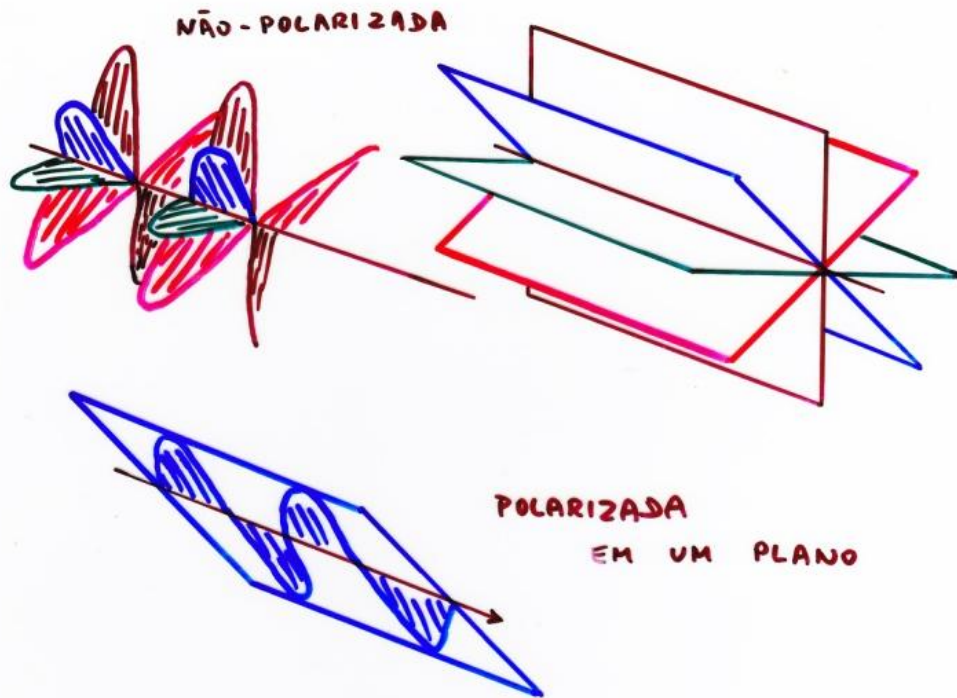


Figure 5: Cross-section of the specimen taken from a mural painting of the Baroque period with a thin layer of gold on the surface (layer 1).

X-RAYS IN ART AND ARCHAEOLOGY – AN OVERVIEW

M. Schreiner, B. Frühmann, D. Jembrih-Simbürger, R. Linke

LUZ POLARIZADA



ESTRUTURAS CÚBICAS
ESTRUTURAS AMORFAS > NENHUM EFEITO
SOBRE FEIXE
POLARIZADO

ESTRUTURAS NÃO-CÚBICAS → FEIXE ^{POLARIZADO NO PLANO} SAI
POLARIZADO
ELIPTICAMENTE

↓
BIRREFRINGENTES

USO DE LUZ POLARIZADA

a) MATERIAIS TRANSPARENTES

- IDENTIFICAR MATERIAIS (propriedades tabuladas)
- DISTINGUIR MATERIAIS ISOTRÓPICOS E ANISOTRÓPICOS
- INTRODUZ CONTRASTE ENTRE GRÃOS
- TENSÕES INTERNAS

b) MATERIAIS OPACOS

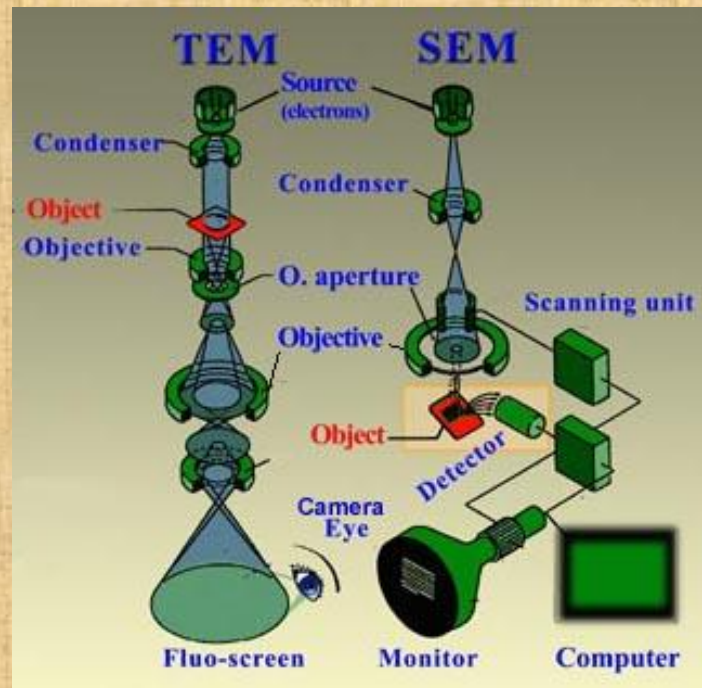
- CONTRASTE ENTRE GRÃOS
- MACLAS, ORIENTAÇÃO PREFERENCIAL
- DISTINGUIR MATERIAIS ISOTRÓPICOS E ANISOTRÓPICOS
- ORIENTAÇÃO DE DOMÍNIOS MAGNÉTICOS EM MATERIAIS FERROMAGNÉTICOS



Neiva – Sm-Fe-Ti

Microscopia eletrônica

MEV e MET



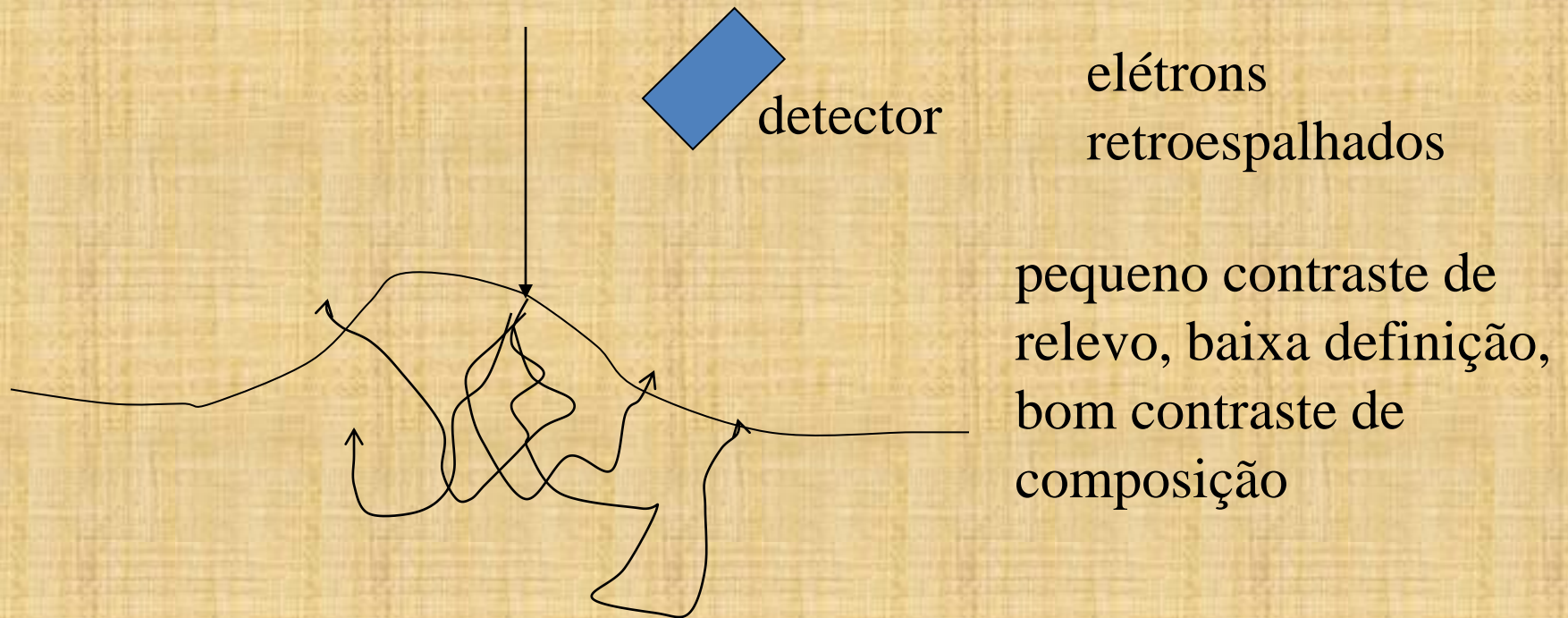
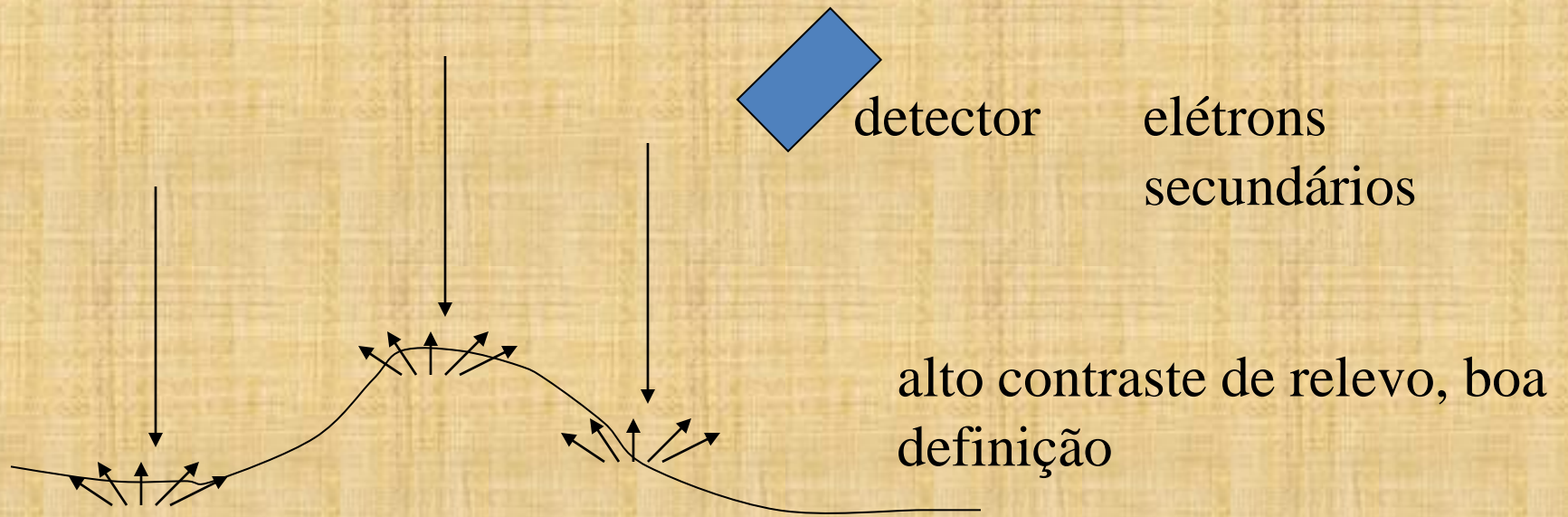
Pr-Fe-B

MEV



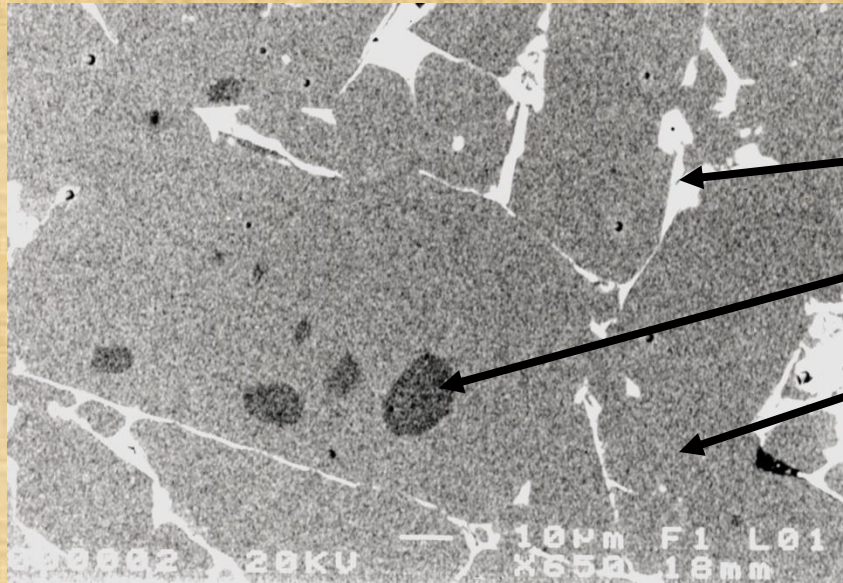
©Eric B. Workman





microscópio eletrônico de varredura

Pr-Fe-B



AED (raios X):

[retornar](#)

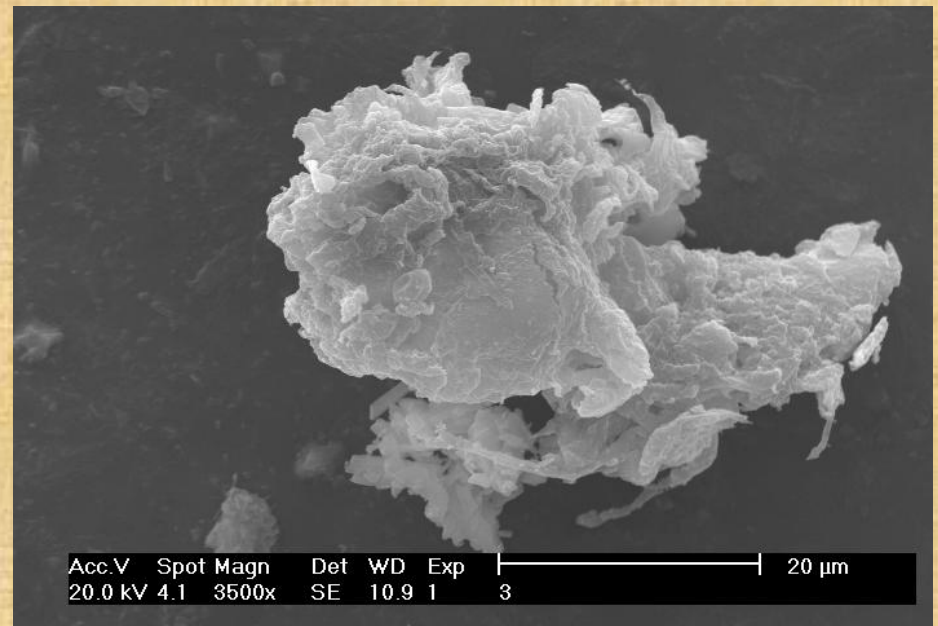
- 97,2%Pr 2,8%Fe
- fase com alto B
- 86,7%Fe 13,3%Pr (e B)

elétrons retroespalhados

Claro – elementos pesados
Escuro – elementos leves

depósito de óxido de
Ce sobre Al

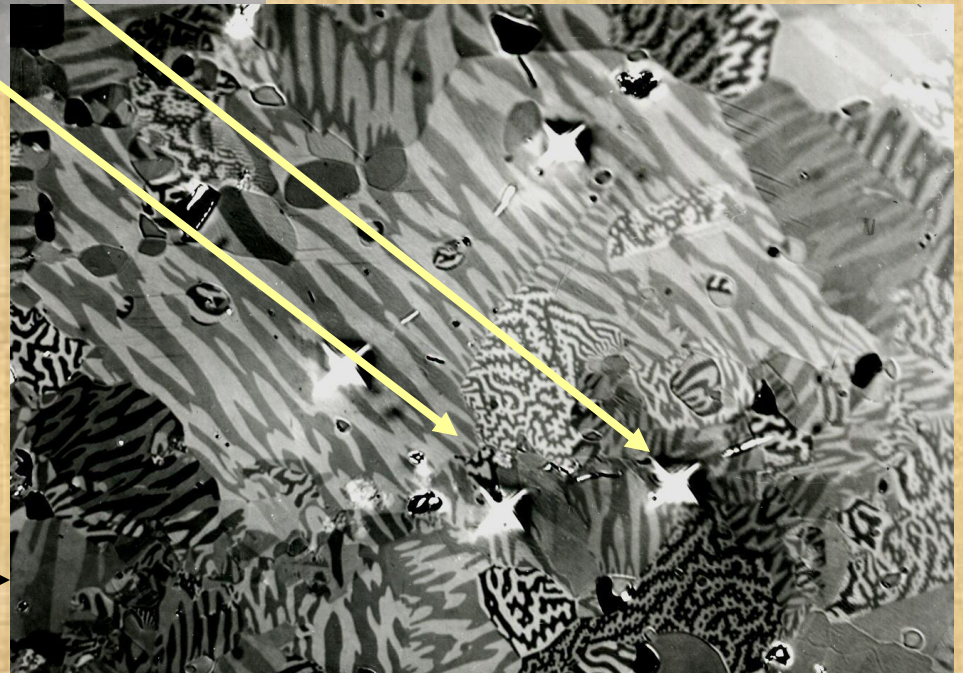
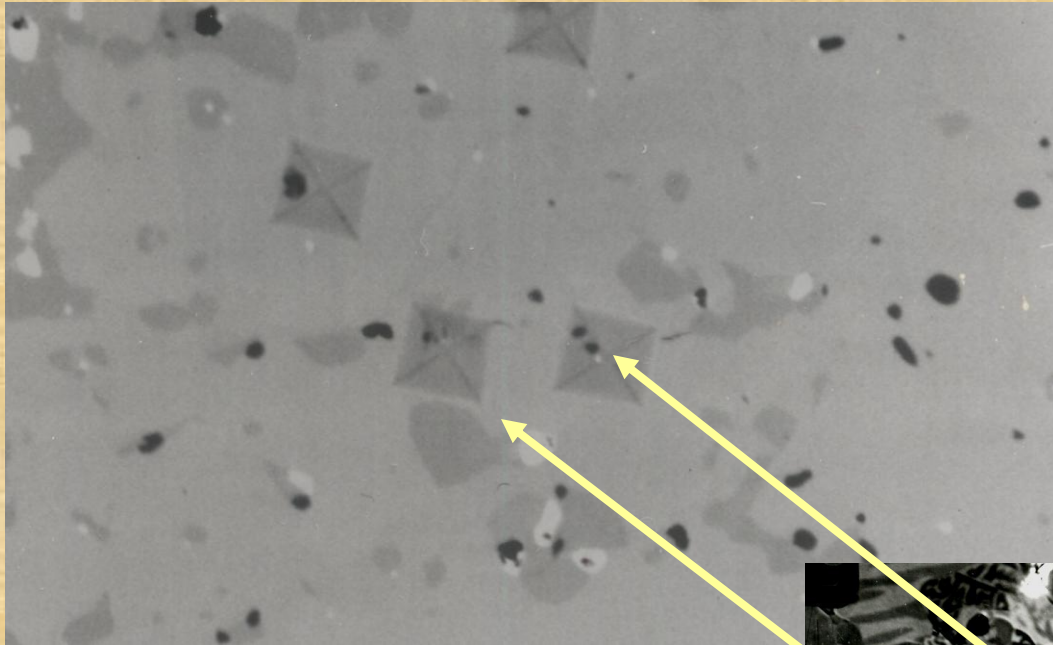
elétrons secundários



Sm-Ti-Fe

MEV com EDXA:
composição de fases

marcas de
microdureza para
localizar a região



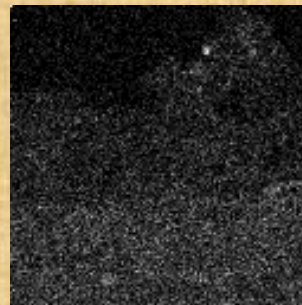
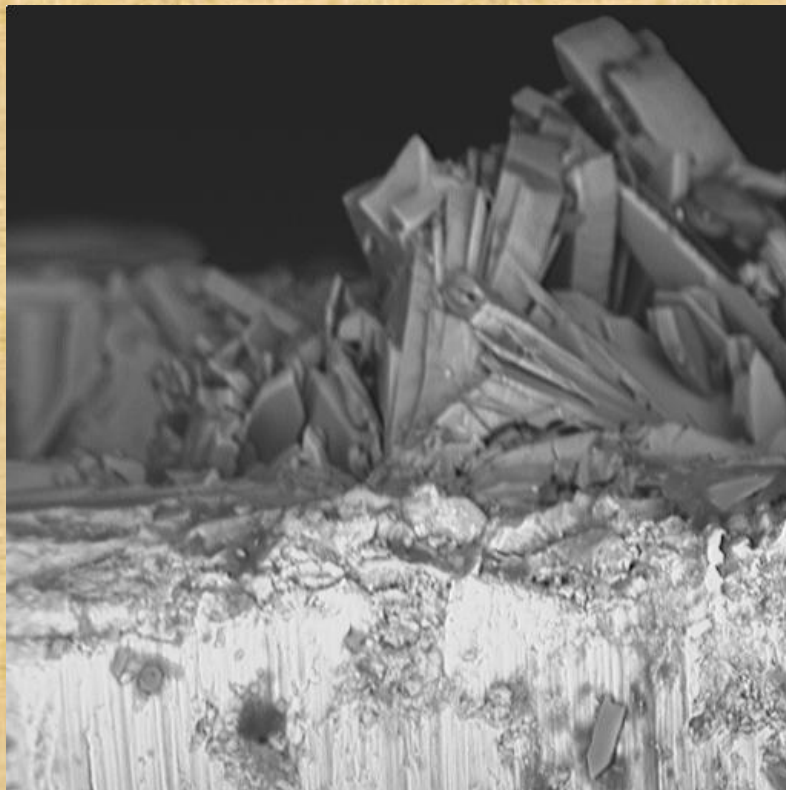
microscopia óptica com
luz polarizada: domínios
magnéticos



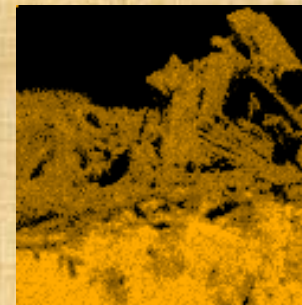
MEV – pátinas artificiais



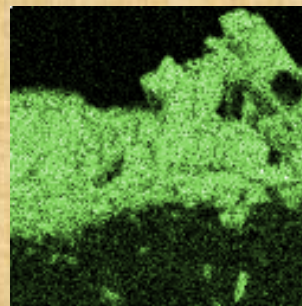
OUTRAS PÁTINAS – mapas de composição (fluorescência de raios X) no MEV



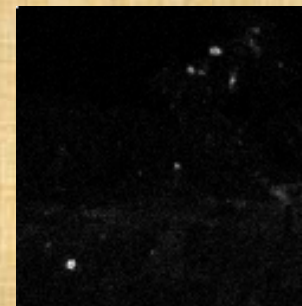
cloro



cobre

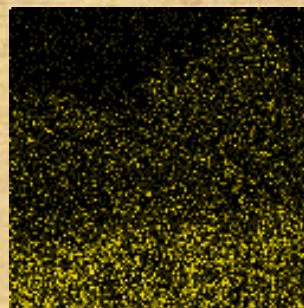


enxofre

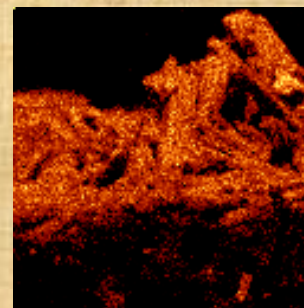


potássio

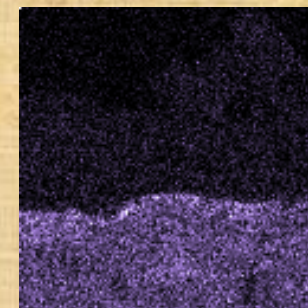
Neiva & Robiolla



zinco



oxigênio



estanho

MEV – o sino

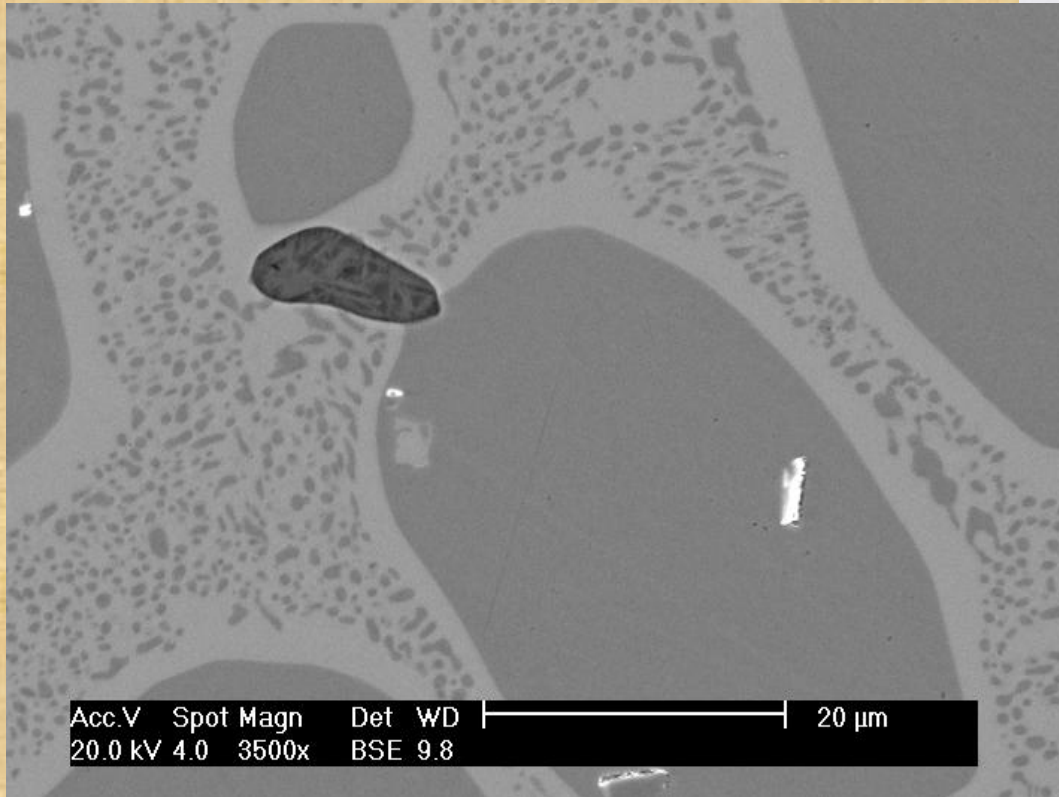
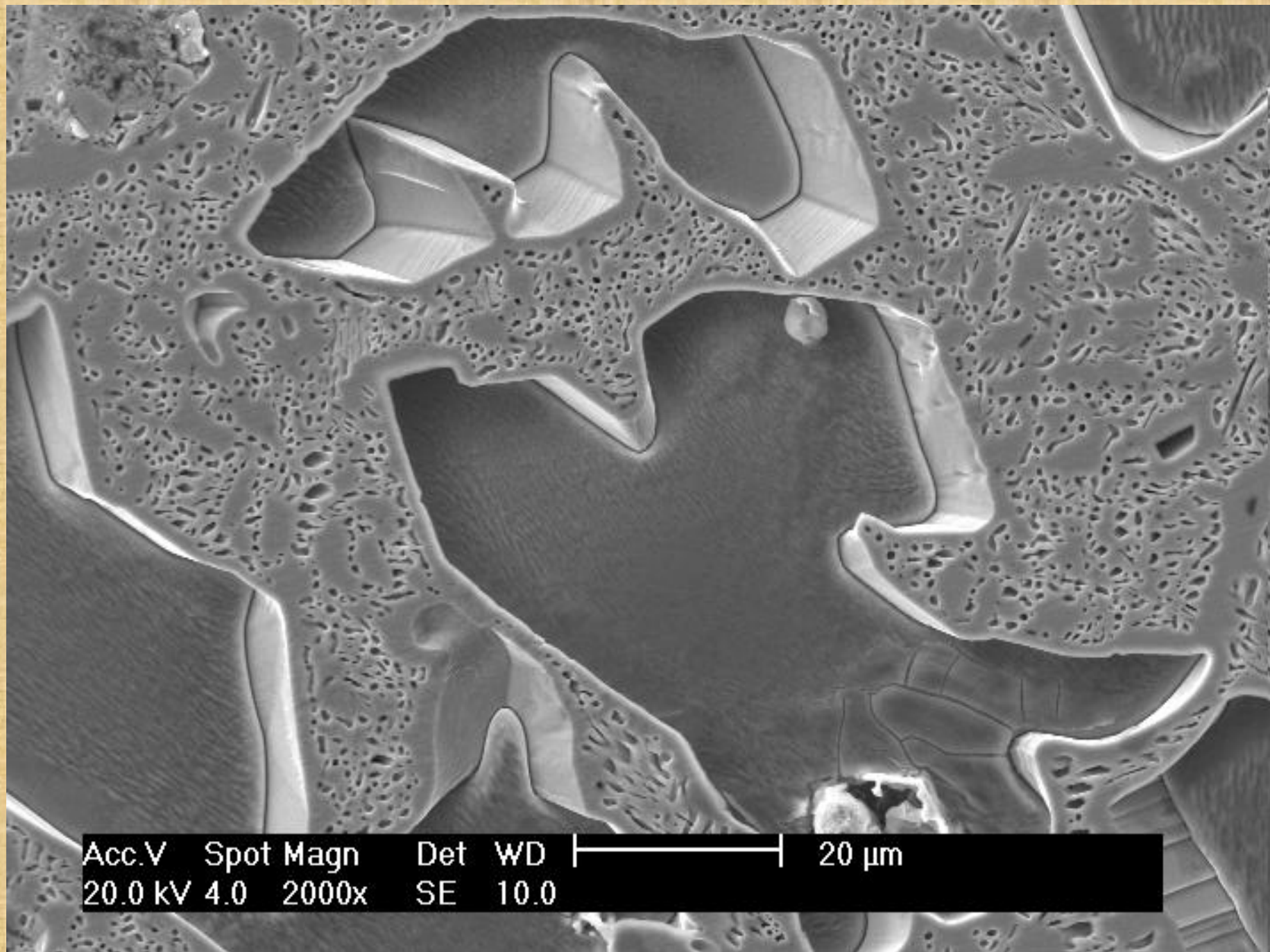
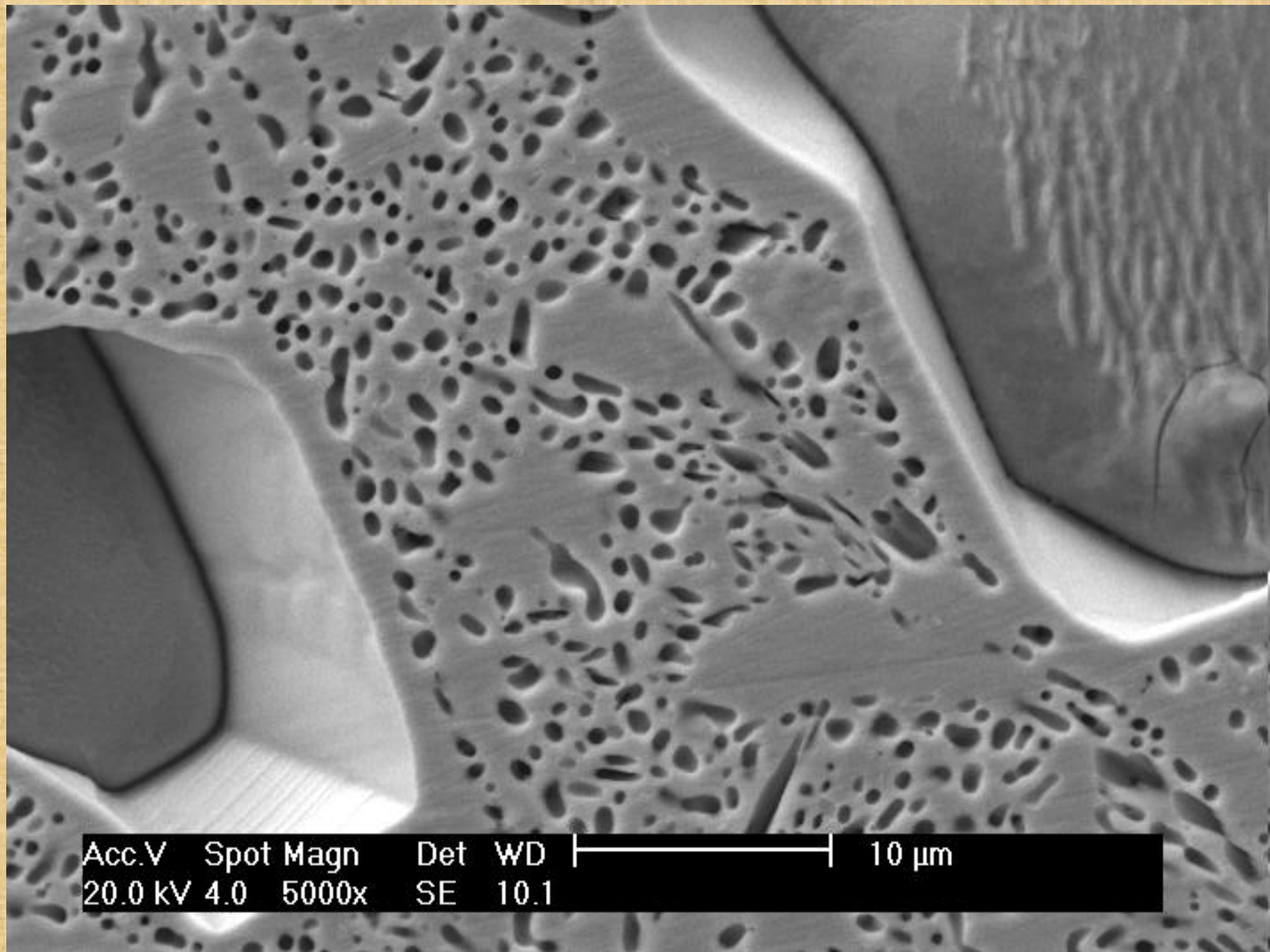


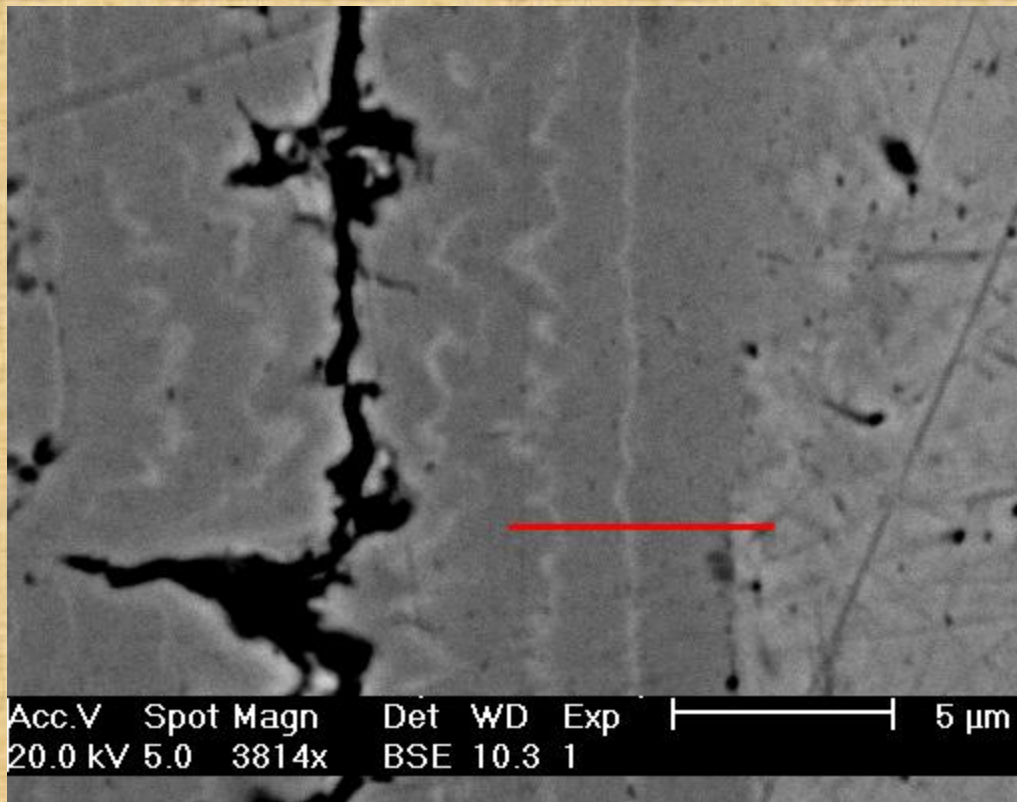
imagem de elétrons
retroespalhados –
contraste por composição
química



Acc.V	Spot	Magn	Det	WD	20 μ m
20.0 kV	4.0	2000x	SE	10.0	

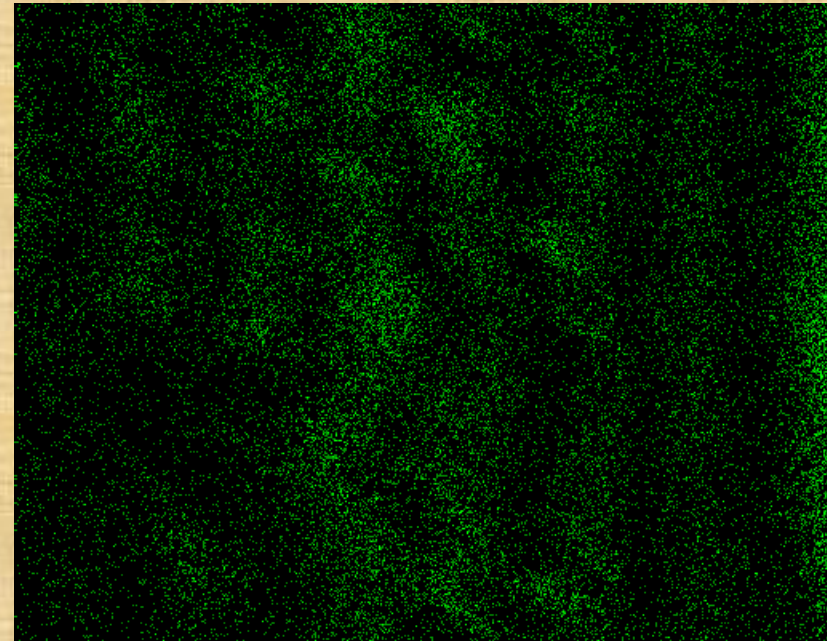


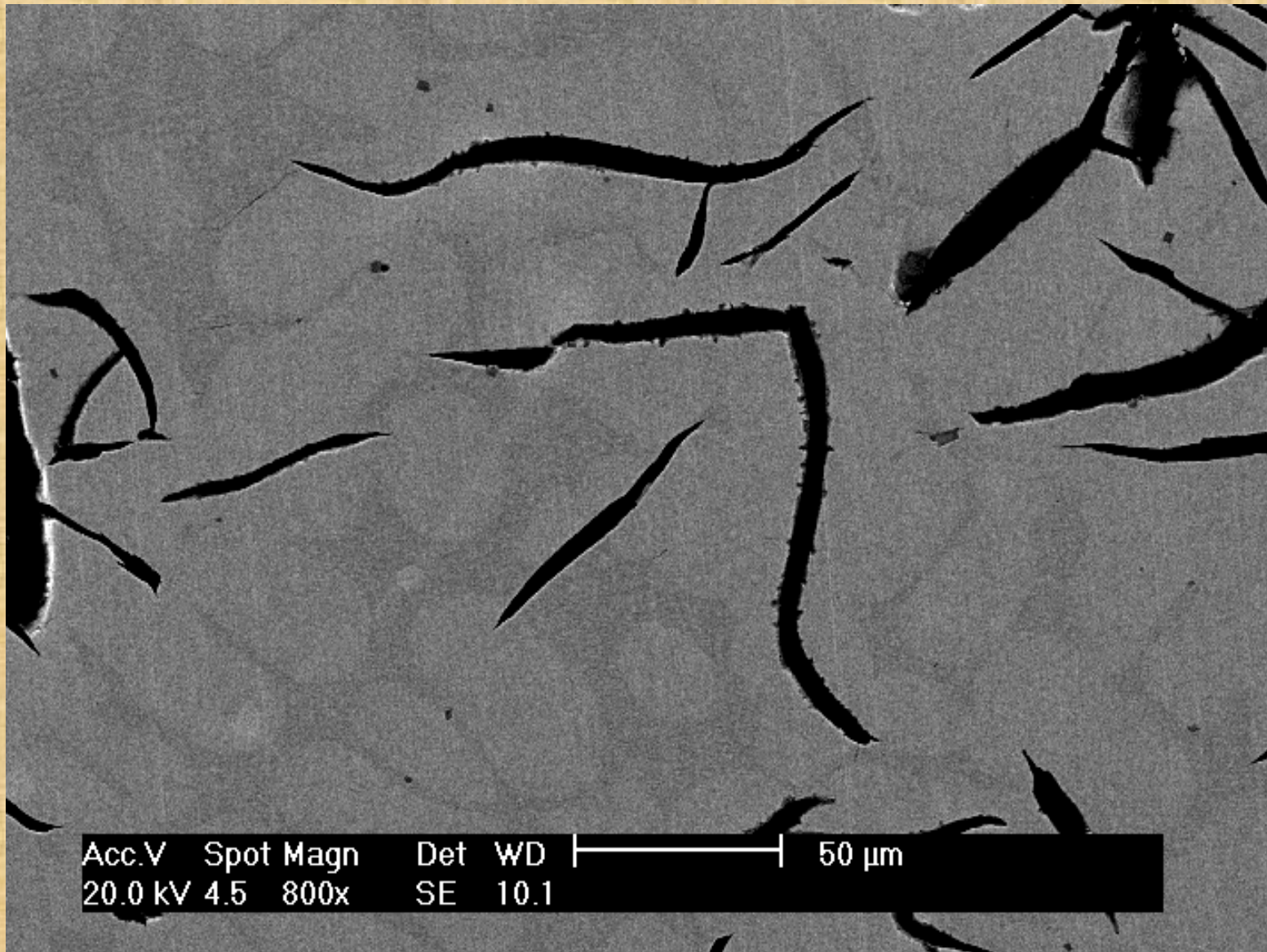
Acc.V Spot Magn Det WD |-----| 10 μm
20.0 kV 4.0 5000x SE 10.1



Claro – elementos pesados
Escuro – elementos leves

Neiva – microcamadas de
cobalto e cobre sobre cobre

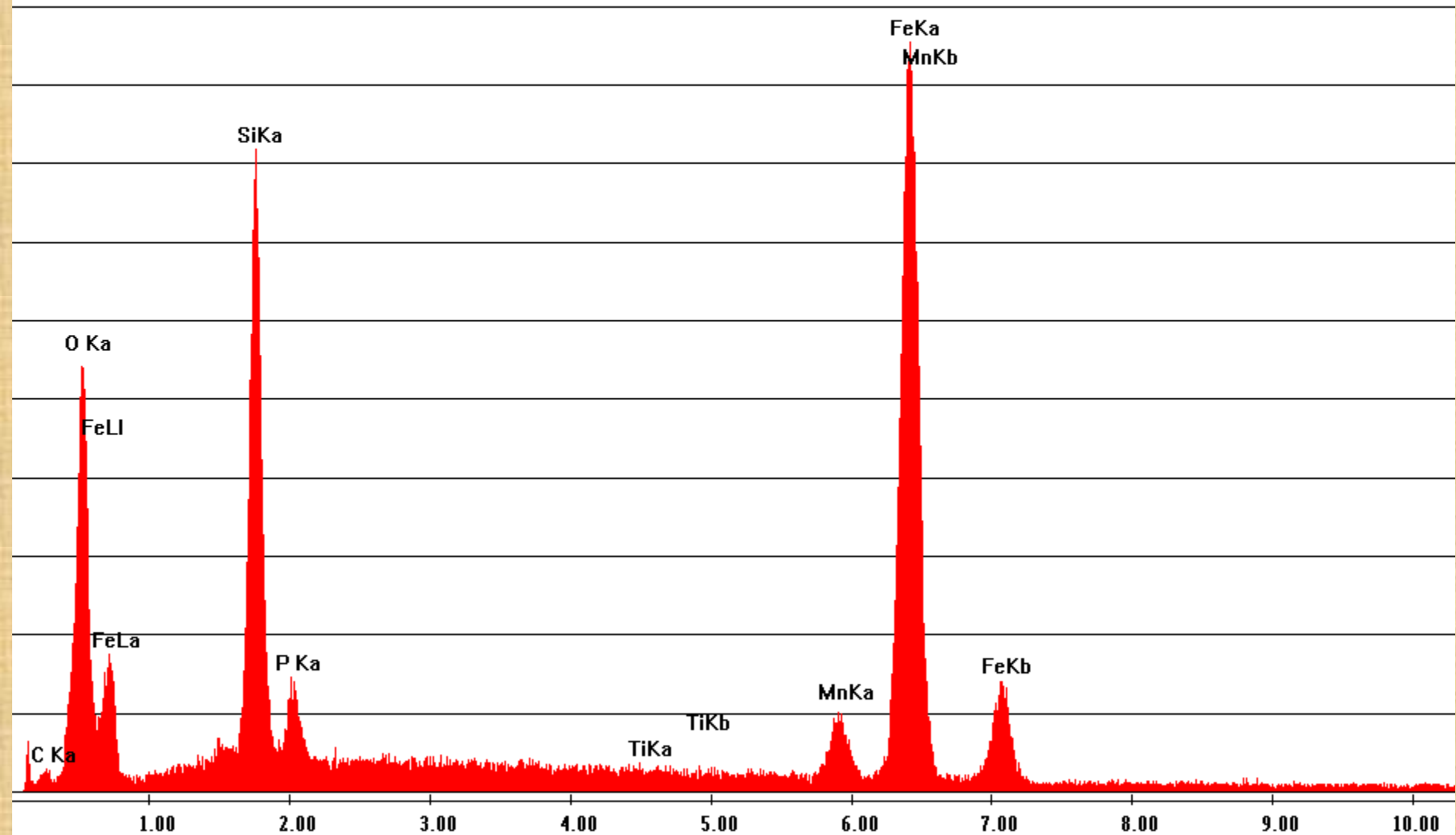




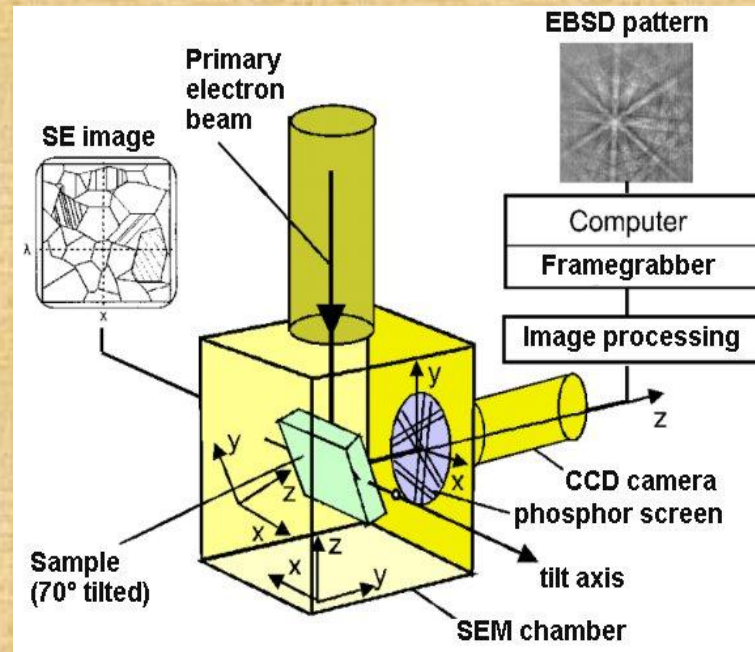
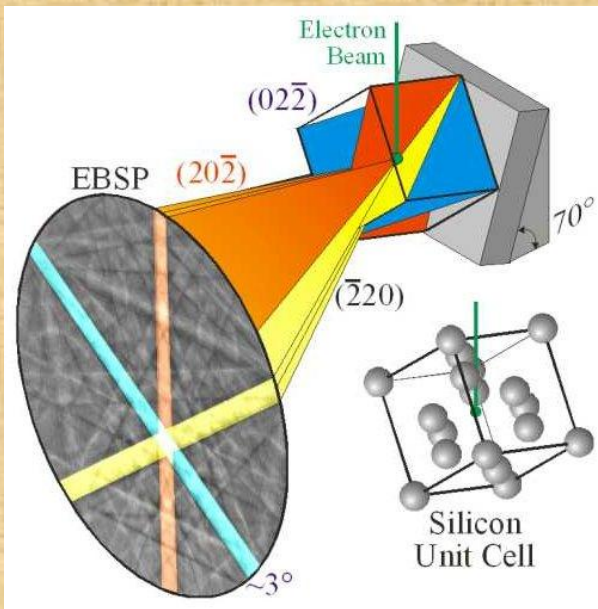
Claro – elementos pesados
Escuro – elementos leves

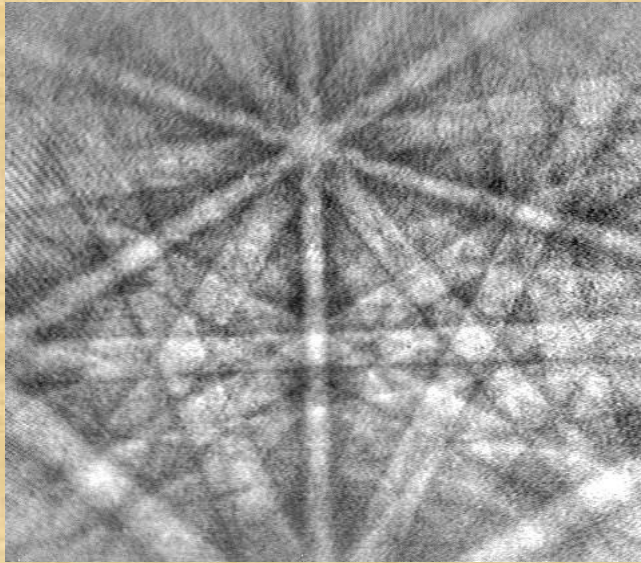
Neiva – Veios de grafita em ferro fundido

Label A: inclusao disco



EBSD (Electron Backscatter Diffraction)





Linhas de Kikuchi

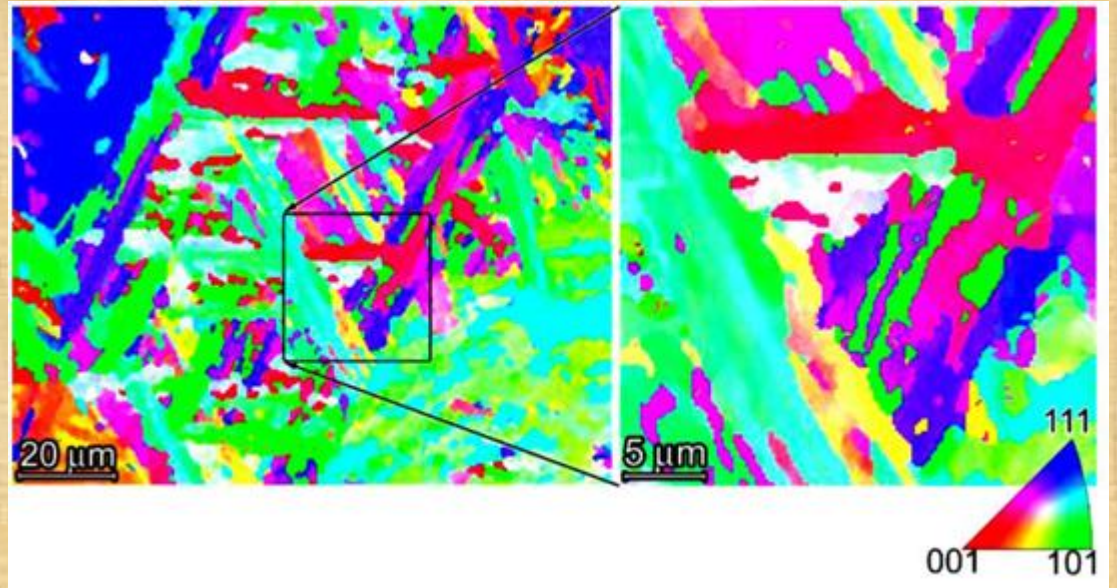
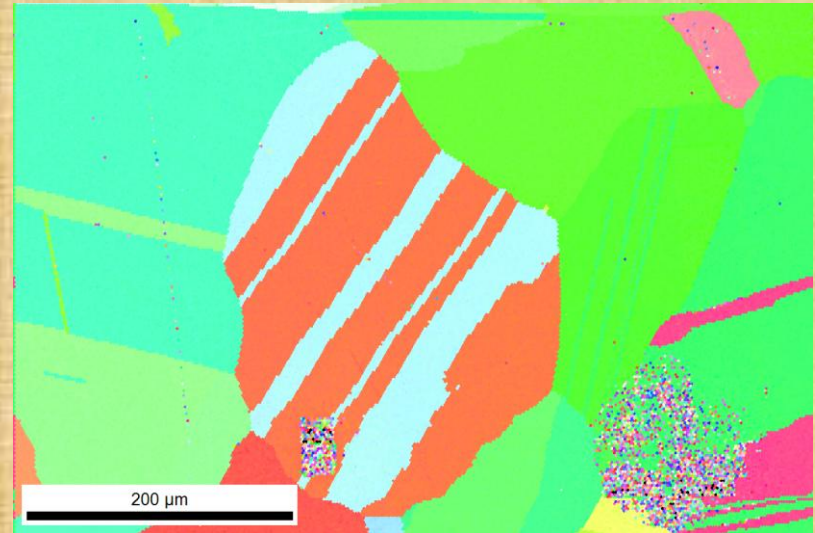
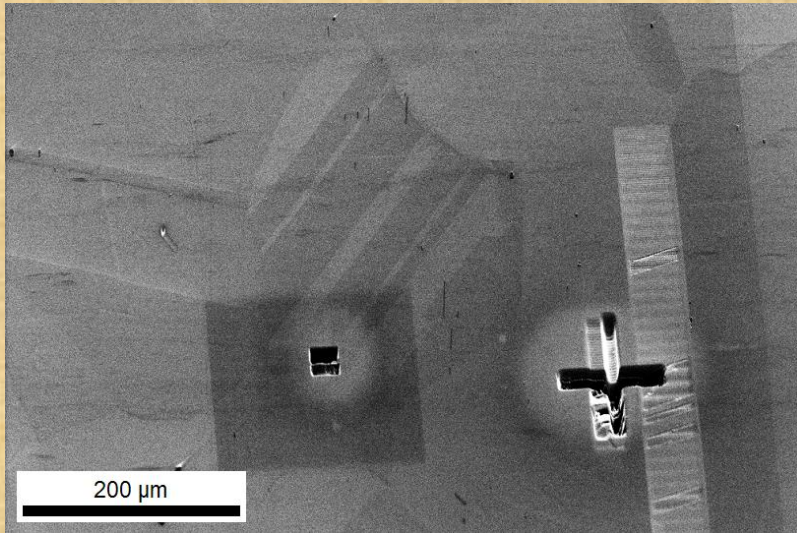
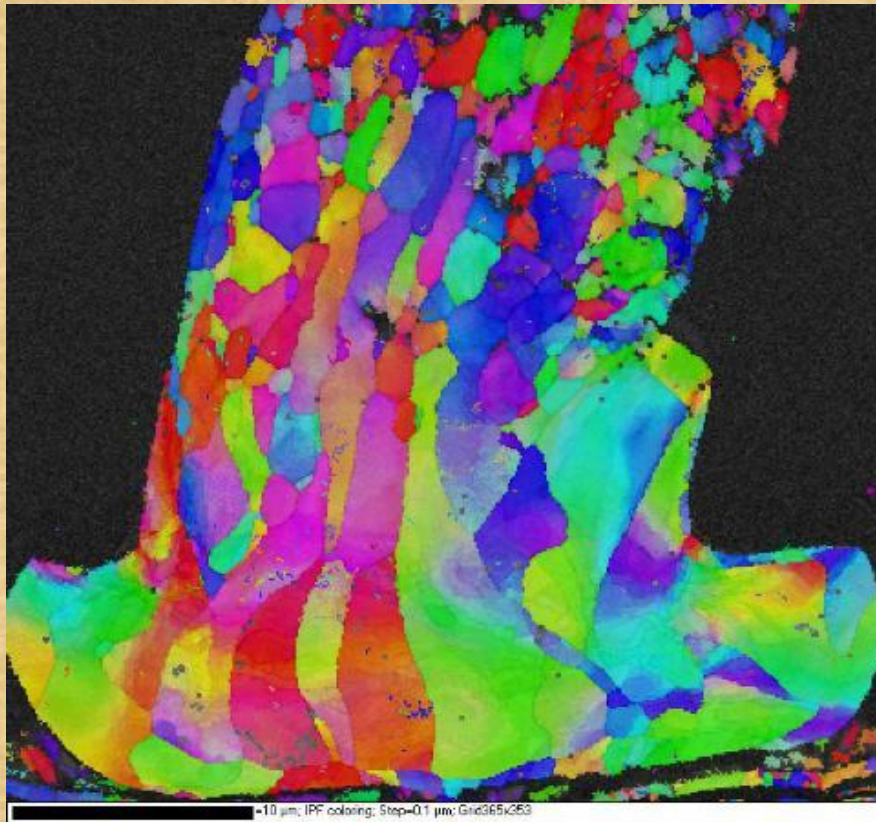


Figura de pólo inversa de estrutura martensídica em um aço
<http://www.Inls.br/Inls/cgi/cgilua.exe/sys/start.htm?sid=507>



http://images.google.com.br/imgres?imgurl=http://web.mit.edu/yildizgroup/research3_pic2.bmp&imgrefurl=http://web.mit.edu/yildizgroup/researchmetal1.htm&usg=__85sIfIGsd6ApiT5PQSxW87I_cxk=&h=850&w=1280&sz=3188&hl=pt-BR&start=56&um=1&tbnid=1qXSVbxoXeDkeM:&tbnh=100&tbnw=150&prev=/images%3F



Wirebond – EBSD

<http://www.jeolusa.com/SERVICESTUPPORT/ApplicationsResources/SamplePreparation/ImageGallery/tabid/345/AlbumID/744-7/Page/1/Default.aspx>



Characterisation and the diagenetic transformation of nonand
micro-crystalline silica minerals

DAVID R. LEE

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Brownlow Street,
Liverpool L69 3GP, UK (e-mail: d.r.lee@liverpool.ac.uk)

ESEM – Environmental Scanning Electron Microscopy

- ESEM diferencia-se de um SEM convencional pela presença de um gás na câmara da amostra. Portanto, as amostras não são vistas sob vácuo, mas sob um vácuo deteriorado, ou “baixo” vácuo.
- O gás atua como um condutor de cargas elétricas evitando o carregamento da amostra e facilitando a detecção do sinal.

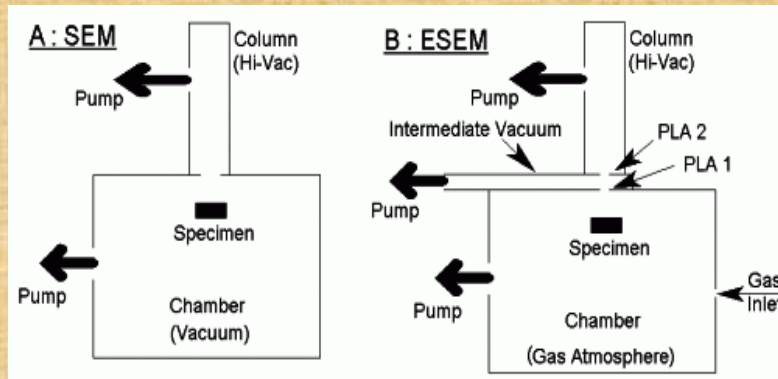
A possibilidade de se obter imagens com a presença de gás na câmara deve-se a duas modificações:

- 1) A coluna é dividida em diferentes zonas de pressão separadas por aberturas limitadoras de pressão.

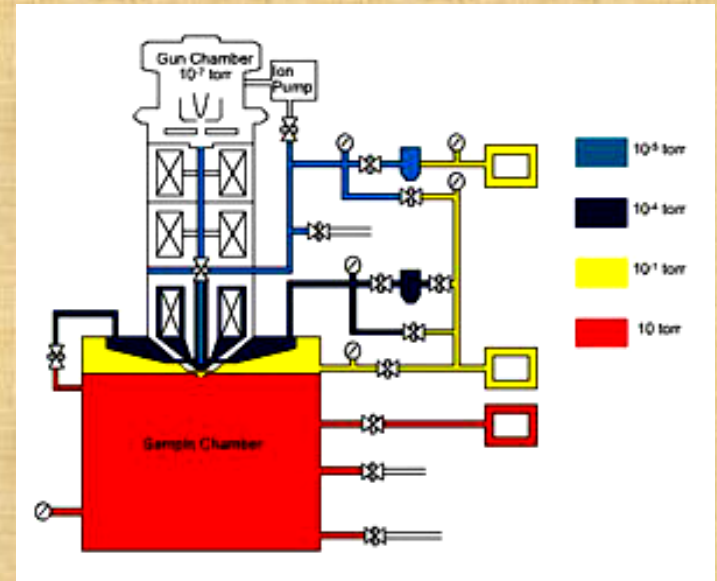
Dados baseados em seminário de Patrícia Kaji Yasumura na disciplina “Princípios de Caracterização de Materiais por Espectroscopia, Difração e Imagem”

ESEM

- Vantagem: não é necessária nenhuma preparação da amostra que possa causar alguma destruição da superfície e as amostras podem ser arquivadas mais facilmente.

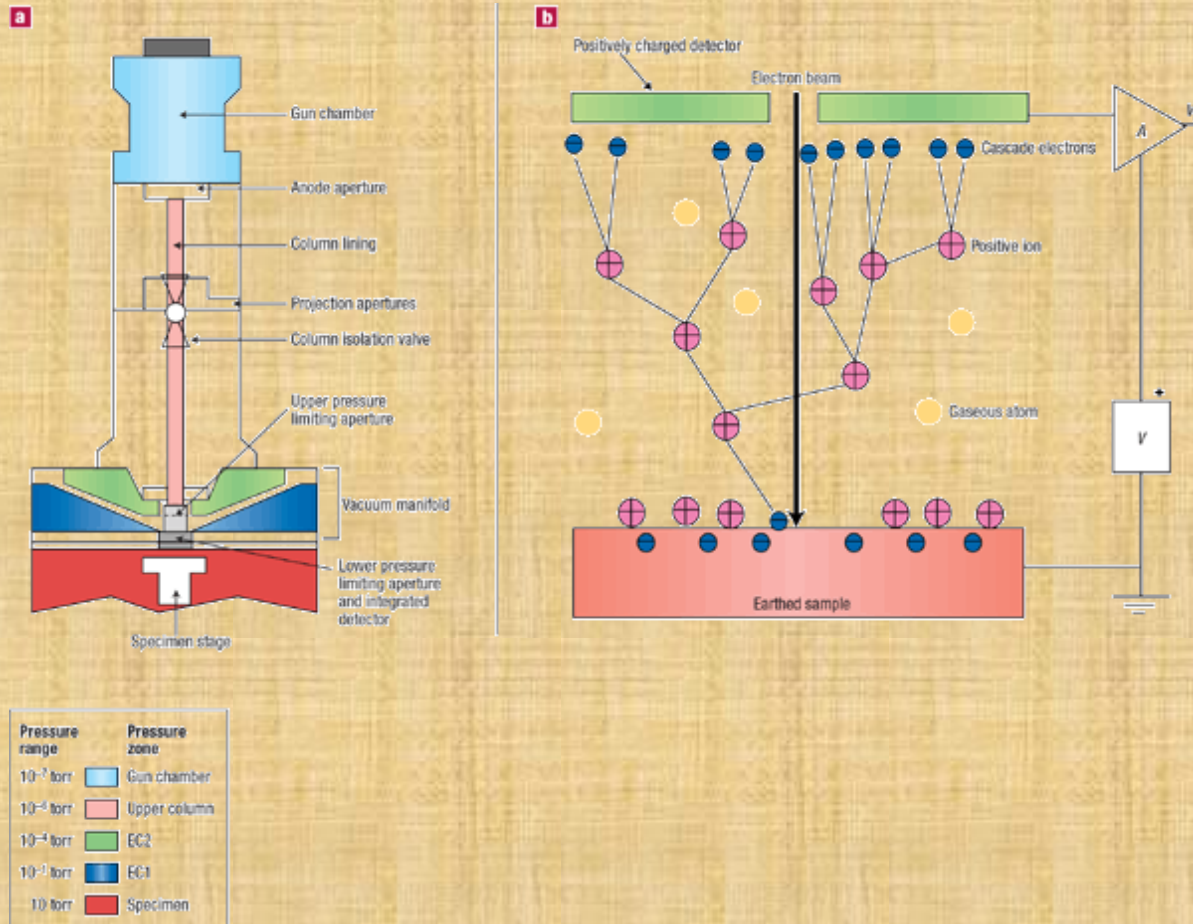


<http://www.shu.ac.uk/research/meri/e-news/issue4/technique.html>



<http://www.phy.cam.ac.uk/research/emsuite/Pictures/ESEM%20Schematic.jpg>

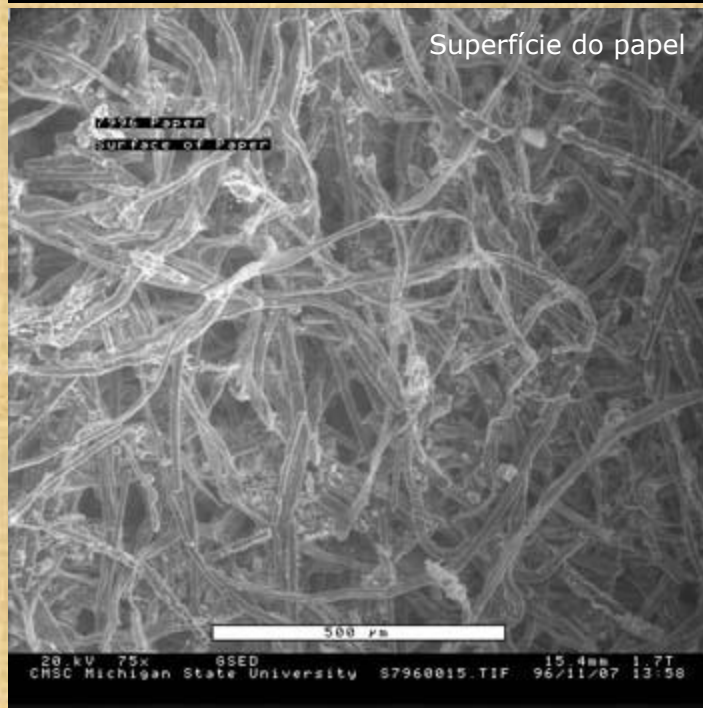
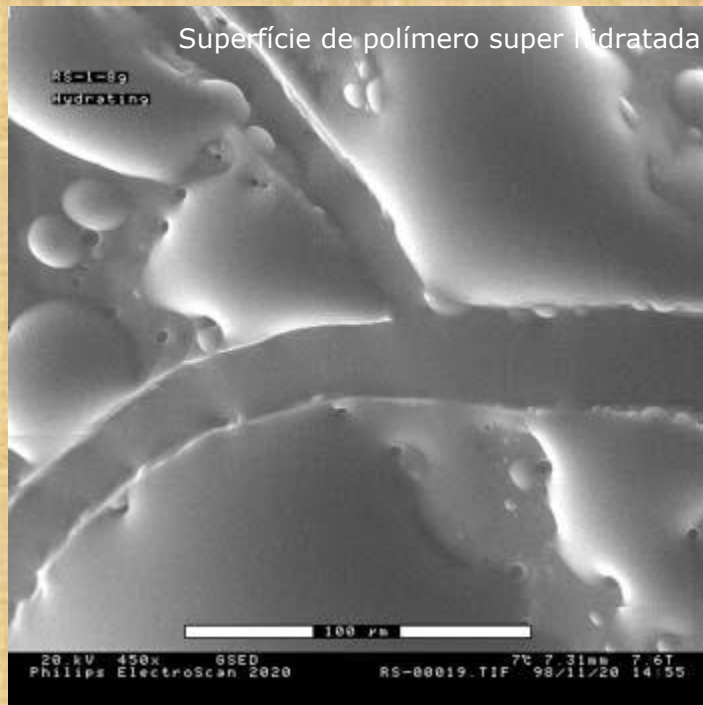
ESEM – Princípio de funcionamento



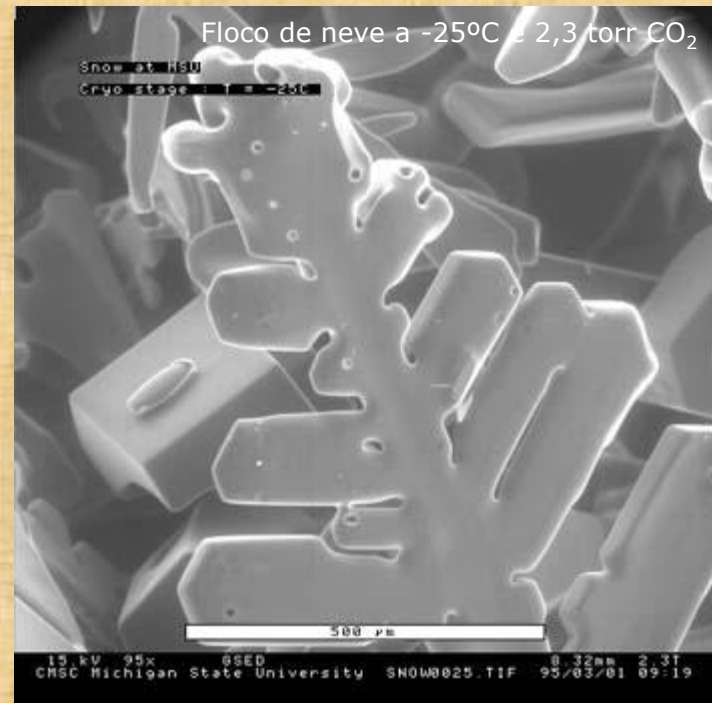
XL 30 ESEM FEG

Scanning Electron Microscope





Exemplos de micrografias ESEM



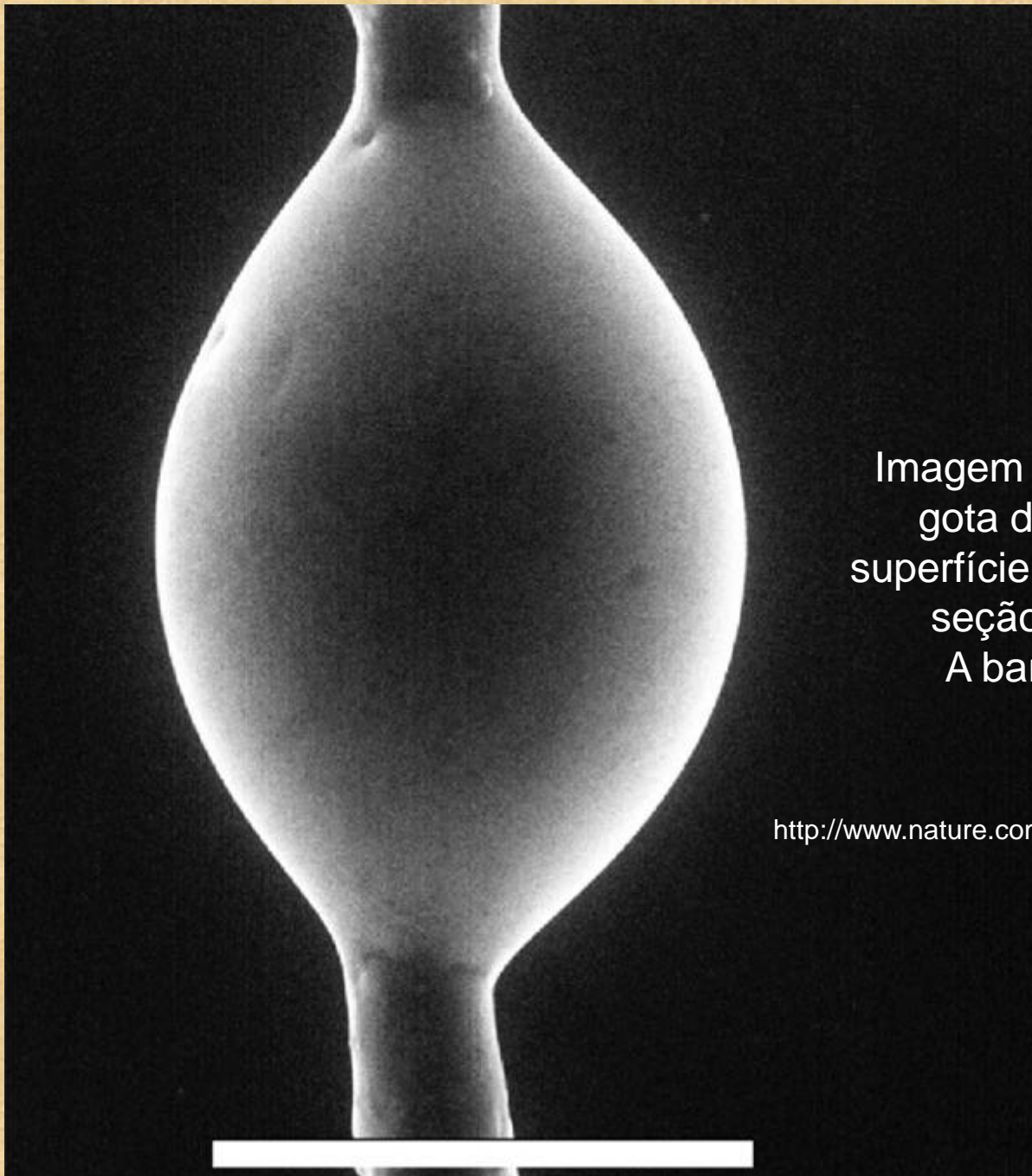


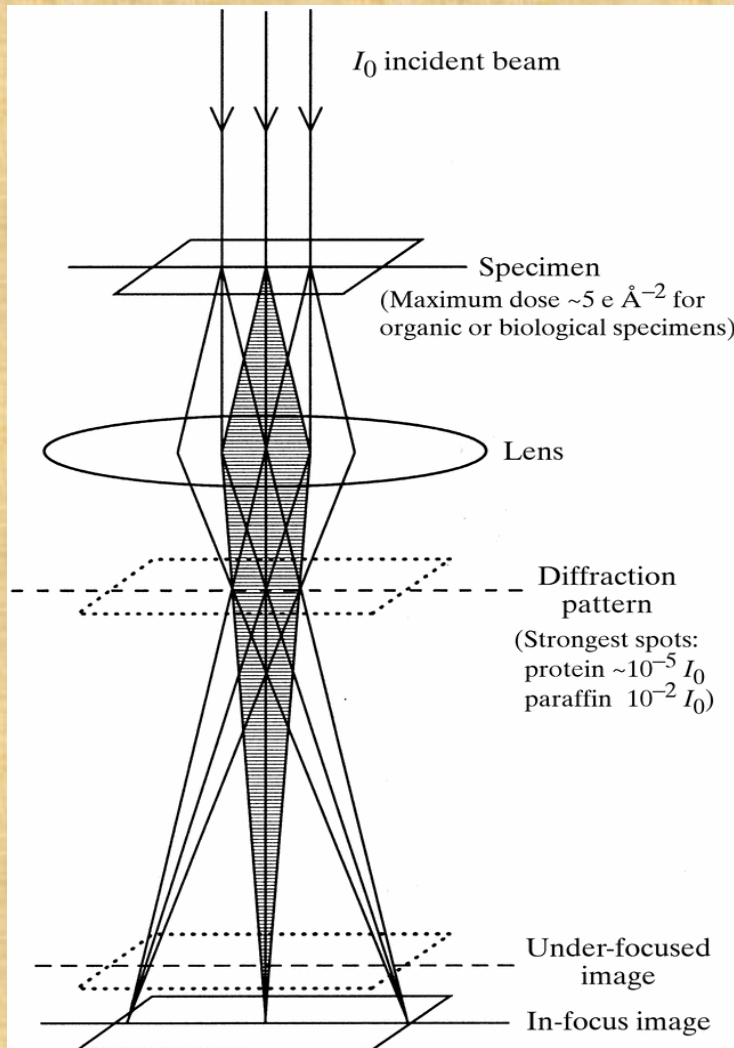
Imagem obtida por ESEM de uma gota de água condensada na superfície de uma fibra celulósica de seção transversal cilíndrica. A barra representa 50 μm .

http://www.nature.com/nmat/journal/v2/n8/fig_tab/nmat898_f5.html

Bibliografia de ESEM

- Bogner, A., Jouneau, P.-H., Thollet, G., Basset, D., Gauthier, C., A history of scanning electron microscopy developments: Towards “wet-STEM” imaging, **Micron** 38 (2007) pp. 390-401.
- Danilatos, G.D., Review and outline of environmental SEM at present, **Journal of Microscopy**, vol. 162, Pt 3, Junho 1991, pp. 391-402.
- Danilatos, G.D., Introduction to the ESEM instrument, **Microscopy Research and Technique** 25 (1993), pp. 354-361
- Forsberg, P., Lepoutre, P., Environmental Scanning electron microscope examination of paper in high moisture environment: surface structural changes and electron beam damage, **Scanning Microscopy**, vol. 8, No.1, 1994, pp. 31-34
- Prack, E.R., An introduction to process visualization capabilities and considerations in the environmental scanning electron microscope (ESEM), **Microscopy Research and Technique** 25 (1993), pp. 487-492

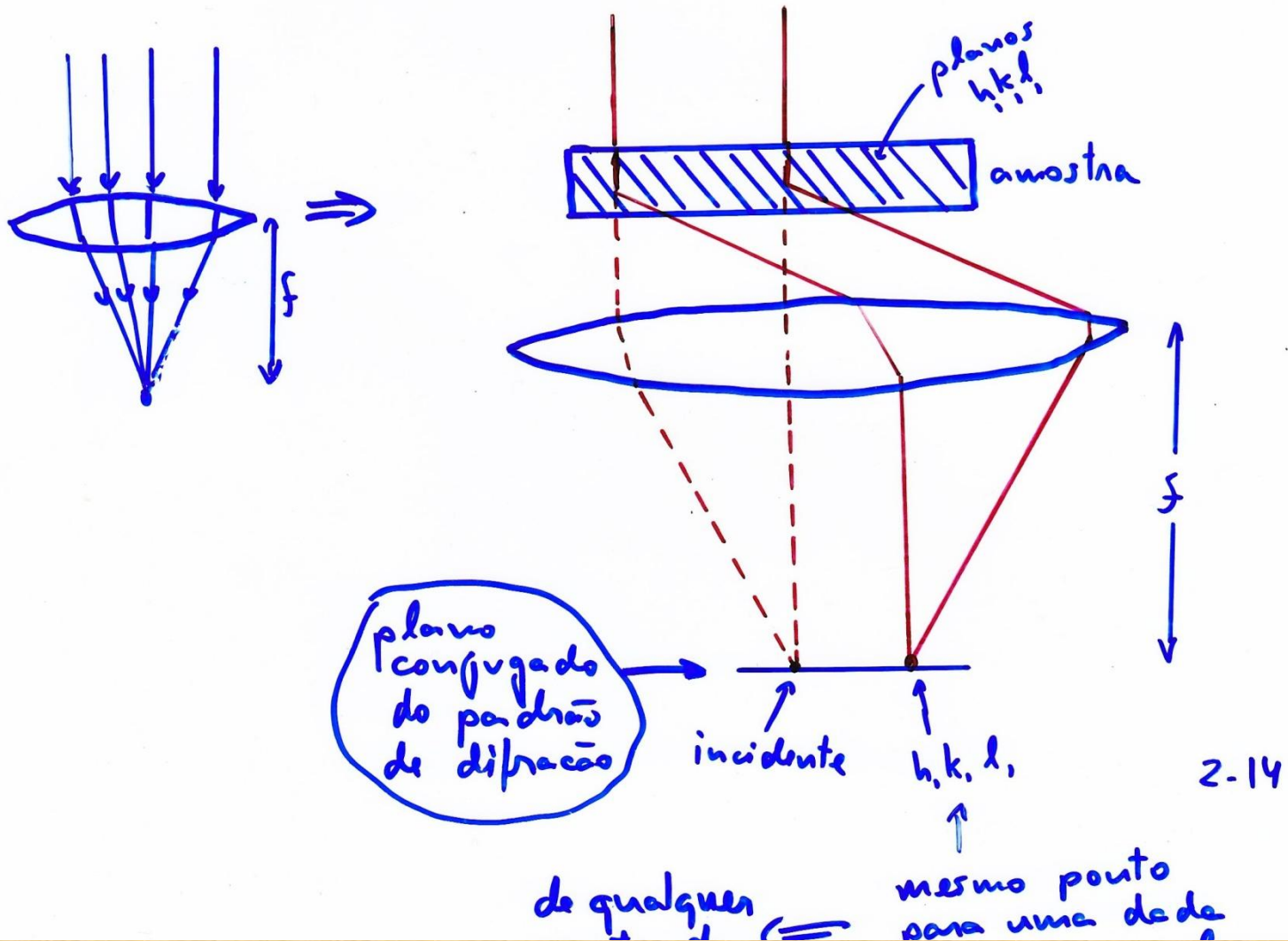
Microscopia Eletrônica de Transmissão - TEM



* TEM

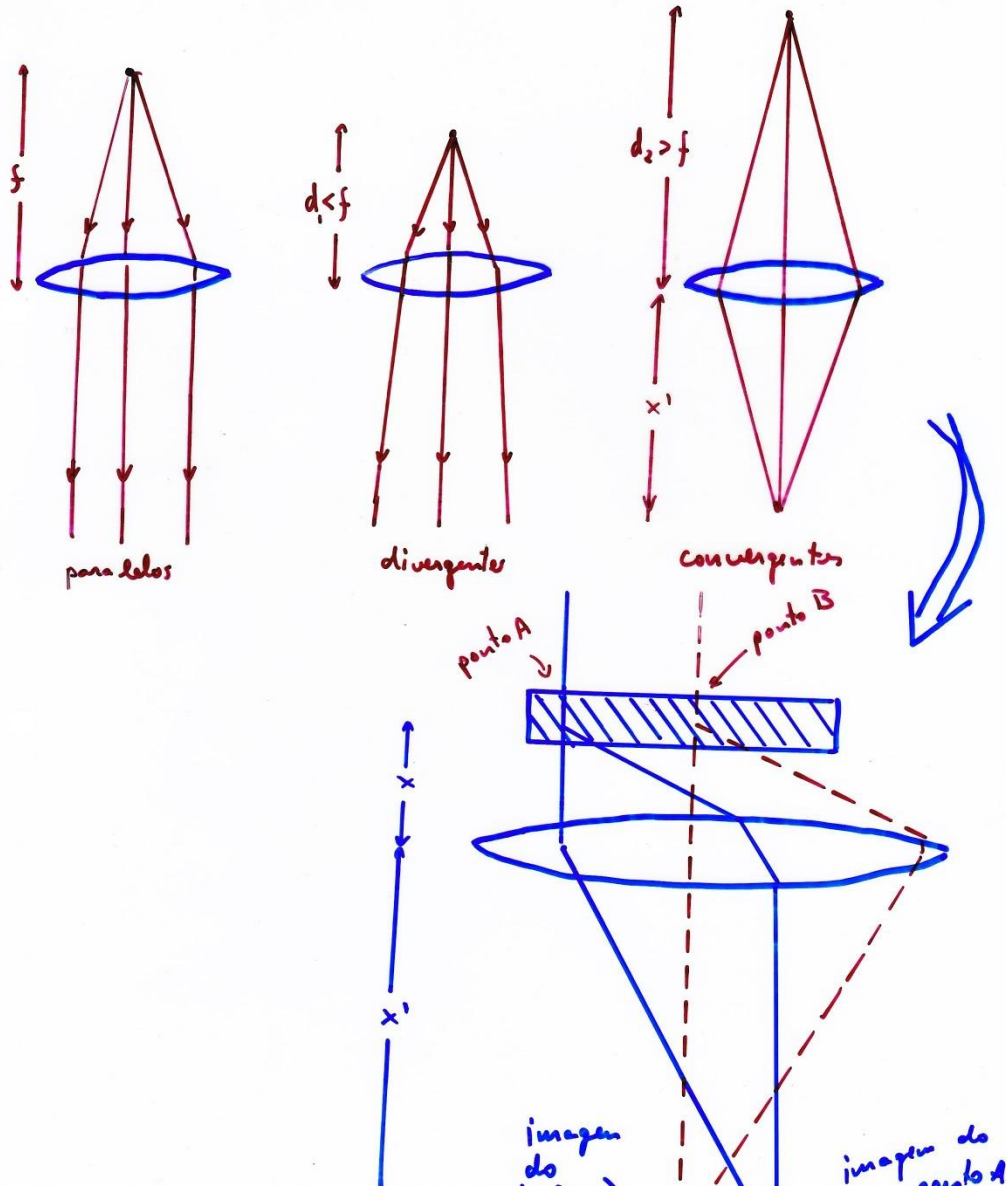


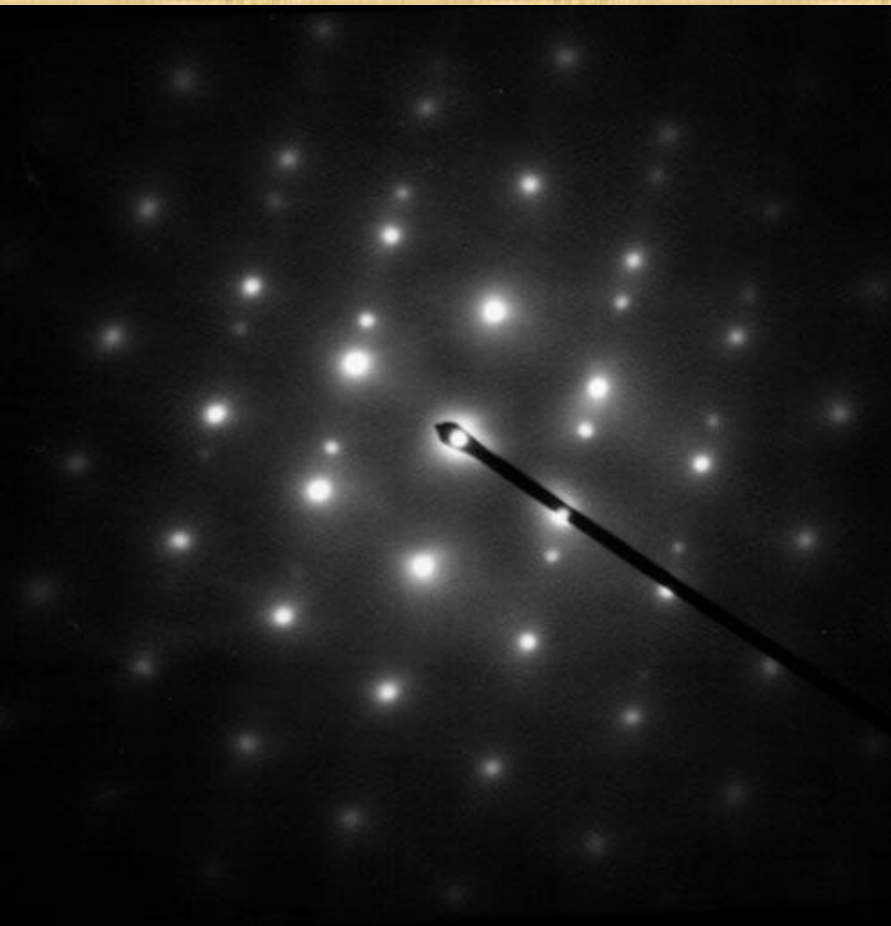
FIGURA DE DIFRAÇÃO



FORMAÇÃO DE IMAGEM

Agora interessa o que ocorre a partir de cada ponto da amostra.

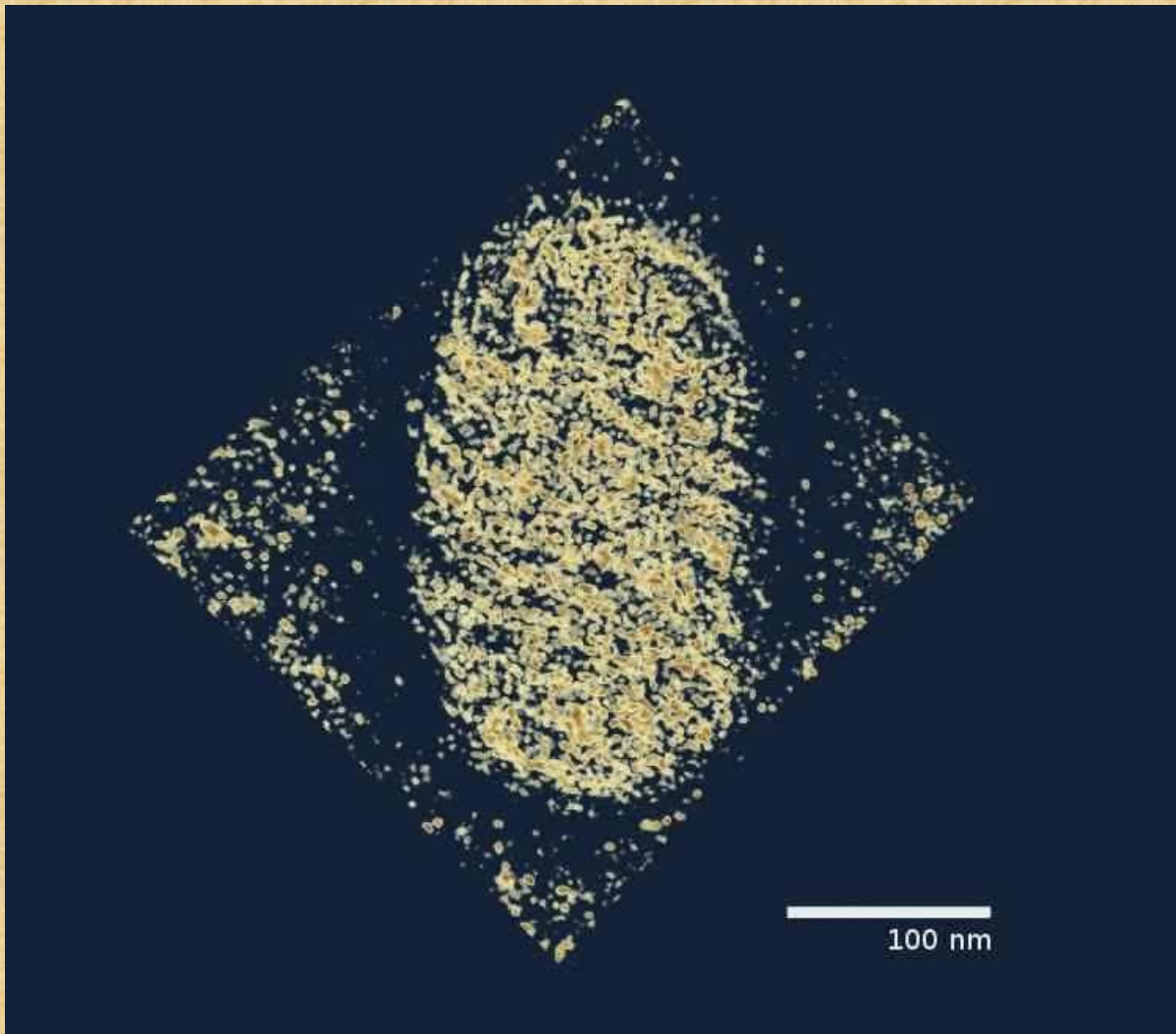




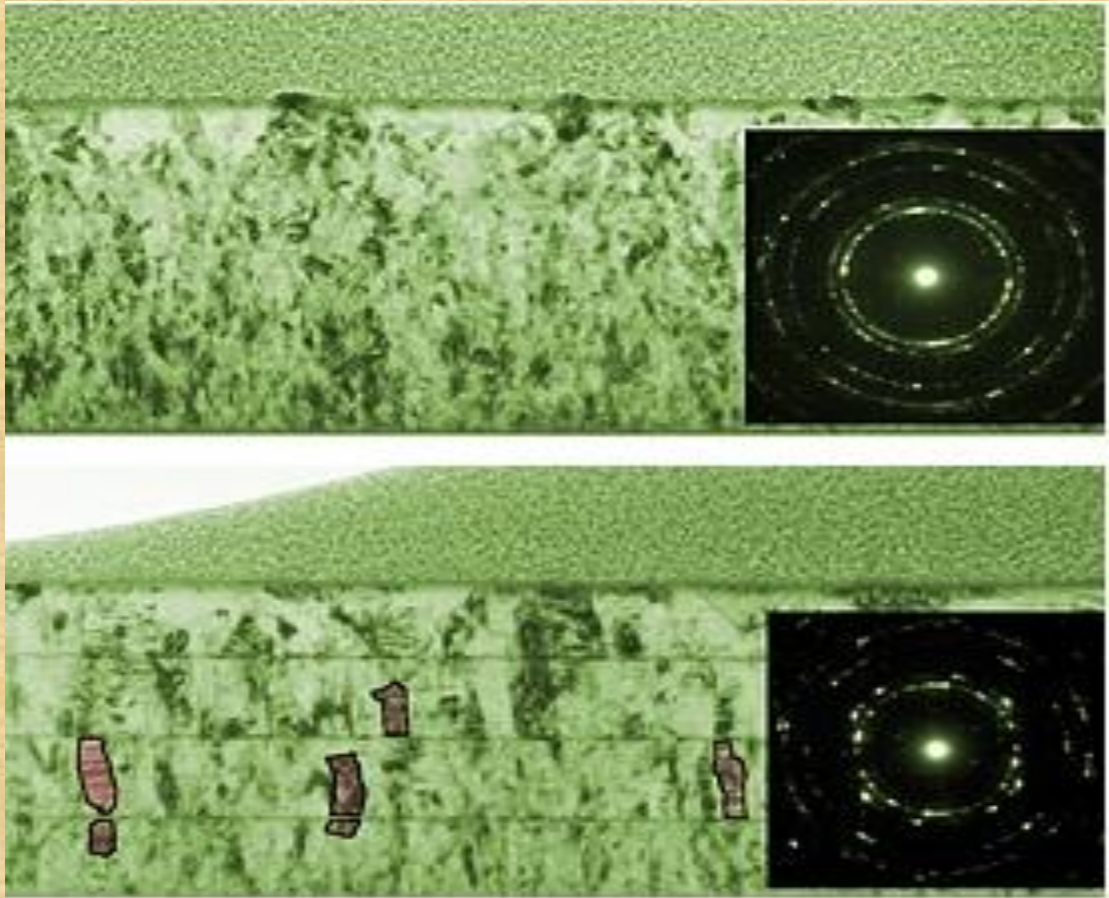
Zone axis diffraction pattern of twinned austenite in steel
wiki



TEM Micrograph of Dislocations 1 (precipitate and dislocations in austenitic stainless steel)
Photomicrograph by Wikityke



A three dimensional TEM image of a parapoxavirus: multiple views of the same specimen can be obtained by rotating the angle of the sample along an axis perpendicular to the beam
wiki



Bonevich, NIST

Sections of a continuous 400-nanometer-thick magnetic film of a nickel-iron-copper-molybdenum alloy (top) and a film of the same alloy layered with silver every 100 nanometers (bottom).

By relieving strain in the film, the silver layers promote the growth of notably larger crystal grains in the layered material as compared to the monolithic film (several are highlighted for emphasis)

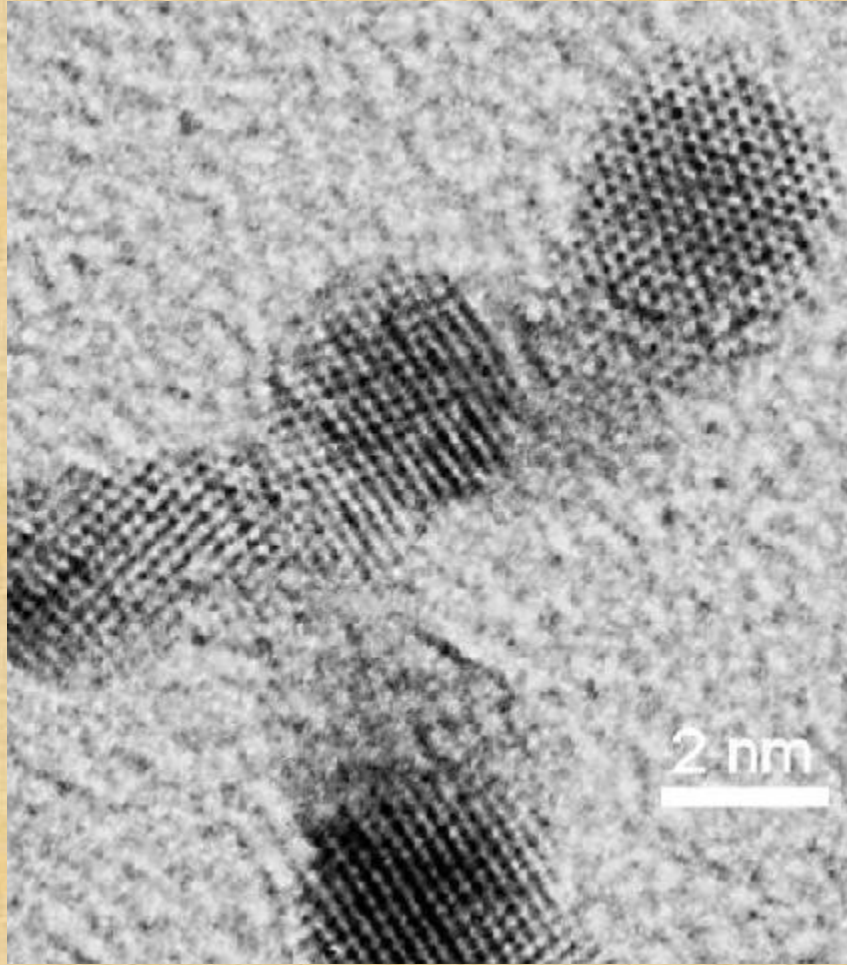
HRTEM

Resolution of the HRTEM is limited by [spherical](#) and [chromatic aberration](#), but a new generation of aberration correctors has been able to overcome spherical aberration.¹

Software correction of spherical aberration has allowed the production of images with sufficient resolution to show carbon atoms in diamond separated by only 0.89 [Ångströms](#) and atoms in silicon at 0.78 Ångströms (78 pm) at magnifications of 50 million times.

wiki

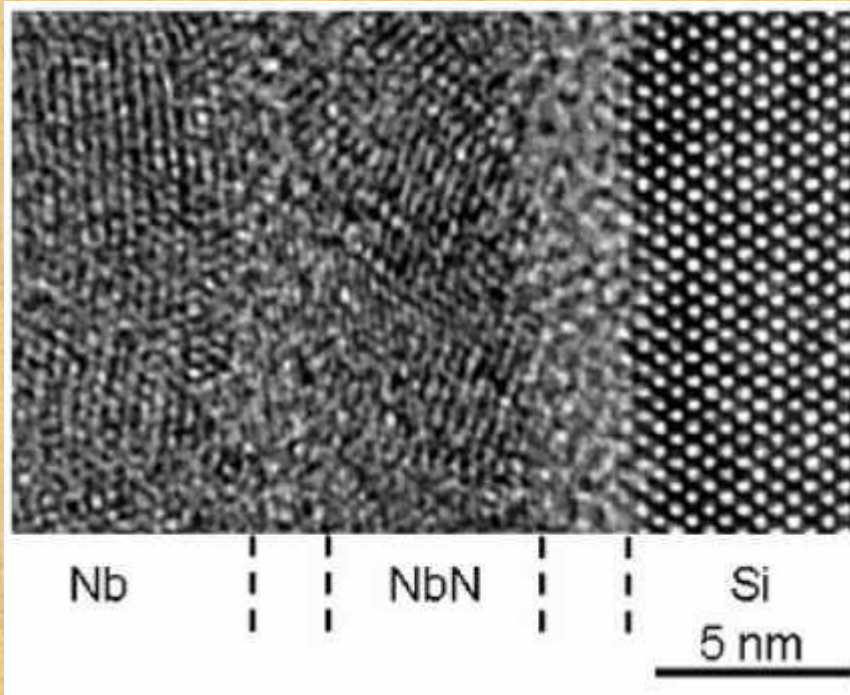
HRTEM



atomic planes within
nanoparticles of metal

CfAM

HRTEM



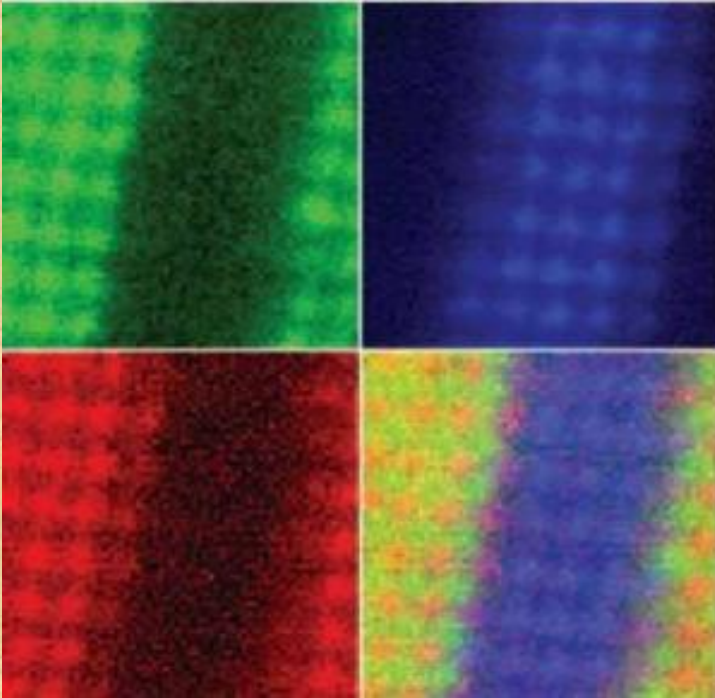
Layer structure on a silicon substrate: The material serves as low-noise receiver of microwave radiation in the universe. The sample originates from the KIT Institute for Micro- and Nanoelectronic Systems.
(Photo by: Siegel/Gerthsen).

STEM

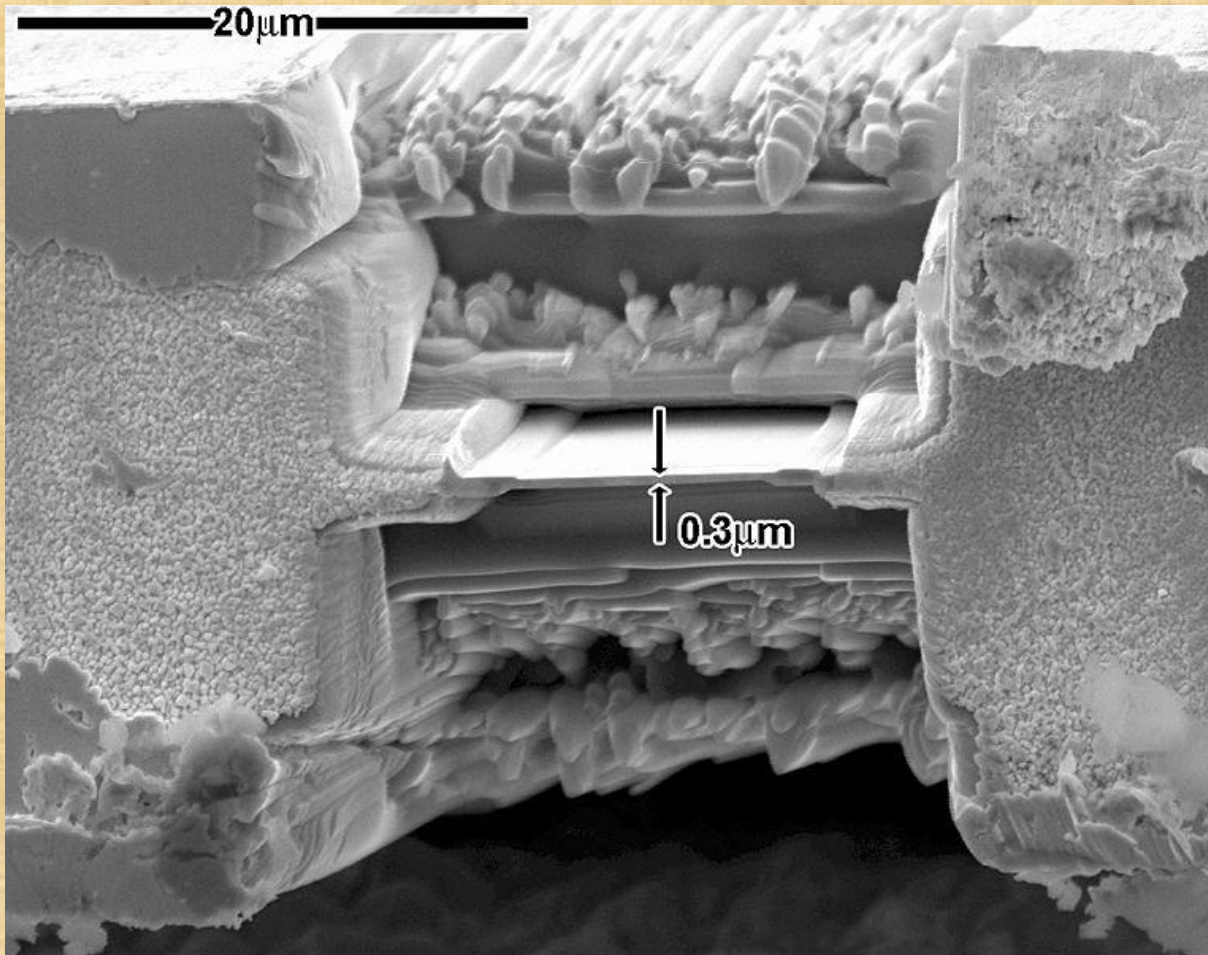
New type of scanning transmission electron microscope (STEM) incorporating new aberration-correction technology focuses a beam of electrons on a spot smaller than a single atom -- more sharply and with greater intensity than previously possible.

The STEM shoots an electron beam through a thin-film sample and scans the beam across the sample in subatomic steps.

The microscope can actually identify atoms using electron energy-loss spectrometry as it scans. Atoms in the path of the beam absorb energy from some of its electrons to kick their own electrons into higher orbits. The amount of energy this takes is different for each kind of atom giving a unique fingerprint



preparação



[SEM](#) image of a thin TEM sample milled by [FIB \(focussed ion beam\)](#). The thin membrane shown here is suitable for TEM examination; however, at $\sim 300\text{-nm}$ thick, it would not be suitable for high-resolution TEM without further milling.

wiki