

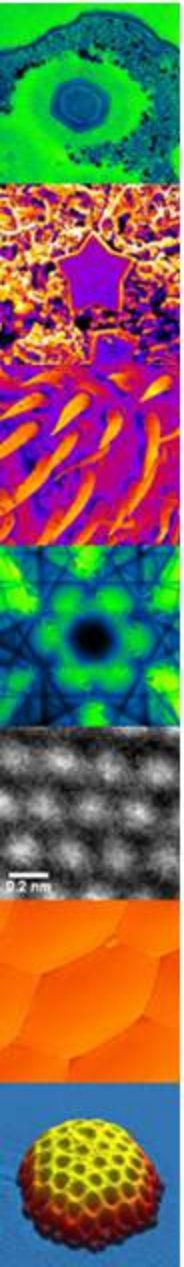
# Cristalografia

Karla Balzuweit

Dep. De Física – ICEX – UFMG

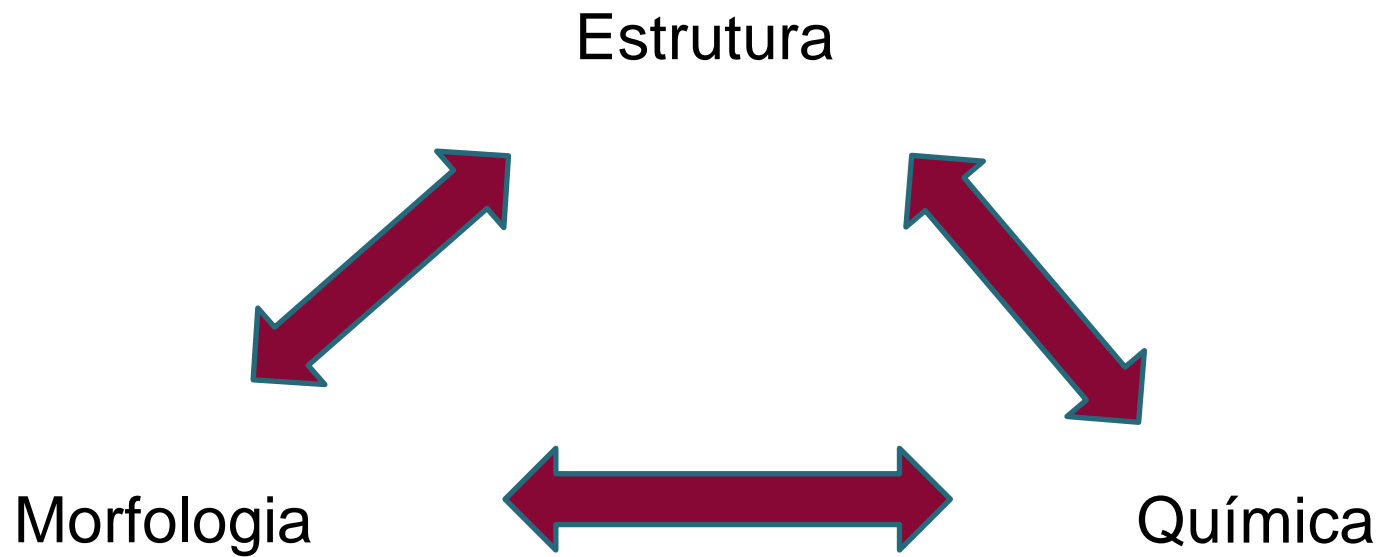
Centro de Microscopia da UFMG





Hoje em dia:

MUITO MAIS QUE UMA GRANDE LUPA!!



# Classificações dos Estados da Matéria

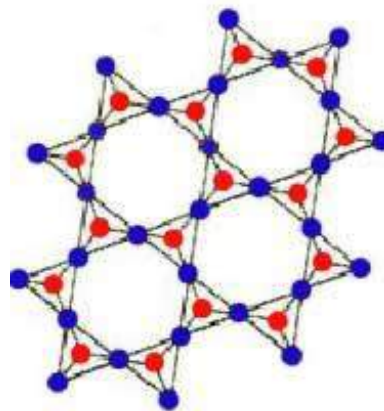
- Os três “clássicos” – sólido, líquido, gasoso;
- Mais recentemente: 4º estado - plasmático ( $T \gg$ ),  
5º estado - condensado de Bose-Einstein ( $T \ll$ );
- Outros: supersólido, superfluido, cristal líquido, fluido supercrítico, coloidal, matéria degenerada, sólido amorfo (vítreo, não-vítreo) x sólido cristalino, condensado fermiônico, plasma quark-glúon, matéria fracamente simétrica, fortemente simétrica, etc.

Estado sólido:

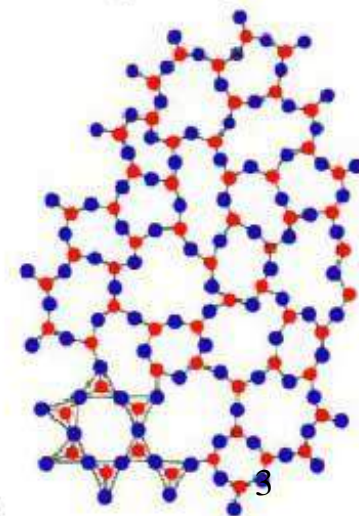
sólidos cristalinos x não-cristalinos  
(= amorfos).

Sólidos vítreos: caso particular de não-cristalino

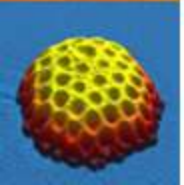
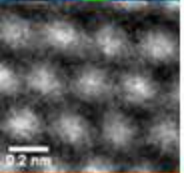
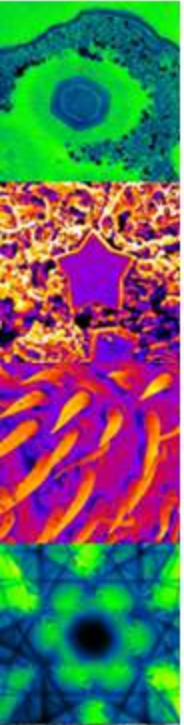
Crystalline SiO<sub>2</sub>  
(Quartz)



Amorphous SiO<sub>2</sub>  
(Glass)



● Si ● O



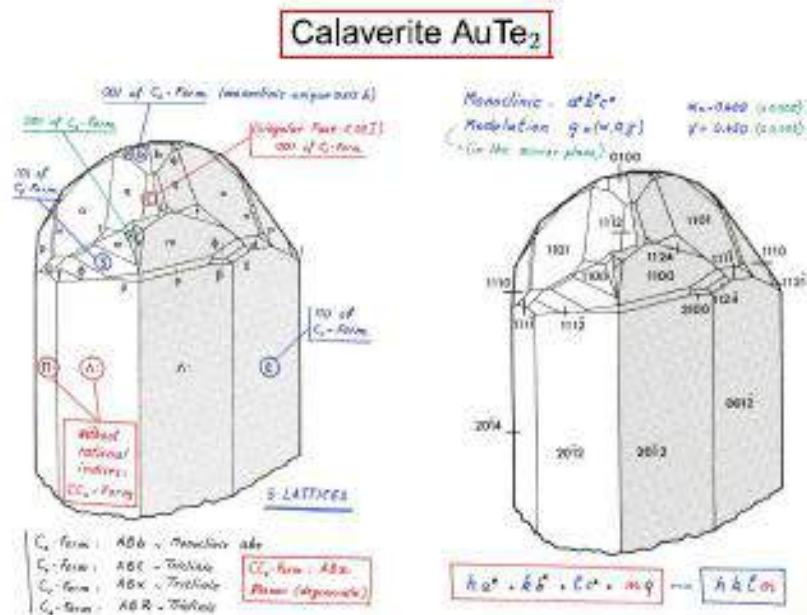
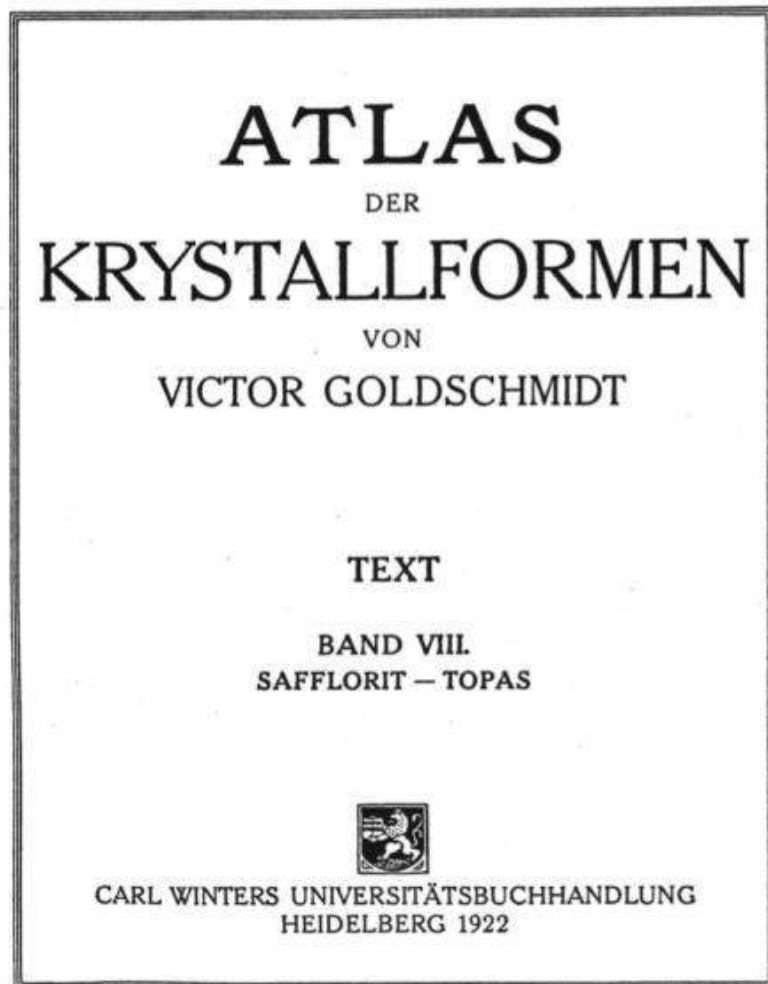
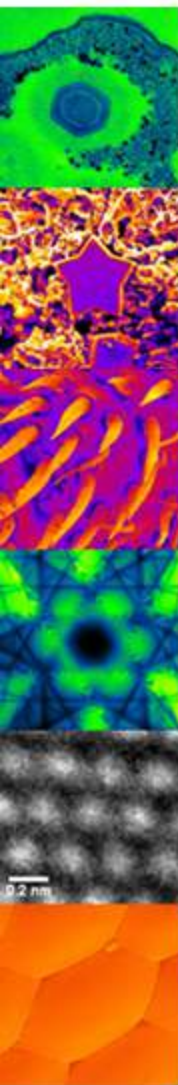
Água marinha



Topázio Imperial

Janet Anneberg Hooker Hall of Geology, Gems and Minerals:  
<http://www.si.edu/resource/faq/nmnh/mineralsciences.htm>

# Como a matéria se organiza?

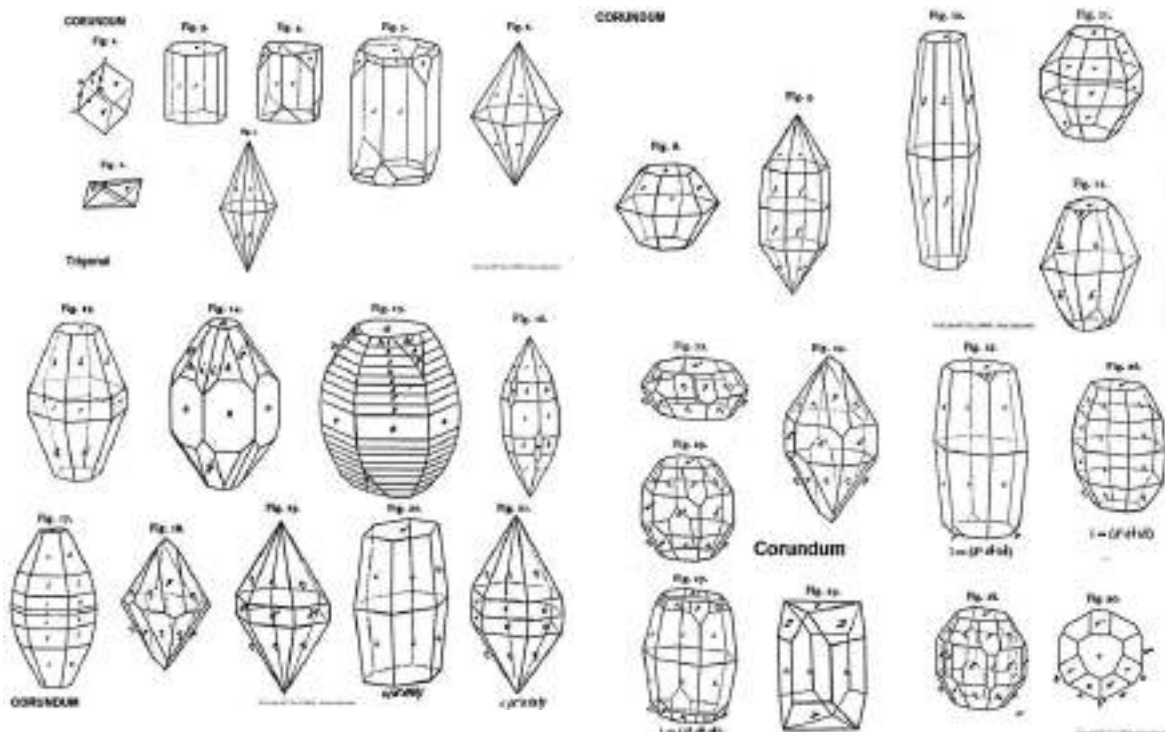


Goldschmidt, Palache, Peacock

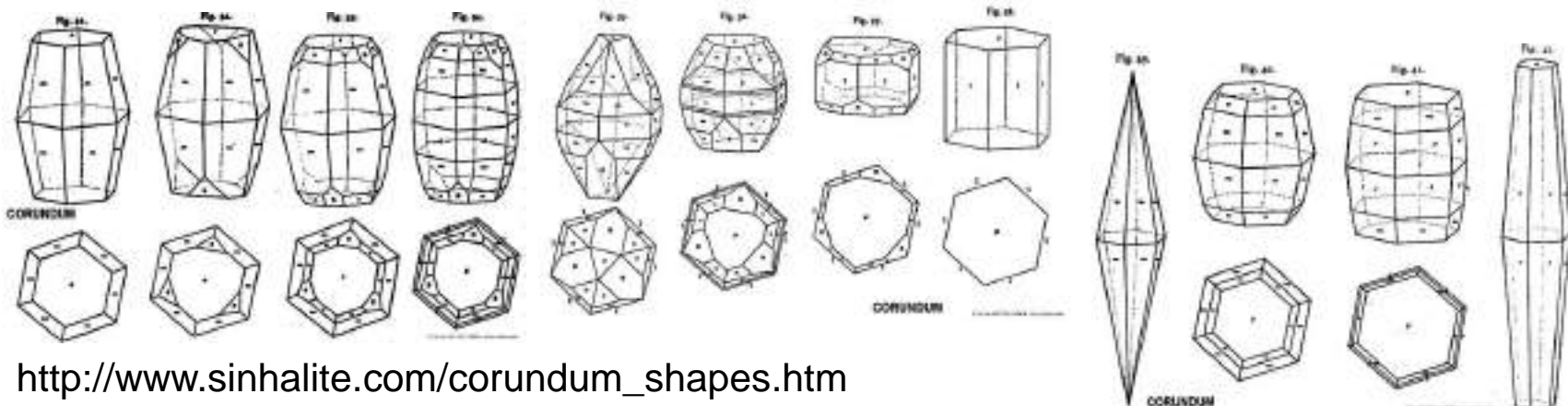
Dam, Janner, Donnay

<http://docenten.science.ru.nl/Janner/le/ape.pdf>

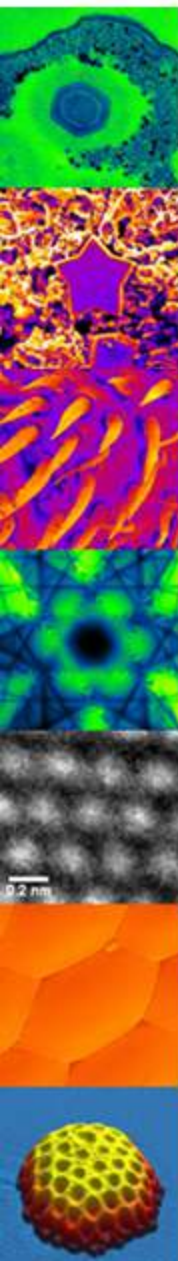
# Corundum – $Al_2O_3$



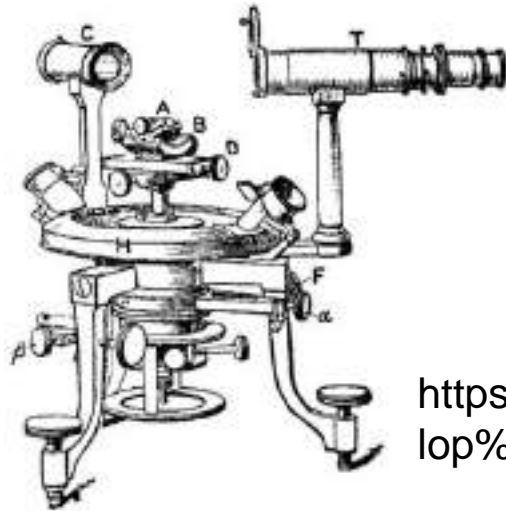
<https://pt.wikipedia.org/wiki/Corundum>



[http://www.sinhalite.com/corundum\\_shapes.htm](http://www.sinhalite.com/corundum_shapes.htm)



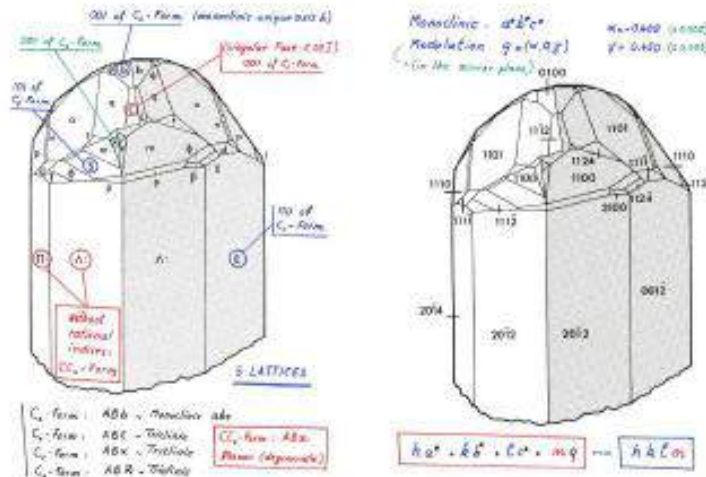
# Goniômetro óptico



[https://en.wikisource.org/wiki/1911\\_Encyclop%C3%A6dia\\_Britannica/Goniometer](https://en.wikisource.org/wiki/1911_Encyclop%C3%A6dia_Britannica/Goniometer)

<http://www.mineralogy.eu/gonio.html>

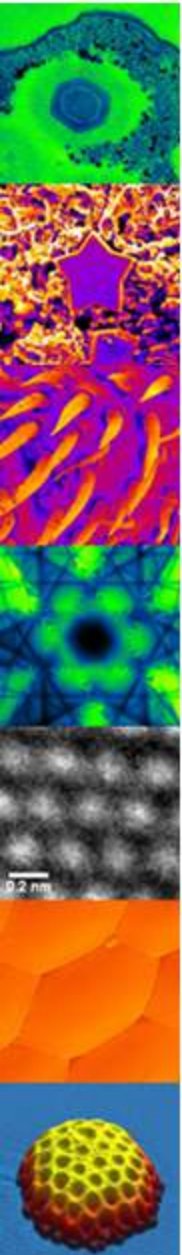
Calaverite  $AuTe_2$

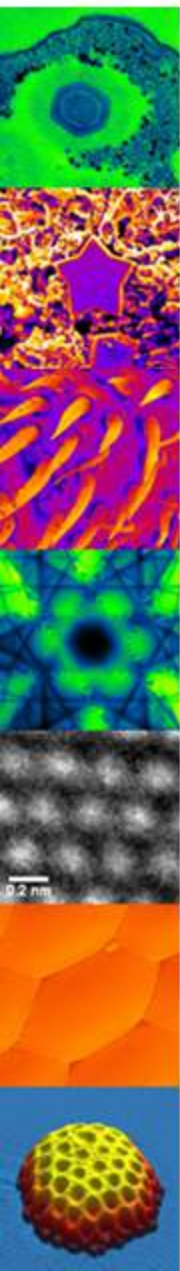


Goldschmidt, Palache, Peacock

Dam, Janner, Donnay

<http://docenten.science.ru.nl/Janner/le/ape.pdf>





# Cristalografia

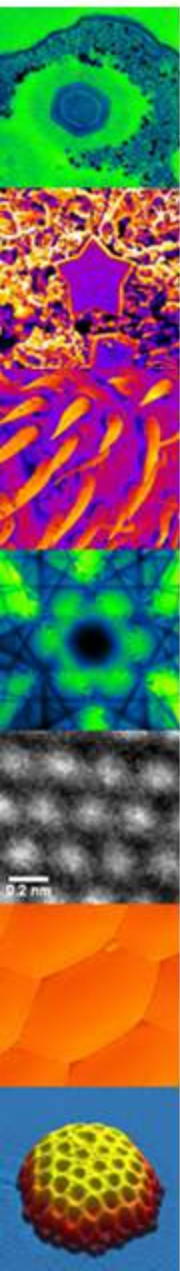
- Cristalografia = descrição de cristais
- Cristal – do grego “krystallos” = gelo
- Cristal – sólido homogêneo, que possui arranjo interno (atômico) tridimensional, com ordem de longo alcance (= *long-range order*).
- Cristalografia: originalmente um ramo da Mineralogia, relativamente tardio entre as Ciências – iniciou-se no século XVII (Steno, Carangeot, de l’Isle, Haüy)



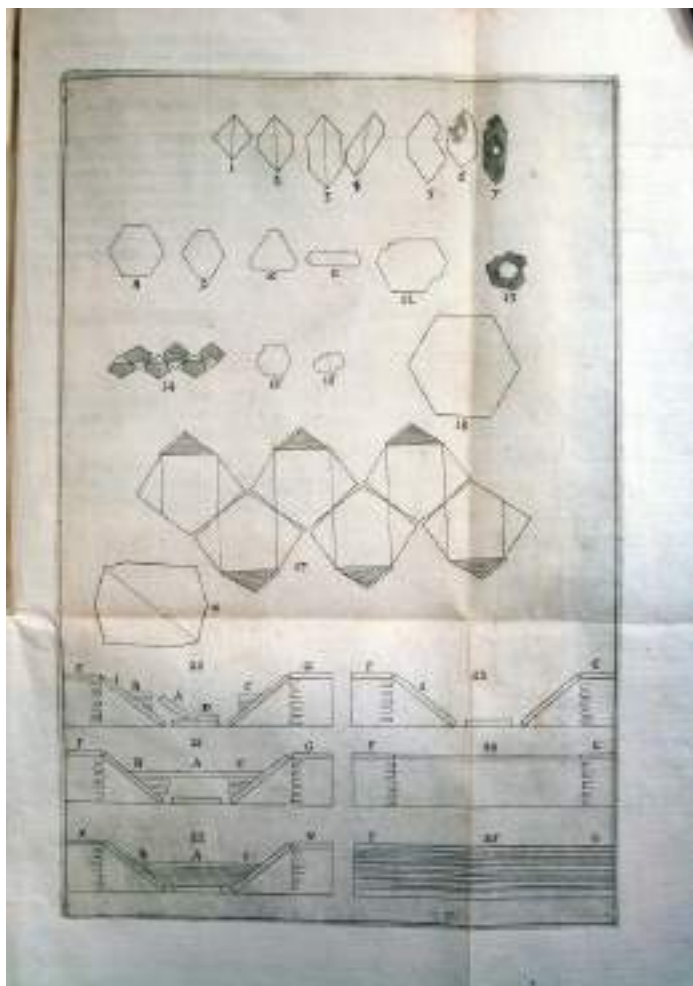
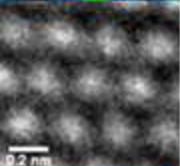
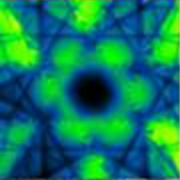
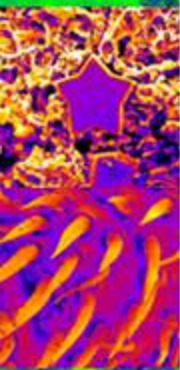
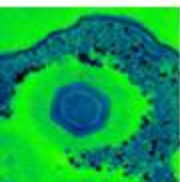
# Nicolau Steno (Niels Stensen)

1638 – 1686

- médico, naturalista e teólogo
- estudo sobre a constância dos ângulos entre as faces dos cristais
- contribuições fundamentais para a paleontologia e para a estratigrafia
- grande anatomista, descobriu as glândulas lacrimais (*glândulas de Steno*)



# Lei da Constância dos Ângulos por Nicolas Steno (Niels Stensen)



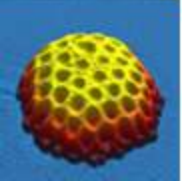
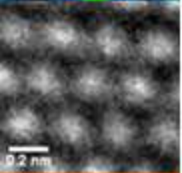
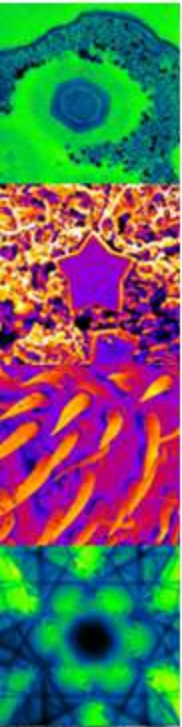
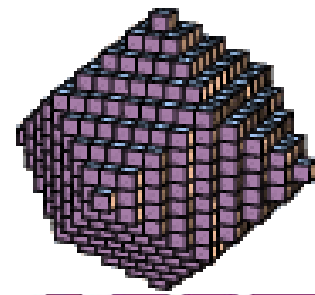
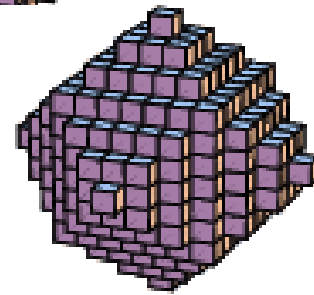
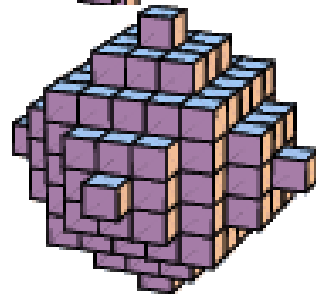
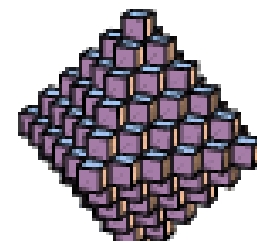
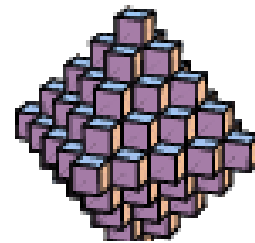
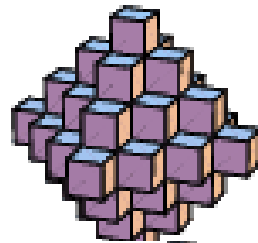
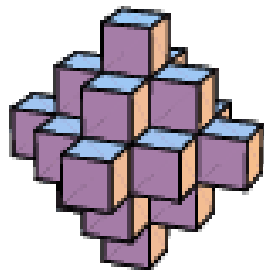
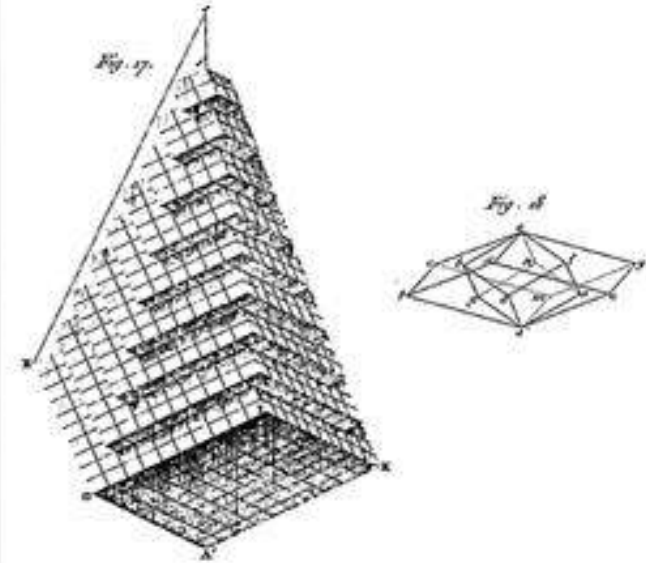
“Início da Cristalografia como  
Ciência”:

Descoberta da lei da Constância dos Ângulos por Nicolas Steno (Niels Stensen) em 1669, observado em cristais de quartzo. Apesar dos diversos espécimes de cristais de quartzo não serem perfeitamente iguais quanto ao tamanho e contorno das faces, o ângulo entre faces equivalentes de vários espécimes era sempre constante.



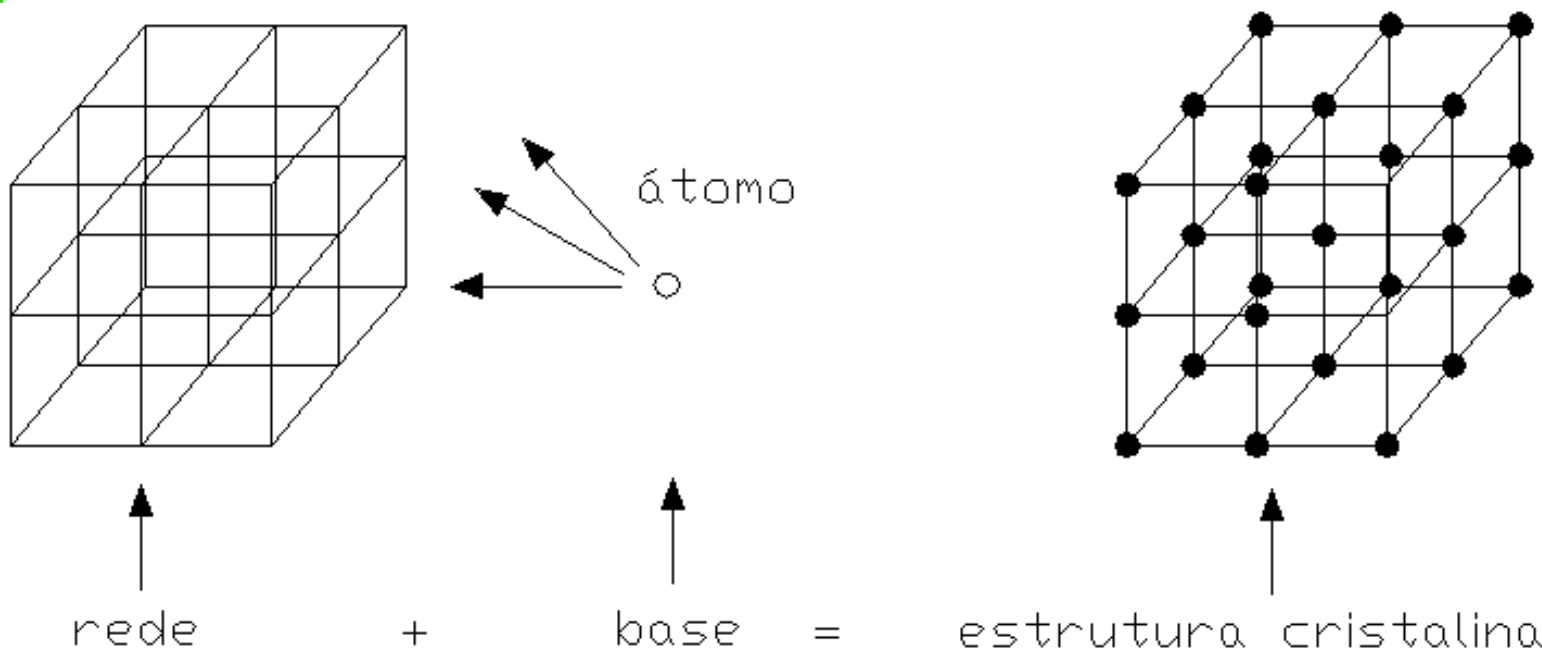
# René J. Haüy - 1784

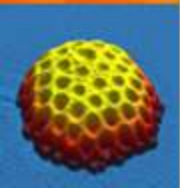
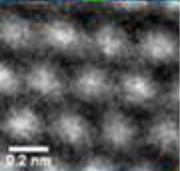
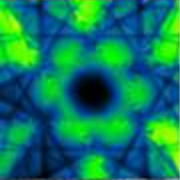
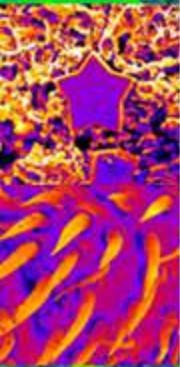
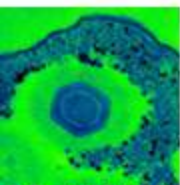
Cristais formados pelo empacotamentos de minúsculos blocos idênticos (conceito precursor das “celas unitárias”)



# Estrutura cristalina

- Átomos estão dispostos em posições regulares no espaço.
- Descrição: rede + base
  - Rede = estrutura geométrica
  - Base = distribuição dos átomos ou conjunto de átomos em cada ponto da rede.





# Geometria

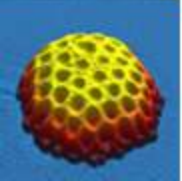
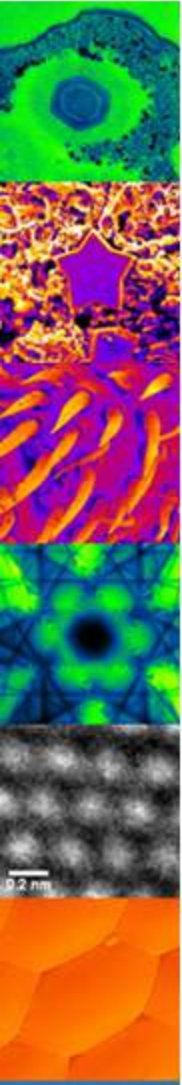
## Teoria de grupo

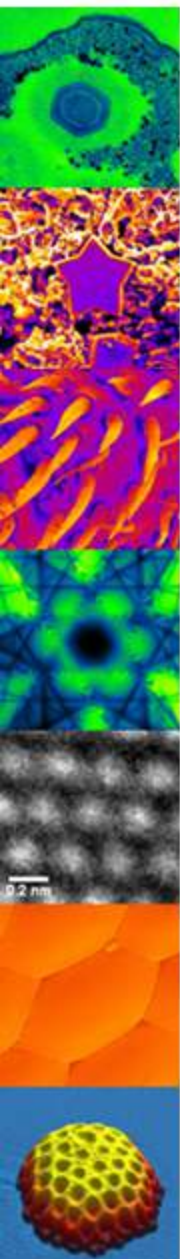
- Padrões
- Simetria
- Regras
- Definições

Livro Escher



# Tessellation - tilings





Existe um número finito de organizar os “átomos” ou “padrões” de tal forma que se preenche todo o plano ou o espaço.

## 1 - DIMENSÃO

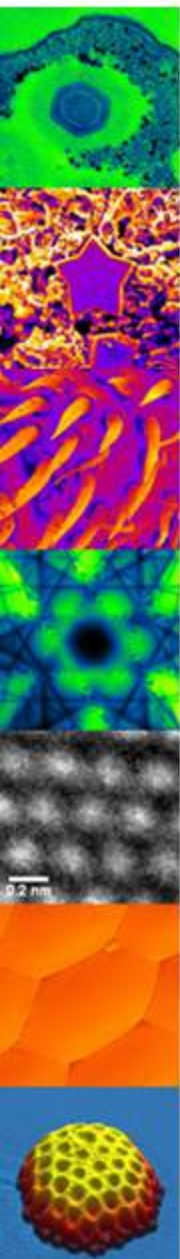
- 7 grupos de espaço

## 3 - DIMENSÕES

- 7 sistemas cristalinos
- 14 redes de Bravais
- 32 grupos de ponto
- 230 grupos de espaço

## 2 - DIMENSÕES

- 4 sistemas cristalinos
- 5 redes de Bravais
- 10 grupos de ponto
- 17 grupos de espaço



Uma rede é definido por 3 vetores tal que:

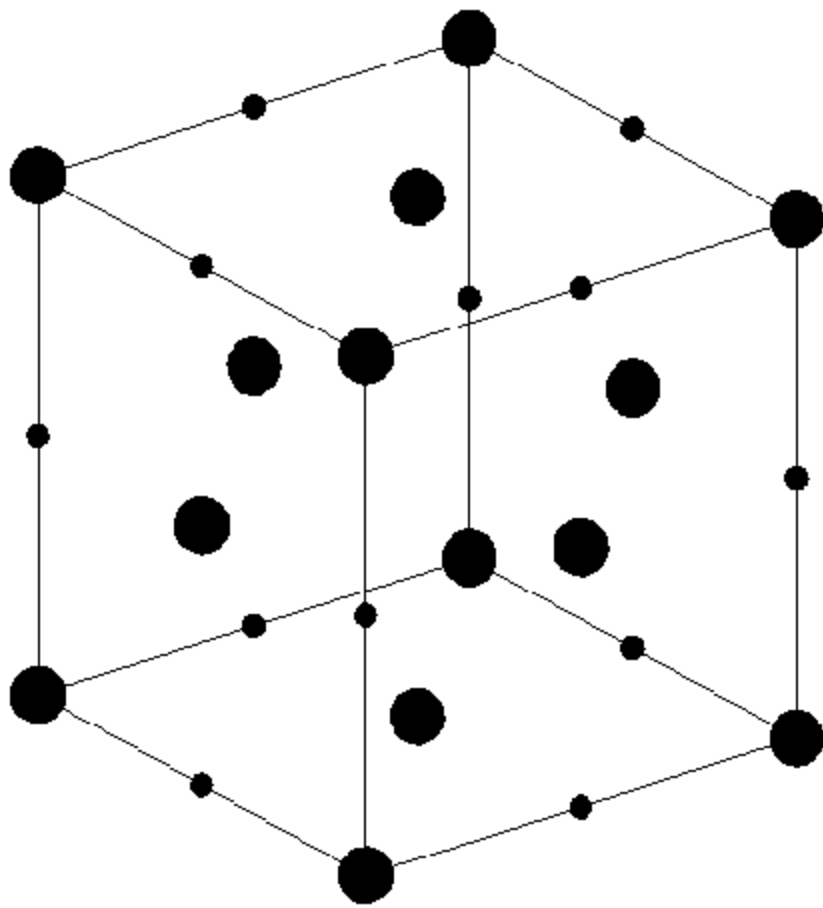
$$\mathbf{r}' = \mathbf{r} + u\mathbf{a} + v\mathbf{b} + w\mathbf{c}$$

onde,  $u, v, w =$  inteiros

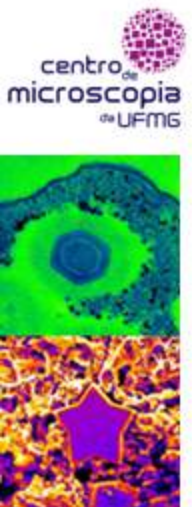
Ponto  $\mathbf{r}'$  é idêntico ao  
ponto  $\mathbf{r}$ .

Ex. Rede cúbica de face  
centrada com 2 átomos  
por ponto de rede.

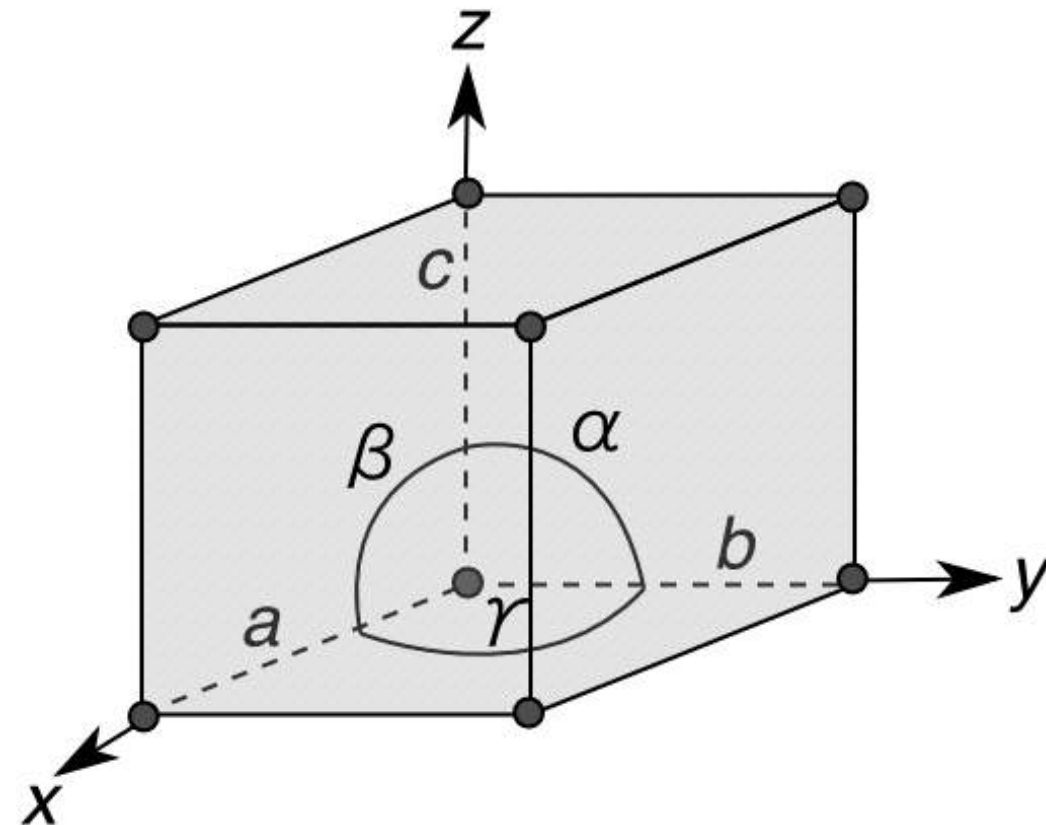
(cristal de NaCl)







# Definição dos parâmetros de rede

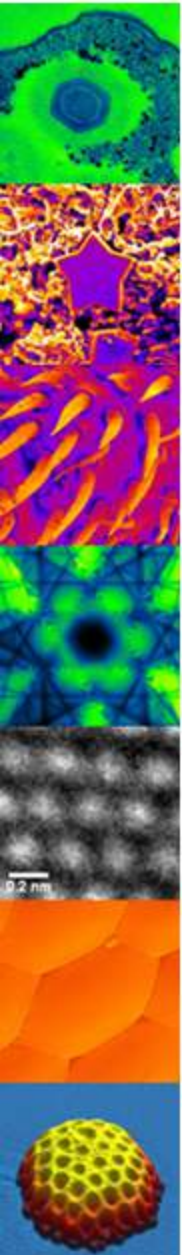


$$b \wedge c = \alpha$$

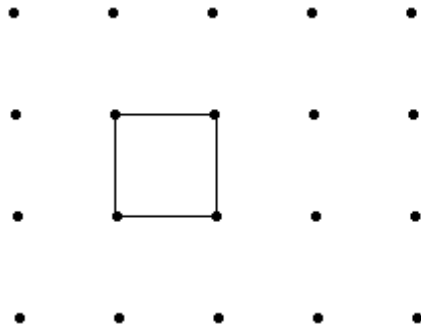
$$a \wedge c = \beta$$

$$a \wedge b = \gamma$$

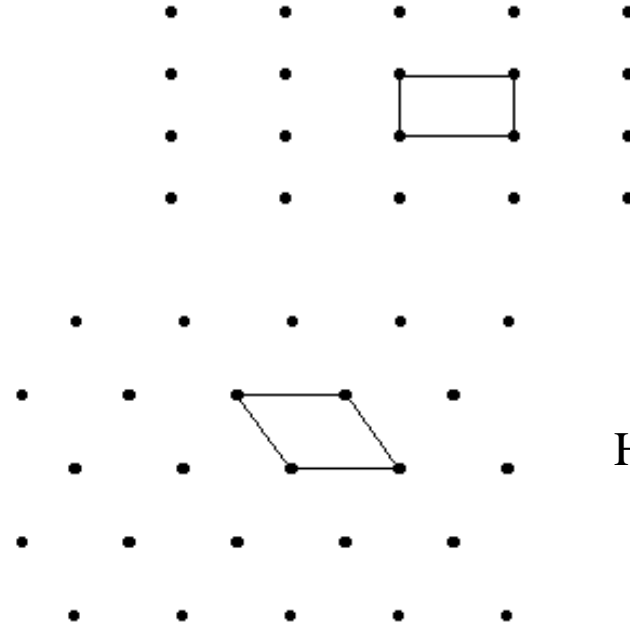
# Redes em 2 dimensões



Quadrada

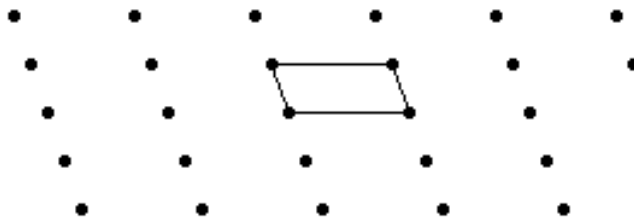


Retangular

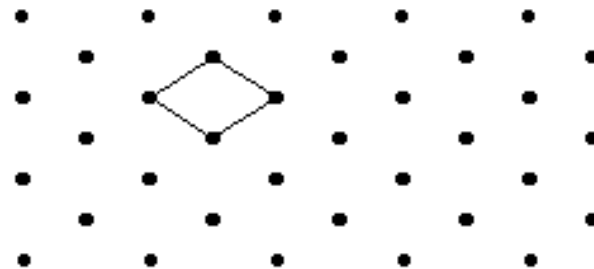


Hexagonal

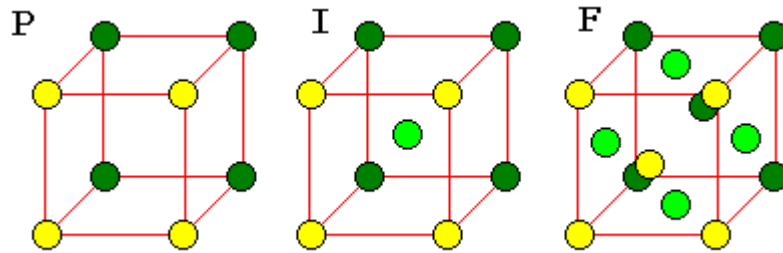
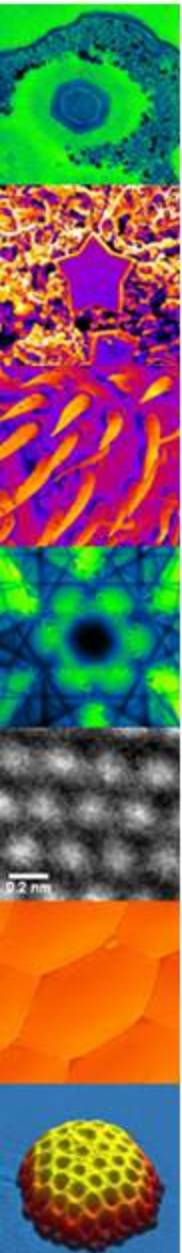
Paralelogramo



Romboedral

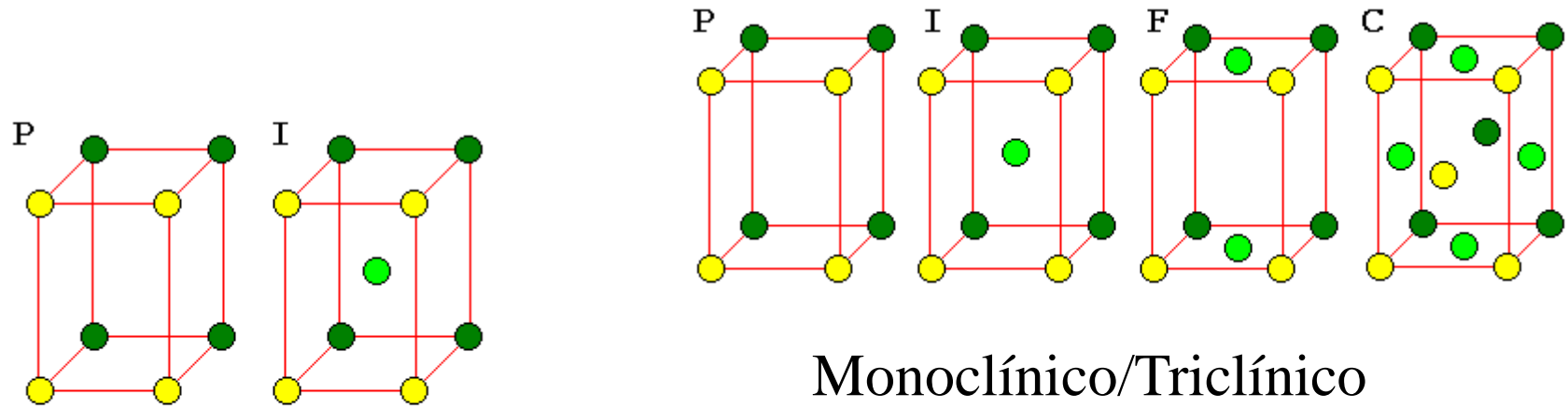


# Redes em 3-dimensões - 14 Redes de Bravais

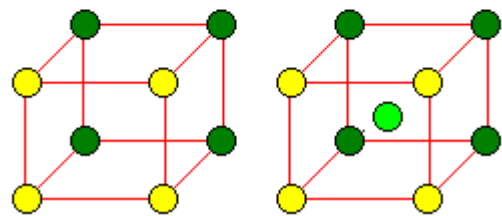


Cúbico

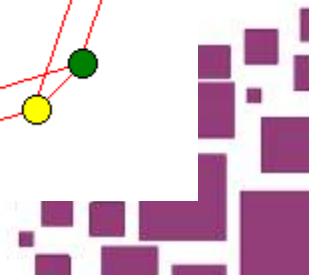
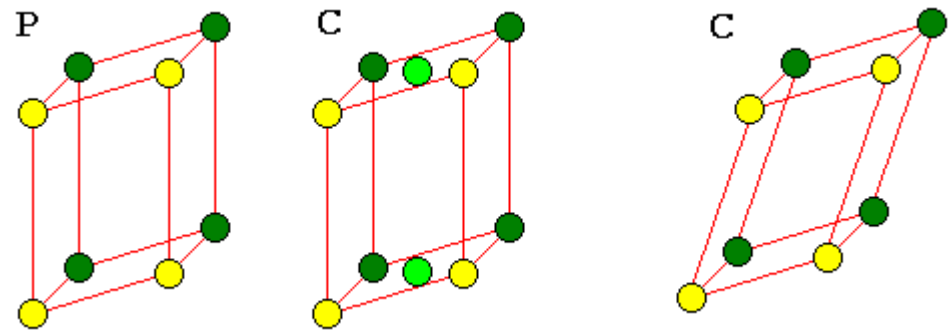
Orthorômbico



Monoclínico/Triclínico



Tetragonal

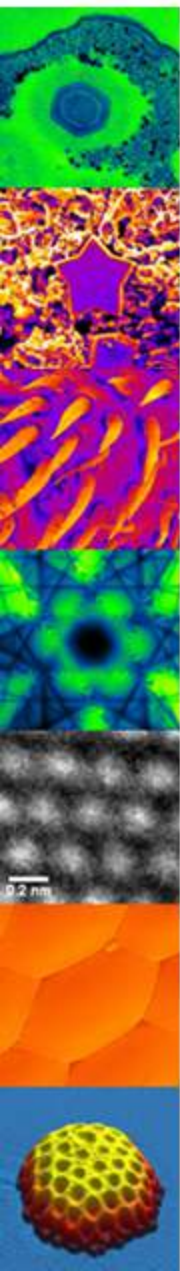


# Simetria

Simetria: é a propriedade decorrente da repetição ordenada das partes de um todo.

A simetria pode ser descrita através dos padrões de repetição ordenada das partes equivalentes: o mecanismo responsável por esta repetição se denomina operador de simetria.

Os operadores de simetria observados em substâncias cristalinas podem ser: translação, rotação, inversão, reflexão e (glide) deslizamento que podem ocorrer combinados.

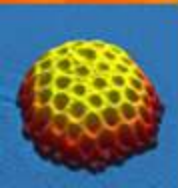
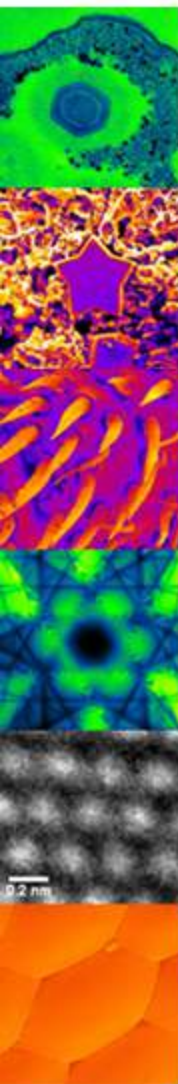


# Operações de Simetria

- 1) **Translação**  $\rho(\mathbf{r}) = \rho(\mathbf{r} + u.\mathbf{a} + v.\mathbf{b} + w.\mathbf{c})$ ;
- 2) **Rotação**: eixos de simetria ( $E_n$  - em inglês,  $A_n$ , de *axis*);
- 3) **Reflexão ou espelho**: planos de simetria ( $m$  – de *mirror*, espelho);
- 4) **Inversão ou centro de inversão**: centro de simetria ( $i$  – de inversão ou  $-1$ );
- 5) Rotoinversão ou combinação de rotação com inversão: ( $-3$ ,  $-4$ ,  $-6$ ). A operação  $-2$  é equivalente à reflexão, logo não é uma simetria nova.
- 6) Parafuso: combinação de rotação com translação (screw axis) de ordem 2 ( $2_1$ ), ordem 3 ( $3_1$  e  $3_2$ ), ordem 4 ( $4_1$ ,  $4_2$ ,  $4_3$ ) e de ordem 6 ( $6_1$ ,  $6_2$ ,  $6_3$ ,  $6_4$ ,  $6_5$ )
- 7) Combinação de rotação com reflexão de ordem 2 ( $2/m$ ), ordem 4 ( $4/m$ ), ordem 6 ( $6/m$ ).
- 8) Combinação de um eixo parafuso (screw axis) com reflexão: ordem 2 ( $2_1/m$ ), ordem 4 ( $4_2/m$ ) e ordem 6 ( $6_3/m$ ).
- 9) Plano de deslizamento: combinação de uma reflexão com translação:  $a, b, c, n, e, d$

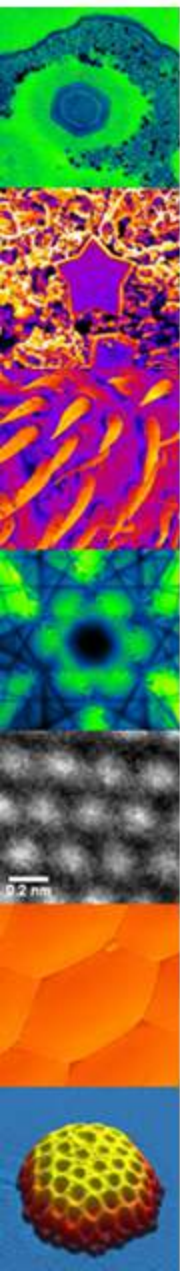
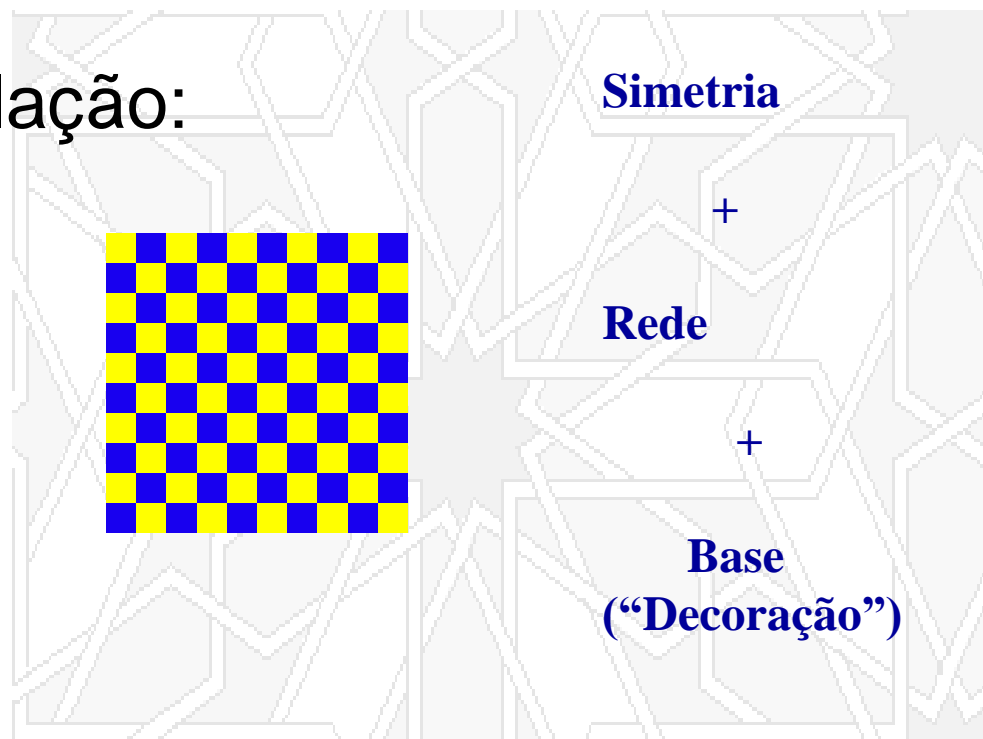
Os elementos de simetria podem ser:

- a) próprios (objetos diretos geram apenas objetos diretos, e vice-versa) – eixos de simetria simples;
- b) impróprios (objetos diretos geram objetos esquerdos, e vice-versa) – planos, centro de simetria e eixos de roto-inversão

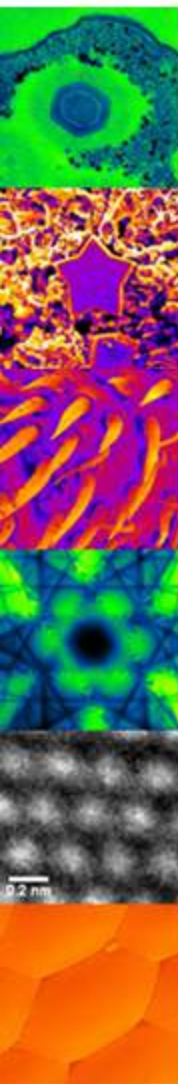


# CRISTALOGRAFIA

- “DEFINIÇÃO CLÁSSICA”
- Ordem de longo alcance
- Ordenamento periódico dos átomos em 3-dimensões
- Simetria de translação:  
 $R = ua + vb + wc$



# 3D



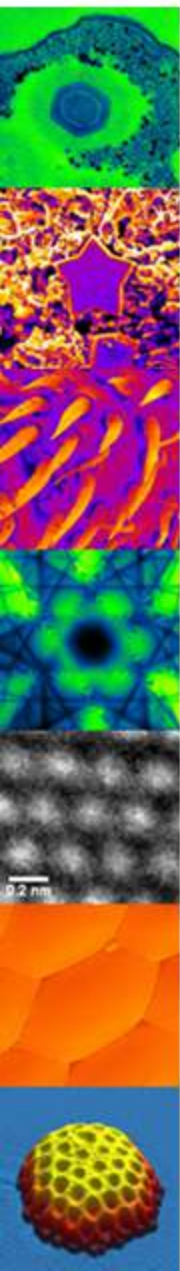
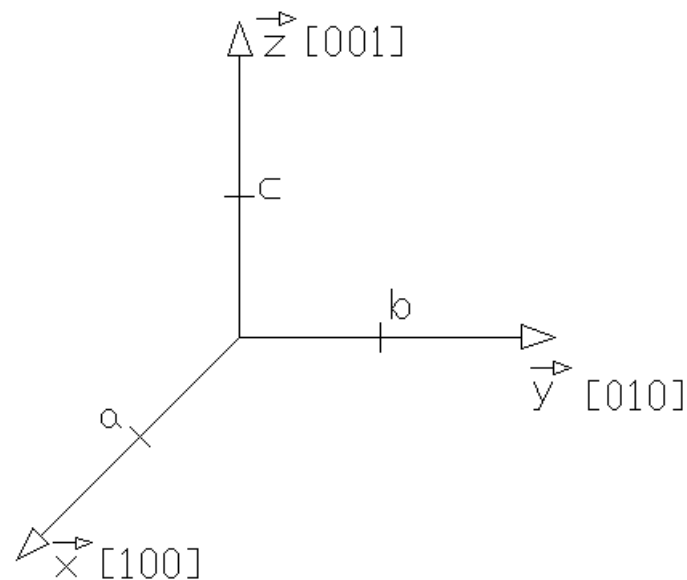
Crystal family (6)	Crystal system (7)	Required symmetries of point group	Point groups	Space groups	Bravais lattices	Lattice system
	Triclinic	None	2	2	1	Triclinic
	monoclinic	1 twofold axis of rotation or 1 mirror plane	3	13	2	monoclinic
	Orthorhombic	3 twofold axes of rotation or 1 twofold axis of rotation and 2 mirror planes.	3	59	4	Orthorhombic
	Tetragonal	1 fourfold axis of rotation	7	68	2	Tetragonal
Hexagonal	Trigonal	1 threefold axis of rotation	5	7	1	Rhombohedral
	Hexagonal	1 sixfold axis of rotation		18		Hexagonal
	Cubic	4 threefold axes of rotation	7	27	3	Cubic
6	7	<b>Total</b>	32	230	14	7



# Direções cristalográficas [l,m,n]:

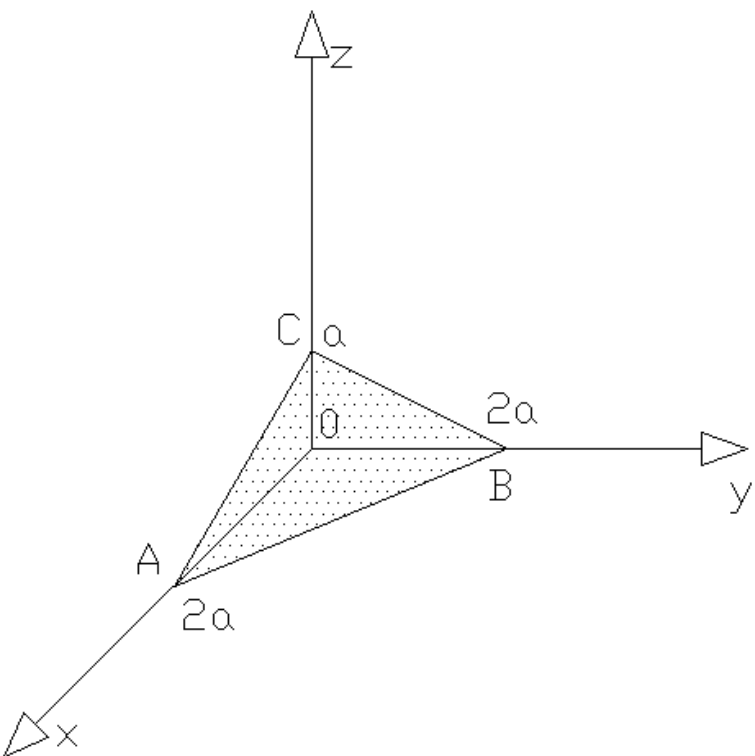
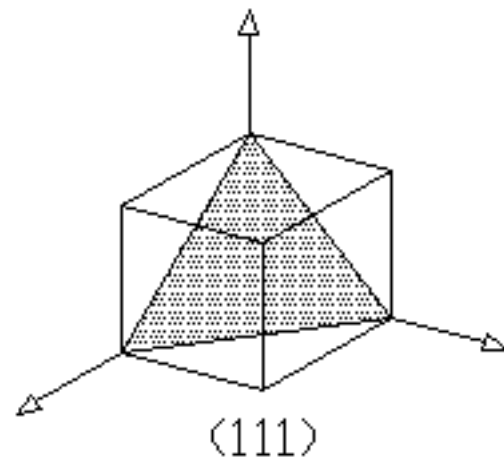
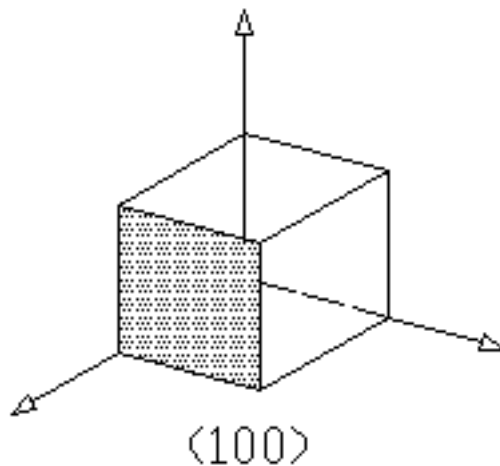
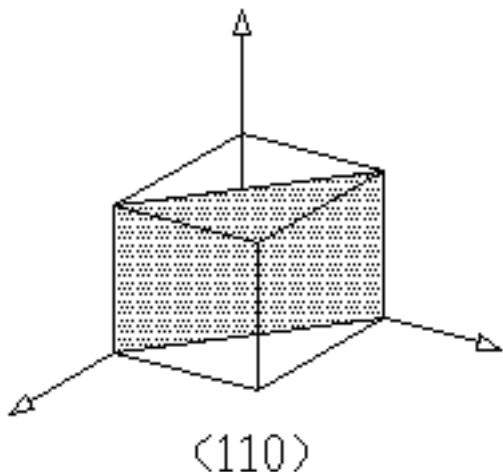
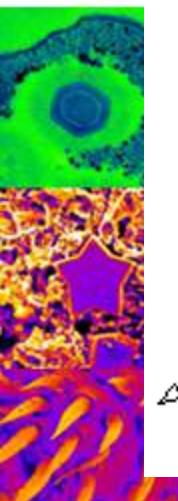
- São expressos por 3 n<sup>os</sup> inteiros com a mesma relação de um vetor naquela direção.

- Os componentes do vetor são dados como múltiplos dos vetores de base.
- A direção da diagonal em sistema tipo paralelepípedo tem as componentes 1a, 1b, 1c, ou seja: [111]
- Em cristal cúbico, a direção [l,m,n] é perpendicular ao plano (l,m,n). Ex. [100] é perpendicular ao plano (100)





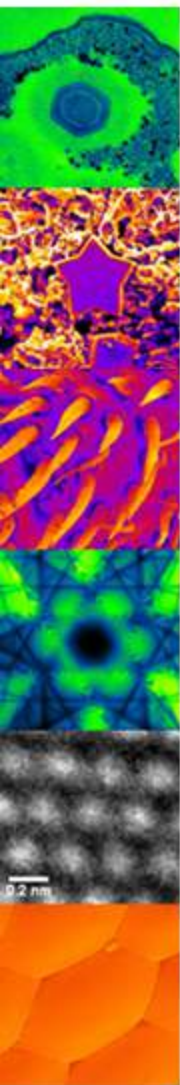
# Planos e Direções Cristalográficas



Índices de Miller:

- Distâncias das intersecções
  - Tomar inversos dos valores
  - Reduzir os resultados a números inteiros com a mesma relação entre si
- Ex.:  $2 \times \frac{1}{2} = 1$ ;  $2 \times \frac{1}{2} = 1$ ;  $2 \times 1 = 2$   
 $\Rightarrow$  plano (1,1,2) ou (112)

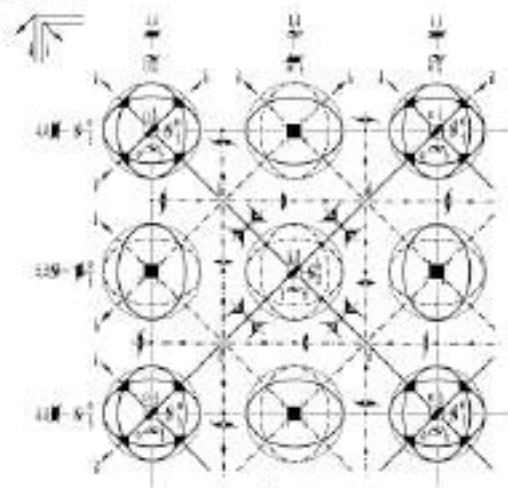
# Grupo de espaço (227) $Fd\bar{3}m$ (silício, alumínio, etc)



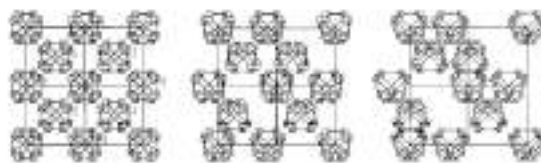
International Tables for Crystallography (2006), Vol. A, Space group 227, pp. 696-701.

$Fd\bar{3}m$   $O_h^f$   $m\bar{3}m$  Cubic  
No. 227  $F, 4, /d \bar{3} 2/m$  Full icosahedral symmetry  $Fm\bar{3}m$

ORIGIN CHOICE 1



Upper half of unit cell only



Origin at  $F$  in  $(x, y, z) = (0, 0, 0)$  from center  $O$   
Asymmetric unit  $0 \leq x < 0.5, 0 \leq y < 0.5, -0.5 \leq z < 0.5$   
Volume  $0.00, 1.00, 1.00, 1.00, 1.00, 1.00, 1.00, 1.00$   
Symmetry operations  
(0.00.00 page 699)

CONTINUED No. 227  $Fd\bar{3}m$

Generators identical  $(0, 0, 0, 0, 0, 0), (0, 0, 0, 1, 0, 0), (0, 0, 1, 0, 0, 0), (0, 1, 0, 0, 0, 0), (0, 0, 0, 0, 1, 0), (0, 0, 0, 0, 0, 1)$

Position	Coordinates	Full icosahedral	
Multiplicity	Wyckoff letter	Site symmetry	
192	$4f$	$(0, 0, 0, 0, 0, 0)$	$O_h$
192	$4c$	$(0, 0, 0, 1/2, 0, 0)$	$O_h$
192	$4b$	$(0, 0, 1/2, 0, 0, 0)$	$O_h$
192	$4a$	$(0, 1/2, 0, 0, 0, 0)$	$O_h$

Special position	Coordinates	Site symmetry	
192	$4f$	$(0, 0, 0, 0, 0, 0)$	$O_h$
192	$4c$	$(0, 0, 0, 1/2, 0, 0)$	$O_h$
192	$4b$	$(0, 0, 1/2, 0, 0, 0)$	$O_h$
192	$4a$	$(0, 1/2, 0, 0, 0, 0)$	$O_h$

Special position	Coordinates	Site symmetry	
192	$4f$	$(0, 0, 0, 0, 0, 0)$	$O_h$
192	$4c$	$(0, 0, 0, 1/2, 0, 0)$	$O_h$
192	$4b$	$(0, 0, 1/2, 0, 0, 0)$	$O_h$
192	$4a$	$(0, 1/2, 0, 0, 0, 0)$	$O_h$

Special position	Coordinates	Site symmetry	
192	$4f$	$(0, 0, 0, 0, 0, 0)$	$O_h$
192	$4c$	$(0, 0, 0, 1/2, 0, 0)$	$O_h$
192	$4b$	$(0, 0, 1/2, 0, 0, 0)$	$O_h$
192	$4a$	$(0, 1/2, 0, 0, 0, 0)$	$O_h$

Symmetry of special projections  
Along [001] plane  $C_{4v}$   
Along [111] plane  $C_{3v}$   
Along [110] plane  $C_{2v}$

# Crystallographic Information File (CIF) - silício

file|Users/pierrestadelmann/Desktop/jemsNetBeans/  
jemsData/Cubic/Si.txt

name|Si

creator|pierrestadelmann

date|Tue Sep 27 16:06:06 CEST 2011

system|cubic

superCell|false

HMSymbol|227|48|0|1|0| F d 3 m

rps|0|x, y, z

rps|1|z, x, y

rps|2|y, z, x

rps|3|x, z, y

rps|4|y, x, z

rps|5|z, y, x

rps|6|x, -y, -z

rps|7|z, -x, -y

rps|8|y, -z, -x

rps|9|x, -z, -y

rps|10|y, -x, -z

rps|11|z, -y, -x

rps|12|-x, y, -z

rps|13|-z, x, -y

rps|14|-y, z, -x

rps|15|-x, z, -y

rps|16|-y, x, -z

rps|17|-z, y, -x

rps|18|-x, -y, z

rps|19|-z, -x, y

rps|20|-y, -z, x

rps|21|-x, -z, y

rps|22|-y, -x, z

rps|23|-z, -y, x

rps|24|1/4 - x, 1/4 - y, 1/4 - z

rps|25|1/4 - z, 1/4 - x, 1/4 - y

rps|26|1/4 - y, 1/4 - z, 1/4 - x

rps|27|1/4 - x, 1/4 + y, 1/4 + z

rps|28|1/4 - z, 1/4 + x, 1/4 + y

rps|29|1/4 - y, 1/4 + z, 1/4 + x

rps|30|1/4 + x, 1/4 - y, 1/4 + z

rps|31|1/4 + z, 1/4 - x, 1/4 + y

rps|32|1/4 + y, 1/4 - z, 1/4 + x

rps|33|1/4 + x, 1/4 + y, 1/4 - z

rps|34|1/4 + z, 1/4 + x, 1/4 - y

rps|35|1/4 + y, 1/4 + z, 1/4 - x

rps|36|1/4 - x, 1/4 - z, 1/4 - y

rps|37|1/4 - y, 1/4 - x, 1/4 - z

rps|38|1/4 - z, 1/4 - y, 1/4 - x

rps|39|1/4 - x, 1/4 + z, 1/4 + y

rps|40|1/4 - y, 1/4 + x, 1/4 + z

rps|41|1/4 - z, 1/4 + y, 1/4 + x

rps|42|1/4 + x, 1/4 - z, 1/4 + y

rps|43|1/4 + y, 1/4 - x, 1/4 + z

rps|44|1/4 + z, 1/4 - y, 1/4 + x

rps|45|1/4 + x, 1/4 + z, 1/4 - y

rps|46|1/4 + y, 1/4 + x, 1/4 - z

rps|47|1/4 + z, 1/4 + y, 1/4 - x

lattice|0|0.54309

lattice|1|0.54309

lattice|2|0.54309

lattice|3|90.0

lattice|4|90.0

lattice|5|90.0

atom|0|Si,a,0.000,0.000,0.000,0.0049,1.000,0.029,Def,0

aff|0|Si|2.129,57.775,2.533,16.476,0.835,2.88,0.322,0.386|Doyle - Turner Acta Cryst. A24 (1968), 390

aff|0|Si|0.120120145,70.63101,1.0649803,1.0460037,0.1822563,0.086886690.03060761,0.2147628,1.1086769,3.6920595,1.5825809

,9.931198|Earl J. Kirkland, Advanced Computing in Electron Microscopy

nsl|0|Si|0.415

aff|0|Si|0.0567,0.0582,0.3365,0.6155,0.8104,3.2522,2.496,16.7929,2.1186,57.6767|L. Peng et al., Acta Cryst. A52 (1996) 257-276::Def

aff|0|Si|6.2915,2.4386,3.0353,32.3337,1.9891,0.6785,1.541,81.6937,1.1407|XRay::: RHF:::Def

CIF Si-file – JEMS – Pierre Stadelmann

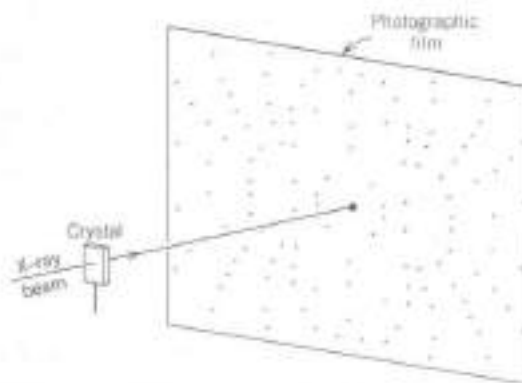
<http://www.jems-saas.ch/>

<http://www.crystallography.net/cod/>

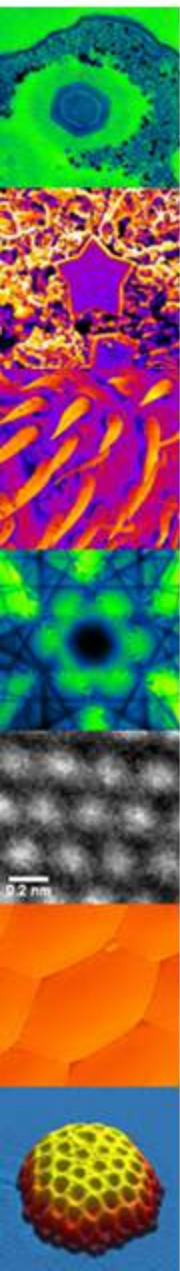
# Cristalografia moderna



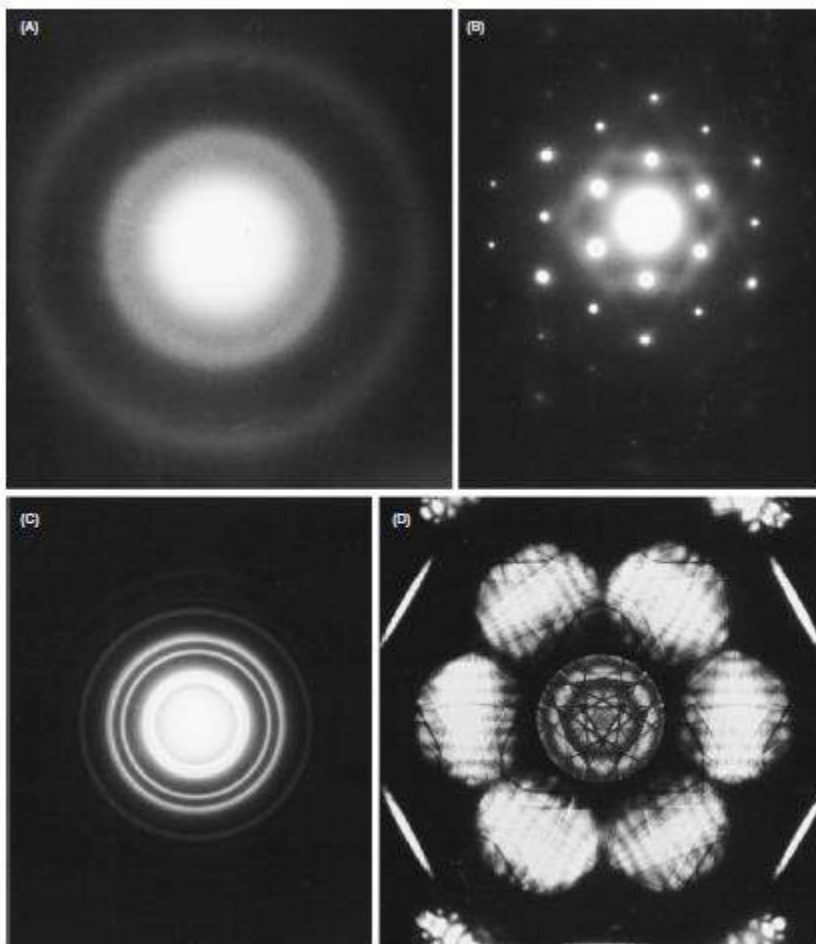
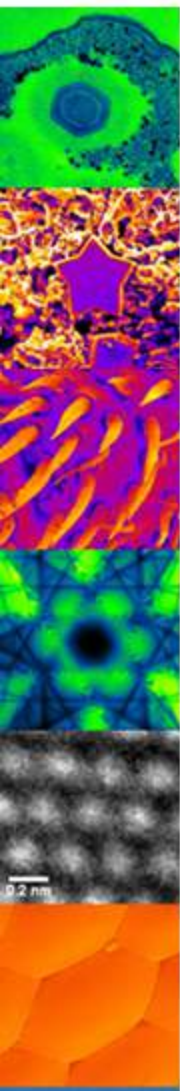
- Wilhelm Roentgen (1895)
- descoberta dos raios X



- Max von Laue
- 1912 - primeiros experimentos de difração de raios X em cristais;
- prêmio Nobel de Física em 1914



# Difração em diversas formas cristalinas



Carbono Amorfo  
Silício monocristalino  
Ouro policristalino  
Silício monocristalino – CBED

FIGURE 2.1.3. Several kinds of DPs obtained from a range of materials in a conventional 100-kV TEM: (A) amorphous carbon, (B) as (A) single crystal, (C) polycrystalline Au, (D) Si illuminated with a convergent beam of electrons. In all cases the direct beam of electrons is responsible for the bright intensity at the center of the pattern and the scattered beams account for the spots or rings that appear around the direct beam.

Transmission Electron Microscopy  
Williams and Carter 2009

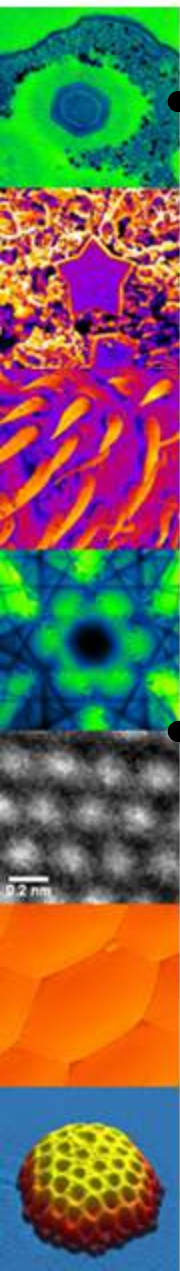
# Difração

## • Difração de Fraunhofer

- espalhamento por ondas planas
  - aproximação longe da amostra
  - plano focal da lente objetiva no TEM
  - elétrons retroespalhados pela amostra

## • Difração de Fresnel

- espalhamento por ondas não planas, por exemplo, esféricas



# Interação radiação amostra

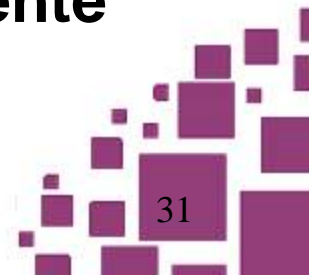
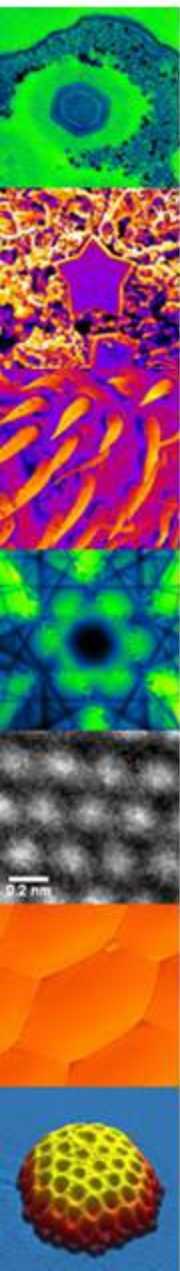
## Raios X

- Interação com a nuvem eletrônica
- Densidade eletrônica

## Elétrons

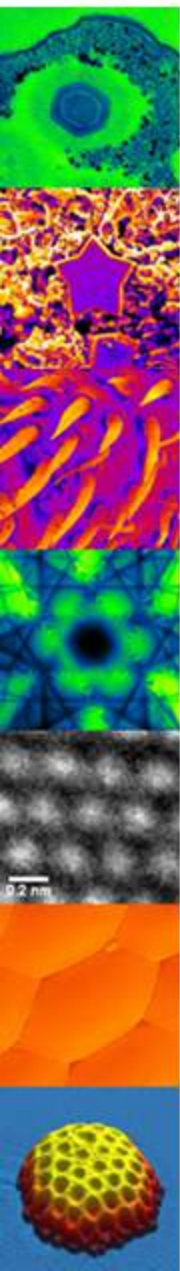
- Interação com a nuvem eletrônica e o núcleo
- Potencial eletrostático

**A intensidade dos “pontos” tem origem diferente**



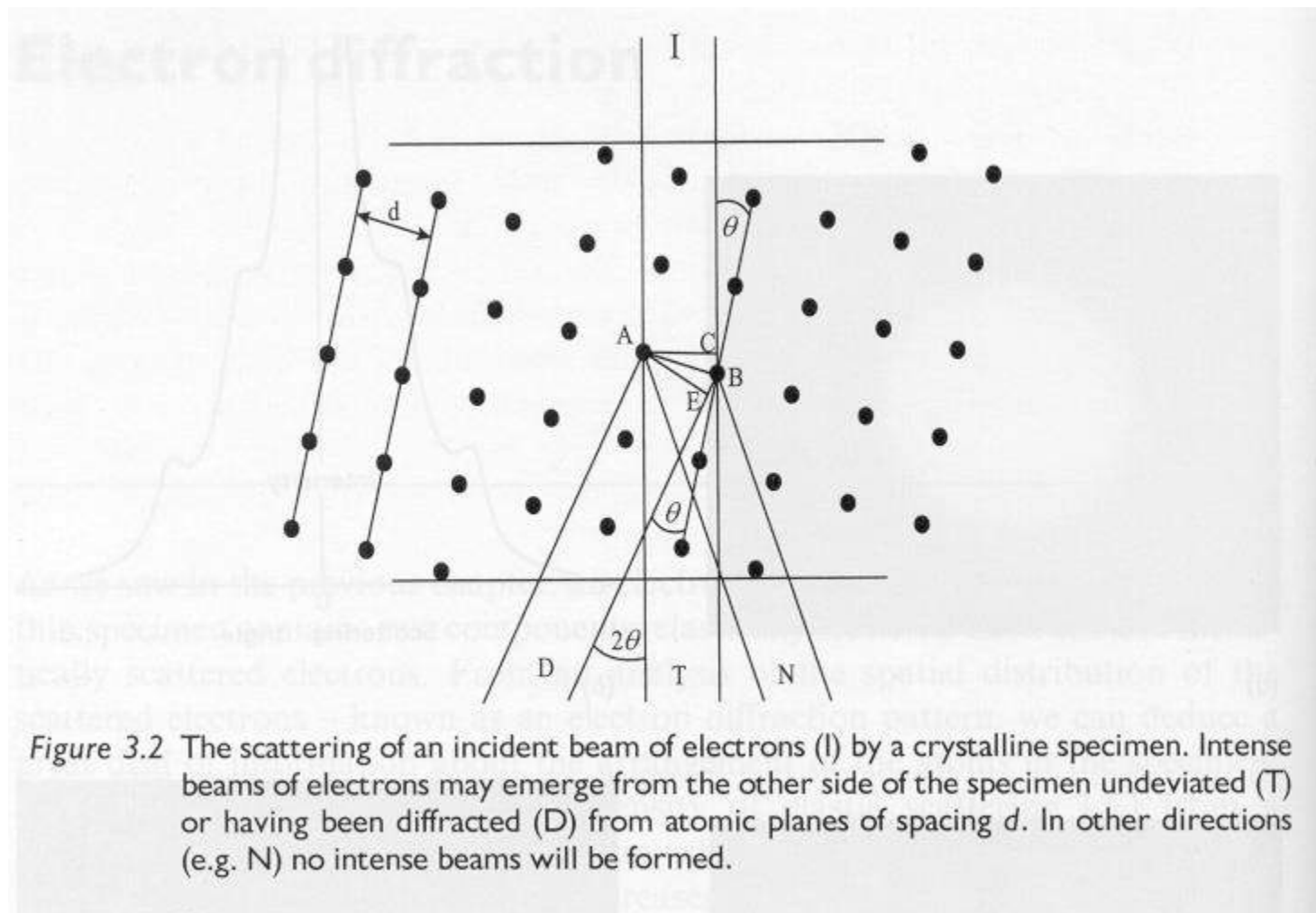
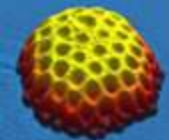
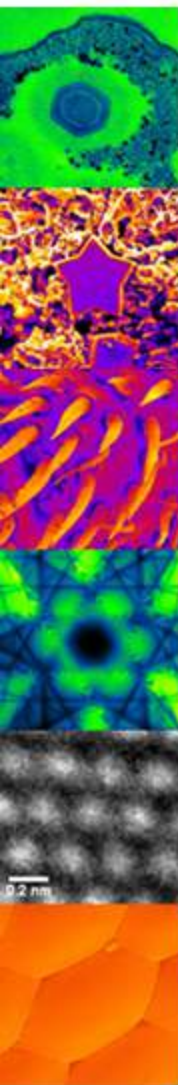
# Difração de elétrons

- Microscopia Eletrônica de Varredura
  - Channeling
  - Difração de elétrons retroespalhados (EBSD)
- Microscopia Eletrônica de Transmissão
  - Difração de área selecionada (SAD)
  - Dark field – bright field
  - Microdifração
  - Difração de feixe convergente (CBED)
  - Difração de feixe convergente de altos ângulos (LACBED)
  - Determinação de estrutura cristalina
    - Precession electron diffraction (PED)
    - Template matching
    - Structure determination
    - Rotation electron diffraction (RED)
    - etc.....

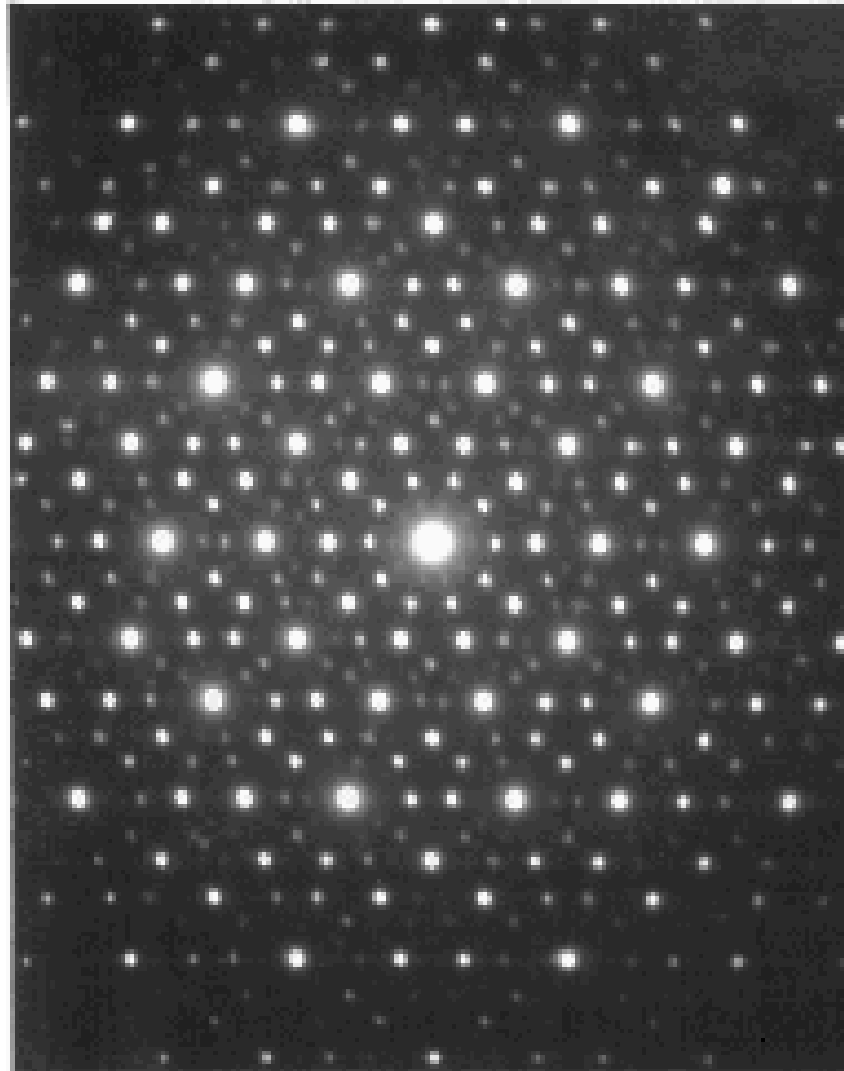
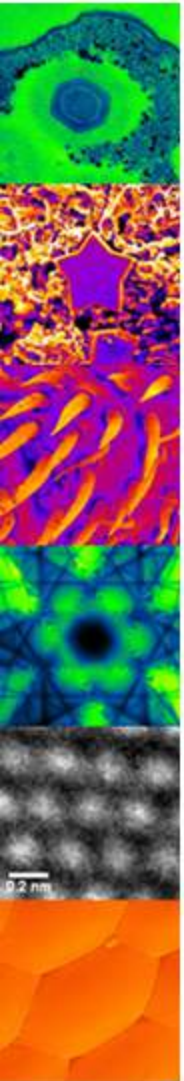




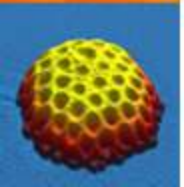
# Difração de elétrons: padrão de difração

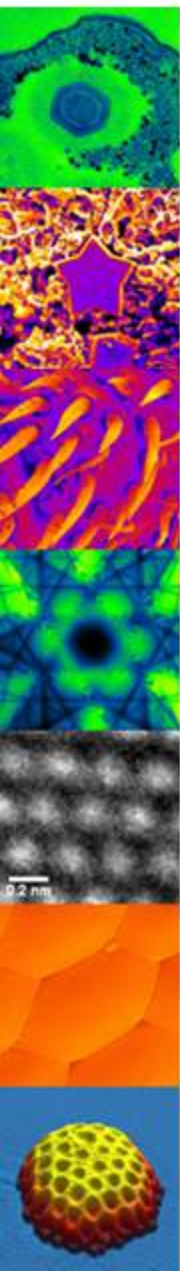


# 1984



Padrão de difração de elétrons de um quasicristal de AlCuFe –  
Karla Balzuweit e Gustaav van Tendeloo 1991





# Difração: microscópio eletrônico e transmissão

## Convergência do feixe

- Paralelo: difração de Fraunhofer
- Convergente
  - Microdifração
  - CBED
  - Determinação de grupos de ponto e de espaço
  - LACBED
  - Estudo de defeitos

## Feixe paralelo: difração de Fraunhofer – matemática de Fourier

- Posição dos “spots”:

- fator de estrutura

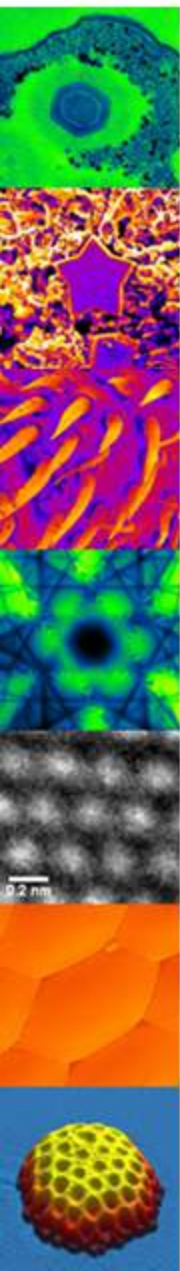
$$F(\theta) = \sum_i f_i e^{2\pi i(hx_i + ky_i + lz_i)}$$

- difração convencional

- Intensidade dos “spots”:

- fator de espalhamento atômico

- Determinação de estrutura

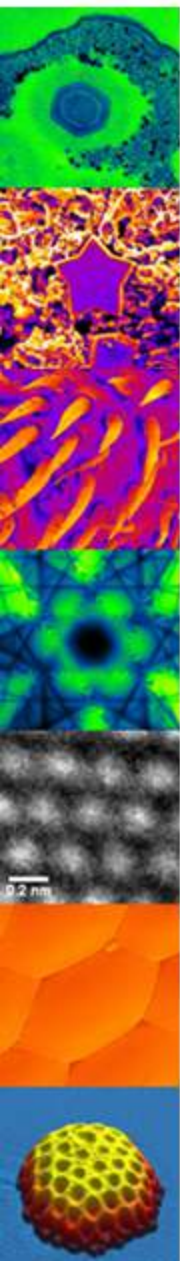


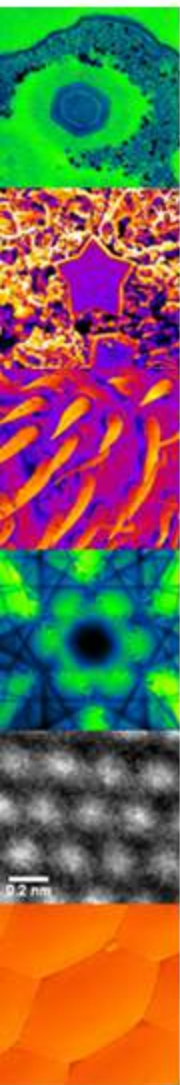
# Interação radiação-amostra

- Conservação de energia mecânica
  - Elásticas
    - não há perda de energia, ou a mesma é desprezível
  - Inelásticas
    - há perda de energia
- Conservação de momento linear e angular
- Colisões de uma única partícula
  
- **Colisões de muitas partículas ou colisões múltiplas (situação na amostra).**

# Espalhamento elástico

- Coerente, se a amostra for fina e cristalina
  - Baixos ângulos de espalhamento ( 1 a 10 graus)
  - Ângulos maiores que 10 graus o espalhamento se torna incoerente
  - Espalhamento inelástico é em geral incoerente, mesmo menor de 10graus.
- 
- Aproximação cinética – espalhamento único
  - Aproximação dinâmica – espalhamento plural ou múltiplo





# Espalhamento múltiplo

Transmission Electron  
Microscopy - L. Reimer -  
Springer Series in Optical  
Sciences vol. 36  
Springer Verlag - 1997

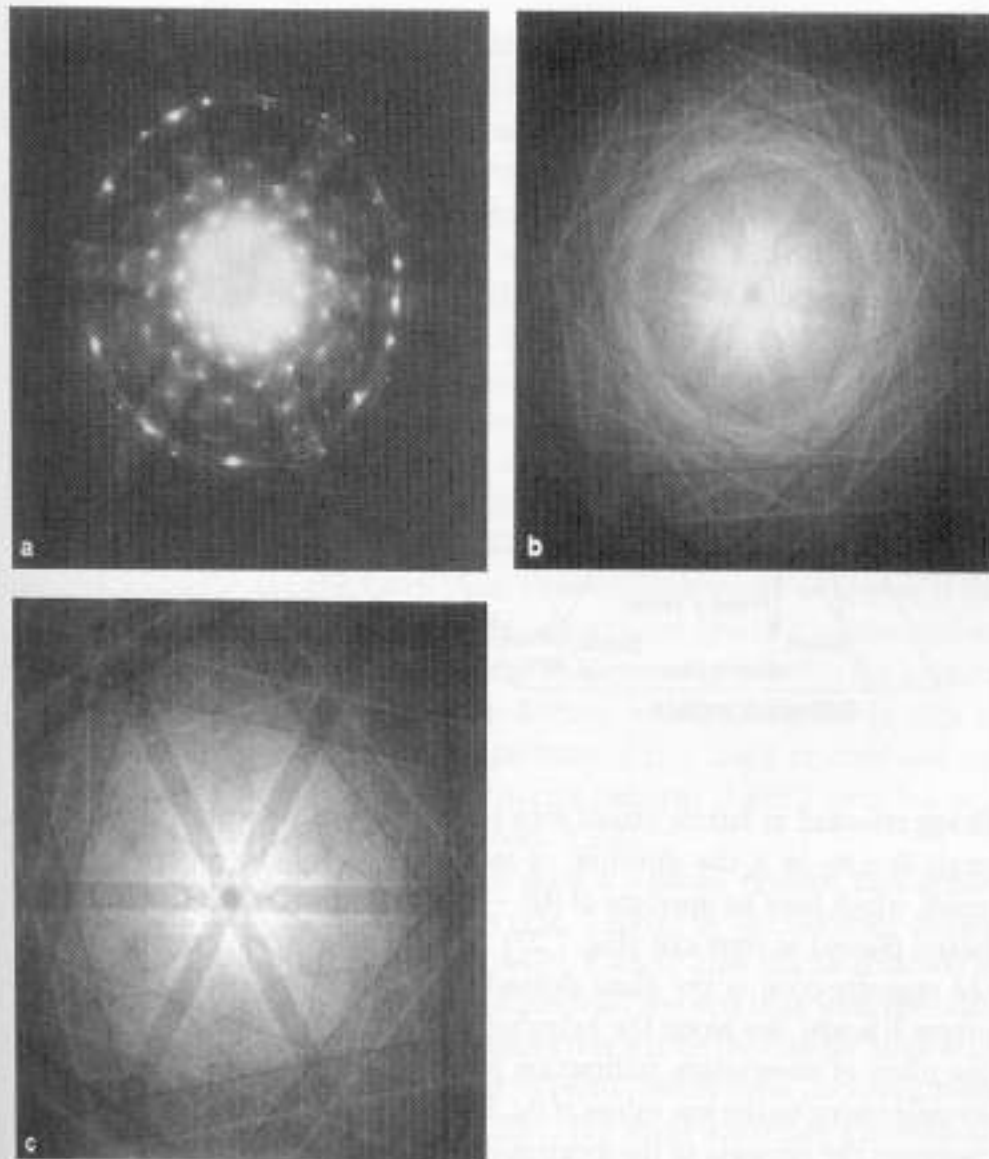
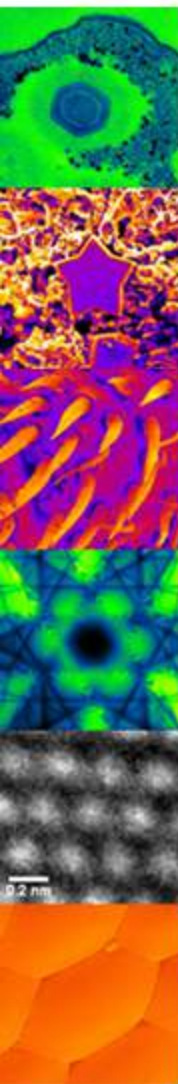
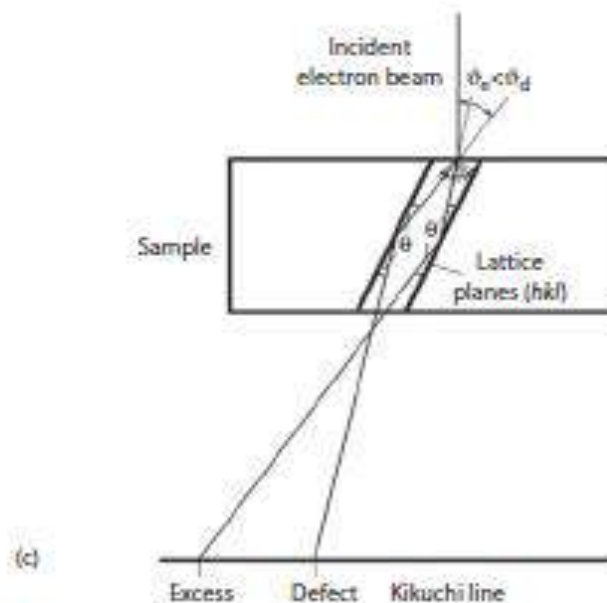
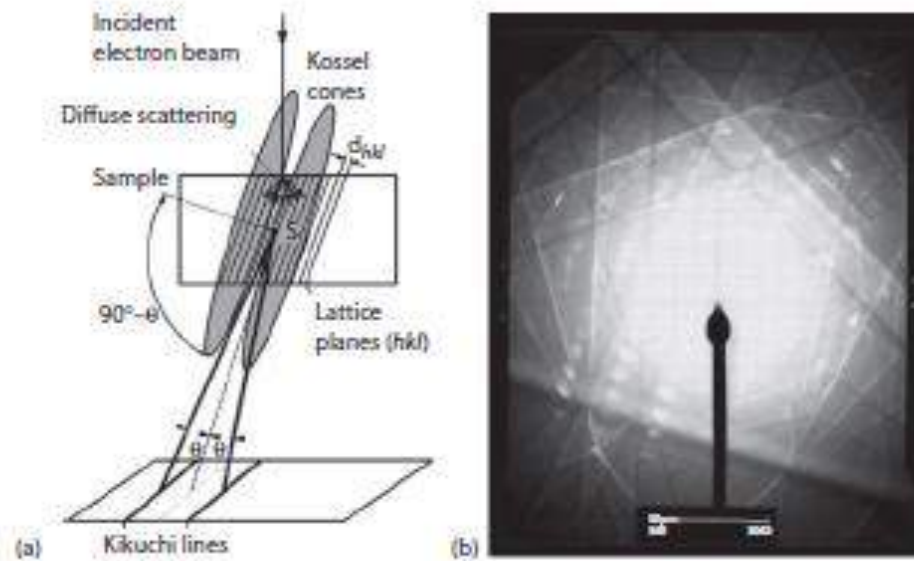


Fig. 7.26. Electron diffraction patterns of Si foils at  $E = 100$  keV with increasing thickness (a)  $t = 80$  nm, (b) 800 nm, (c) 1500 nm with the electron beam parallel to [111]. Pattern (a) shows diffraction spots of the zero- and first-order Laue zones; (b) shows defect and excess Kikuchi lines at medium angles and defect Kikuchi bands at low angles. In (c) the centre shows only Kikuchi bands and the region of excess and defect Kikuchi lines is shifted towards larger angles



# Padrões de Kikuchi em um MET

Introduction to Texture Analysis:  
Macrotecture, Microtexture and  
Orientation Mapping,  
Olaf Engler and Valerie Randle –  
CRC Press - 2010



**FIGURE 6.1**  
Formation of Kikuchi patterns in transmission geometry in TEM. (a) Origin of Kikuchi lines by inelastic scatter of the electrons, giving Bragg diffraction at source  $S$  on lattice planes  $(hkl)$ . (b) Kikuchi pattern from austenitic steel (accelerating voltage 200 kV). (Courtesy of S. Zaeferrer.) (c) Formation of excess and defect Kikuchi lines.

# SAD e linhas de Kikuchi

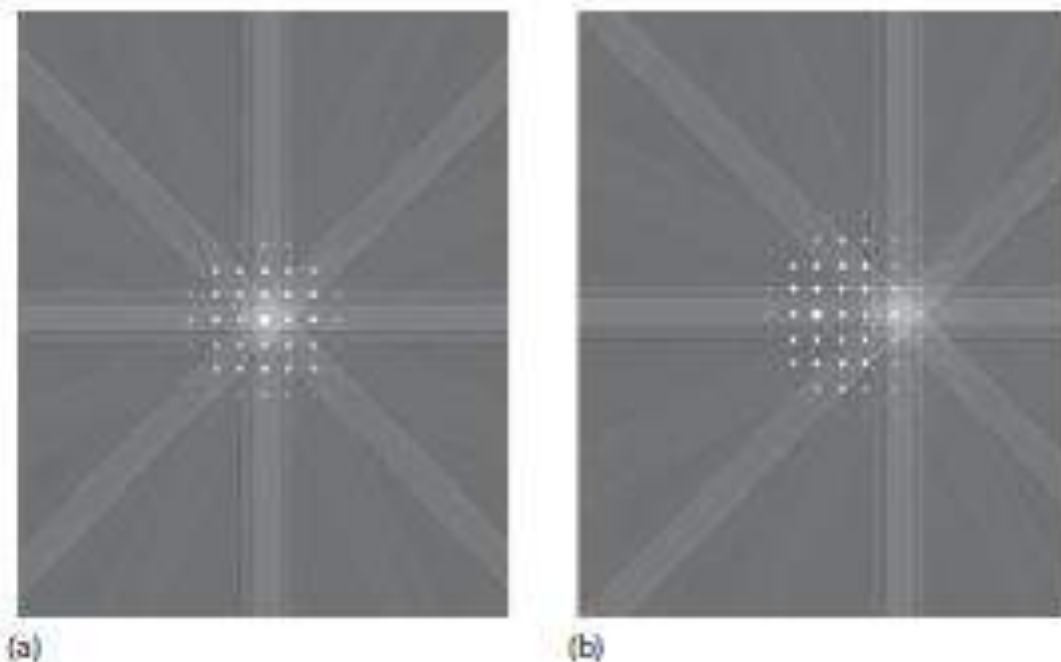
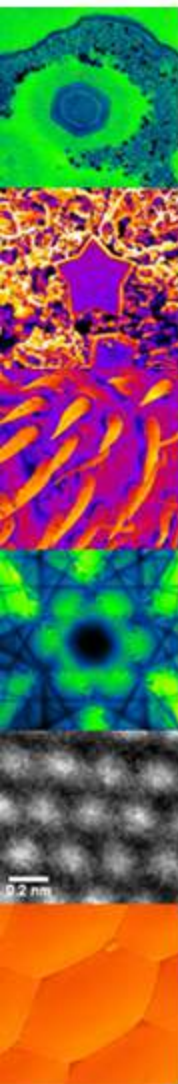


FIGURE 8.6

Simulated diffraction patterns for a  $[100]$  axis showing both SAD spots and Kikuchi lines for (a) untitled, that is, exact  $[100]$  orientation and (b)  $2^\circ$  tilted orientation. These patterns show that Kikuchi lines have much greater sensitivity to crystal orientation than SAD spots (simulation program TOCA by Zaefferer, 2002).





# Padrões de Kikuchi em um MEV

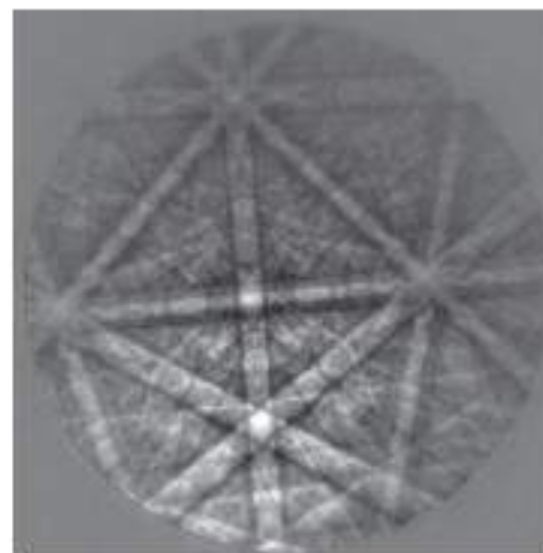
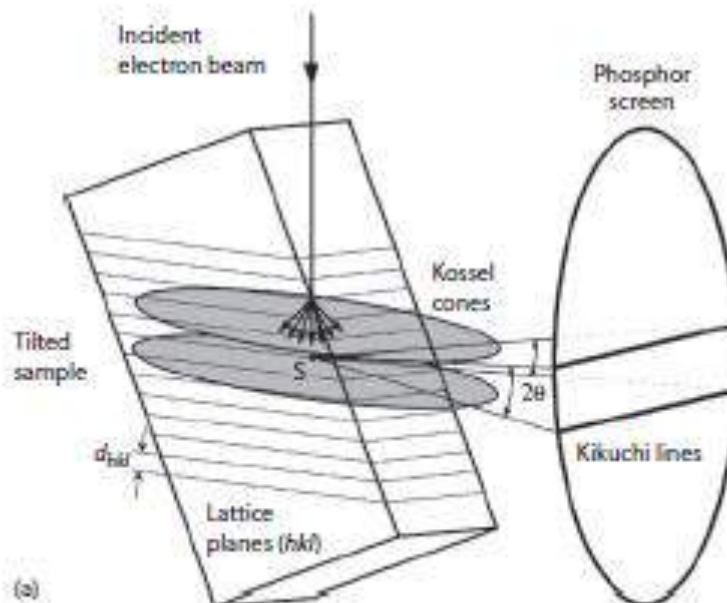
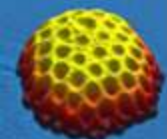


FIGURE 6.2

Formation of backscattered Kikuchi patterns by EBSD in SEM: (a) Origin of Kikuchi lines from the EBSD (i.e., tilted specimen) perspective. (b) EBSD pattern from copper (accelerating voltage 15 kV). (Courtesy of S. Zaeferrer.)

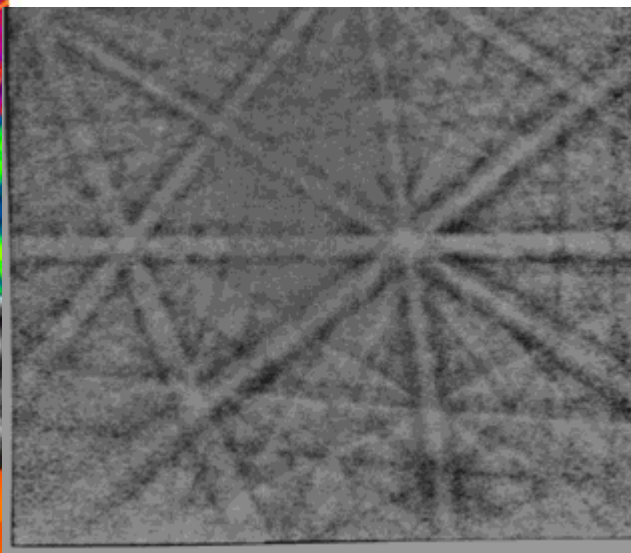
Introduction to Texture Analysis:  
Macrotecture, Microtexture and  
Orientation Mapping,  
Olaf Engler and Valerie Randle –  
CRC Press - 2010



# ELECTRON BACKSCATTER DIFFRACTION - EBSD

## HISTORICAL OVERVIEW

*Kikuchi pattern is a projection of the geometry of the crystal lattice from a volume of specimen. The pattern consists of pairs of band-like parallel lines (Kikuchi bands or lines).*



Kikuchi pattern from the cleavage surface of galena

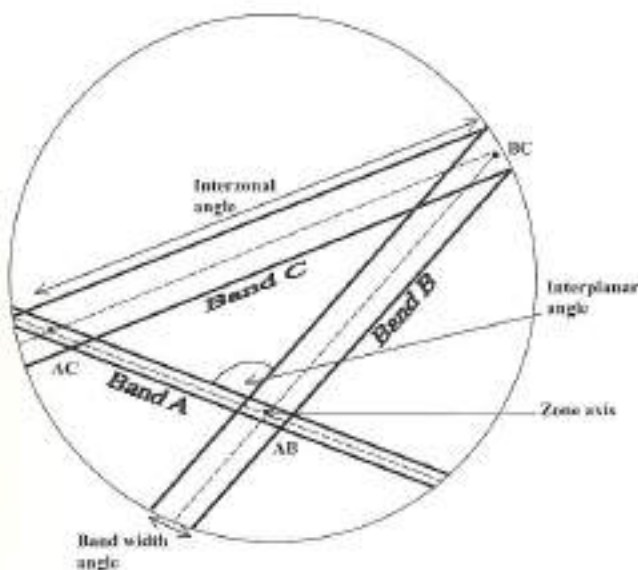
*Magalhães (2003)*

- Similar to “remarkable lines” obtained by Kikuchi (1928 *apud* Coates, 1967).
- Comparable ones were acquired by Alam *et al.* (1954).
- Scanning electron microscopy based observations were reached by Coates (1967): selected area electron channeling patterns (SACP, SAC or ECP).
- Venables & Harland (1972) published the first results achieved by SEM, which were named electron backscatter patterns (EBSPs), although the Backscatter Kikuch Pattern (BKP) was also a known name.

# EBSD

## HISTORICAL OVERVIEW

*Kikuchi pattern is a projection of the geometry of the crystal lattice from a volume of specimen. The pattern consists of pairs of band-like parallel lines (Kikuchi bands or lines).*



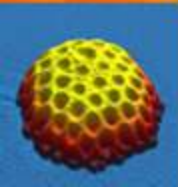
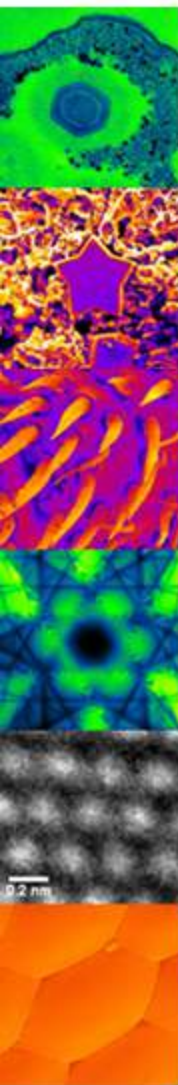
Schematic illustration of the features of Kikuchi pattern

Randle (2003)

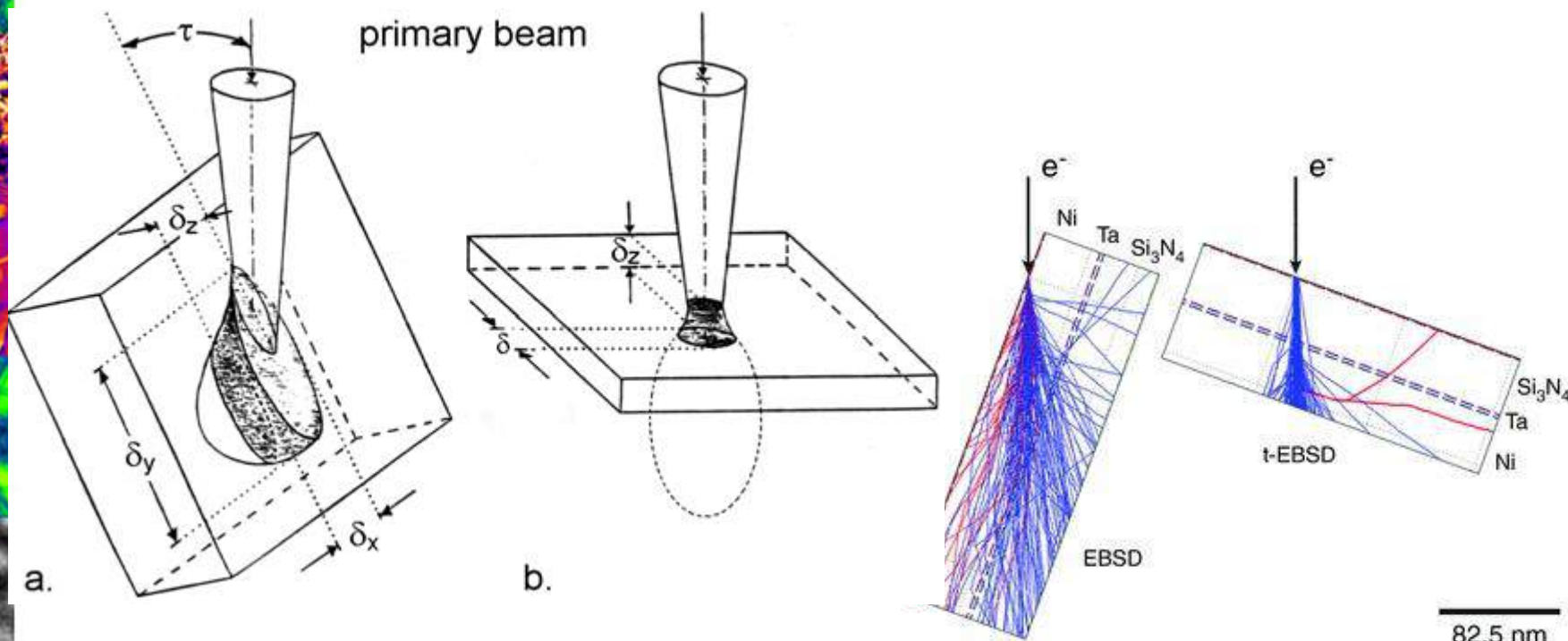
- Great advance with live video imaging and computer assisted indexing (Dingley, 1984).
- 1985: More extensive use of generic computer software — applicable for all crystal systems in theory; however, in practice, it was applied only for cubic and hexagonal systems.
- In the beginning of 1990, fully automatic pattern recognition and indexing was set out.
- The Hough Transform was the procedure suited for shaping the position of Kikuchi bands.
- The full automation led to the mapping of crystallographic orientation, allowing the use of a new term : “*Orientation Image Microscopy*” (Adams *et al.*, 1993).

# SCANNING ELECTRON MICROSCOPY

*FEG – Quanta 200 FEI*



# Volume de interação – “fonte virtual”



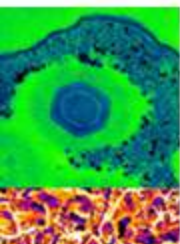
[https://www.ebsd.info/pdf/A99\\_MicrToday\\_16\(2008\)\\_IBP.pdf](https://www.ebsd.info/pdf/A99_MicrToday_16(2008)_IBP.pdf)

<https://www.ebsd.info/TKD-EBSD.htm>

Transmission EBSD from 10 nm domains in a scanning electron microscope (PDF Download Available).

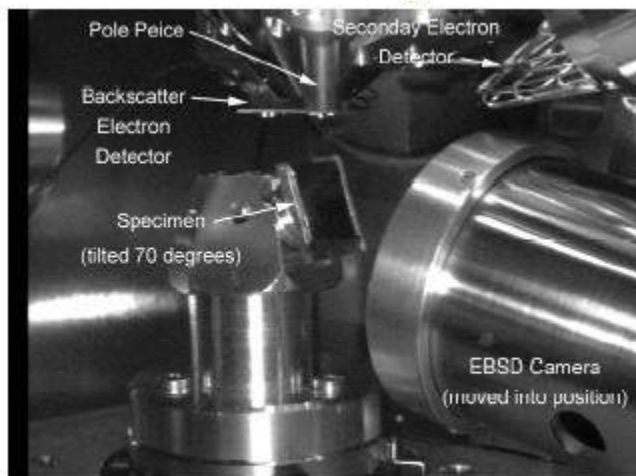
Available from:

[https://www.researchgate.net/publication/51808960\\_Transmission\\_EBSD\\_from\\_10\\_nm\\_domains\\_in\\_a\\_scanning\\_electron\\_microscope](https://www.researchgate.net/publication/51808960_Transmission_EBSD_from_10_nm_domains_in_a_scanning_electron_microscope) [accessed Jun 4, 2017]

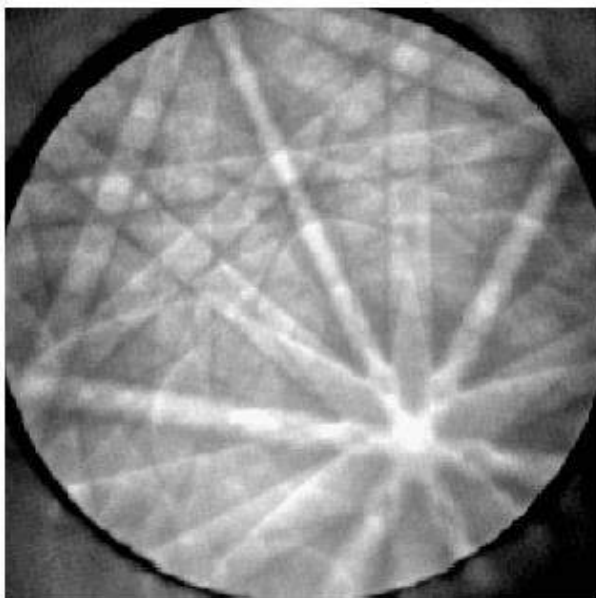


# Análise de grãos:

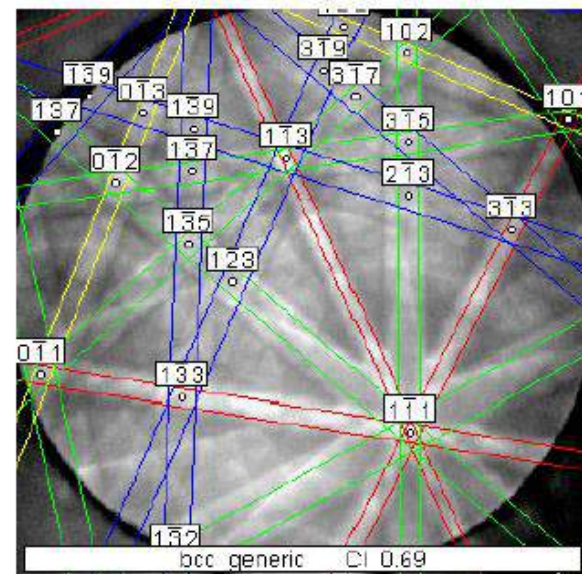
## Difração de elétrons retroespalhados – EBSD



**Figure 1** A view of the inside of the SEM's specimen chamber showing the specimen and the SE, BSE and EBSD detectors.



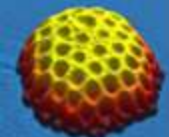
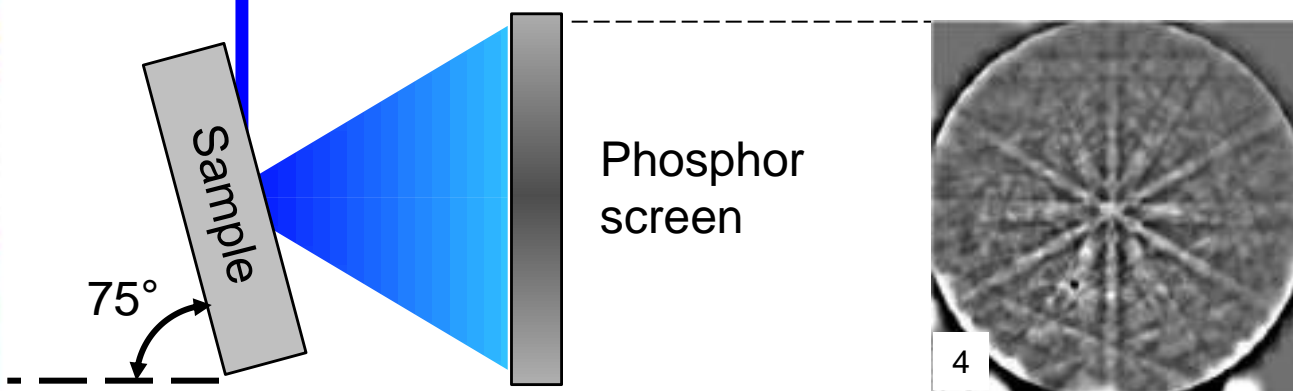
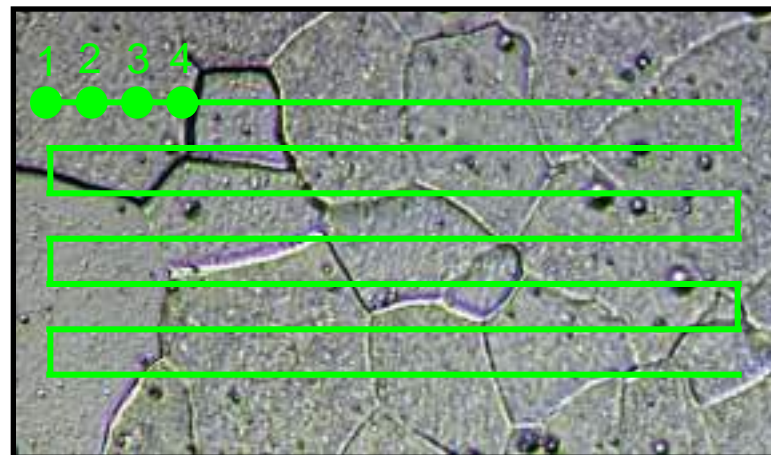
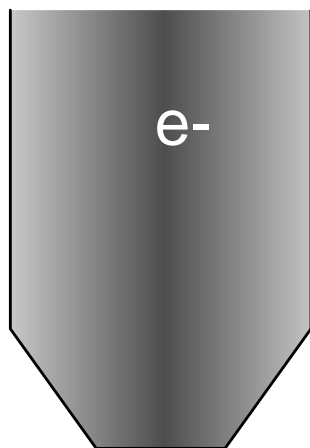
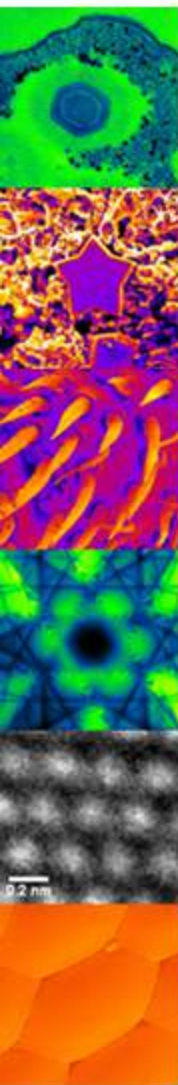
**Figure 3** The EBSD pattern obtained from the position in figure 2 marked by the green cross. Each band in this pattern is produced by a specific set of crystallographic planes. The angles of the bands tell us the orientation of these planes, the width tells us the d-spacing, the angles of the intersections of the bands tell us about the crystal's symmetry and the intersections themselves identify poles.



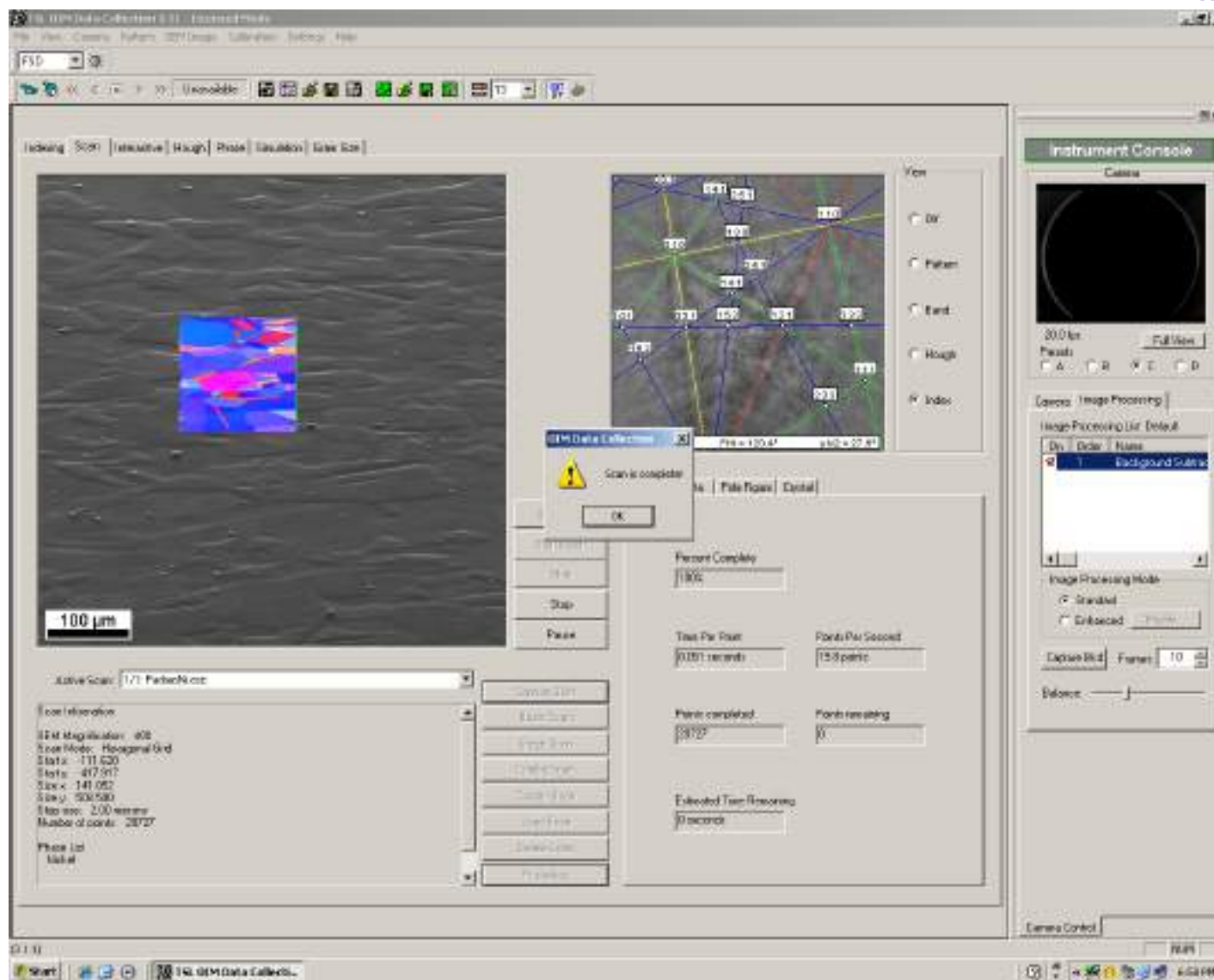
**Figure 5** This is the indexed pattern shown in figure 3. It has been identified as a generic bcc structure that is oriented just off the  $[1 -1 1]$  direction. Note the high confidence index of 0.69.



# ACQUISITION OF KIKUCHI PATTERNS

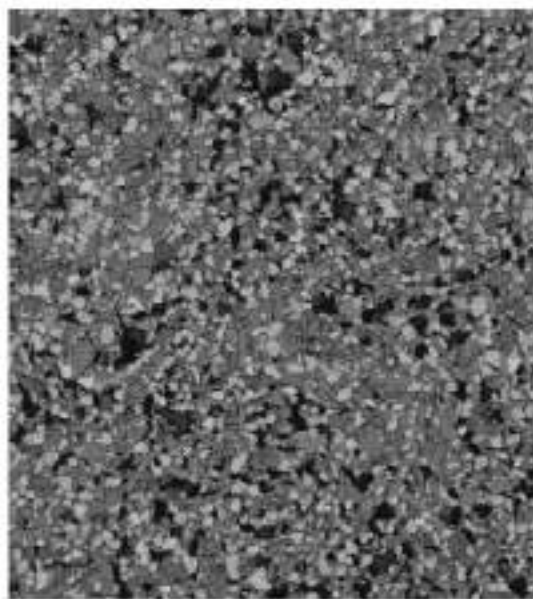
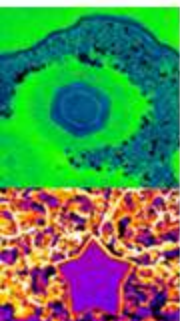


ACQUISITION OF KIKUCHI PATTERNS  
PROCEDURES



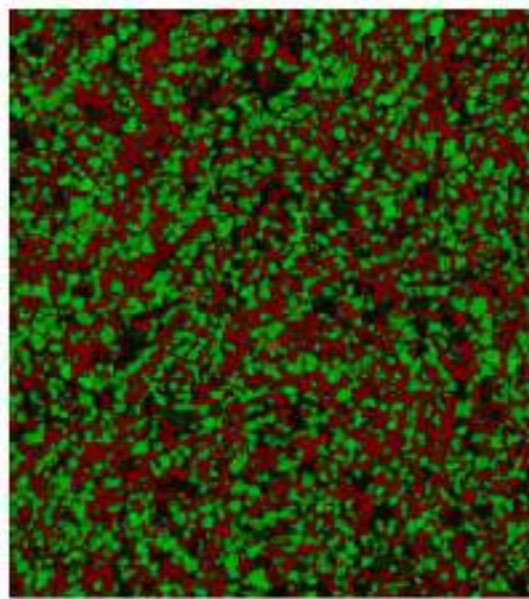


# Difração de elétrons retroespalhados – EBSD



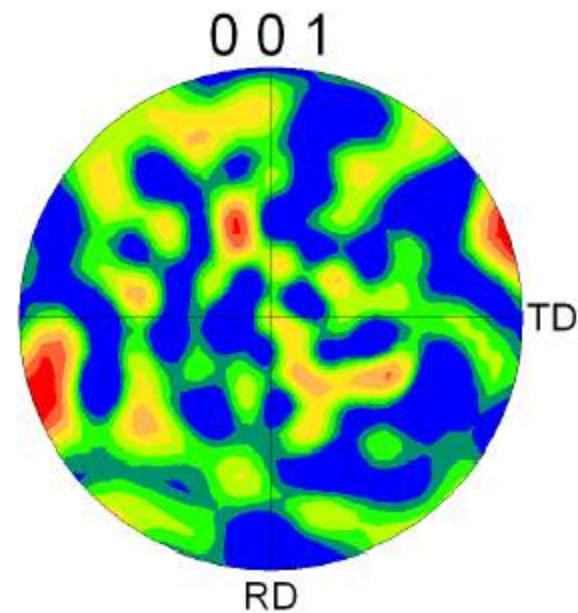
35.00  $\mu\text{m}$  = 70 steps IQ 5.2 / 154

**Figure 9** The pattern image quality map for a TiN-TiB<sub>2</sub> specimen produced by combustion synthesis. Lighter colored areas represent higher image quality and darker areas represent lower image quality. The darkest areas in this sample are either at grain boundaries, where two or more patterns are obtained simultaneously, and in the specimen's pores.

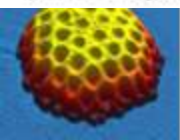


35.00  $\mu\text{m}$  = 70 steps IQ 5.2 / 154, Phase

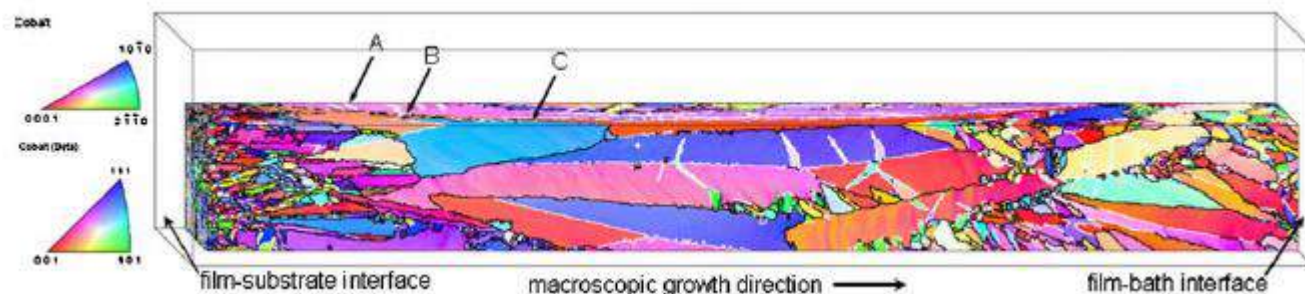
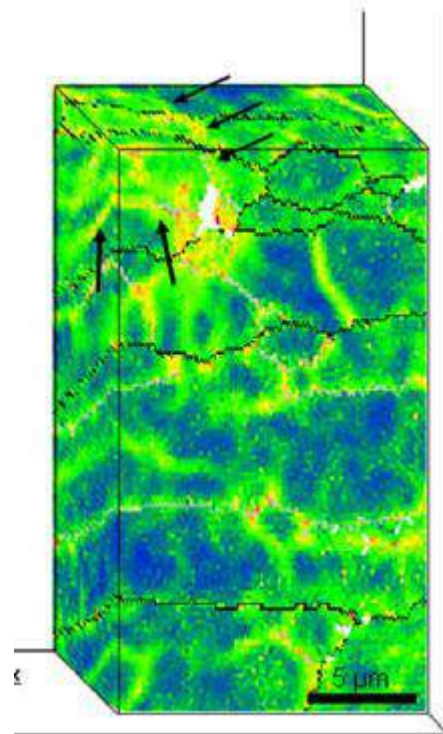
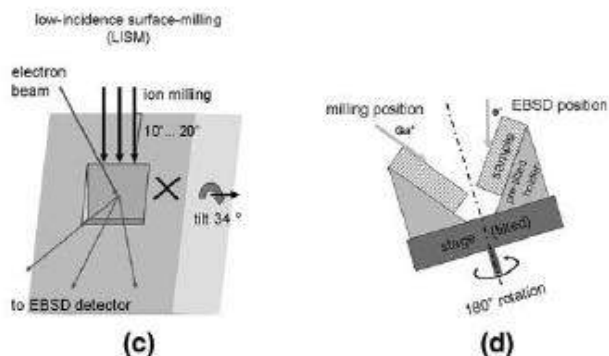
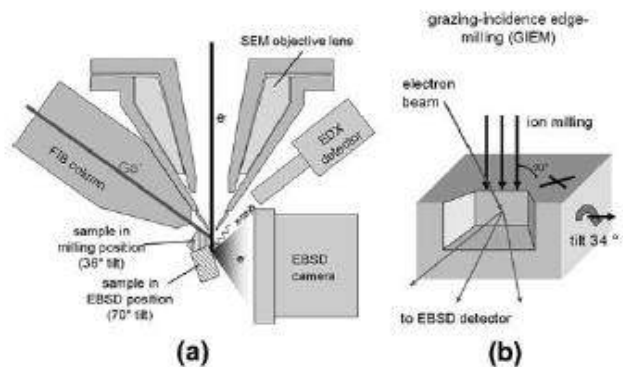
**Figure 10** The phase map for the TiN-TiB<sub>2</sub> specimen. The green areas represent the TiN phase (cubic) and the red areas represent the TiB<sub>2</sub> phase (hexagonal). The black represents areas of poor pattern image quality, such as at grain boundaries and in the pores.



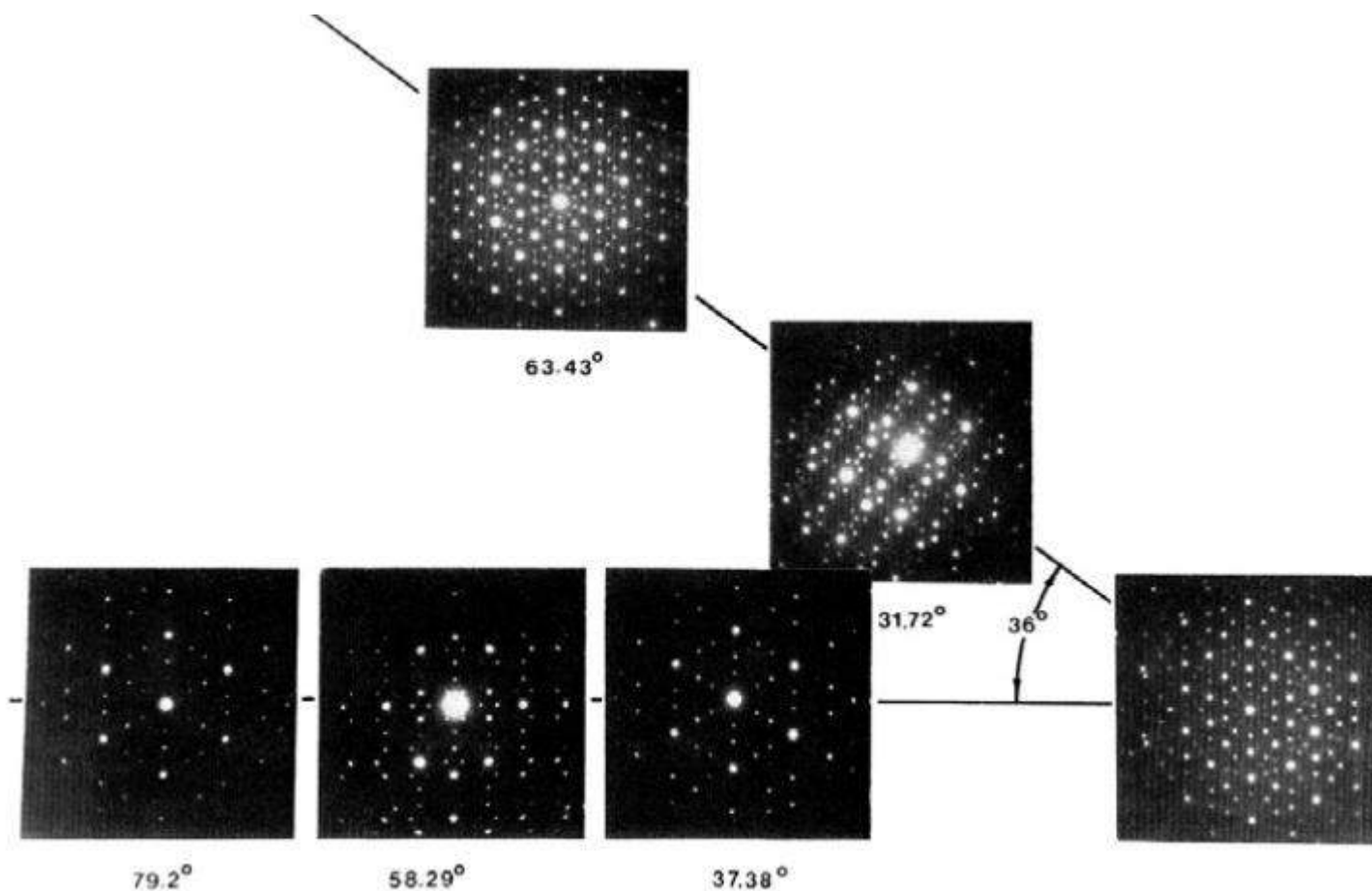
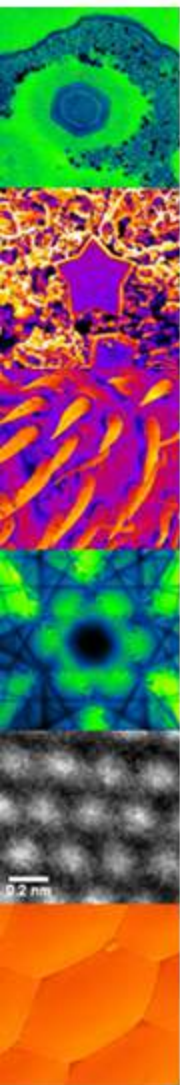
**Figure 12** Pole figure plot showing the crystallographic texture in a calcium cerium titanate ceramic.



# 3D - Electron backscattering diffraction (EBSD)



# Quasicrystals – Dan Schechtman Chemistry Nobel Prize 2011



Al-Mn diffraction patterns in D. Schechtman, I. Blech, D. Gratias, J.W. Cahn (1984) "Metallic phase with long range orientational order and no translation symmetry", *Physical Review Letters* 53 (20), pp 1951 – 1954.



# Softwares

- Vesta: <http://jp-minerals.org/vesta/en/>
- Crystalbox: <https://www.fzu.cz/crystbox>
- JEMS: <http://www.jems-saas.ch/> (pago)
  
- Bancos de dados:
  - <http://www.crystallography.net/cod>
  - <http://rruff.info/>
  
  - <http://scifinder-cas.ez27.periodicos.capes.gov.br/>  
SciFinder da CAS – acessível para as Universidades, pago pela CAPES; necessita de cadastro prévio.

## Alguns outros

- <http://www.freechemical.info/freeSoftware/kinds.php>

