

# Dyfrakcja elektronów wstecznie rozproszonych

## **Analiza punktowa**

**EBSD - Electron Backscatter Diffraction**

**EBSP - Electron Backscatter Pattern**

**BKP - Backscatter Kikuchi Pattern**

## **Obrazowanie orientacji**

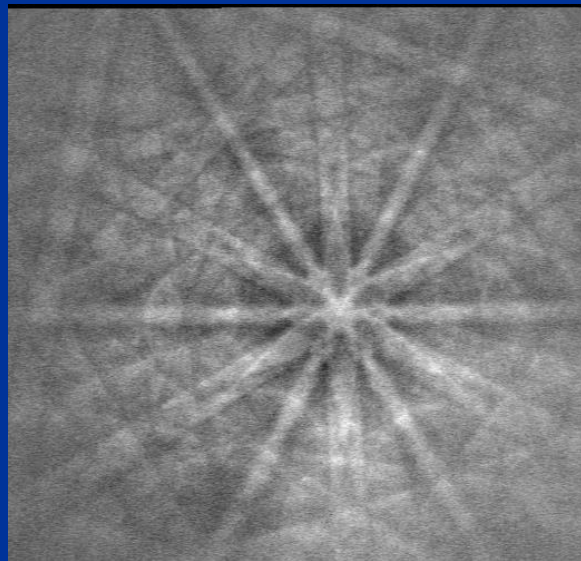
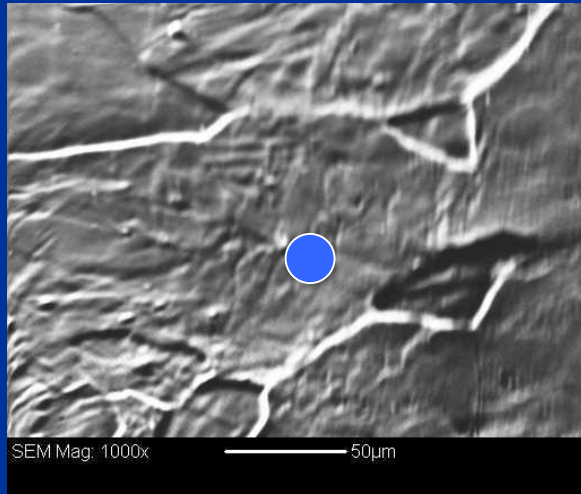
**COM - Crystal Orientation Mapping**

**ACOM - Automatic Crystal Orientation  
Mapping**

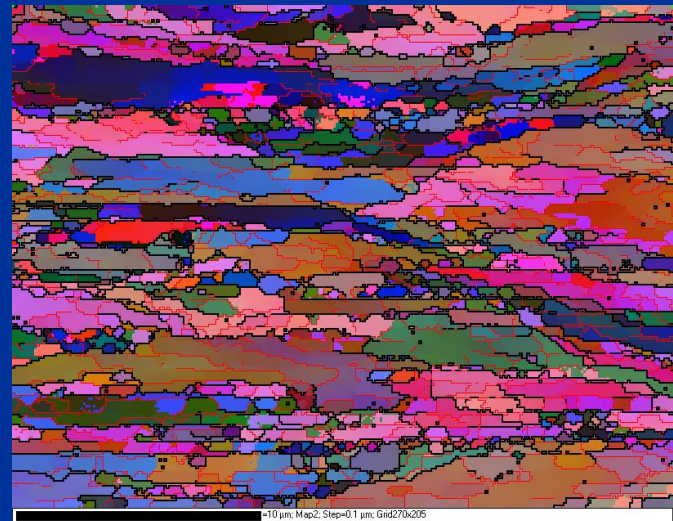
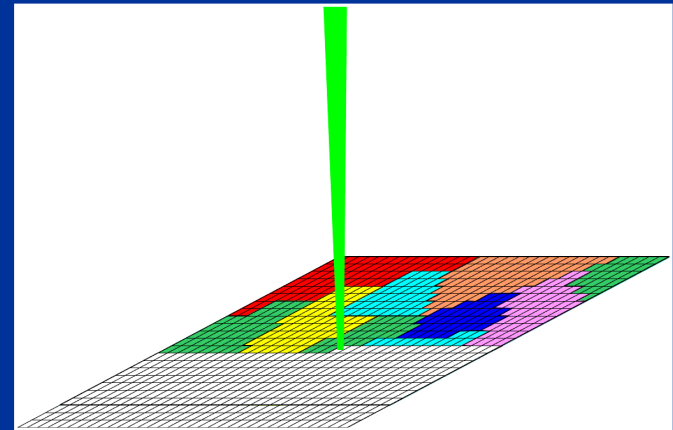
**OIM® - Orientation Imaging Microscopy**

(TexSEM Laboratories trademark)

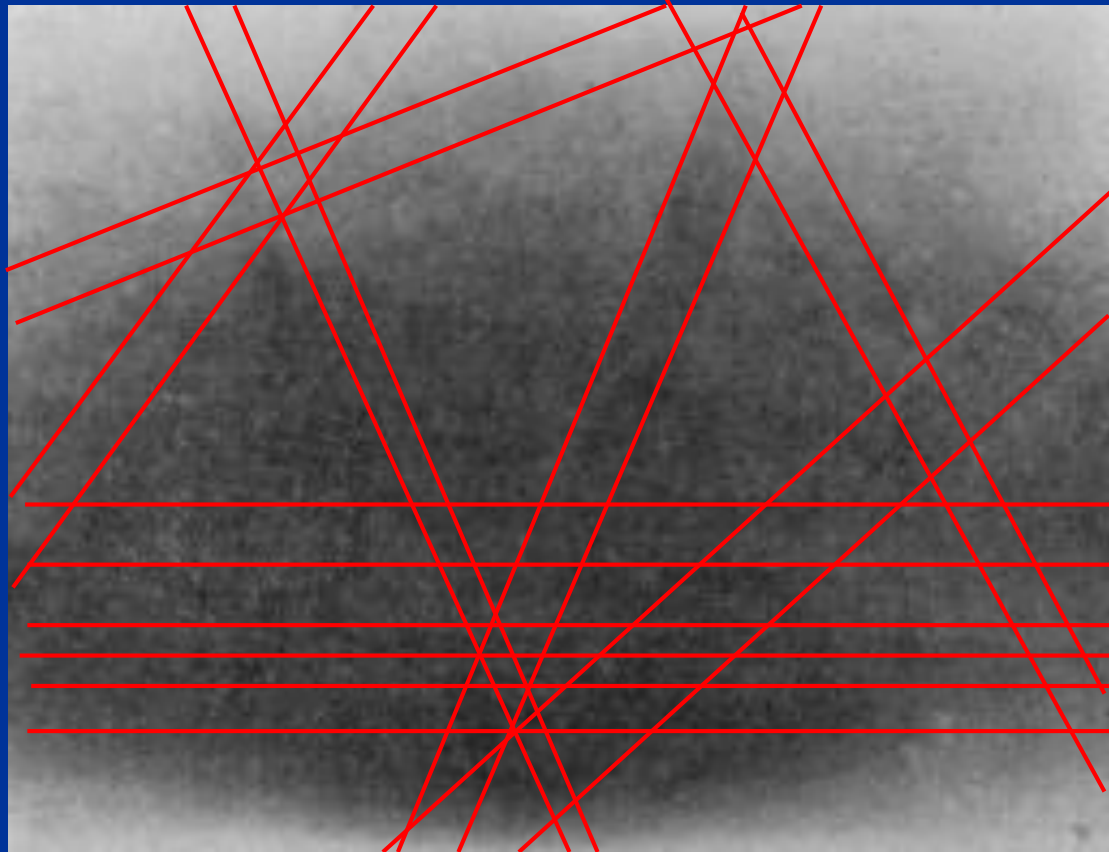
# Analiza punktowa



# Skanywanie wiązką elektronową

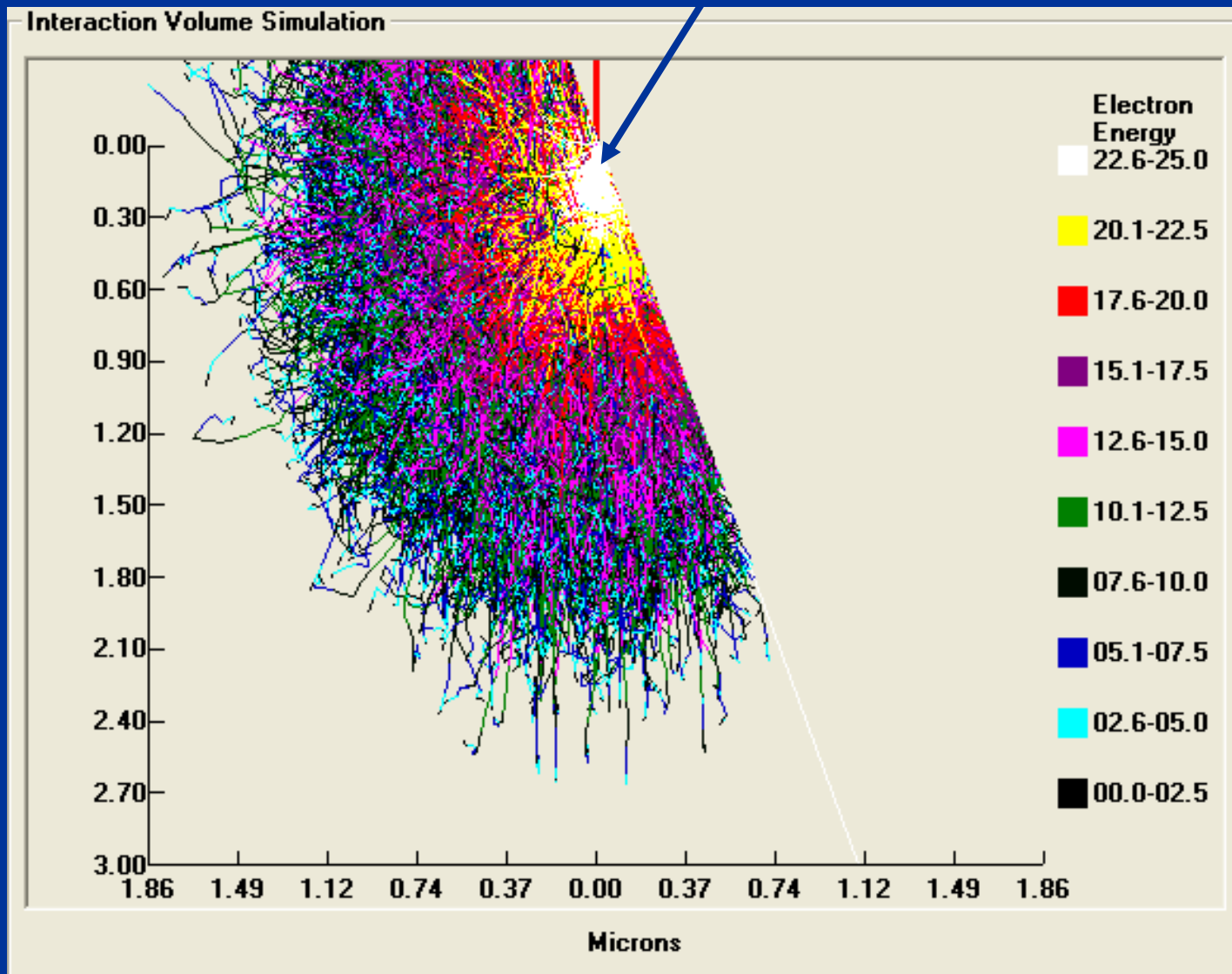


**Pierwszy obraz linii dyfrakcyjnych uzyskany przez Kikuchiego w 1928 roku z kryształu kalcytu  $\text{CaCO}_3$**

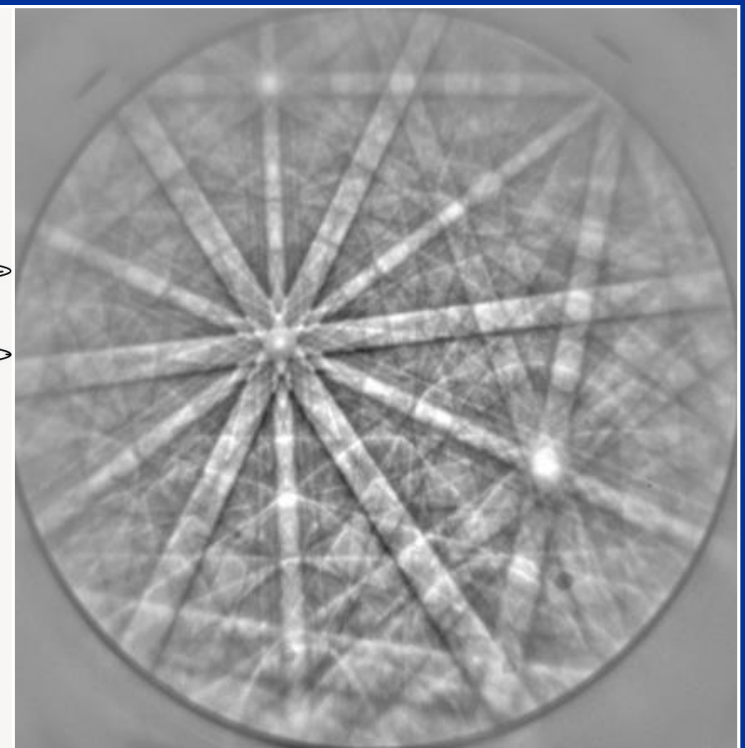
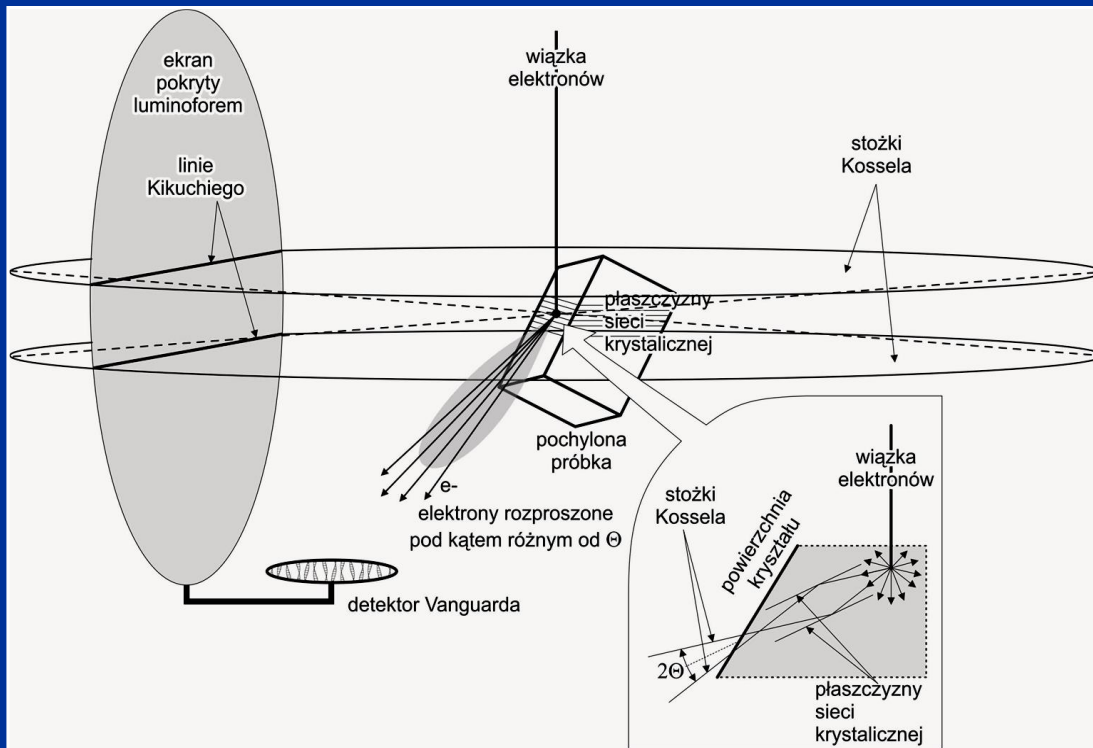


**Shoji Nishikawa and Seishi Kikuchi**  
*„The Diffraction of Cathode Rays by Calcite”*  
**Proc. Imperial Academy (of Japan) 4 (1928) 475-477**

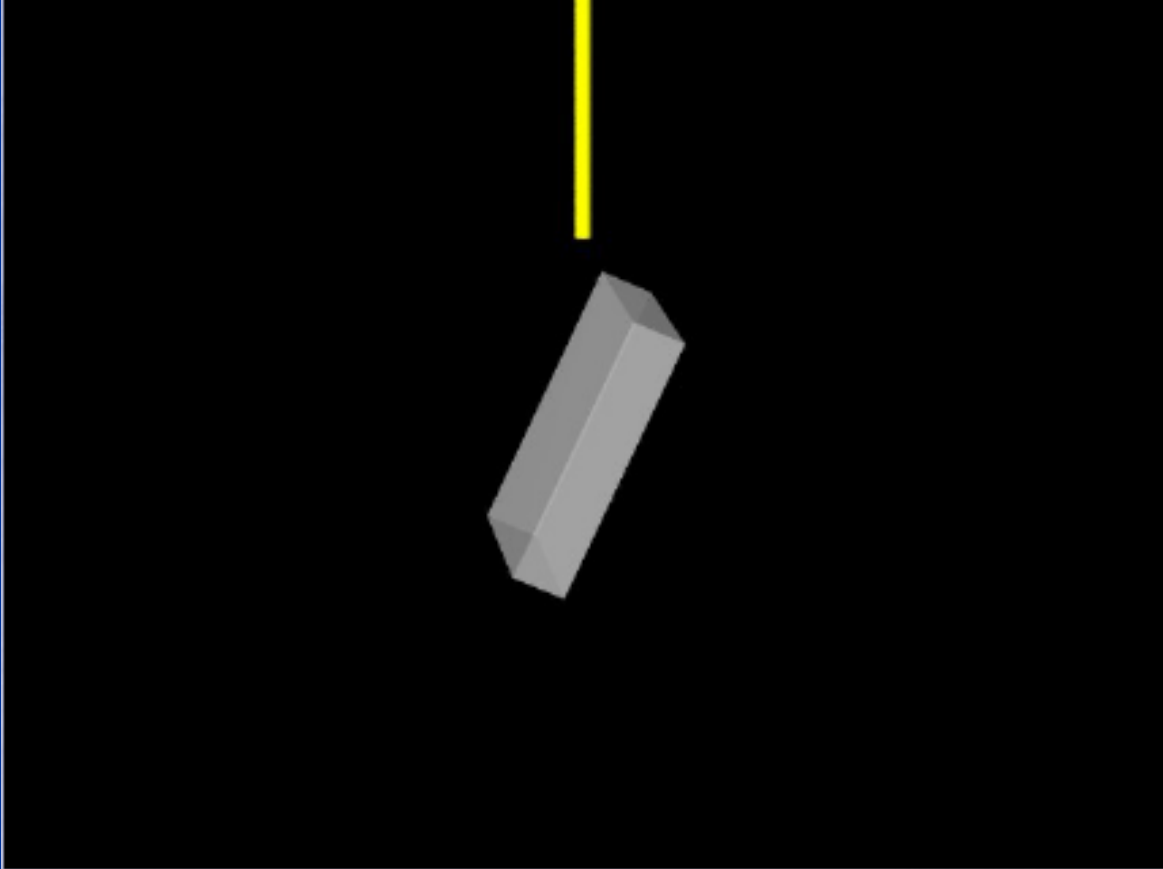
# Punktowe źródło elektronów

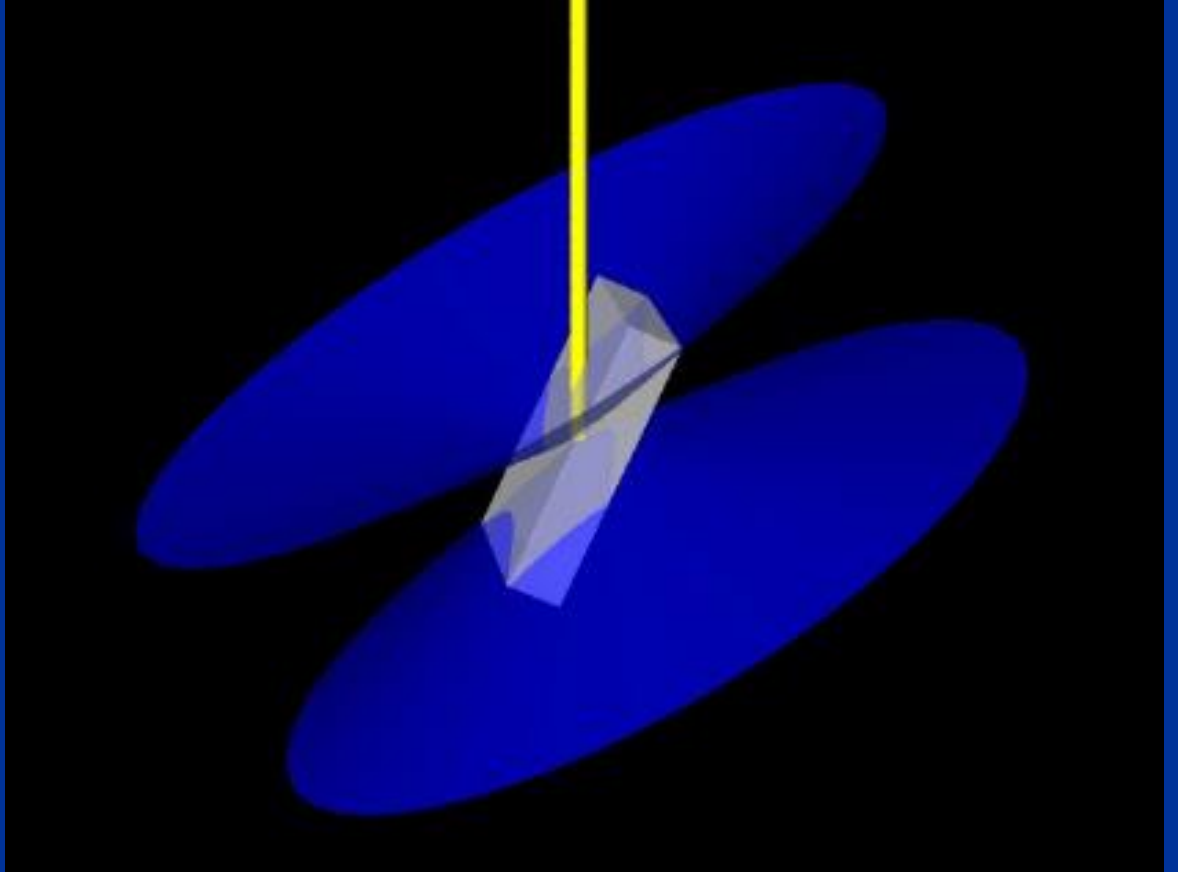
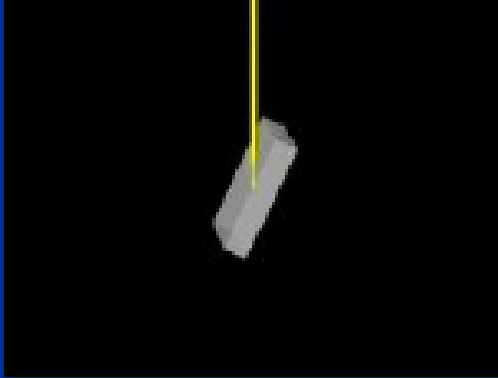


- Elektrony wstecznie rozproszone ulegają dyfrakcji tworząc dla każdej płaszczyzny krystalograficznej stożki dyfrakcyjne o dużym kącie rozwarcia (tzw. stożki Kossela)
- Przecięcie tych stożków z ekranem luminoforu – układ równoległych linii (pasm) zwanych liniami (pasmami) Kikuchiego **unikalny dla danej orientacji krystalitu**

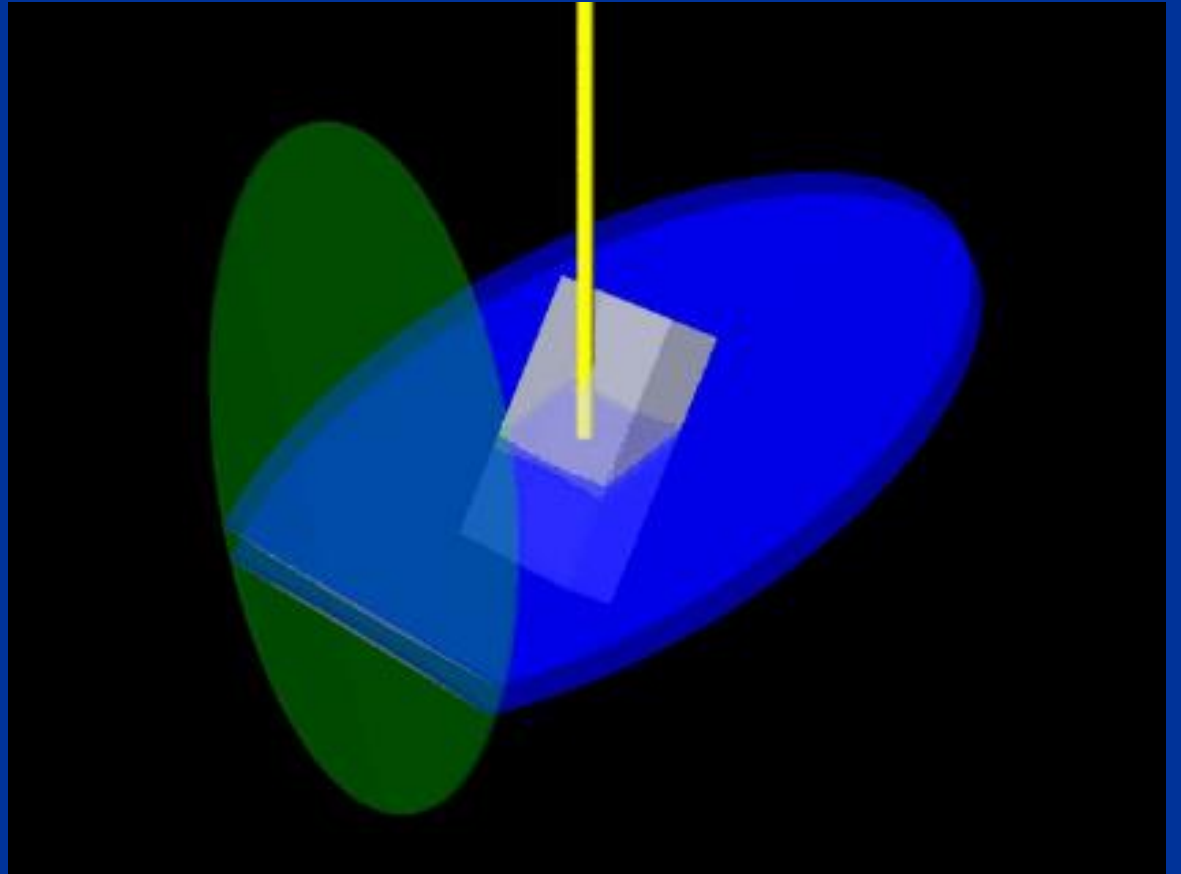
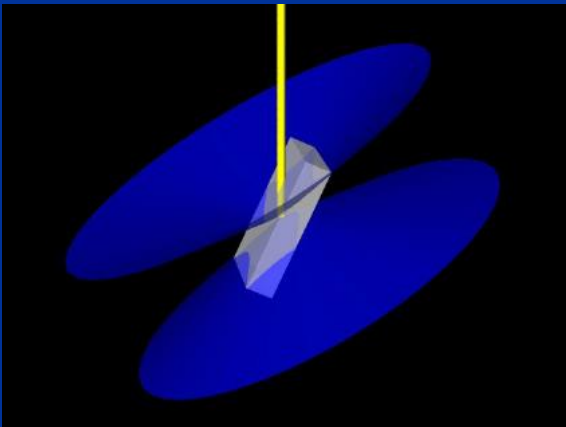
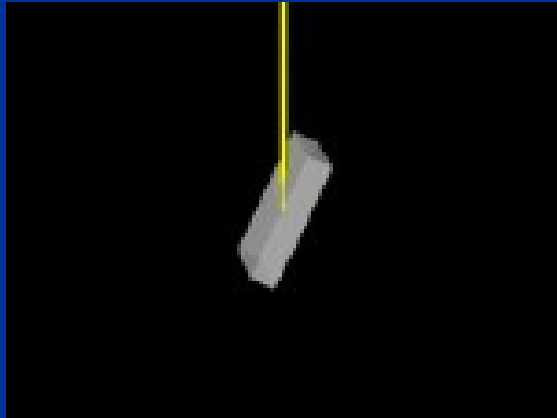


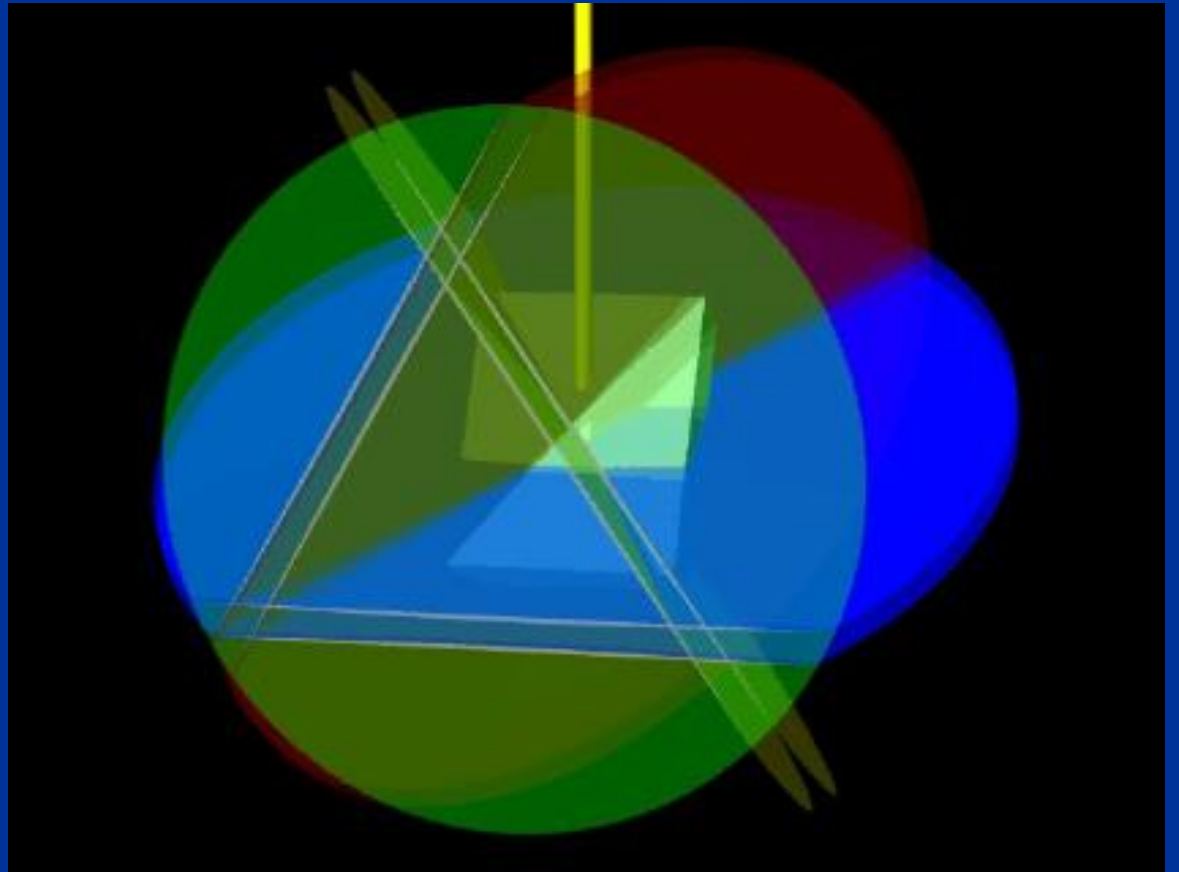
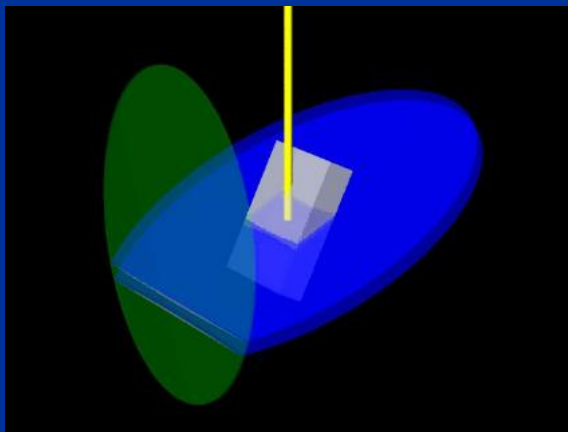
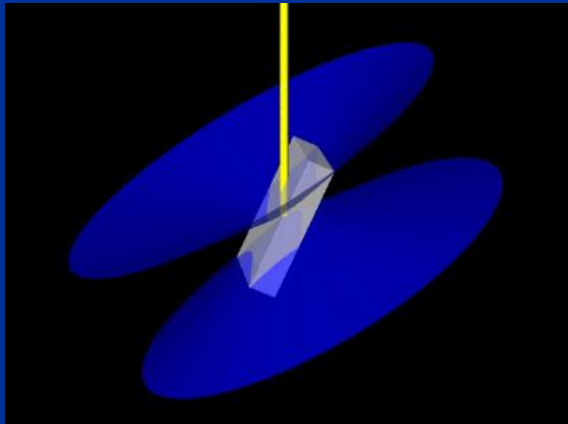
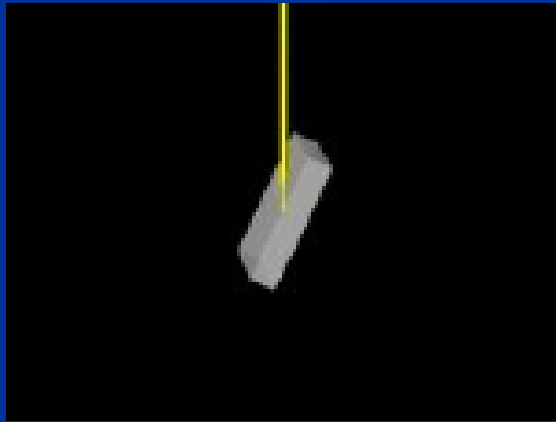
EBSD z krystalitu  $\text{FeTiO}_3$

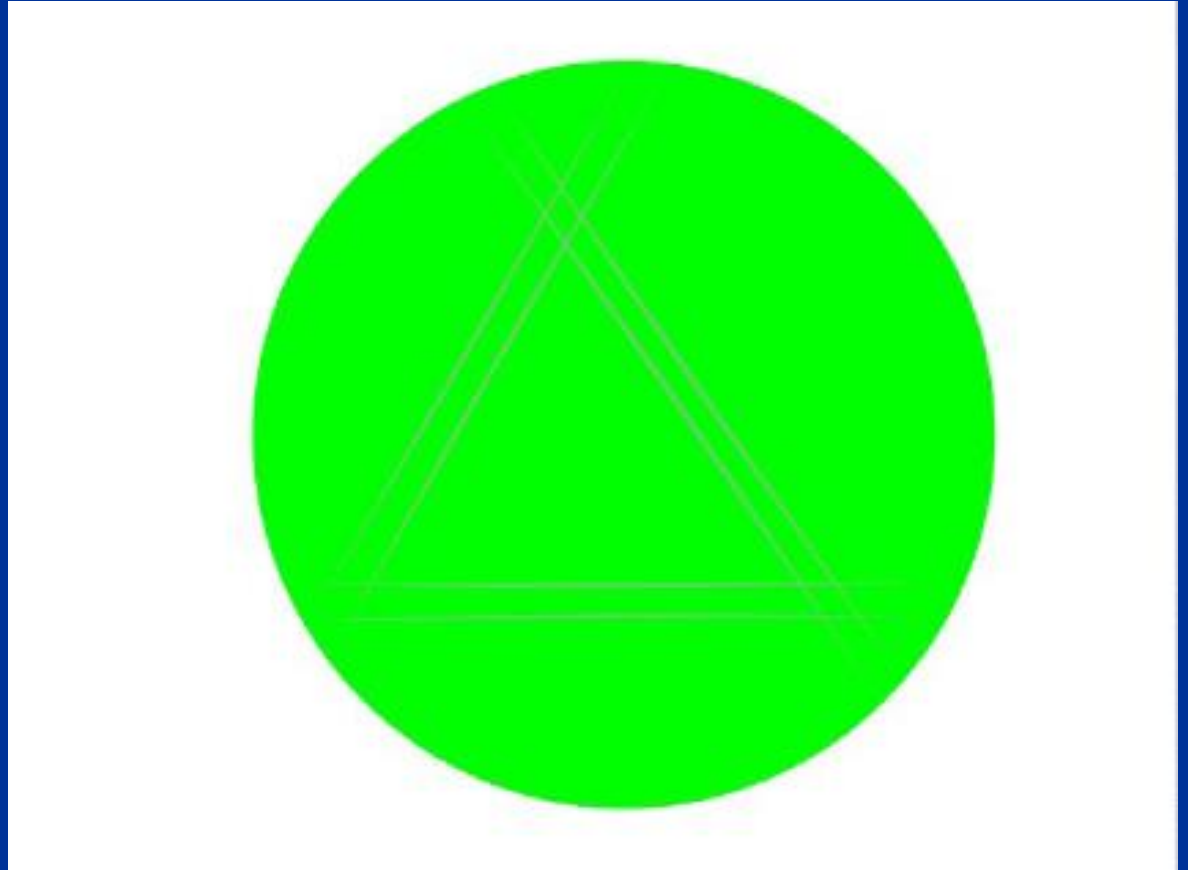
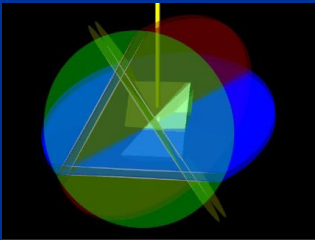
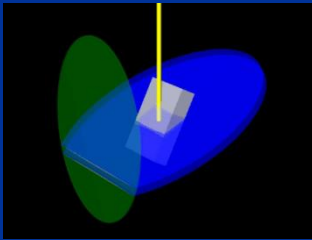
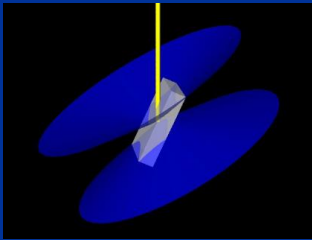
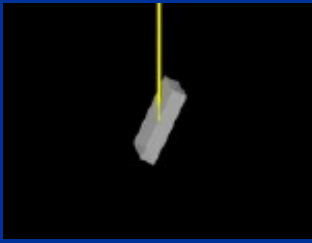


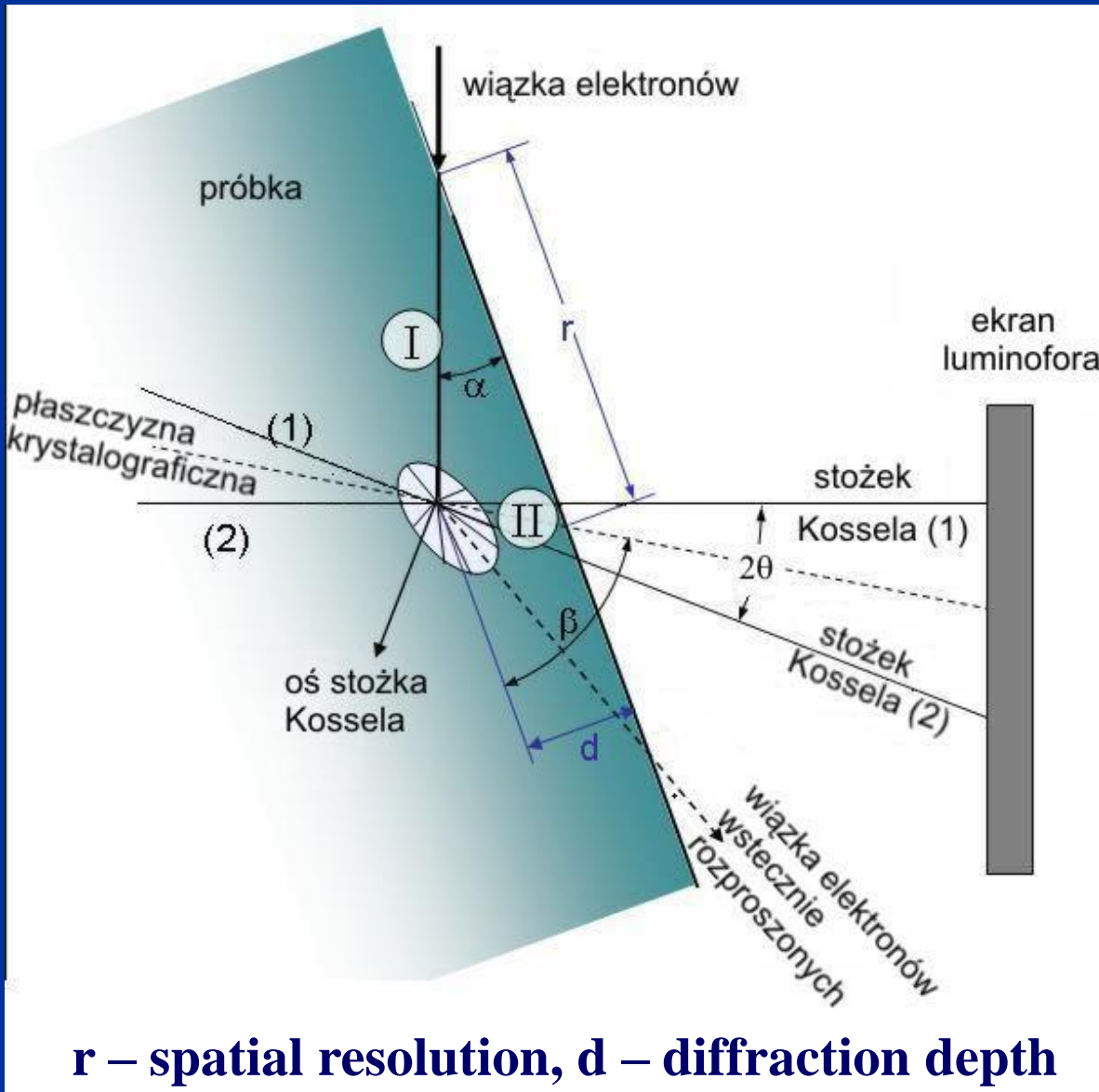










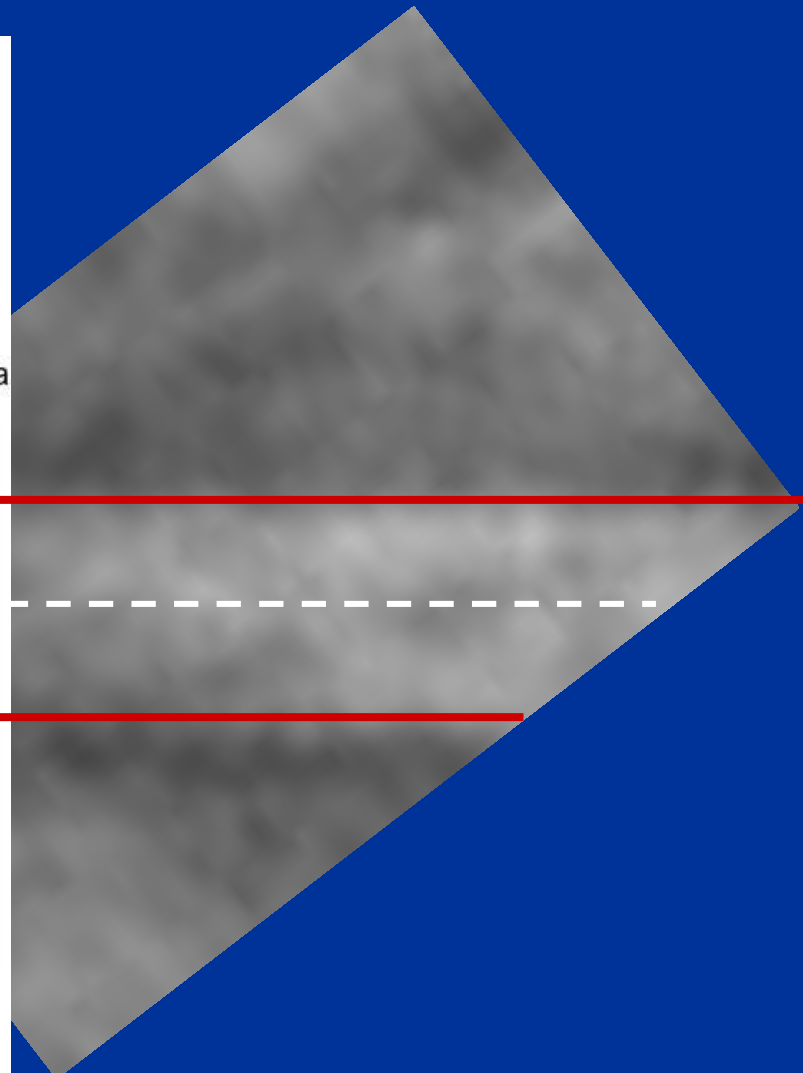
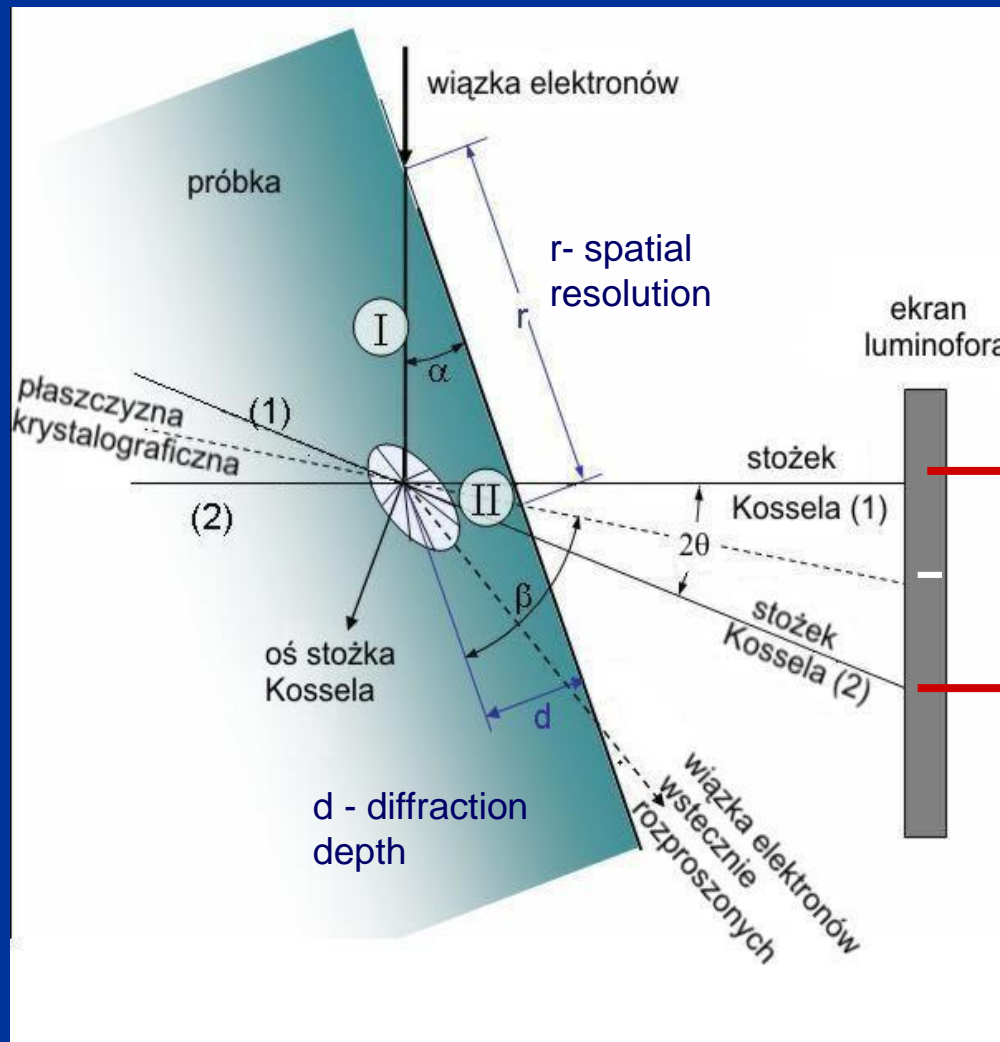


**r – spatial resolution, d – diffraction depth**

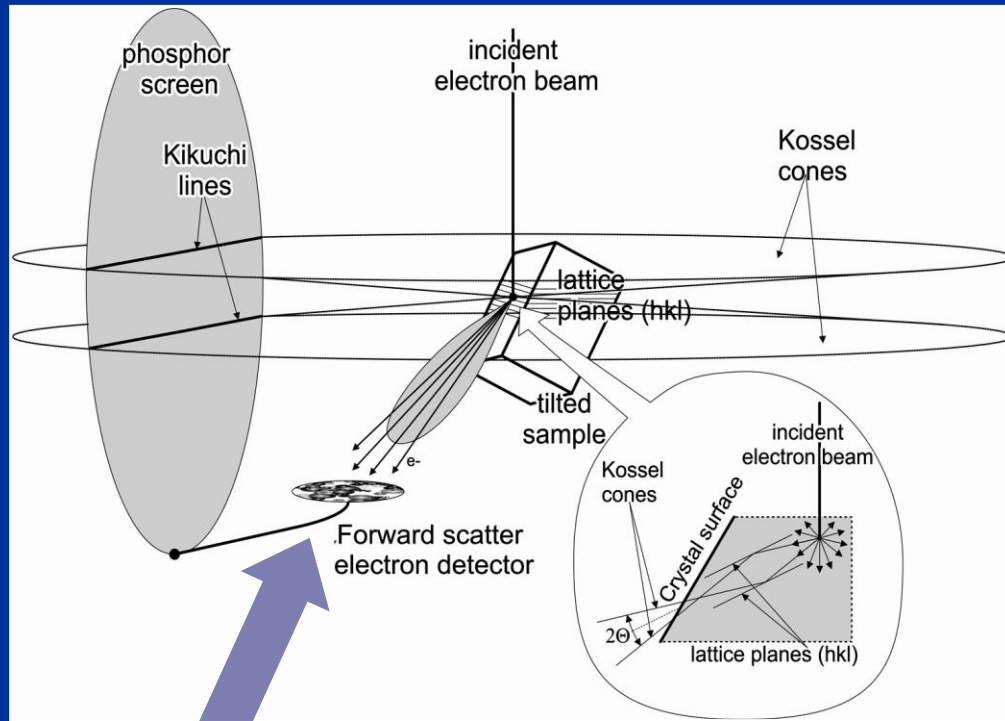
**1** utworzenie źródła elektronów w warstwie podpowierzchniowej kryształu – elektrony rozpraszane są we wszystkich kierunkach. Część ulega dyfrakcji zgodnie z prawem Bragga:

$$\lambda = 2d_{hkl} \sin\theta$$

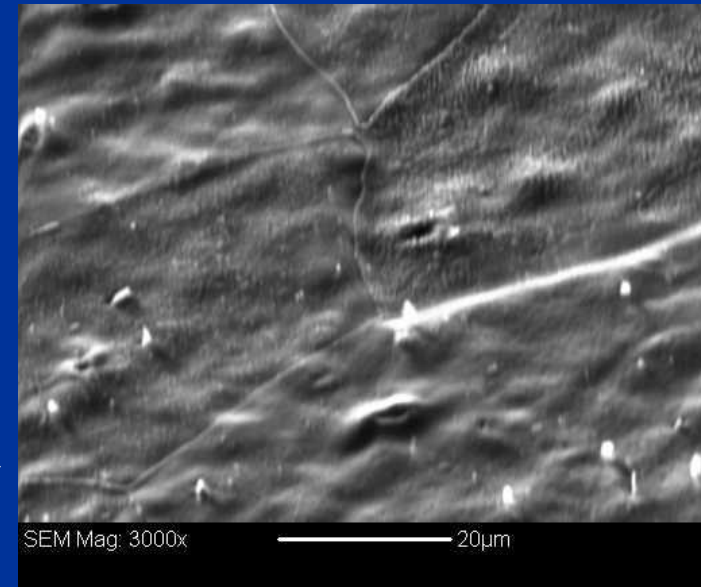
**2** Kąt Bragga jest mały (1° dla 15 kV) – stożki są duże – linie przecięcia z ekranem prawie proste; odległość pomiędzy liniami proporcjonalna do  $\tan(\theta)$  a linia środkowa pomiędzy przecięciami stożków jest rzutem gnomonicznym płaszczyzny krystalograficznej uginającej elektrony



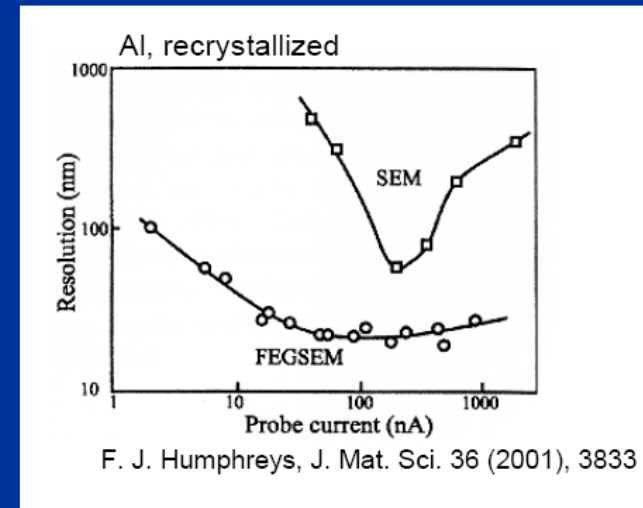
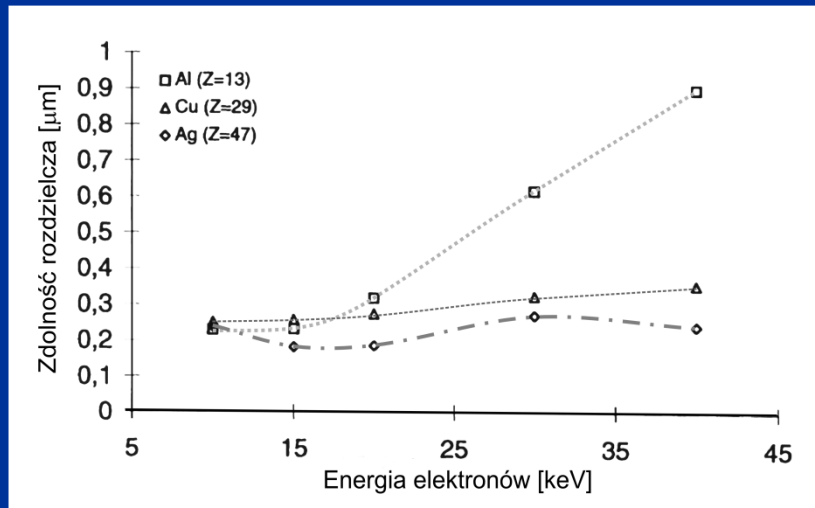
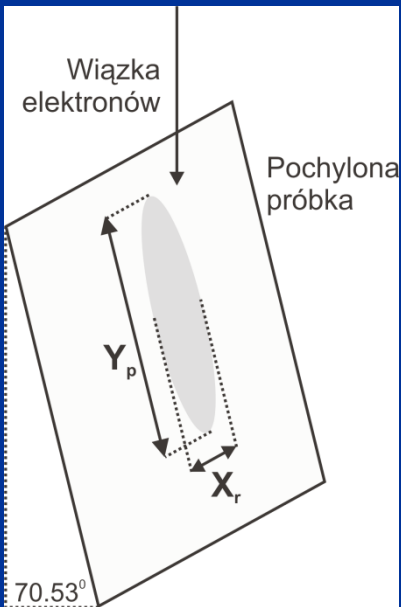
Technika badawcza bardzo czuła powierzchniowo



**Dodatkowy detektor elektronów rozproszonych „do przodu” (forward scattered) umieszczony na kamerze CCD służy do rejestracji obszarów, z których rejestrowane są dyfrakcje**



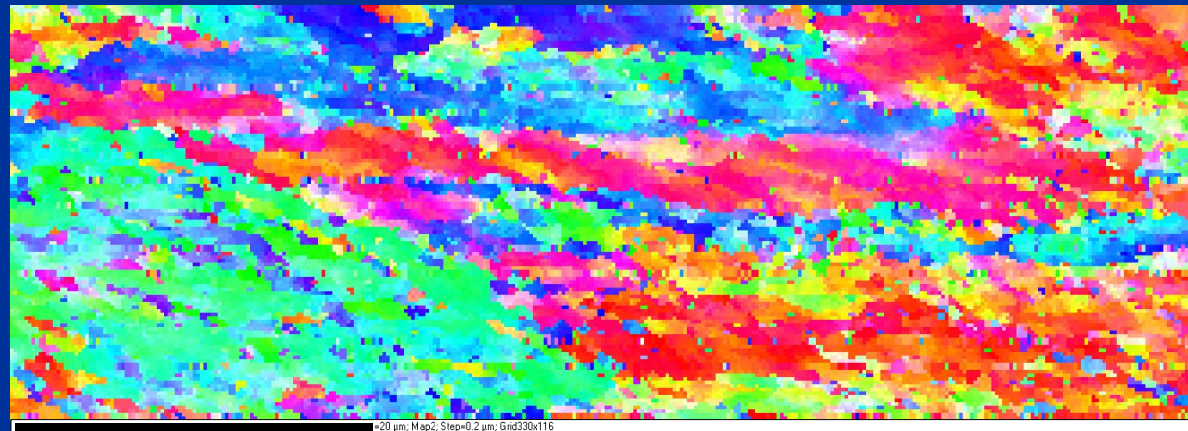
# Przestrzenna zdolność rozdzielcza EBSD



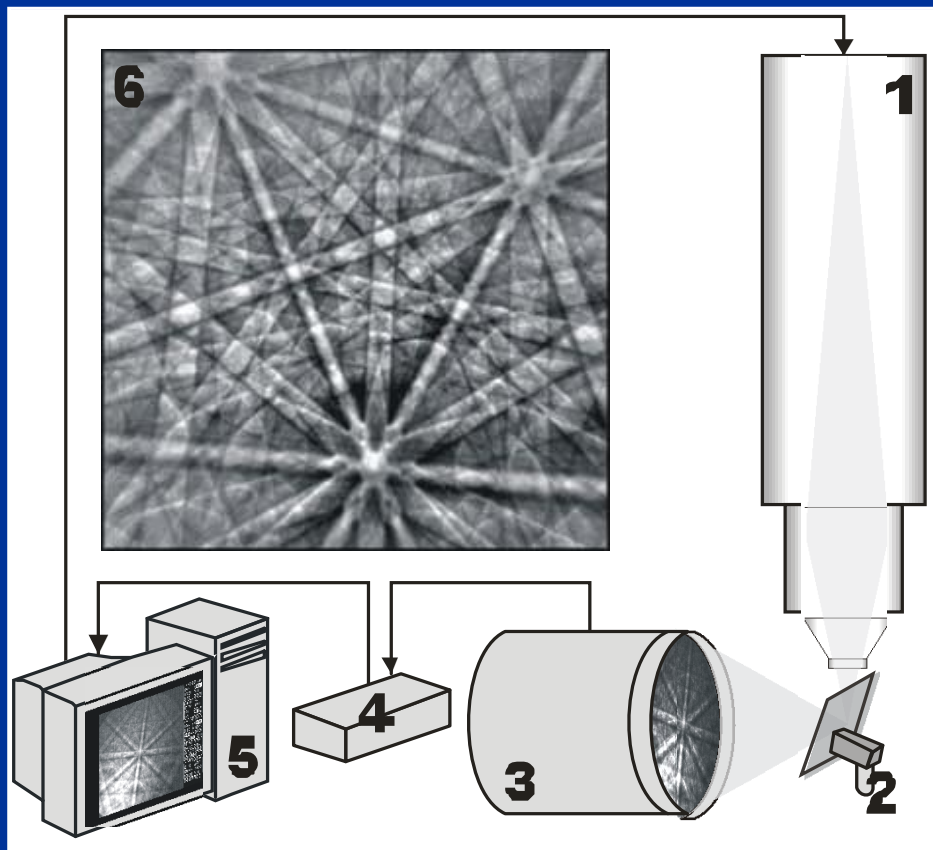
Zależność zdolności rozdzielczej od napięcia przyspieszającego dla Al, Cu i Ag

Zależy:

- od rodzaju materiału
- od źródła elektronów (FEG z termoemisją!)

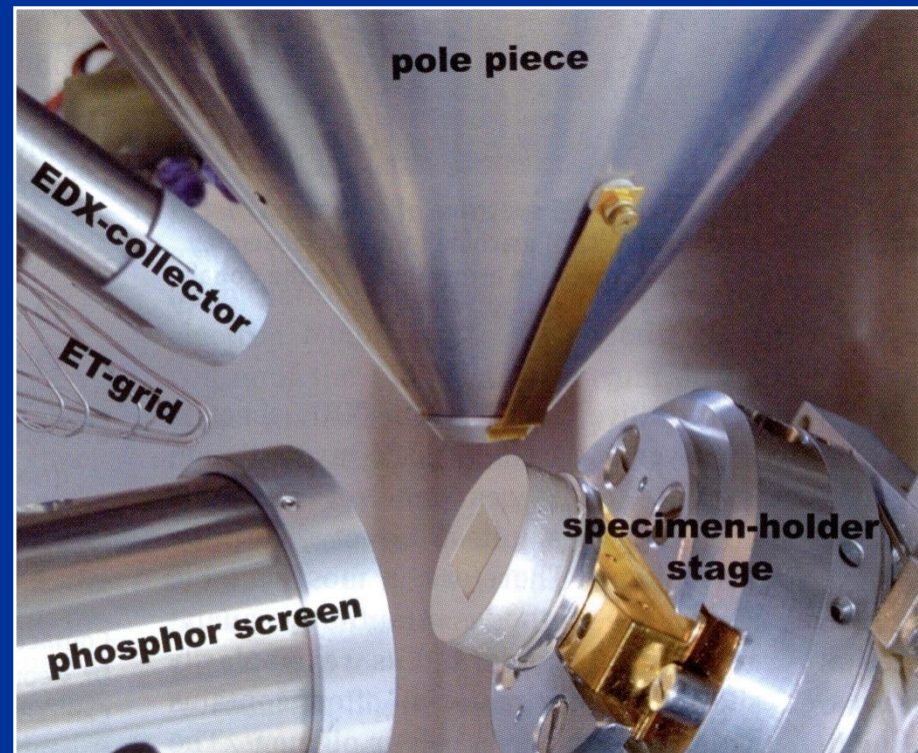


Cu, ECAP step size 200 nm FEI FEGSEM



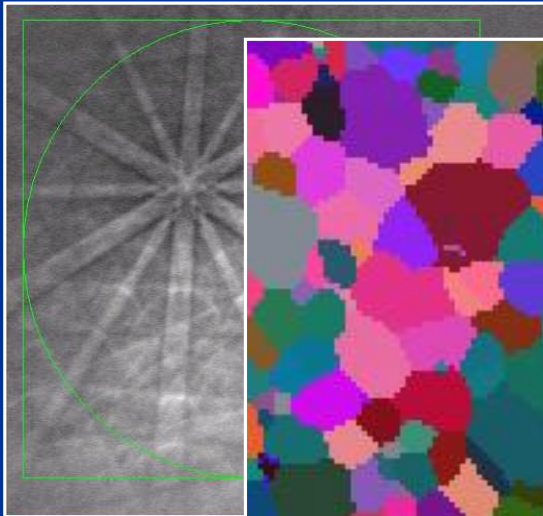
## Schemat systemu EBSD

- 1 – kolumna elektronooptyczna
- 2 – próbka
- 3 – kamera CCD
- 4 – sterownik kamery
- 5 – komputer sterujący wiązką oraz przeprowadzający analizę obrazu
- 6 – obraz dyfrakcyjny

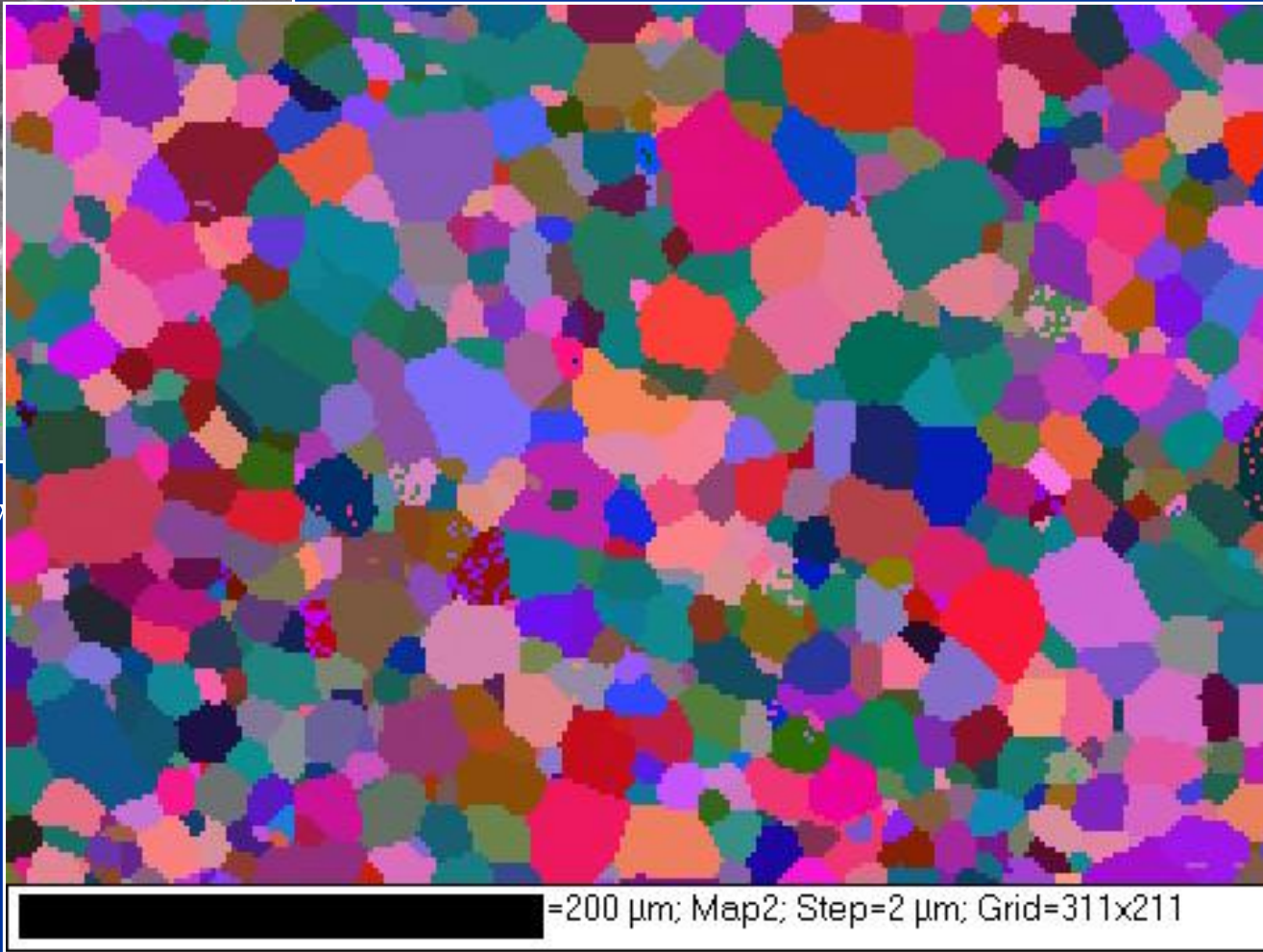




# Jak otrzymać mapę orientacji?



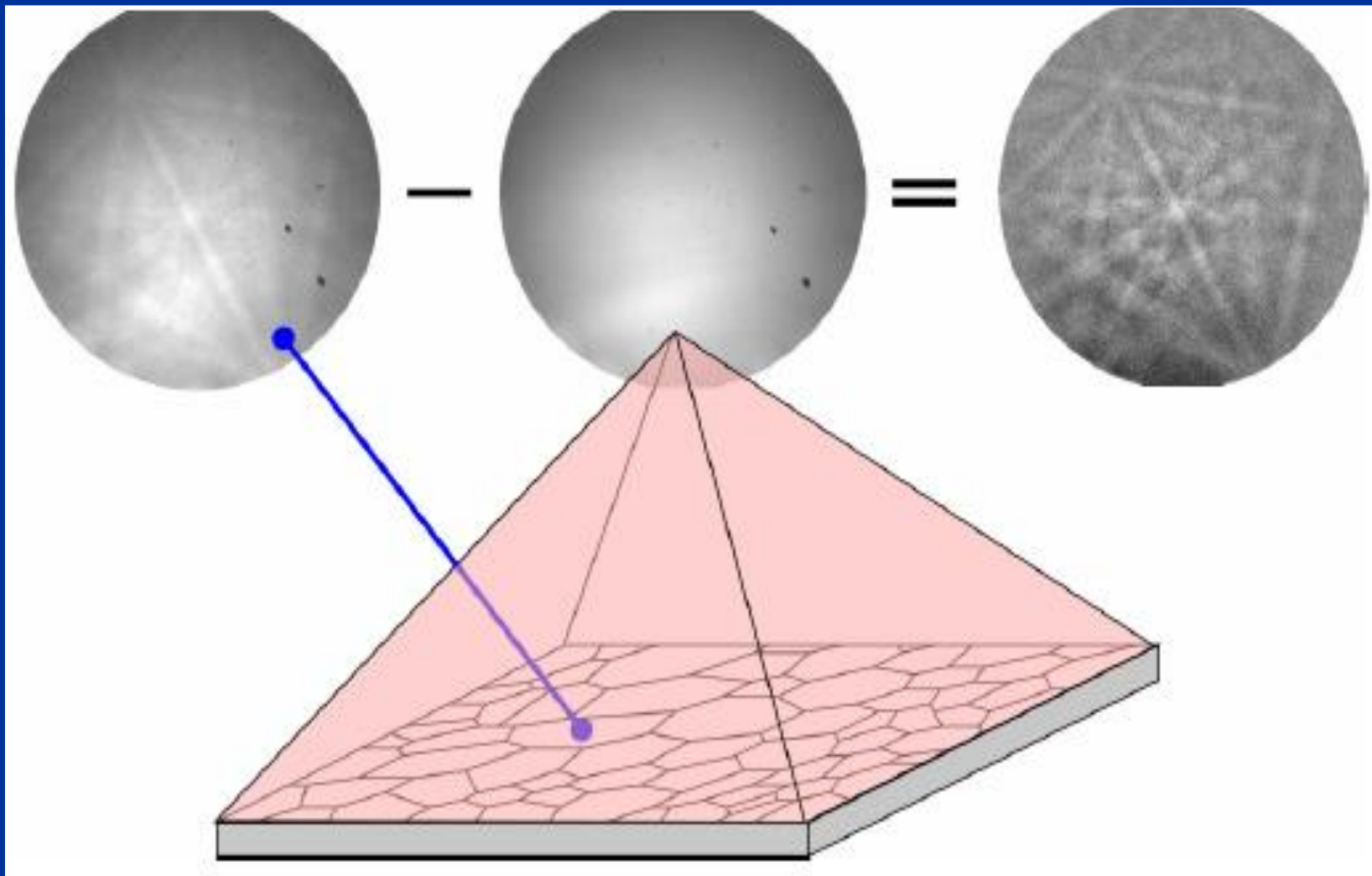
Akw



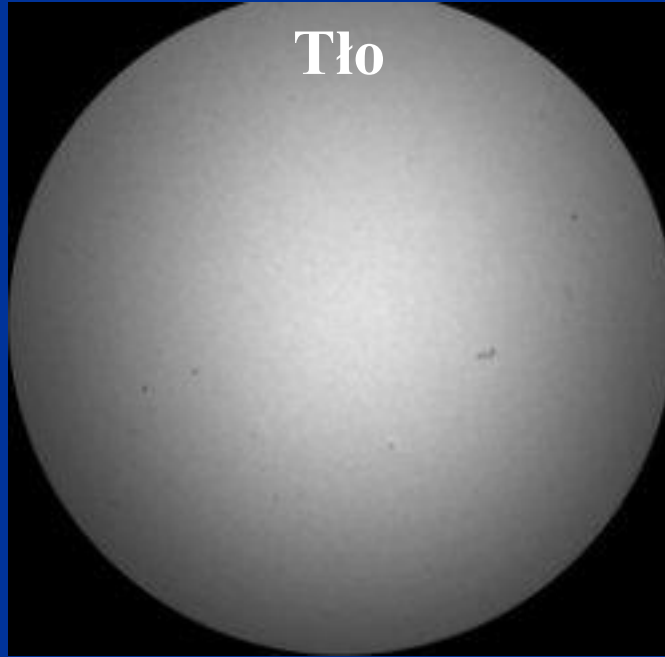
frakcji

**TO NIE JEST TYLKO KOLOROWA MAPA!!!**

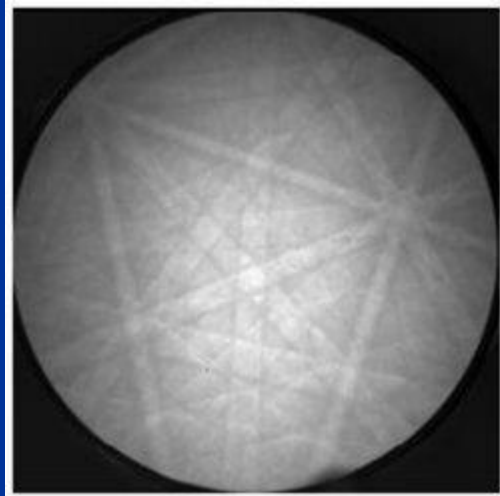
# Akwizycja – odejmowanie tła



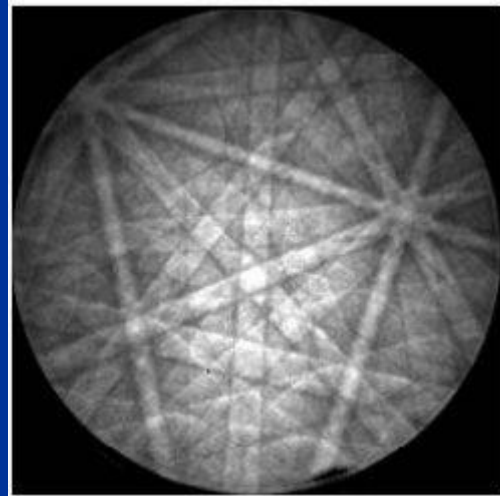
Tło



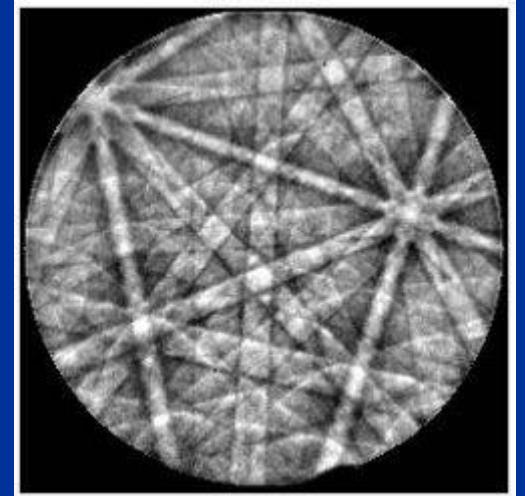
Dyfrakcja wyjściowa



Odejmovanie tła



Dzielenie przez tło



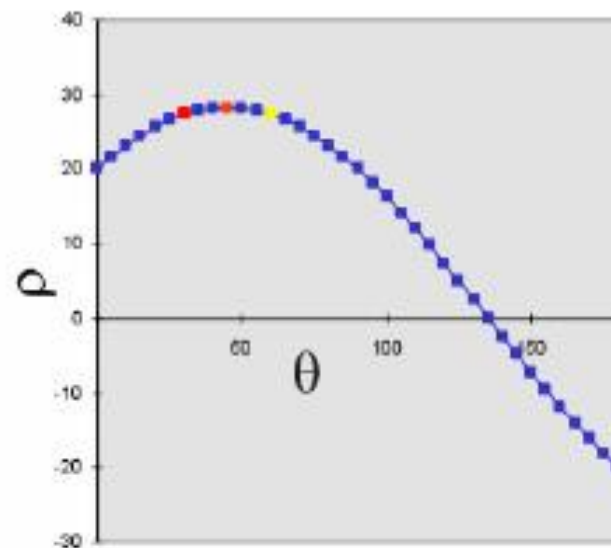
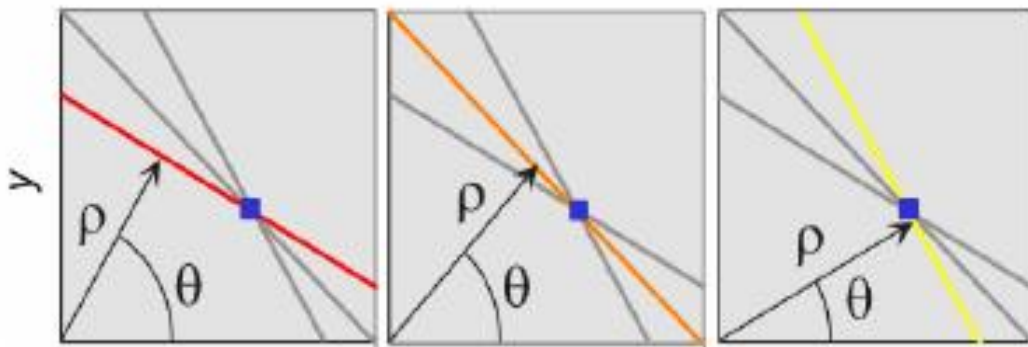
# Detekcja linii na dyfrakcji – transformata Hougha

P.C.V. Hough „Method and means for recognising complex pattern”

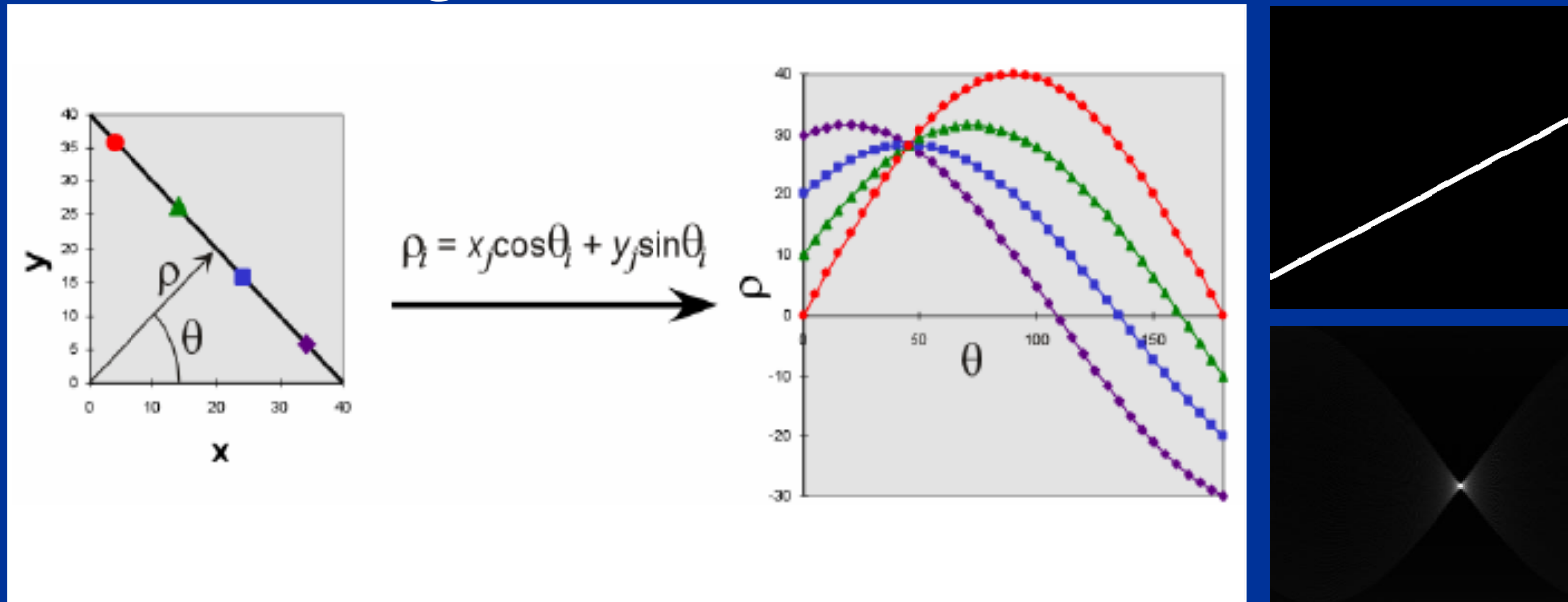
US Patent 3 069 654, 1962

- Przez pojedynczy pixel na układzie X-Y można przeprowadzić nieskończoną liczbę linii
- Linia ta może być opisana przez parametry Hougha „ $\rho$ ” i „ $\theta$ ”, gdzie „ $\rho$ ” reprezentuje odległość linii od początku układu a „ $\theta$ ” – kąt pochylenia linii
- Punkt w przestrzeni obrazu jest przedstawiony jako sinusoida w przestrzeni Hougha

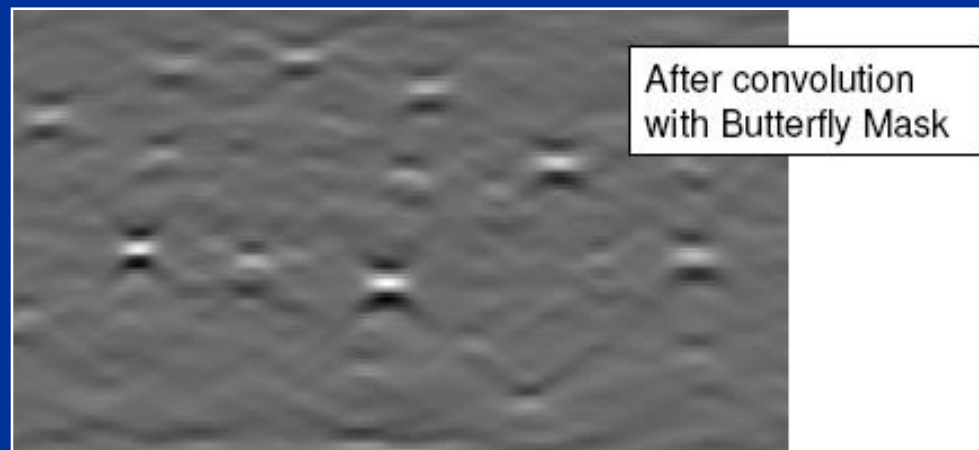
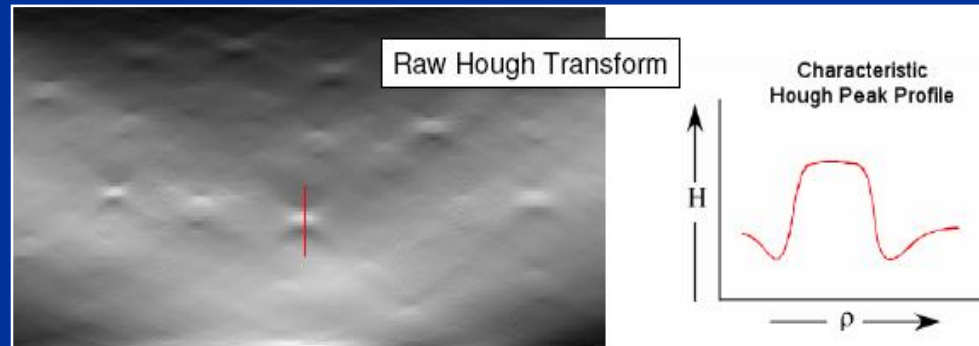
$$\rho_i = x \cos \theta_i + y \sin \theta_i$$



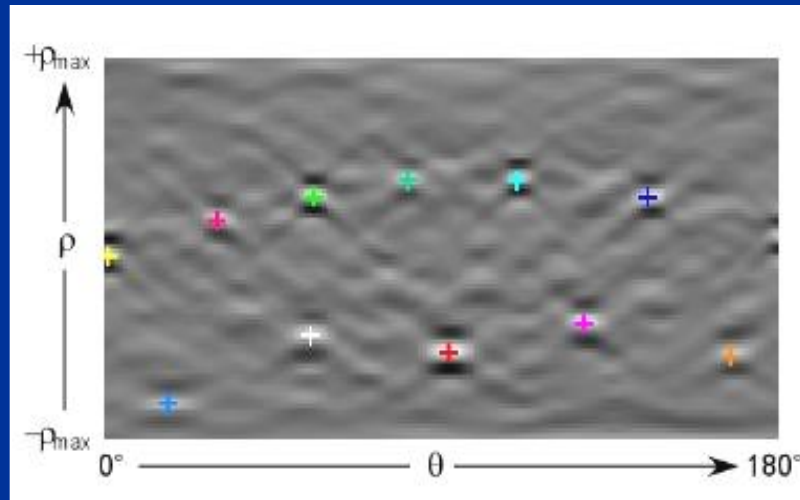
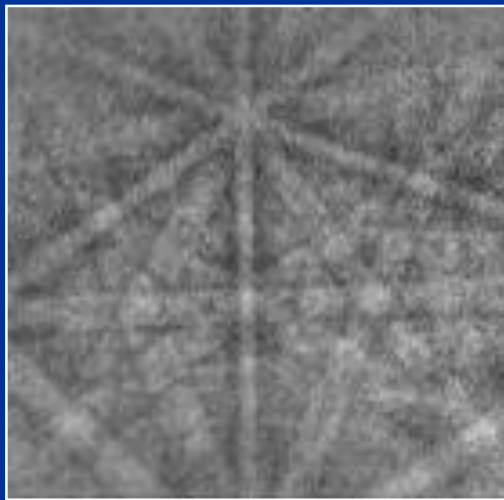
- Rozważmy 4 piksele leżące na jednej linii. Dla każdego piksela obliczamy wszystkie możliwe wartości „ $\rho$ ” dla kąta „ $\theta$ ” w zakresie od  $0^\circ$  do  $180^\circ$  zgodnie z równaniem:  $\rho = x\cos\theta + y\sin\theta$ .
- Otrzymujemy 4 krzywe sinusoidalne przecinające się w punkcie o koordynatach „ $\rho$ ” i „ $\theta$ ”, odpowiadających koordynatom „ $\rho$ ” i „ $\theta$ ” dla linii w układzie X-Y.
- Linia w układzie X-Y przekształca się w punkt w układzie  $\rho$ - $\theta$  (transformata Hougha).

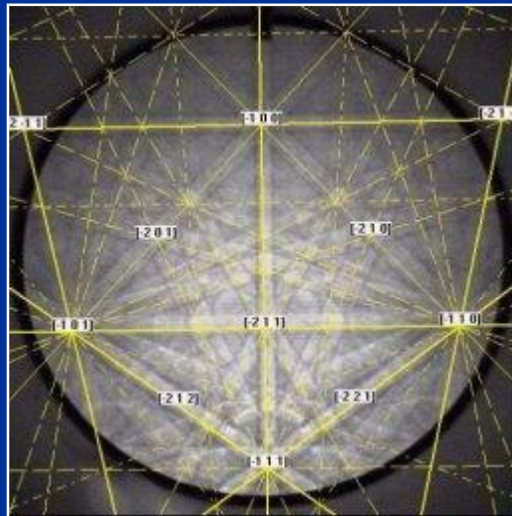
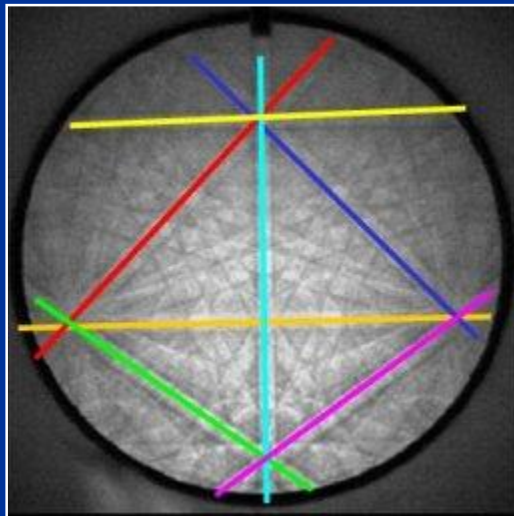
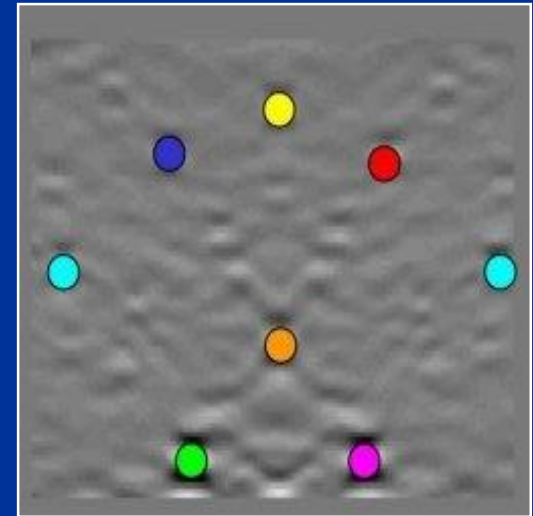
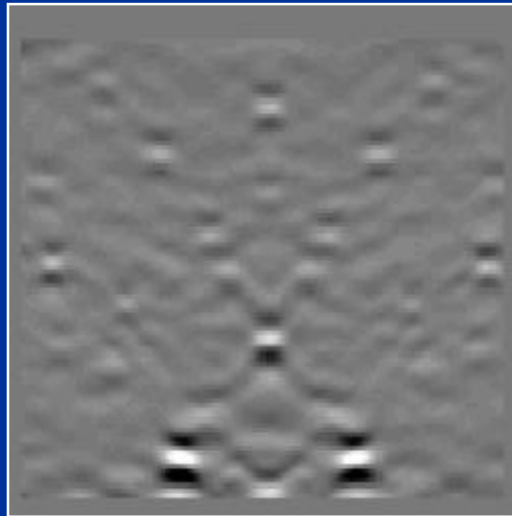
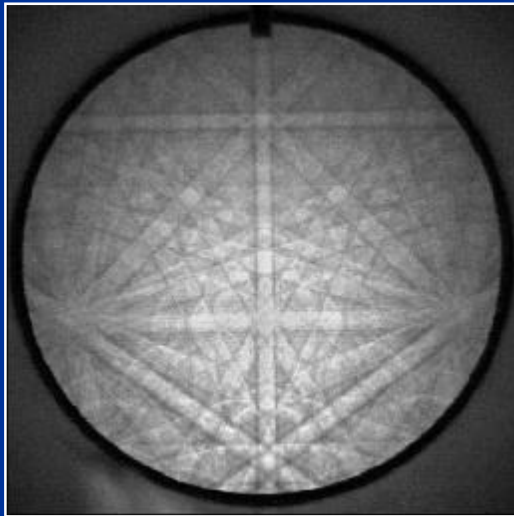


# Transformata Hougha



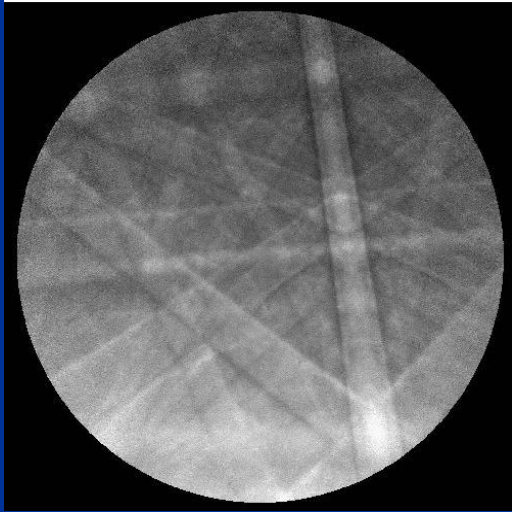
# Detekcija linii - transformata Hougha



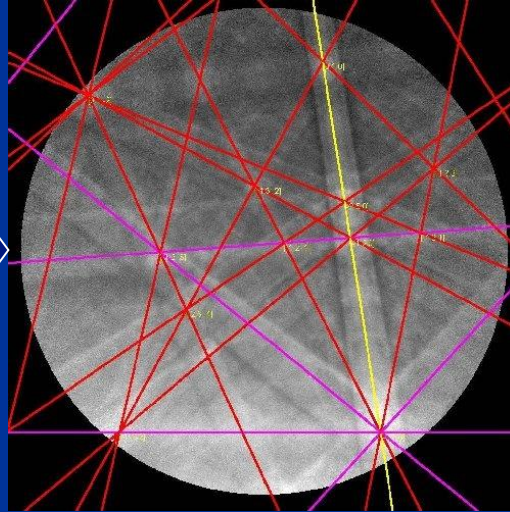


**Detekcja linii za  
pomocą transformaty  
Hougha**

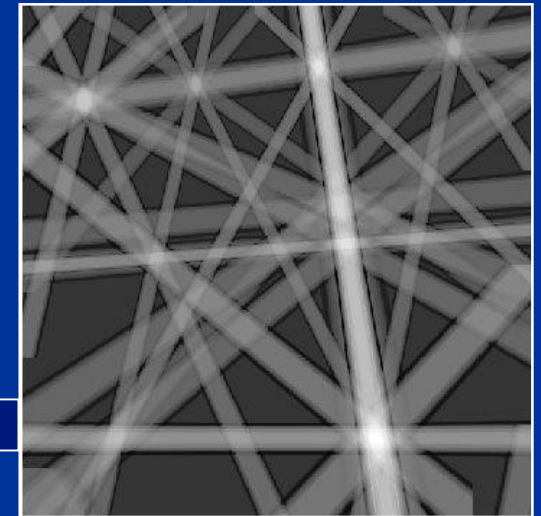
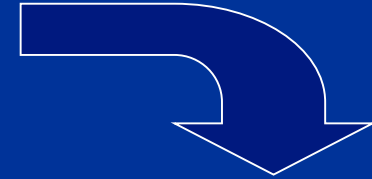




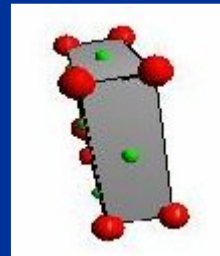
Dyfrakcja z ceramiki  
mullitowej (układ  
ortorombowy) ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ),  
10 kV



Rozwiązanie dyfrakcji  
nałożone na realną dyfrakcję  
orientacja  $\{370\}\langle 7-34\rangle$



Symulacja dyfrakcji – pasma  
Kikuchiego o intensywności  
większej niż 10% najbardziej  
intensywnych pasm

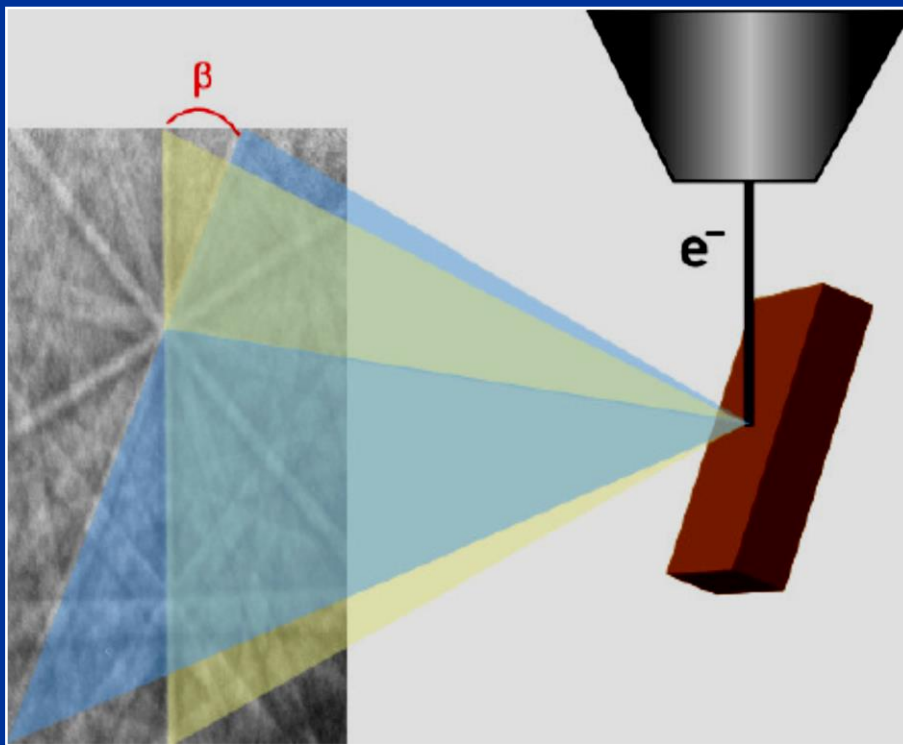


Symulacja orientacji  
kryształitu mullitu



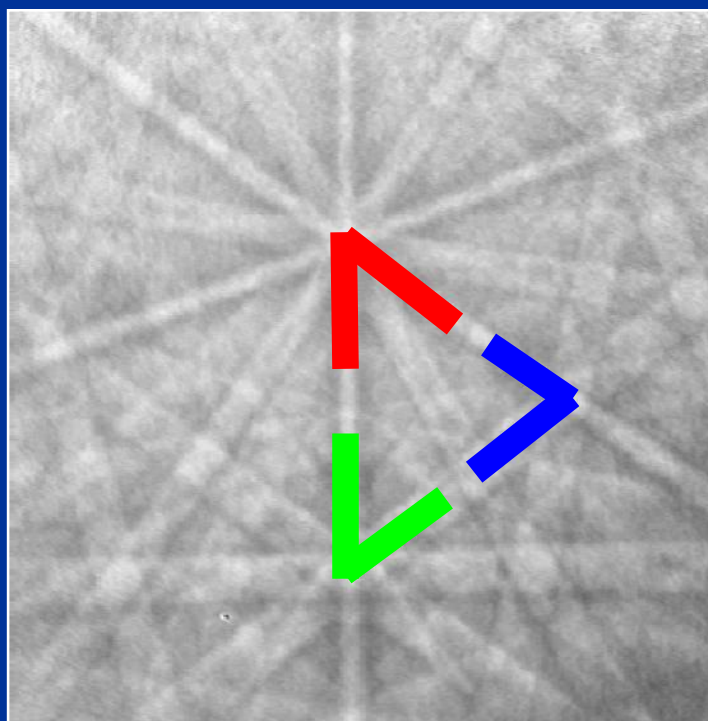
# **EBSD – analiza dyfrakcji**

# Identyfikacja odpowiednich wskaźników $hkl$ przez porównanie wartości kątów pomiędzy dwoma pasmami z wartościami kątów z Tablic



Angle	$(hkl)_1$	$(hkl)_2$	Angle	$(hkl)_1$	$(hkl)_2$
25.2	200	311	64.8	220	$3\bar{1}1$
29.5	111	311	70.5	111	$11\bar{1}$
31.5	220	311	72.5	200	131
35.1	311	$31\bar{1}$	80.0	111	$3\bar{1}\bar{1}$
35.3	111	220	84.8	311	$1\bar{3}1$
45.0	200	220	90.0	111	$2\bar{2}0$
50.5	311	$3\bar{1}\bar{1}$	90.0	200	020
54.7	111	200	90.0	200	$0\bar{2}2$
58.5	111	$31\bar{1}$	90.0	220	$1\bar{1}3$
60.0	220	202	90.0	220	$2\bar{2}0$
63.0	311	$13\bar{1}$			

Zestaw możliwych rozwiązań dla 3 pasm Kikuchiego otrzymamy poprzez porównanie wartości zmierzonych kątów na realnej dyfrakcji z wartościami z Tablic



Angle	(hkl)1	(hkl)2
25.2	200	311
29.5	111	311
31.5	220	311
35.1	311	31-1
35.3	111	220
45.0	200	220
50.5	311	3-1-1
<u>54.7</u>	<u>111</u>	<u>200</u>
<u>58.5</u>	<u>111</u>	<u>31-1</u>
60.0	220	202
63.0	311	13-1
64.8	220	3-11
70.5	111	11-1
<u>72.5</u>	<u>200</u>	<u>131</u>
80.0	111	3-1-1
84.8	311	1-31
90.0	111	2-20
90.0	200	020
90.0	200	022
90.0	220	1-13
90.0	220	2-20

1) Identify the  $hkl$  of the high contrast bands (bands likely to be detected by the Hough transform).

200  
111  
220  
311

2) Determine all of the symmetrically equivalent  $hkl$ 's.

200, 020, 002  
111, 11 $\bar{1}$ , 1 $\bar{1}$ 1,  $\bar{1}$ 11  
220, 2 $\bar{2}$ 0, 202, 20 $\bar{2}$ , 022, 02 $\bar{2}$   
311,  $\bar{3}$ 11, 3 $\bar{1}$ 1, 31 $\bar{1}$ , 131,  $\bar{1}$ 31, 1 $\bar{3}$ 1, 13 $\bar{1}$ , 113,  $\bar{1}$ 13, 1 $\bar{1}$ 3, 11 $\bar{3}$

3) Form all possible pairs.

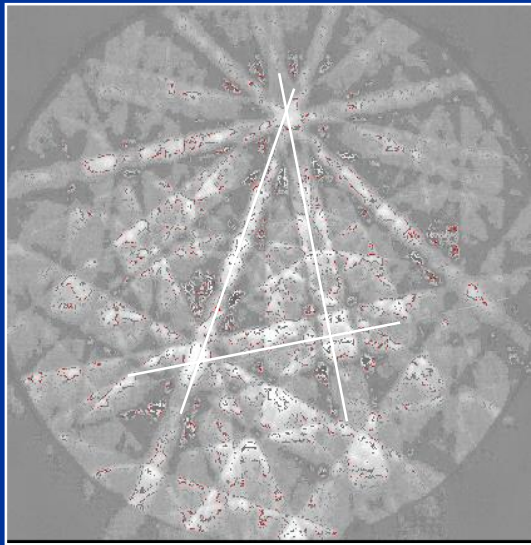
200, 020   200, 002   200, 111   200, 11 $\bar{1}$    ...  
020, 002   020, 111   020, 11 $\bar{1}$    020, 1 $\bar{1}$ 1   ...  
002, 111   002, 11 $\bar{1}$    002, 1 $\bar{1}$ 1   002,  $\bar{1}$ 11   ...  
⋮

4) Calculate the angles between the plane pairs.

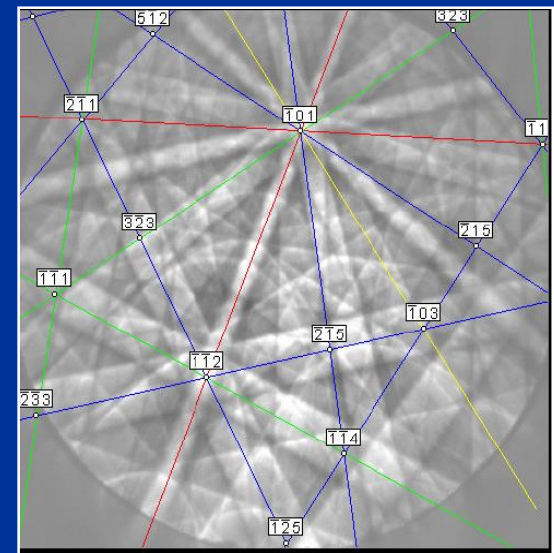
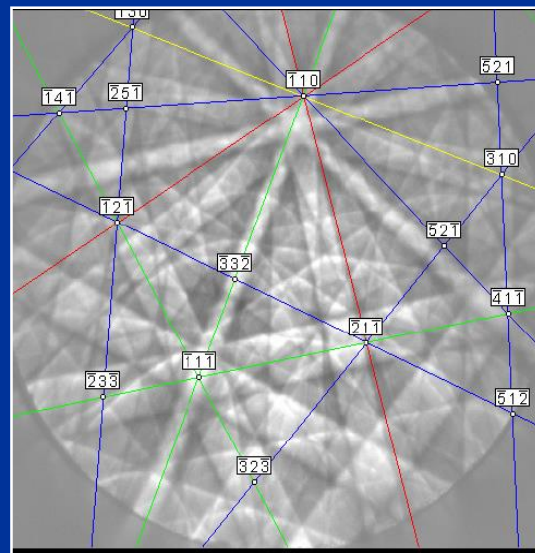
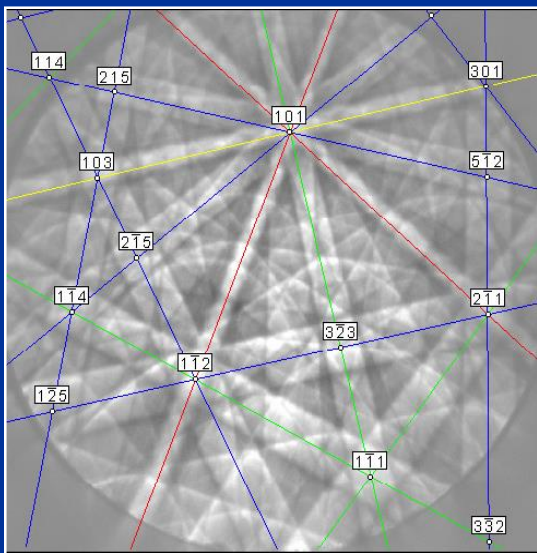
200∠020 = 90°   200∠002 = 90°   200∠111 = 54.7°   200∠11 $\bar{1}$  = 54.7°   ...  
020∠002 = 90°   020∠111 = 54.7°   020∠11 $\bar{1}$  = 54.7°   020∠1 $\bar{1}$ 1 = 54.7°   ...  
002∠111 = 90°   002∠11 $\bar{1}$  = 90°   002∠1 $\bar{1}$ 1 = 90°   002∠ $\bar{1}$ 11 = 90°   ...  
⋮

5) Throw out duplicates and sort.

200∠311 = 25.2°   111∠220 = 35.3°   111∠ $\bar{3}$ 11 = 58.5°   111∠11 $\bar{1}$  = 70.5°  
111∠311 = 29.5°   200∠220 = 45.0°   220∠202 = 60°   200∠131 = 72.5°  
220∠311 = 31.5°   311∠ $\bar{3}$ 11 = 50.5°   311∠13 $\bar{1}$  = 63.0°   111∠ $\bar{3}$ 11 = 80°  
311∠ $\bar{3}$ 11 = 35.1°   200∠111 = 54.7°   220∠3 $\bar{1}$ 1 = 64.8°   ⋮



Dla układu 3 pasm Kikuchiego  
porównanie wartości kątów pomiędzy  
płaszczyznami z realnej dyfrakcji i  
wartościami kątów z Tablic daje  
**3 możliwe rozwiązania**  
Które jest właściwe?

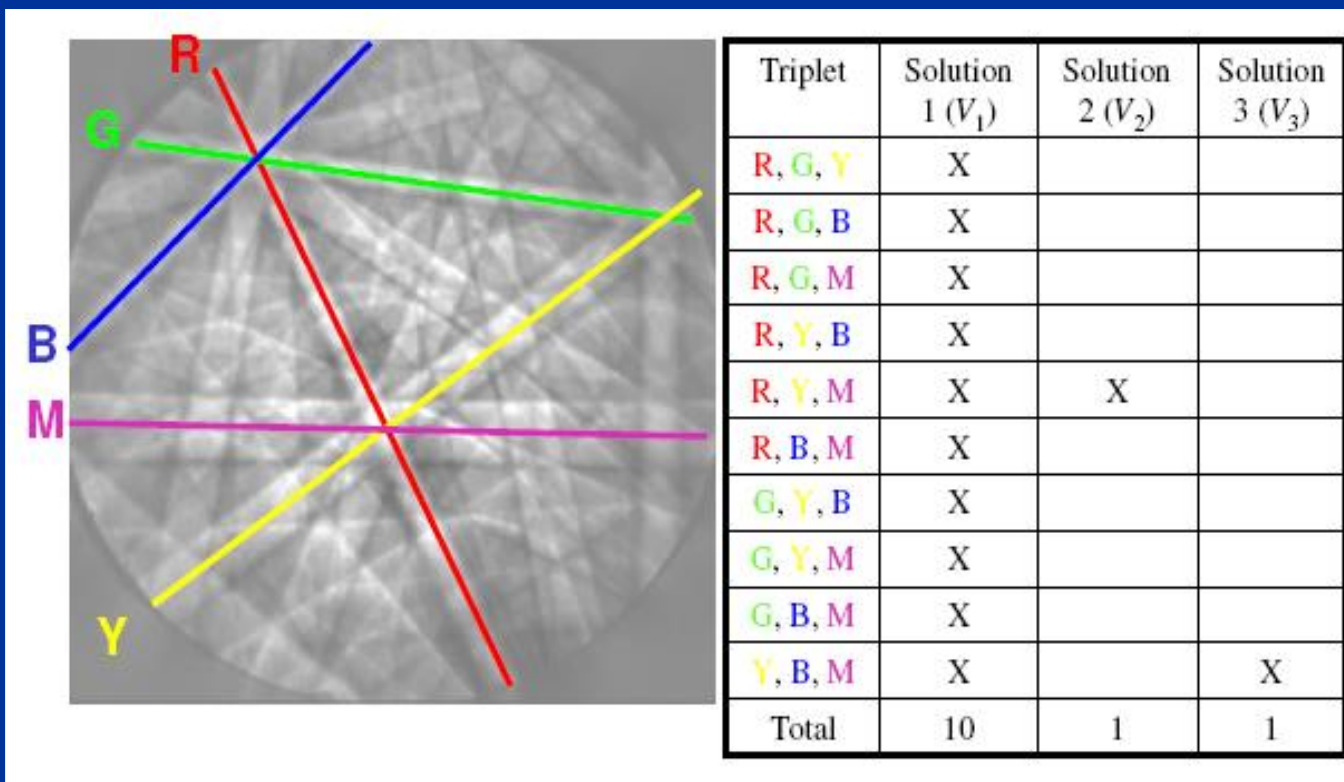


$$\#triplets = \frac{n!}{(n-3)! \cdot 3!}$$

For a given number of bands,  $n$ , used for pattern indexing, the number of band triplets is determined by this formula.

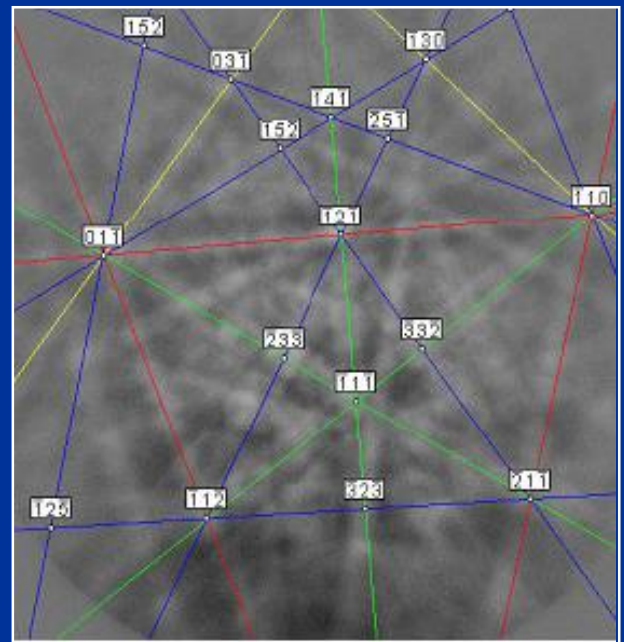
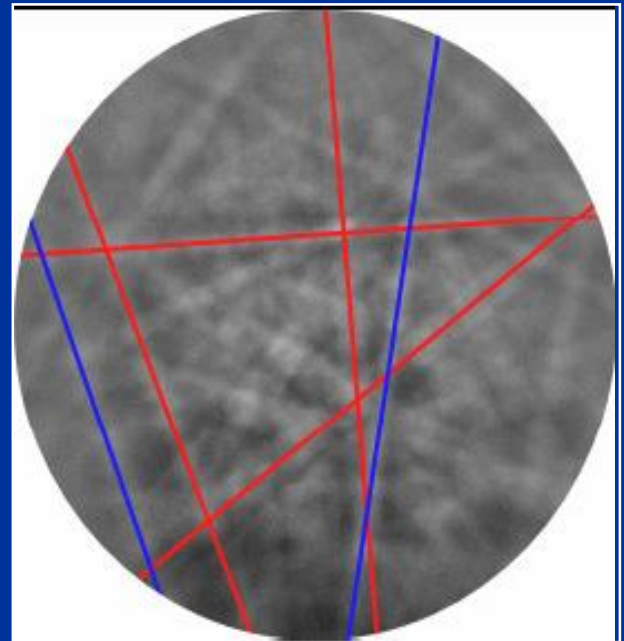
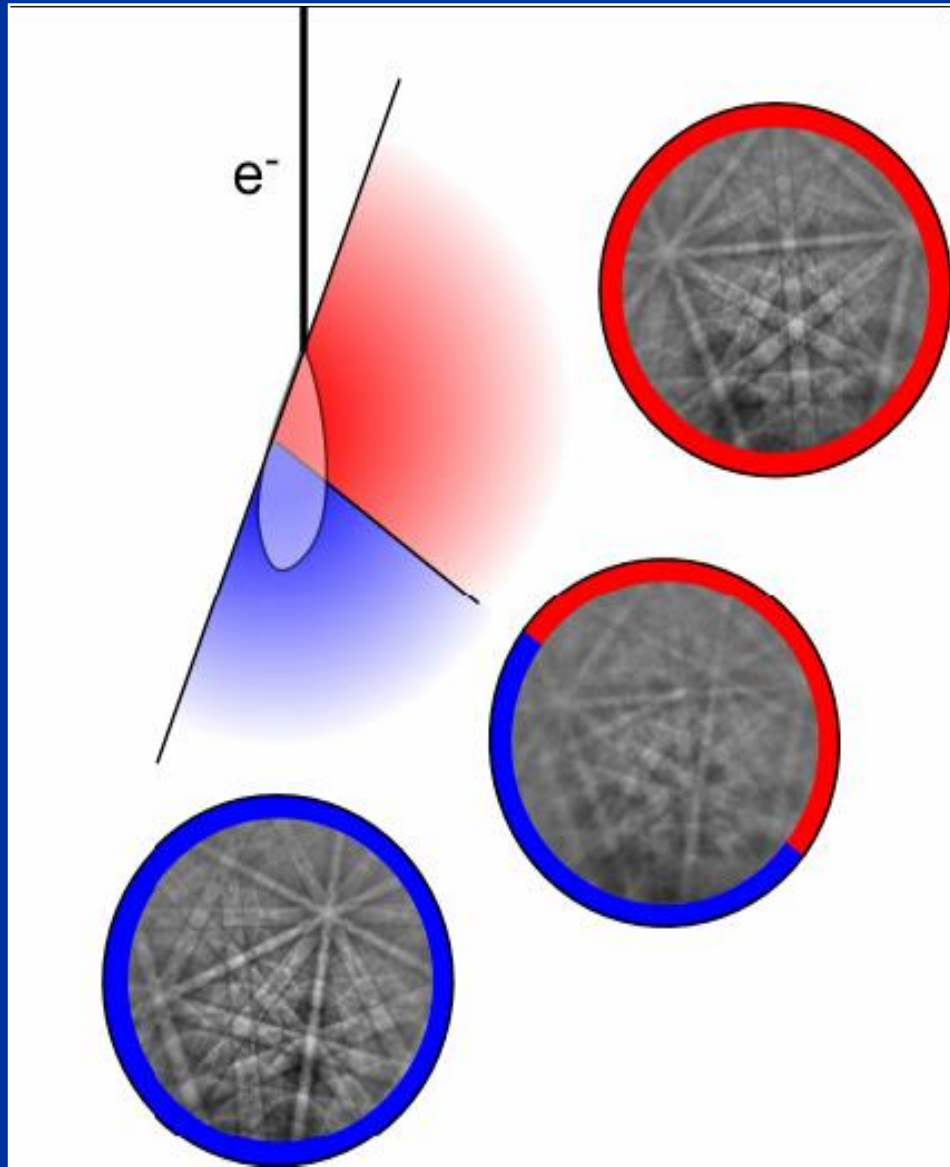
Typically 7 to 9 detected bands are used for automatic indexing.

$n$	$\# triplets$
3	1
4	4
5	10
6	20
7	35
8	56
9	84

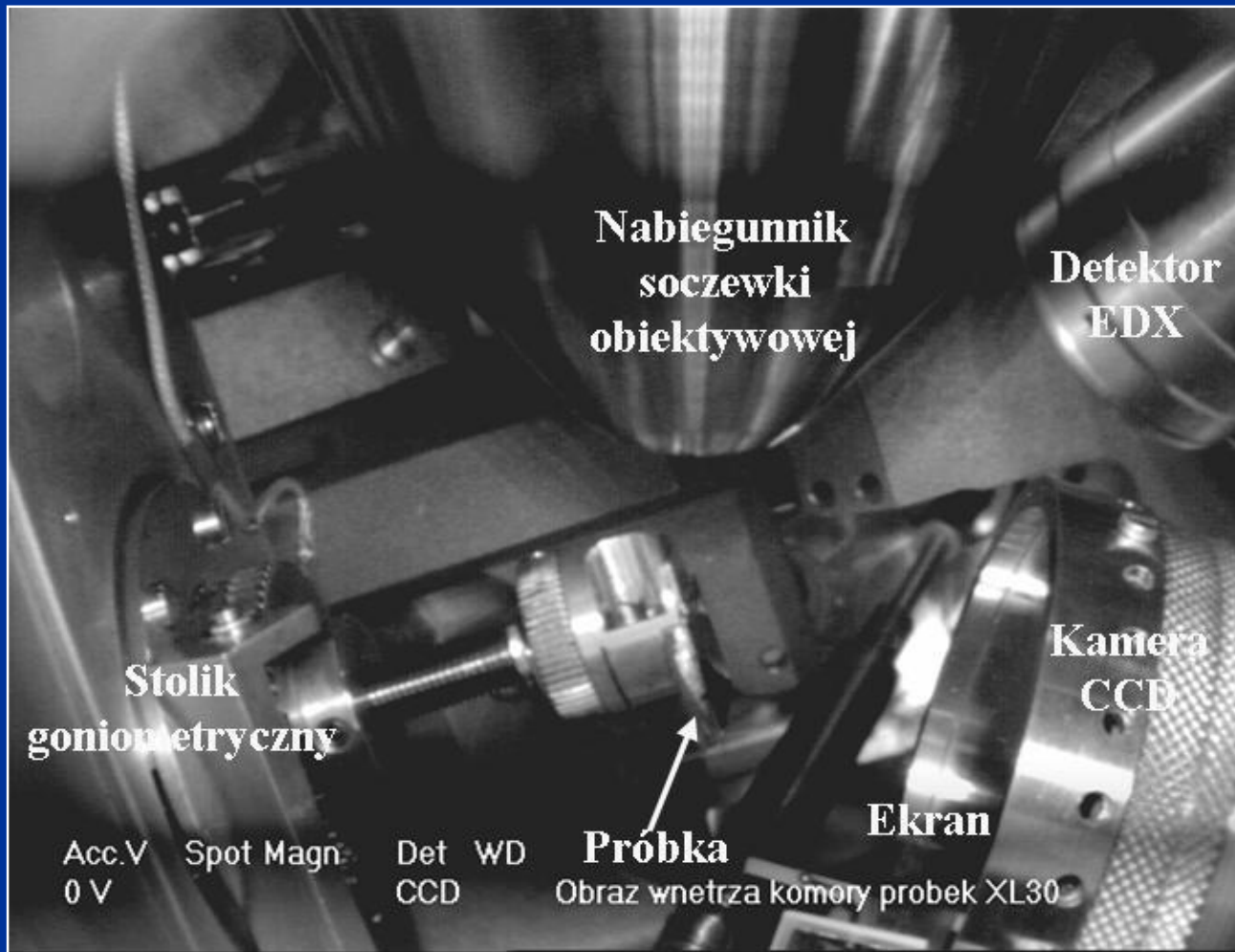


**Dla zestawu 5 pasm – możliwe 10 kombinacji trójkowych**  
**Dla 10 kombinacji trójkowych - tylko rozwiązanie  $V_1$  (10 głosów).**  
**Rozwiązanie  $V_2$  (1 głos) i rozwiązanie  $V_3$  (1 głos)**

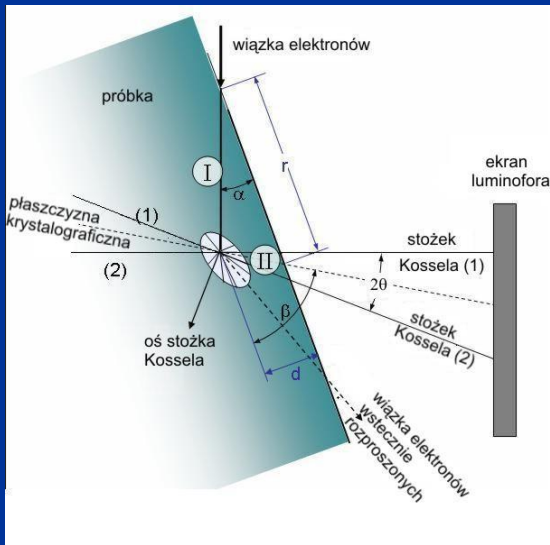




# C-SEM (HV)



# **Kilka uwag praktycznych**

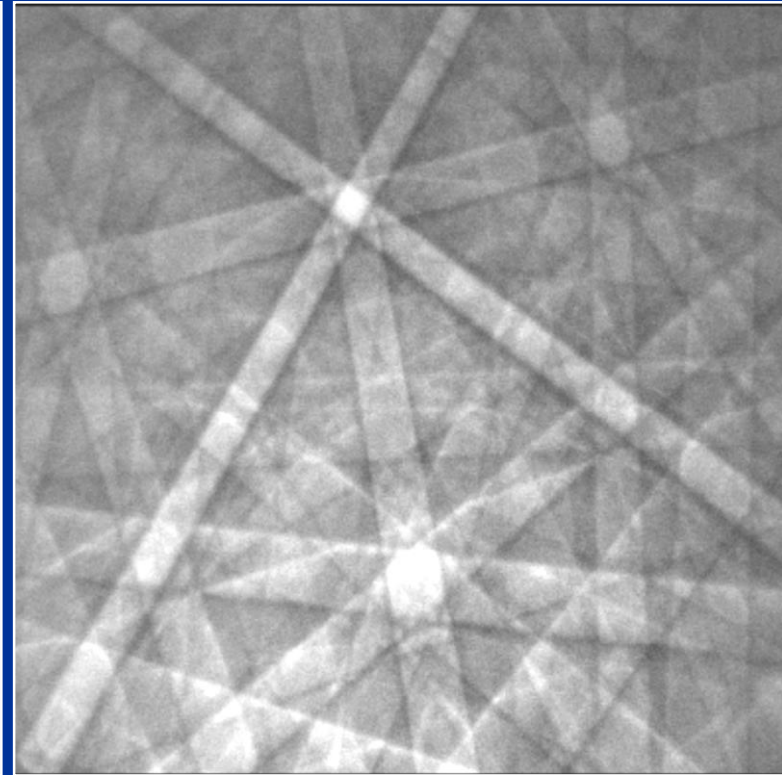
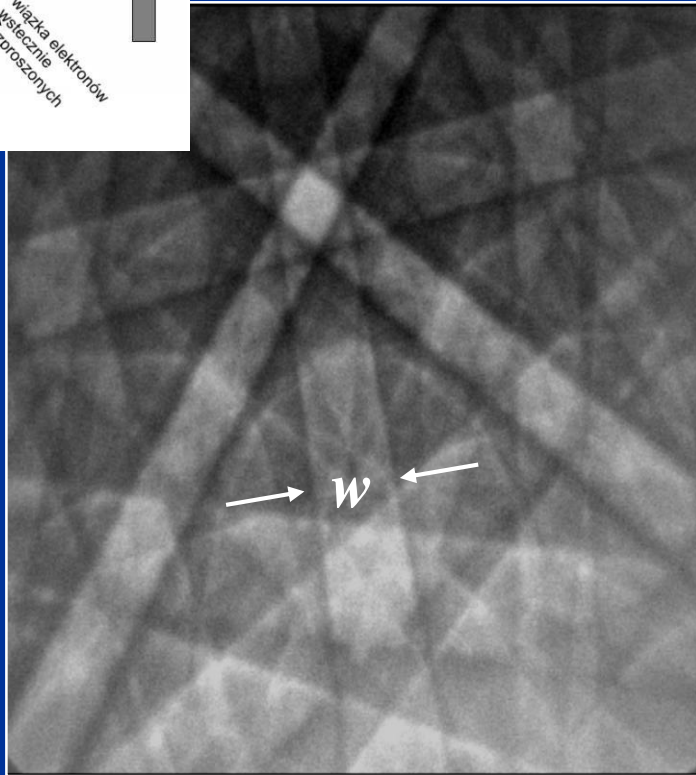


$$w = 2l\theta$$

w - szerokość  
pasma

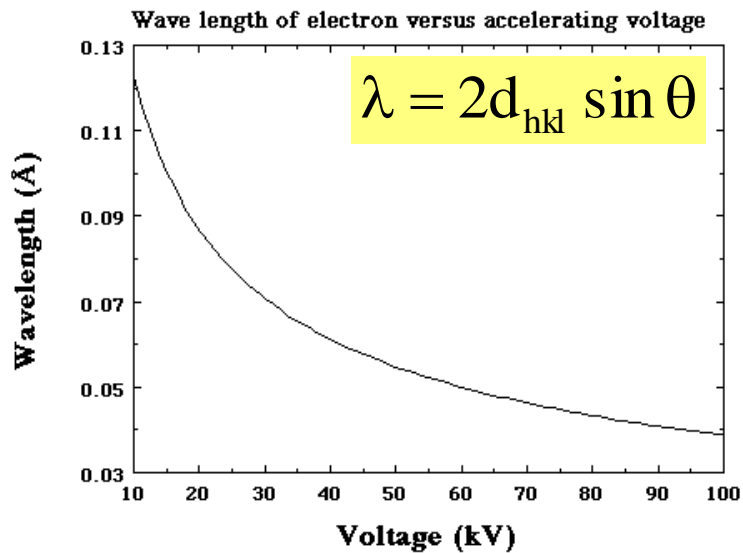
$$n\lambda = 2d_{hkl} \sin\theta = 2d_{hkl} \theta$$

$$w = \frac{l\lambda}{d} \quad \text{gdzie : } l - \text{odległość próbka - ekran}$$



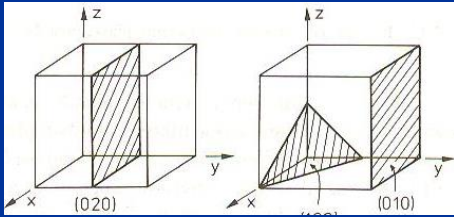
Wpływ napięcia przyspieszającego (zależność w od  $\lambda$ )

EBSD z Si przy 10 kV i 30 kV

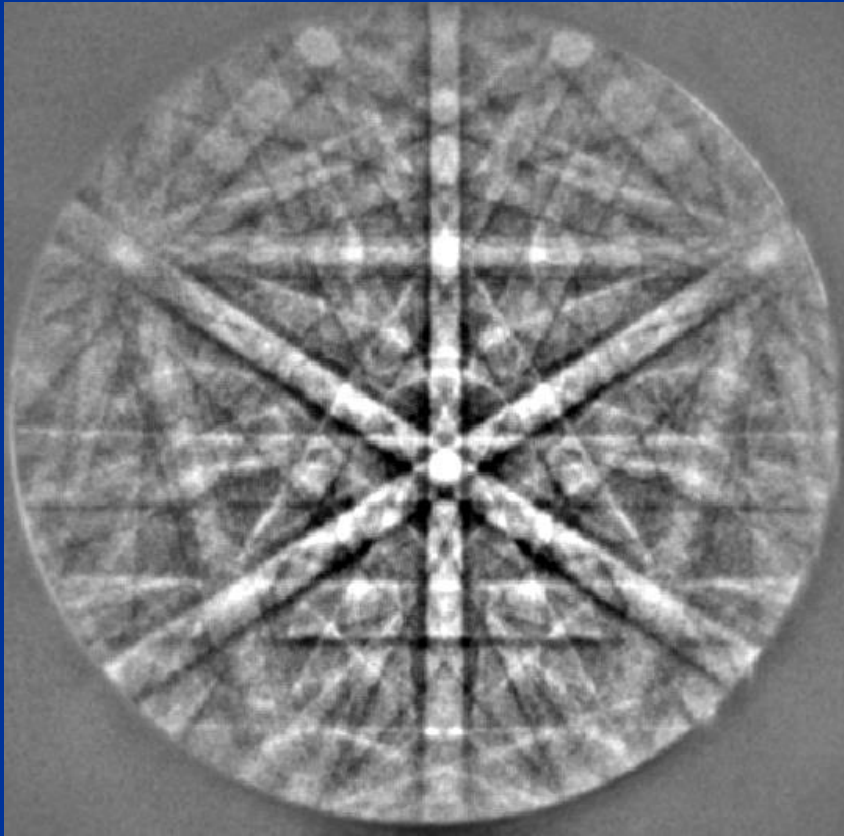


V (kV)	$\lambda$ (nm)
20	0.00859
30	0.00698
40	0.00602
50	0.00536
60	0.00487
70	0.00448
80	0.00418
90	0.00392
100	0.00370
200	0.00251
300	0.00197
400	0.00164
500	0.00142
600	0.00126
700	0.00113
800	0.00103
900	0.00094
1000	0.00087
2000	0.00050
4000	0.00028

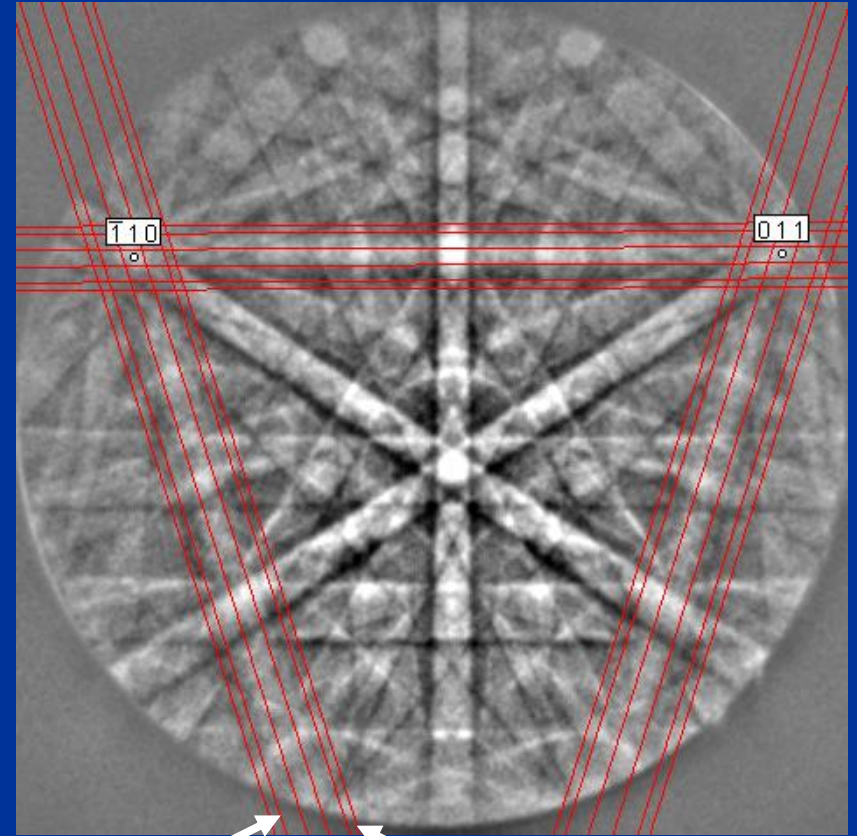
Szerokość pasm Kikuchiego jest odwrotnie proporcjonalna do odległości międzyplaszczynowej „d”



<b>Si 1-1-1</b>	<b>3.135 Å</b>
<b>Si 3-3-3</b>	<b>1.045 Å</b>
<b>Si 4-4-4</b>	<b>0.784 Å</b>



(zależność „w” od „d”)



3-3-3 1-1-1 4-4-4

**Natężenie pasma Kikuchiego pochodzącego od danej płaszczyzny  $hkl$ :**

$$I_{hkl} = \left[ \sum_i f_i(\Theta) \cos 2\pi(hx_i + ky_i + lz_i) \right]^2 + \left[ \sum_i f_i(\Theta) \sin 2\pi(hx_i + ky_i + lz_i) \right]^2$$

gdzie:  $f_i(\Theta)$  atomowy czynnik rozpraszania dla elektronów,  
 $(x_i, y_i, z_i)$  cząstkowe koordynaty dla atomu  $i$  w komórce elementarnej.

**Zarejestrowaną dyfrakcję porównujemy z dyfrakcją wyliczoną na podstawie powyższego równania, uważając aby uwzględniać tylko te płaszczyzny które uginają elektrony, gdyż tylko one biorą udział w tworzeniu dyfrakcji.**

# Czynnik strukturalny

Umożliwia przewidywanie obecności lub nieobecności refleksów dyfrakcyjnych od różnych płaszczyzn krystalograficznych a także proporcje ich intensywności. Opisywany przez niego efekt spowodowany jest interferencją fal cząstkowych ugiętych na poszczególnych atomach komórki elementarnej.

$$\mathbf{A}_{hkl} \propto \mathbf{F}_{hkl} \mathbf{A}_0$$

$A_{hkl}$  – amplituda wiązki elektronowej ugiętej na płaszczyźnie  $hkl$

$A_0$  – amplituda wiązki elektronowej padającej

$F_{hkl}$  – czynnik strukturalny dla płaszczyzny  $hkl$

$$\begin{aligned} \mathbf{F}_{hkl} = & f_1(\theta) \exp[-2\pi i(hu_1 + kv_1 + lw_1)] \\ & + f_2(\theta) \exp[-2\pi i(hu_2 + kv_2 + lw_2)] \\ & + \dots \\ & + f_n(\theta) \exp[-2\pi i(hu_n + kv_n + lw_n)] \end{aligned}$$



gdzie:

$Z$  – liczba atomowa,

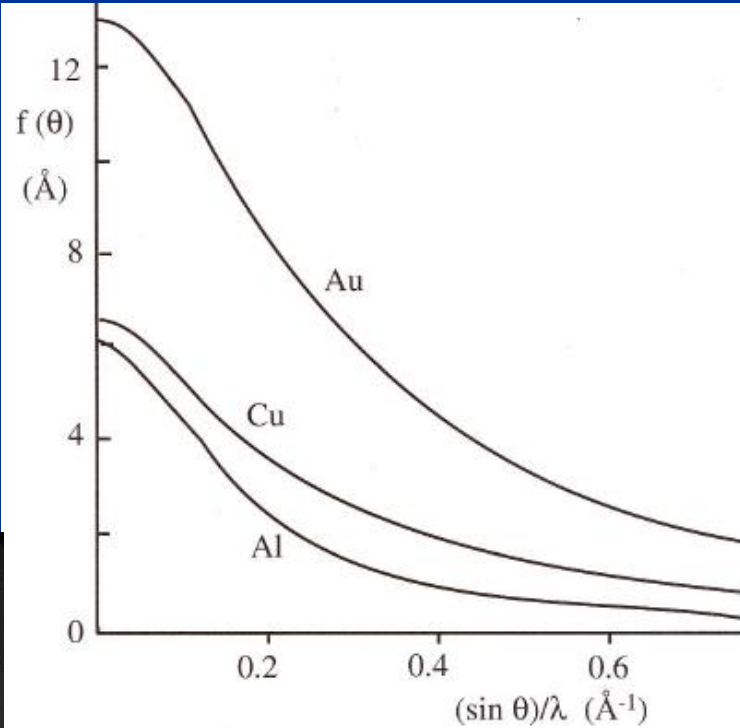
$f_x$  – czynnik atomowy na rozpraszanie promieniowania rentgenowskiego,

$Z-f_x$  – rozpraszanie elektronu na elektronach powłok

$\lambda/\sin\theta$  – opisuje rozpraszanie Rutherforda elektronu na jądrze atomowym

(L.REIMER)

$$f(\theta) = \frac{m_0 e^2}{2h^2} \left( \frac{\lambda}{\sin\theta} \right)^2 (Z - f_x)$$



Al

a

Ta

b

$$\mathbf{F}_{hkl} = \sum_{j=1}^n f_j(\theta) \exp[-2\pi i(hu_j + kv_j + lw_j)]$$

Dla n liczby atomów w komórce elementarnej

W pewnych przypadkach płaszczyzny nie uginają elektronów – dyfrakcja nie zachodzi – *forbidden reflections*

# Czynnik strukturalny

100 dla fcc (Au)

– atomy Au rozmieszczone są na narożach komórki elementarnej i na środkach płaszczyzn.

$n = 4$  (bo cztery atomy tworzą komórkę elementarną zajmując pozycje:  $[0,0,0]$ ,  $[0,1/2,1/2]$ ,  $[1/2,0,1/2]$ ,  $[1/2,1/2,0]$ )

$$F_{hkl} = f_1(\theta) \begin{pmatrix} \exp[-2\pi i(1 \cdot 0 + 0 \cdot 0 + 0 \cdot 0)] \\ + \exp[-2\pi i(1 \cdot 0 + 0 \cdot \frac{1}{2} + 0 \cdot \frac{1}{2})] \\ + \exp[-2\pi i(1 \cdot \frac{1}{2} + 0 \cdot 0 + 0 \cdot \frac{1}{2})] \\ + \exp[-2\pi i(1 \cdot \frac{1}{2} + 0 \cdot \frac{1}{2} + 0 \cdot 0)] \end{pmatrix} = \begin{pmatrix} \exp[0] \\ + \exp[0] \\ + \exp[-\pi i] \\ + \exp[-\pi i] \end{pmatrix} = 0$$

$$F_{hkl} = \sum_{j=1}^n f_j(\Theta) \cos 2\pi(hx_n + ky_n + lz_n) - i \sum_{j=1}^n f_j(\Theta) \sin 2\pi(hx_n + ky_n + lz_n)$$

$x, y, z$  – położenia atomu  $n$

$hkl$  – wskaźniki Millerowskie płaszczyzn

## $F_{hkl}$ dla FCC

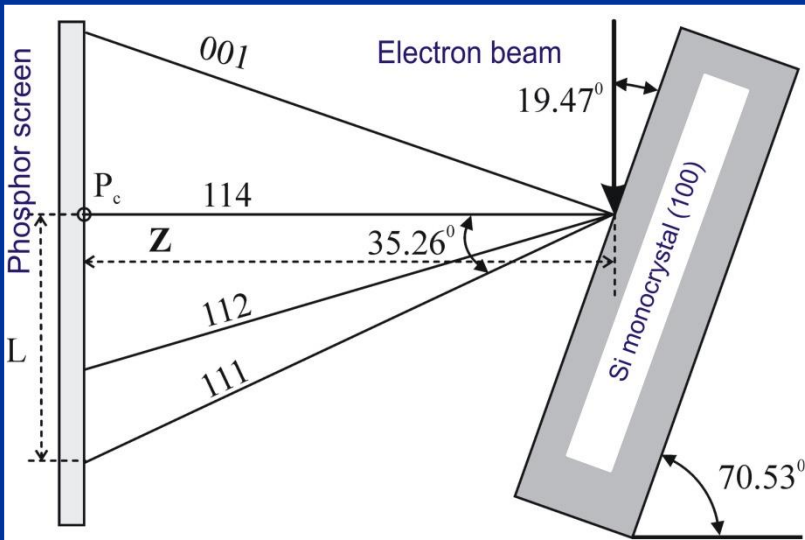
warunkiem wystąpienia refleksu w sieci FCC jest, aby

wskaźniki „h k l” były tego samego rodzaju (parzyste lub nieparzyste),

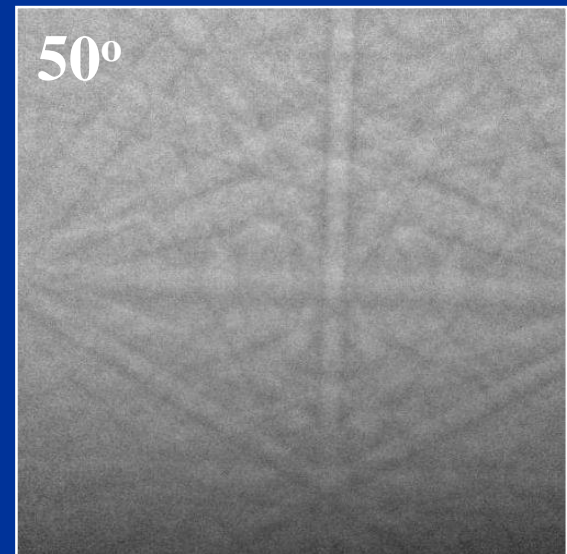
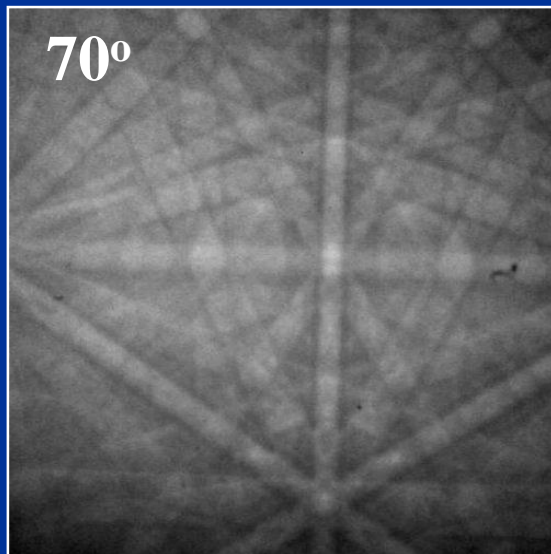
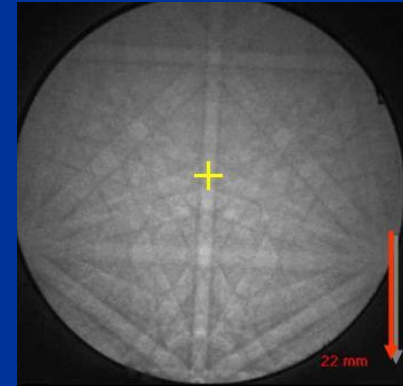
wtedy suma dwóch wskaźników będzie zawsze parzysta (w tym przypadku  $F_{hkl} = 4f$ ), dlatego eliminujemy płaszczyzny: 100, 110, 210, 211...., a zostają płaszczyzny: 111, 200, 220, 311, 222, 331, 422, 333, 440, 531, 442, 533

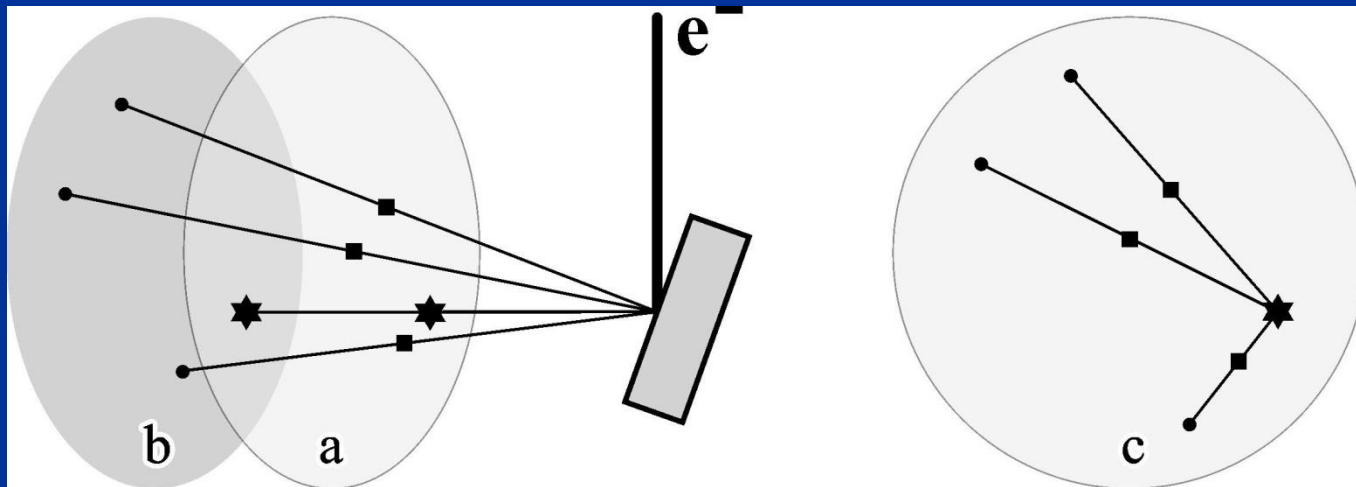
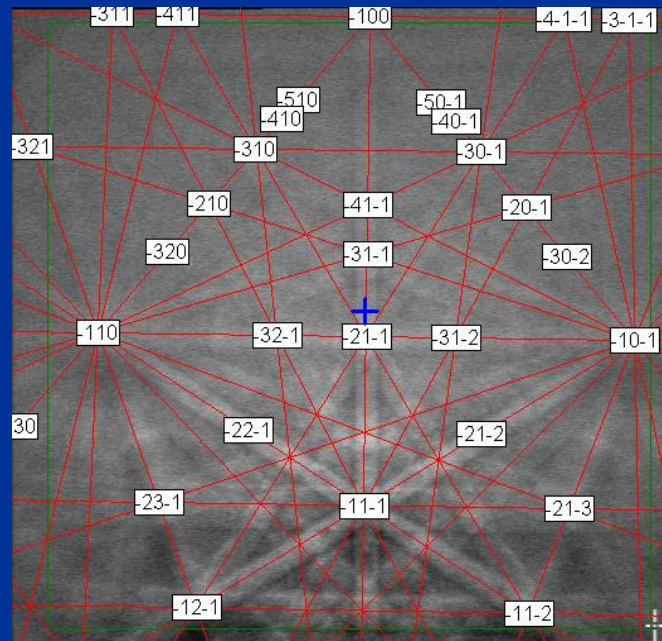
Reflectors	No.	d-spacing, Å	Intensity %
{111}	4	2.338	100.0
{200}	3	2.025	69.4
{220}	6	1.432	27.6
{311}	12	1.221	18.2
{222}	4	1.169	16.2
{331}	12	0.929	9.0
{422}	12	0.827	6.6

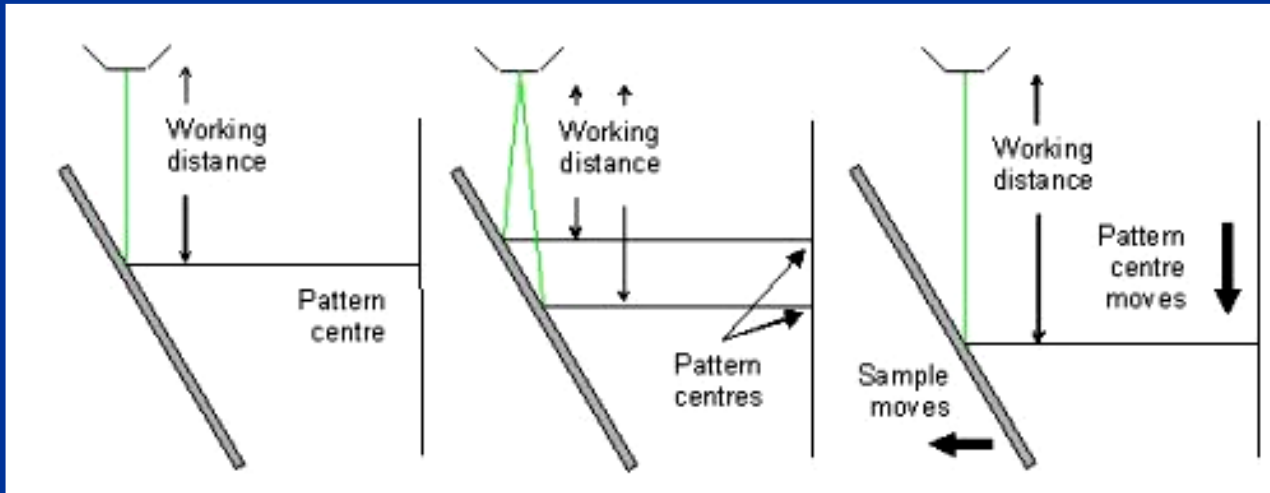
# *Pattern Centre* – dlaczego pochylamy próbkę o $70^\circ$ ?



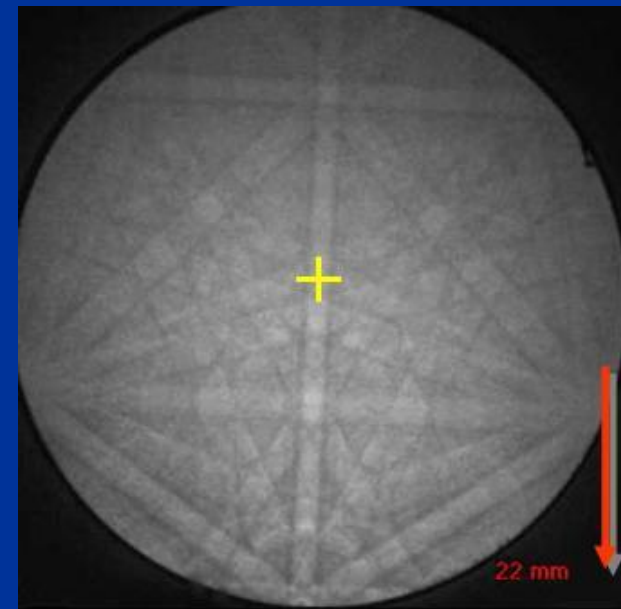
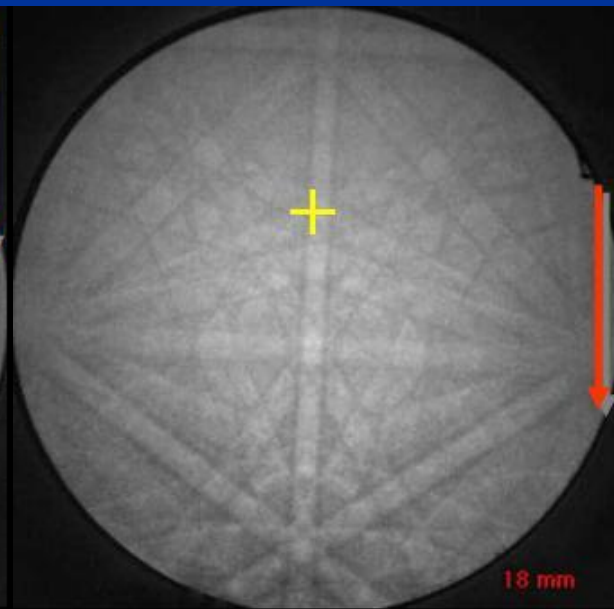
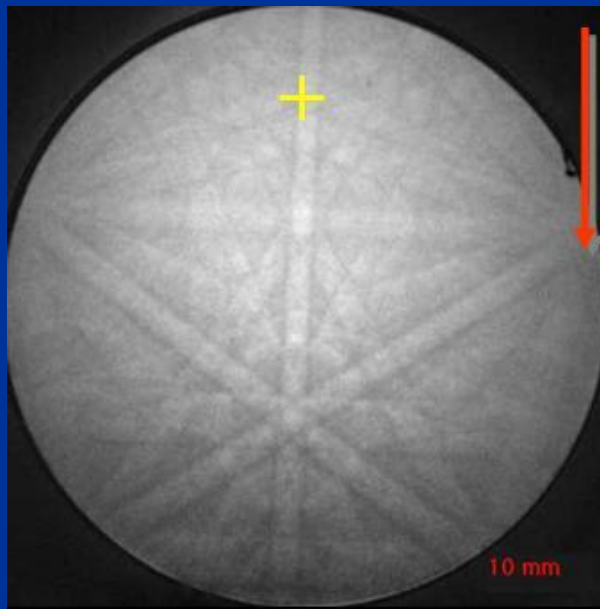
*Pattern Centre (PC)*  
Punkt na ekranie  
luminoforu leżący  
najbliżej źródła  
elektronów  
generującego dyfrakcje

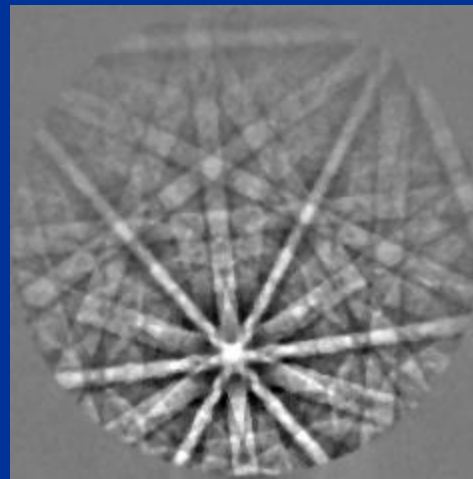
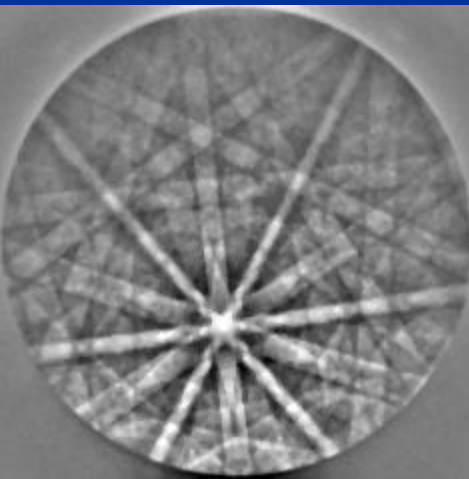
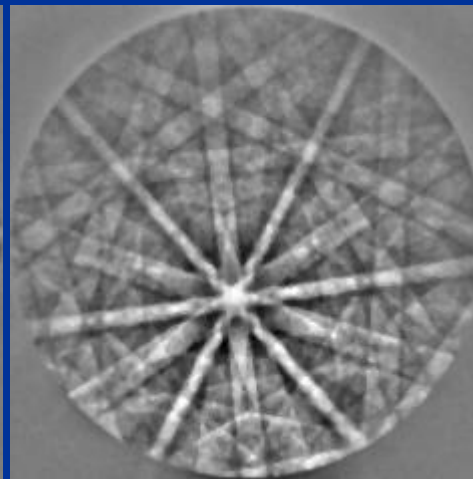
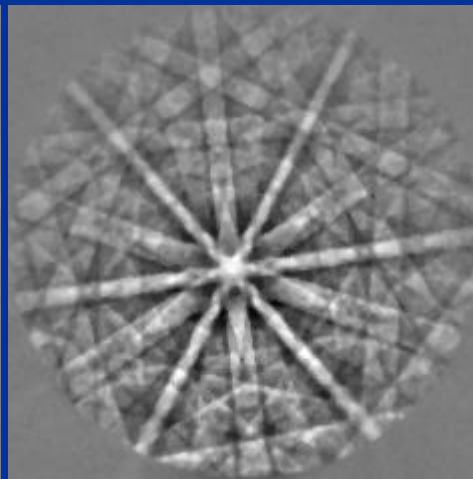
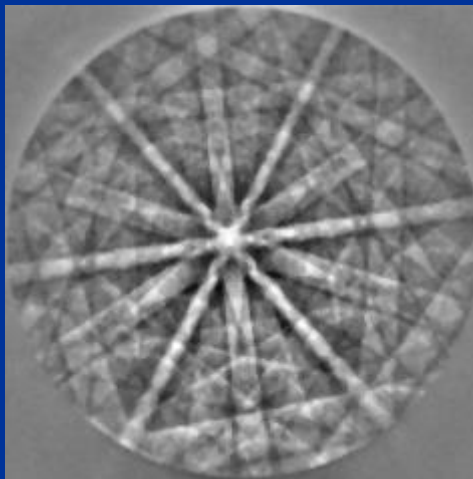
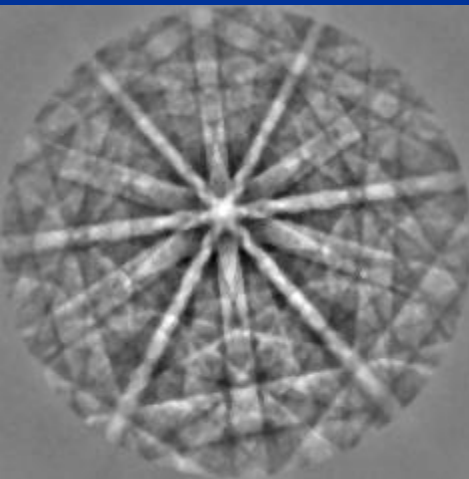
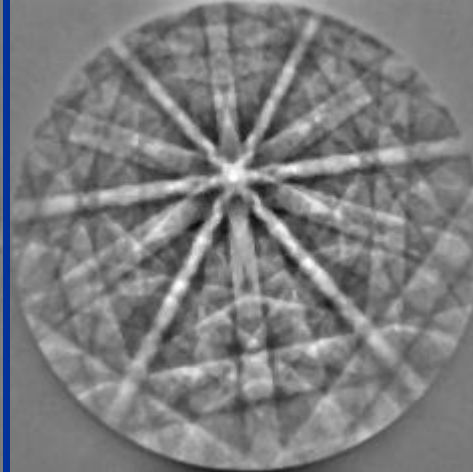
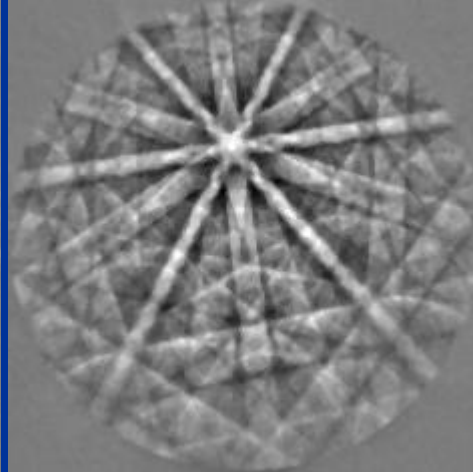
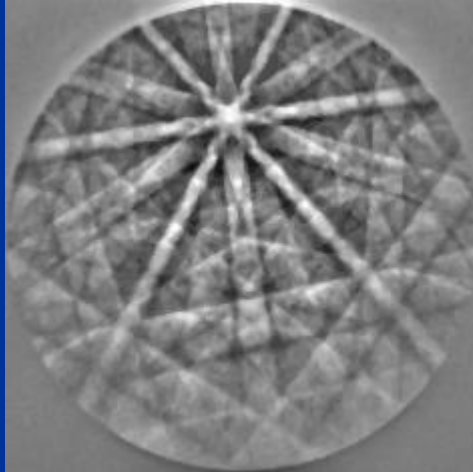
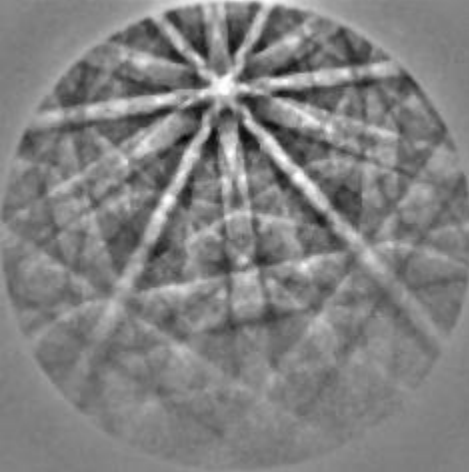






Gdy zmienia się  
WD (*Working Distance*) ulega  
zmianie położenie  
PC (*Pattern Centre*)

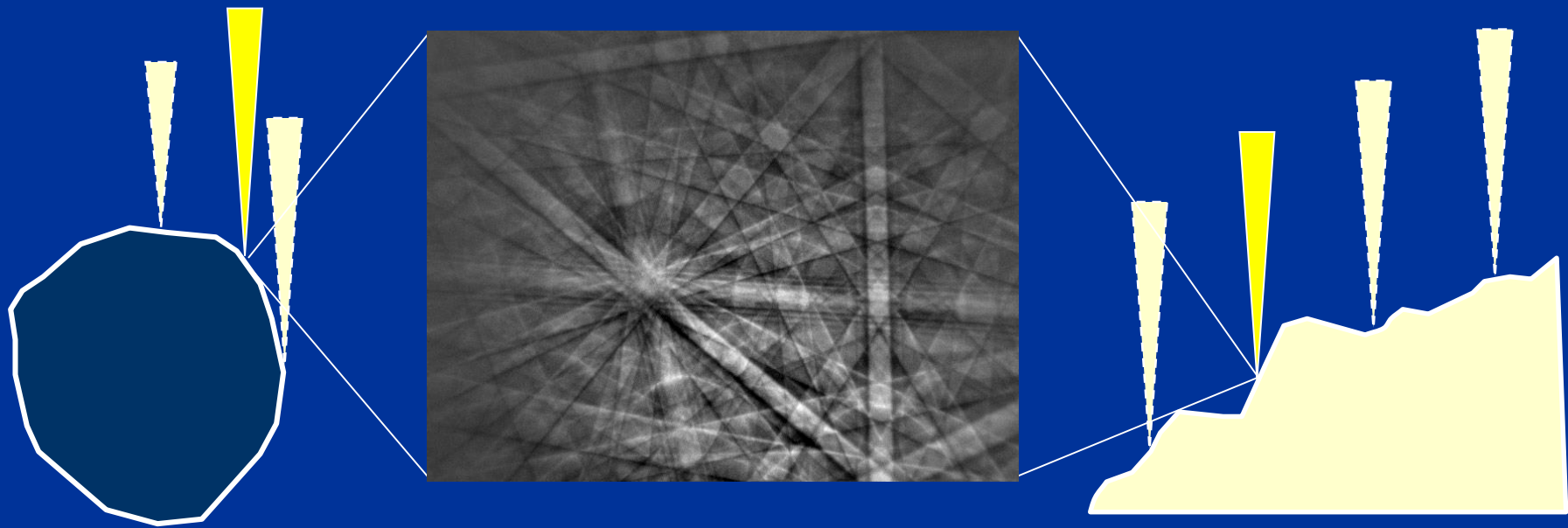




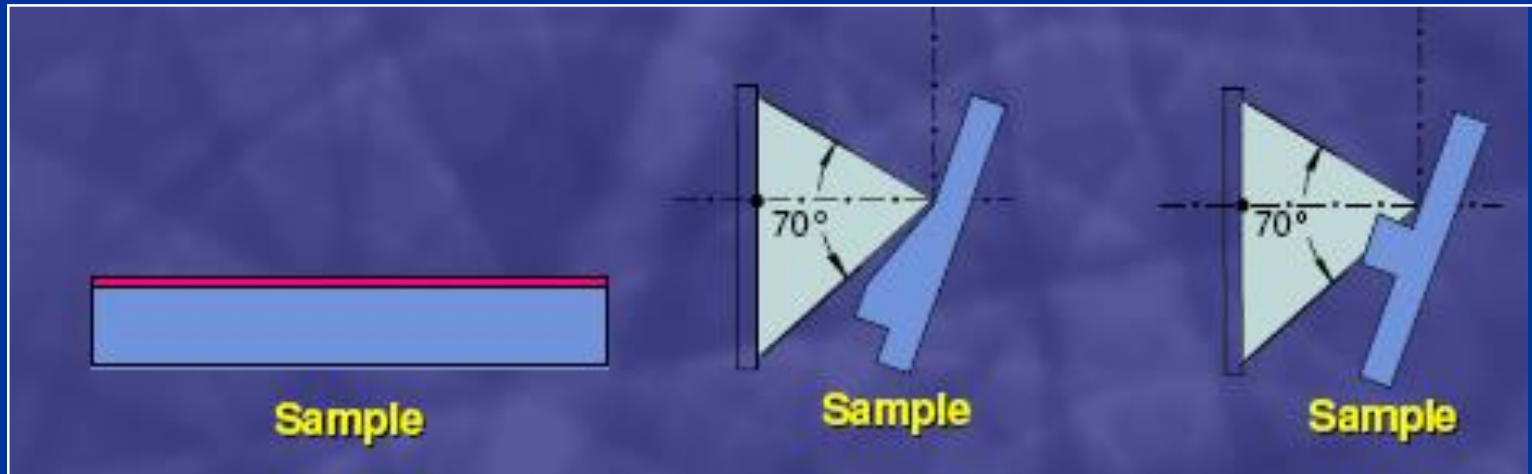
**Zmiana położenia PC (*Pattern Centre*) wraz ze zmianą WD (*Working Distance*)**

**Austenit – WD od 7 mm do 25 mm  
(zmiana co 2 mm)**



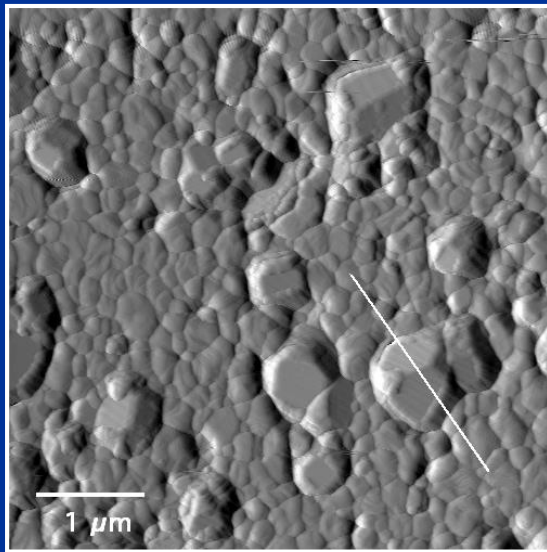


# Przygotowanie próbek do badań

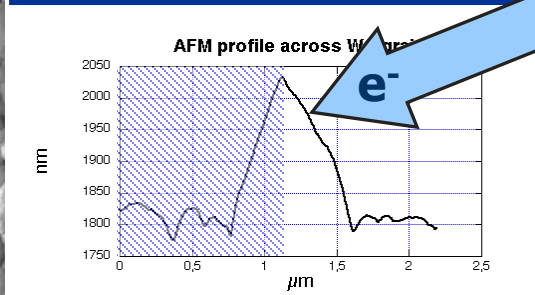


Ponieważ informacja pochodzi z głębokości  $\sim 50$  nm, dlatego konieczna jest:

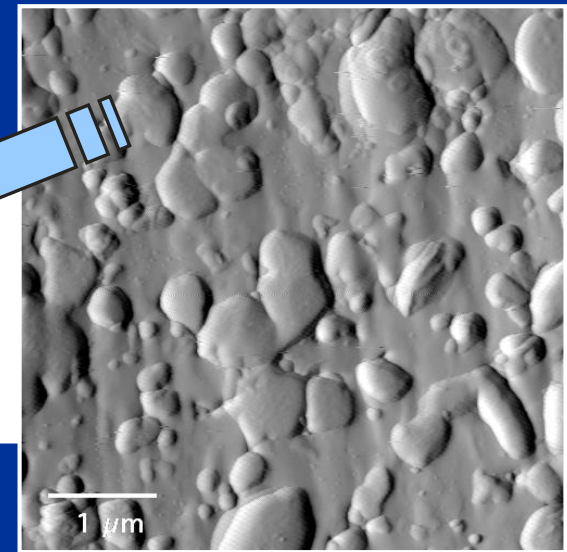
- Ciągłość struktury krystalograficznej aż do powierzchni próbki
- Gładka powierzchnia bez wypukłości („*shadowing*”)
- Brak warstwy zdeformowanej, tlenkowej, napylonej



**Trawienie termiczne**  
**Y-TZP (0.2 - 0.3 μm)/WC**



**AFM profile across WC**  
**grain**

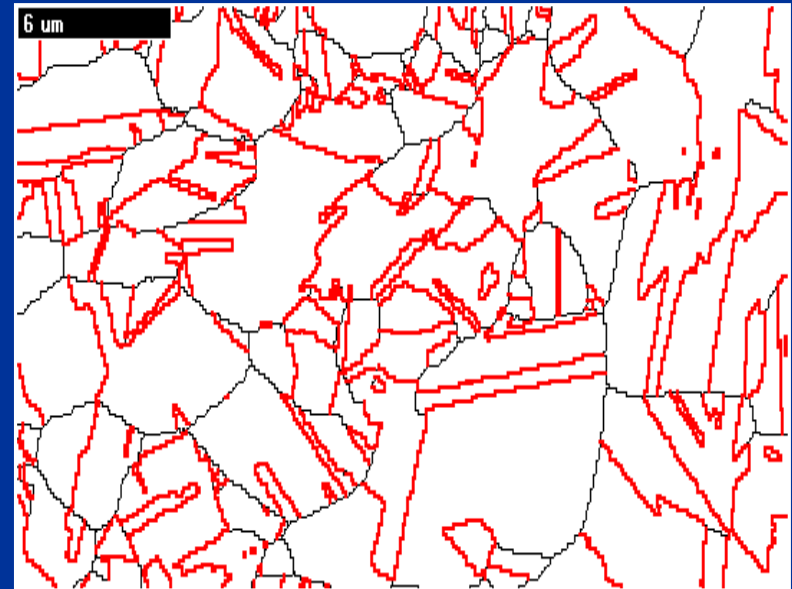


**Polerowanie koloidalną**  
**krzemionką**  
**Y-TZP (0.2 - 0.3 μm)/WC**

# Przykłady

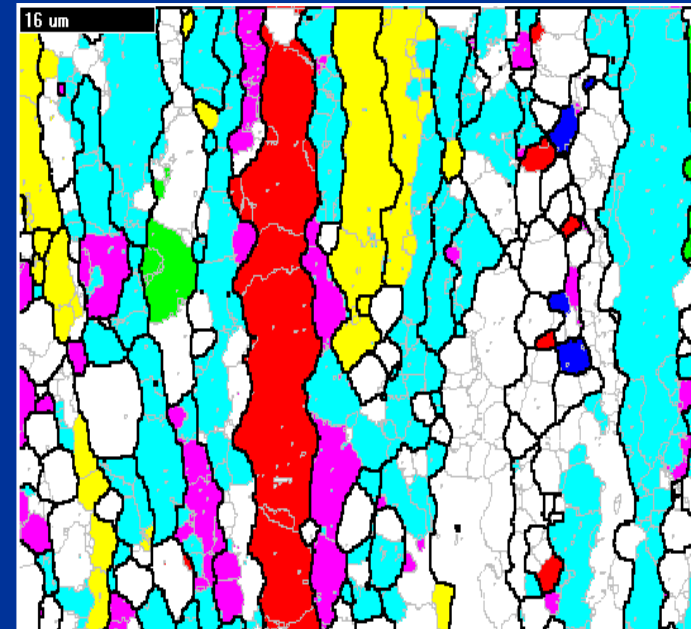
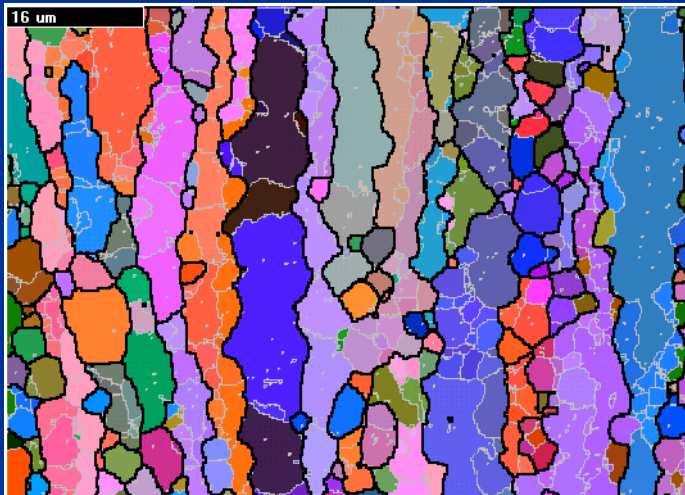


**Granice międzyziarnowe  
w mosiądzu  
Mapa orientacji**

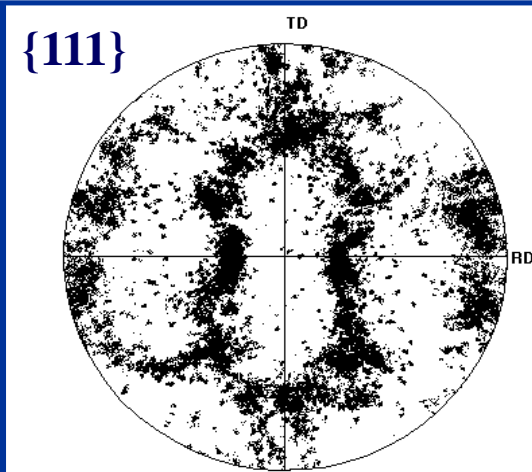


**Granice CSL (Coincidence Site  
Lattice) w mosiądzu  
 $\Sigma 3$  (granice bliźniacze) – 67%**

# Analiza tekstury



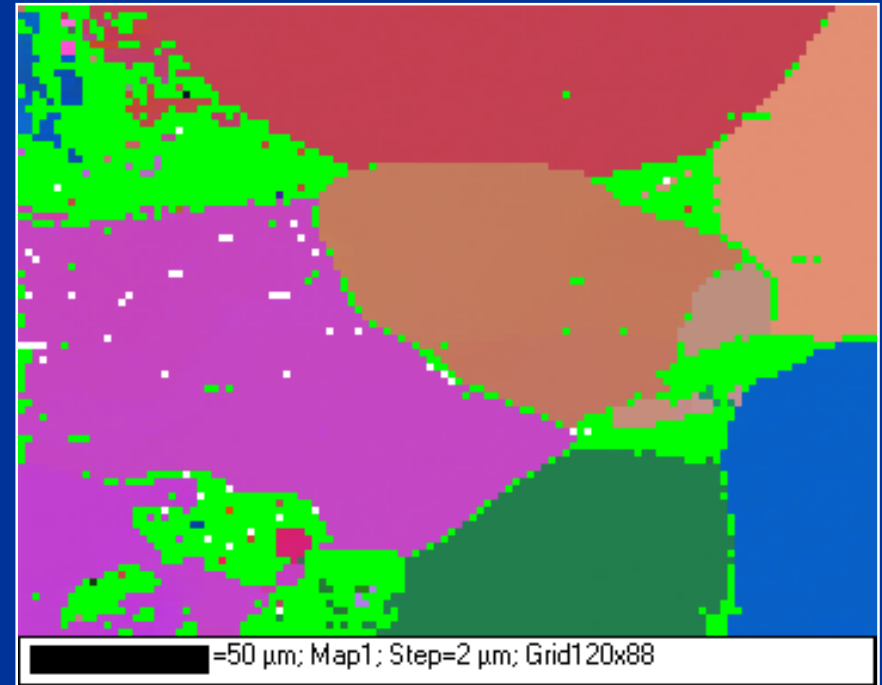
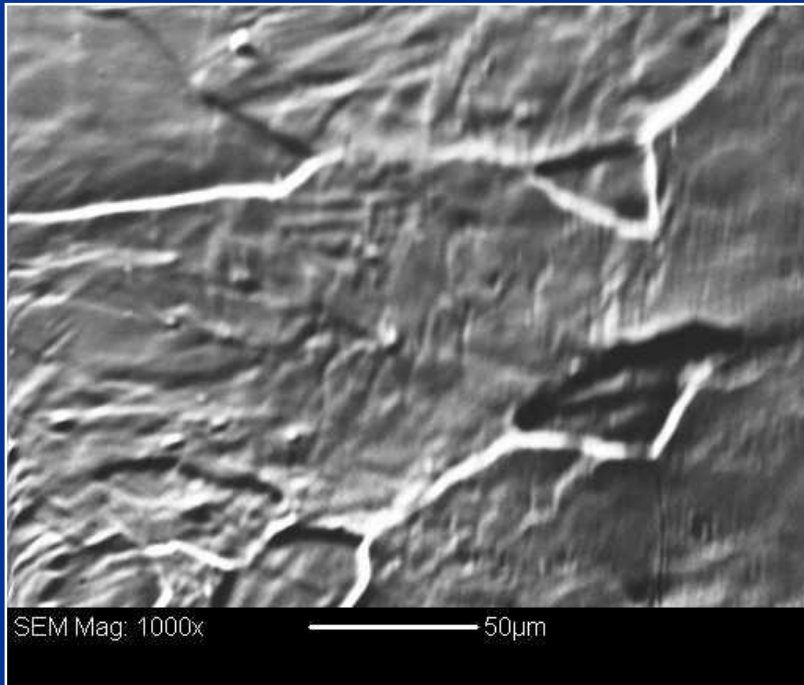
- Brass  $\{011\}\langle 112\rangle$
- Copper  $\{112\}\langle 111\rangle$
- Cube  $\{001\}\langle 100\rangle$
- Goss  $\{011\}\langle 100\rangle$
- P  $\{011\}\langle 122\rangle$
- S  $\{123\}\langle 634\rangle$



Stop AA5182 walcowany na gorąco

*Prof. J.Humphries, UM*

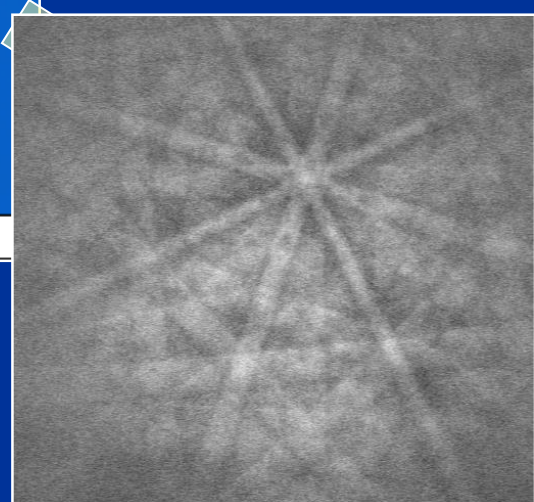
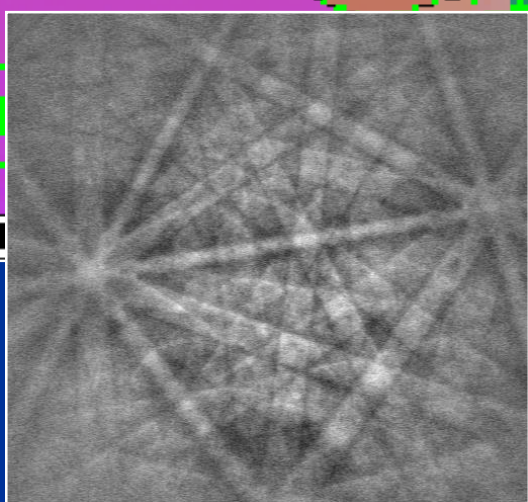
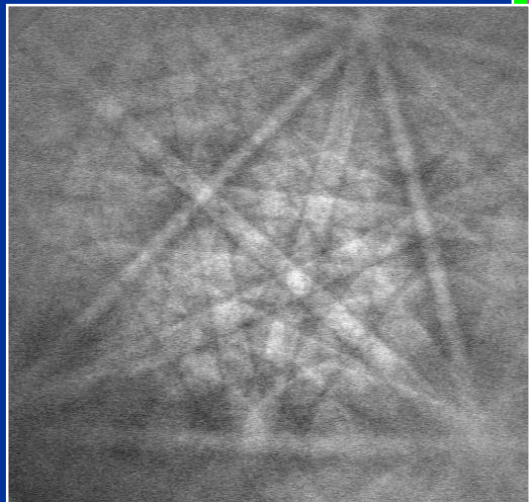
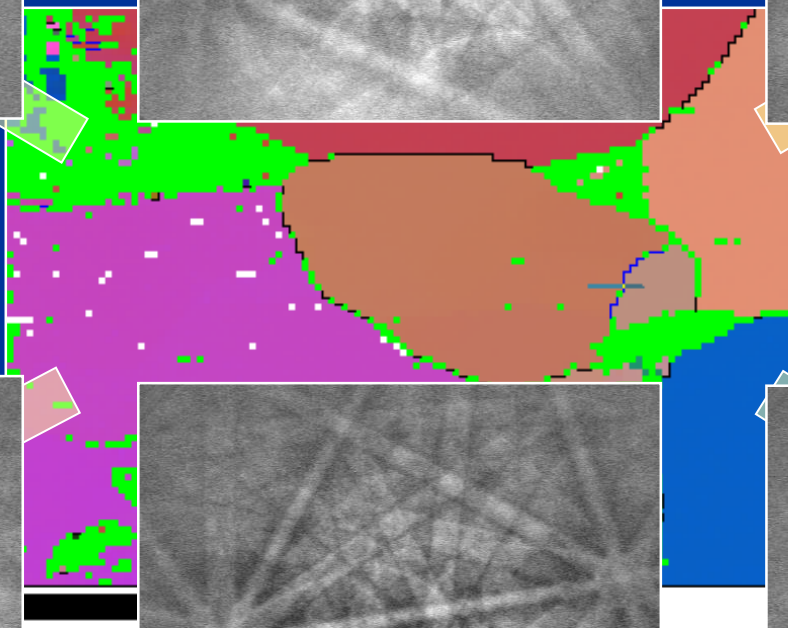
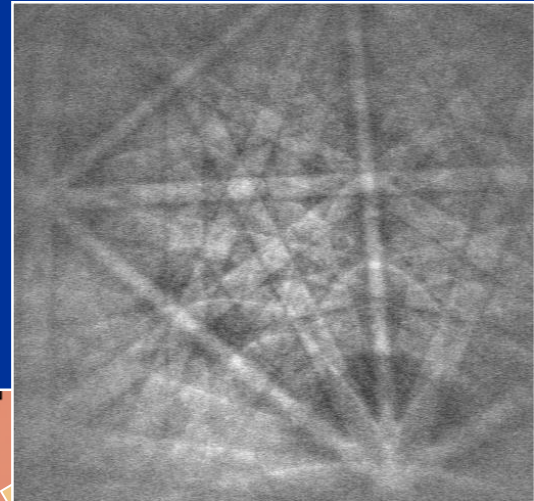
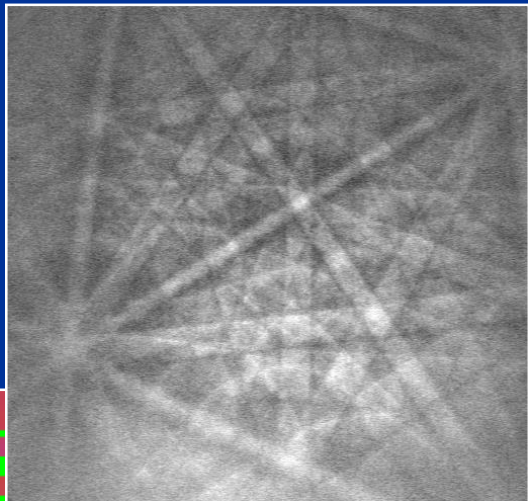
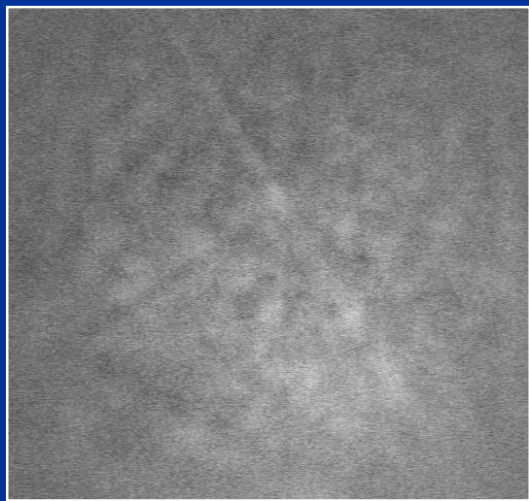
# Fe0.05wt%C 725°C 554h



**Dodatkowy detektor elektronów rozproszonych do przodu (forward scattered electrons) do obrazowania miejsc na próbce, z których rejestrowane są dyfrakcje**

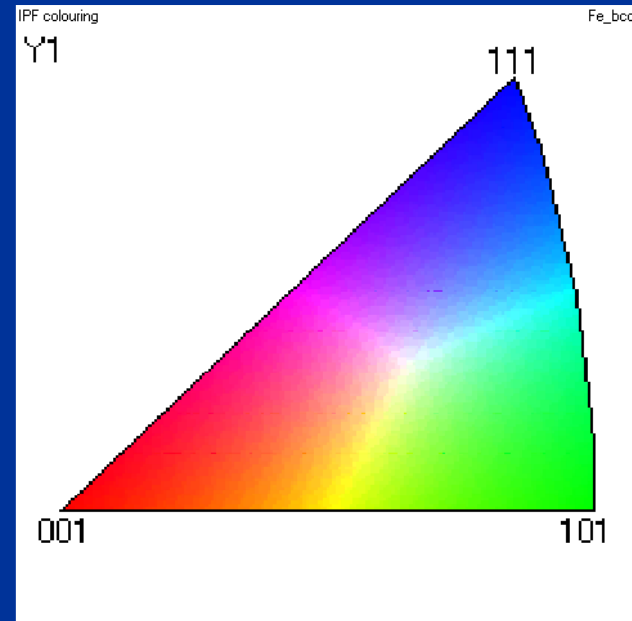
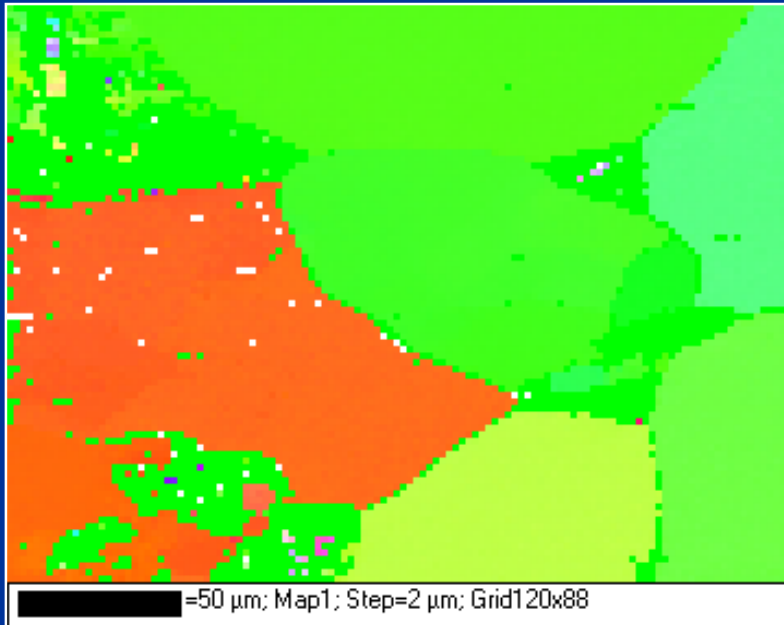
**Mapa zmian orientacji**

**All Euler Map**



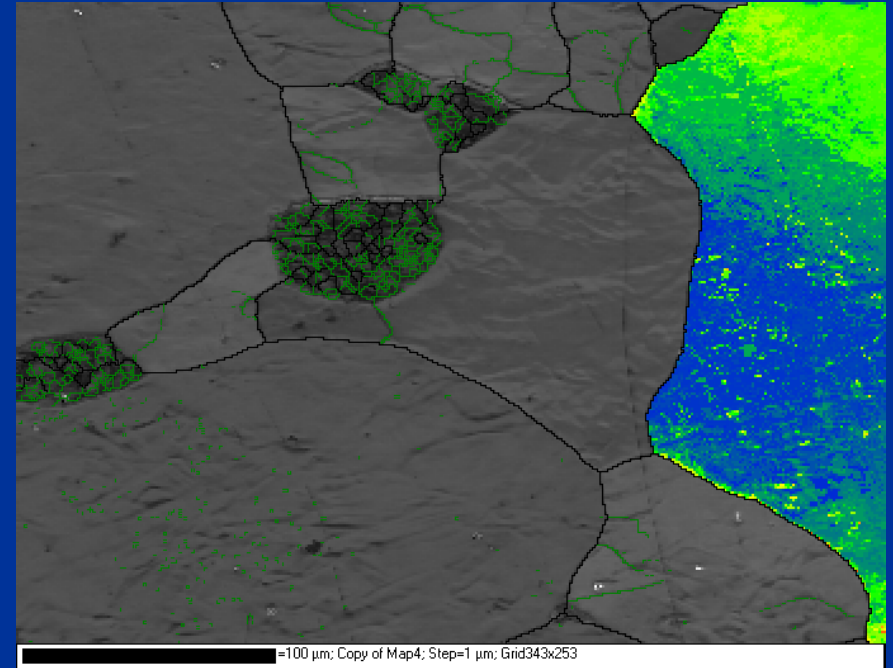
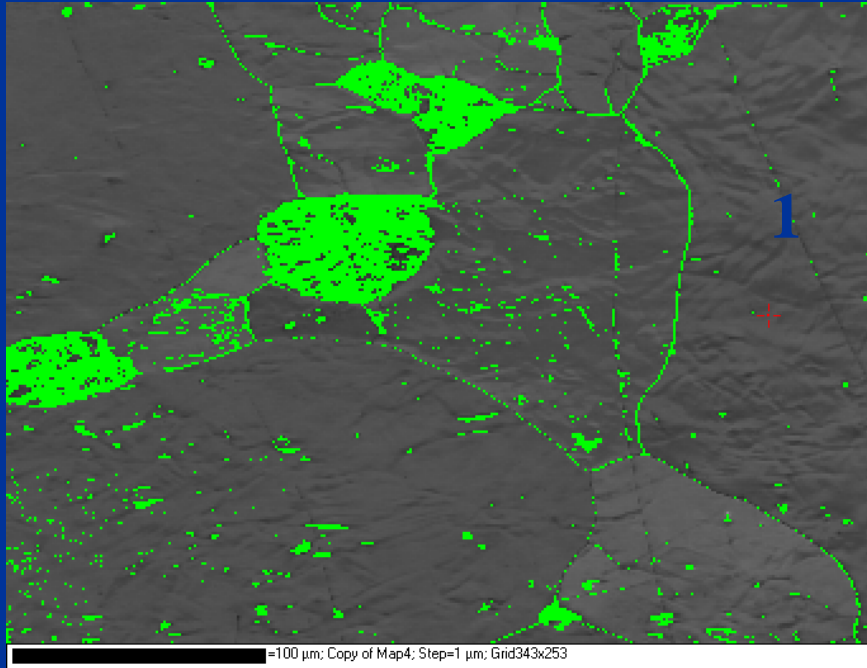


# Fe0.05wt%C 725°C 554h



**Mapa IPF (*Inverse Pole Figure Map*)**  
**Kodowanie kolorów wskazuje kierunek kryształu**  
**równoległy do kierunku referencyjnego**

# Fe0.05wt%C 725°C 554h



Mapa BC (*Band Contrast*)

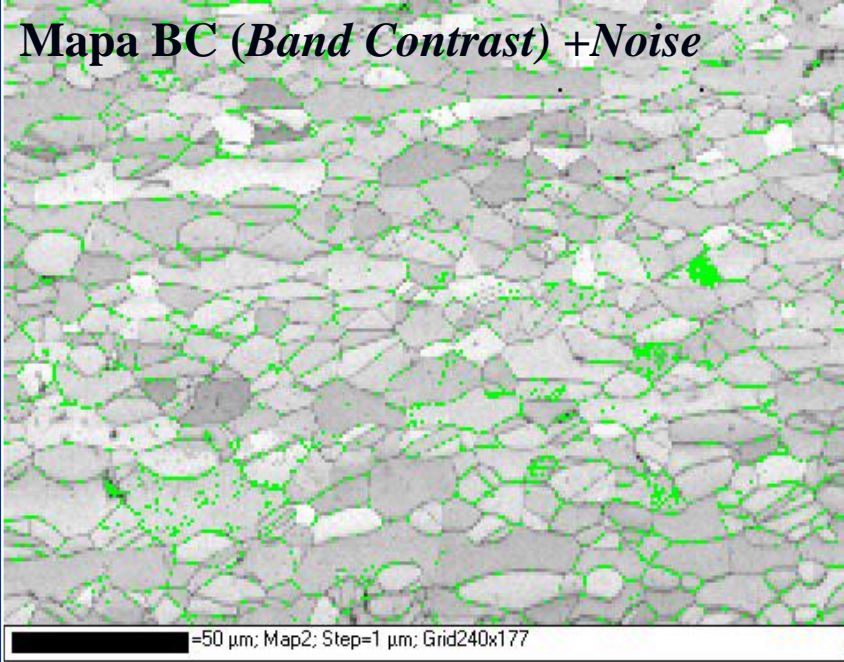
Mapa TC (*Texture Component Map*)

Idealna orientacja ziarna 1:

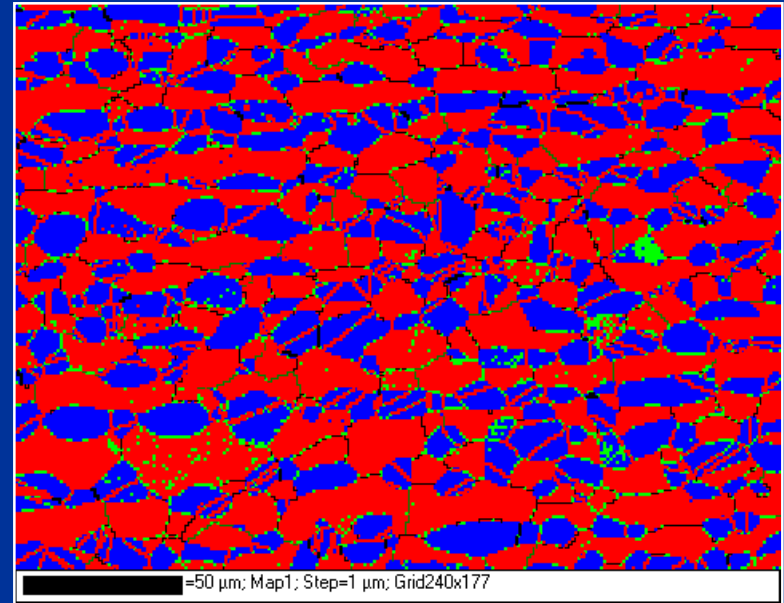
$$\varphi_1=130.6^\circ, \Phi=40.1^\circ, \varphi_2=67.0^\circ,$$

odchylenie od idealnej orientacji  $1^\circ$

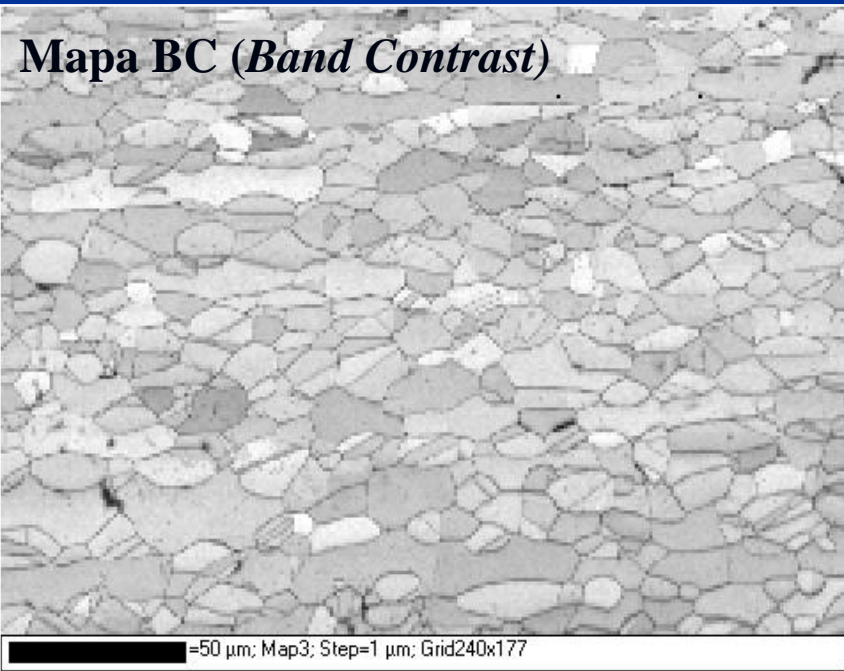
Mapa BC (*Band Contrast*) +Noise



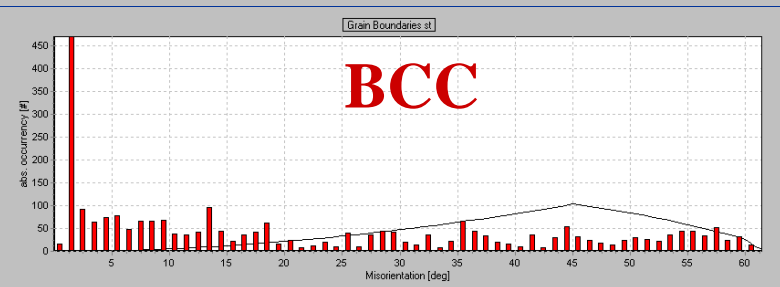
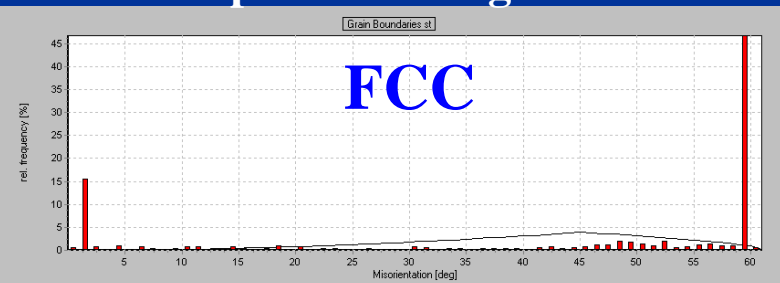
STAL DUPLEX

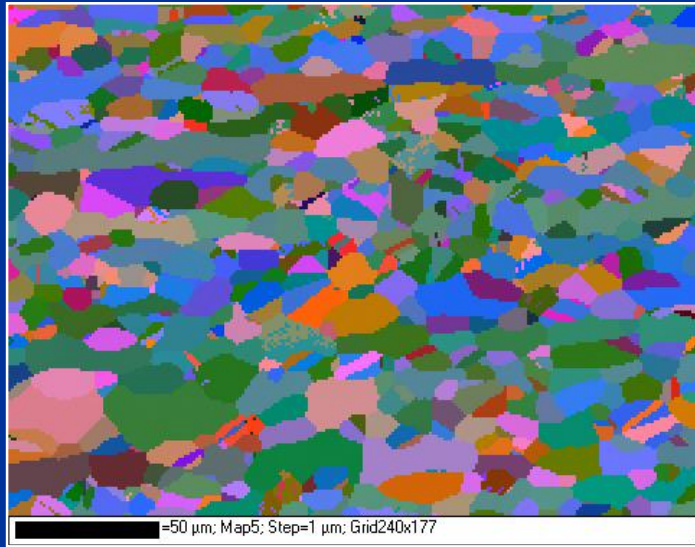


Mapa BC (*Band Contrast*)

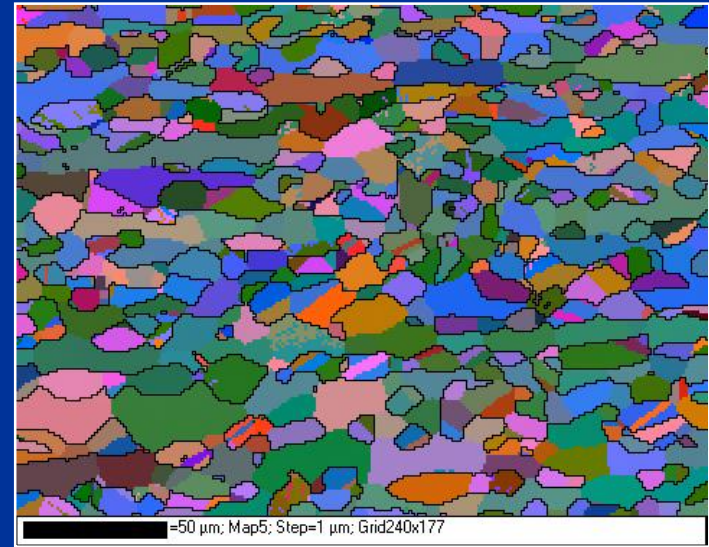


Mapa fazowa + granice

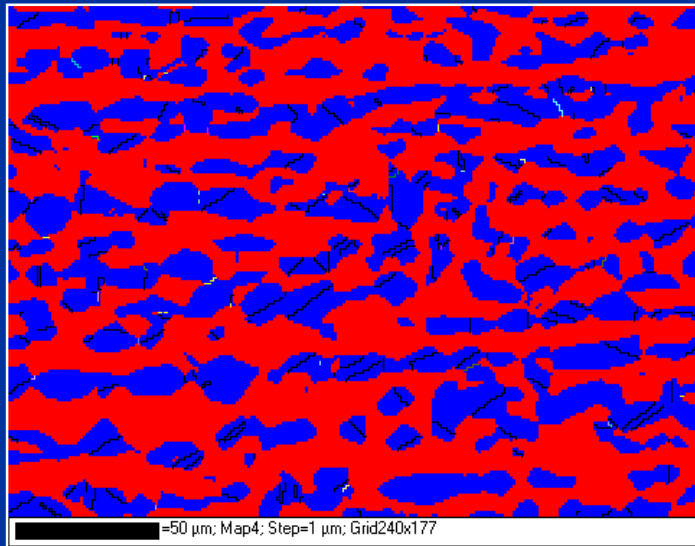




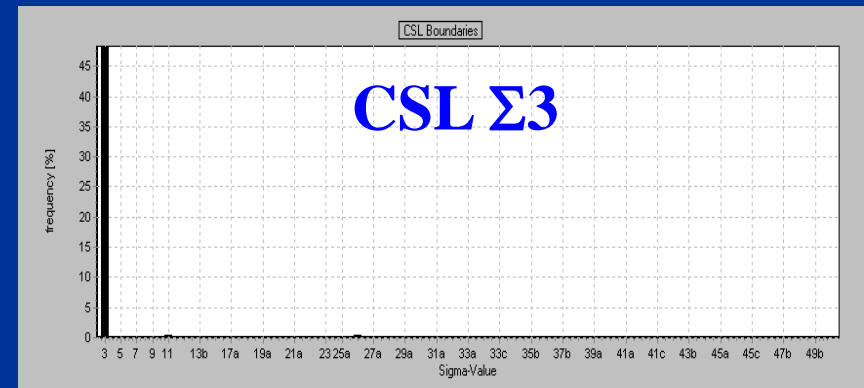
Mapa orientacji



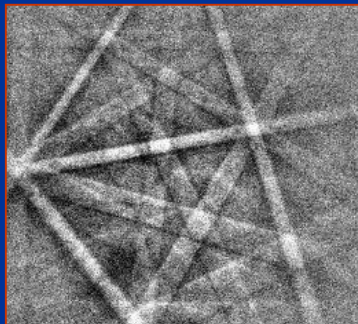
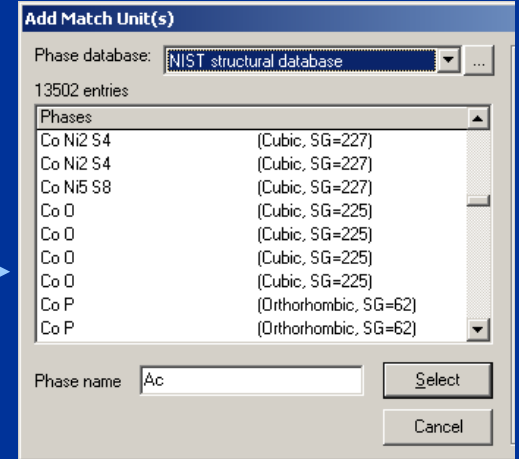
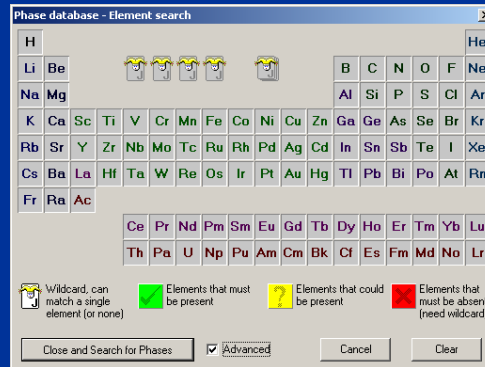
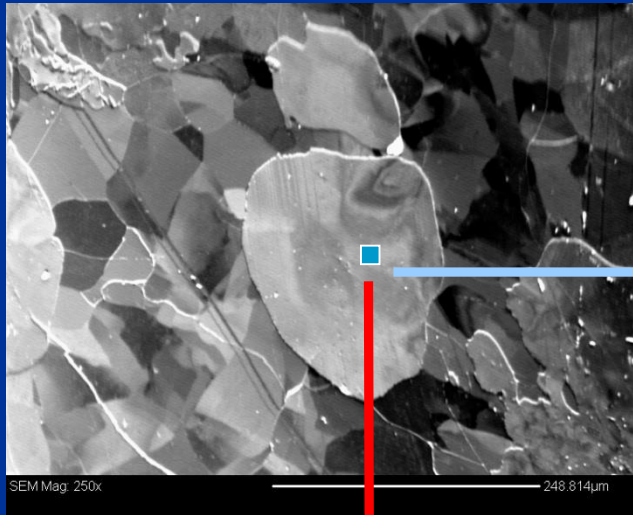
Mapa orientacji + granice fazowe



Mapa fazowa



# EBSD/OIM – technika wymagająca wzorców



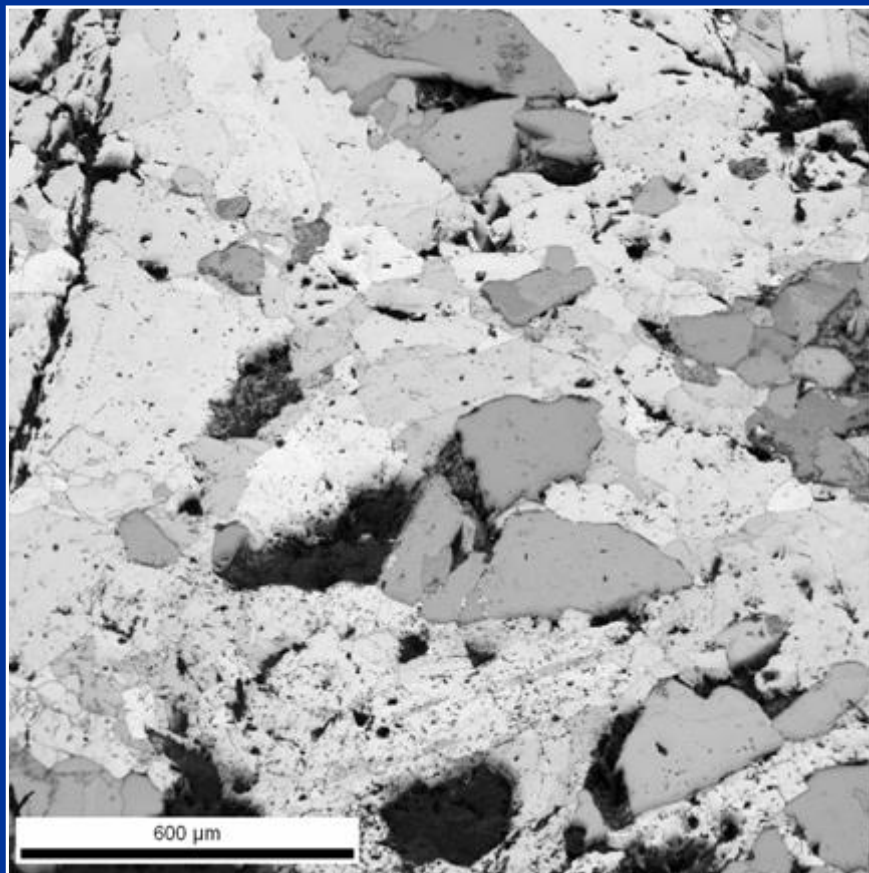
0-2-1  
1-2-1  
0-3-1  
1-10  
1-20  
1-30  
0-10  
0-31

Solutions:

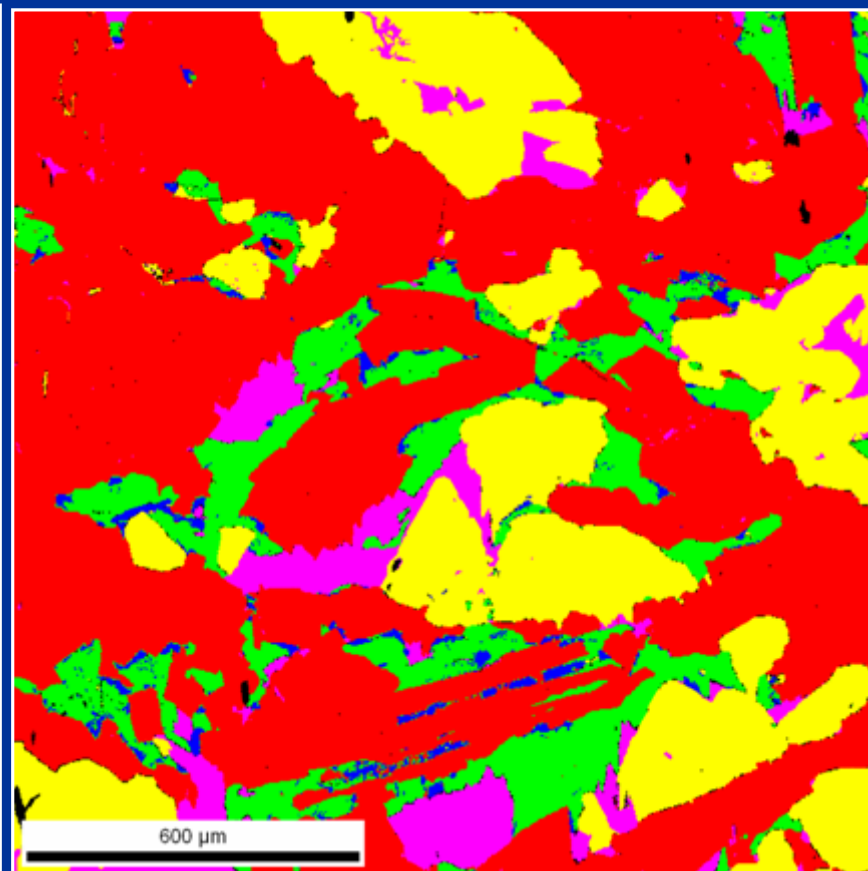
Phase	MAD	Bands
Aluminium	0.0521	6
AlAs2 Cs3	1.9693	6
AlAs2 Cs3	1.9697	6
AlAs2 Cs3	1.9829	6

# SZMERGIEL (skała zawierająca m.in. korund i magnetyt)

Mapa BC (*Band Contrast*)

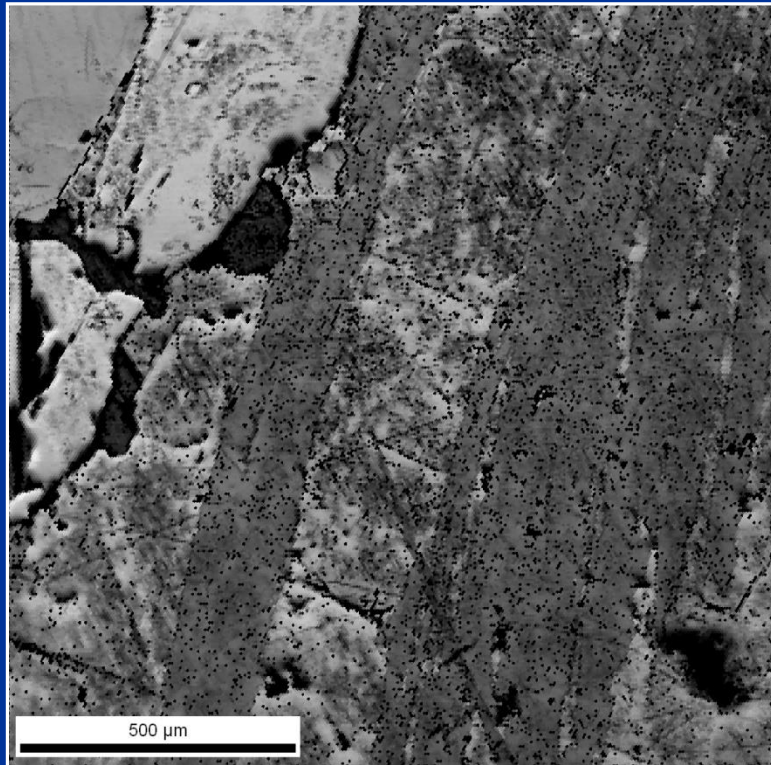


Mapa fazowa

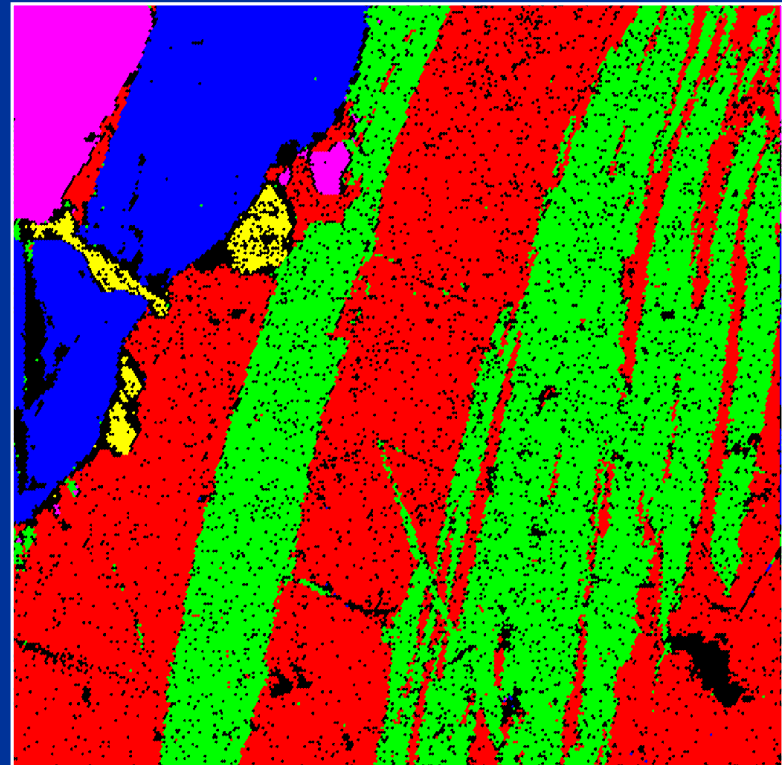


- Fe<sub>3</sub>O<sub>4</sub>
- FeTiO<sub>3</sub>
- Al<sub>2</sub>O<sub>3</sub>
- TiO<sub>2</sub>
- Magnesium Iron Aluminum Silicate Hydroxide

# Mapa BC (*Band Contrast*)

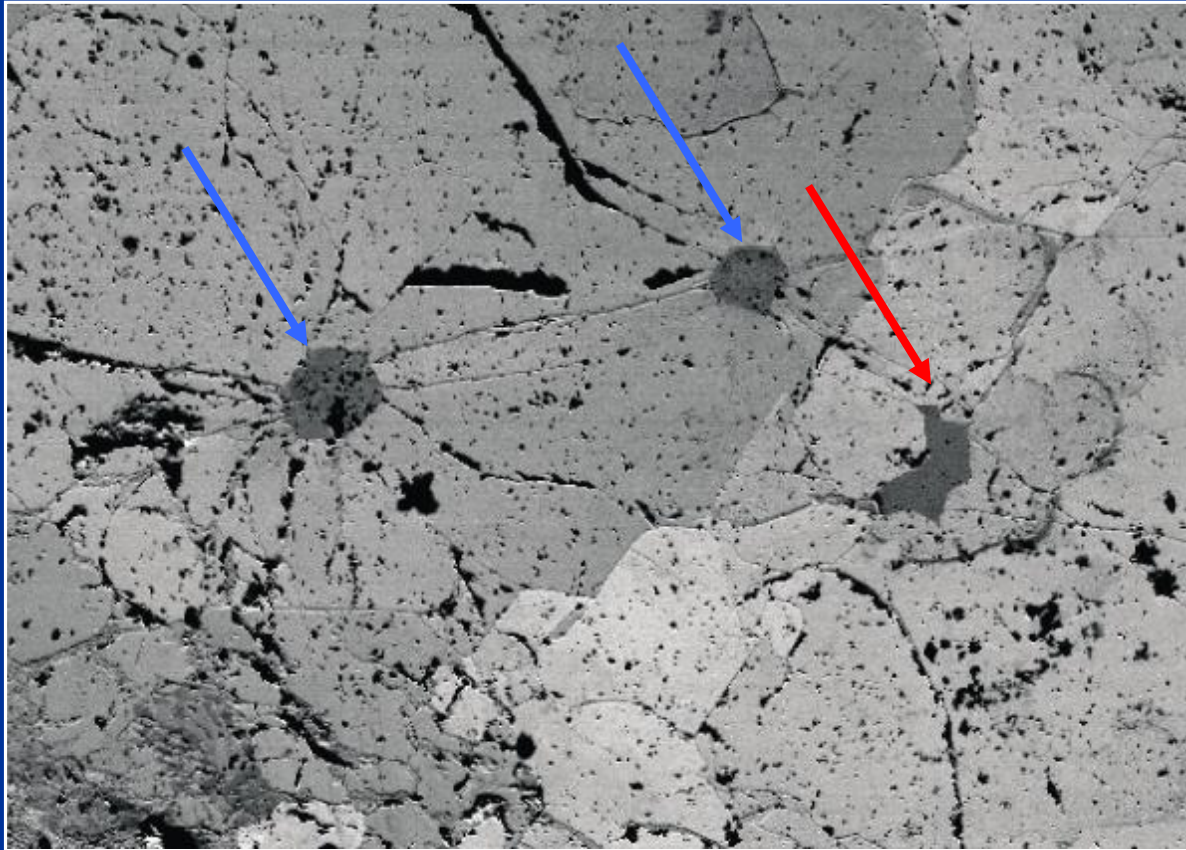


# Mapa fazowa



Phase	Total Fraction	Partition Fraction
Chalcopyrite	0.395	0.445
Cubanite	0.330	0.372
Amphibole	0.010	0.011
Iron Oxide	0.115	0.130
Iron Sulfide	0.039	0.044

# Forescatter electron image



**General microstructure of the analyzed area.**

**Three prominent inclusions in dark grey, two of which shows the coesite-quartz transformation (radial fractures visible as dark lines).**

**Grey scale variations in the surrounding garnet represent differences in crystal orientations („channelling contrast”)**

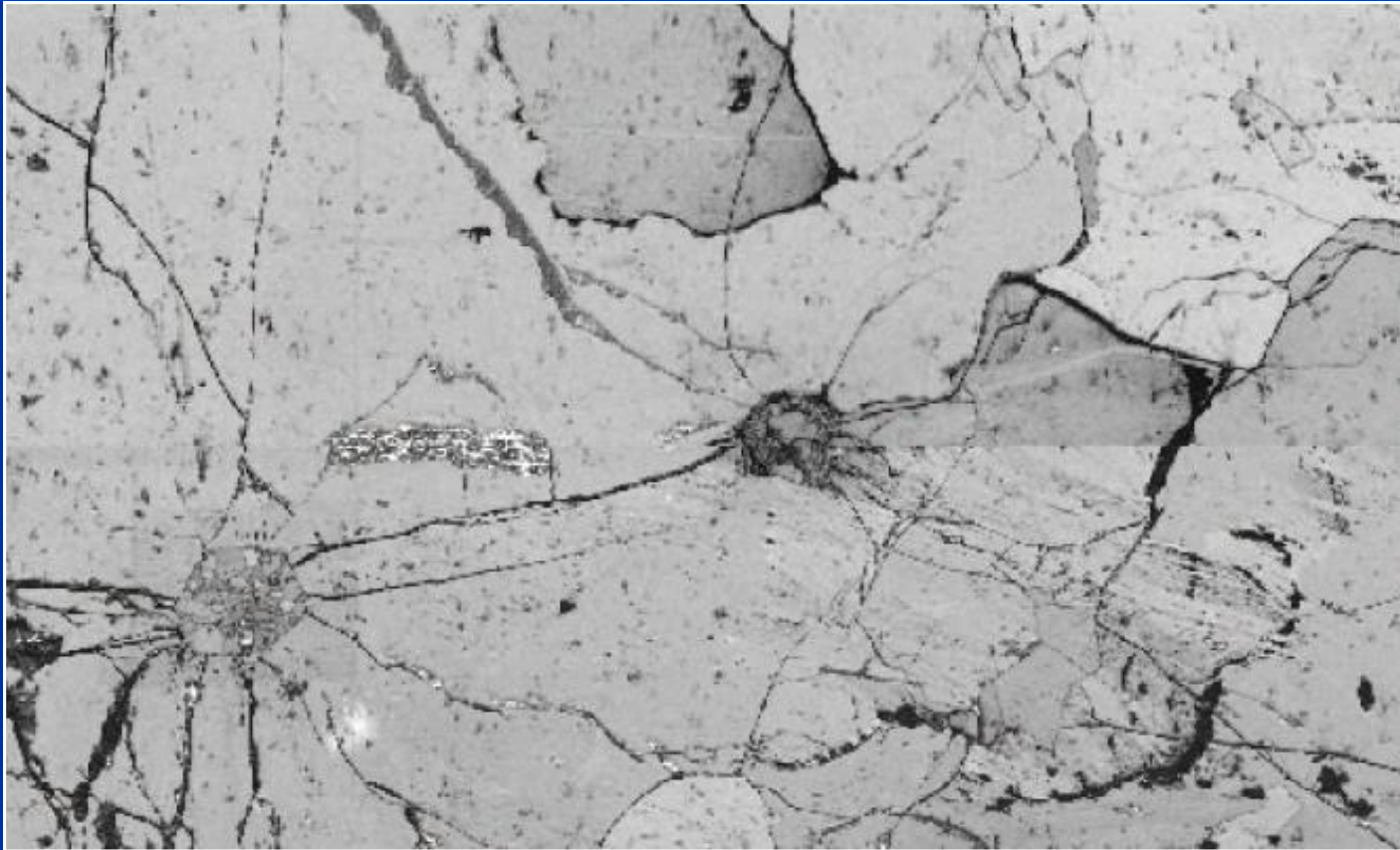
Koezyt – odmiana wysokociśnieniowa (20-80 kbar)  $\text{SiO}_2$  o strukturze jednoskośnej C2/c

Kwarc – odmiana niskociśnieniowa  $\text{SiO}_2$  o strukturze trygonalnej  $\text{P3}_22$

Granat -  $(\text{Mg,Fe})_3\text{Al}_2[\text{SiO}_4]_3$



# EBSD quality map

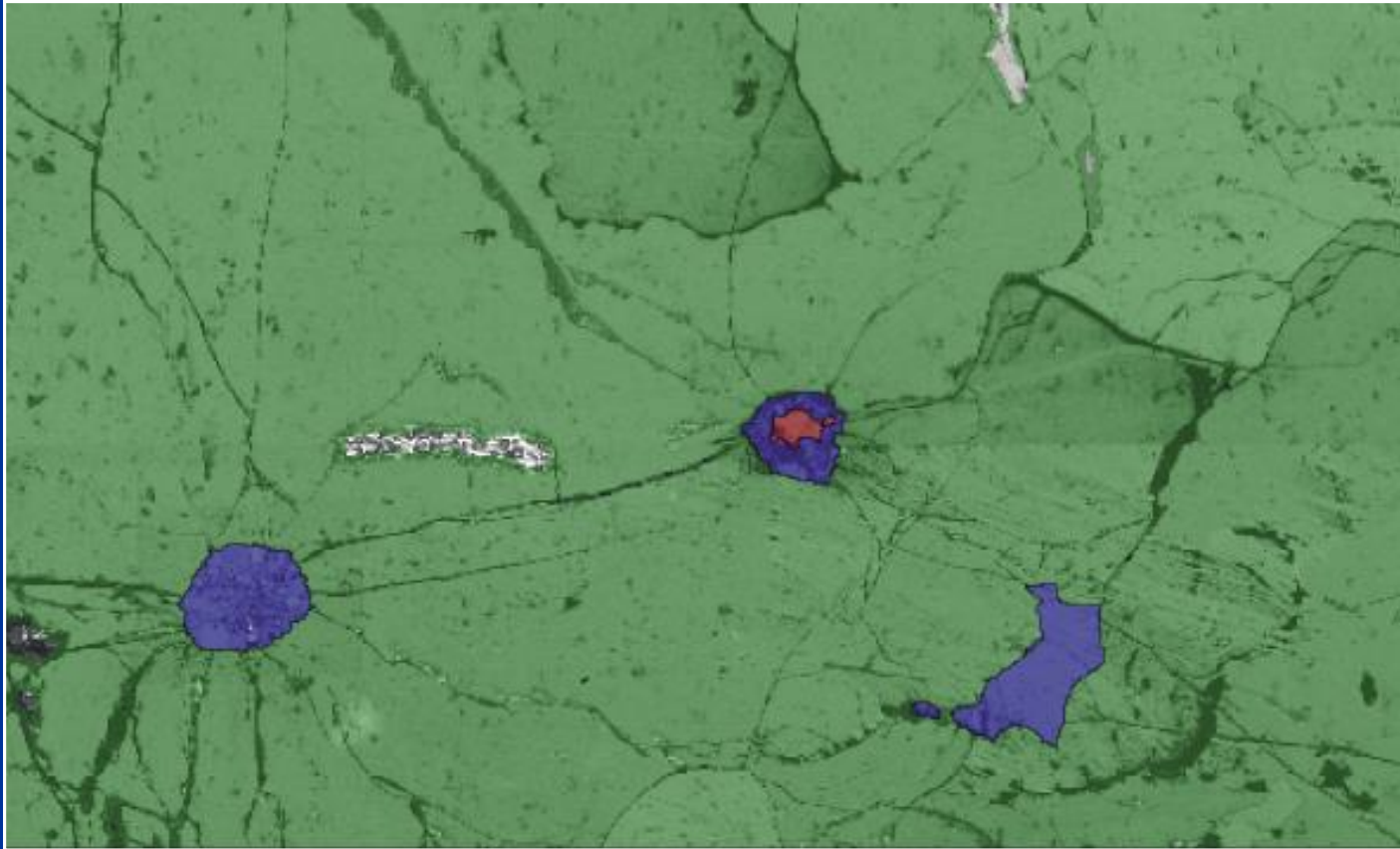


**General microstructure of the analyzed area**

**Two of the inclusions (in the centre and lower left part of the image) are prominent with radiating fractures, visible as black lines (poor EBSD quality)**

**Horizontal join of the 2 maps is visible across the centre of the image. Scale bar = 400 $\mu$ m**

# Phase map



## Distribution of three phases across the area

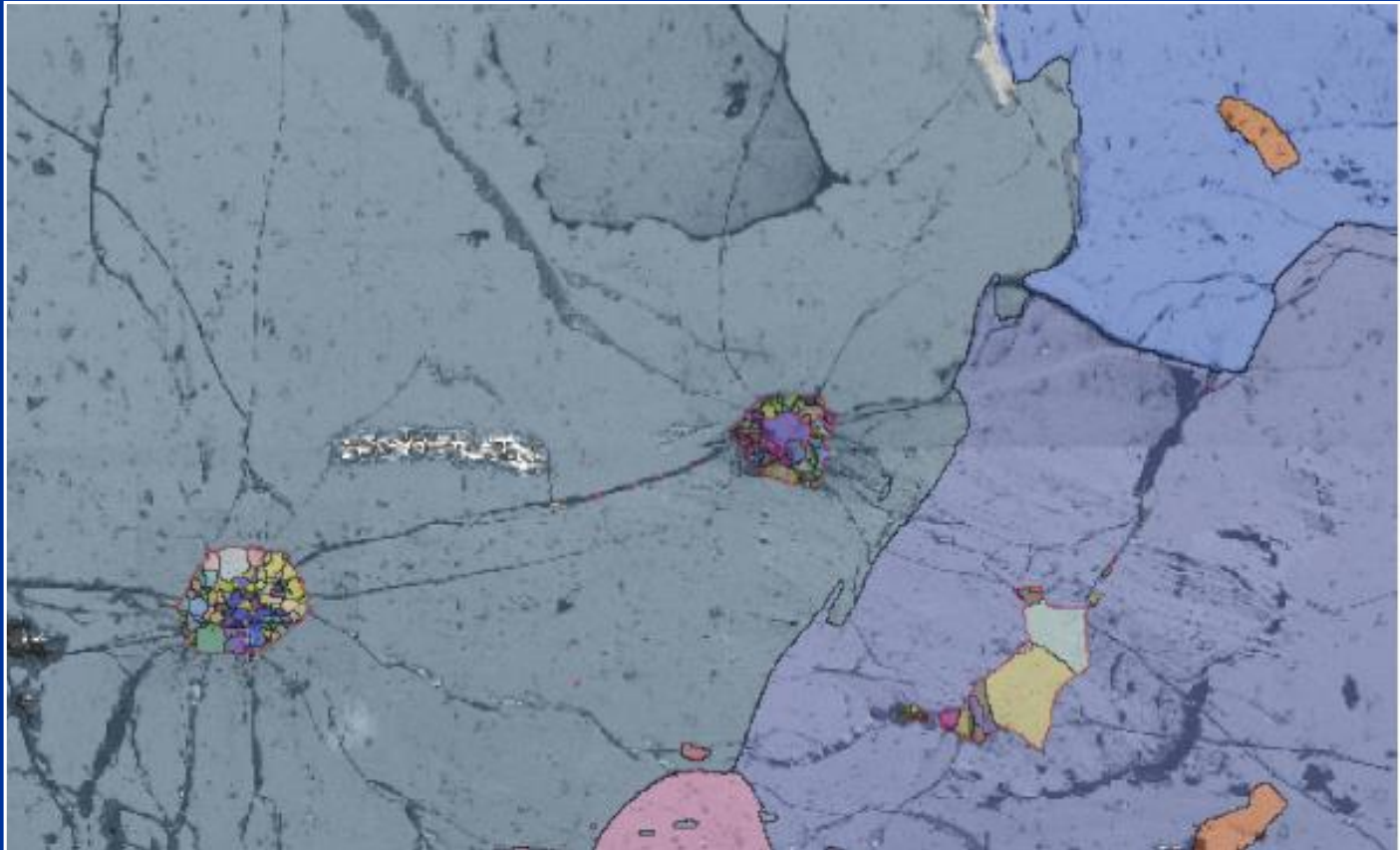
Garnet is marked in green, quartz in blue and coesite in red.

The central inclusion has a core of coesite surrounded by a rim of quartz.

The lower-left inclusion has completely reverted to quartz.

Radiating from the prominent inclusions are fractures, visible as black lines  
(poor EBSD quality)

# Orientation map



**Crystallographic orientations of all three minerals**

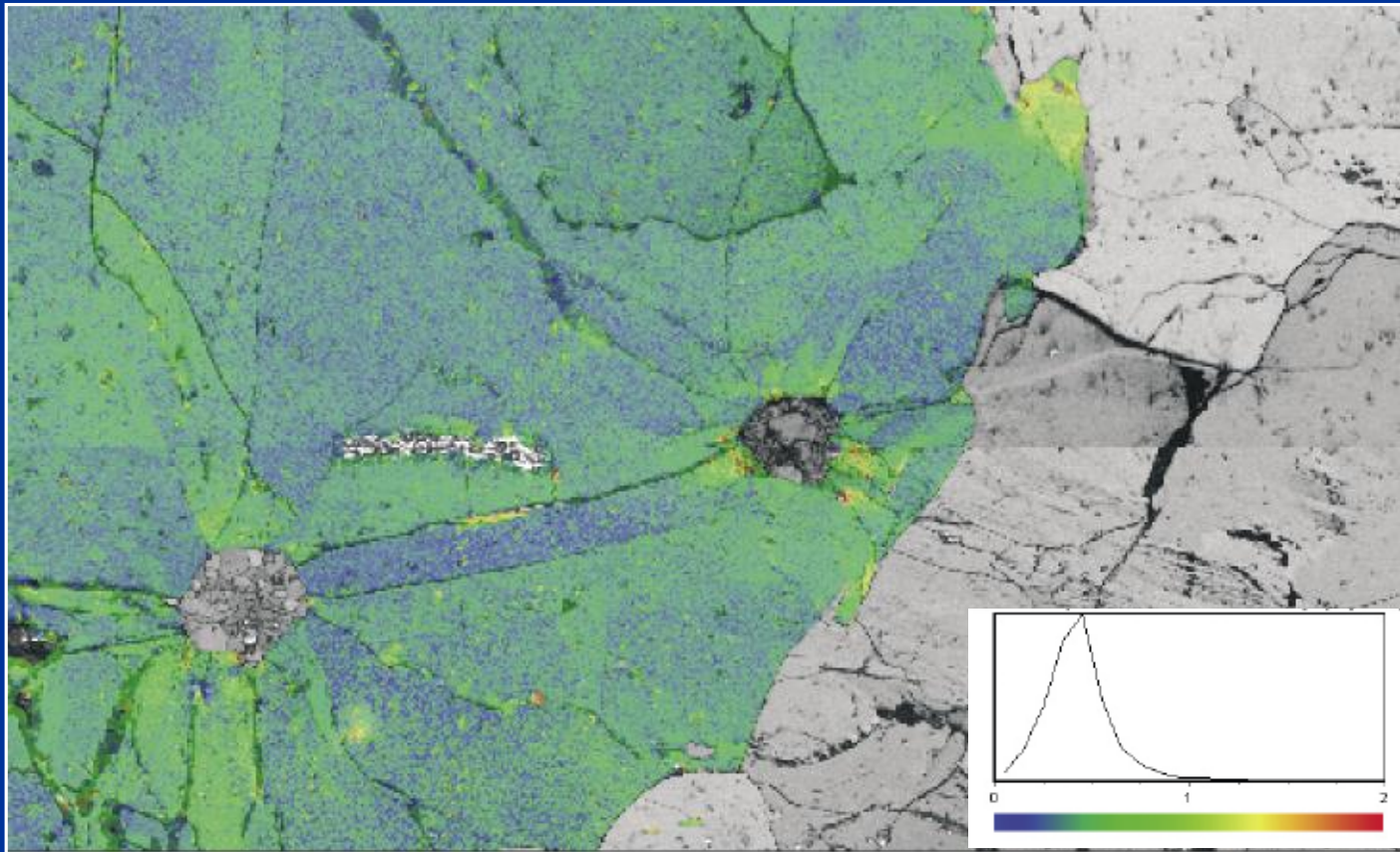
**Colors corresponding to the Euler angles.**

**Grain boundaries are marked in black, phase boundaries are marked in red**

**The whole area is comprised of only a few garnet grains. The quartz in the inclusions is polycrystalline with grain boundaries radiating from the centre.**

**The coesite in the central inclusion is a single crystal**

# Deformation map



## Deformation of the garnet around the coesite/quartz inclusions

The attempted change from coesite to quartz has tried to expand the inclusion, thereby causing radial cracks and deformation in the garnet.

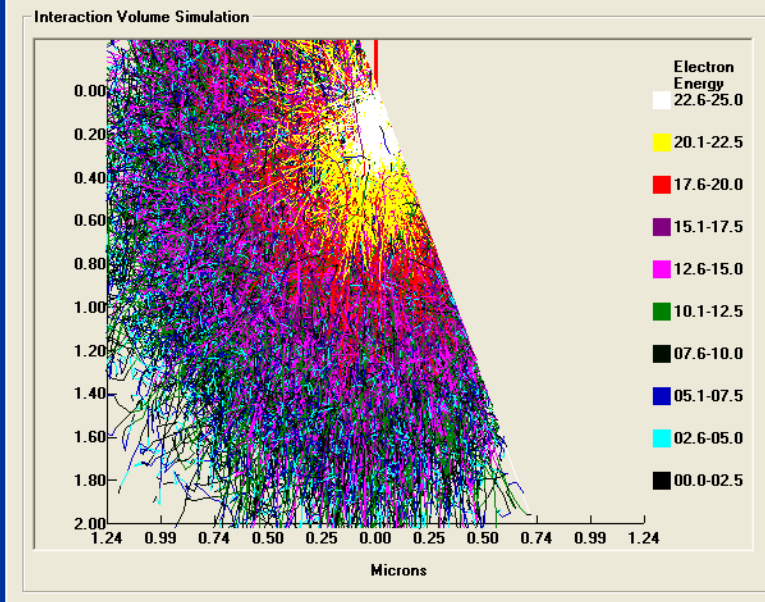
This means that the garnet crystal has acted as a protective pressure vessel so that pieces of coesite have been preserved.

Small scale of deformation in the large garnet grain (less than  $2^\circ$ )

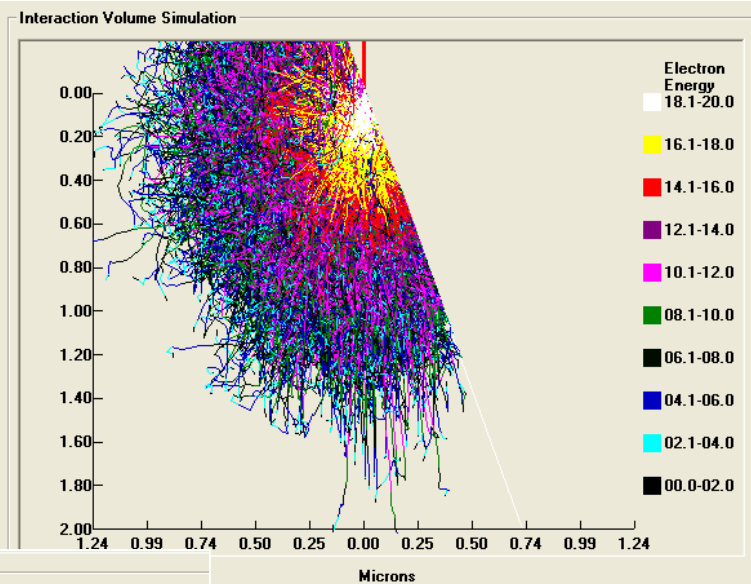
**The EBSD technique is limited in spatial resolution by the electron-solid state interaction volume.**

**The best achievable spatial resolution on a material of medium atomic number is currently in the order of about 30 nm!!!**

*Towards better spatial  
resolution  
(below 1  $\mu\text{m}$ )*



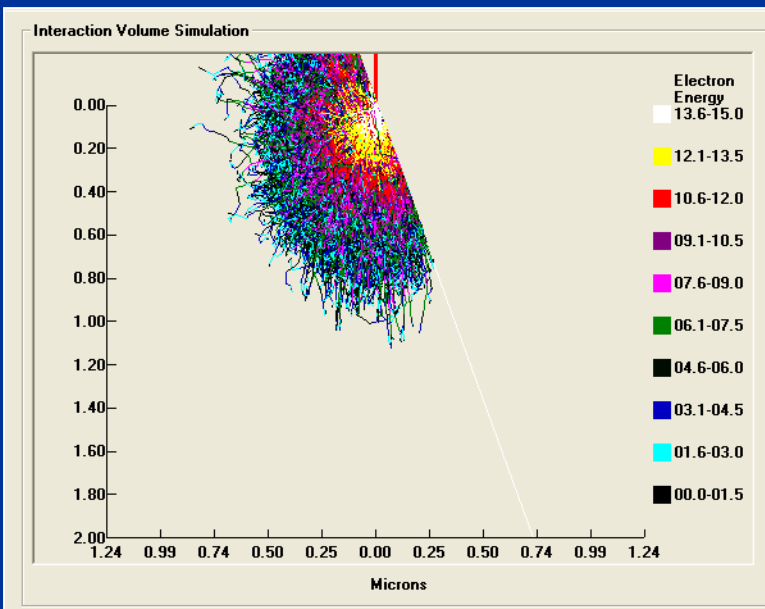
**Sample Conditions**  
 kV: 25.0 Tilt: 70  
 No. Trajectories: 30000  
 B.S. Coefficient: 0.5184



**Sample Conditions**  
 kV: 20.0 Tilt: 70  
 No. Trajectories: 30000  
 B.S. Coefficient: 0.5171

Bulk Ni

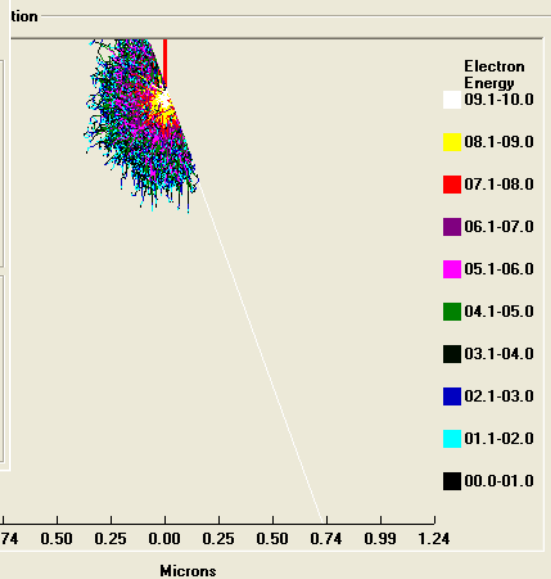
Comments



**Sample Conditions**  
 kV: 15.0 Tilt: 70  
 No. Trajectories: 30000  
 B.S. Coefficient: 0.5233

Bulk Ni

Comments

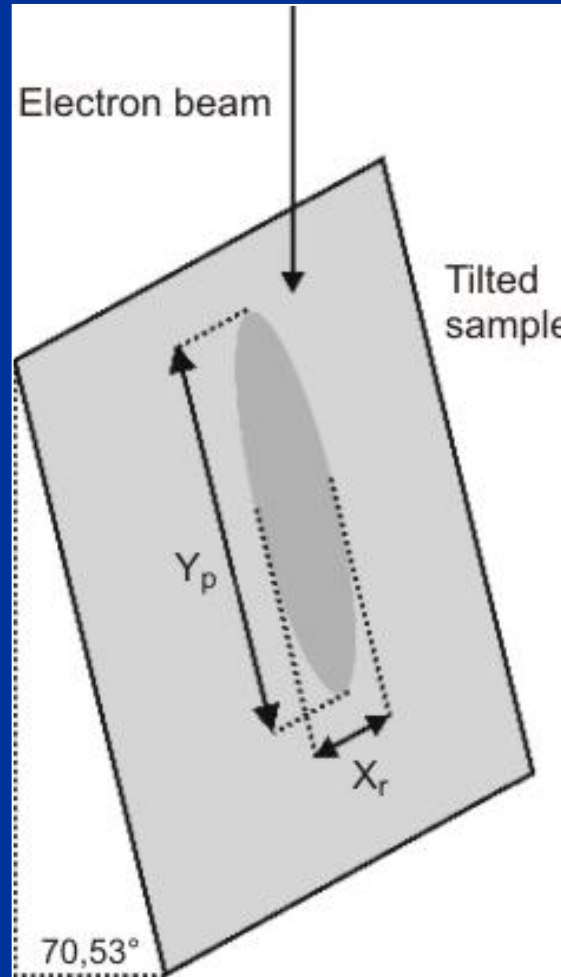
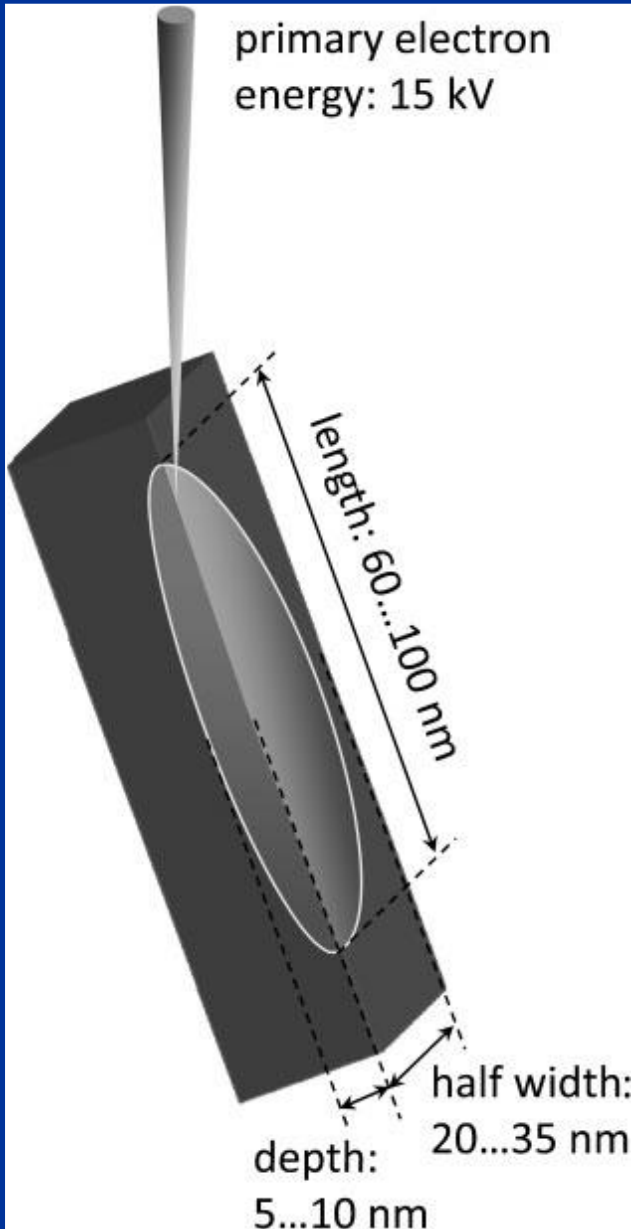


**Sample Conditions**  
 kV: 10.0 Tilt: 70  
 No. Trajectories: 30000  
 B.S. Coefficient: 0.5218

Bulk Ni

Comments

# Interaction volume of the primary electron beam with the sample



$X_r$  spatial resolution  
parallel to the sample tilt  
axis  
 $Y_p$  spatial resolution  
perpendicular to the  
sample tilt axis



# Physical spatial resolution

is determined by the size of the volume which contributes to the diffraction signal.

It depends on:

- accelerating voltage
- probe current

**Higher spatial resolution means:**

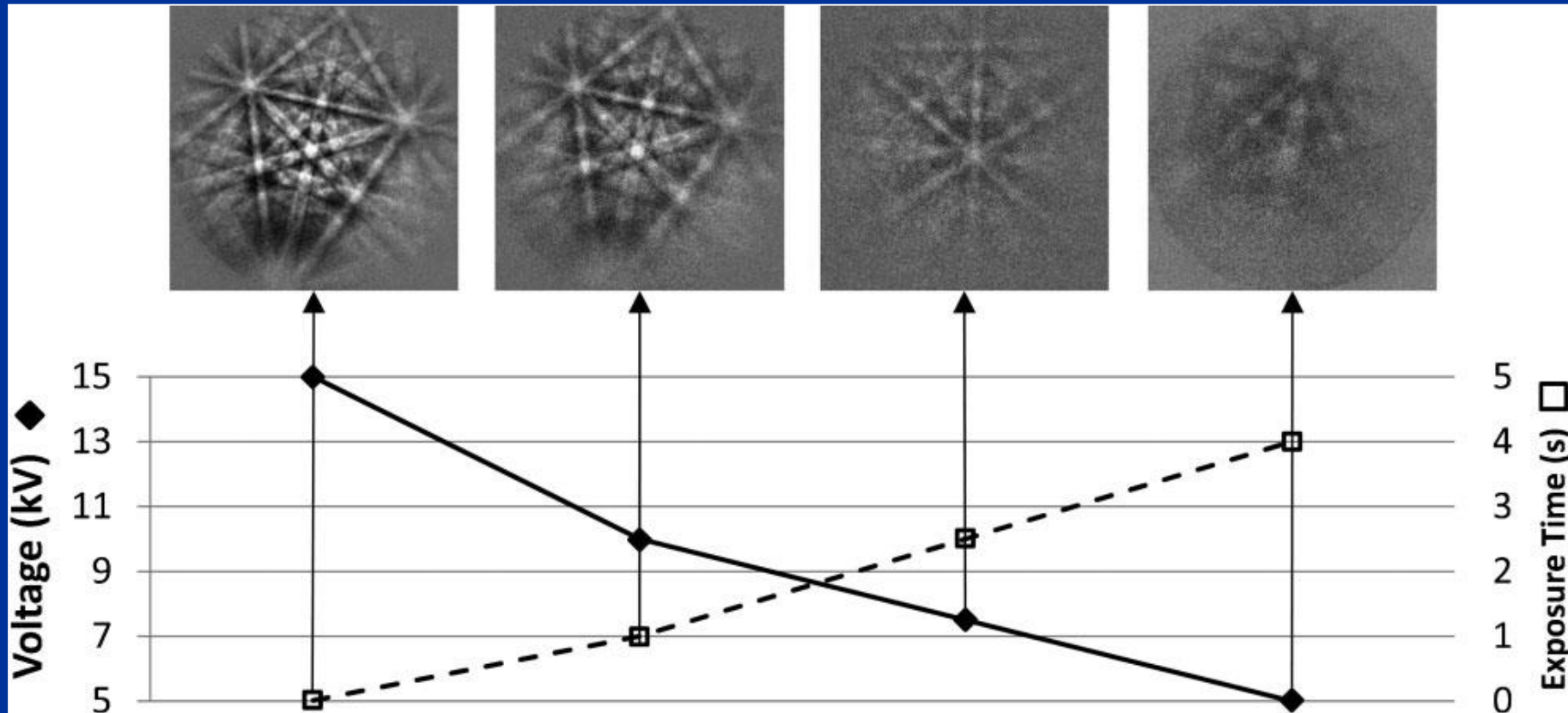
**lower kV & beam current**

**The challenge for improving physical spatial resolution is EBSD operated at low kV !!!**

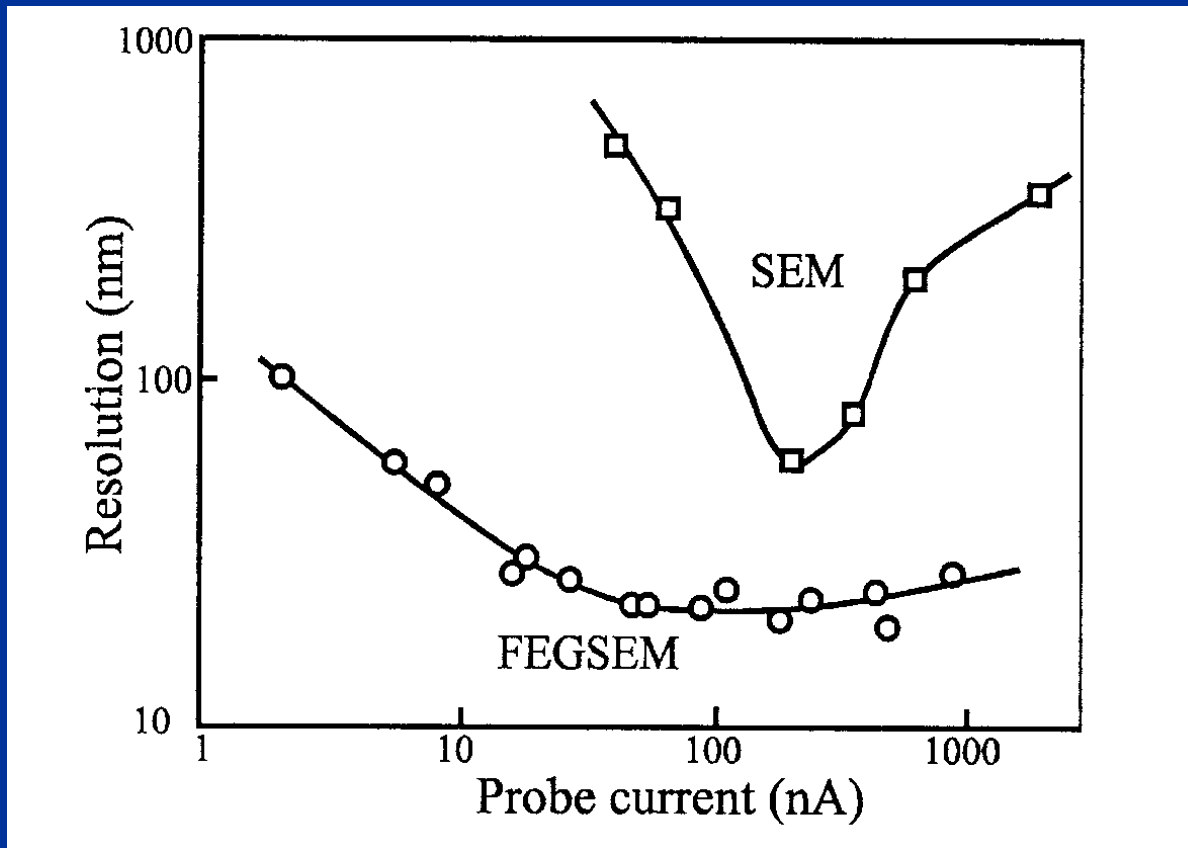
**because:**

- **Low signal**
- **Weaker patterns especially the edges**
- **Broaden bands**

# Influence of accelerating voltage on exposure time

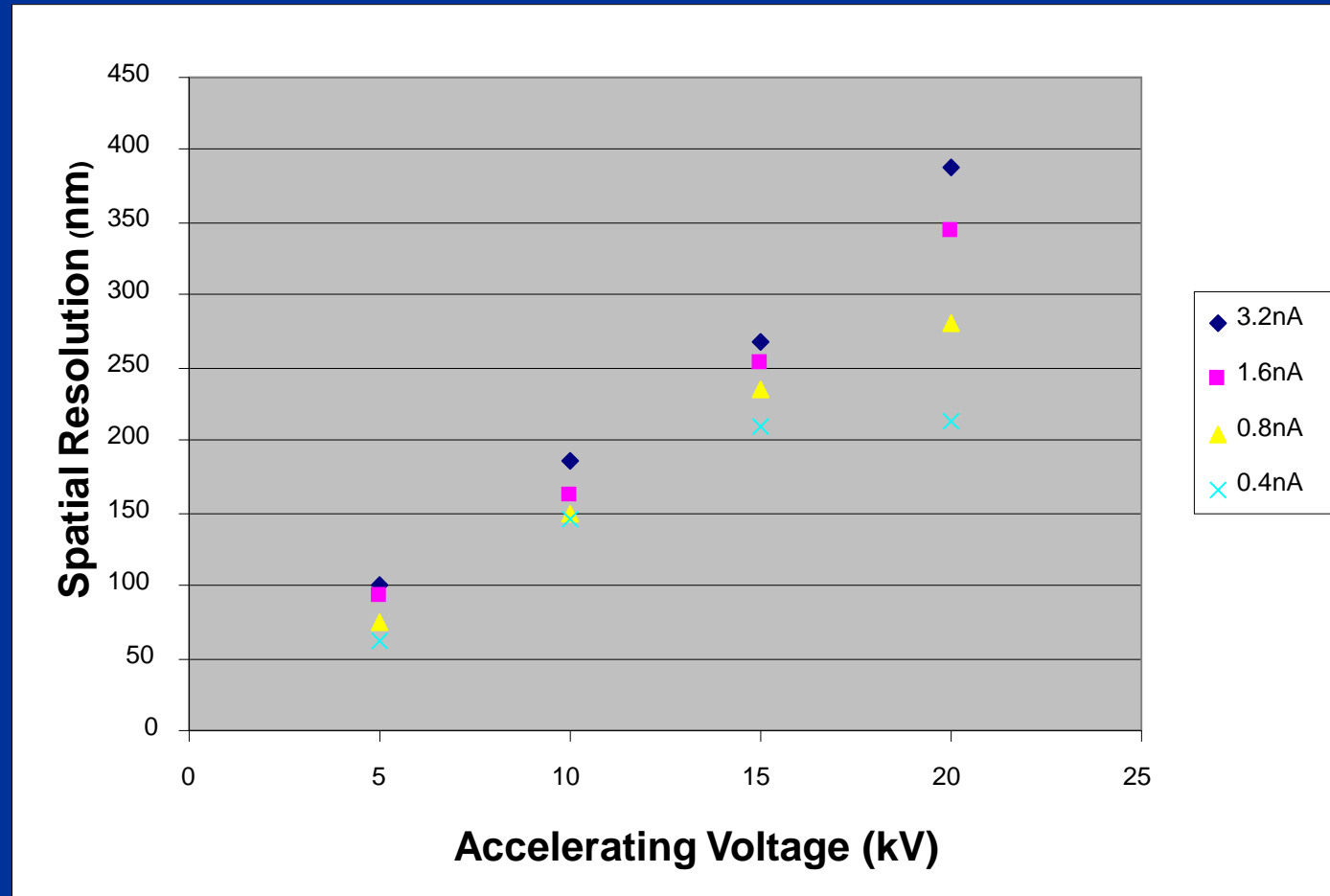


# Probe Current vs Physical Spatial Resolution

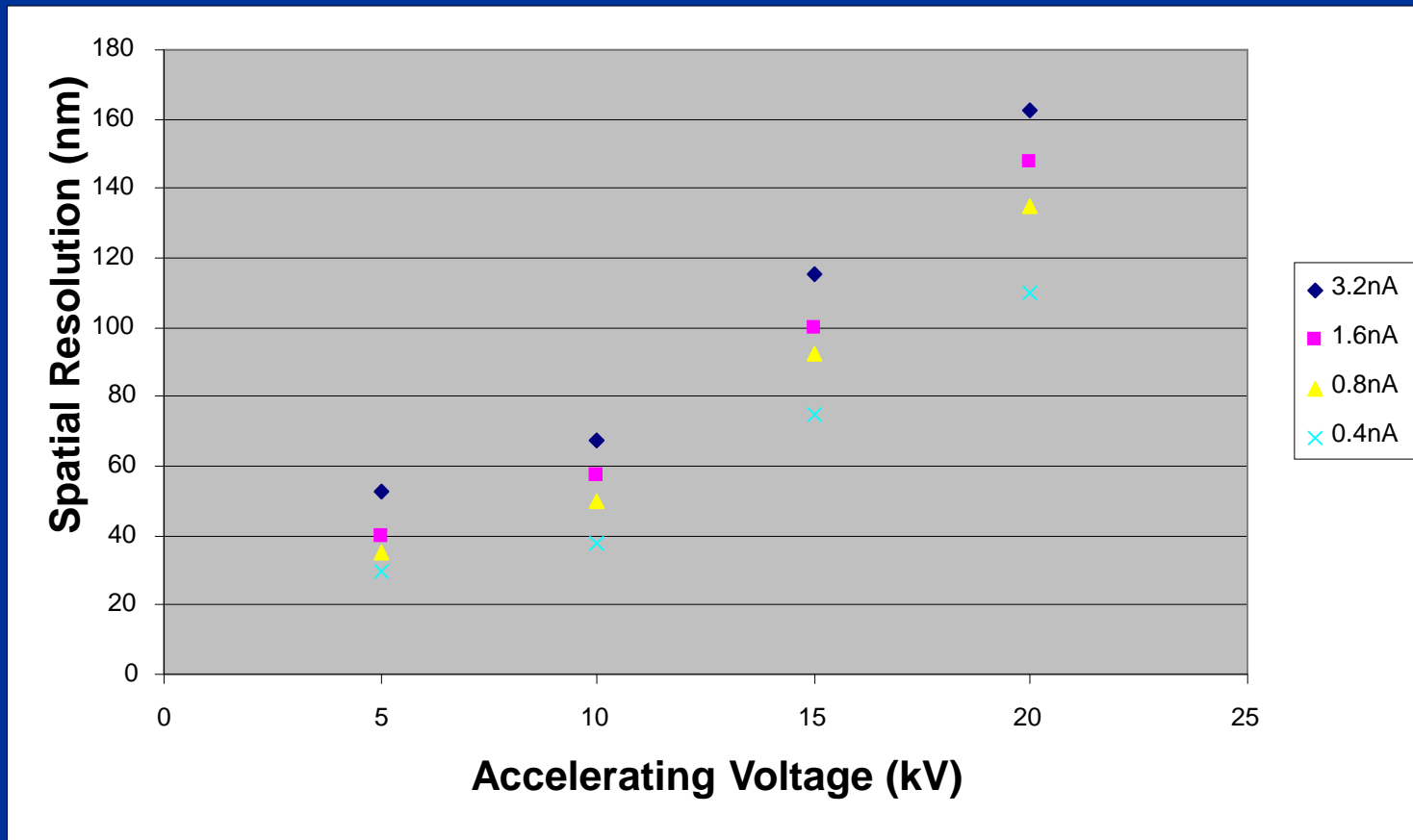


Spatial resolution is improved by using lower probe currents, but must have ability to obtain good Kikuchi patterns.

# Physical spatial resolution as a function of voltage and probe current for aluminum

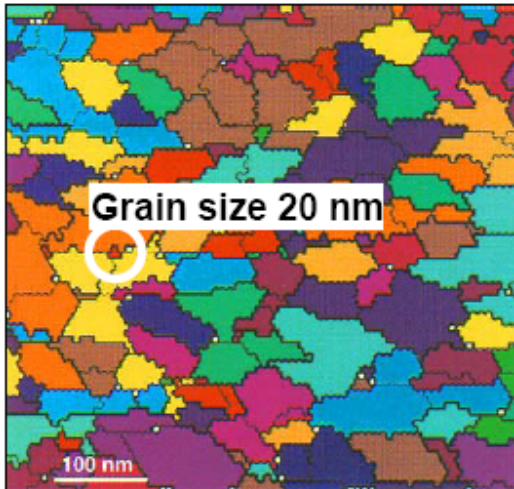


# Spatial resolution as a function of voltage and probe current for copper

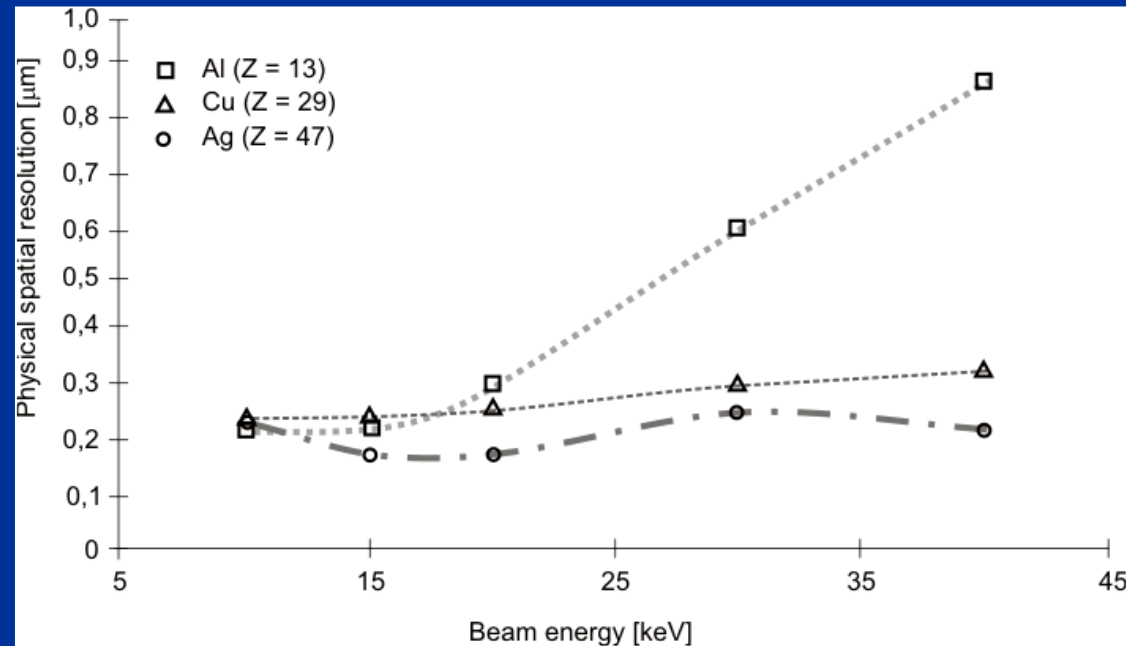


# Influence of atomic number on physical spatial resolution

Pt, recrystallized

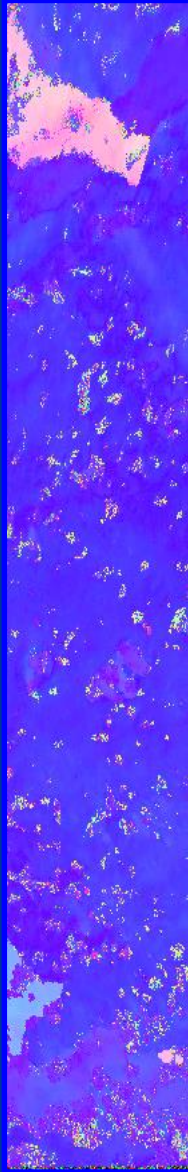


D. Dingley, in "Electron Backscatter Diffraction in Material Science", Kluwer, 2000

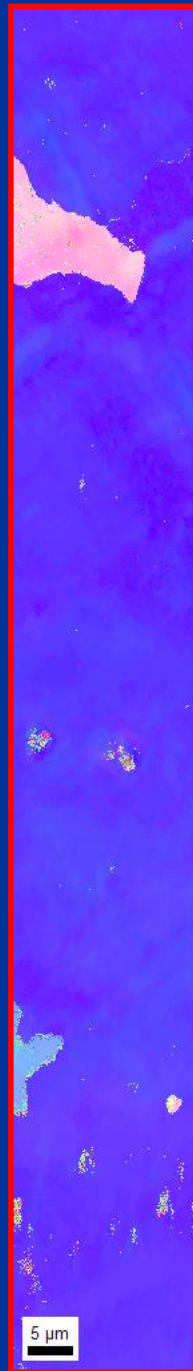


T.Petersen, G.Heilberg, J.Hjelen  
(1998) ICEM 14 Proceedings, 775-776

100 nm step size

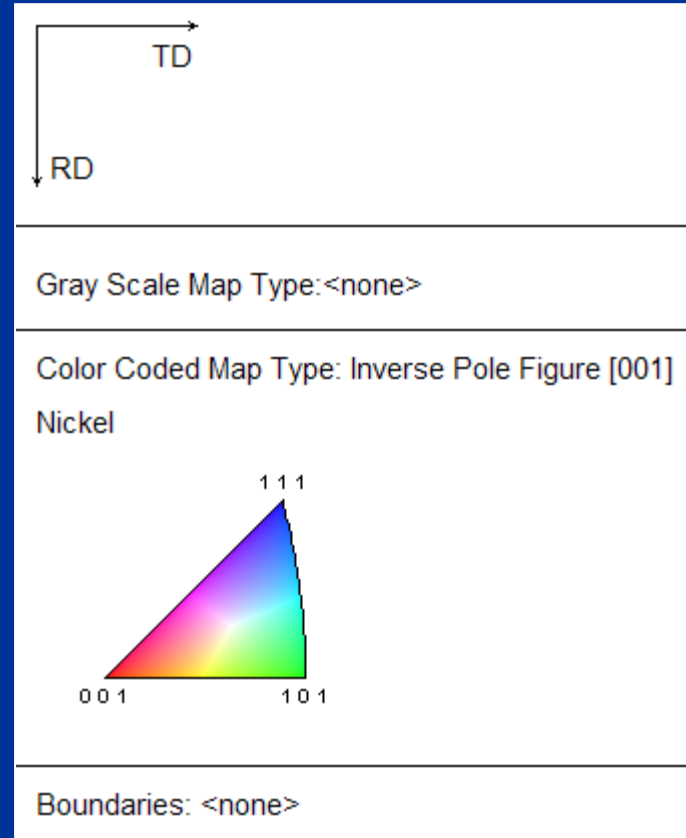


20 keV



10 keV

# Nickel







**15keV**



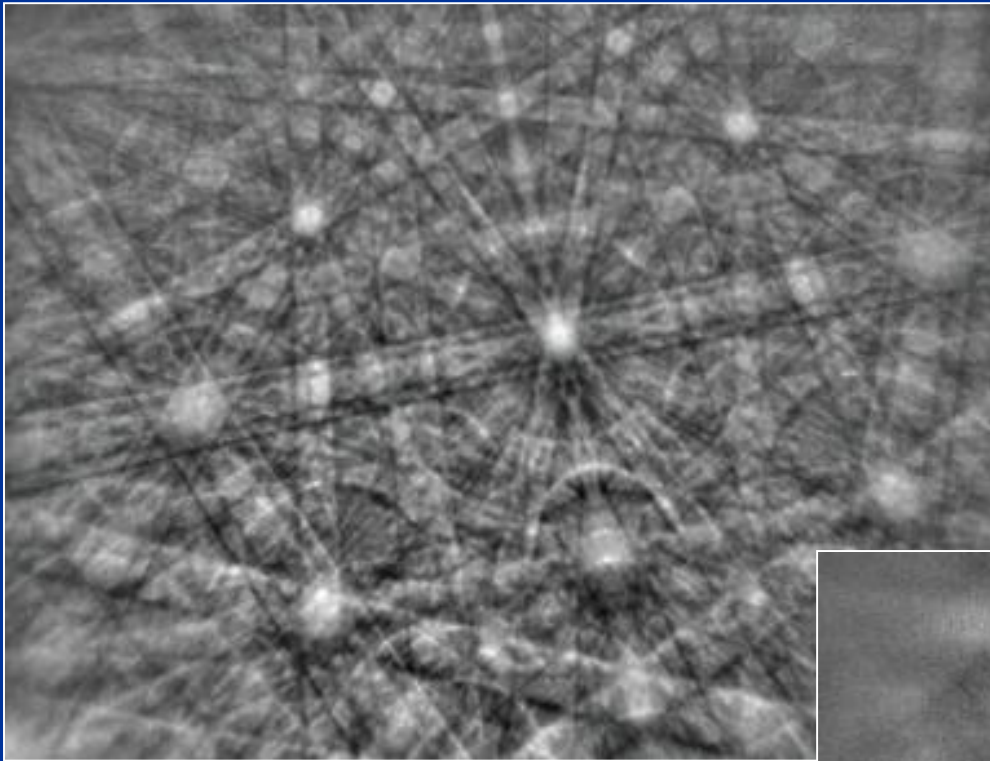
**7.5keV**

10 nm step size

**Significant improvement of physical spatial resolution of EBSD is possible !!!**

**At 15 keV only 2 pixels and a slight decrease of pattern quality indicate of the presence of a small twin in a lightly deformed TWIP steel sample**

**At 7.5 keV the twin is clearly visible!**



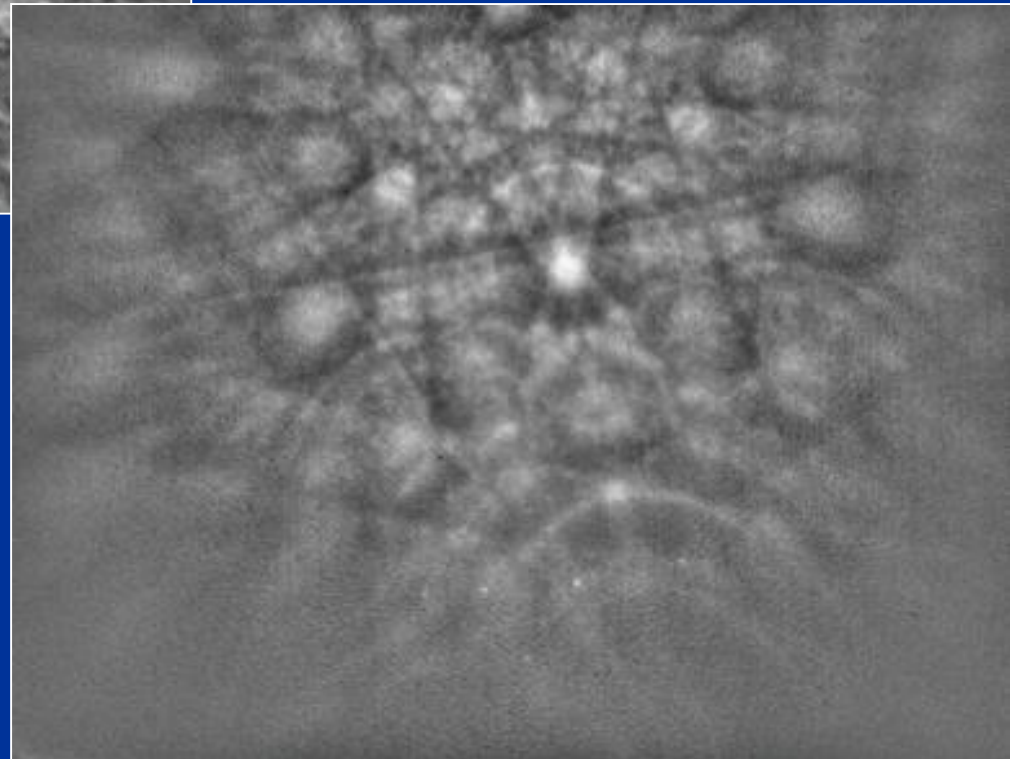
**Iron pyrite at 20 keV**

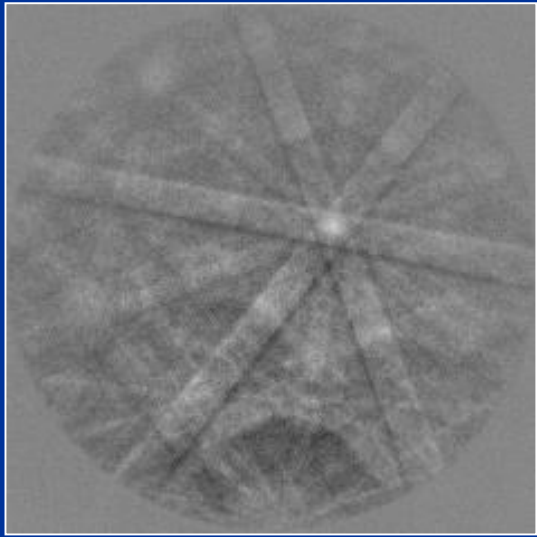
*At 20keV excellent detail  
can be seen within the pattern.*

*At 5keV, the patterns remain clear  
and are readily indexed.*

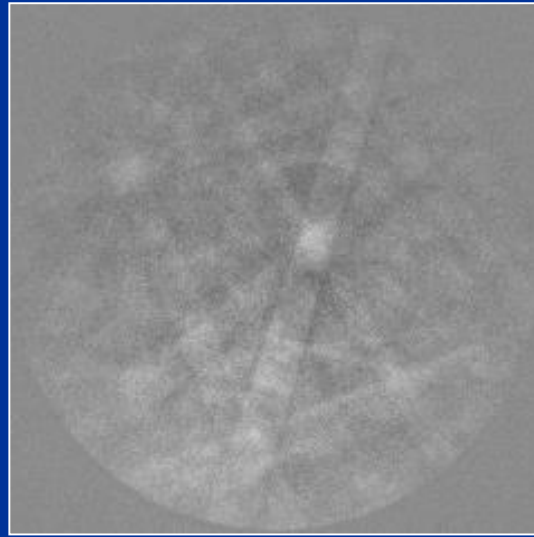
Courtesy of Harvinder Singh Ubhi,  
Oxford Instruments

**Iron pyrite at 5 keV**

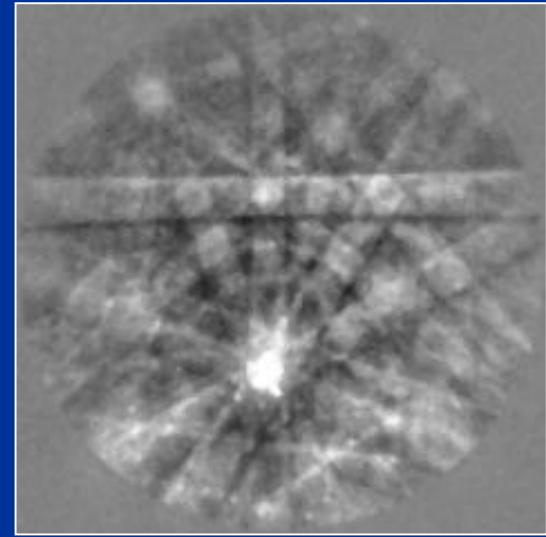




15 keV

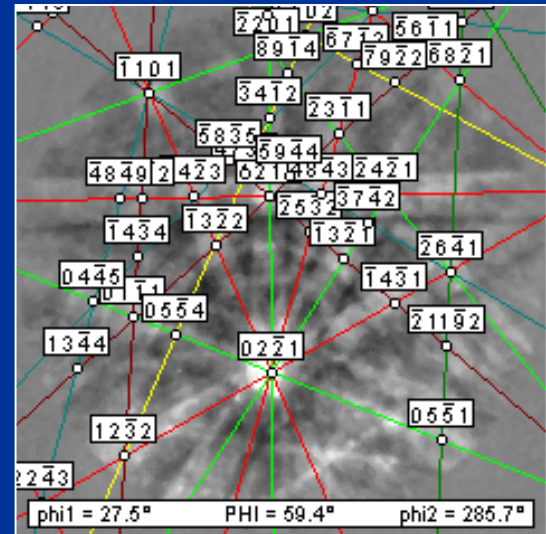


10 keV

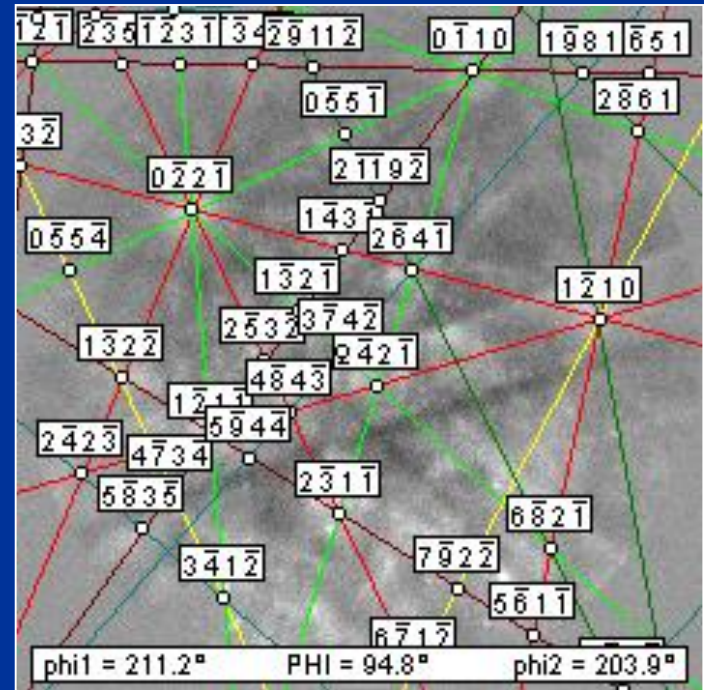
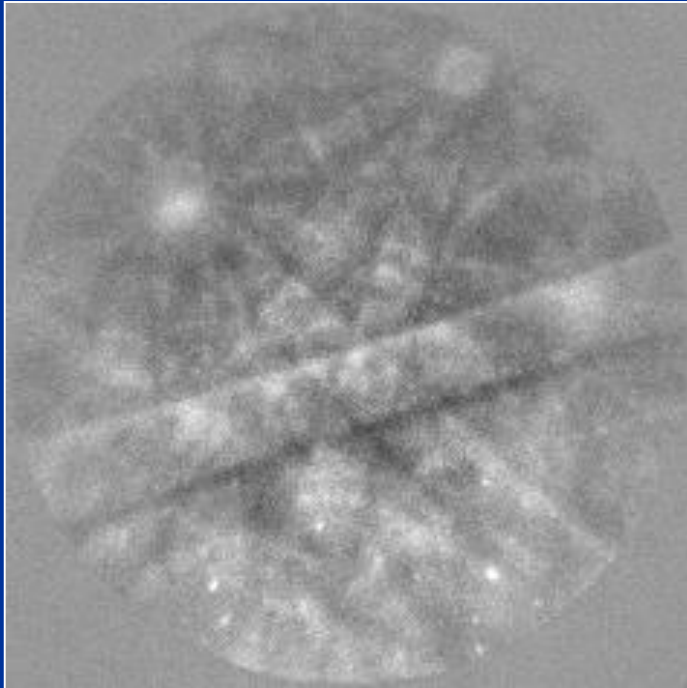


7.5 keV

# EBSD from $Al_2O_3$

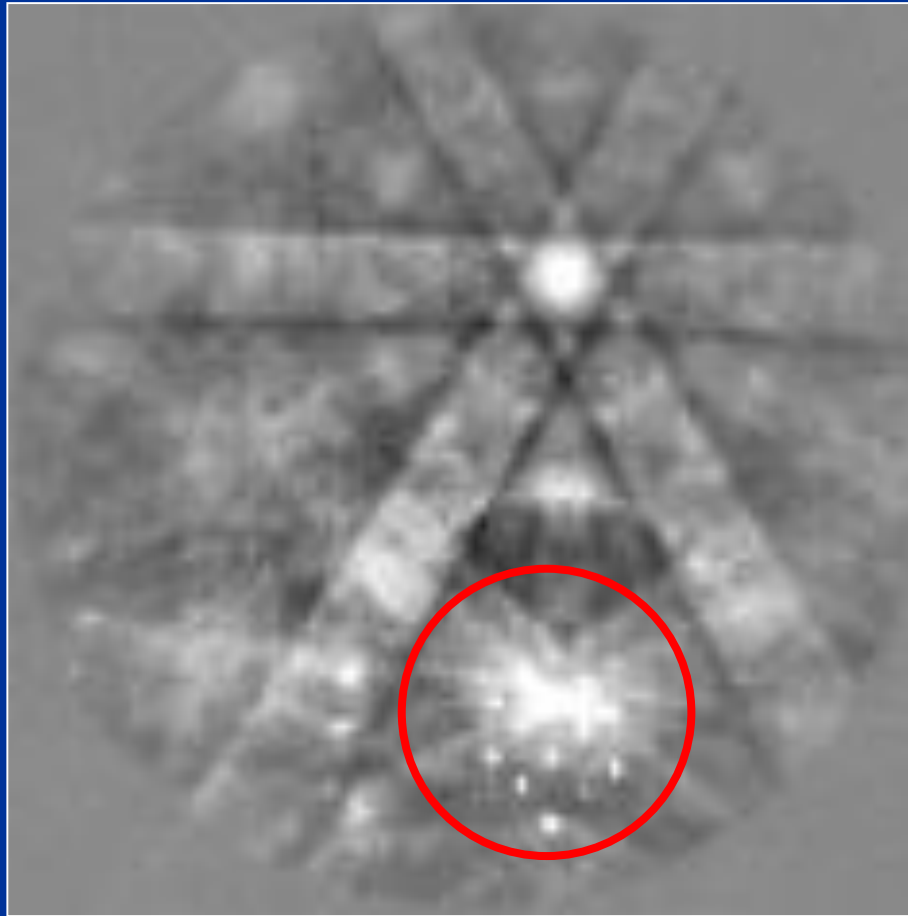


# EBSD from $\text{Al}_2\text{O}_3$ 5 keV !!!



Long exposure time: 6 fps, binning 1x1

# EBSD from $\text{Al}_2\text{O}_3$ 5 keV !!!



**RHEED pattern is visible !!!**

**Reflection High Energy Electron Diffraction RHEED:**

- provides large elastic scattering cross-section for forward scattered electrons
- keeps penetration depth small by using grazing incidence

# Orientation maps from $\text{Al}_2\text{O}_3$ 5 keV

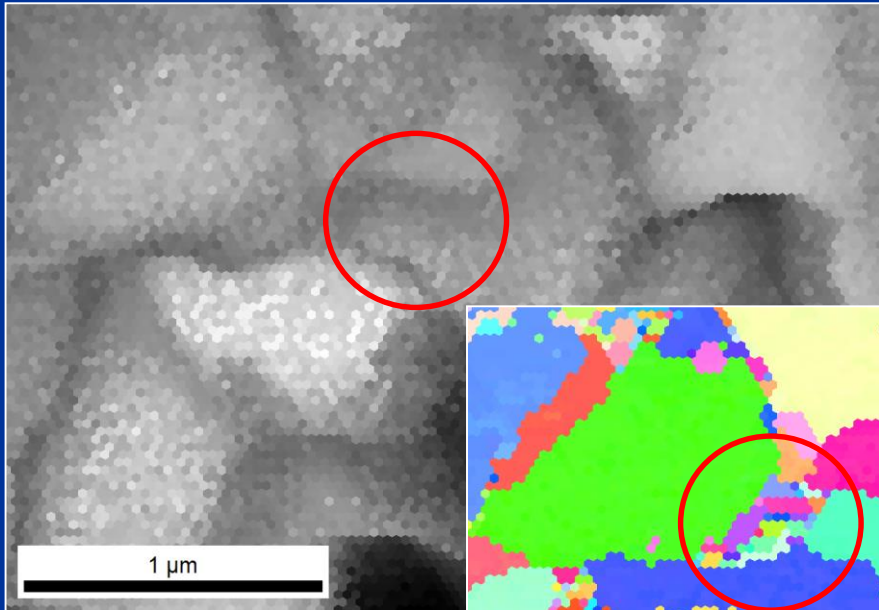
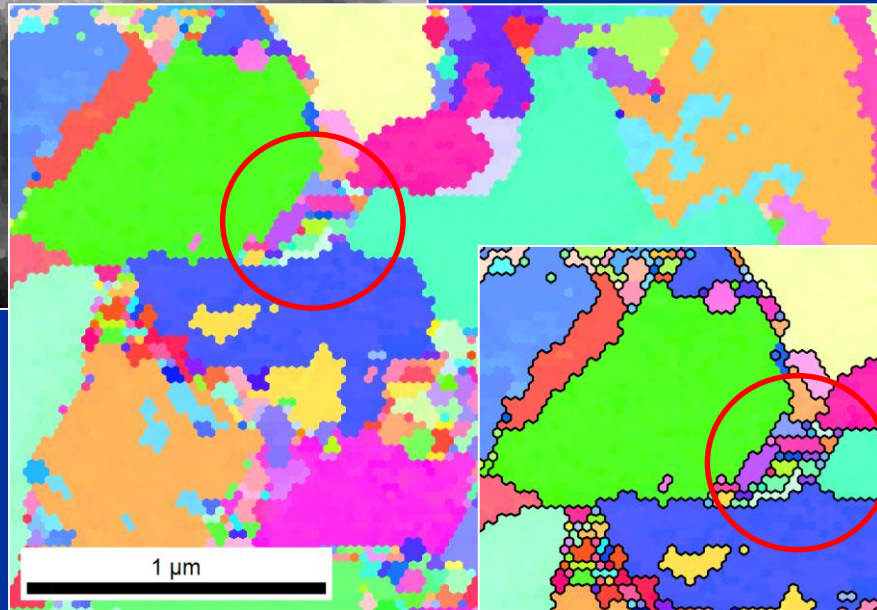
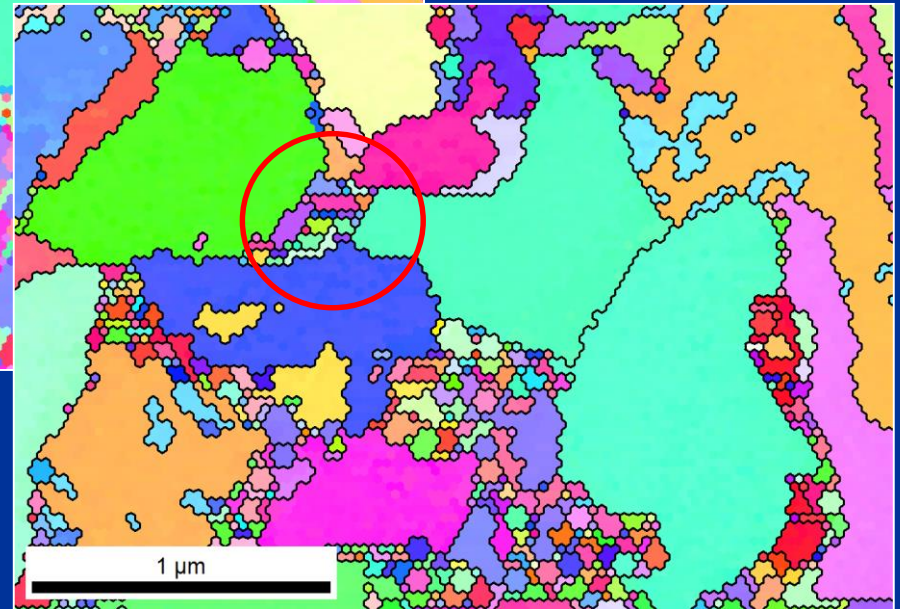


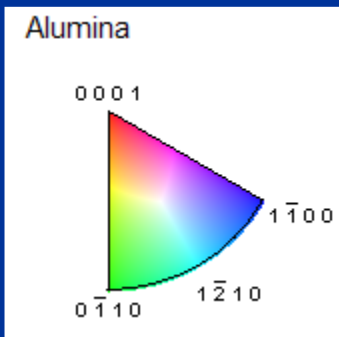
Image Quality map



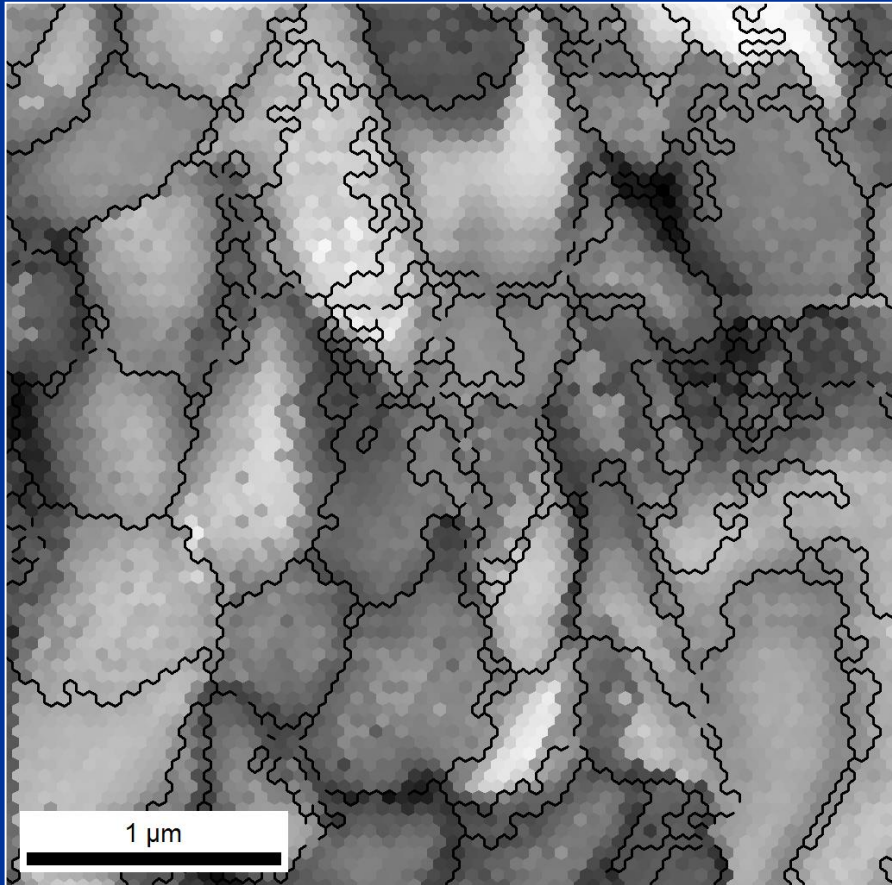
Inverse Pole Figure map



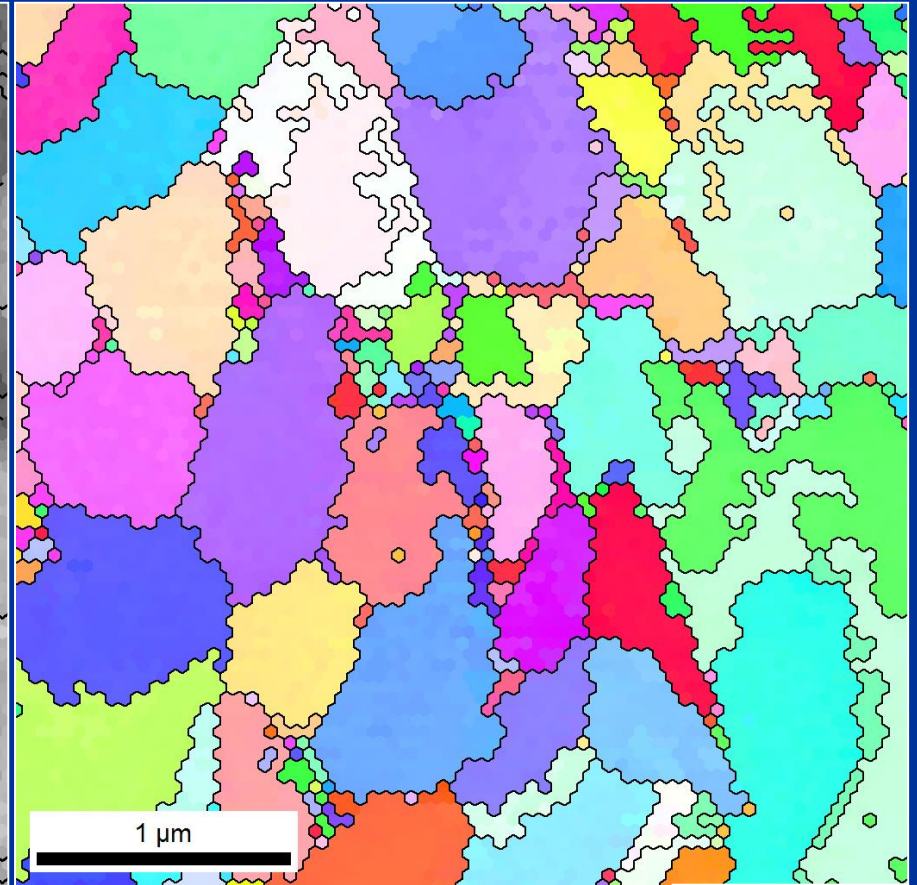
IPF + HAGB



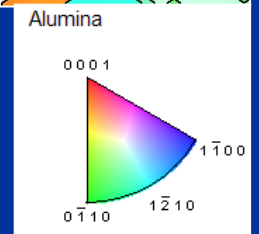
# Orientation maps from $\text{Al}_2\text{O}_3$ 5 keV



**IQ + HAGB**



**IPF + HAGB**



# Effective spatial resolution

is given by the smallest structure that can be discerned by the EBSD system.

It depends on:

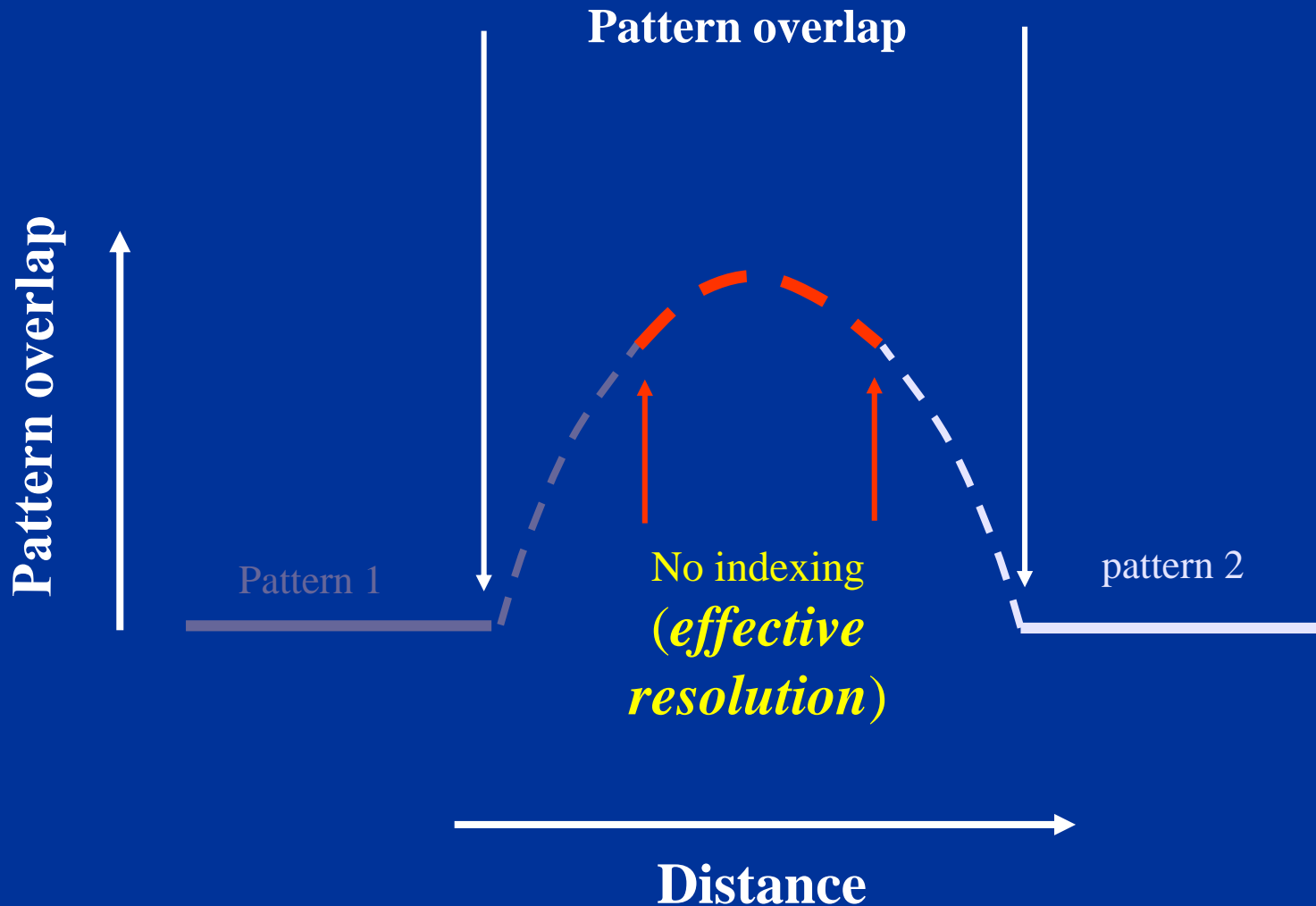
- the misorientation between two points
- software algorithms used for pattern indexing and pattern deconvolution

Effective spatial resolution:

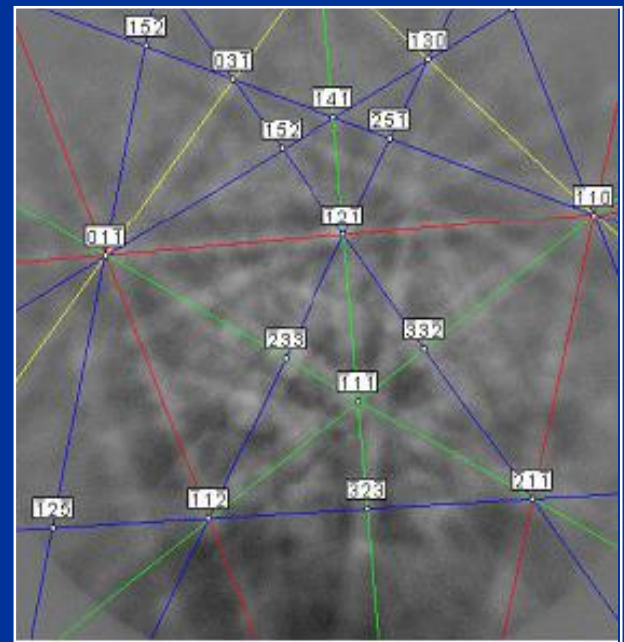
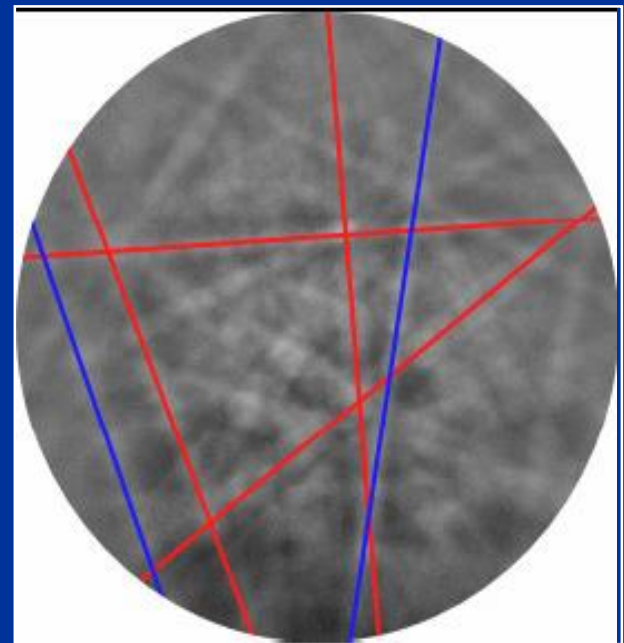
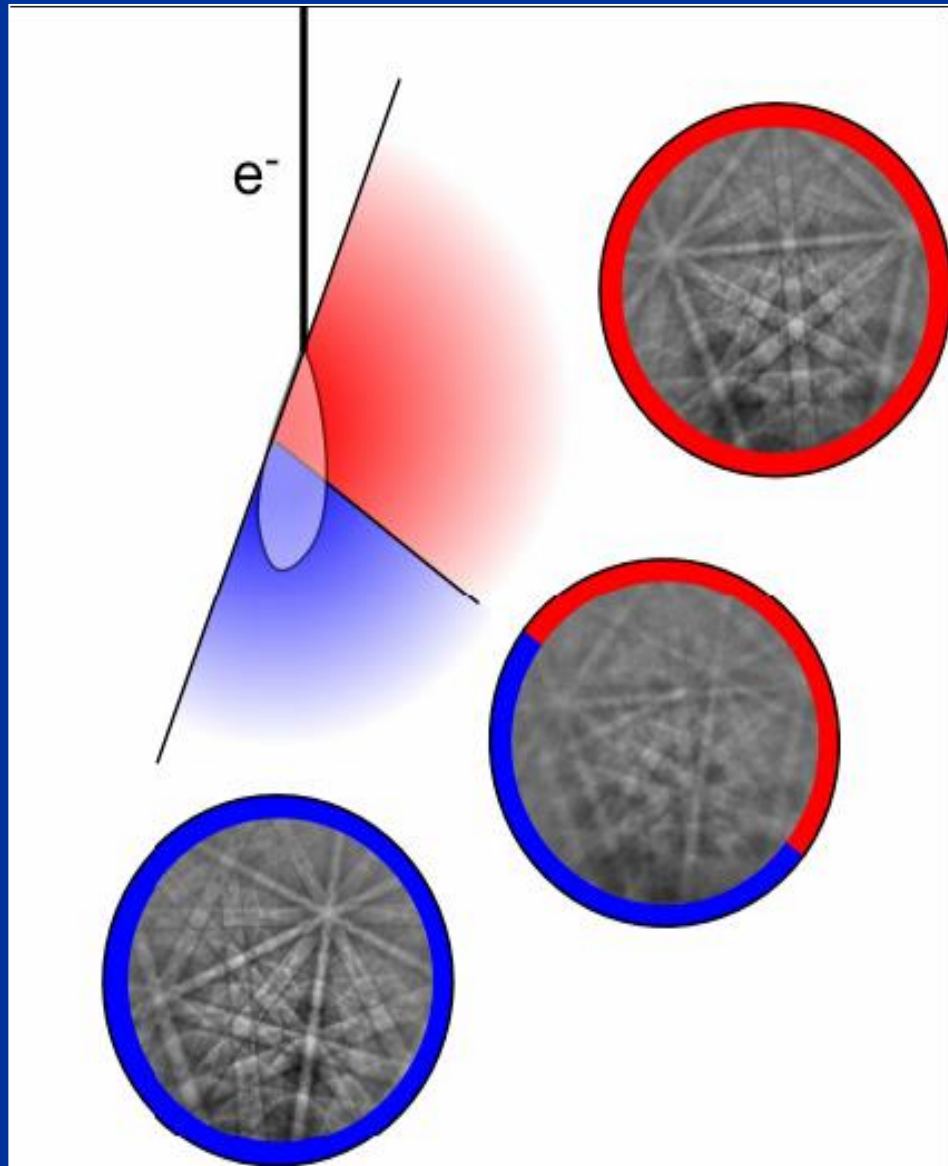
*the ability to deconvolute overlapping patterns at crystallite boundaries*

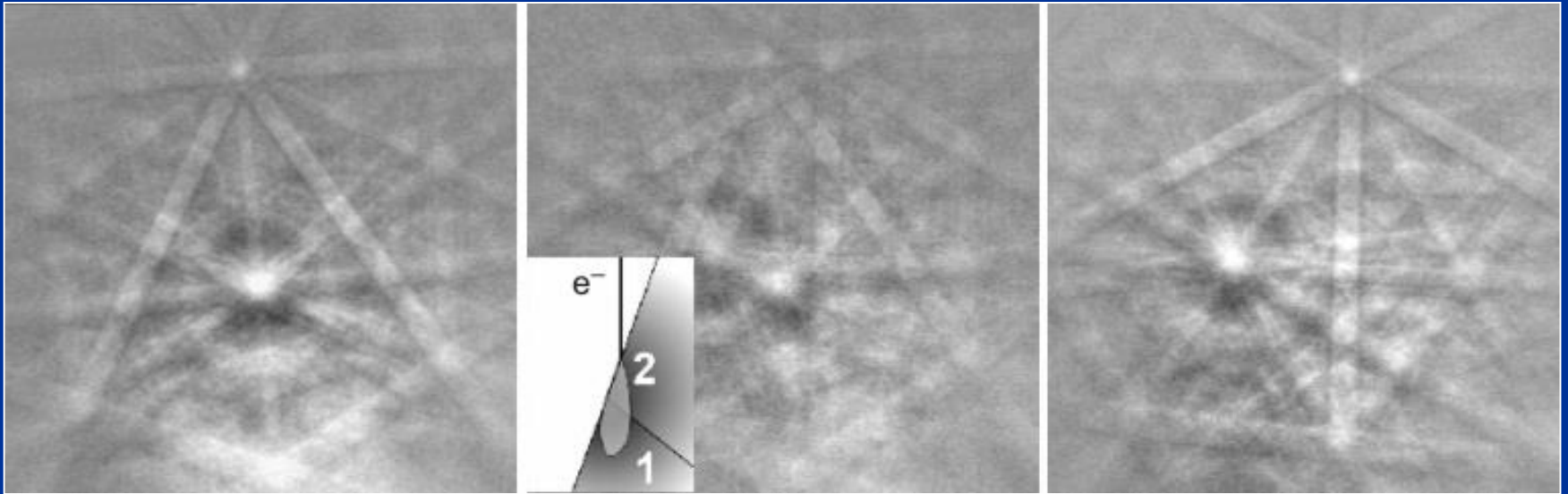


# Absolute Spatial Resolution



Can be measured by traversing the beam across a boundary normal to the sample surface





## **EBSD from Al<sub>2</sub>O<sub>3</sub> acquired in the Variable Pressure SEM**

Non-indexed points are in  
a narrow region at the grain boundary,  
where there are overlapping patterns

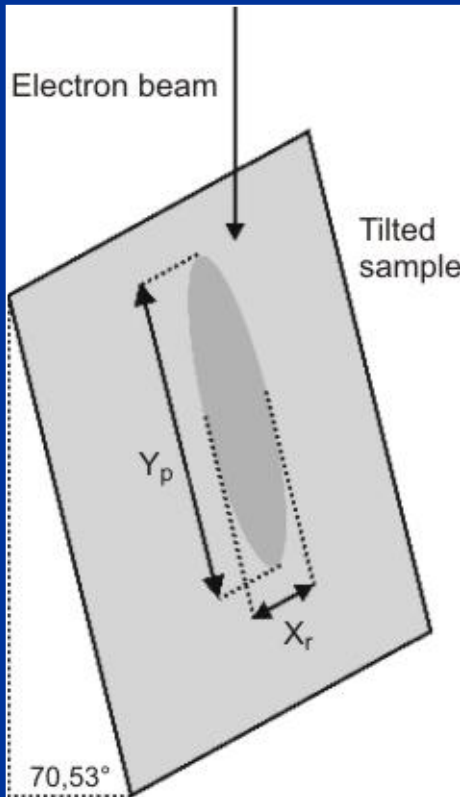
# Calculation of Effective Spatial Resolution

$$X_r = \frac{D(1 - H)}{4}$$

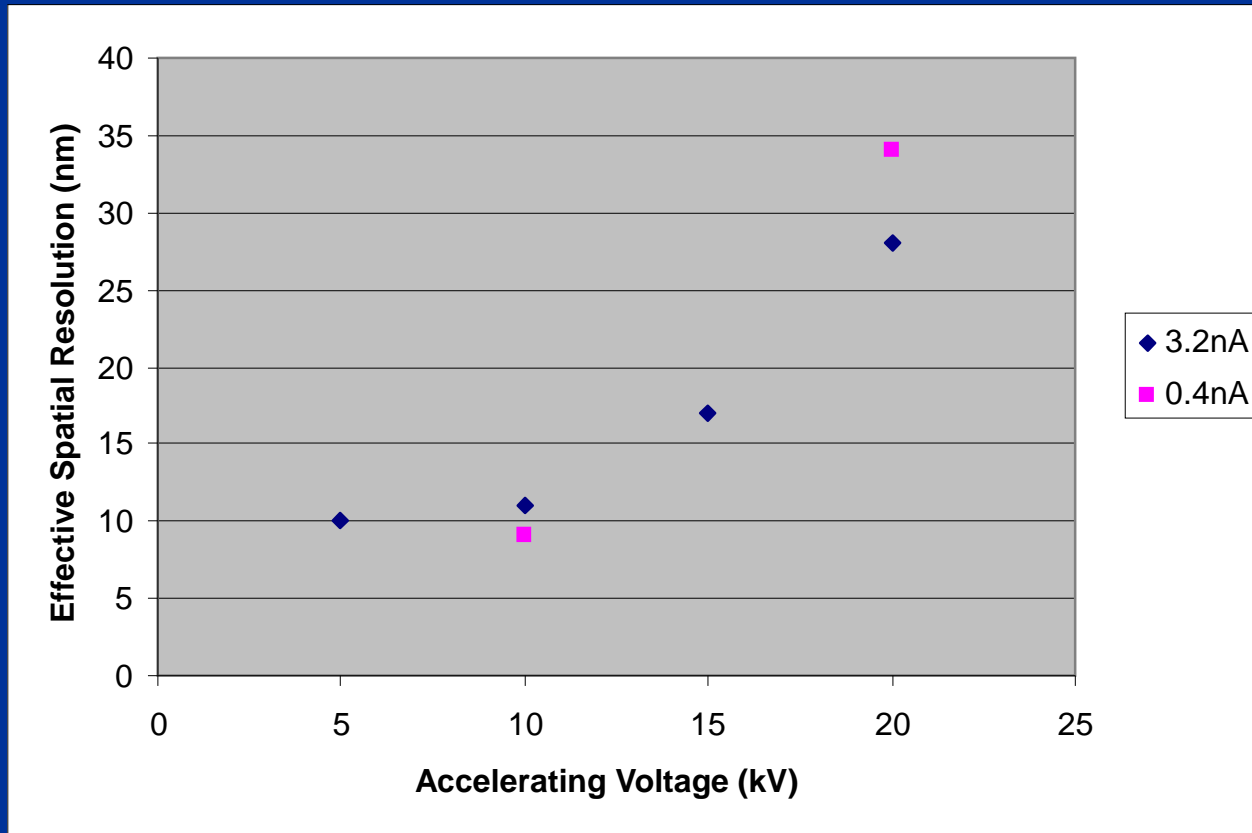
where:

**D** is the grain size of the sample

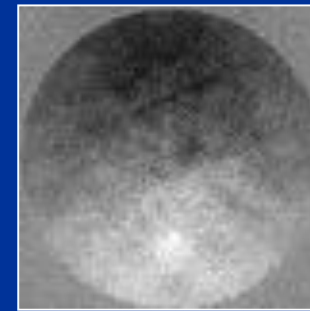
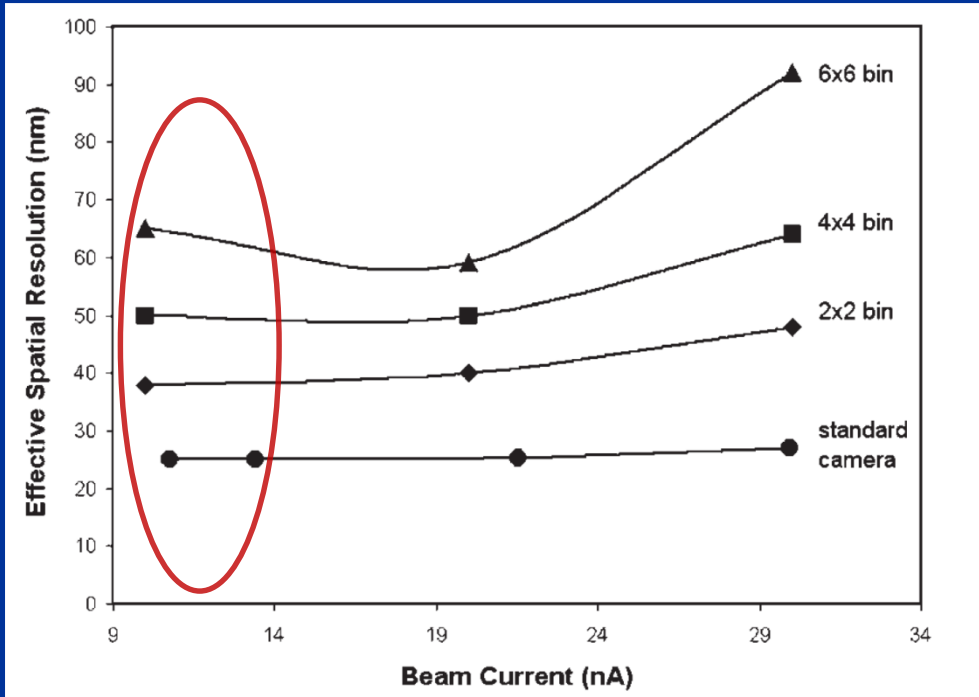
**H** is the fraction of patterns indexed (fractional hit rate).



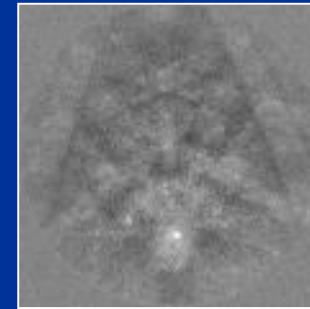
# Effective Spatial Resolution calculated for Al as a function of accelerating voltage and beam current



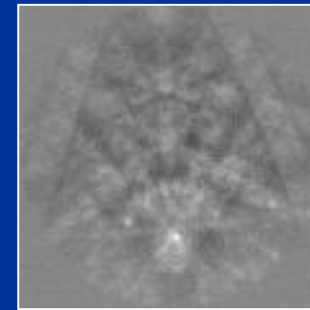
# Effective spatial resolution degrades with increased binning



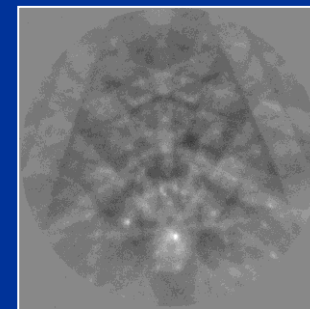
Binning 6x6



Binning 4x4



Binning 2x2



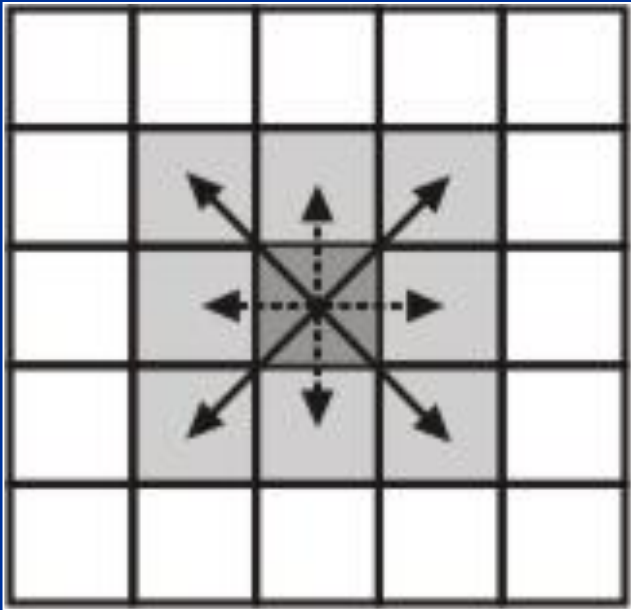
Binning 1x1

As binning increases, the bands become less well defined, the pattern quality is reduced and the **effective spatial resolution**, which depends on the ability to **deconvolute overlapping patterns at boundaries**, deteriorates.

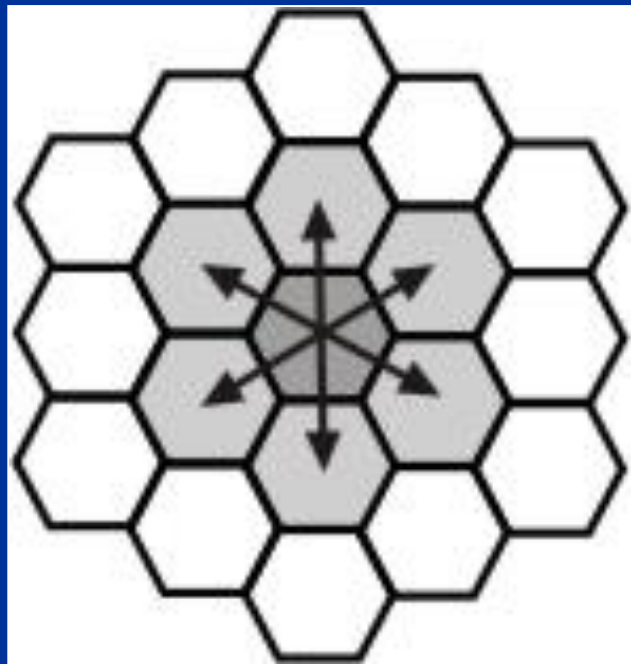
**The step size selected for a particular EBSD  
measurement is  
NO INDICATOR  
of spatial resolution!!!**

**If step size is smaller than the physical or effective  
resolution then it depends only on the ability of the  
software system to deconvolute the overlapping  
patterns of neighbouring volumes.**

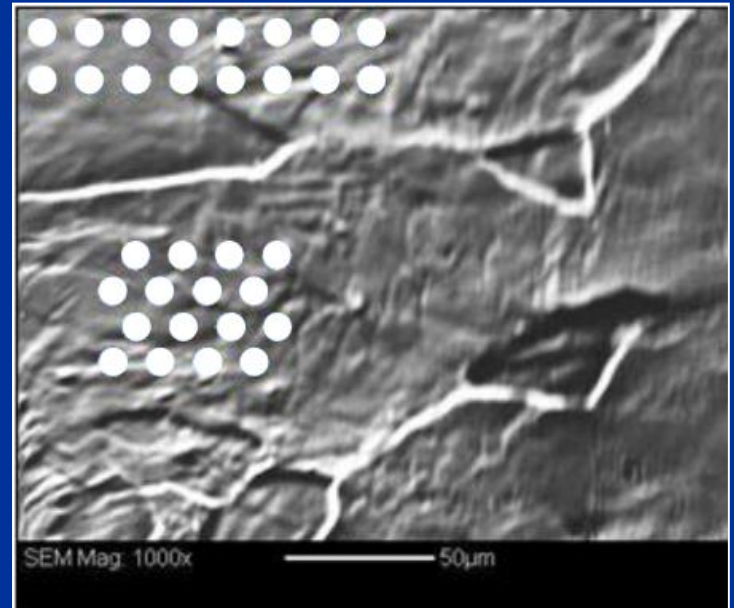
# Scanning mode versus spatial resolution



Square grid



Hexagonal grid





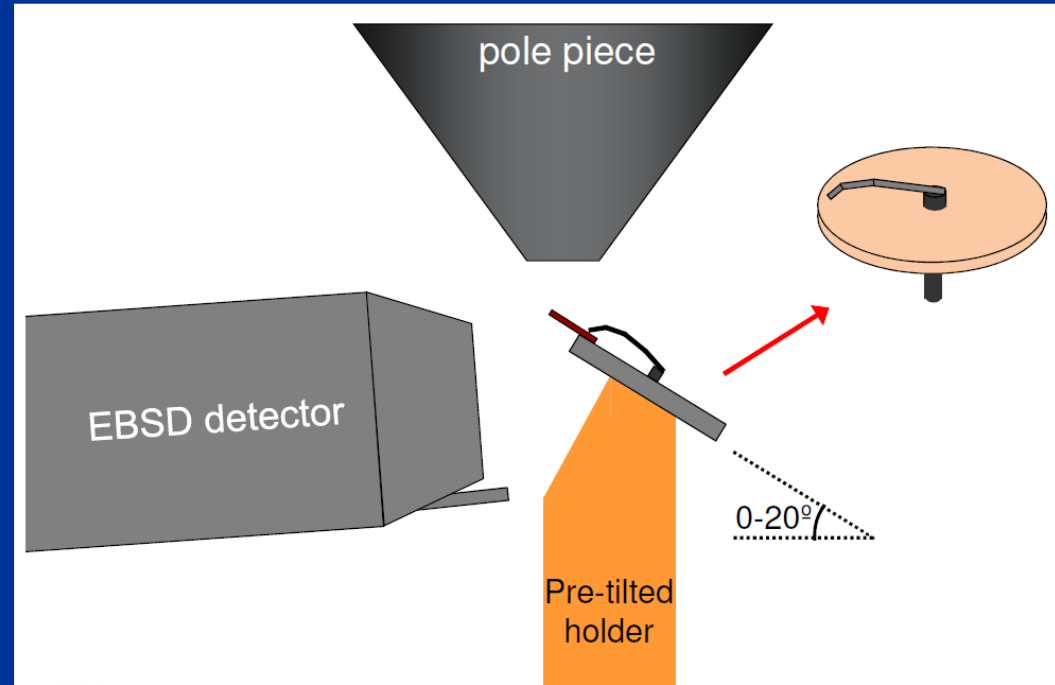
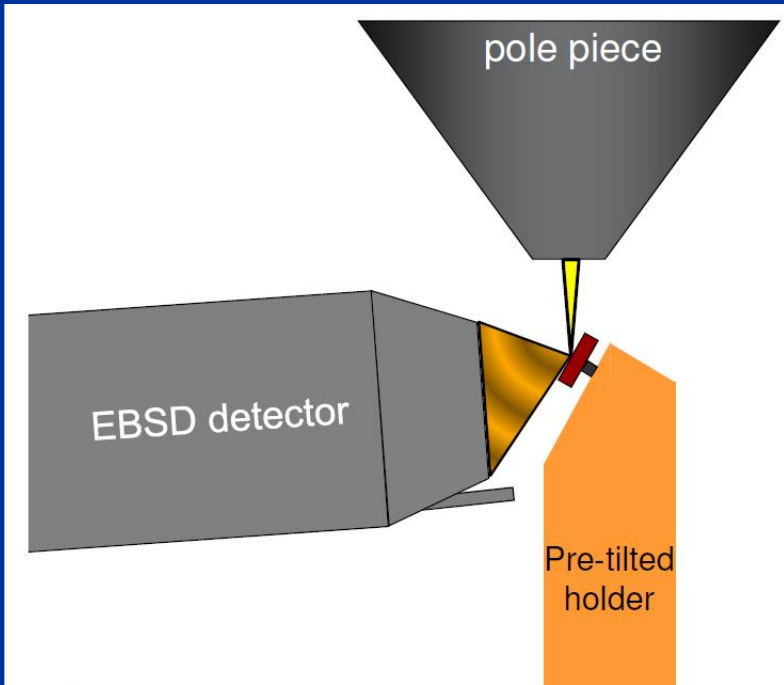
# *TKD versus EBSD*

# **t – EBSD or TKD**

**(transmission electron backscatter diffraction)**

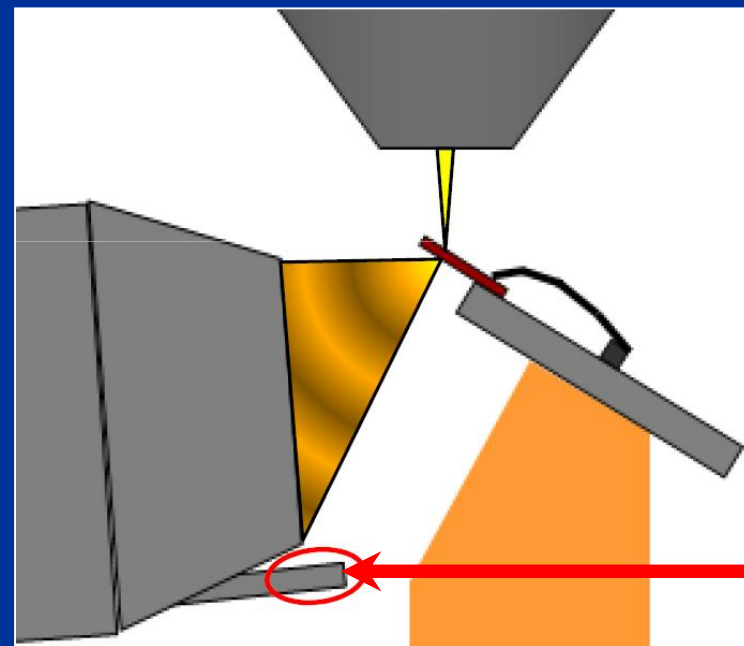
**(Kikuchi Diffraction Pattern in the SEM)**

- **t-EBSD/TKD uses a conventional EBSD system, including mounting, hardware and software**
- **FEG-SEM platform is an important component for the highest resolution work**
- **The difference (and innovation) between EBSD and t-EBSD/TKD is the placement and orientation of the sample in the SEM chamber**
- **Requires a thin sample !!!**



## Geometry: TKD in the SEM

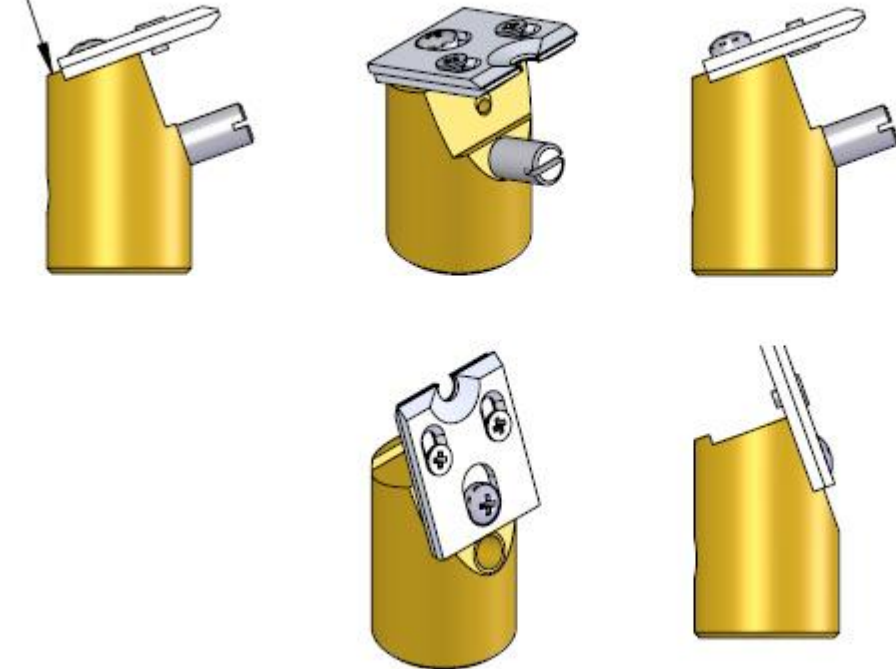
Courtesy of Scott D. Sitzmann,  
Oxford Instruments



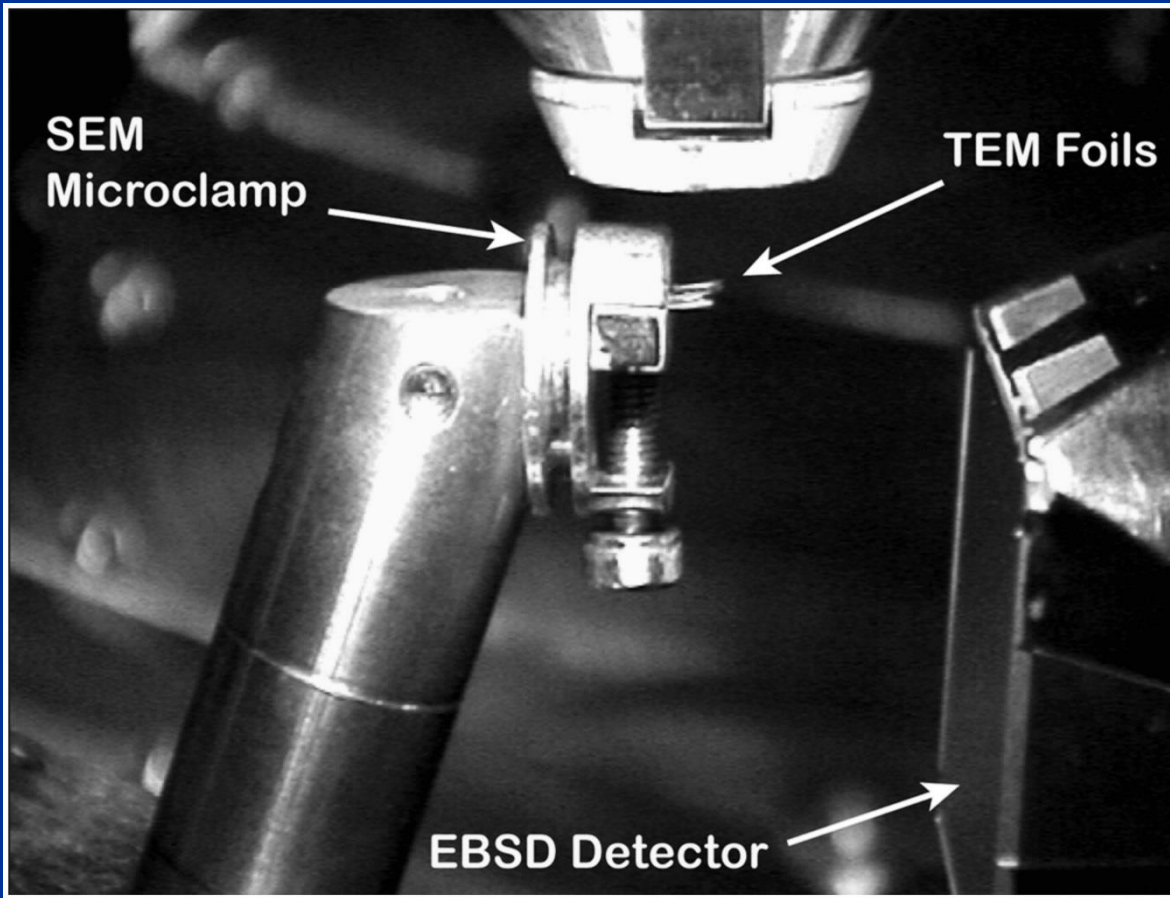
FSD  
(here:  
DF-STEM)

# SEM-TKD holder – a microclamp

SHOULDER TO  
PREVENT ROTATION

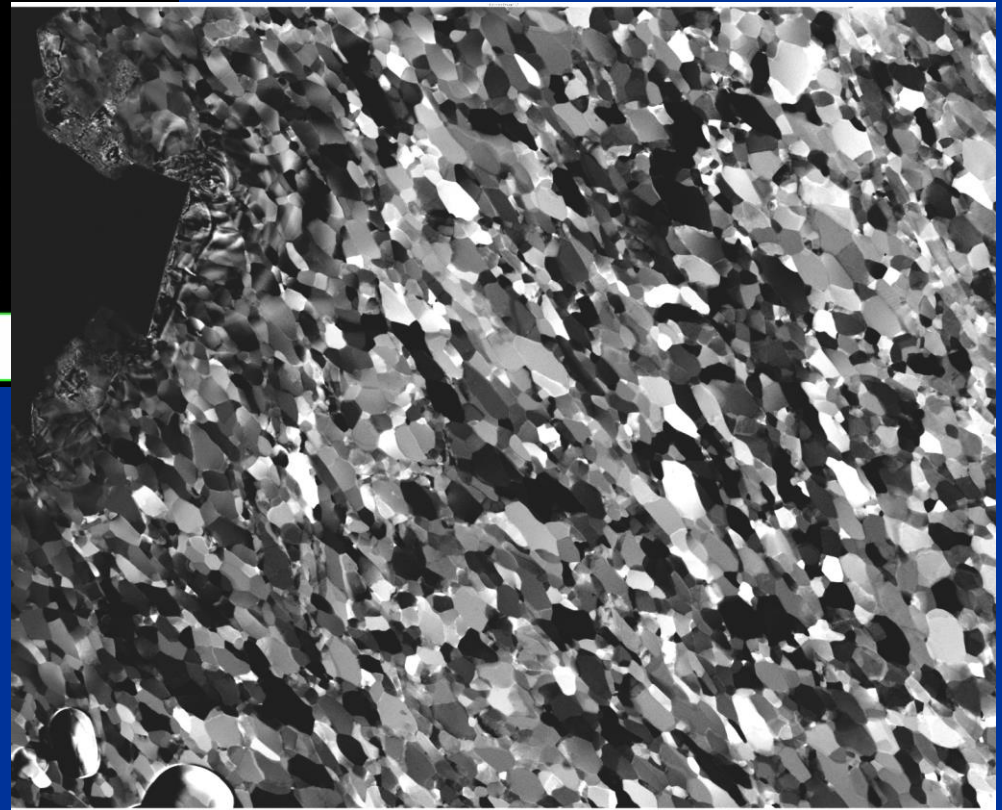
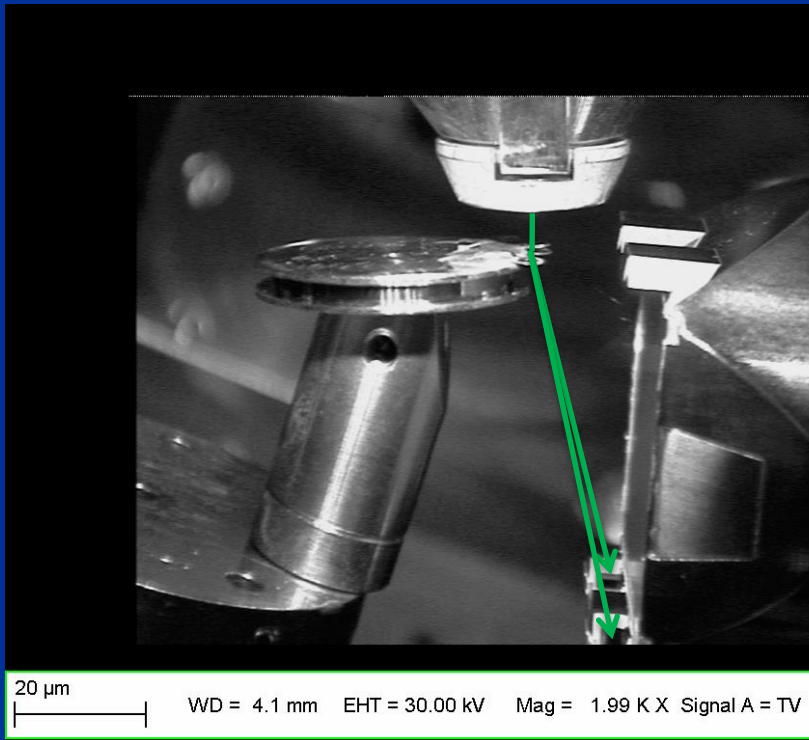


Sample holder  
In EBSD position

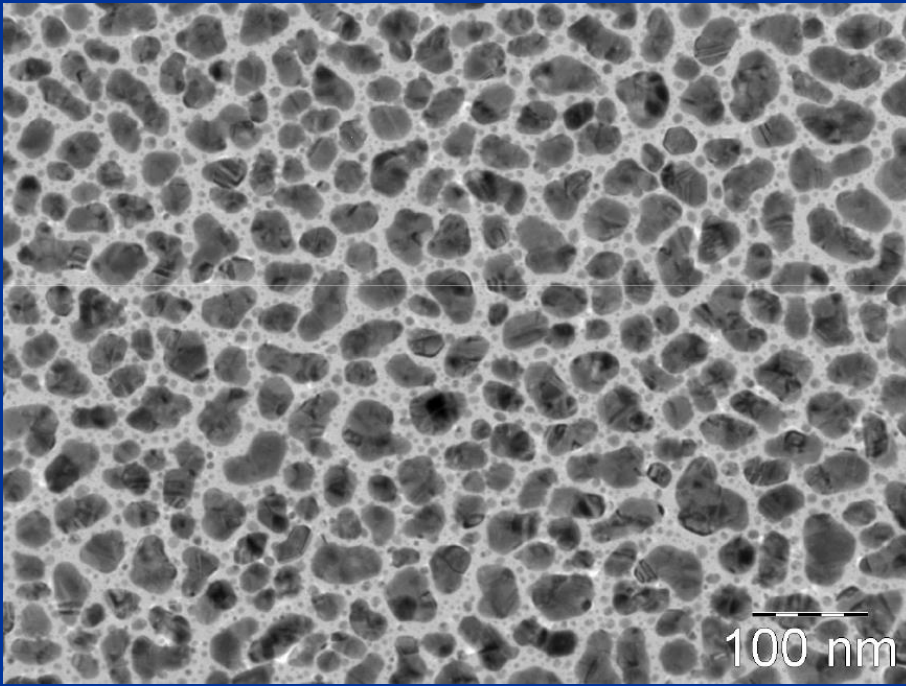


**Experimental set up for SEM-TKD, with the pre-tilted EBSD sample holder, the TEM foils held in a microclamp in a horizontal position, and the EBSD detector to the right.**

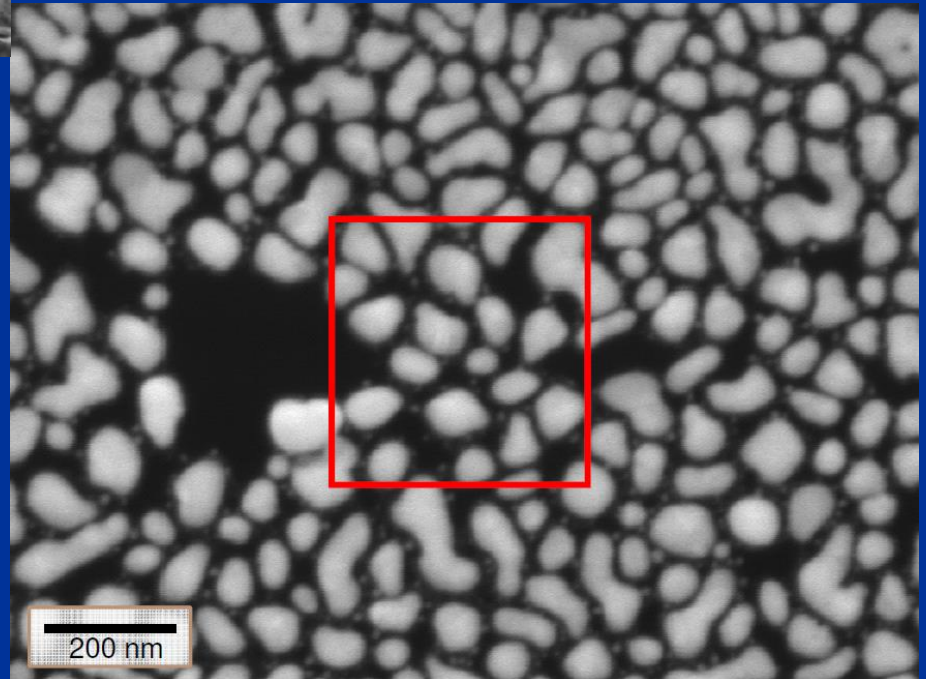
# FSE from Al after severe plastic deformation



**BF-TEM image  
gold islands**

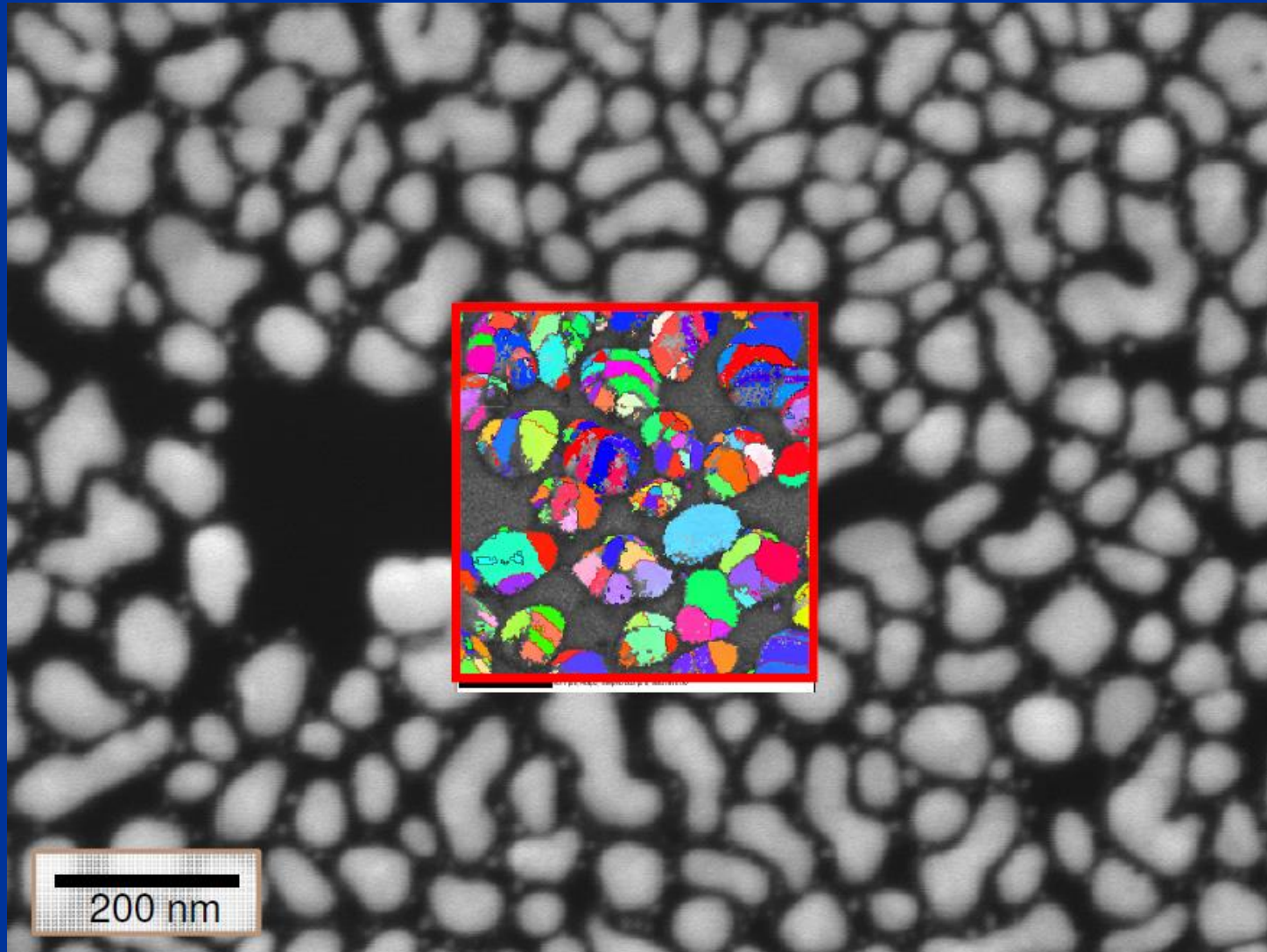


**STEM-FSE image  
gold islands**



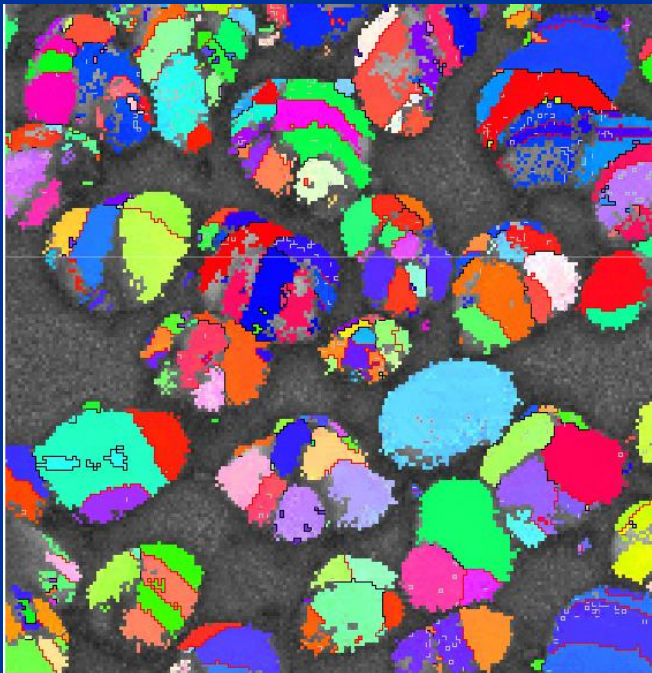
Sample courtesy S. Prihodko (UCLA),  
TEM work by Roy Boulder (NIST)

# STEM-FSD image with IPF map

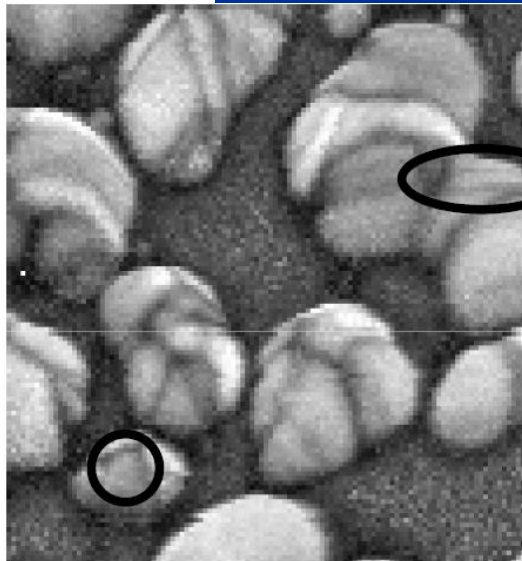
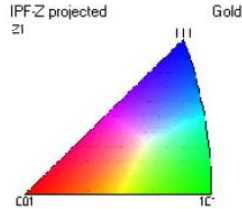


Courtesy of Scott D.Sitzmann,  
Oxford Instruments

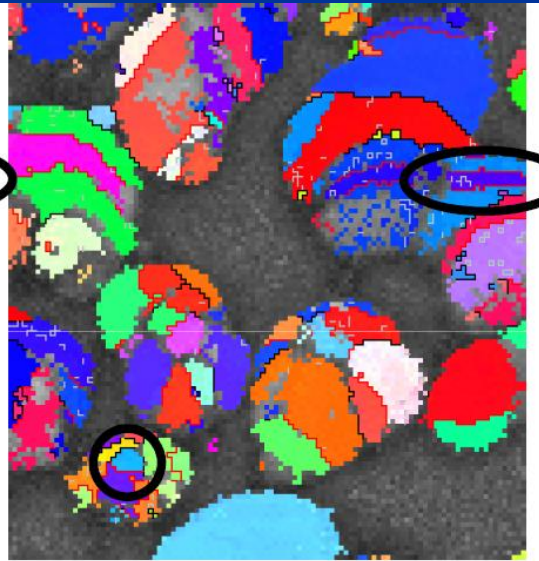




=0.1  $\mu\text{m}$ ; Map2; Step=0.002  $\mu\text{m}$ ; Grid191x197



100 nm



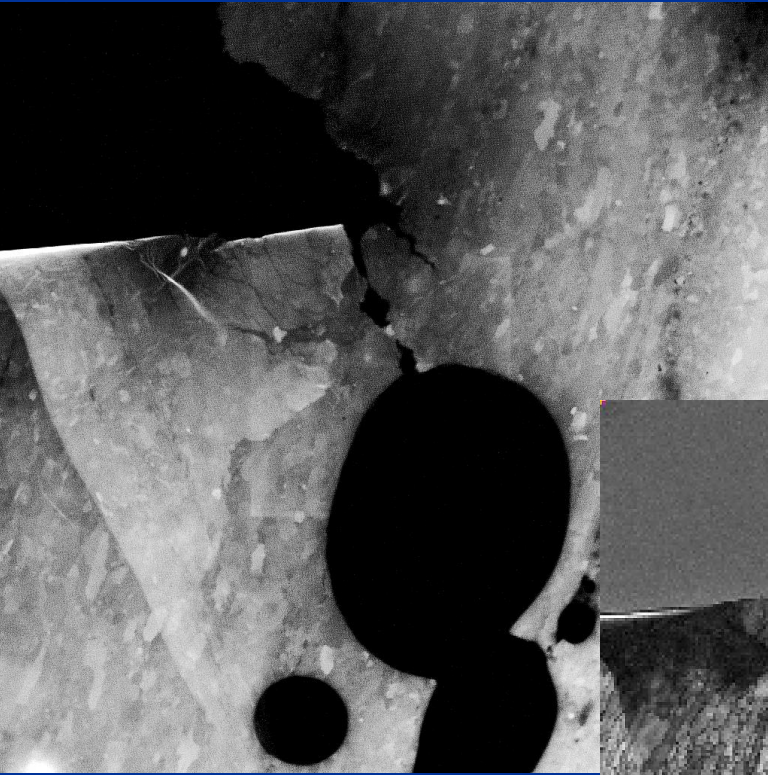
Each pixel = 2nm



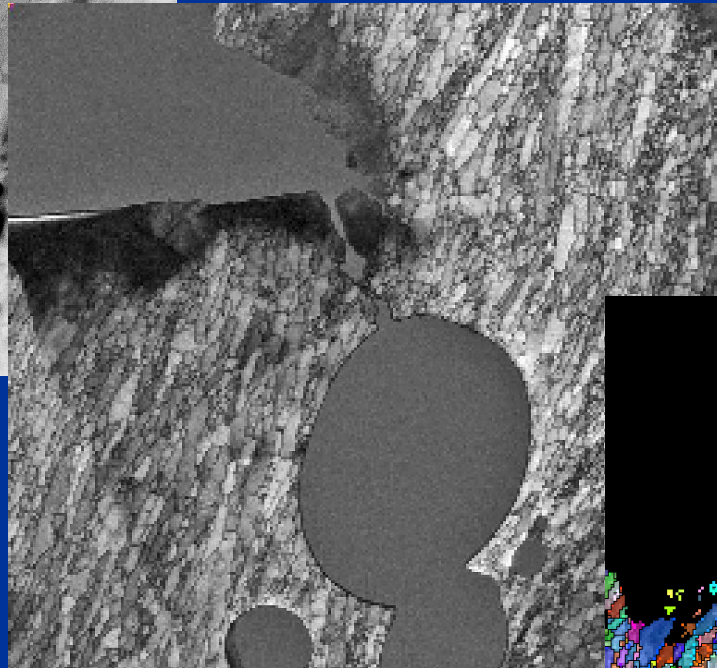
~10 X 15nm twin domain

Courtesy of Scott D.Sitzmann  
Oxford Instruments

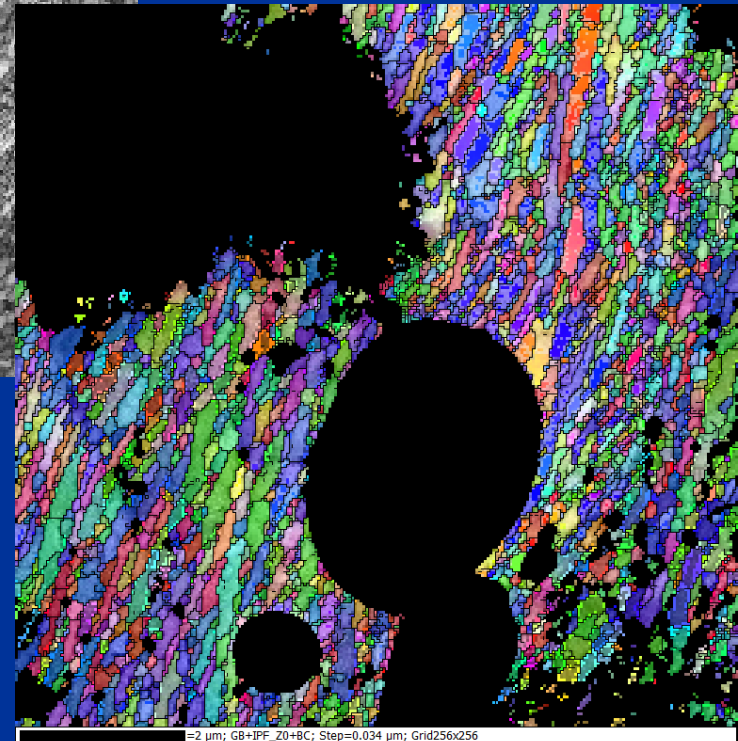
# Highly deformed Al 6060 alloy



FSE image

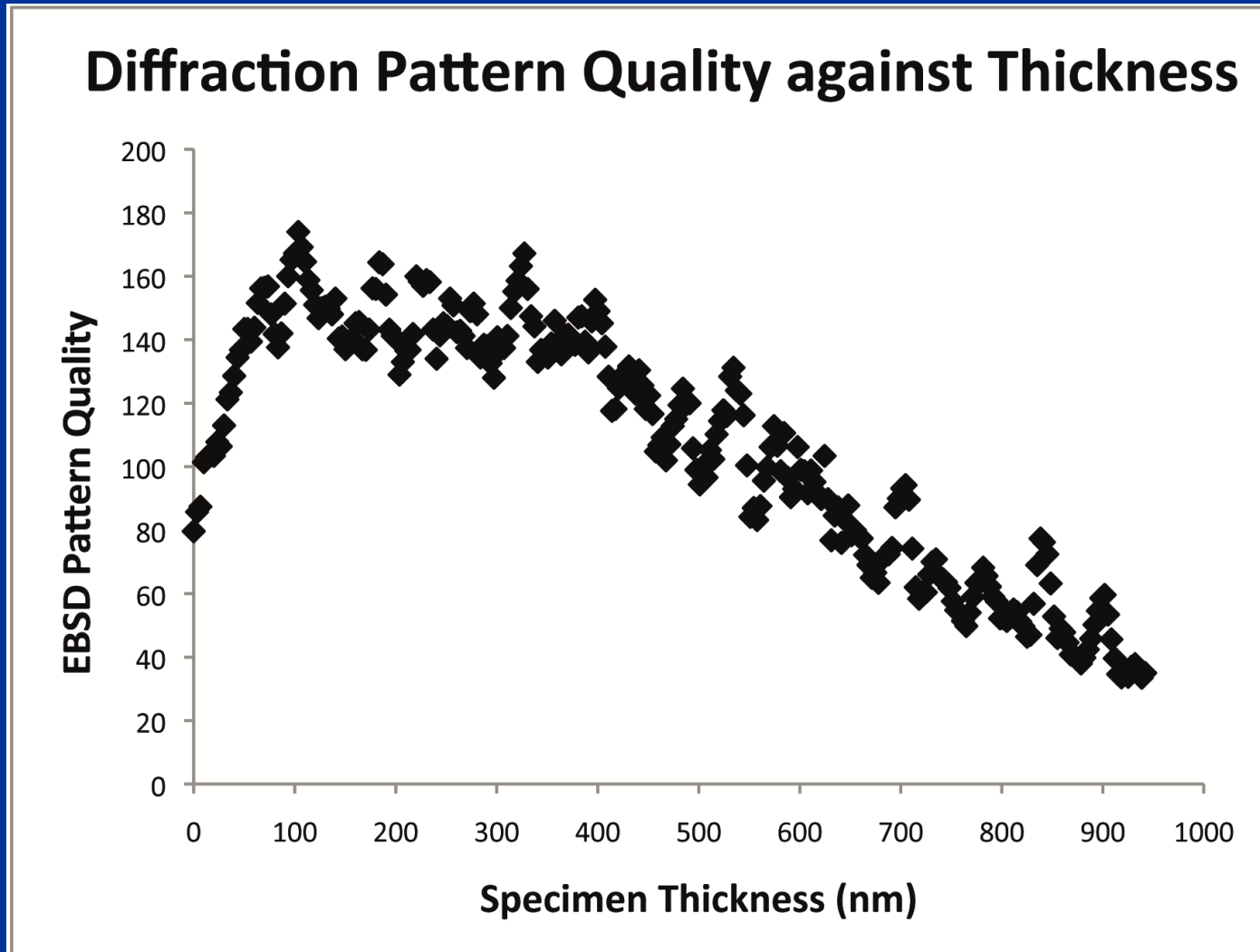


Band contrast map

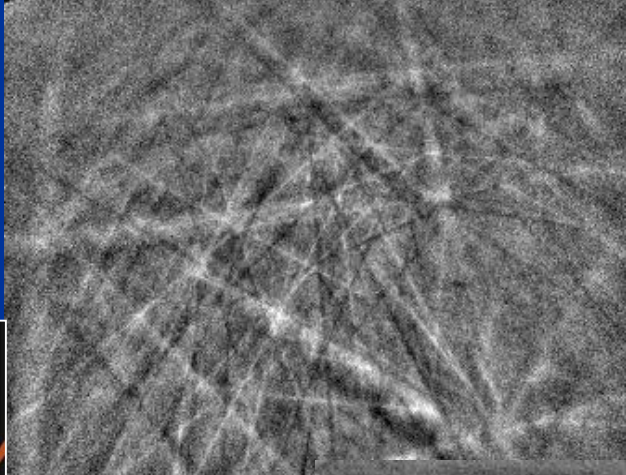


IPF orientation map

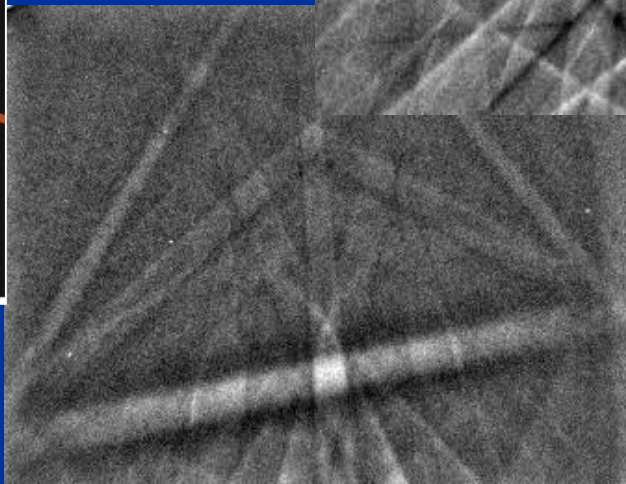
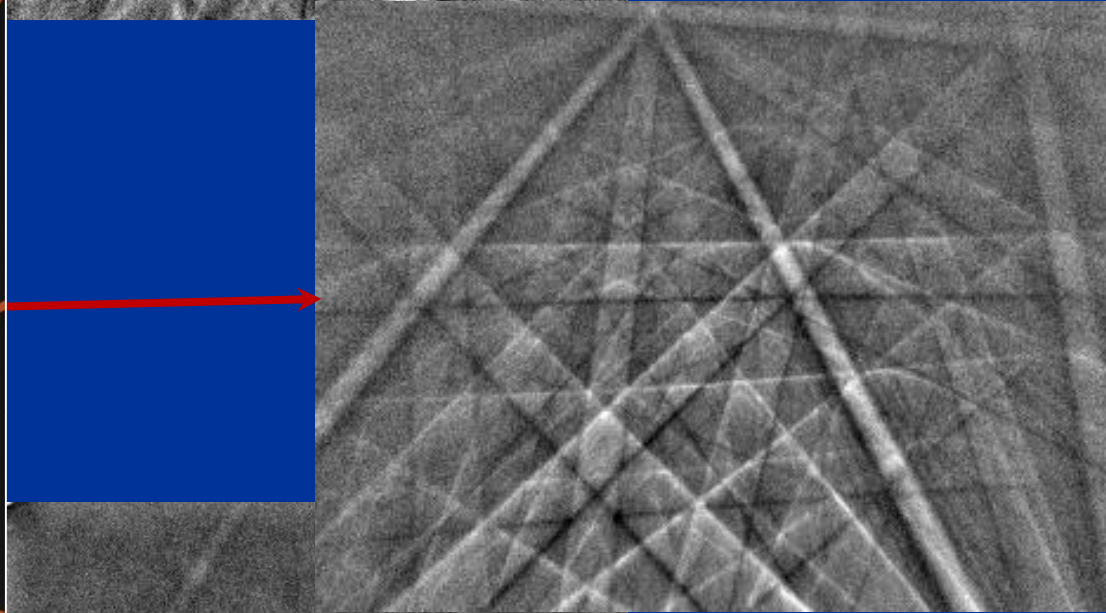
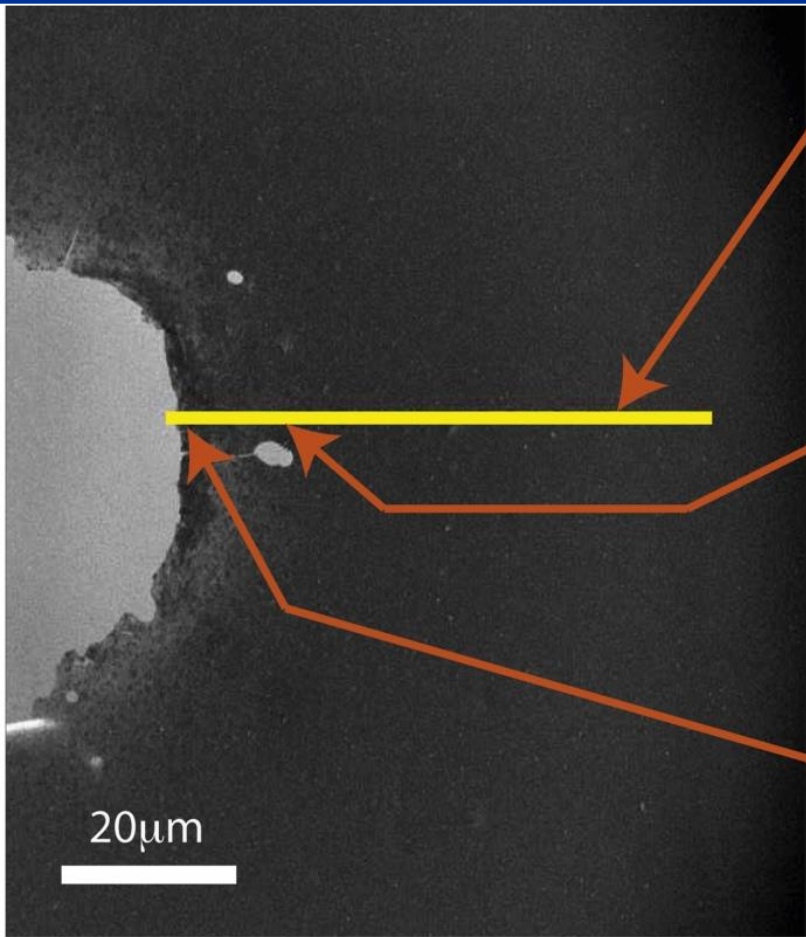
# EBSP quality vs thickness for Al thin foil



**Too thick:** Lateral beam spread may produce **overlapped patterns**, depending on grain size, Z, kV

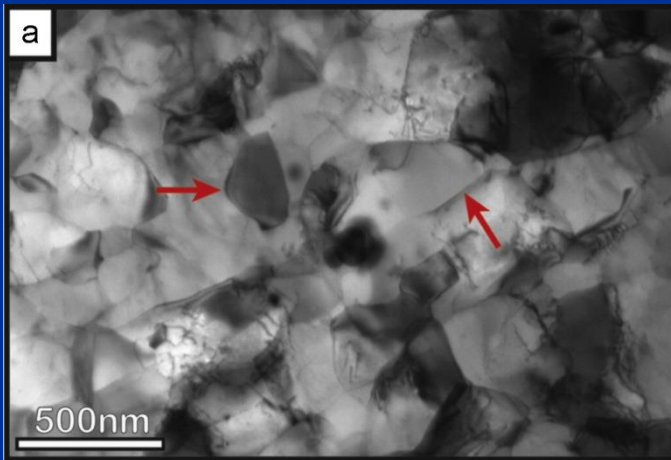


Optimal range



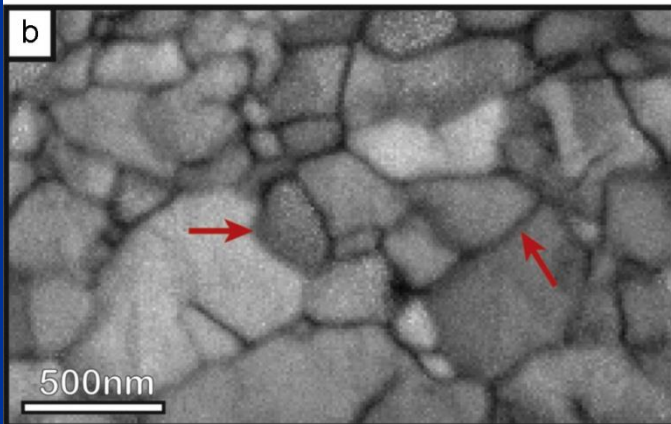
**Too thin**  
(weak signal but still workable)

Secondary electron (SE) image of the central perforation in an **Al 6060** TEM foil. The yellow line marks a transect along which diffraction patterns were stored and analyzed.

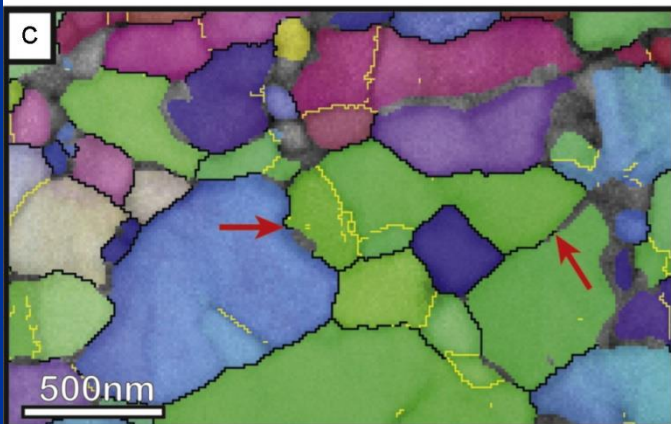


**A comparison between STEM imaging in the SEM and TKD orientation mapping in aluminium alloy Al6060.**

(a) A BF STEM image collected in the SEM.



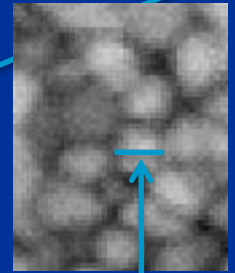
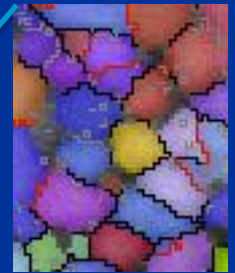
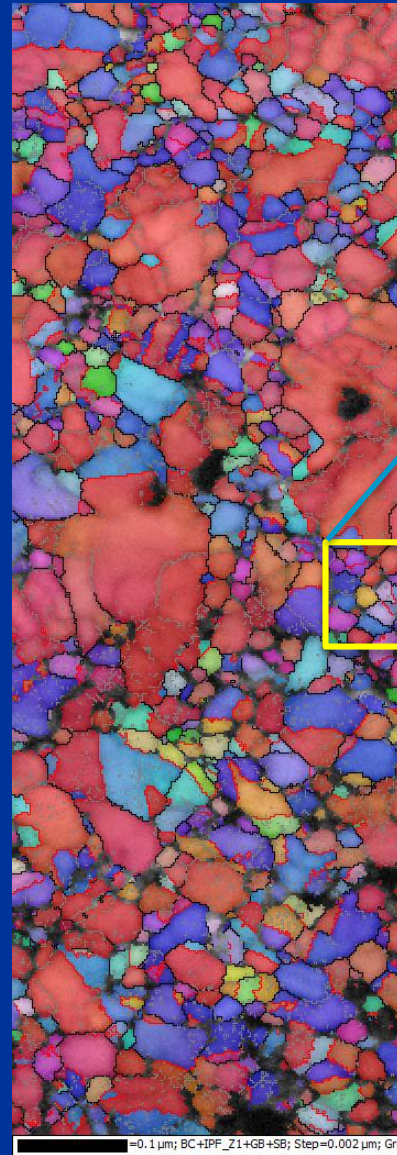
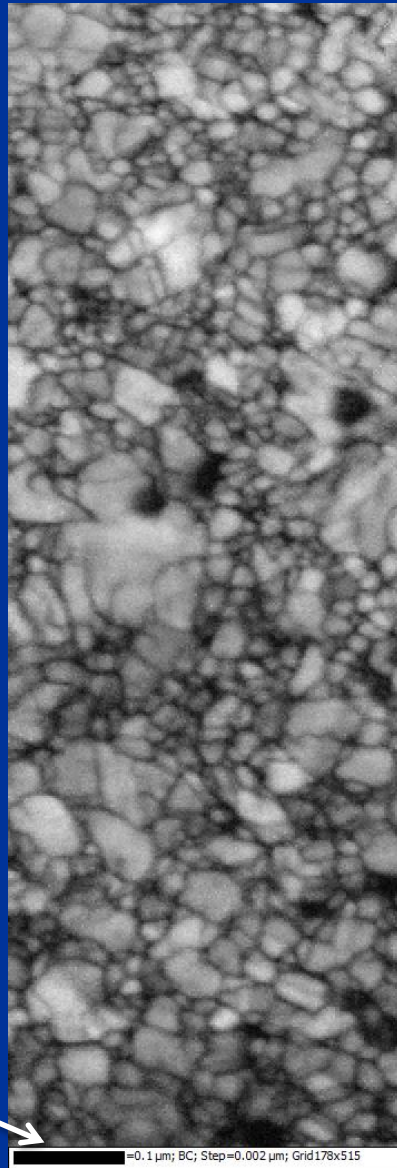
(b) TKD pattern quality map (band contrast) of the same area, collected using a 10 nm step size.



(c) Cleaned orientation map (IPF z-direction coloring scheme), showing high angle boundaries ( $>10^\circ$ ) in black, and low angle boundaries ( $2-10^\circ$ ) in yellow.

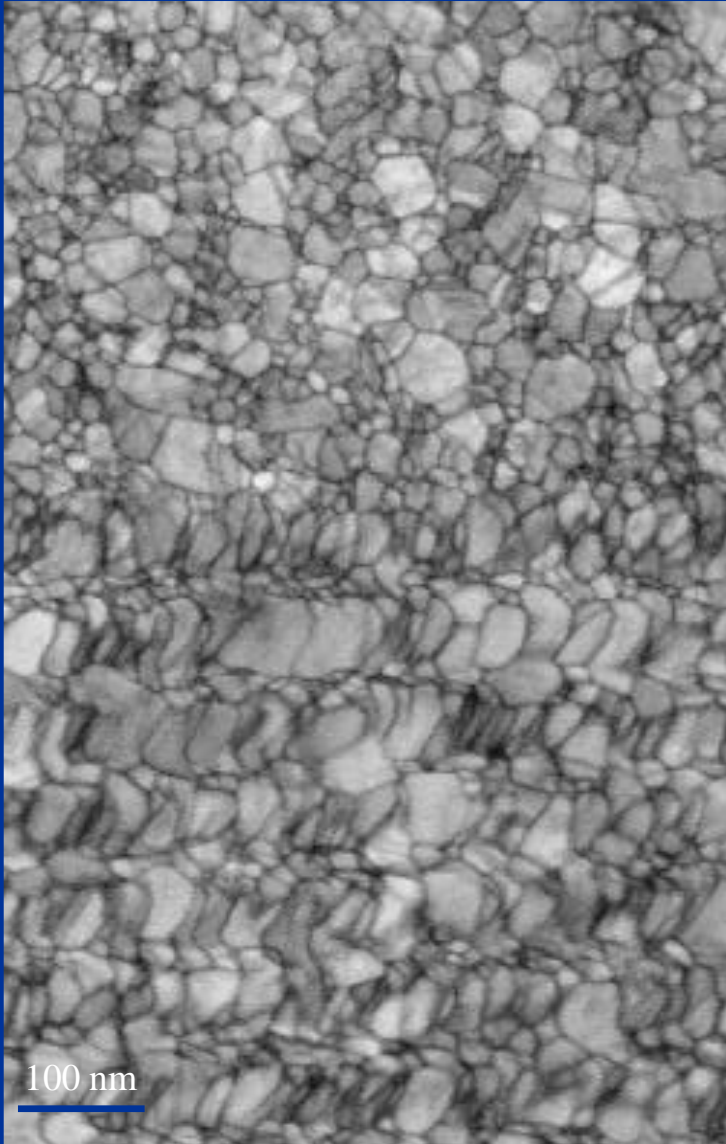
# Electrodeposited nano-Ni

100 nm  
(Step: 2 nm)



18nm

Since TKD generally uses very small step sizes with high electron current, it is especially vulnerable to drift !!!



#### Drift

Best results at highest resolution after

letting stage stabilize for 1-3 hours

Vertical drift: image out of focus

Good depth of field beneficial

#### Contamination

Clean samples and good vacuum  
necessary

Scanning adjacent to region of interest for  
10-60 minutes can help

Sensible choice of map shape (short rows)

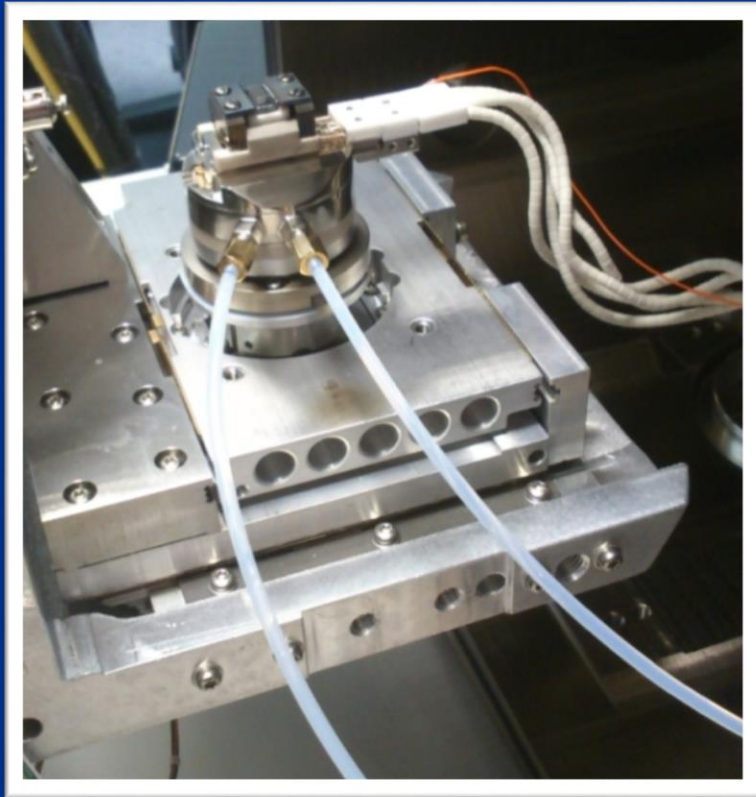
#### Lab instabilities

Air conditioning and coolant temperature

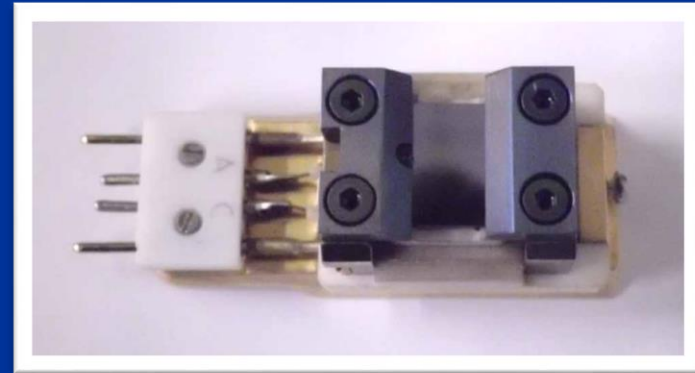
*In situ EBSD  
experiments*



# In situ experiments in the SEM



*Gatan 950 °C Murano 525 heating stage with UBS Temperature Controller in the microscope chamber*

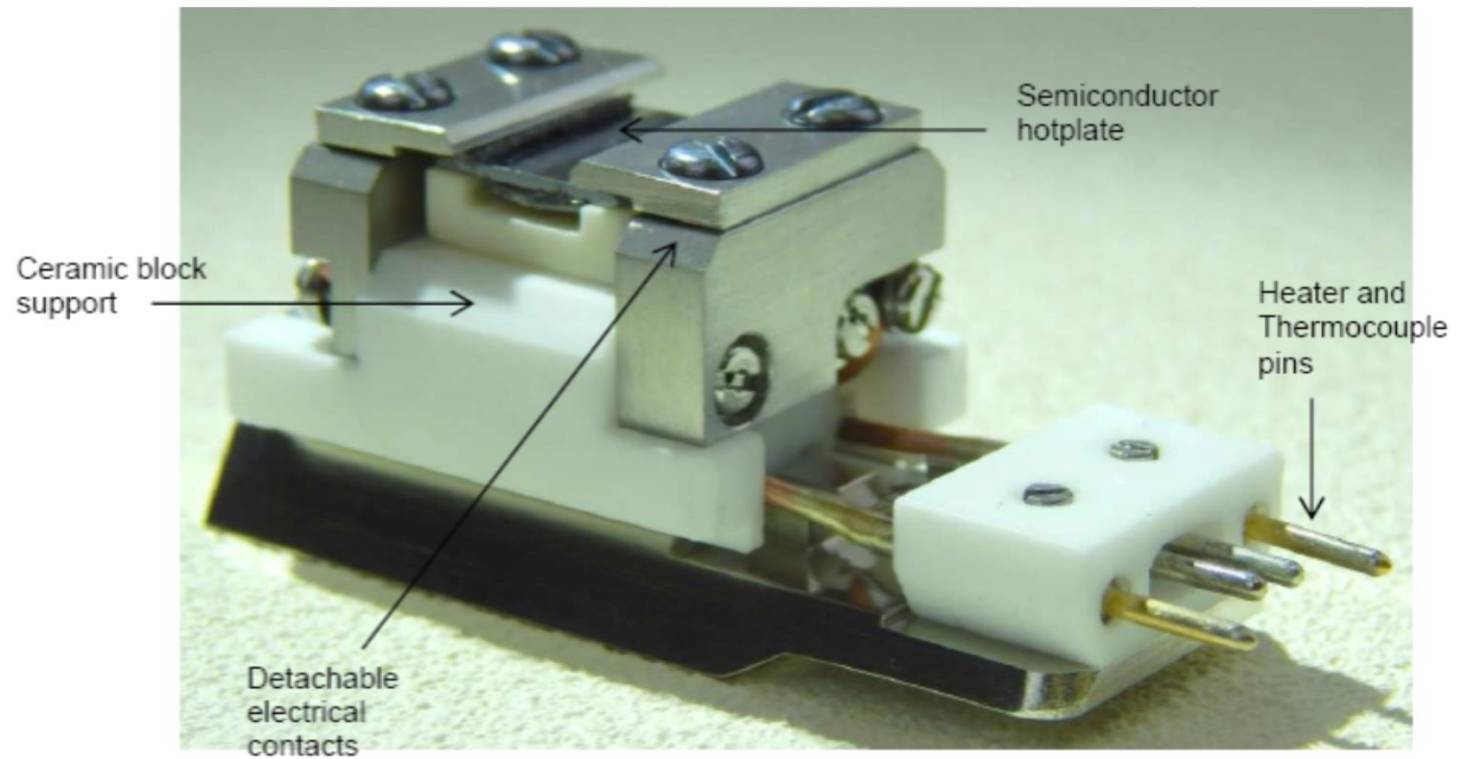


*Specimen plate*

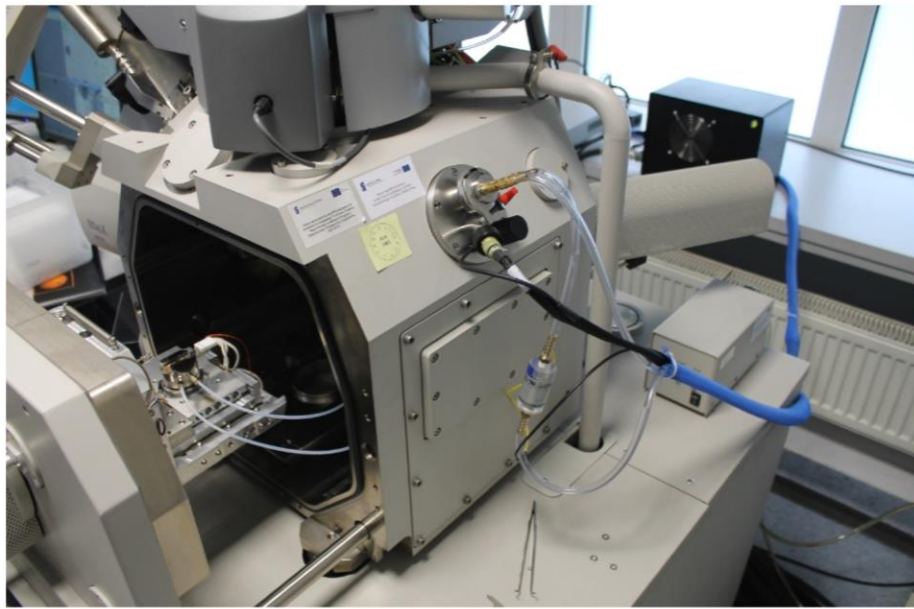


*Main window of temperature control software*

# In situ experiments in the SEM

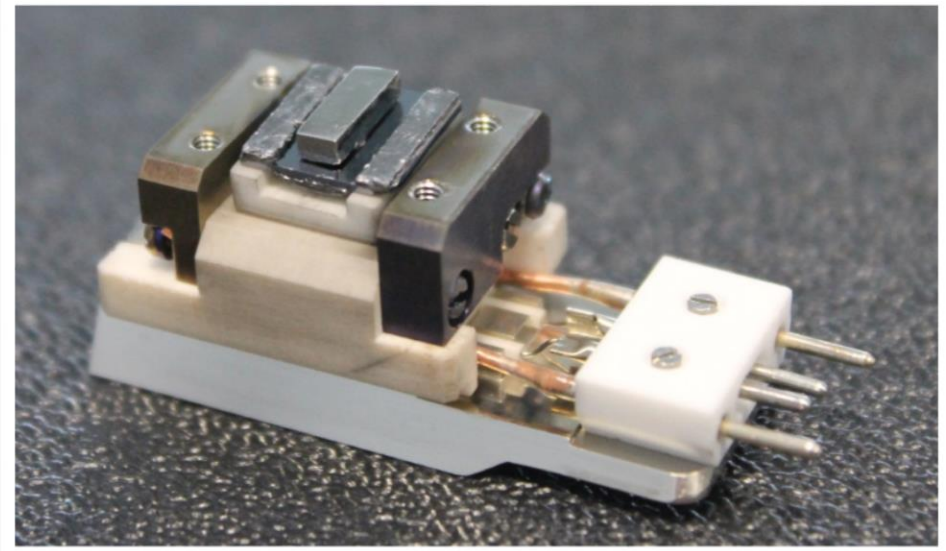
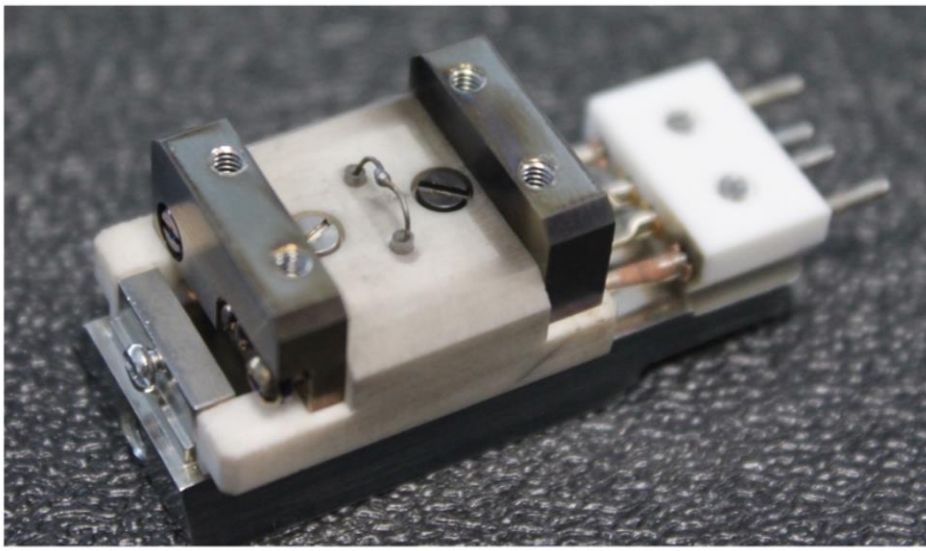


*Gatan 950 °C Murano 525 heating stage*



## Heating stage parameters

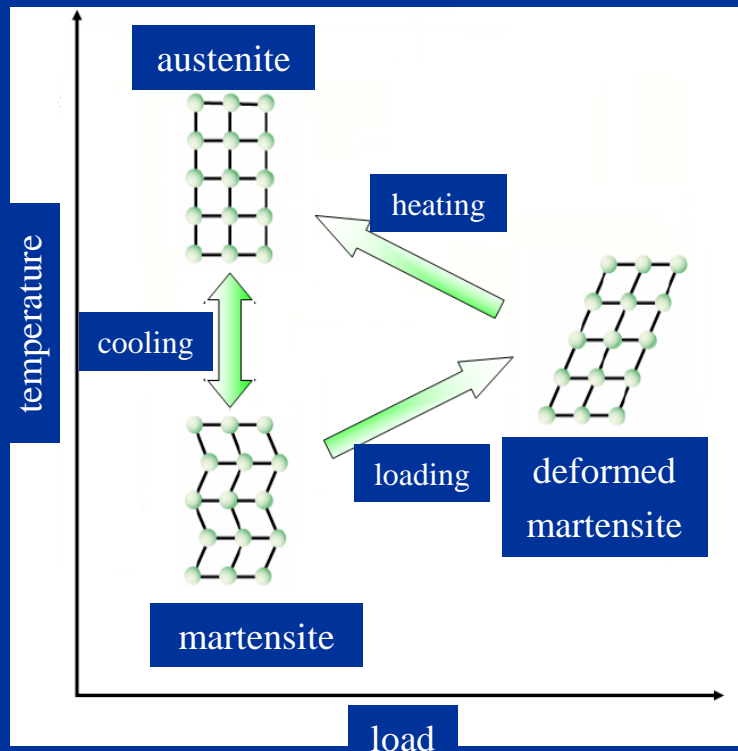
- First heating stage for rapid *in-situ* specimen characterization
- Monitor phase transformation from ambient to 950 °C
- Single specimen for entire heating range, provides continuity and optimizes speed of characterization
- Controlled specimen heating to >100 °C per minute for 9 mm (L) x 4 mm (W) x 1.5 mm (H) samples



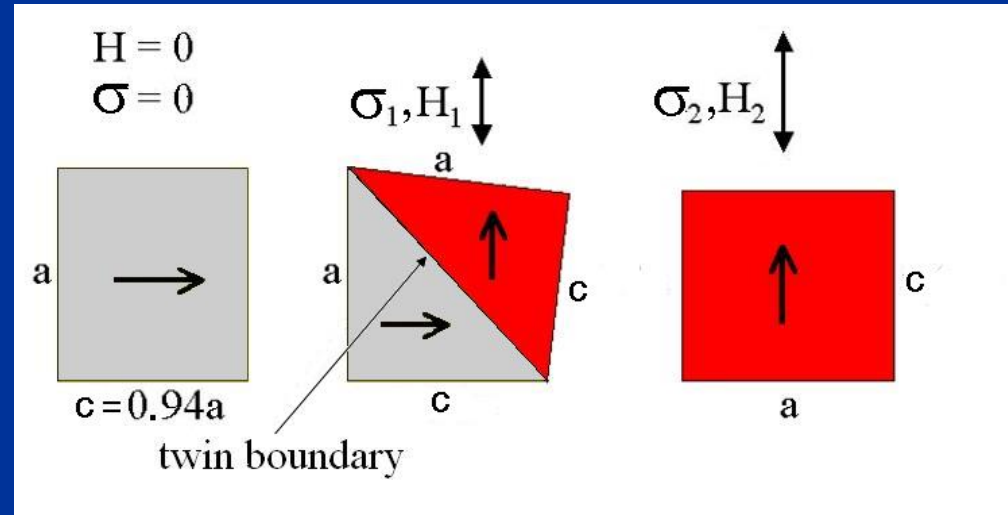
Gatan 950 °C Murano 525 heating stage

# Magnetic field induced strain

Requirements: high magnetocrystalline anisotropy, highly mobile twin boundaries



Shape-memory effect



Magnetic shape-memory effect  
(Magnetic Field Induced Strain)

**MFIS is based on the easy twin boundary motion and a high magnetocrystalline anisotropy in the martensitic state. Due to the tetragonal NM (non-modulated) or 7M or 5M (modulated) monoclinic structures, a macroscopic strain occurs by twin boundary motion exchanging the a- and c-axes.**

# System Ni-Mn-Ga

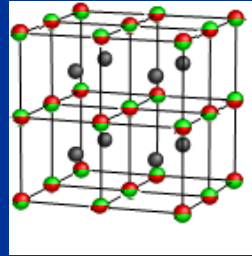
B2' structure

Random  
distribution of  
Mn and Ga

1100°C

≈750°C

$a=b=c$



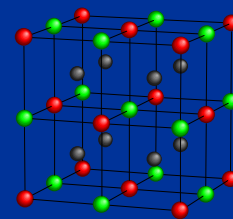
Ni ● Mn/Ga ●

austenite

L2<sub>1</sub> ordered  
structure

105°C

$a=b=c$



Ni ● Mn ● Ga ●

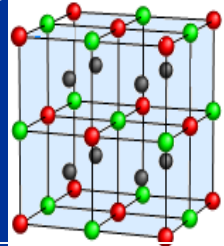
Martensite

3 variants

7M

$a>b>c$ , modulated  
c easy axis

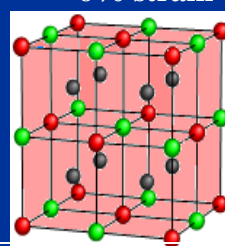
10% strain (single crystal)



5M

$a\approx b>c$ , modulated  
c easy axis

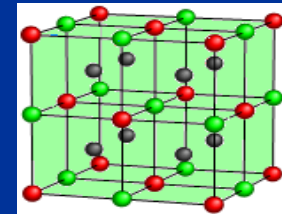
6% strain



NM

$a=b<c$ , unmodulated  
(100) (010) easy plane

20% strain (only mech.)

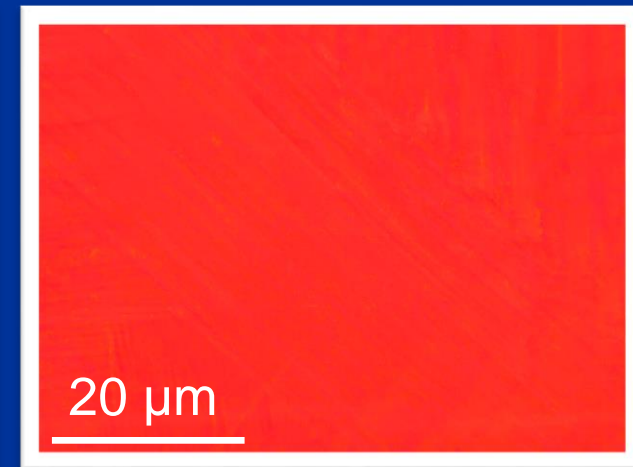
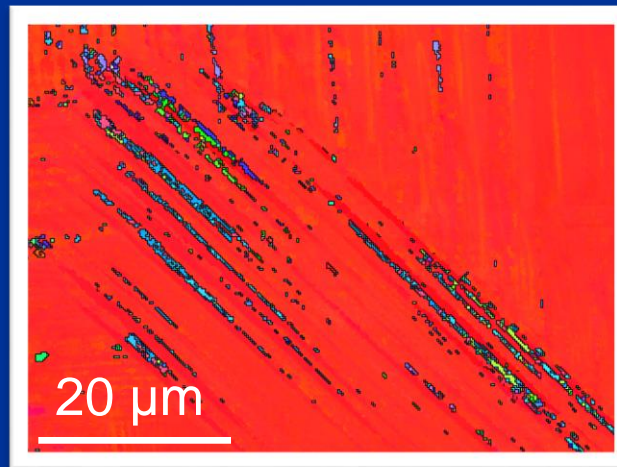
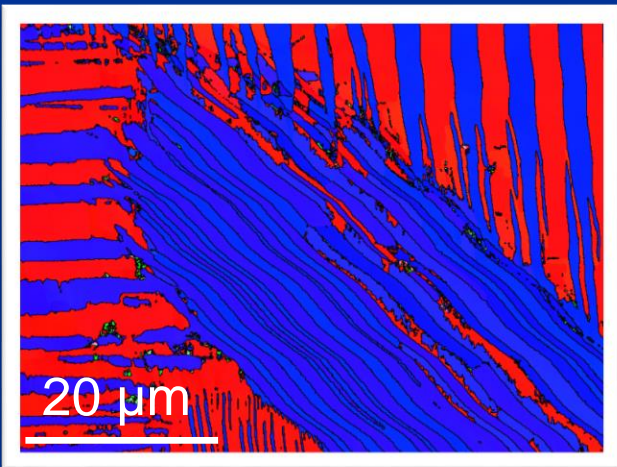


# $\text{Ni}_{50}\text{Mn}_{30}\text{Ga}_{20}$ non-modulated structure ( $A_s \sim 80^\circ\text{C}$ , $A_f \sim 90^\circ\text{C}$ )

25°C

90°C

105°C

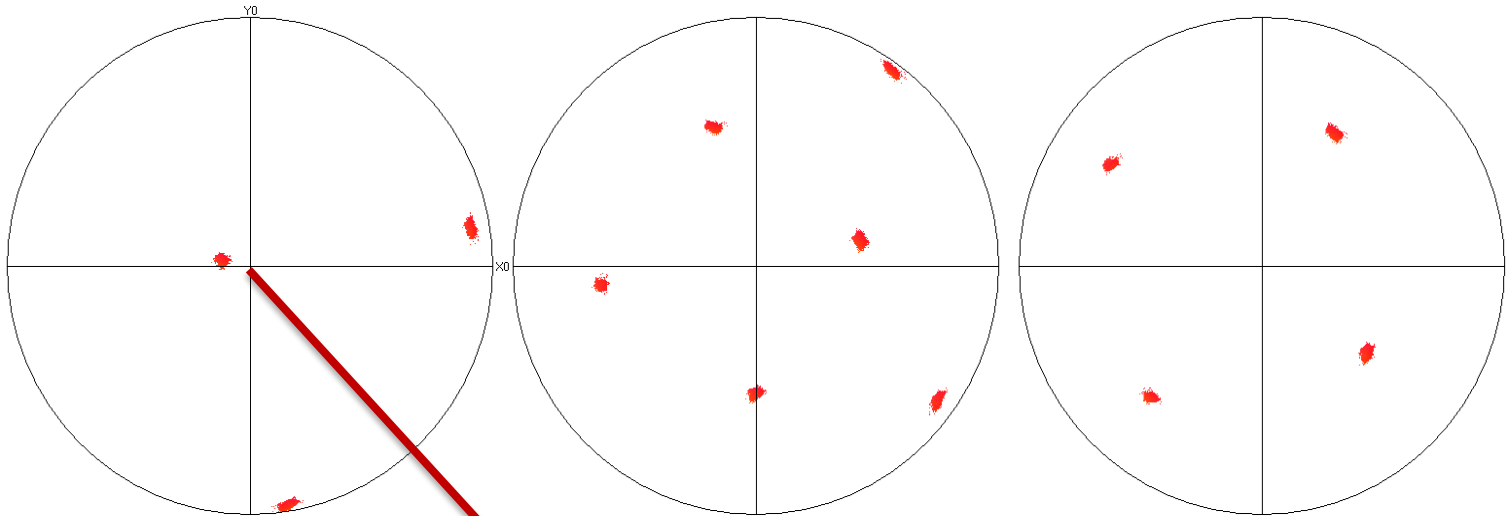


IPF color coding,  
step size 0.15  $\mu\text{m}$

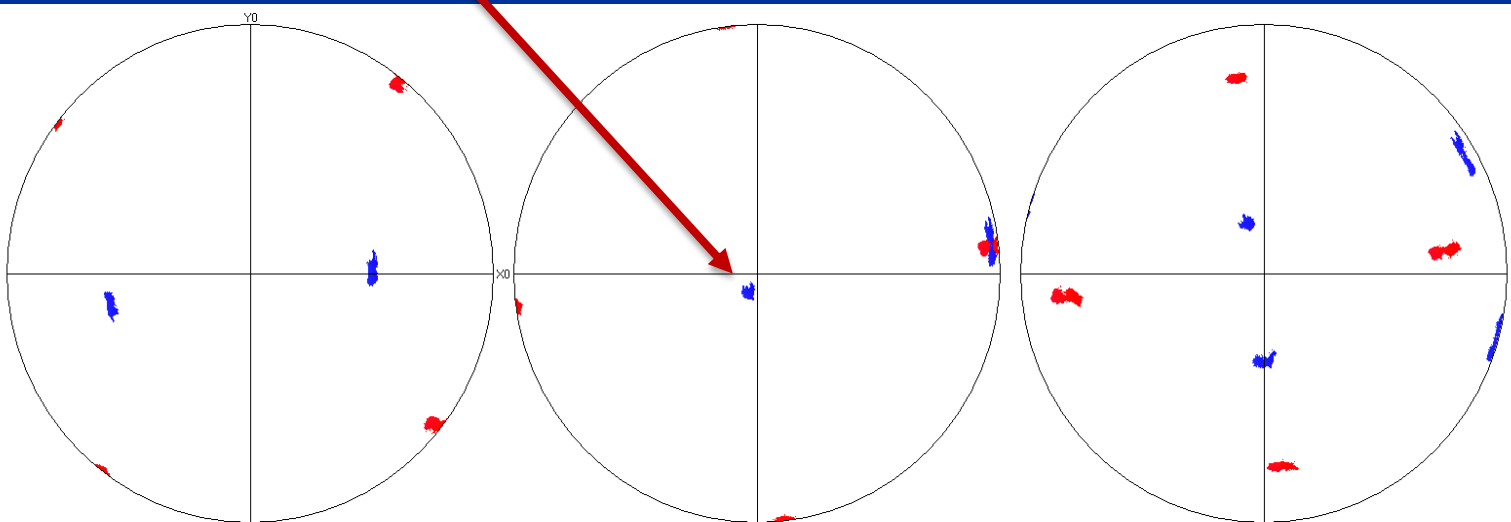
**Phase map shows only one  
phase – i.e. complete  
transformation has occurred**

# Orientation relationship

Austenite  
(105°C)



Martensite  
(25°C)



PF

{100}

{110}

{111}

In-situ studies of martensitic phase transformation allow to capture details of the orientation relationship between austenite and martensite

# $\text{Ni}_{50}\text{Mn}_{29}\text{Ga}_{21}$ modulated 5M structure

( $A_s \approx 74^\circ\text{C}$ ,  $A_f \approx 81^\circ\text{C}$ )

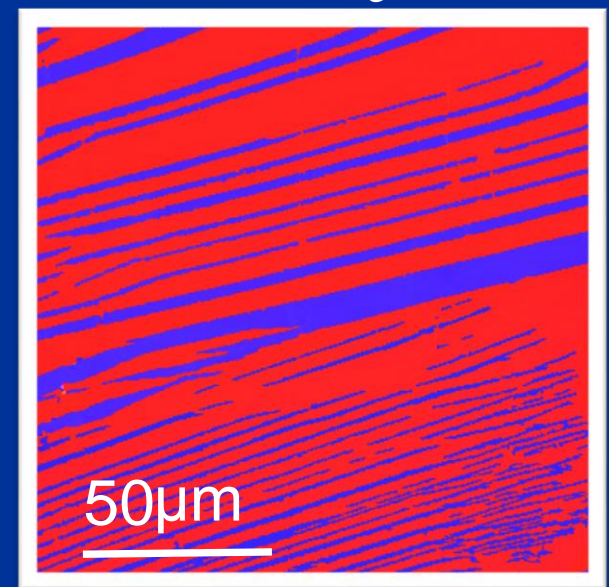
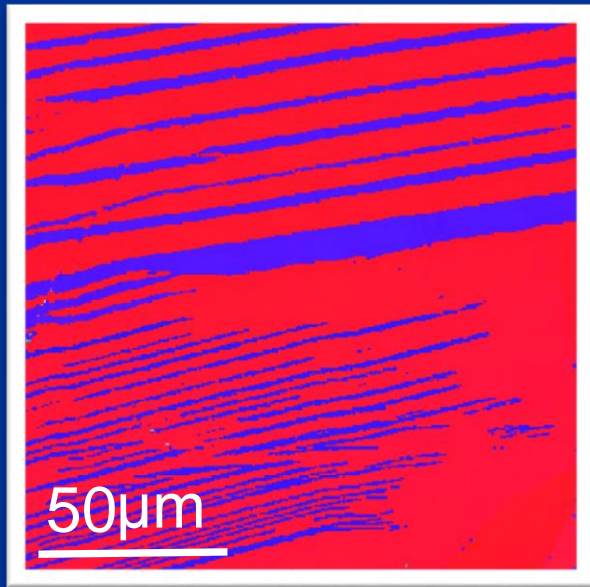
## Thermal cycling!

25°C

90°C

25°C

after cooling



IPF color coding, step size  
0.4  $\mu\text{m}$

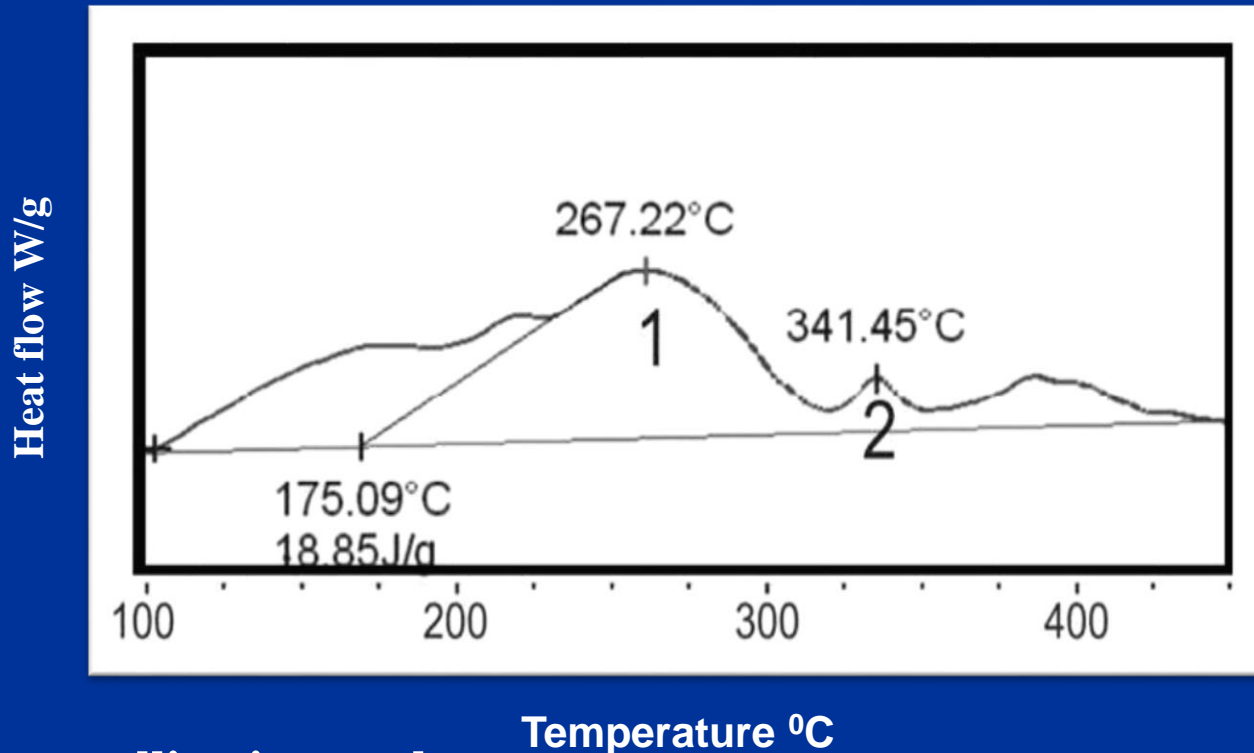
### Reverse transition

Distribution of martensitic  
laths (plates) after cooling  
is not the same as before  
phase transformation



# Aluminum alloy 6013 after cold rolling

## Calorimetric studies

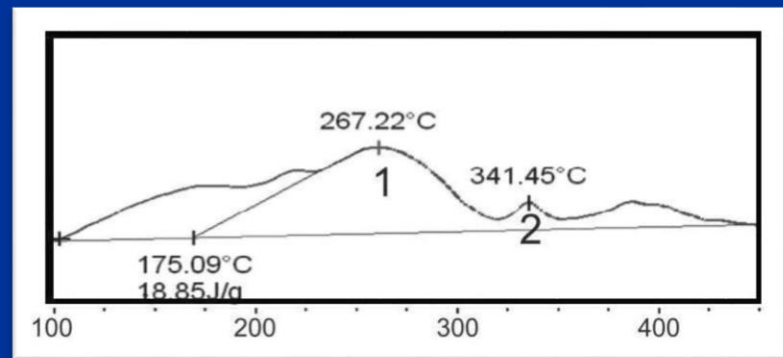
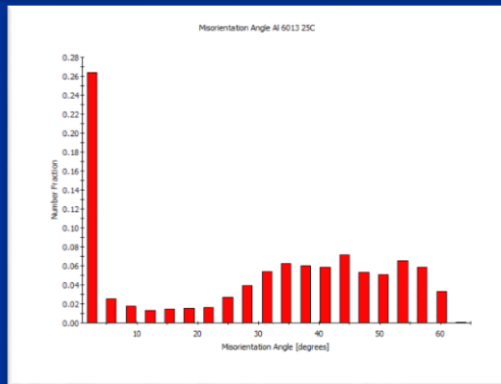
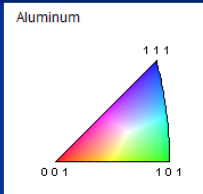
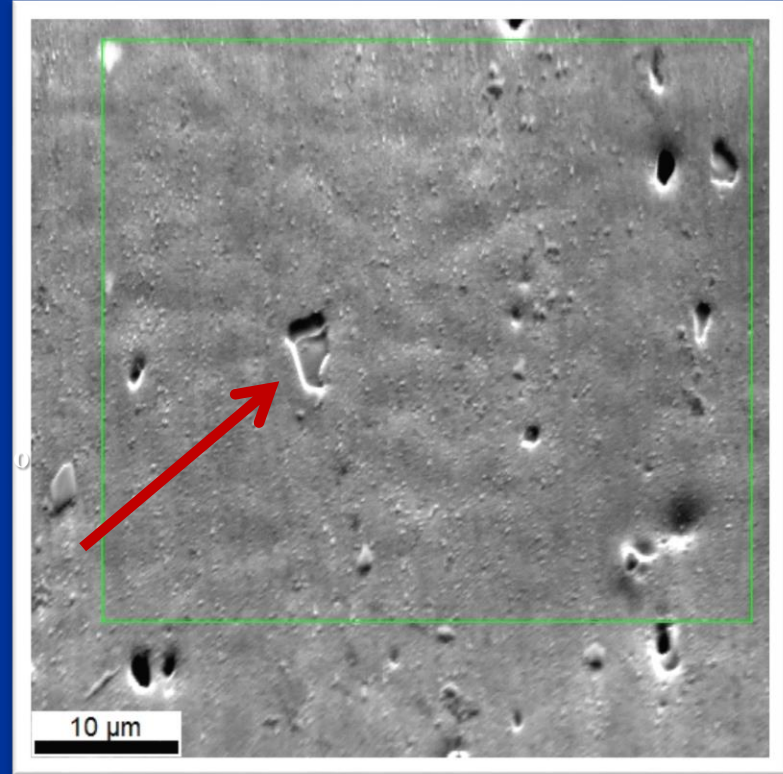
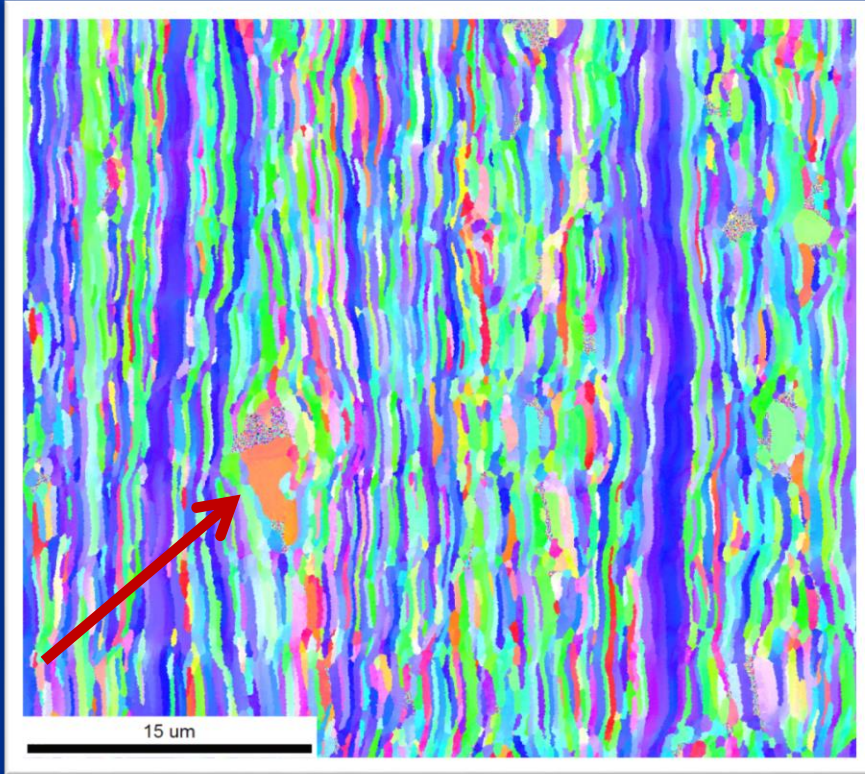
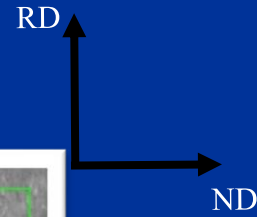


### Recrystallization peaks:

- 1 first recrystallization takes place in highly deformed zones in the vicinity of **large particles** (particle stimulated nucleation PSD)
- 2 proper recrystallization is overlapped by dissolution of **small ( $\ll 1\mu\text{m}$ ) second-phase particles**.

Recrystallization is fully completed after reaching 380°C.

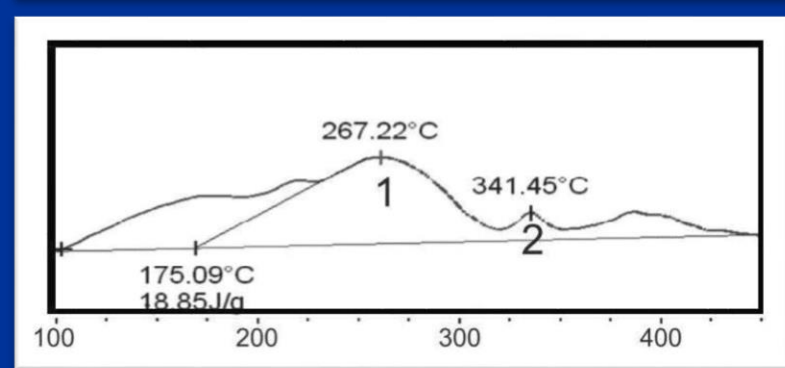
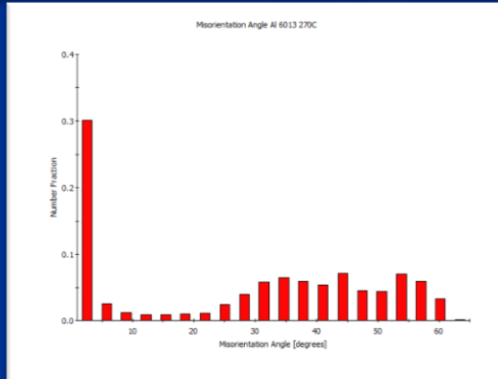
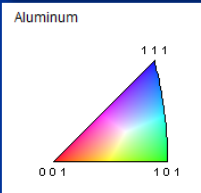
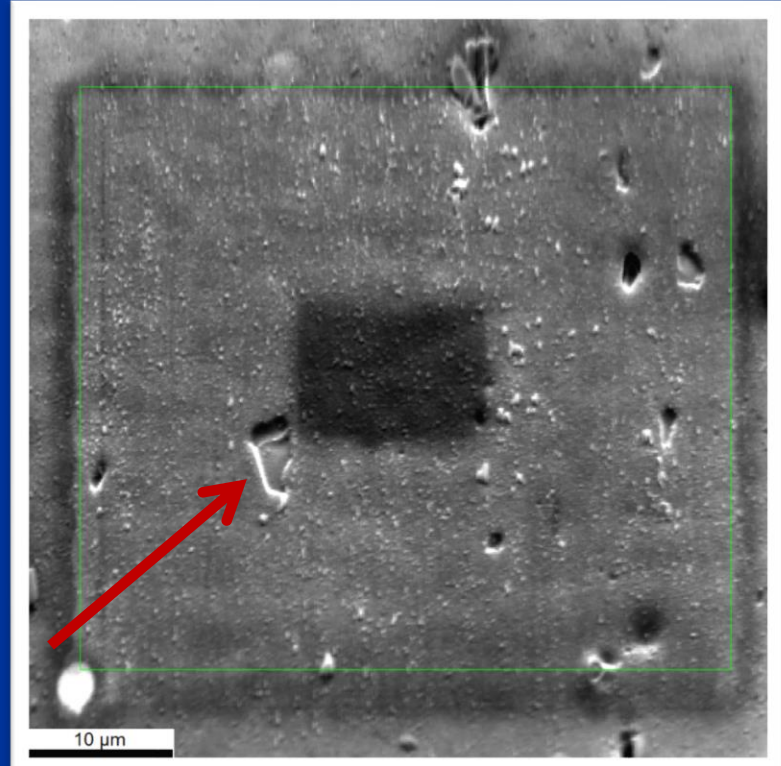
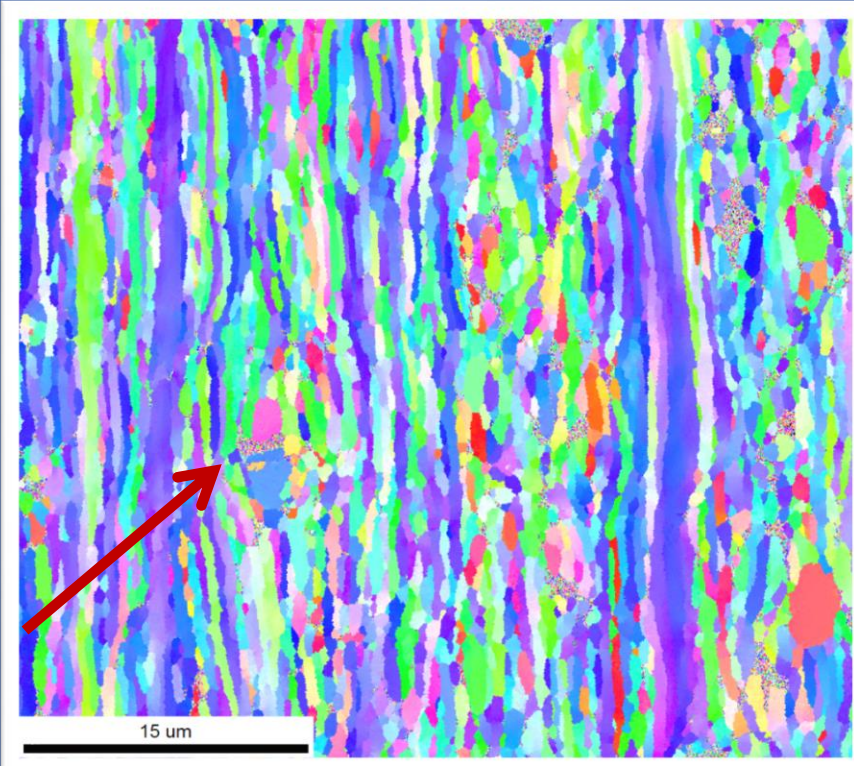
# Aluminum alloy 6013 SEM 25°C



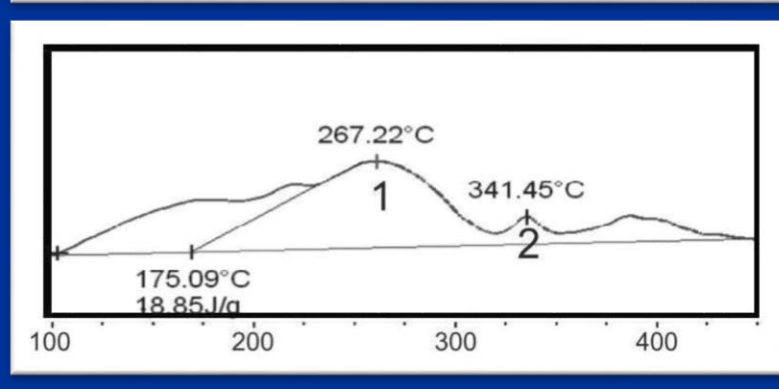
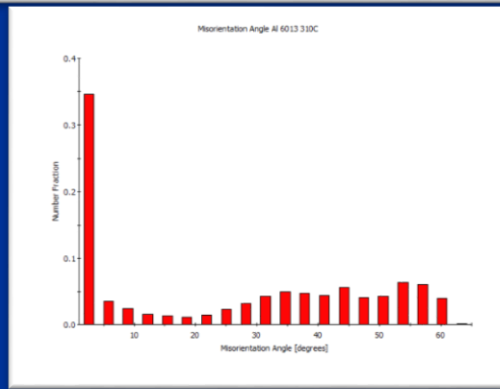
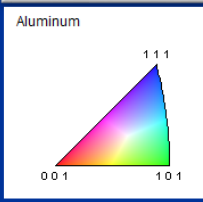
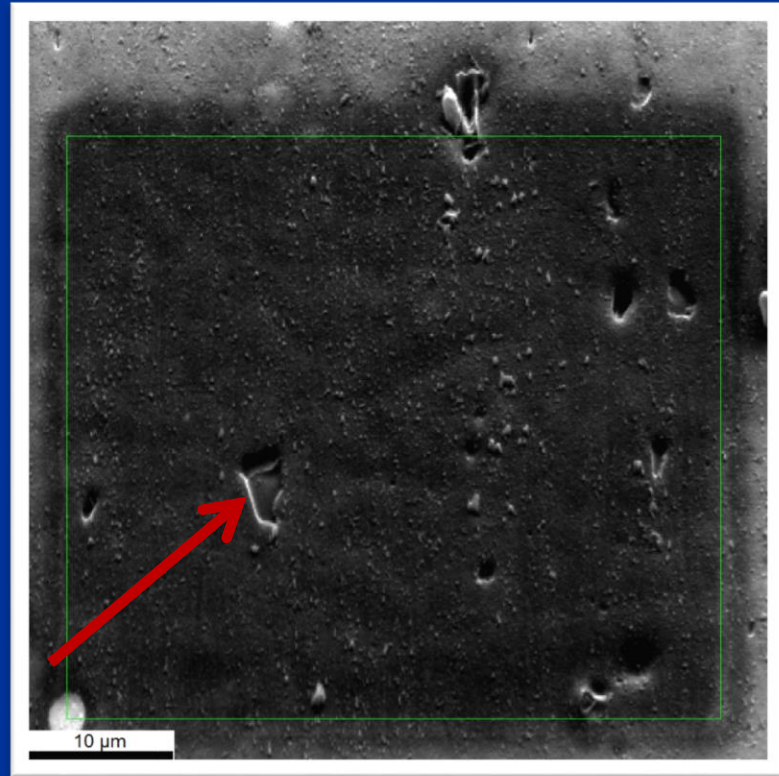
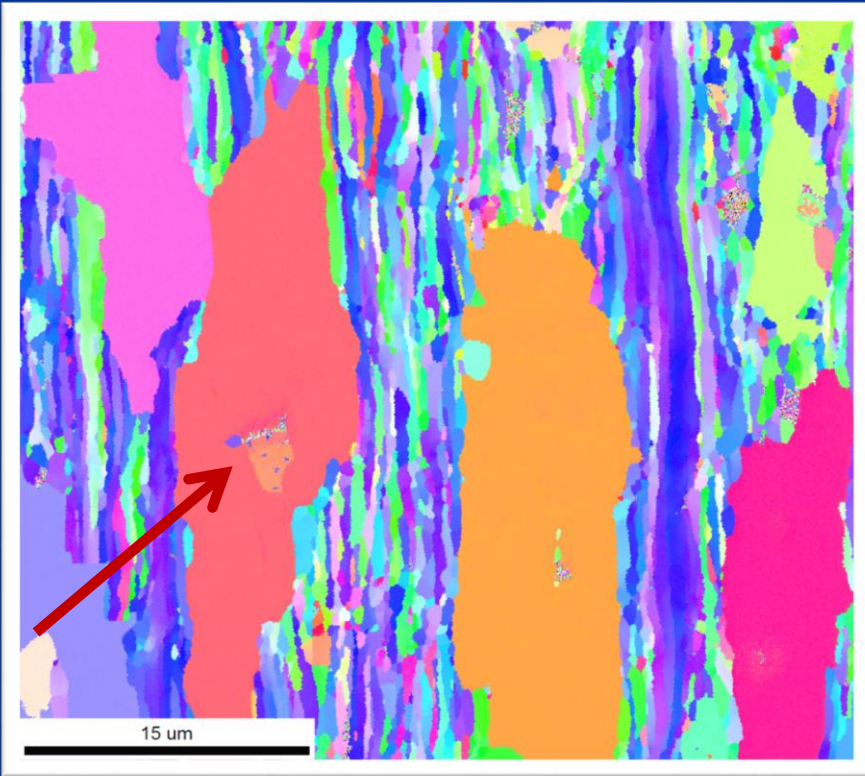
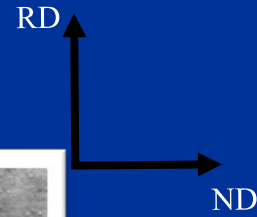
# Aluminum alloy 6013 SEM 270°C

RD

ND



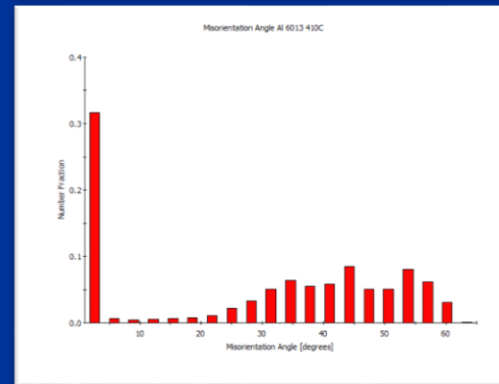
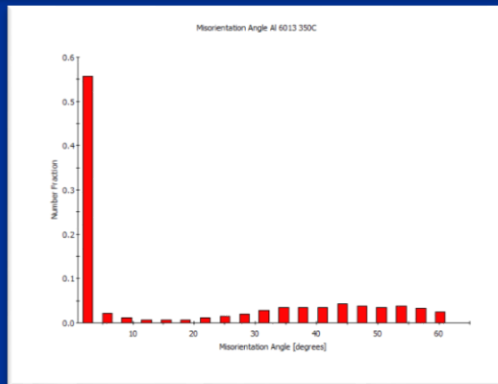
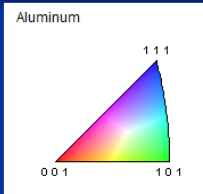
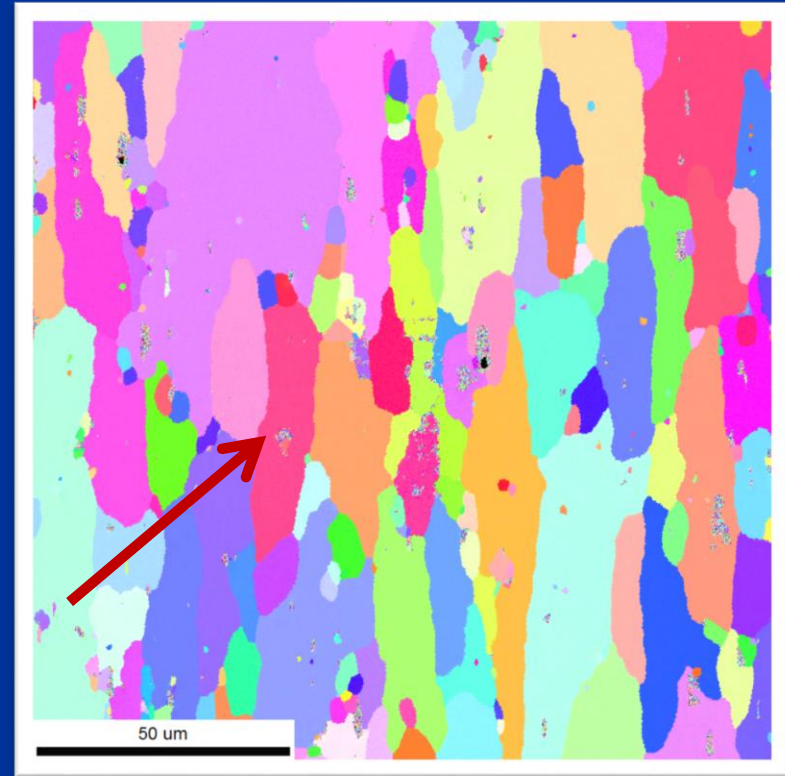
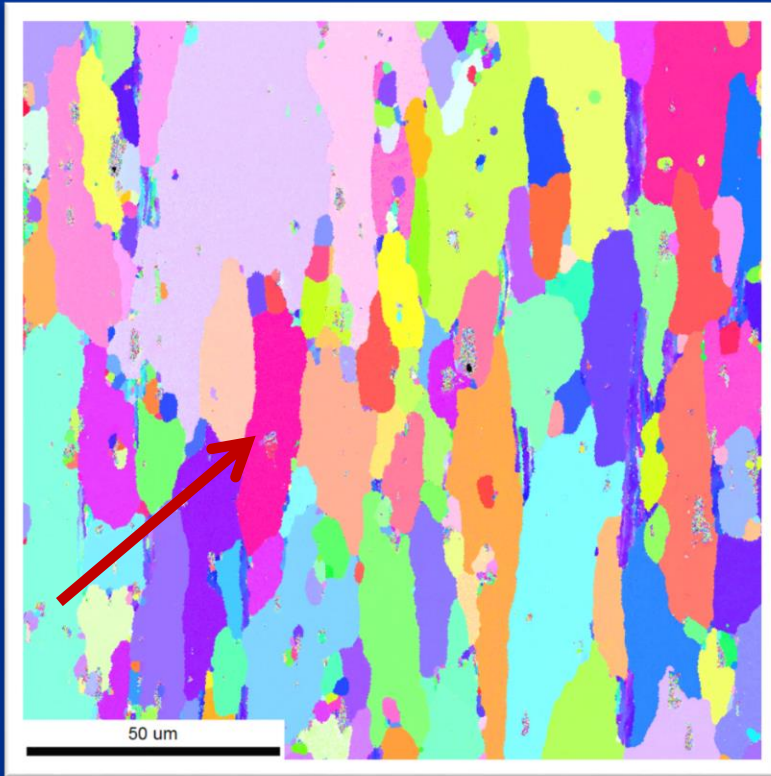
# Aluminum alloy 6013 SEM 310°C



# Aluminum alloy 6013 SEM 350°C & 410°C

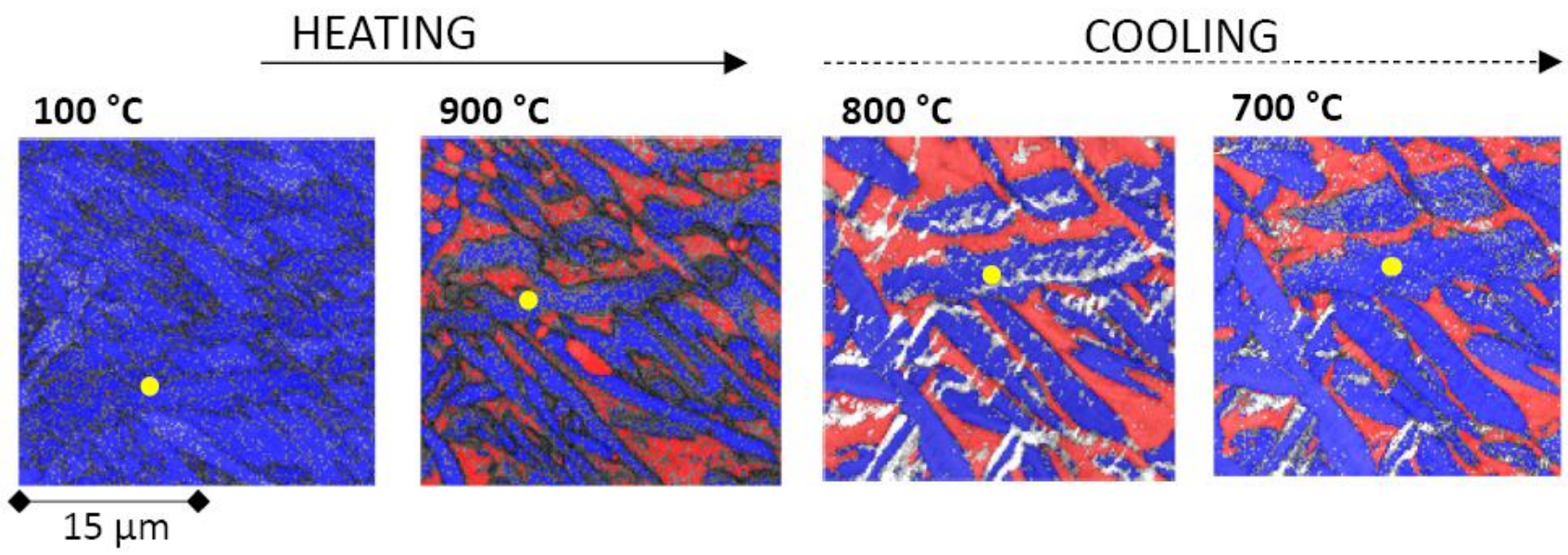
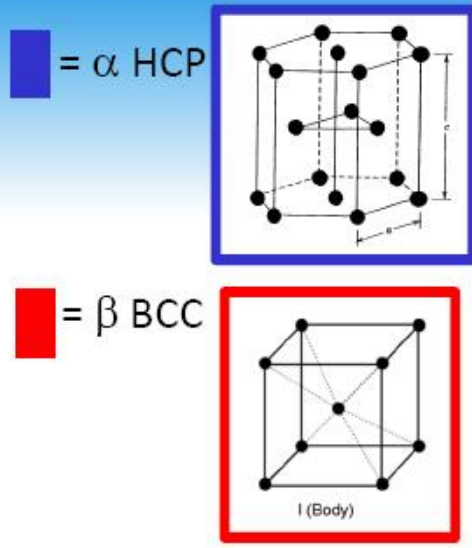
RD

ND



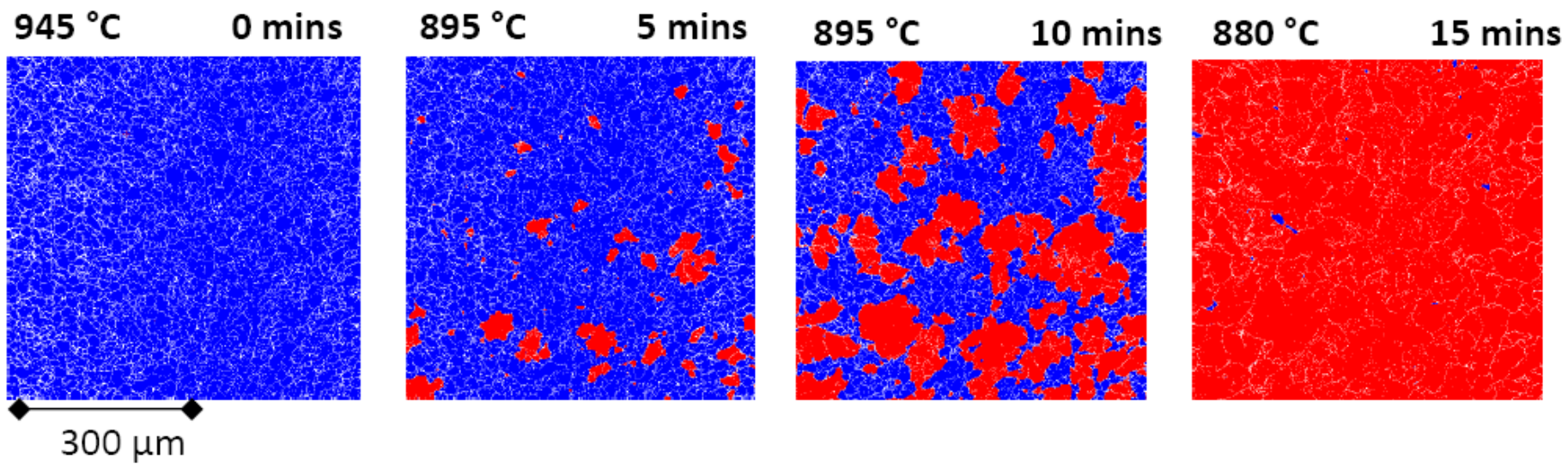
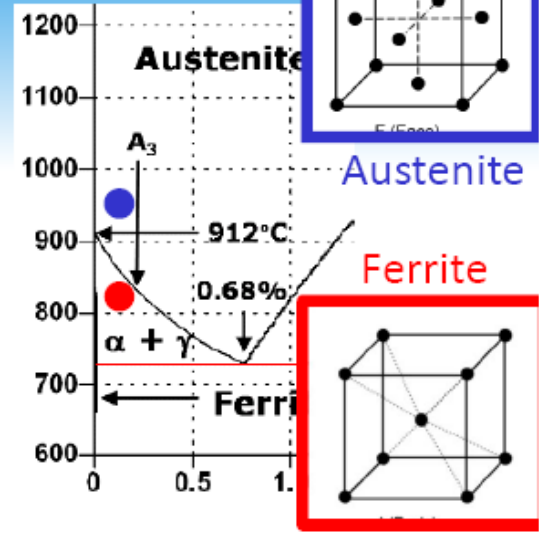
# In-situ phase transformation $\alpha \rightarrow \beta$ in Titanium

- ❖ **Specimen:** 5 mm x 5 mm x 1 mm pure electropolished Ti.
- ❖ At 100 °C Ti exists in the HCP phase .
- ❖ Close to 880°C, Ti undergoes an allotropic transformation to BCC.
- ❖ Upon cooling, Ti structure gradually reverts back to HCP (equiaxed / Widmanstatten structure).
- ❖ Yellow marker shows minimal drift at elevated temperatures



# In-situ phase transformation $\gamma \rightarrow \alpha$ in low carbon steel

- ❖ **Specimen:** 5 mm x 5 mm x 1 mm electropolished steel.
- ❖ Heating to 945 °C : transformation to austenite.
- ❖ Controlled cooling to 880 °C: transformation to ferrite
- ❖ Microstructure and mechanical properties  $\rightarrow$  grain size.
- ❖ Murano allows steel heat treatment cycles to be optimised in real time, in-situ.

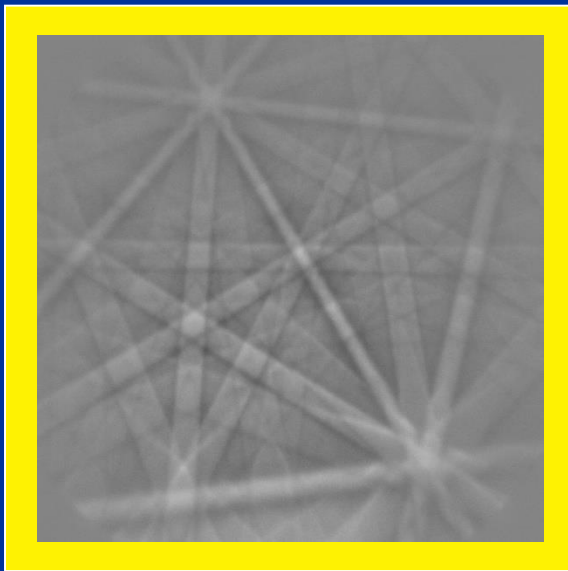


$\gamma$  Austenite FCC *Murano used for a controlled cooling rate.*  $\alpha$  Ferrite BCC

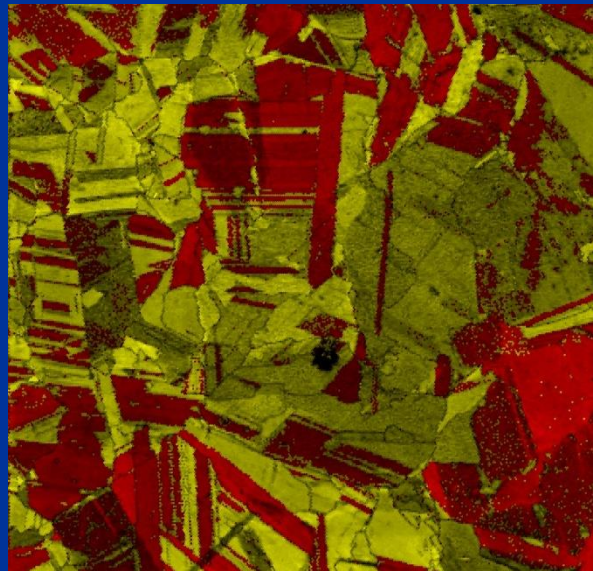


# EBSD - Analiza przemian fazowych

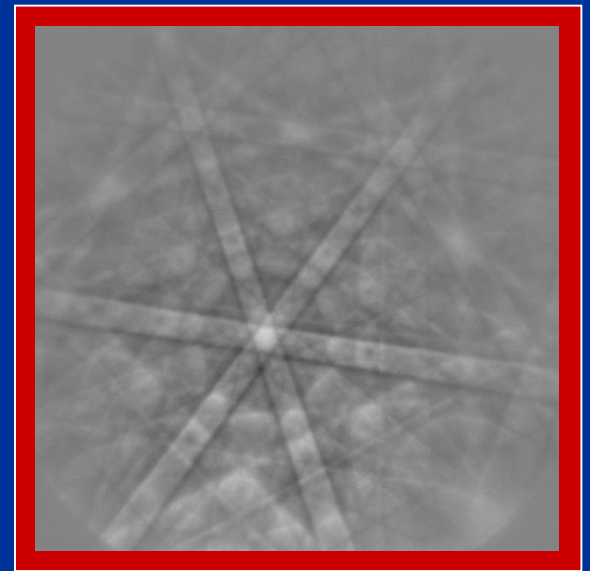
Kobalt  $\beta$ : faza  
wysokotemperaturowa  
sieć FCC



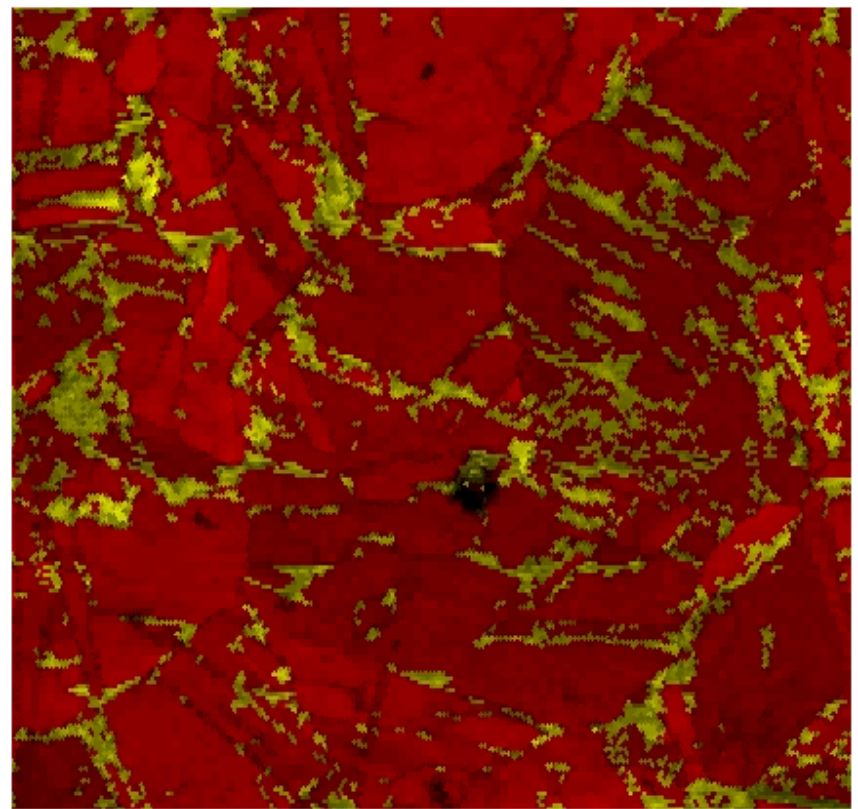
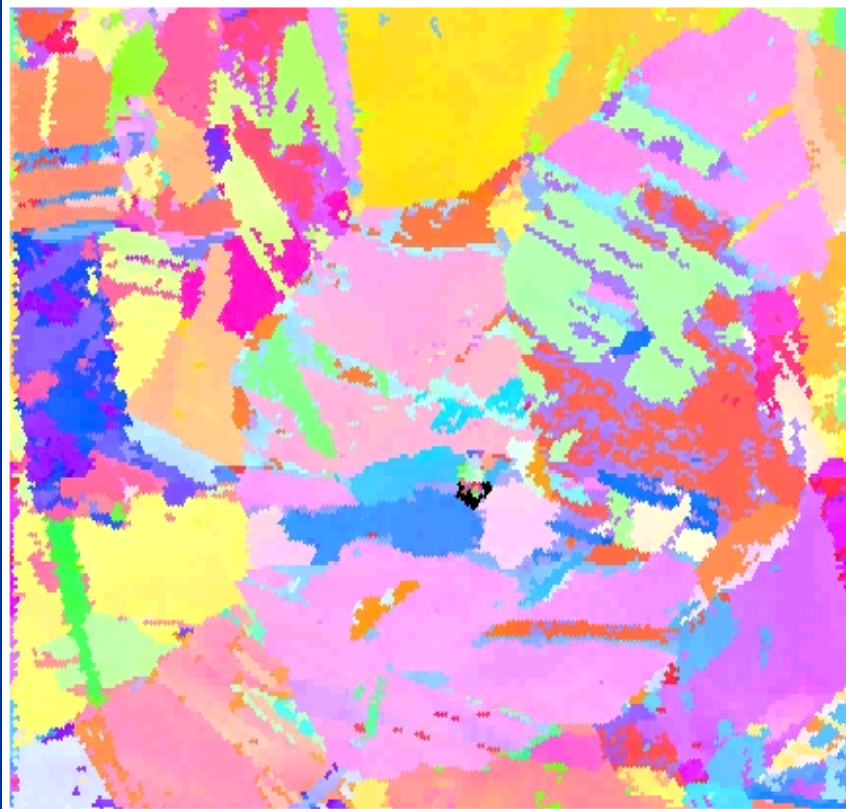
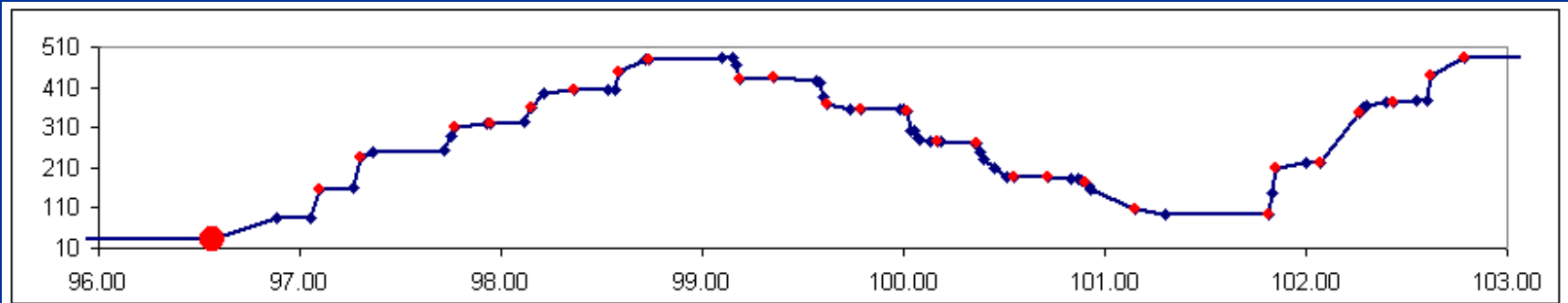
Temperatura przemiany  
alotropowej 417°C

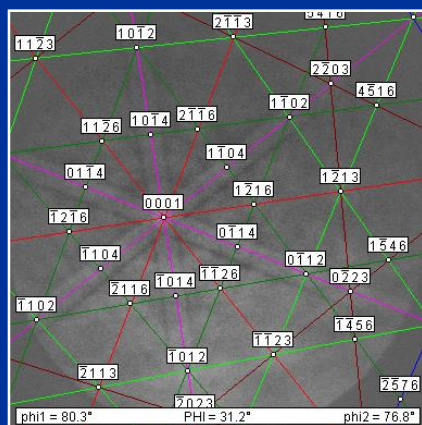
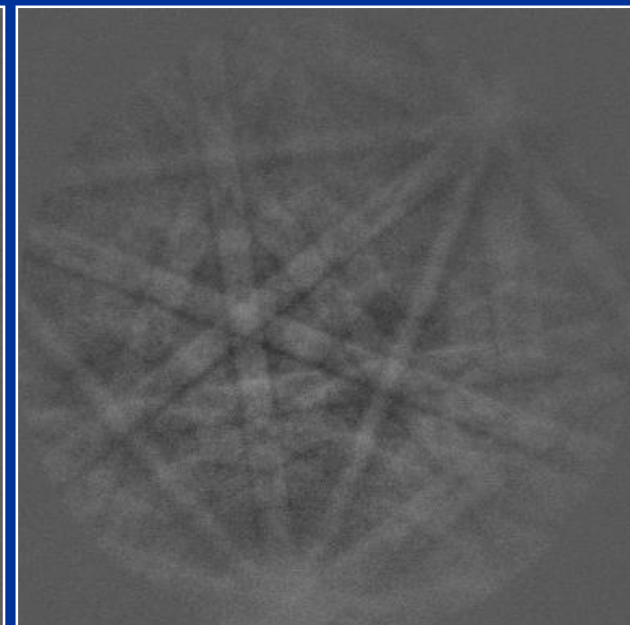
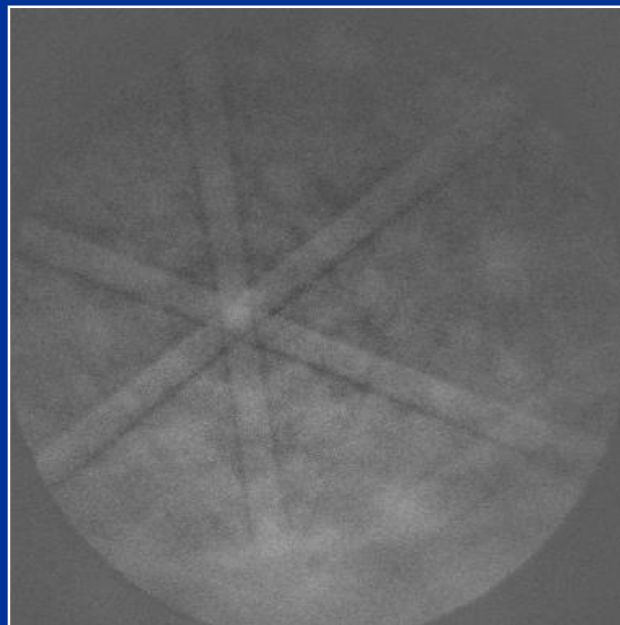
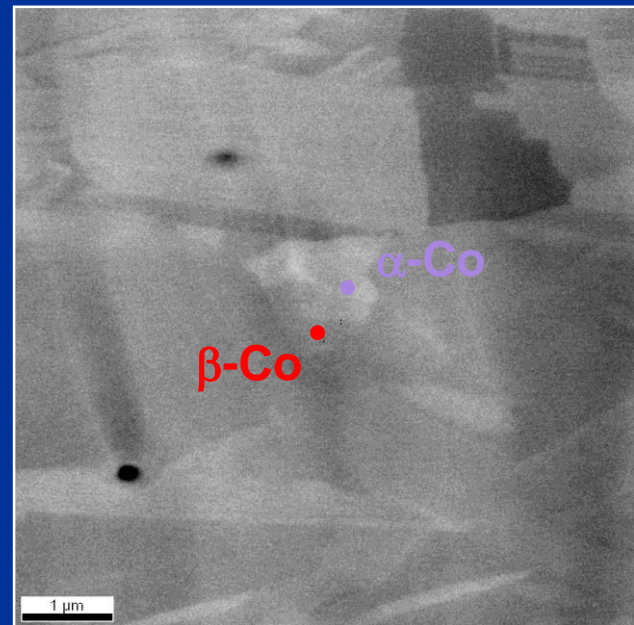


Kobalt  $\alpha$  sieć HCP

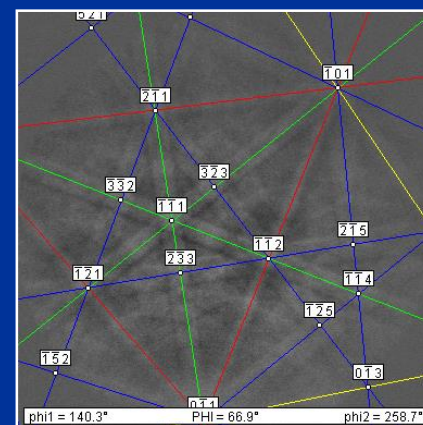








$\alpha$ -Co  
(HPC)

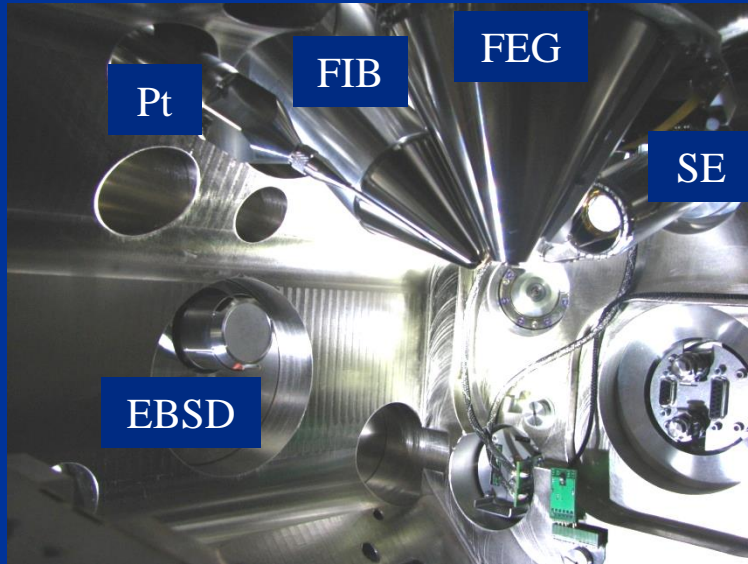


$\beta$ -Co  
(FCC)

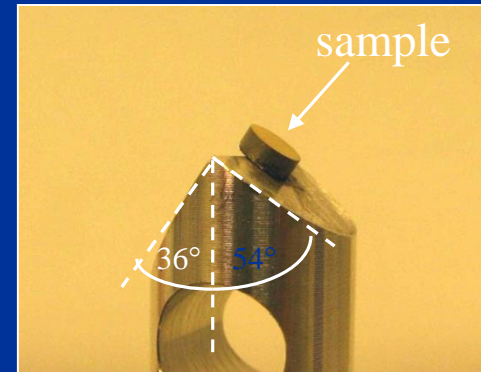
Równoległość osi [0001] i [111] faz  $\alpha$  i  $\beta$  dla Co

# *3D EBSD*

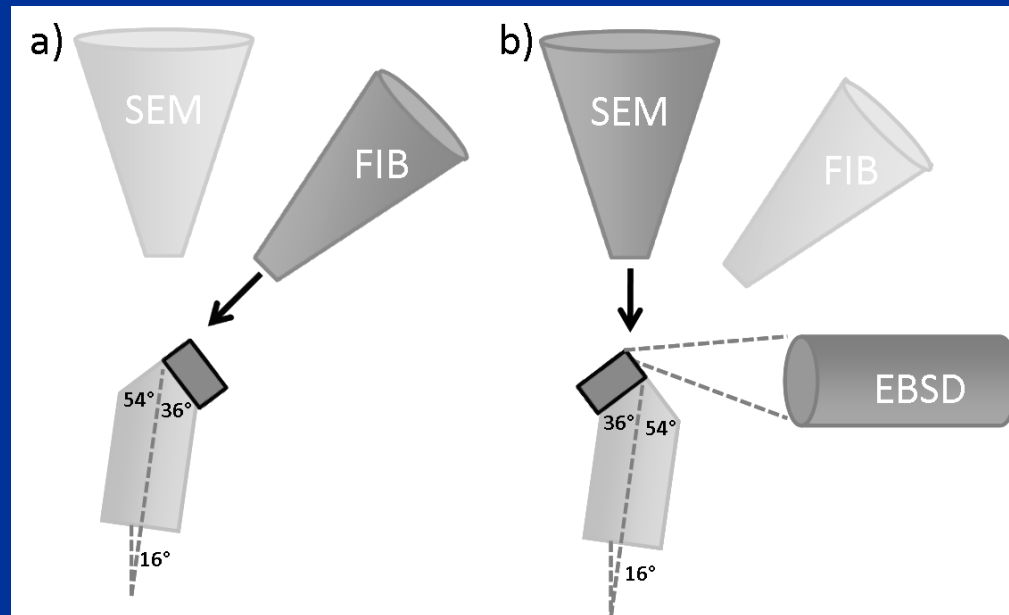
# 3D-EBSD - Geometrical setup



microscope chamber

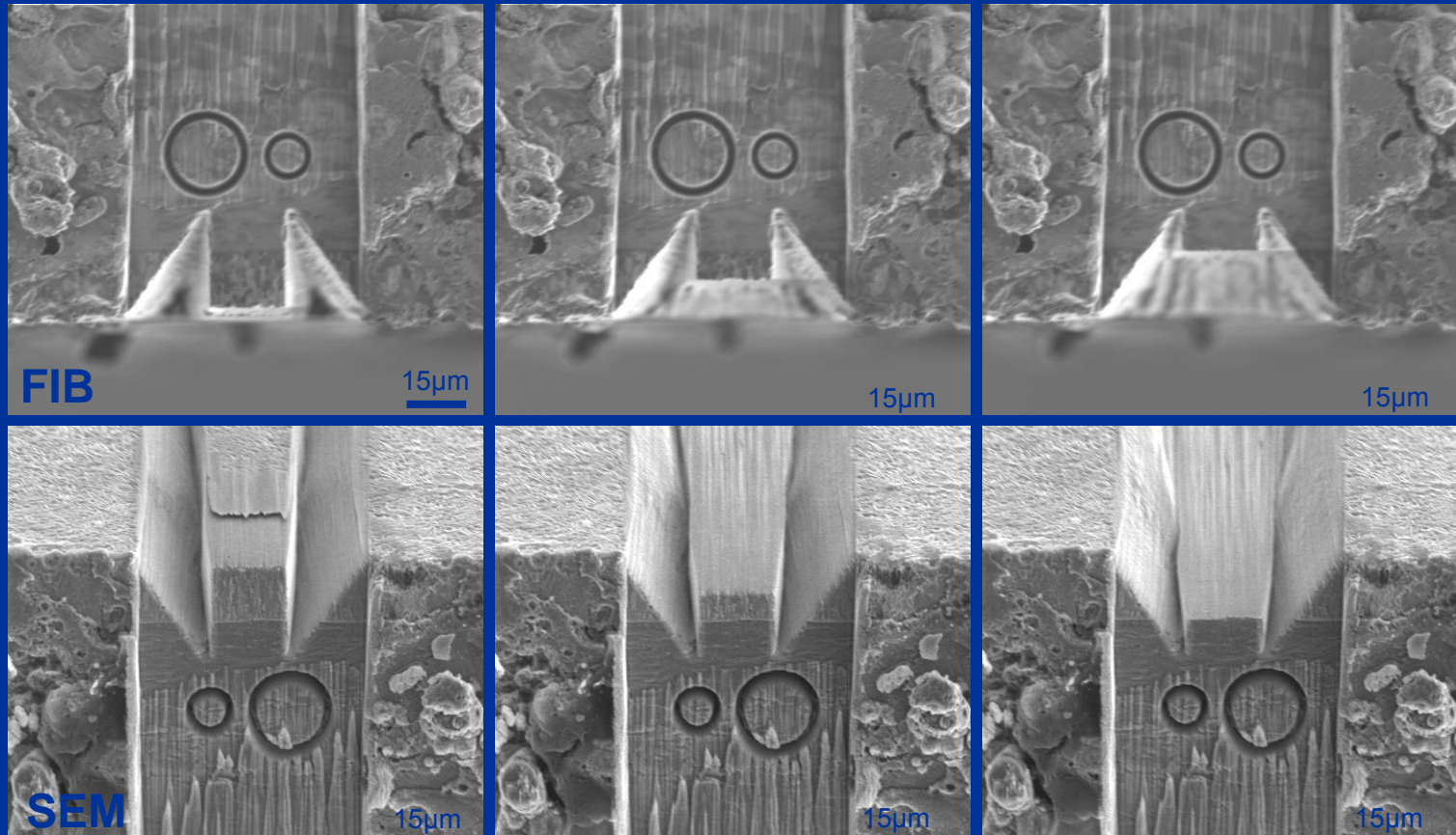


pre-tilted sample holder

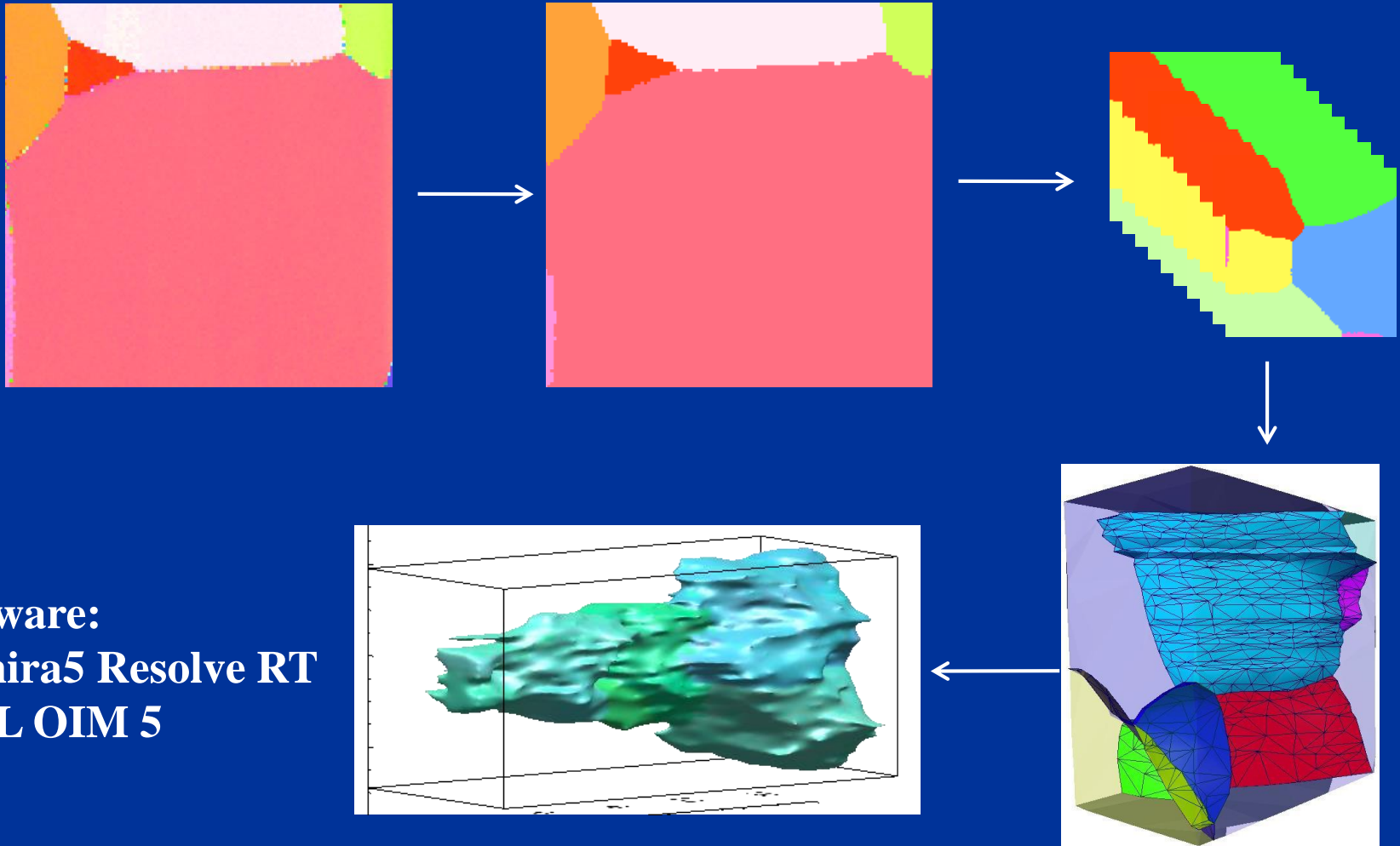


When a pre-tilted holder is used, the sample rotation around tilt axis by  $180^\circ$  is only needed.

# Ion milling



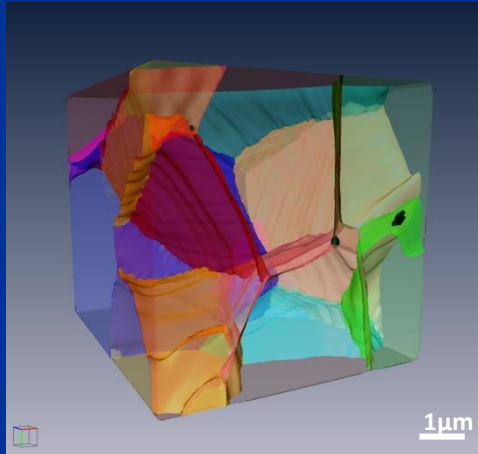
# Data processing



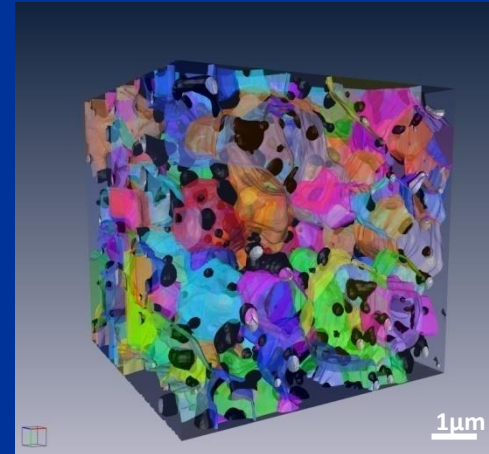
## Software:

- Amira5 Resolve RT
- TSL OIM 5

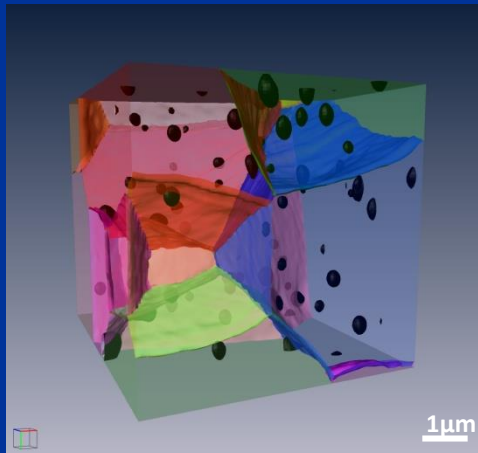
# Results: 3D reconstructions



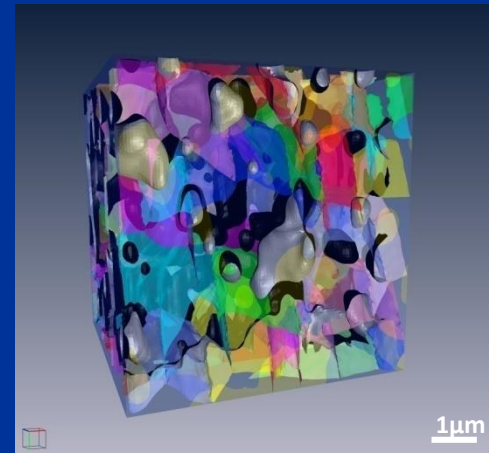
T1600



S1600



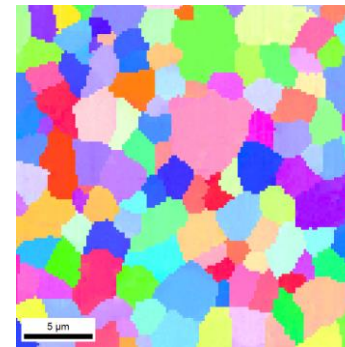
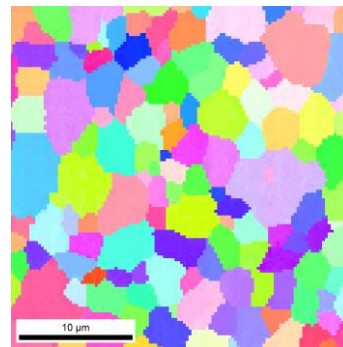
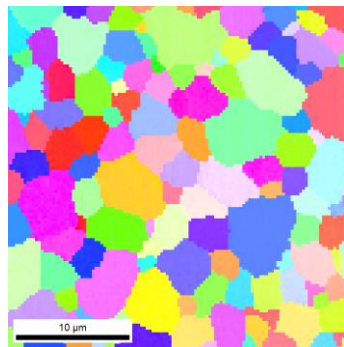
T1650



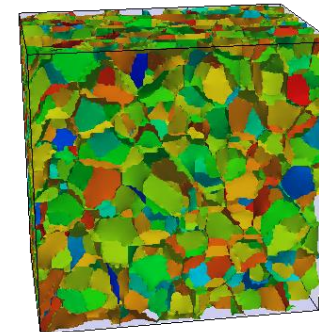
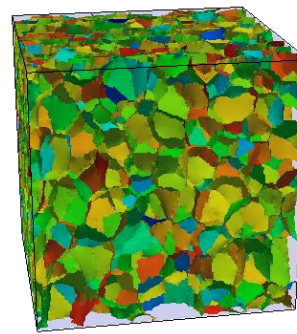
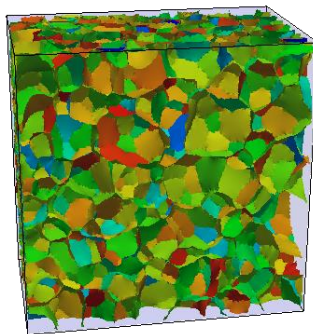
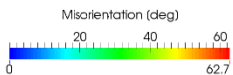
S1650

# 2D & 3D EBSD results from $\text{ZrO}_2$

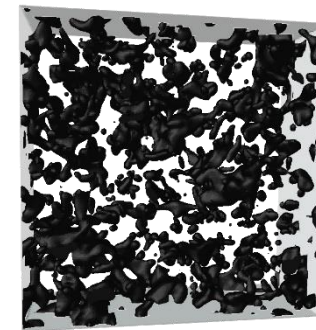
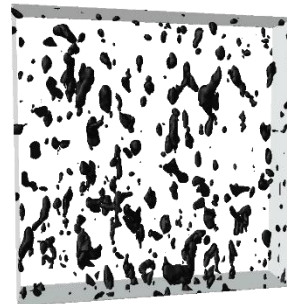
2D Inverse Pole Figure map



3D grain boundary reconstruction

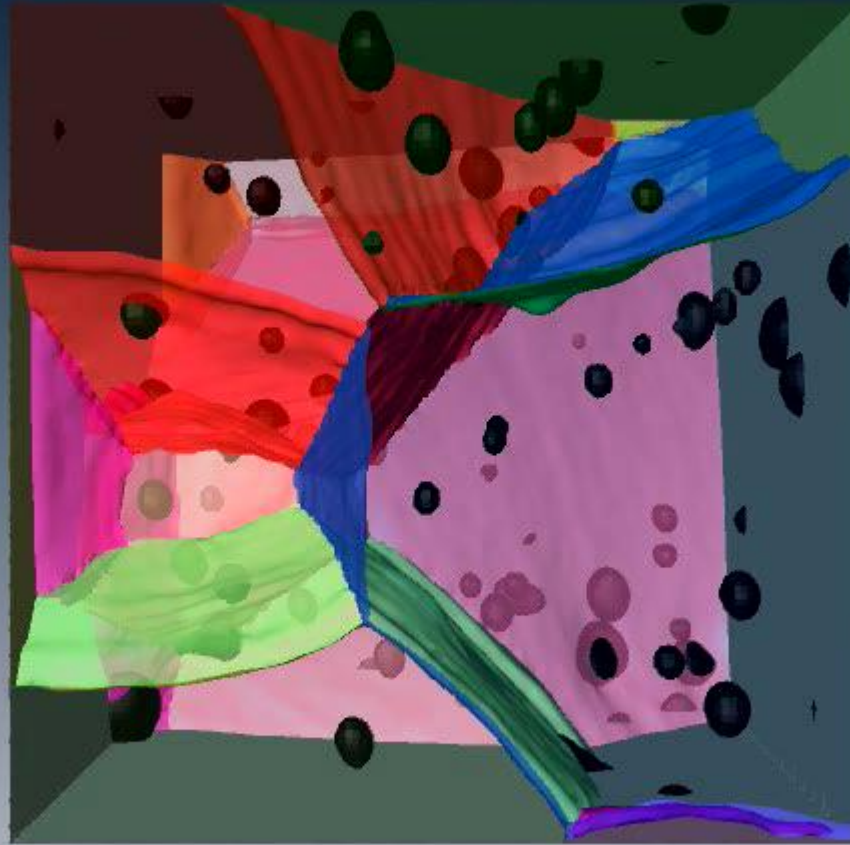


3D pore structure reconstruction

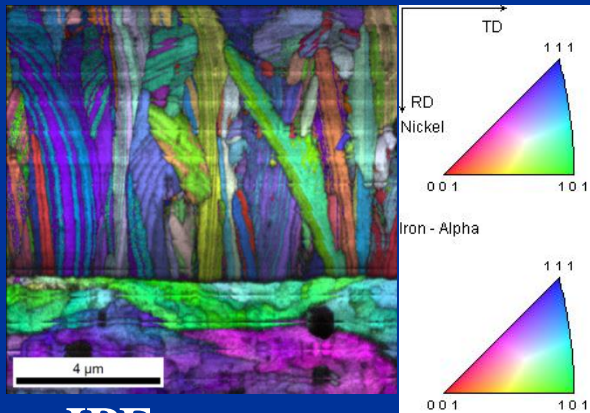




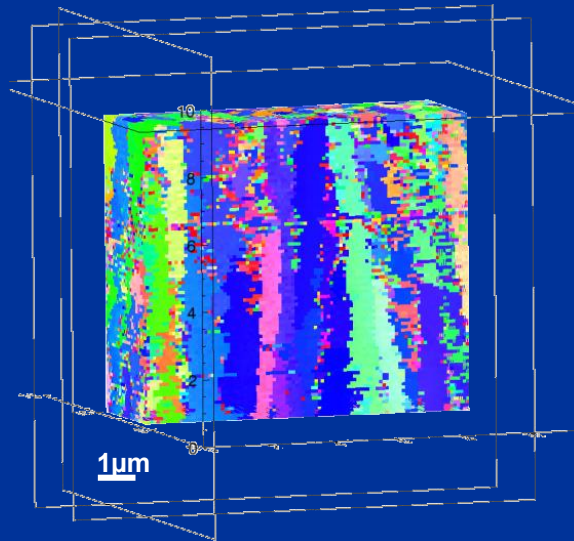
# 3D pores distribution in $\text{ZrO}_2$



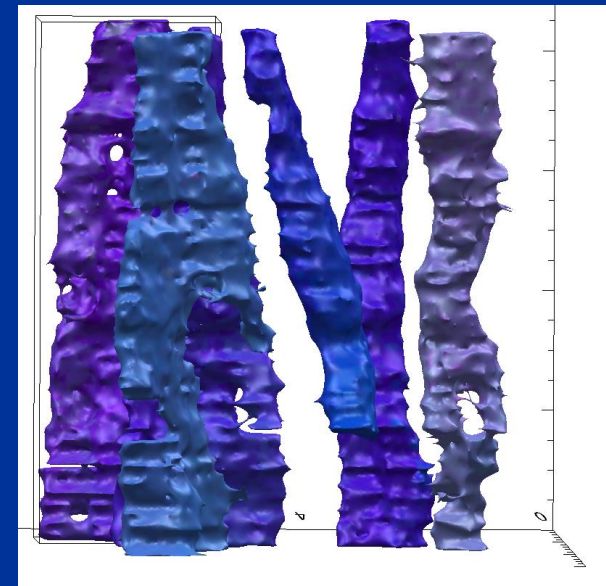
# Other applications: Ni-Mo/Fe



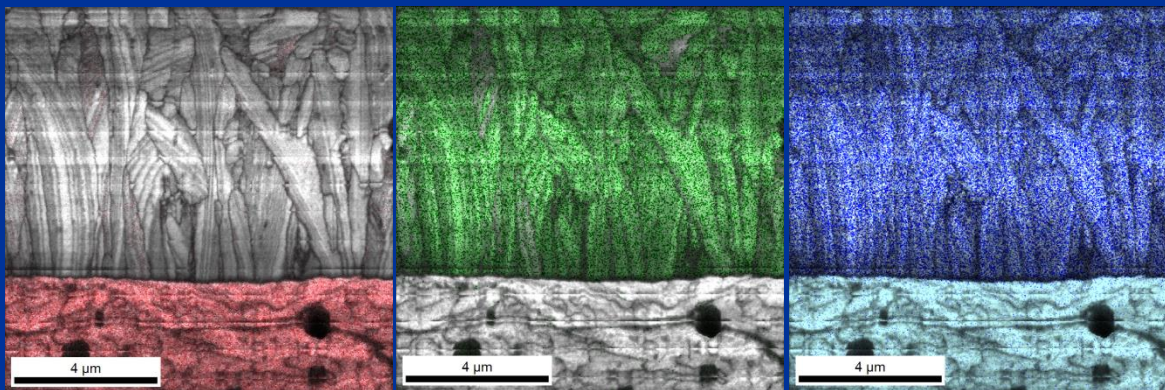
IPF map



3D IPF map

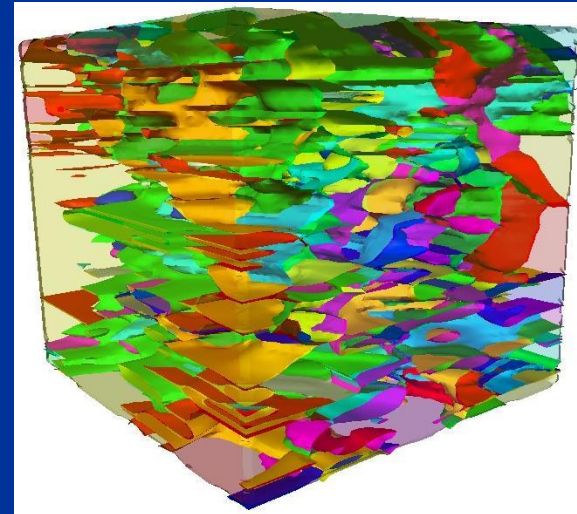
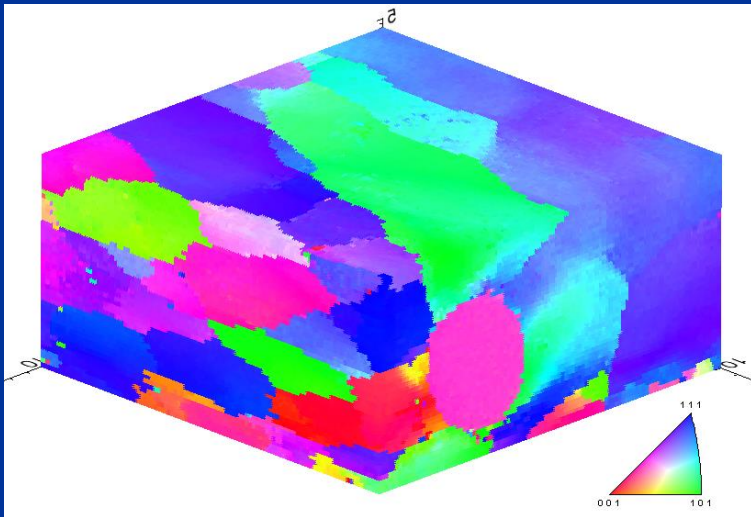


3D grain reconstruction

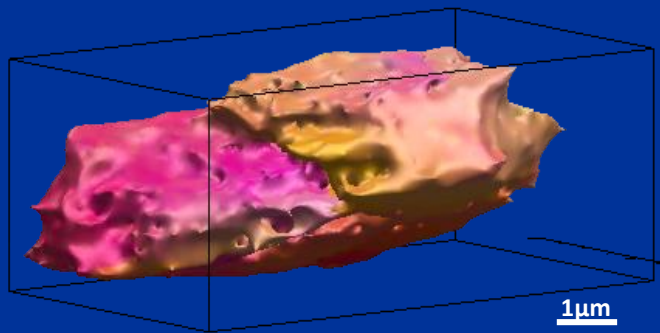


Fe Mo Ni  
Chemical composition evaluation by EDS

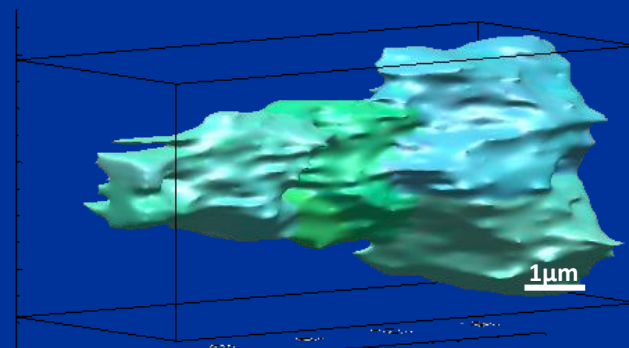
# Other applications: Al6013



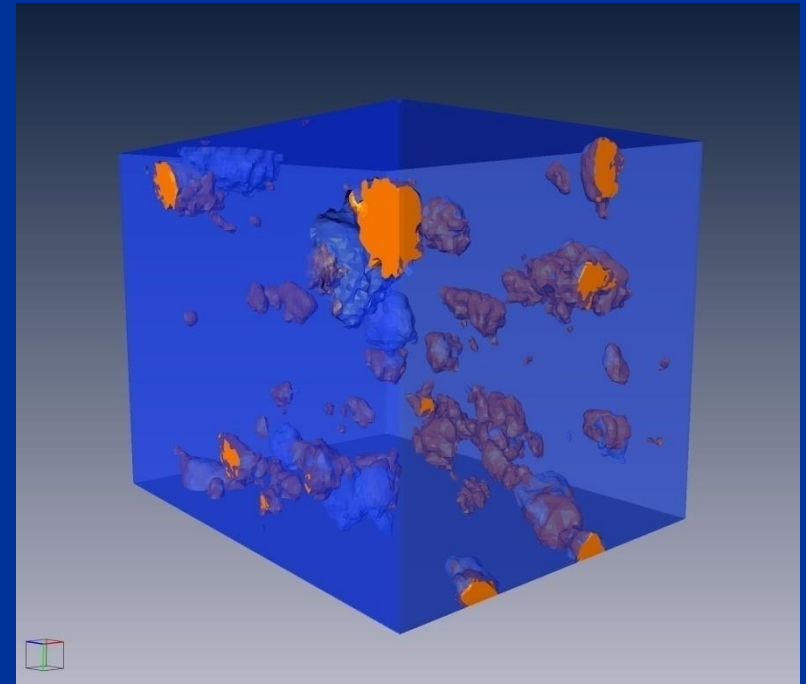
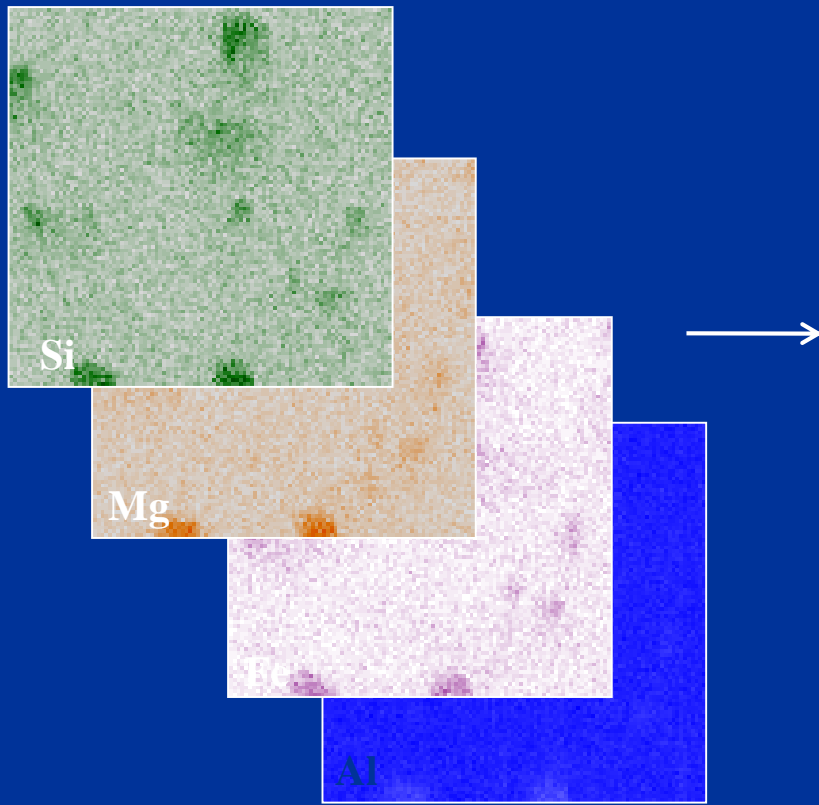
Grain boundary reconstruction  
(colors are randomly chosen)



3D grain reconstruction



# 3D EDS Al6013



EDS maps of elements distribution

3D-EDS reconstruction

# 3D-EBSD technique – pros and cons

## Applications:

- 3D microstructure and microtexture of metals and ceramics
- microstructure analysis with the grain size of ca. 1-5  $\mu\text{m}$
- analysis of grain boundary character
- investigations of pore morphology

## Limitations:

- destructive technique!
- effects of anisotropic sputtering
- effects of amorphisation
- sample charging (in ceramics!)
- resolution: 50x50x50  $\text{nm}^3$
- region of interest: 25x25x25  $\mu\text{m}^3$
- time per slice: 5-30 min