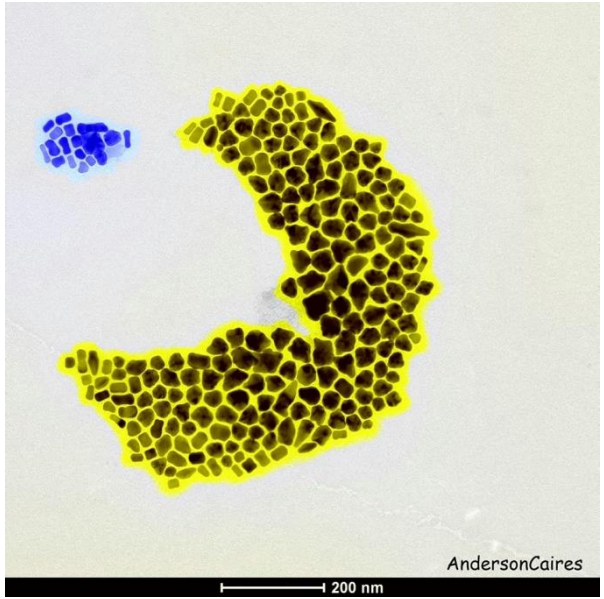
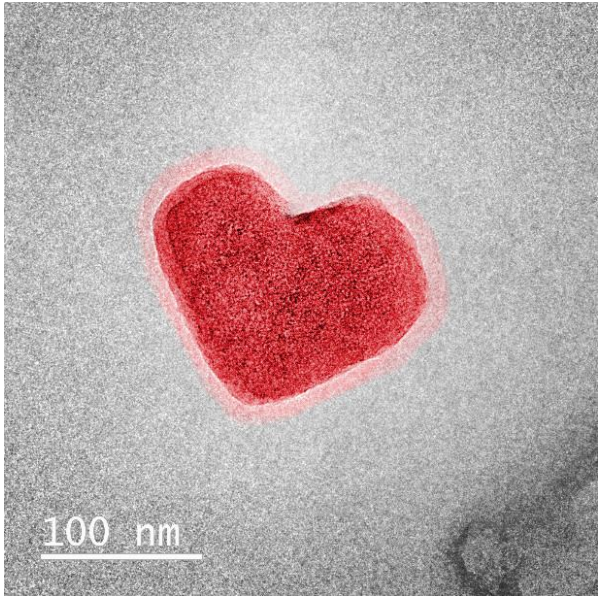
AFM

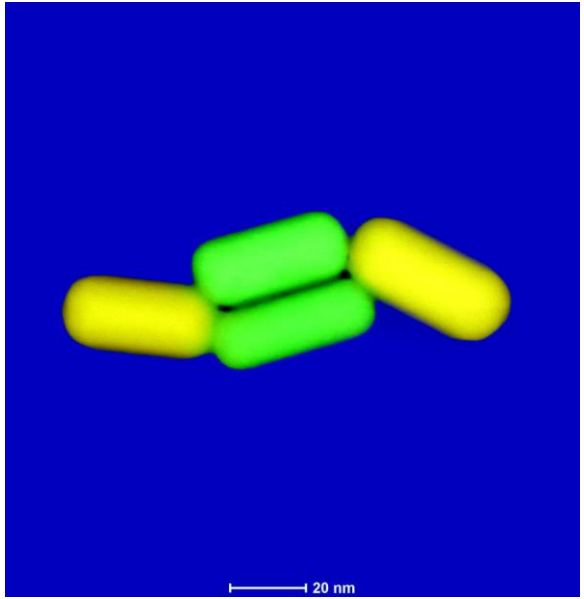
ANDERSON CAIRES

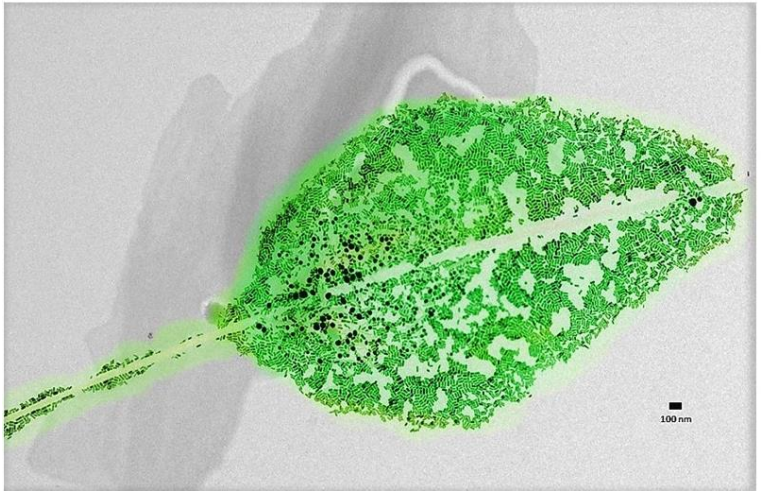
“A (outra) Aula”

TEM


AndersonCaires
200 nm


100 nm


20 nm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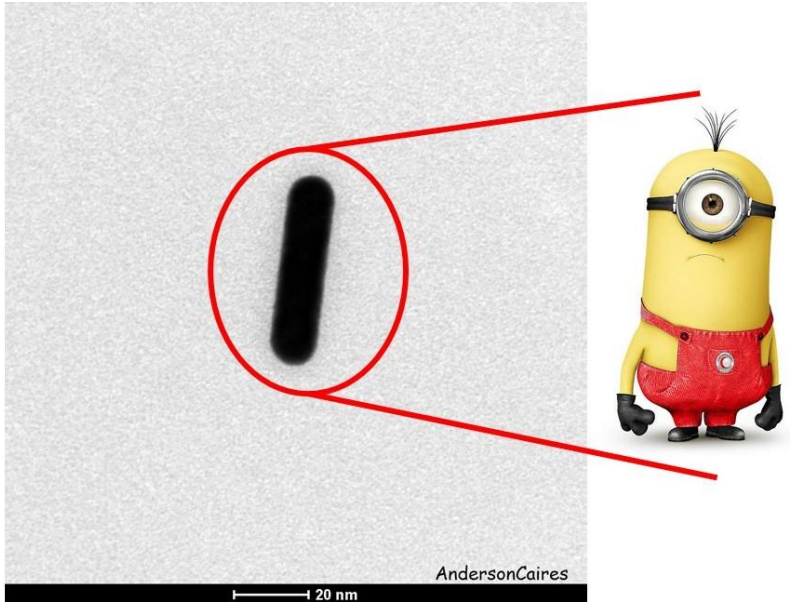
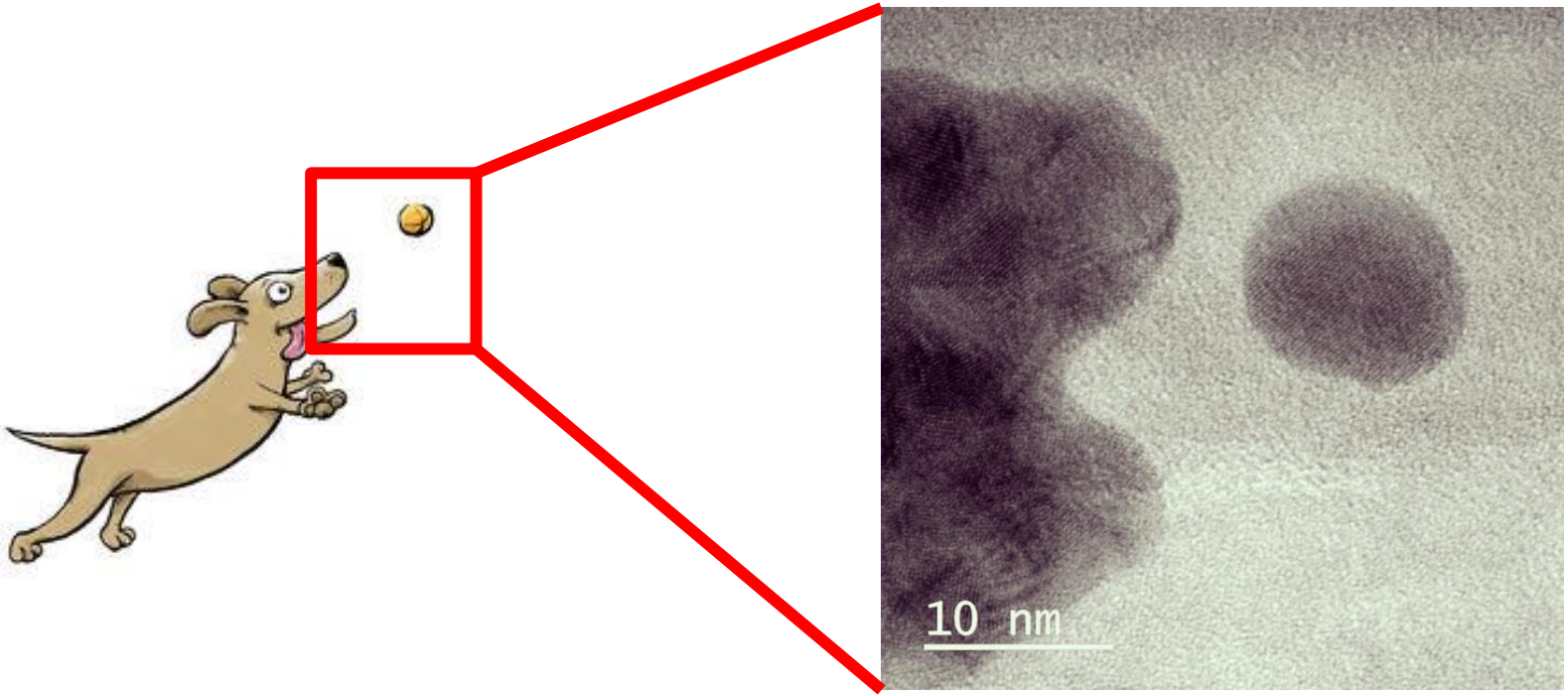
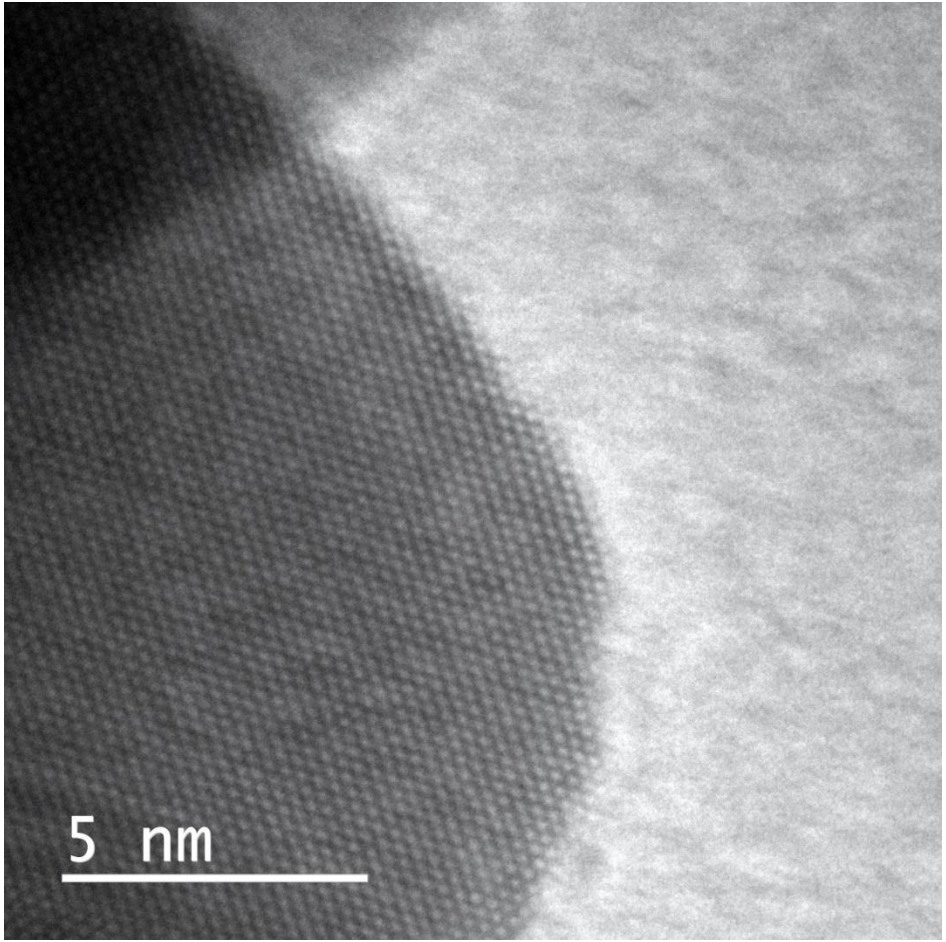
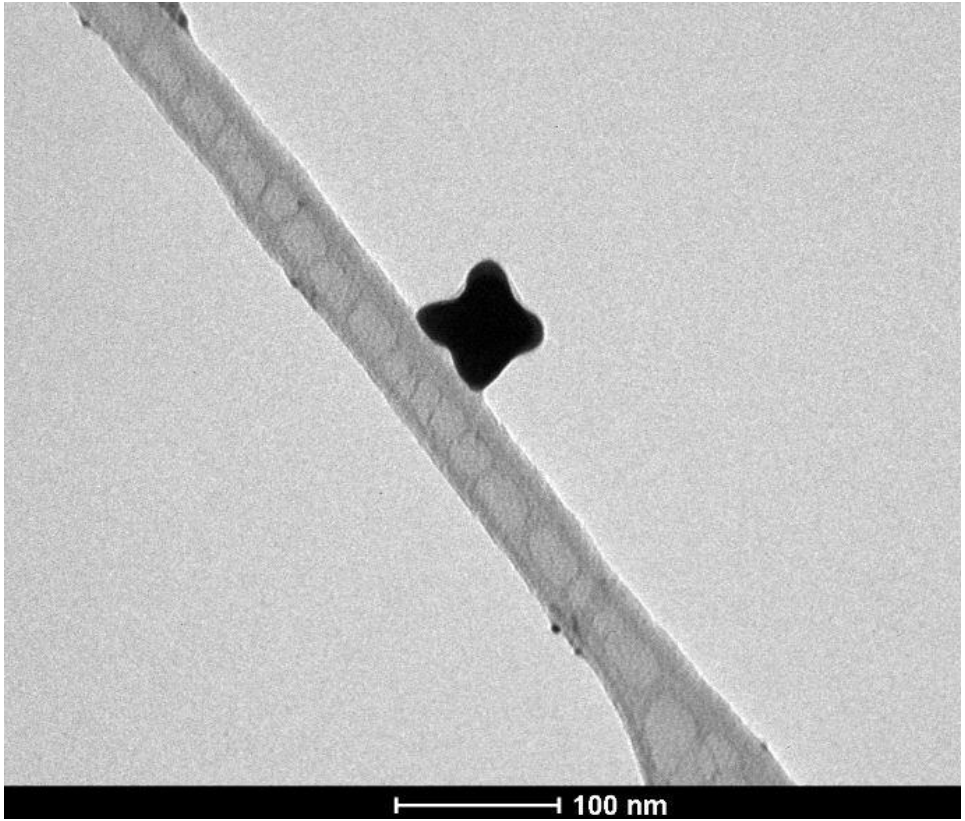
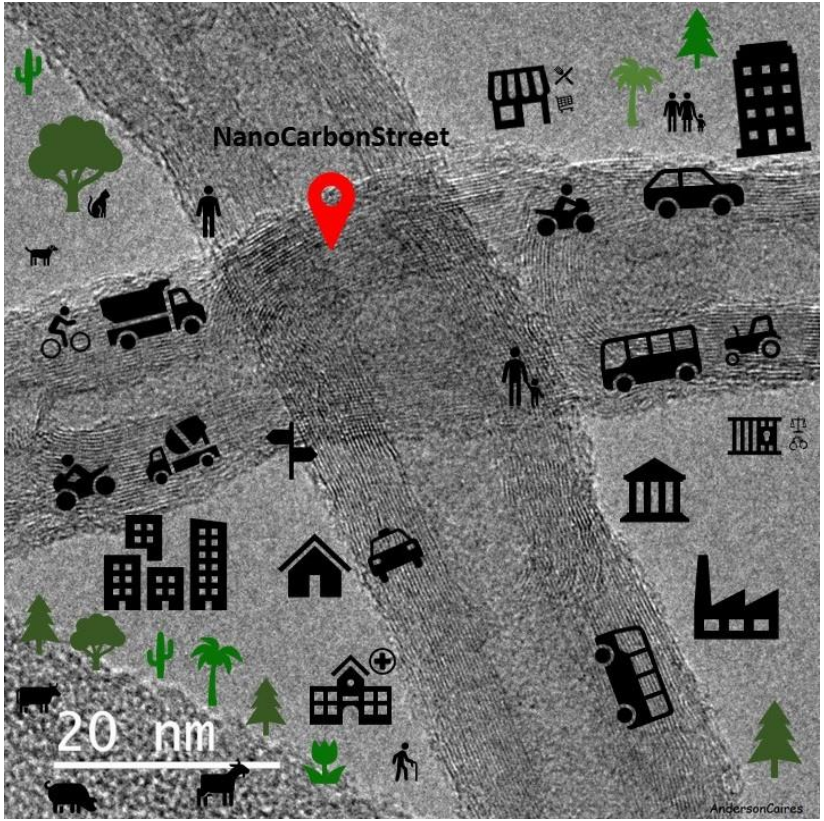

AndersonCaires
20 nm

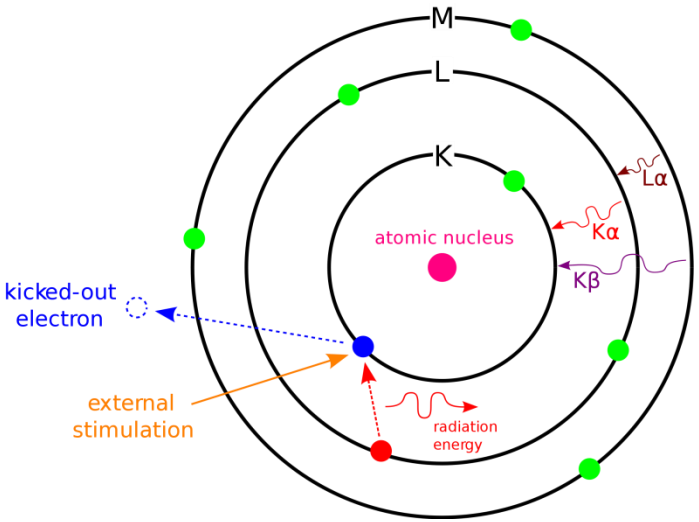

10 nm


5 nm


100 nm


NanoCarbonStreet
20 nm
AndersonCaires

EDS - Light elements



Hydrogen, **helium**, **lithium**, **beryllium** and **boron**

EDS - Light elements

- Low cross section
- Low fluorescence yield (less X-rays are produced because the formation of Auger electrons is favoured)
- Due to their low energies, many of the few X-rays produced are not able to leave the sample because they are absorbed before they can reach the sample surface
- Absorption and peak overlaps with L, M and N lines of heavier elements

EDS - Light elements

Analysis of Boron with Energy Dispersive X-ray Spectrometry

Advances in Light Element Analysis with SDD Technology

The analysis of light elements (from Be to F, refer to periodic table in fig. 1) presents a special challenge for energy dispersive X-ray spectrometry (EDS). Some of the problems are due to inherent physical effects, while others are technical in nature, relating to the design of the instrument used for analysis and the measurement procedure. Along with the demand for fast and efficient tools for analysis at the micro- and nanometer scale, the need for light element analysis with EDS has

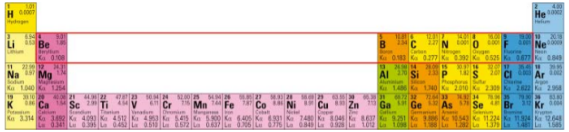
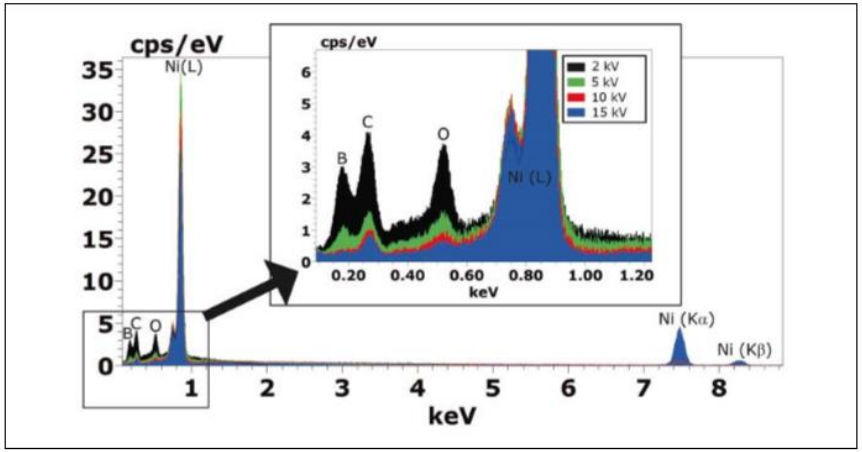


Fig. 1: Upper part of the periodic table: The light elements beryllium, boron, carbon, nitrogen, oxygen and fluorine present a challenge for X-ray spectrometry.



Electron diffraction - Reciprocal space

Basis vectors

Basis vectors in reciprocal space

$$\overrightarrow{a_1}^* = \overrightarrow{a}^*$$

$$\overrightarrow{a_2}^* = \overrightarrow{b}^*$$

$$\overrightarrow{a_3}^* = \overrightarrow{c}^*$$

$$\overrightarrow{a_4}^* = \overrightarrow{e_4} + \vec{q}$$

$$\vec{q} = \alpha \overrightarrow{a}^* + \beta \overrightarrow{b}^* + \gamma \overrightarrow{c}^*$$

Basis vectors in direct space

$$\overrightarrow{a_1} = \vec{a} - \alpha \overrightarrow{e_4}$$

$$\overrightarrow{a_2} = \vec{b} - \beta \overrightarrow{e_4}$$

$$\overrightarrow{a_3} = \vec{c} - \gamma \overrightarrow{e_4}$$

$$\overrightarrow{a_4} = \overrightarrow{e_4}$$

$$\overrightarrow{a_i}\,\overrightarrow{a_j}^* = \delta_{ij}$$

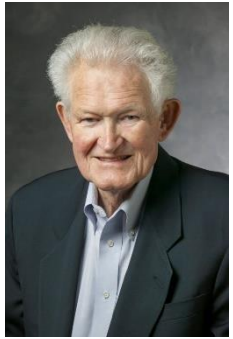
$$x = x_1 \overrightarrow{a_1} + x_2 \overrightarrow{a_2} + x_3 \overrightarrow{a_3} + x_4 \overrightarrow{a_4}$$

TEM 3D

Video AuNPs tomografia

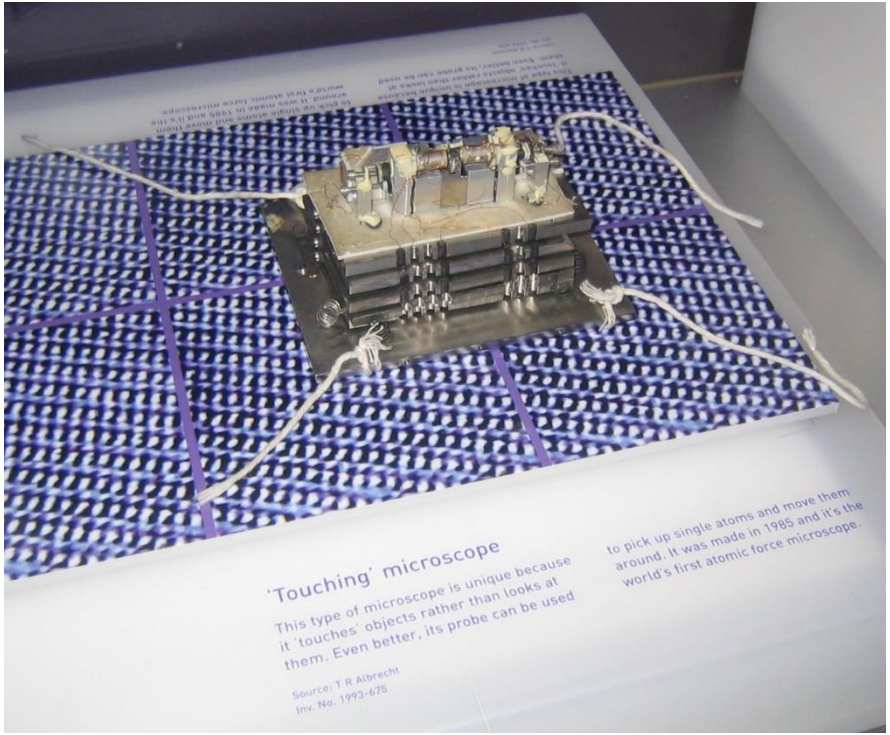
AFM

Primeiro Microscópio de Força Atômica (AFM)



Binnig, Calvin Quate and Christoph Gerber
1986

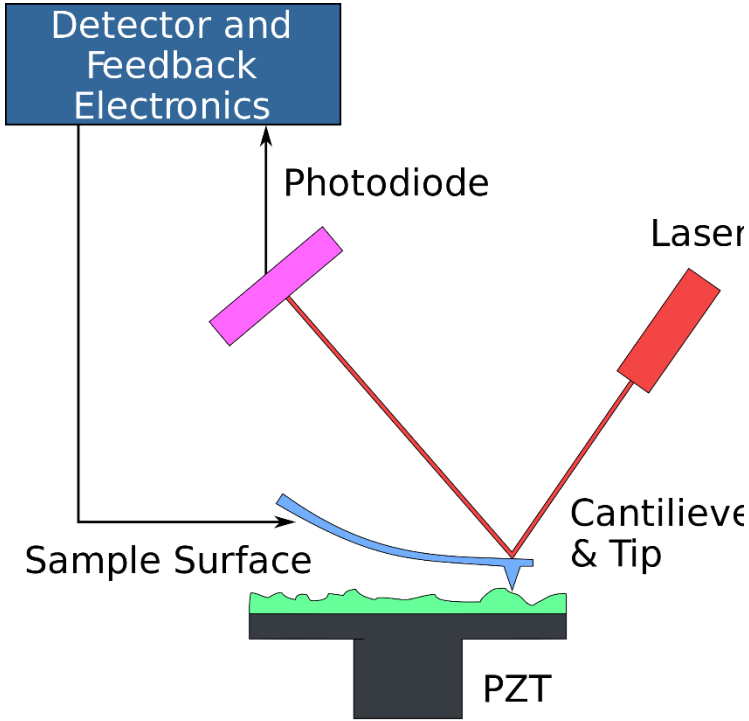
“Com objetivo de medir forças menores que 1µN entre a superfície da ponteira (tip) e a superfície da amostra"

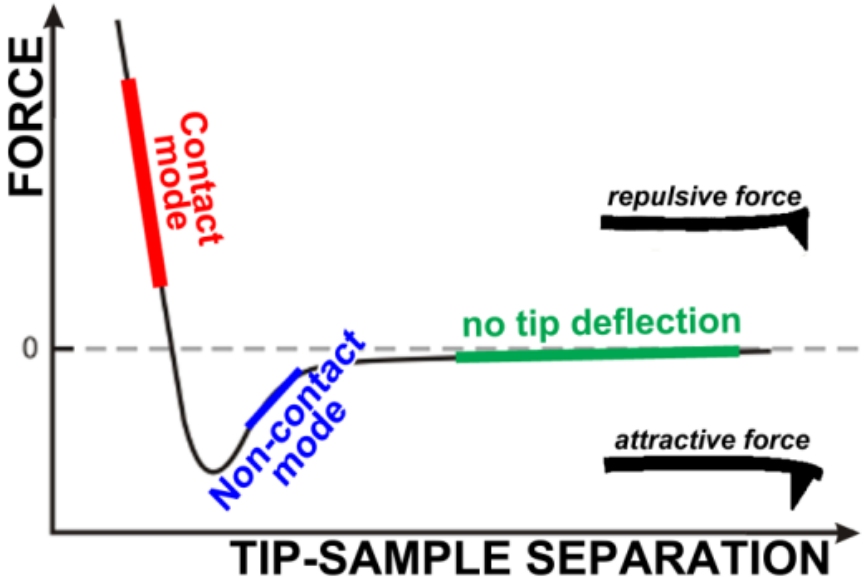


Springer Handbook of Nanotechnology,Bhushan, B., Ed. Springer ,2004, p.361,isbn=3-540-01218-4

https://pt.wikipedia.org/wiki/Microsc%C3%B3pio_de_for%C3%A7a_at%C3%B4mica#cite_note-1

Atomic Force Microscopy (AFM): Princípios básicos





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http://www.intechopen.com/source/html/42103/media/image2.png

Atomic Force Microscopy (AFM): Princípios básicos

LENNARD-JONES potential

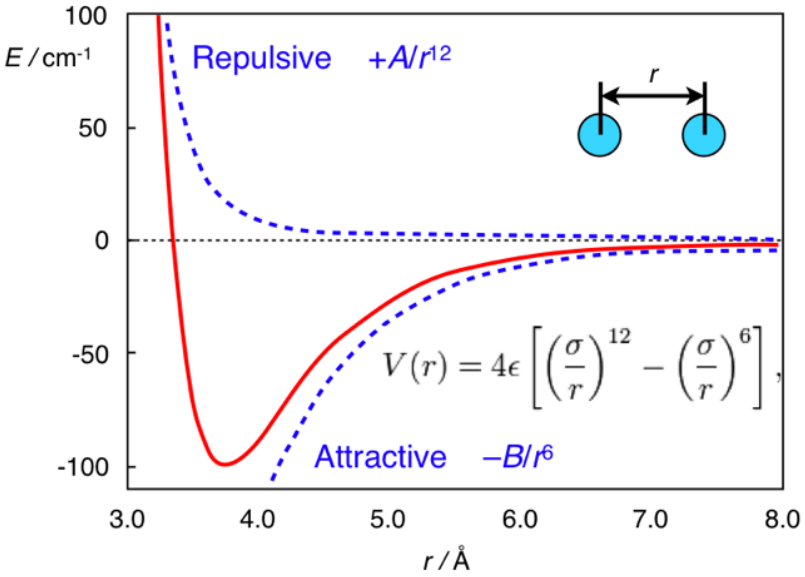
$$V(d) = 4\varepsilon \left(\underbrace{\left(\frac{\sigma}{d}\right)^{12}}_{\text{repulsion (short range)}} - \underbrace{\left(\frac{\sigma}{d}\right)^{6}}_{\text{attraction (long range)}} \right)$$

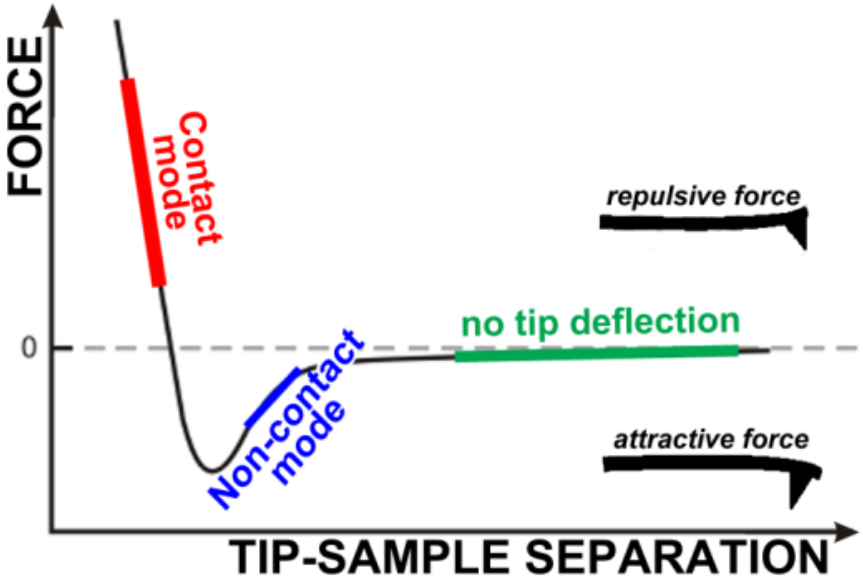
$$\varepsilon, \sigma = \text{const}$$

E = F Δs
E ~ eV; Δs~ Å
F ~ 2.10-9N

Em grandes distâncias, as interações são predominantemente atrativas, devido às forças de Van der Waals. Se aproximarmos ainda mais a ponta com a superfície, as interações são repulsivas devido a repulsão entre os orbitais eletrônicos dos átomos da superfície da amostra e os da ponta do microscópio de força atômica.

Atomic Force Microscopy (AFM): Princípios básicos





https://upload.wikimedia.org/wikipedia/commons/thumb/7/7c/Atomic_force_microscope_block_diagram.svg/2000px-Atomic_force_microscope_block_diagram.svg.png

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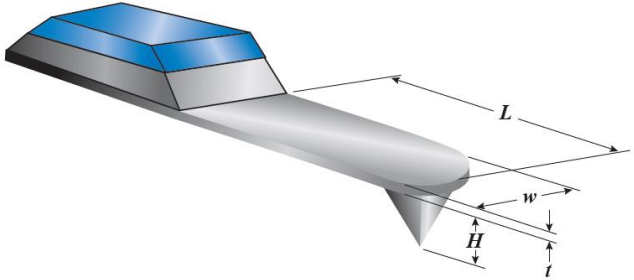
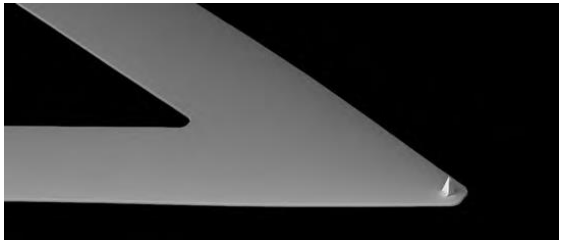
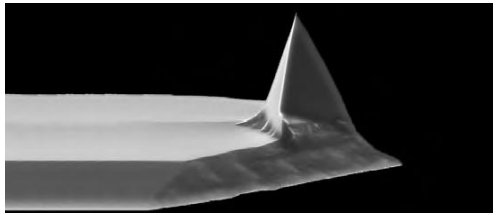
Vídeo (principal) parte 1

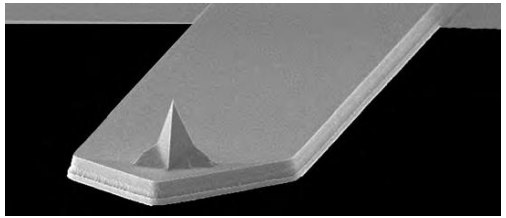
The Modern AFM

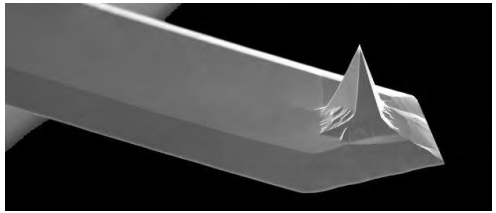
Cantilever











- Pontas reais e características

Vídeo Ponta (SEM)

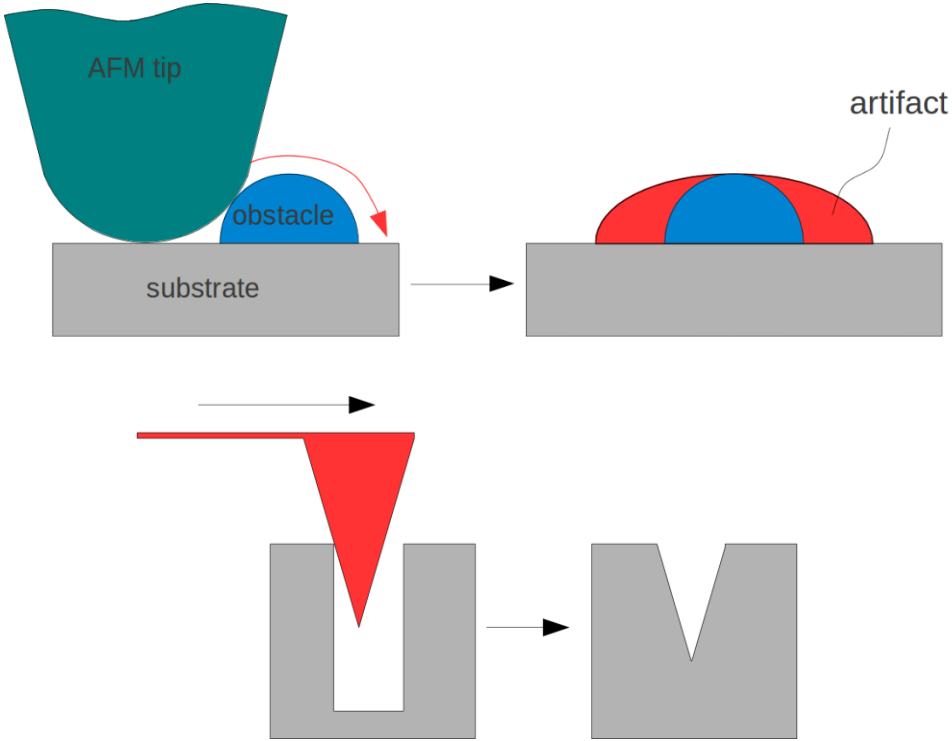
Contato

Não-contato

Tapping

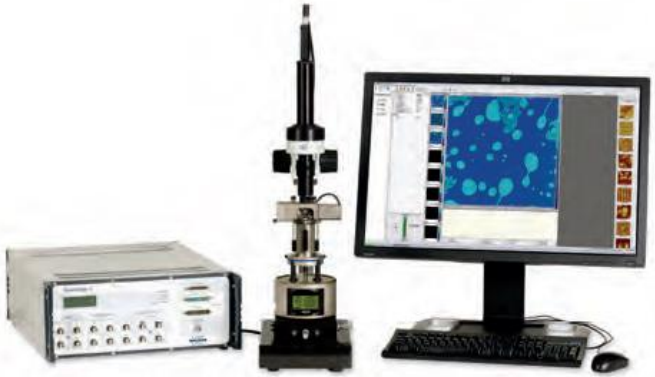
Vídeo (principal) parte 2

AFM Artefatos



Vídeo imagem AFM

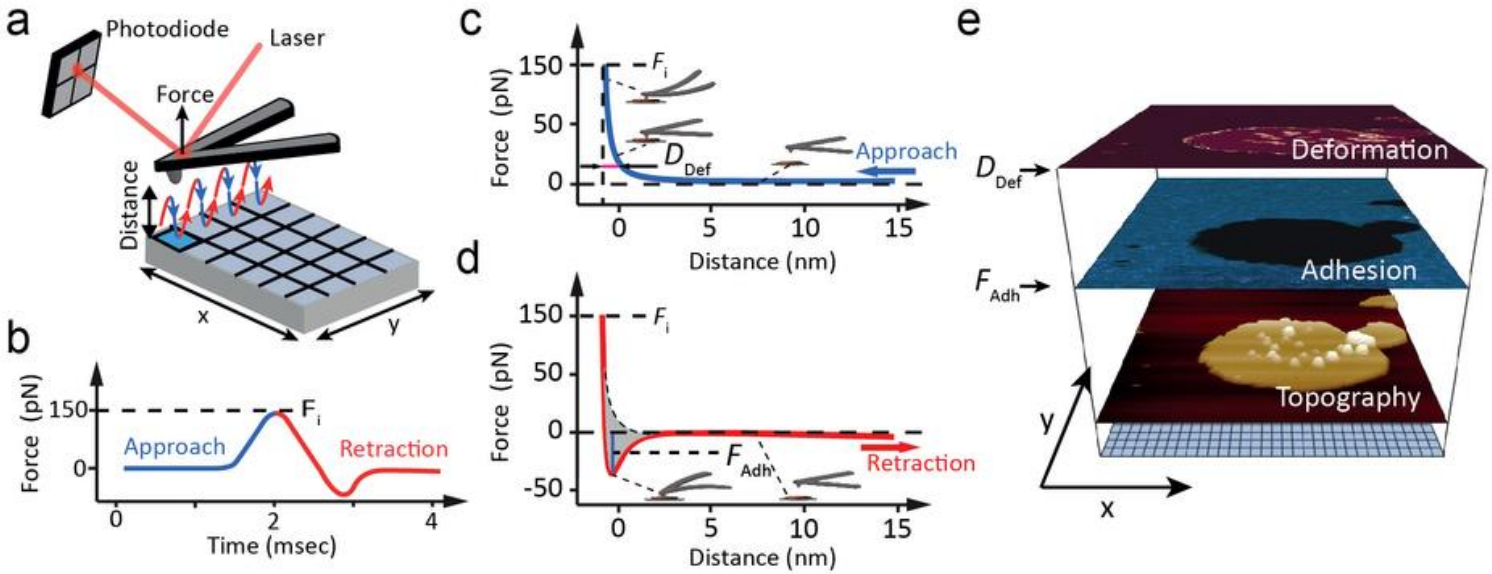
The Modern AFM



Vídeo (principal) parte 3

QNM

Quantitative Nanomechanical Mapping



Nature Methods 12, 2015, 845–851

Quantitative Nanomechanical Mapping

Adhesion: The peak force below the baseline.

Deformation: The maximum deformation of the sample (defined as the distance from the base of the **Deformation Fit Region** position to the peak interaction force position) caused by the probe.

DMT Modulus:

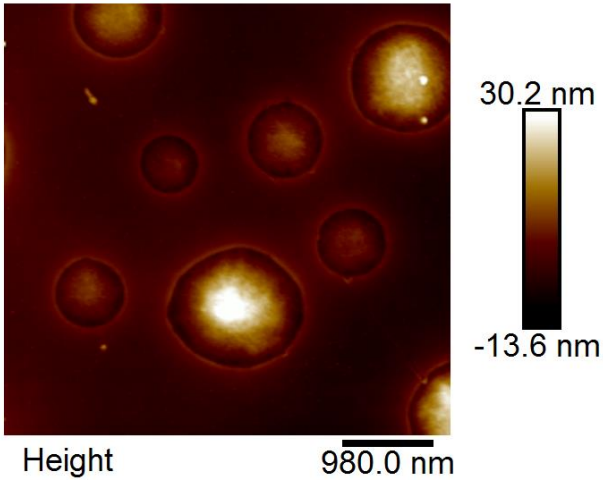
$$F_{tip} = \frac{4}{3}E^{*}\sqrt{Rd^{3}} + F_{adh}$$

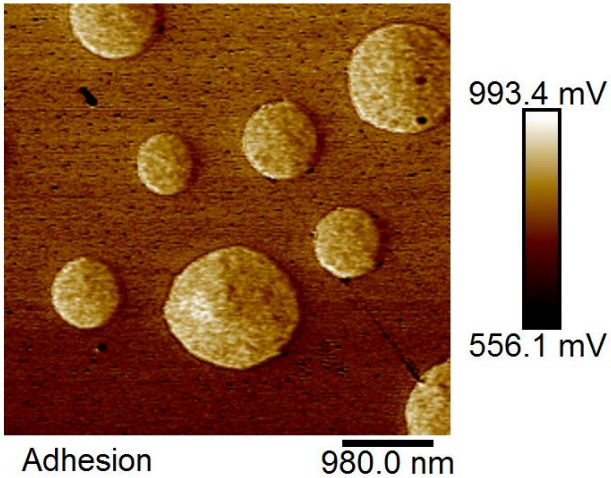
Dissipation:

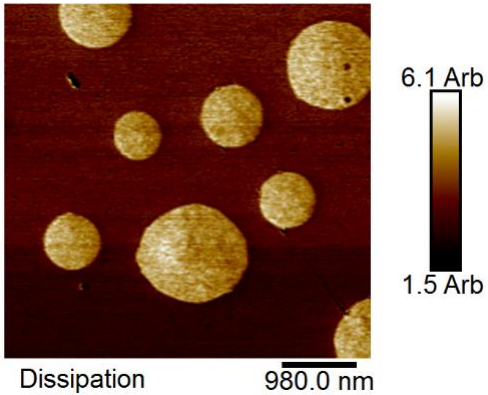
$$W = \int_{0}^{T} \bar{F} \bullet \bar{v}dt = \int \bar{F} \bullet d\bar{Z}$$

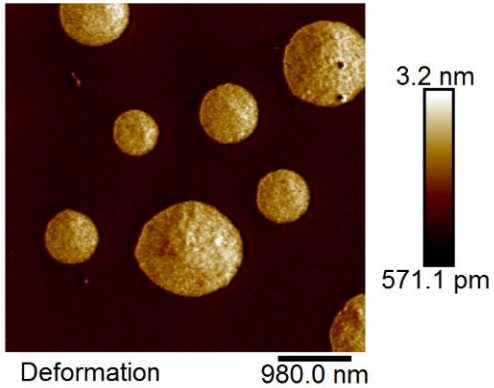
Quantitative Nanomechanical Mapping

PS+LDPE blend









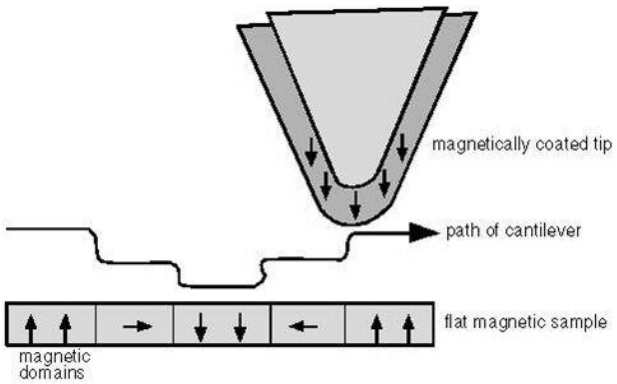
LAS MAT

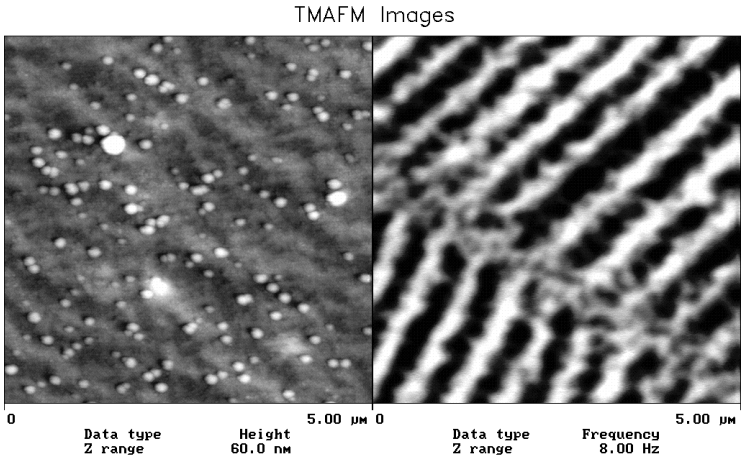
CeNano²I

MFM

Microscopia de força magnética

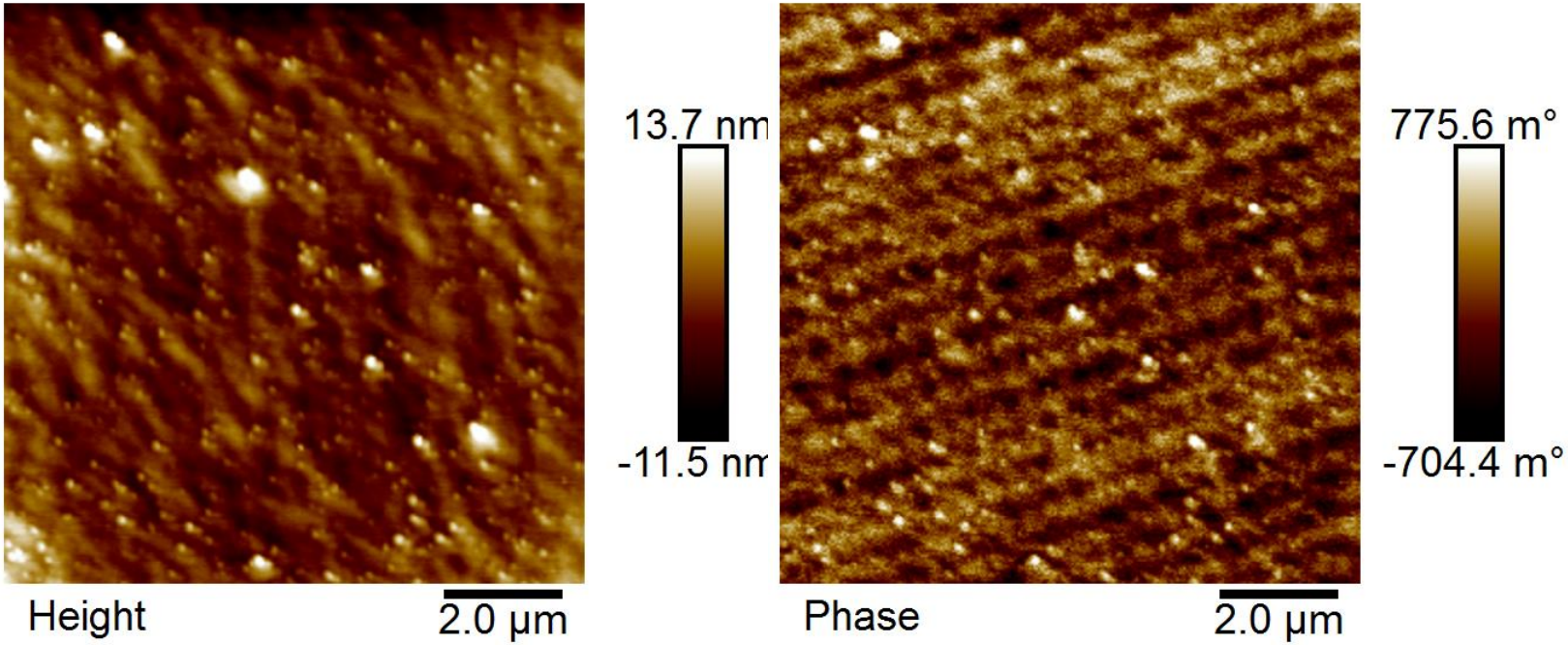
In the absence of magnetic forces, the cantilever has a resonant frequency f_0. This frequency is shifted by an amount Δf proportional to vertical gradients in the magnetic forces on the tip.





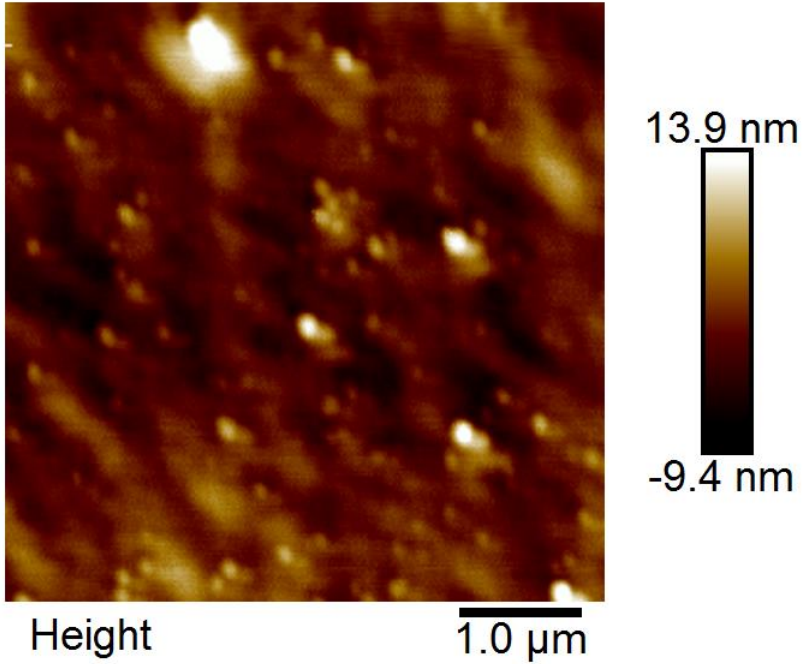
Microscopia de força magnética

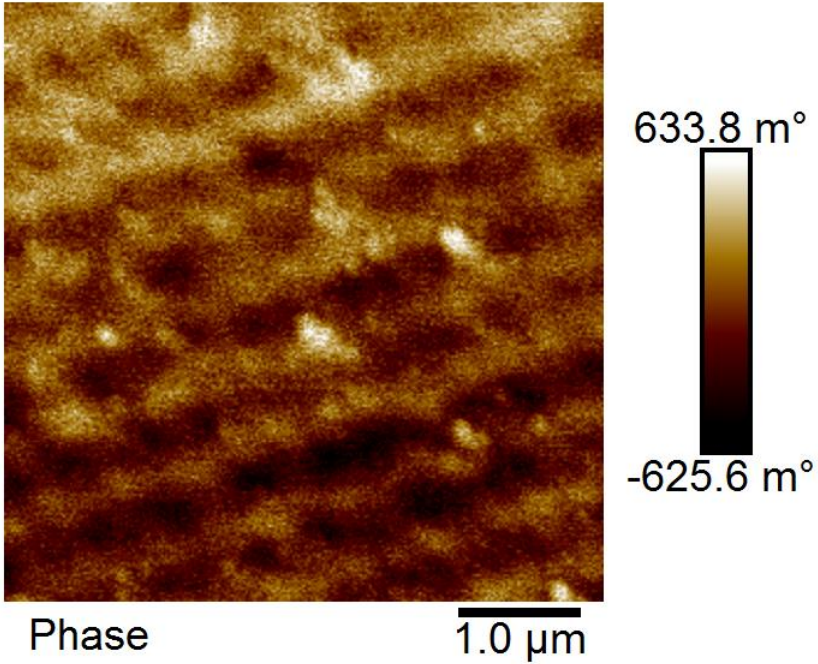
CeNano²I



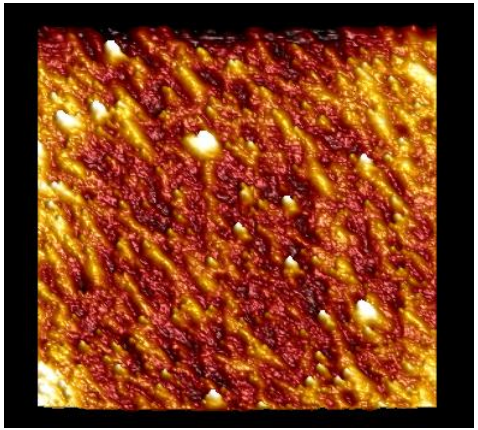
Microscopia de força magnética

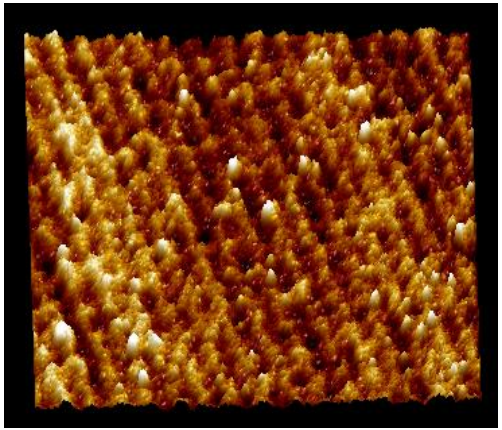
CeNano²I

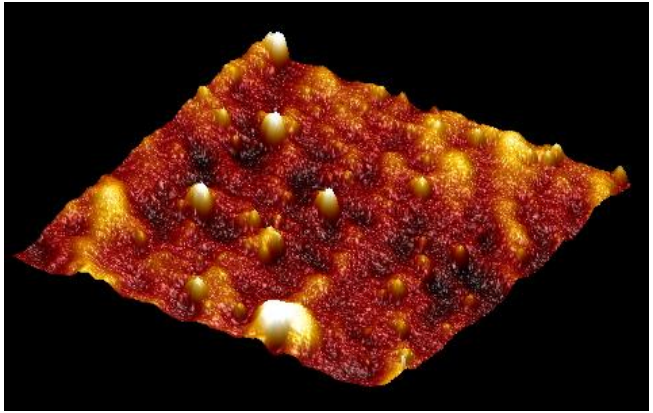


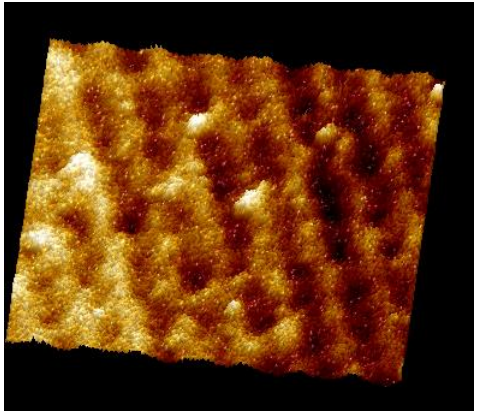


Microscopia de força magnética







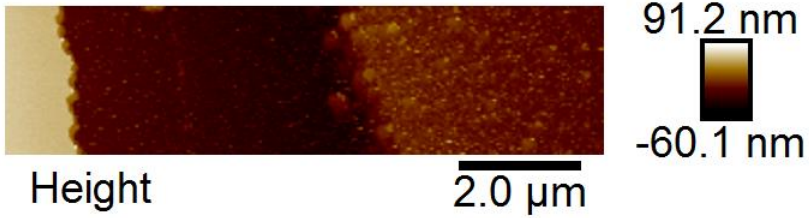


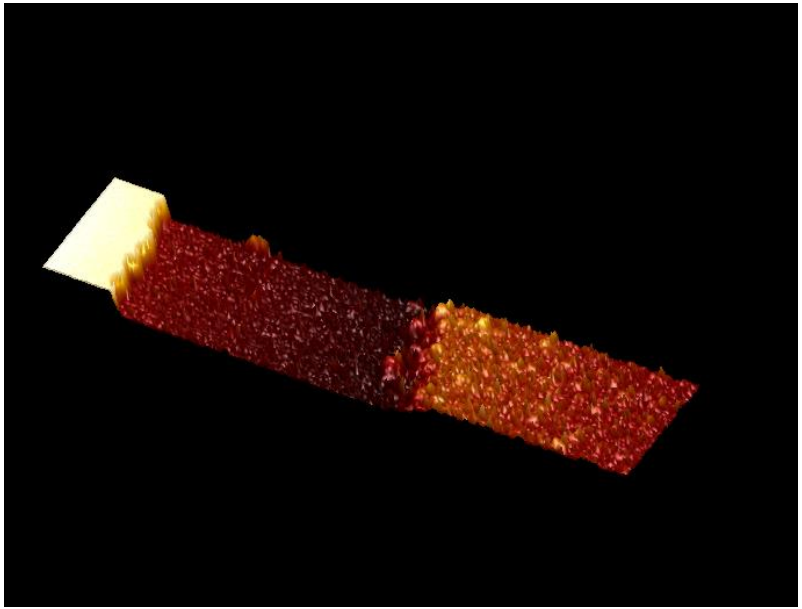
LAS MAT

CeNano²I

EFM

Microscopia de força elétrica



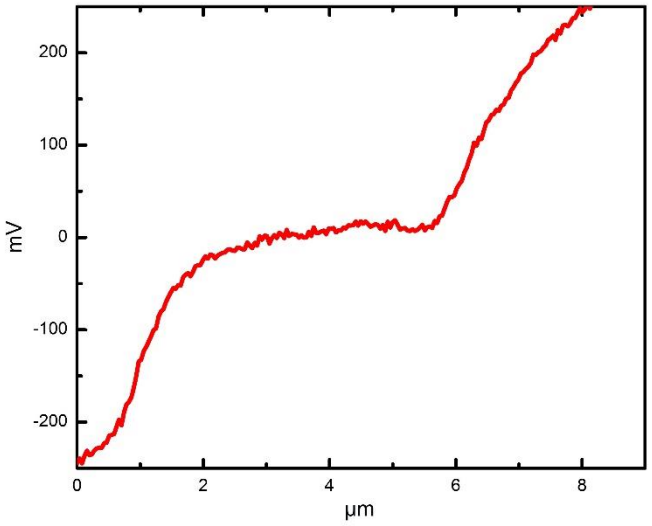


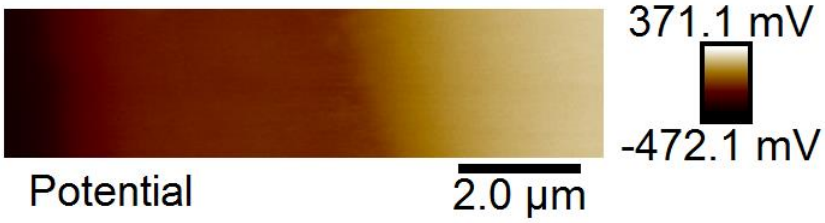
LAS MAT

CeNano²I

Microscopia de força elétrica

CeNano²I

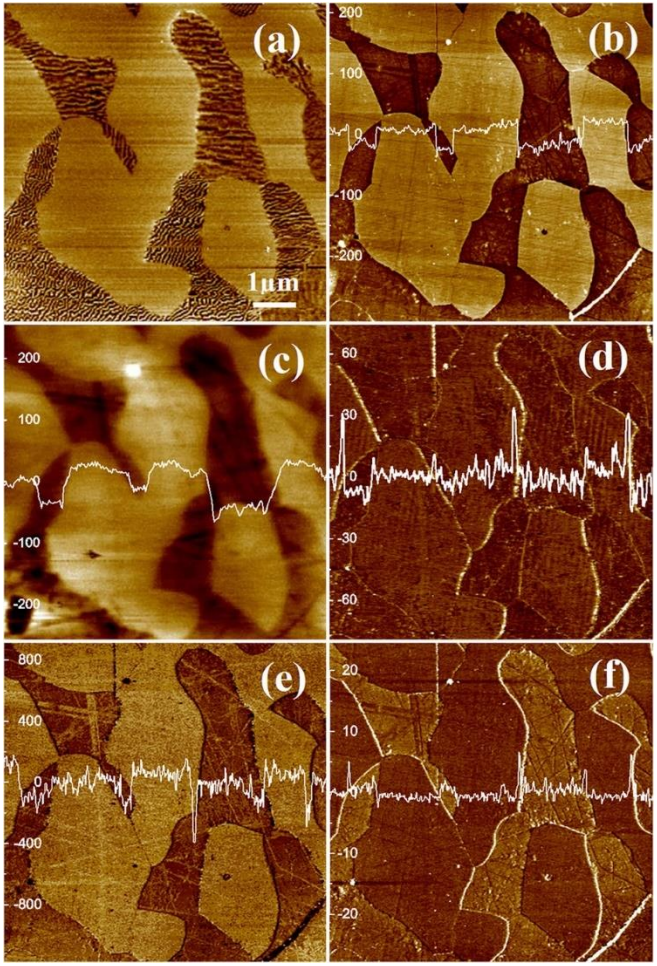




Aplicação em metalurgia

Ferrite and austenite phases in a duplex stainless

(a) A MFM image of the duplex stainless steel; (b) An AFM topography image (c) EFM potential mapping; (d) Adhesion mapping (nN); (e) Modulus (GPa) mapping; and (f) Deformation mapping (nm).



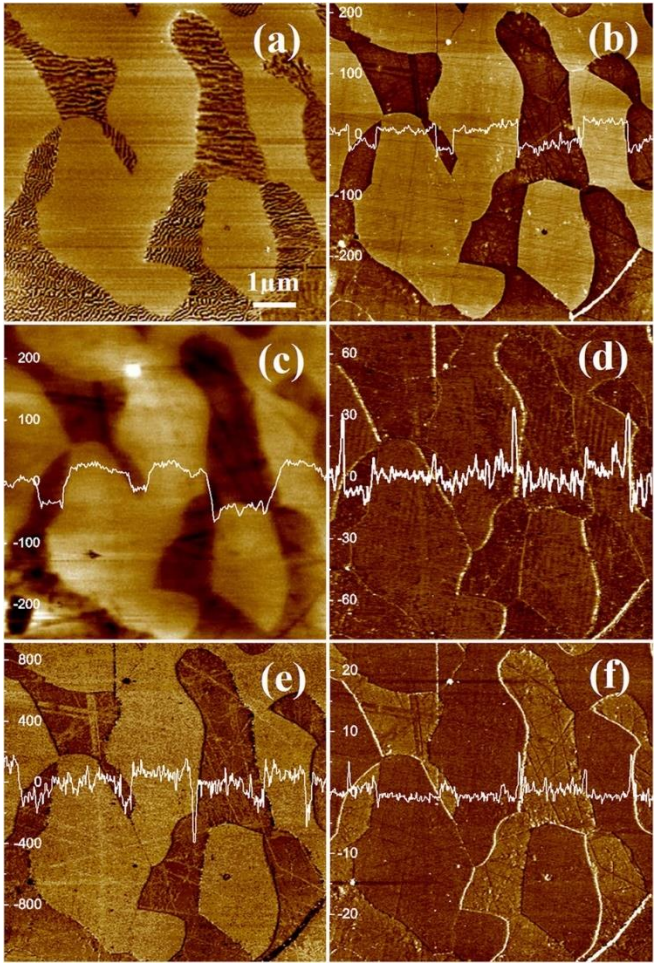
Nature Scientific Reports 6, 20660 (2016)

Aplicação em metalurgia

The ferrite phase has a striped appearance due to its ferromagnetic behavior, while the paramagnetic austenite phase shows a uniform appearance.

Topography (Fig. 2(b)) of the same area reveals that austenite (lighter) is higher than ferrite (darker). **The difference in height is caused by the electrochemical polishing during which the ferrite phase dissolved faster than austenite due to its relatively lower corrosion resistance.**

Nature Scientific Reports 6, 20660 (2016)

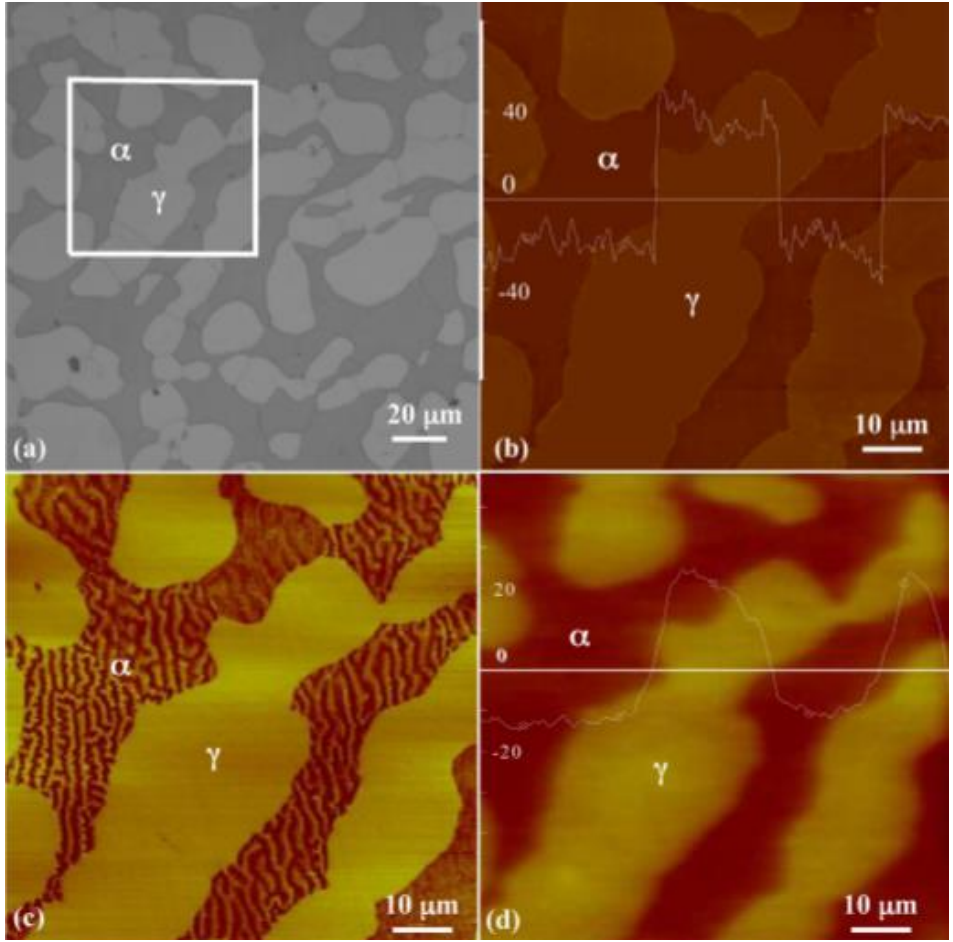


Aplicação em metalurgia

Ferrite and austenite phase identification in duplex stainless steel

Ferrite (α) - Austenite (γ) phases

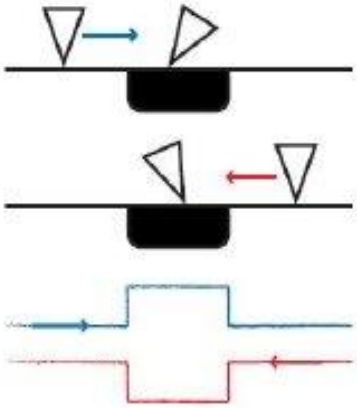
(a) Optical image duplex stainless steel surface; (b) AFM topography (c) MFM image; (d) EFM potential map.

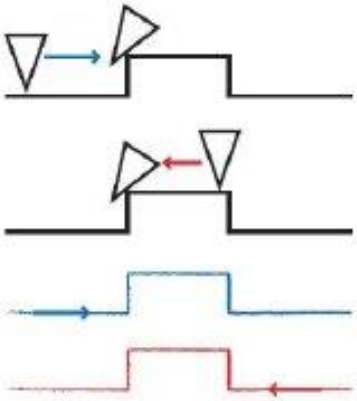


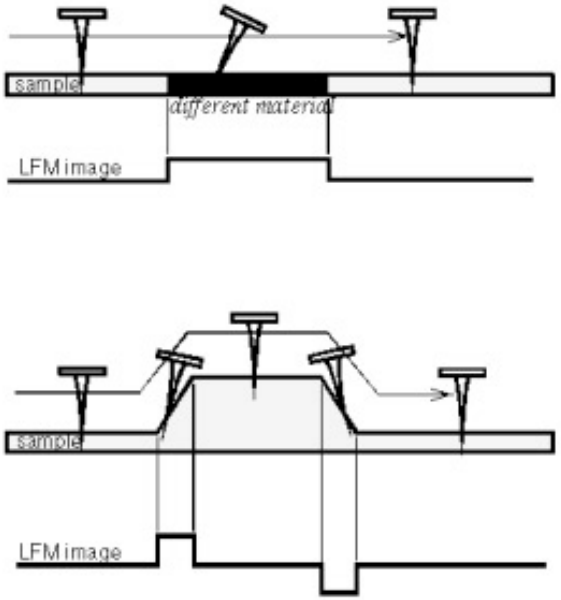
L.Q. Guo et al. /Applied Surface Science 287 (2013) 499– 501

LFM

Lateral Force Microscopy (LFM)

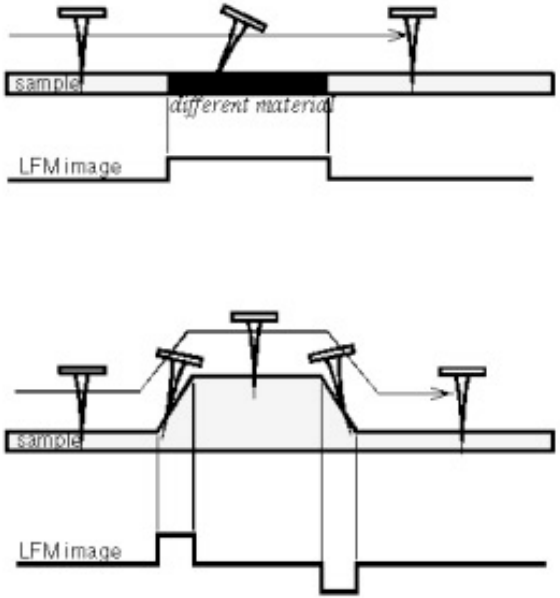


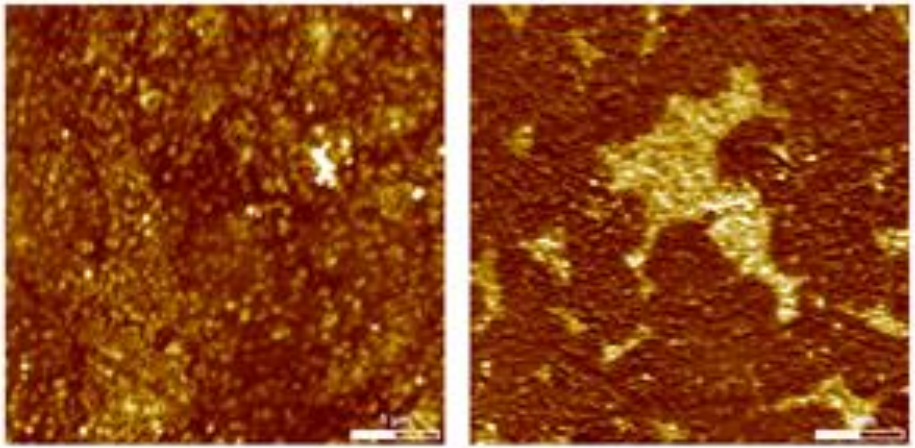




https://mai.ku.edu/lateral-mode

Lateral Force Microscopy (LFM)



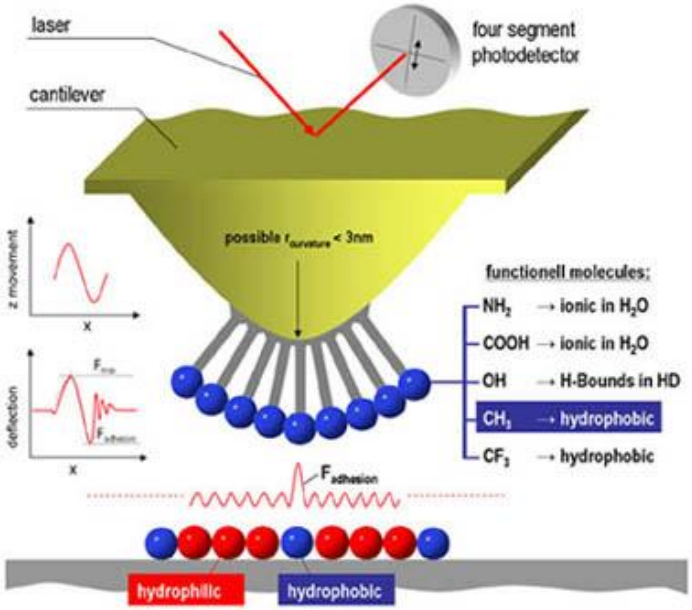


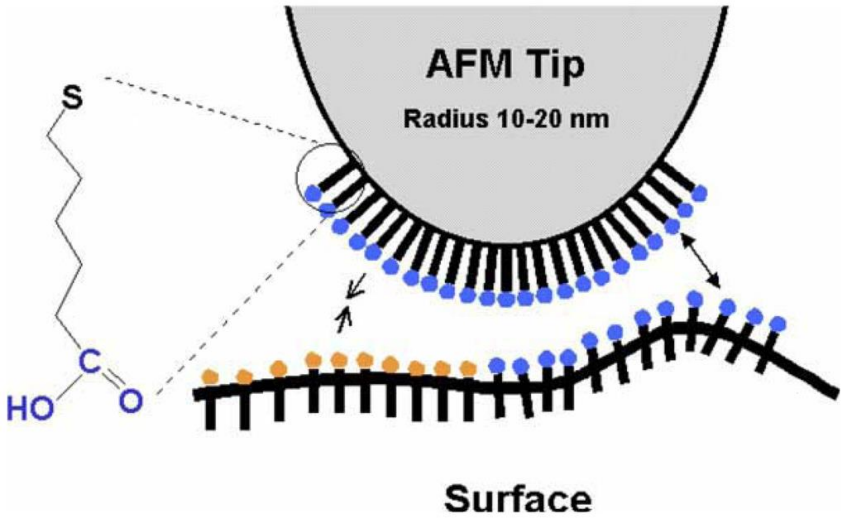
Both topographic (left) and LFM (right) images were acquired simultaneously.

Fonte: Park Systems

CFM

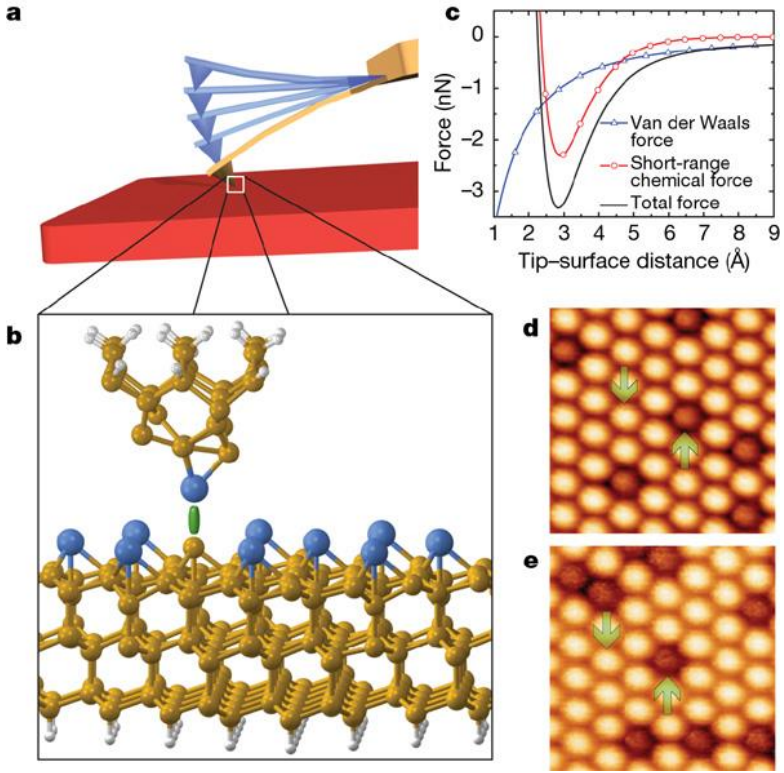
Chemical force microscopy (CFM)





http://www.mdpi.com/sensors/sensors-12-08278/article_deploy/html/images/sensors-12-08278f1-1024.png
http://www.nanocraft.de/stream/images/service/cfmcontrast.jpg

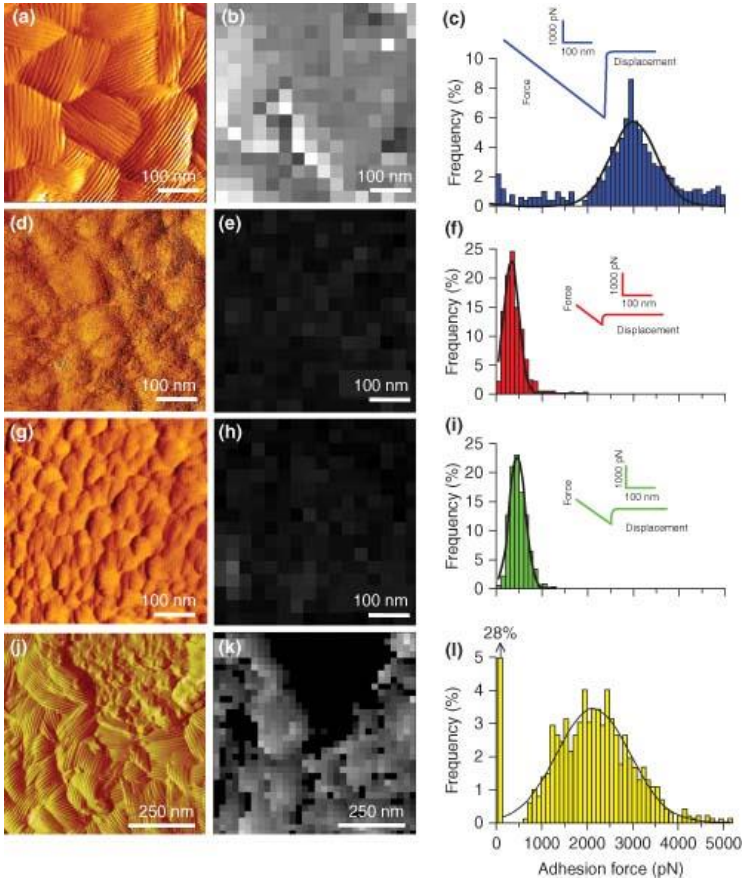
Chemical force microscopy (CFM)



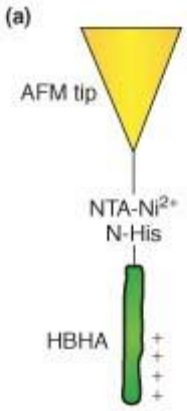
Chemical identification of individual surface atoms by atomic force microscopy

Nature 446, 64-67(1 March 2007)

Chemical force microscopy (CFM)

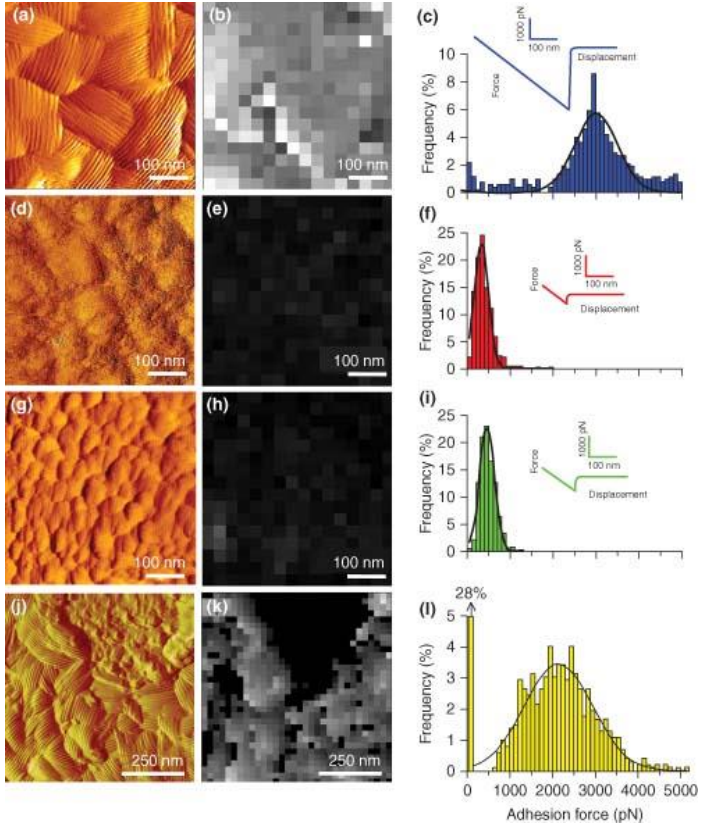


Nanoscale imaging of microbial pathogens using atomic force microscopy



WIREs Nanomed Nanobiotechnol, 1: 168–180. doi:10.1002/wnan.18

Chemical force microscopy (CFM)



Nanoscale imaging of microbial pathogens using atomic force microscopy

Chemical force microscopy (CFM) of *A. fumigatus* conidia.

(a-c) **Hydrophobic tip**, indicating that the rodlet surface is uniformly hydrophobic. **(Bright)**

(d–f) NaOH-treated conidia **(Dark)**

(g–i) The ΔrodA ΔrodB double mutant **(Dark)**

(The purely hydrophilic surface is attributed to cell wall polysaccharides).

(j–l) Detergent-treated conidia, revealing highly correlated structural and hydrophobic heterogeneities **(Bright)**

WIREs Nanomed Nanobiotechnol, 1: 168–180. doi:10.1002/wnan.18

AFM-SEM

Hybrid AFM-SEM

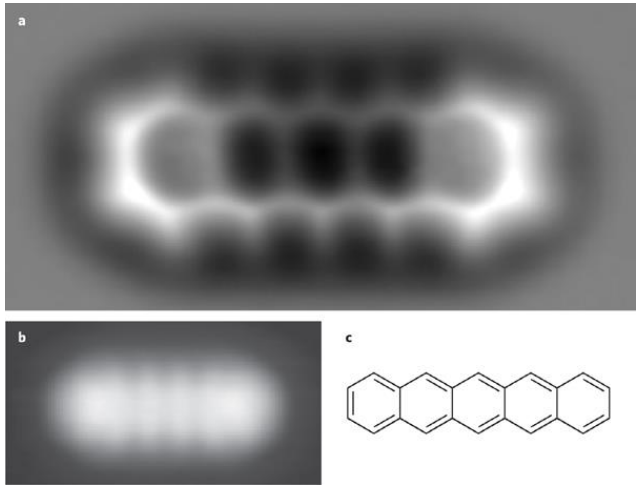
https://www.nanosurf.com/en/products/afsem-afm-for-sem

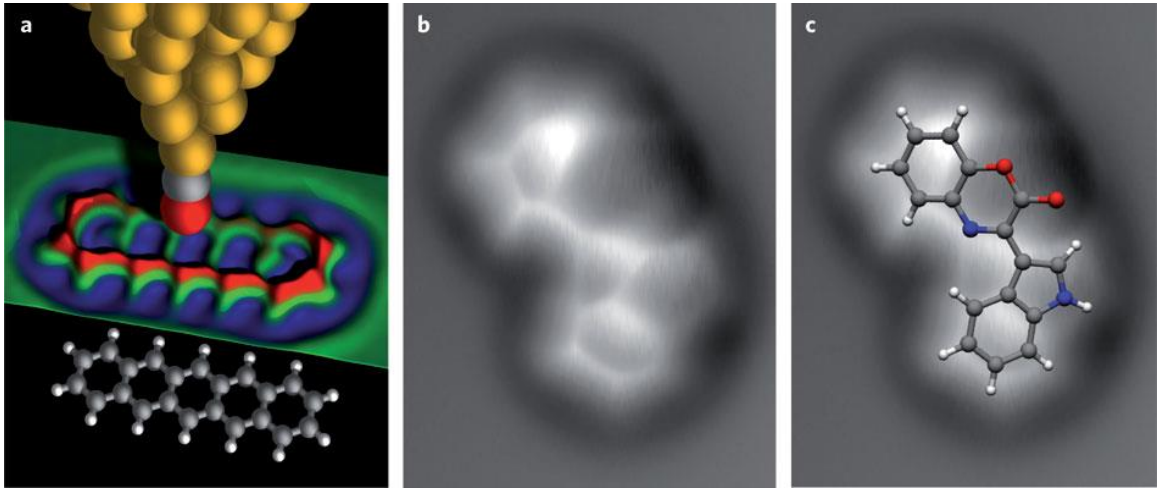
http://www.microscopy-analysis.com/blog/blog-articles/hybrid-afm-sem-%E2%80%93-complementary-not-competitive-techniques

http://www.dme-spm.com/remafm.html

Molecular imaging

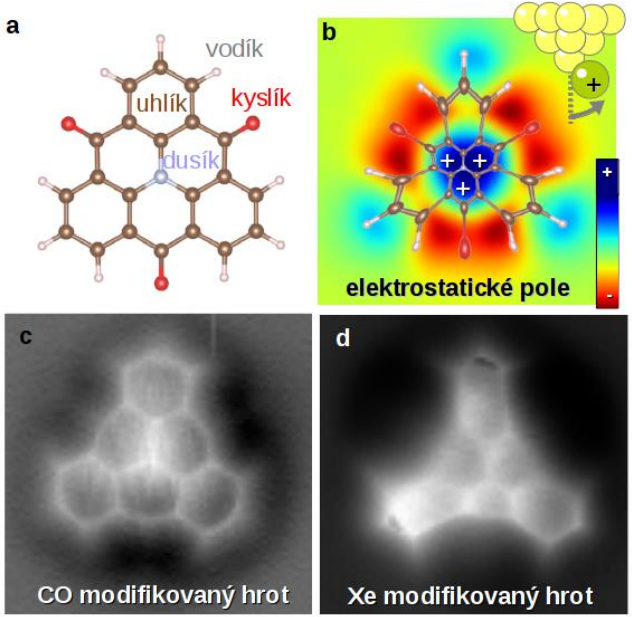
Molecular imaging: The tip of what can be seen

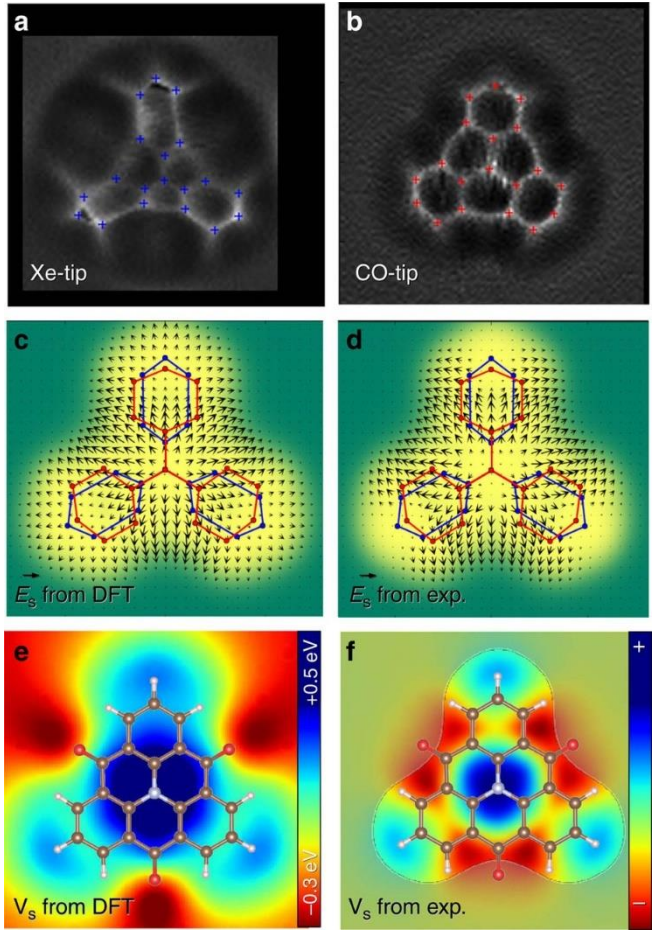




Nature Chemistry 3, 2011, 273–278

Molecular imaging





Nature Communications 7, 11560 (2016)

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