Whose Fault is it?

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The Power of Powder Diffraction

Erice-2011, 2 - 12 June, Erice, Italy

The Effect of Faulting and Twinning on the Profiles of Diffraction Peaks

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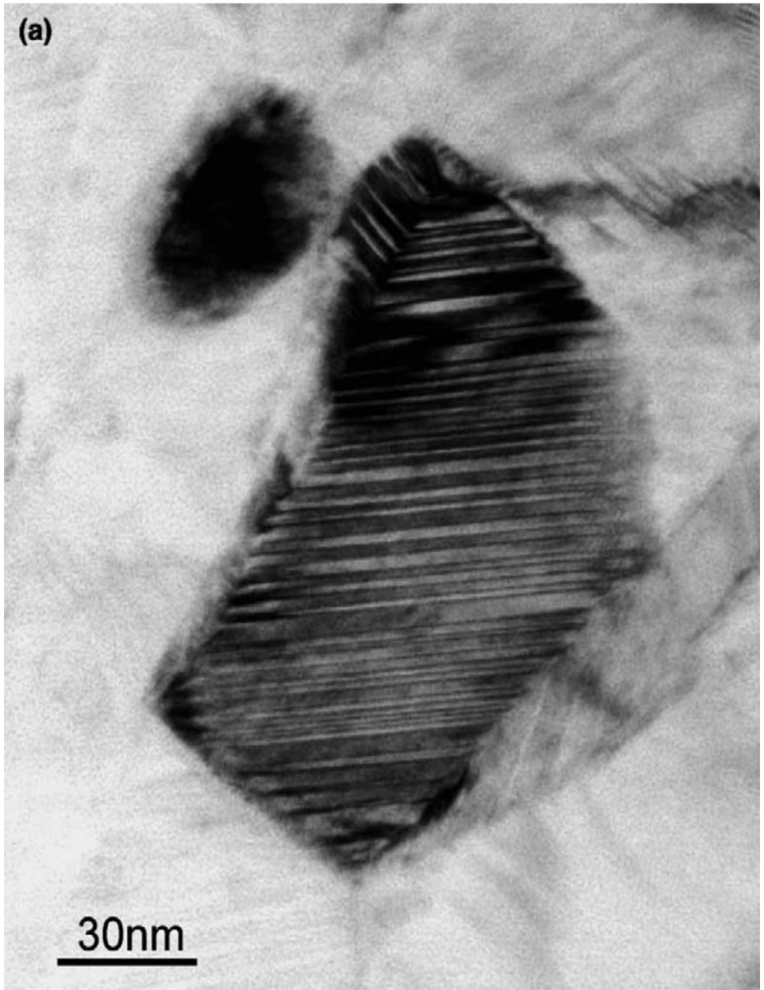
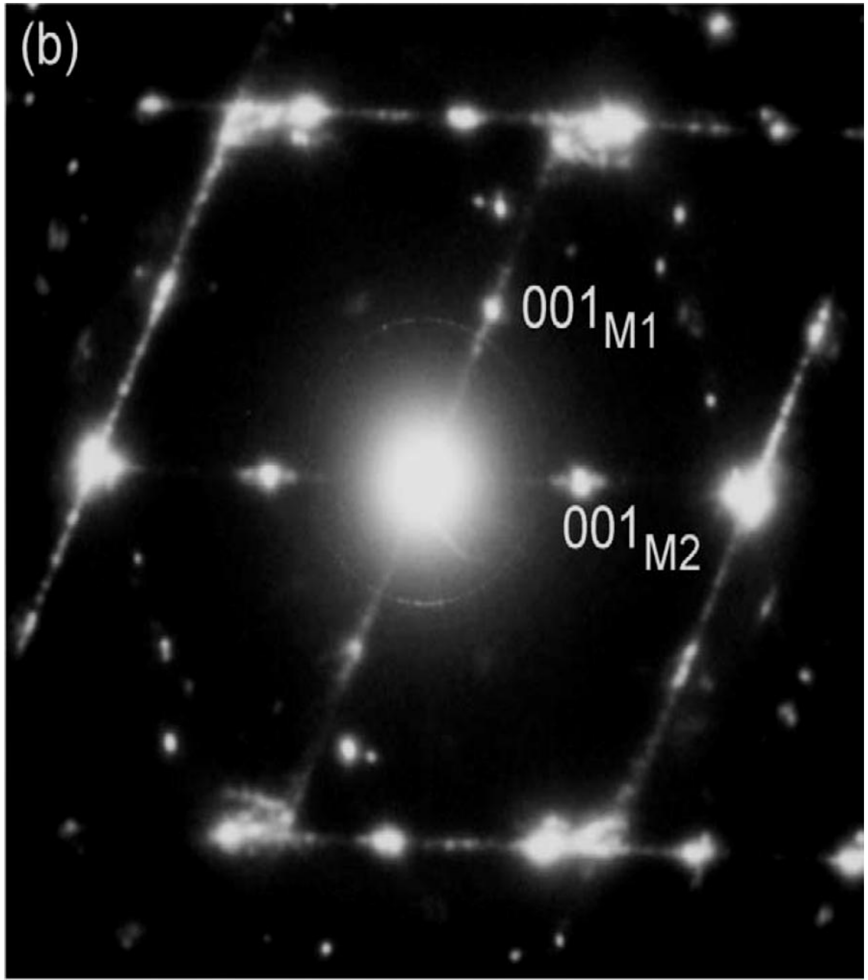
Twinning in NiTi nanograins

T. Waitz, V. Kazykhanov, H.P. Karnthaler, *Acta Materialia* 52 (2004) 137–147

Twinned nanocrystalline grains

streaking is typical for faults





T. Waitz, V. Kazykhanov, H.P. Karnthaler:

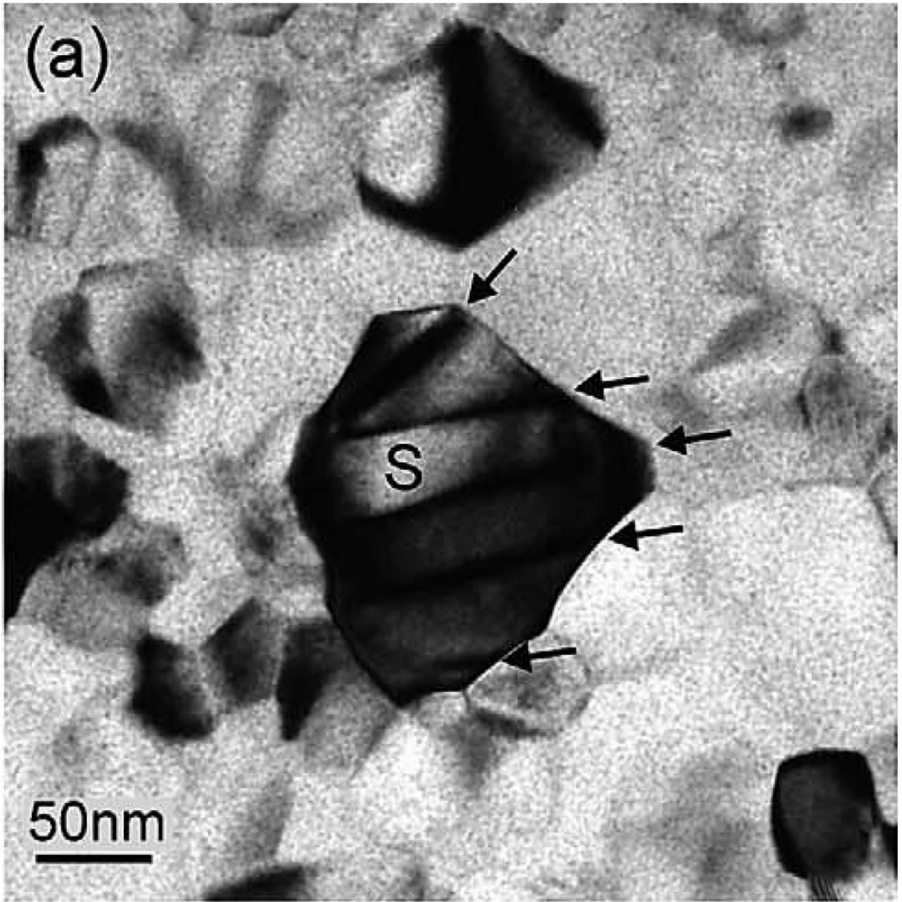
Martensitic phase transformations in nanocrystalline **NiTi** studied by TEM

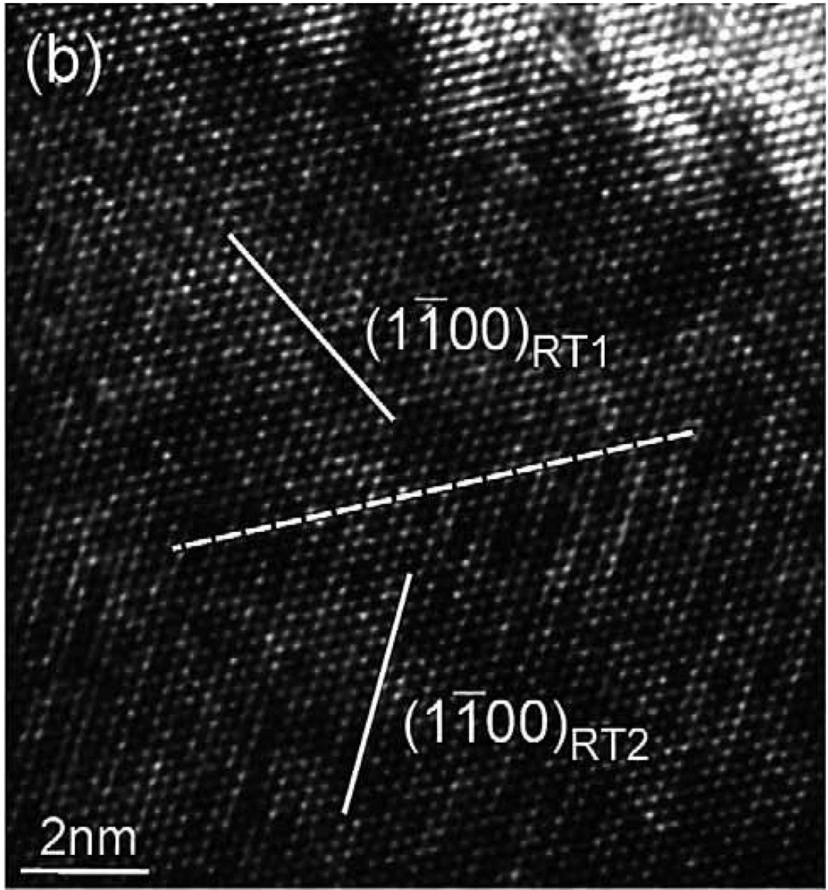
Acta Materialia 52 (2004) 137–147

Perfect matching along the twin plane

Bright field image of a **twinned grain**.

HRTEM image of a grain containing two **twin related variants**: RT1 & RT2

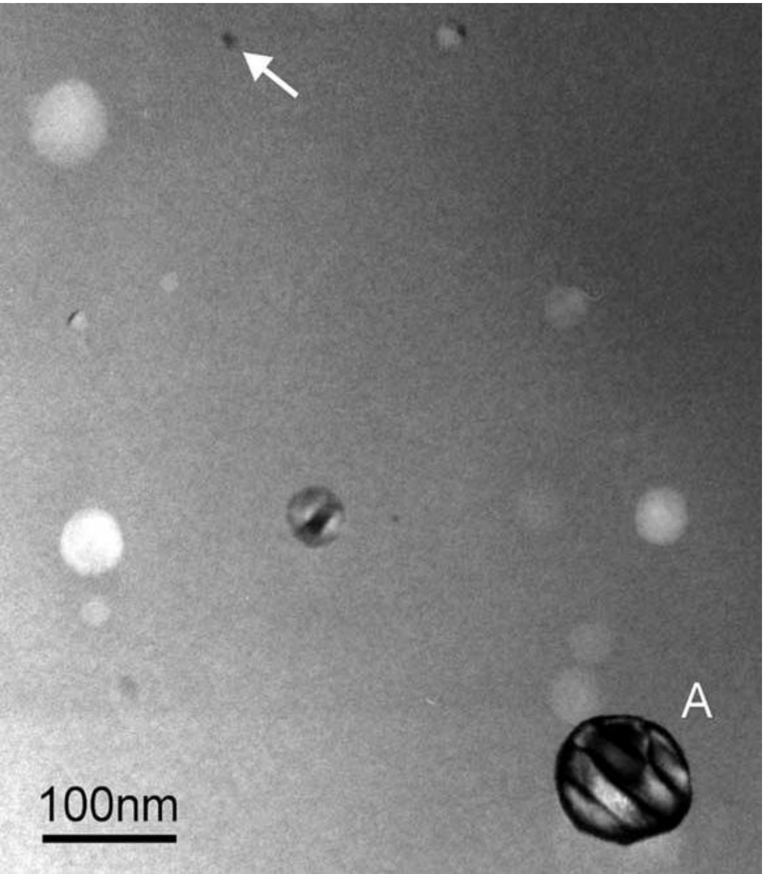




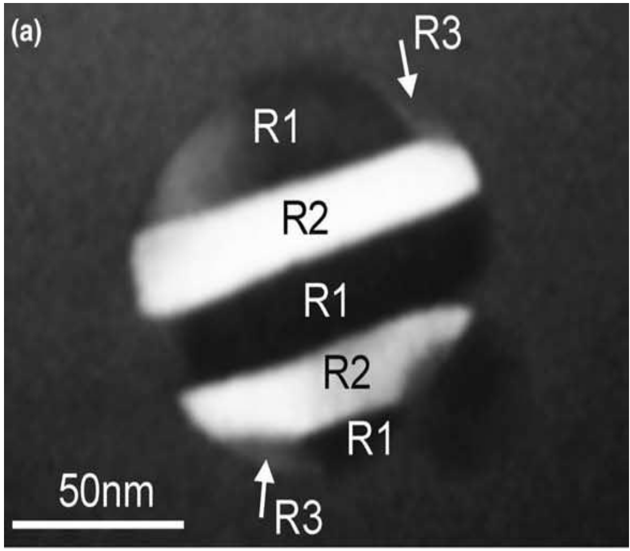
Twinning in NiTi nanograins

T. Waitz, H.P. Karnthaler, *Acta Materialia* 52 (2004) 5461–5469

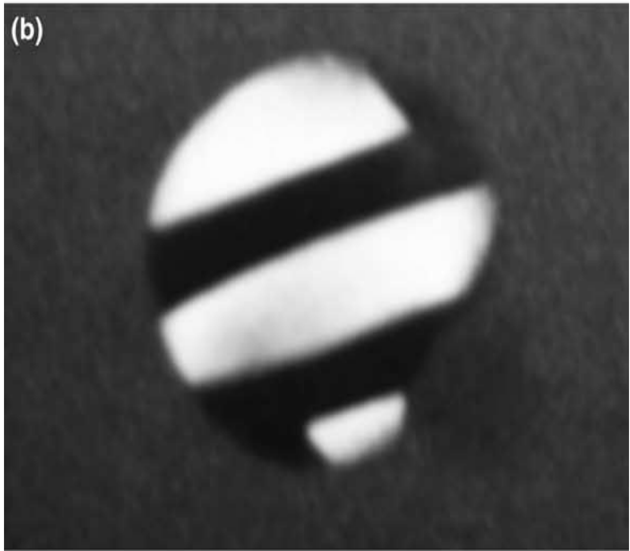
Devitrified naocrystalline particles



twin related
R1-R2-R3
phases



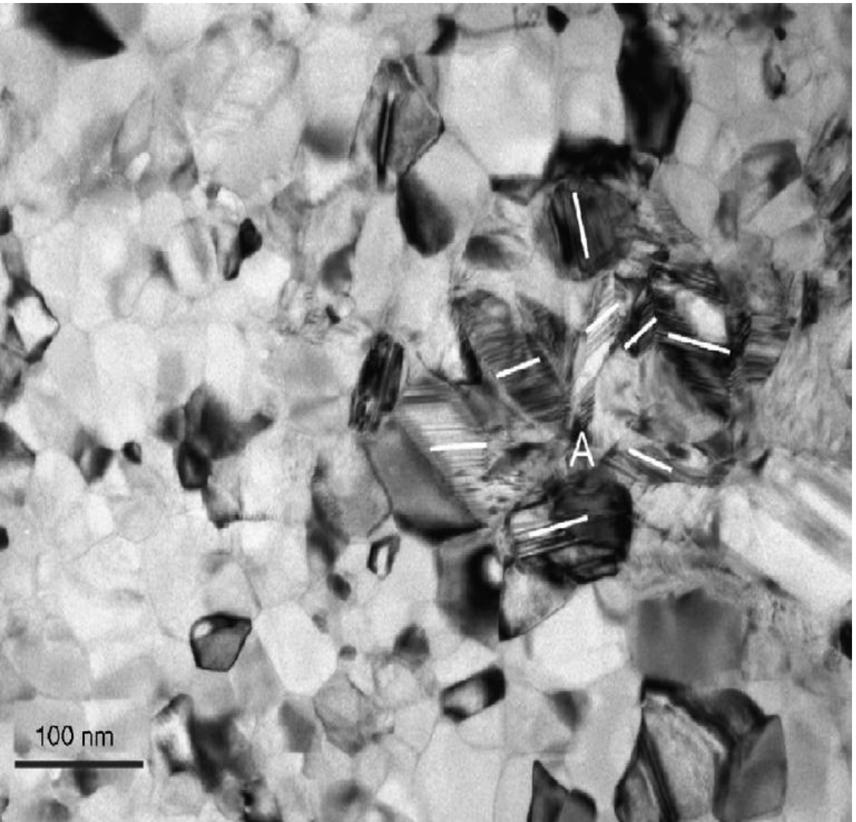
Dark-Field
with **R2** reflection

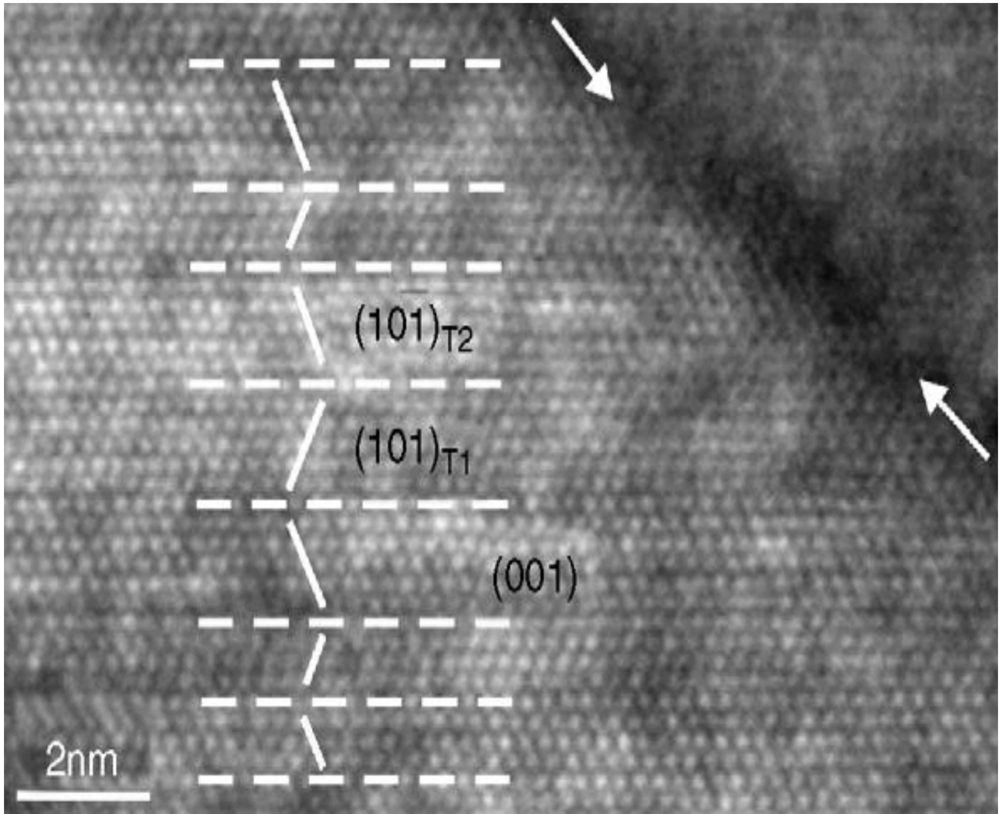


Dark-Field
with **R1** reflection

Ni-Ti shape-memory alloys deform by twinning

T. Waitz, T. Antretter, F.D. Fischer, N.K. Simha, H.P. Karnthaler: *J. Mech. Phys. Solids*, 55 (2007) 419–444

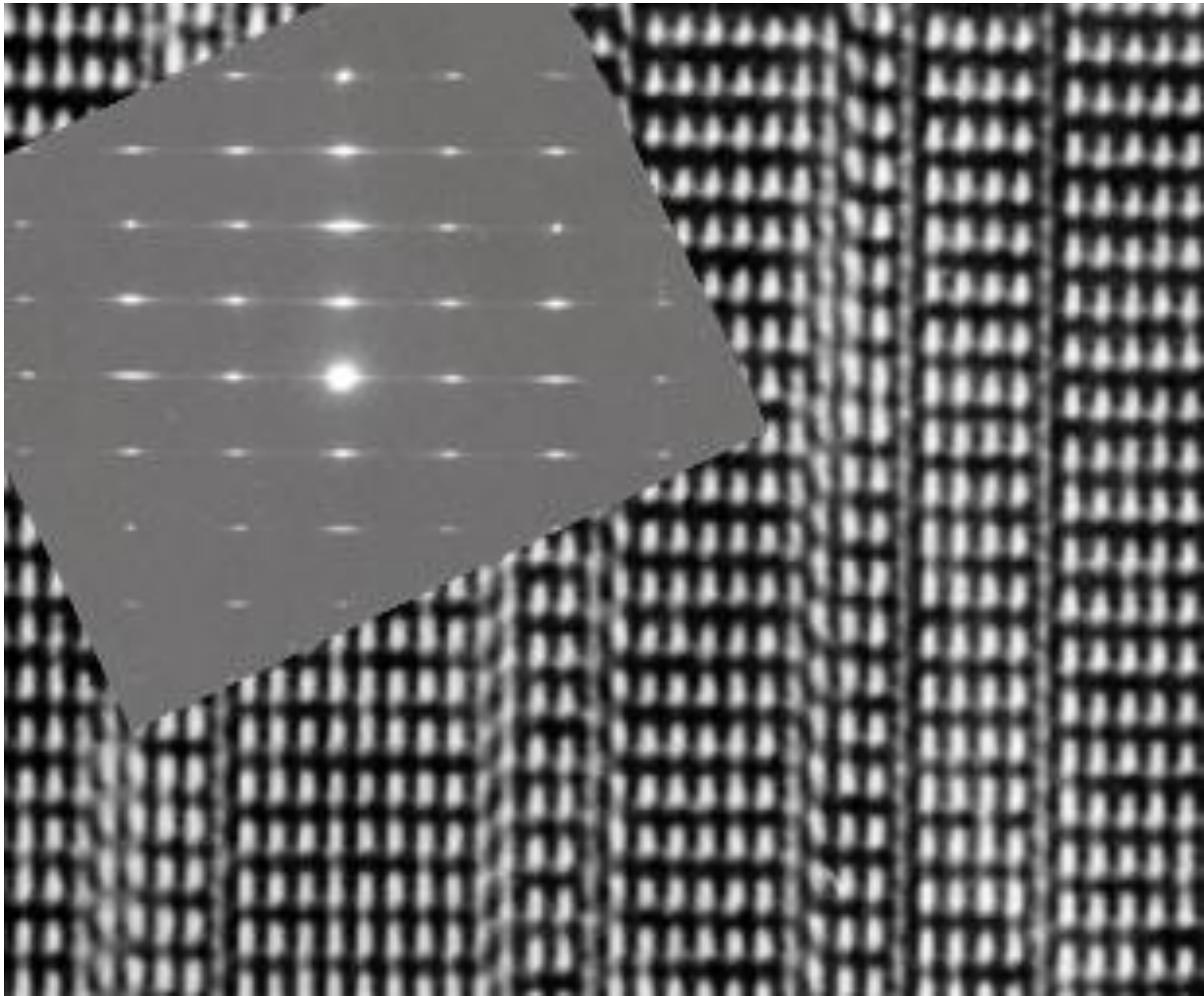




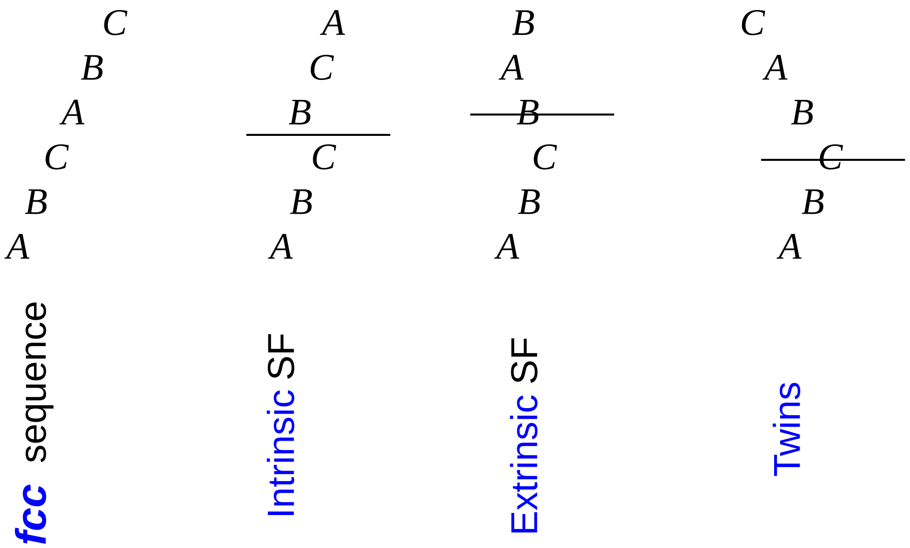
Planar Defects in Magnesium-Fluorogermanate

P. Kunzmann in J.M. Cowley, Acta Cryst. (1976). A32, 83

Streaking normal to the fault planes



Faulting along the close-packed planes in *fcc* or *hcp* crystals



What about ***hcp*** metals like:

Mg, Ti, Zr, Be, Zn ?

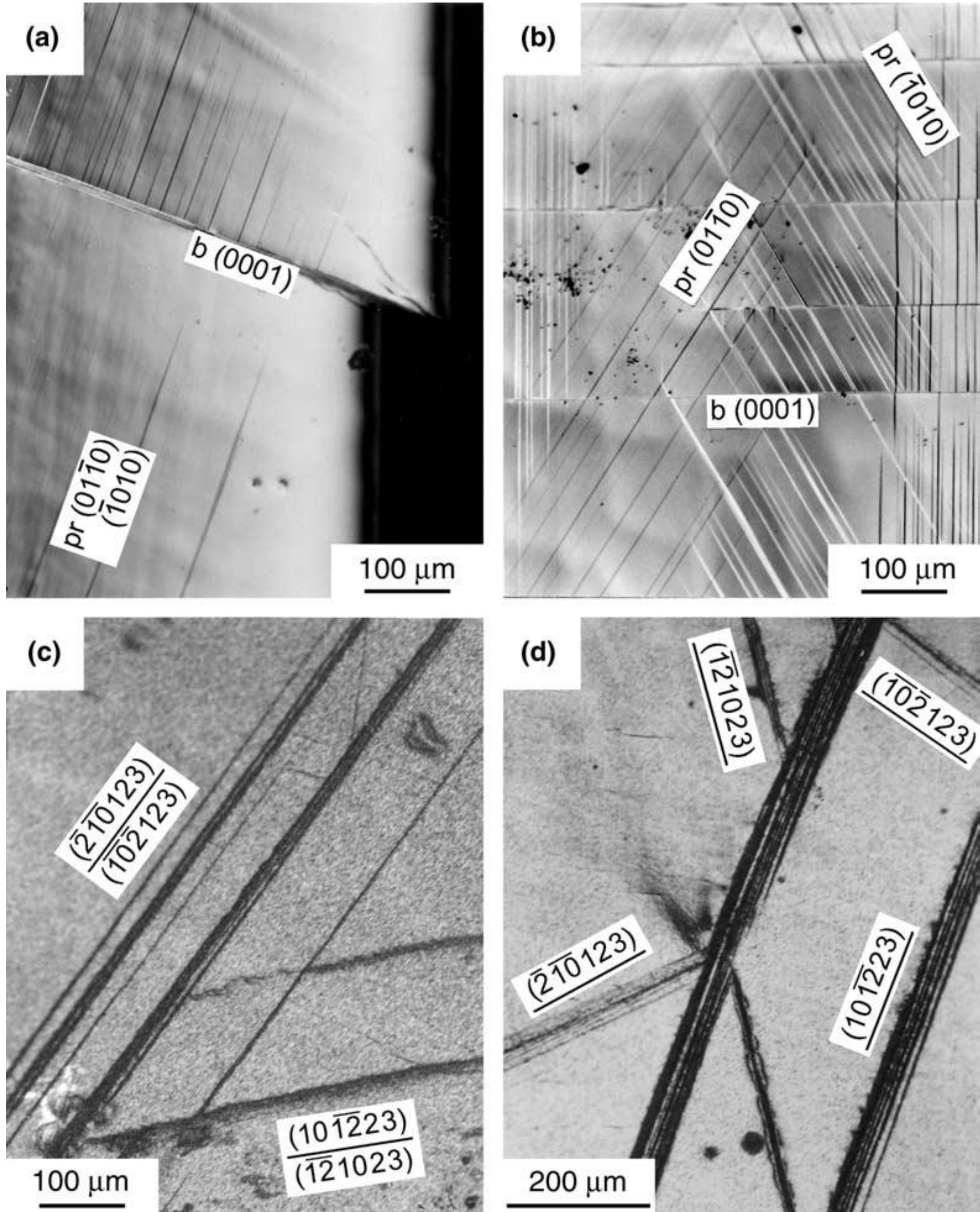
and their alloys

Ti-Al *hcp*-DO_{19}

K. Kishida, Y. Takahama, H. Inui,
Acta Mater, 52 (2004) 4941-4952

prismatic planes

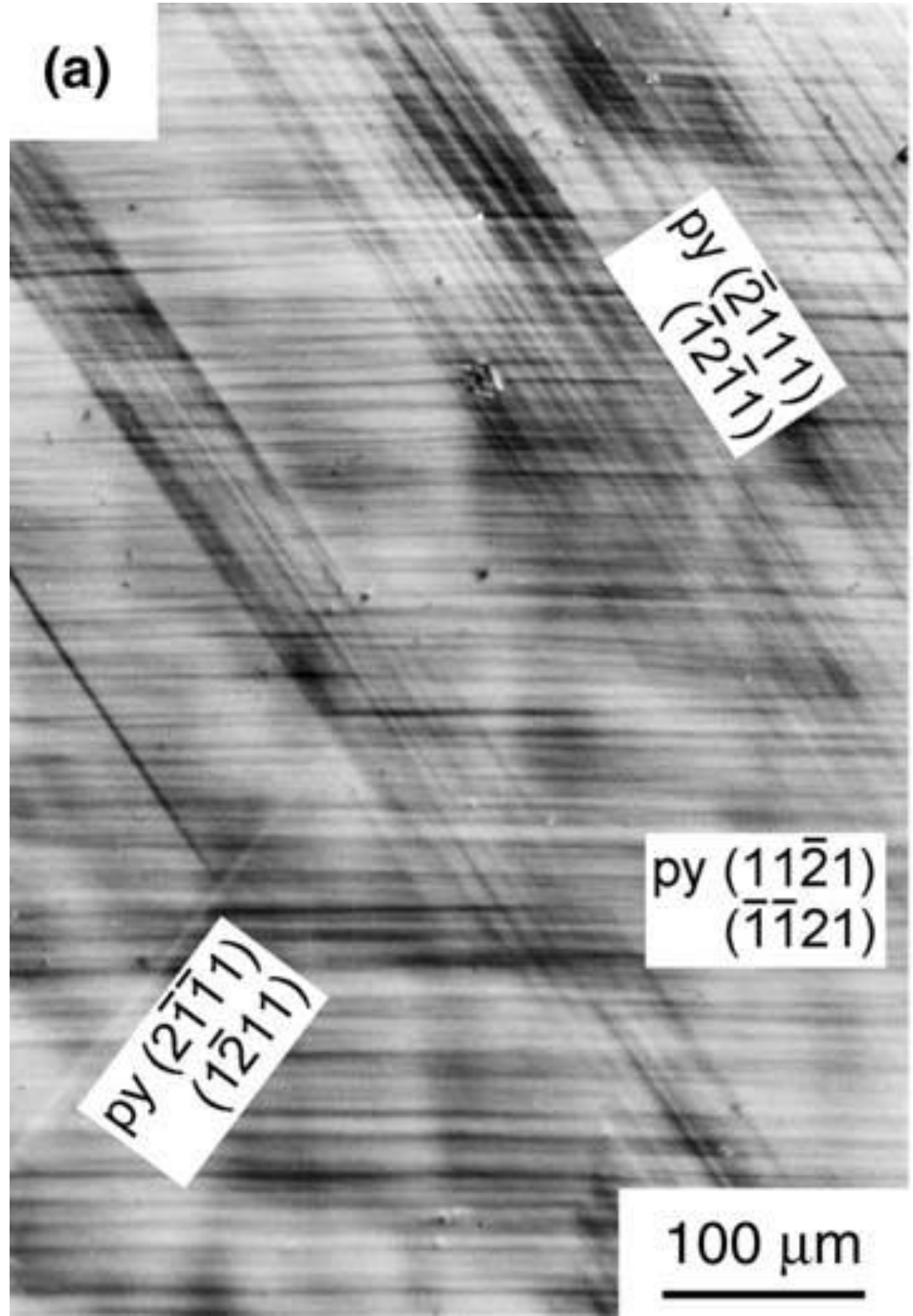
pyramidal planes



optical micrographs

Ti-Al *hcp*-DO_{19}

K. Kishida, Y. Takahama, H. Inui, Acta Mater, 52 (2004) 4941-4952

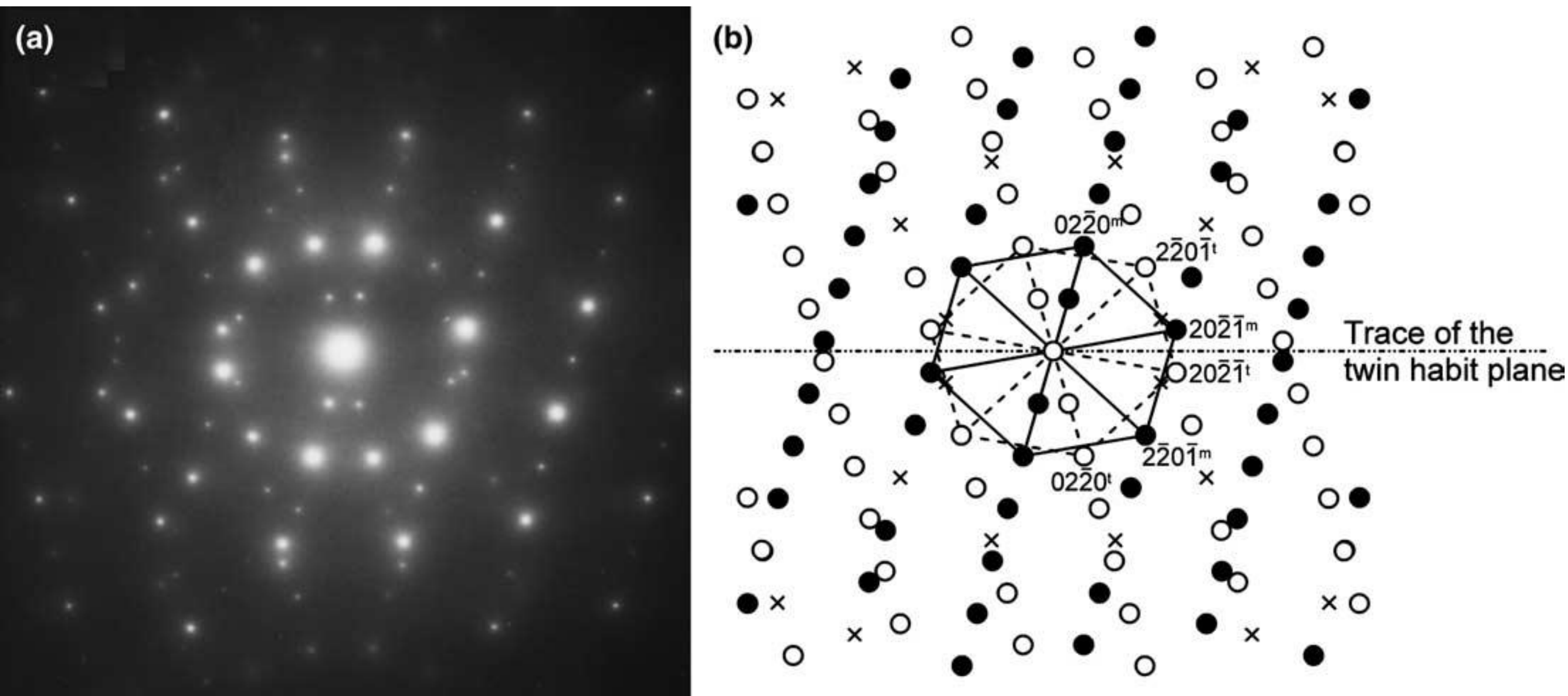


pyramidal planes

Ti-Al *hcp*-DO$_{19}$

K. Kishida, Y. Takahama, H. Inui,
Acta Mater, 52 (2004) 4941-4952

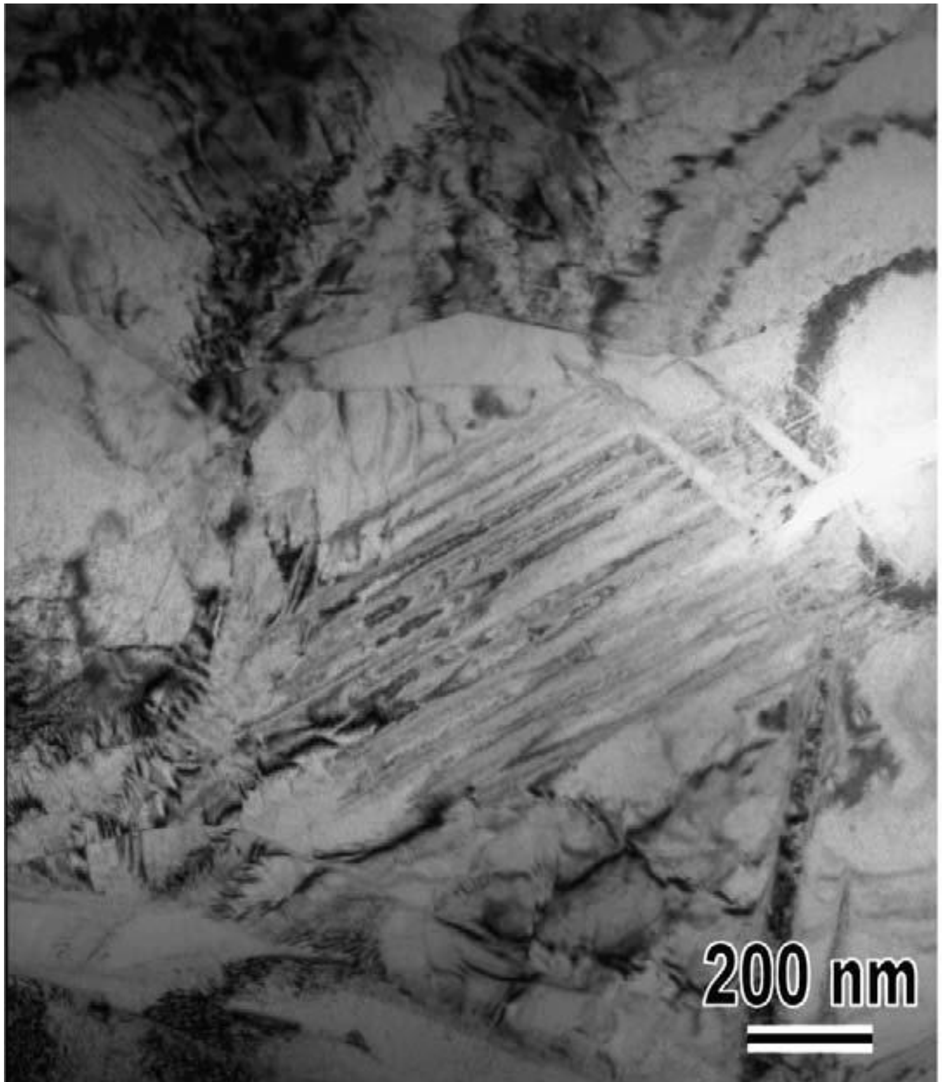
Diffraction spots of
mother and twin crystal
never coincide:
non-merohedral

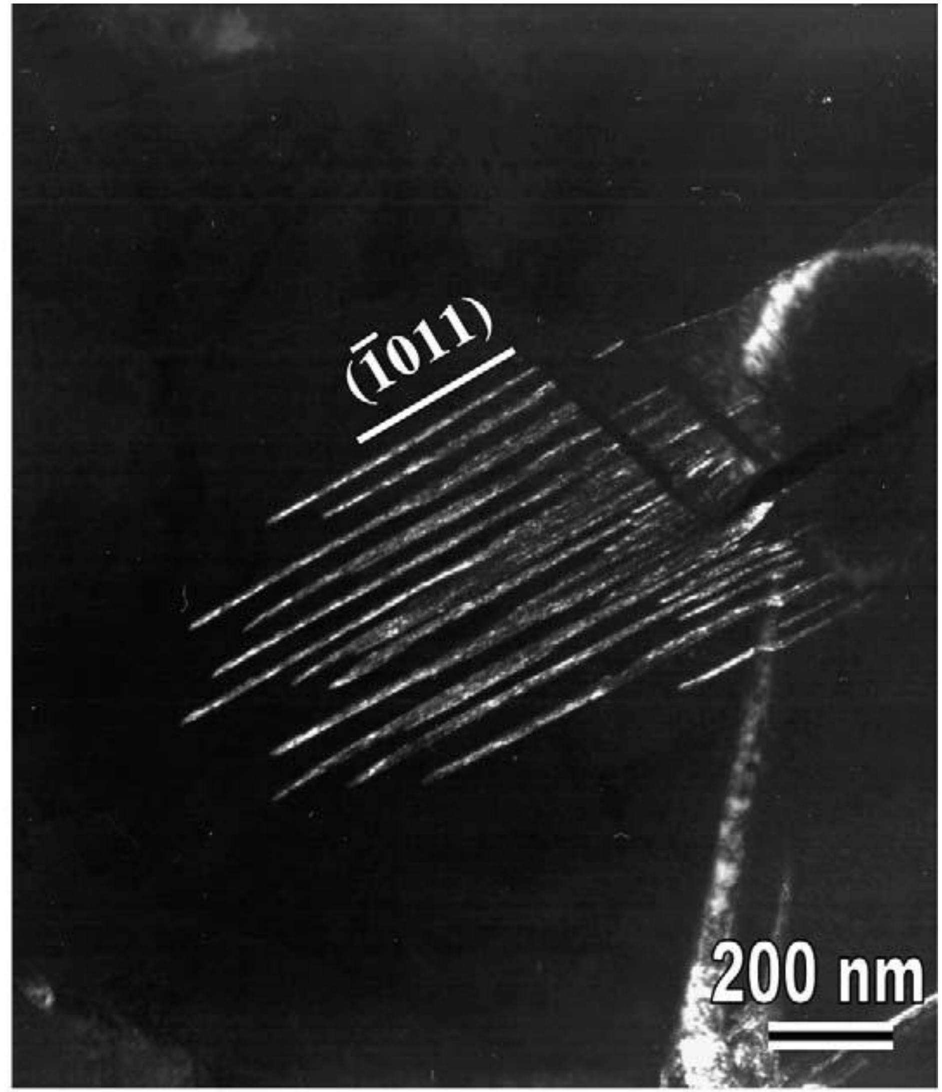


Ti-6Al-4V

G.G.Yapici, I.Karaman,Z.P.Luo, Acta Mater, 54 (2006) 3755

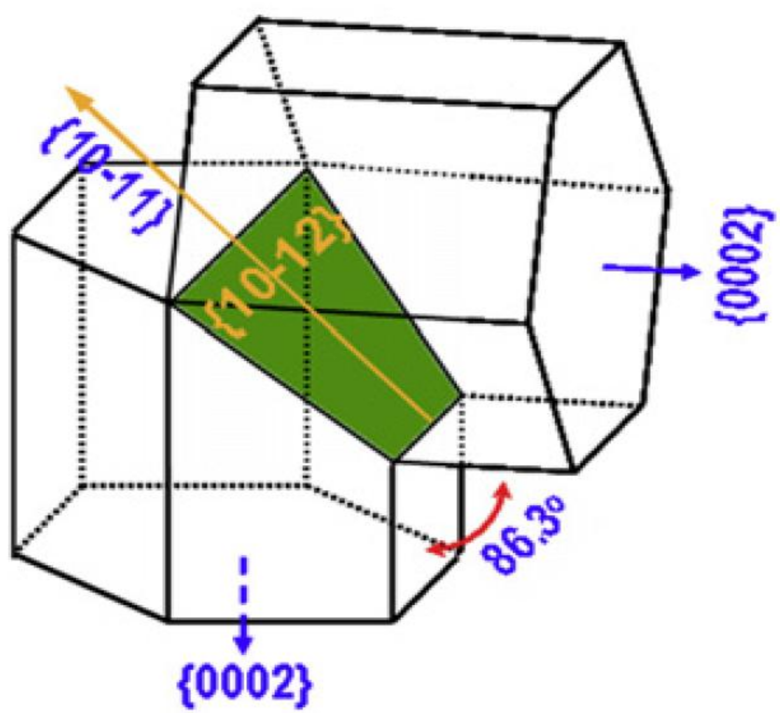
Deformation twins along the $\bar{1}0.1$ pyramidal plane



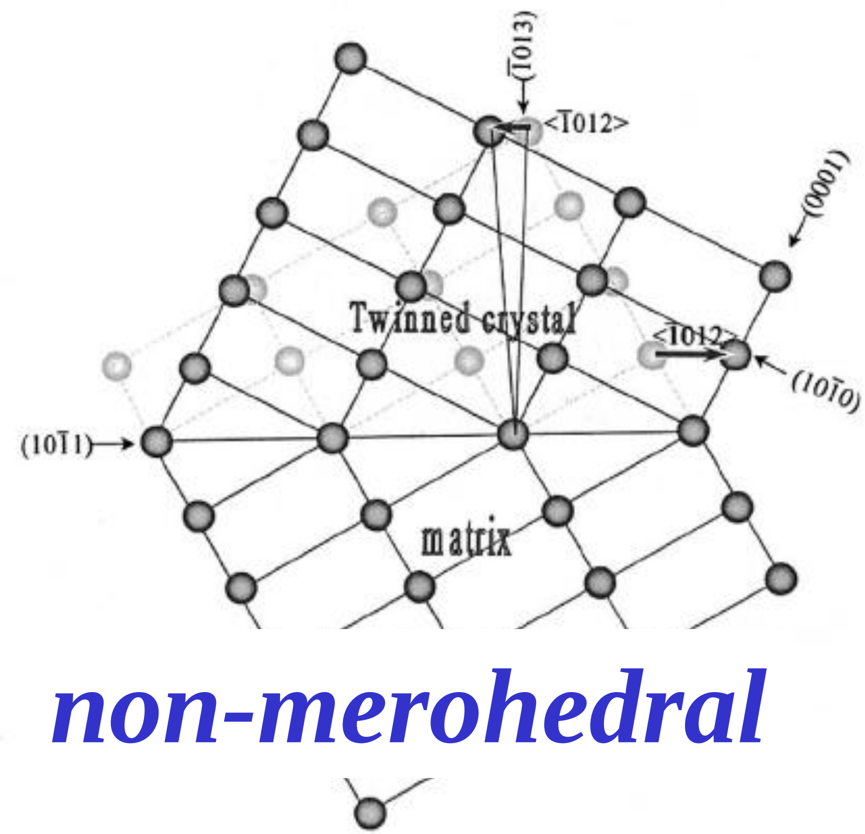


Twinning on pyramidal planes in *hcp* crystals

{10.2}



{10.1}



non-merohedral

L. Wu, A. Jain, D.W. Brown, G.M. Stoica, S.R. Agnew, B. Clausen, D.E. Fielden, P.K. Liaw Acta Materialia 56 (2008) 688–695

I. Kim, W.S. Jeong, J. Kim, K.T. Park, D.H. Shin Scripta Materialia 45 (2001) 575-581

Philosophy of the present line-profile analysis

Bottom-up approach

profile-functions are

created by **theoretical methods**

based on **real lattice defects**

diffraction patterns are **fitted** by

patterns constructued by using

defect-related profile-functions

Philosophy of line profile analysis

$$I_{meas} \Leftrightarrow I_{size} * I_{strain} * I_{planar} * I_{instr} + BG$$

faults

Measured pattern

Physically modeled

Experimentally determined

Non-linear, least squares fitting

I_{Strain} : ρ, M, q

$M = R_e \rho^{1/2}$

I_{Size} : m, σ

I_{planar} : α or β

Strain-broadening: strain-profile

is given by:

$<\varepsilon_{g,L}{}^2>$

mean-square-strain

where the strain Fourier coefficients are:

$$A^D(L) = \exp\{-2\pi^2 g^2 L^2 < \varepsilon^2_{g,L} >\}$$

Dislocation-model for $<\varepsilon_{g,L}{}^2>$: Krivoglaz-Wilkens [1970]:

$$< \varepsilon^2_{L,g} > \ = \frac{\boldsymbol{\rho \cdot C \cdot b^2}}{\mathbf{4\pi}} \quad f(\eta)$$

$\boldsymbol{b}$: Burgers vector

ρ : dislocation density

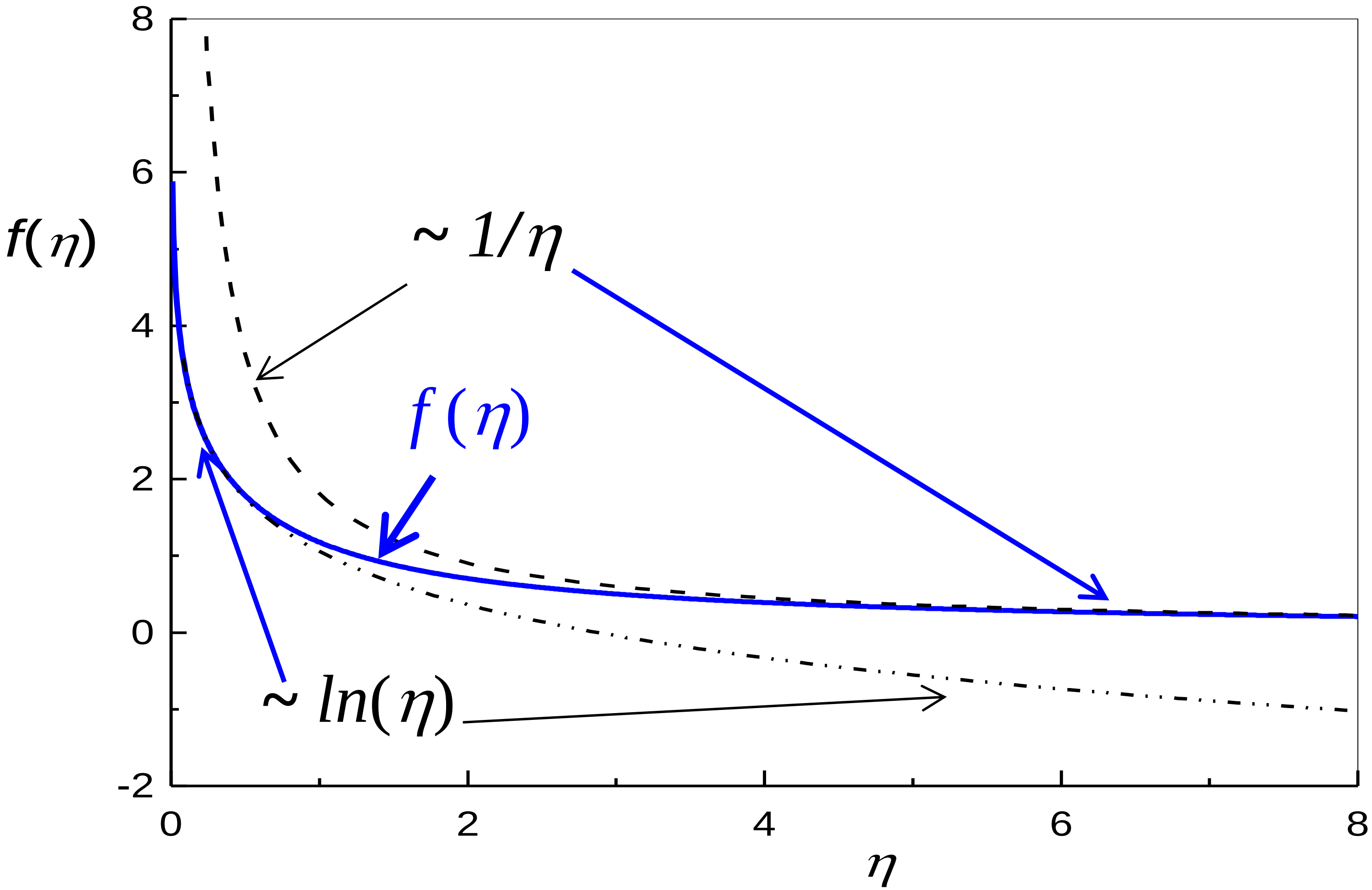
C : Contrast factor of dislocations

$f(\eta)$: Wilkens function [1970]

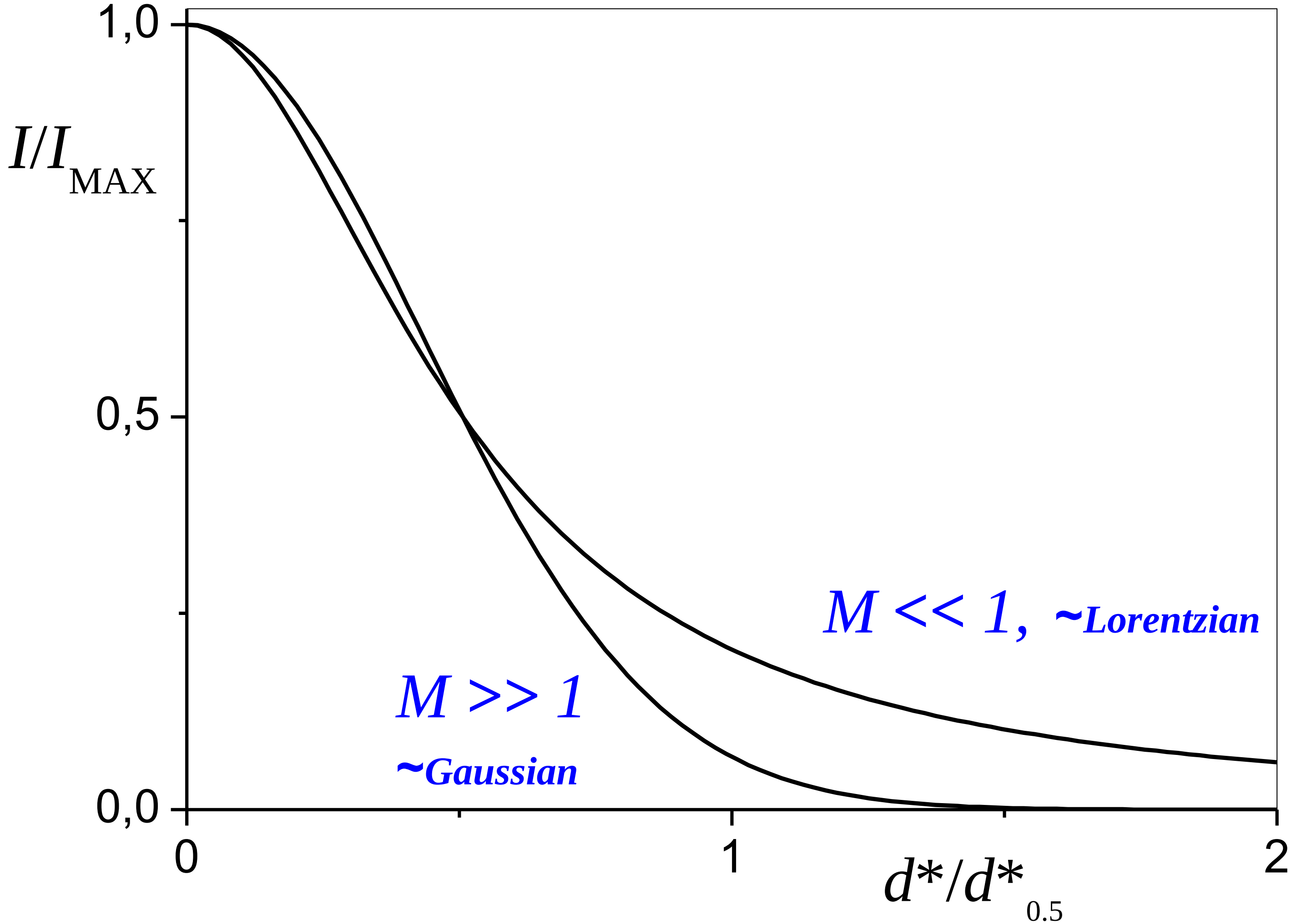
$$\eta = L/\boldsymbol{R}_{\mathbf{e}}$$

The Wilkens function [1970]

$$\eta = L/R_e$$



Strain-profiles normalized



$$M = R_e\sqrt{\rho}$$

Strain profile:

inverse Fourier transform of the *strain Fourier coefficients*

$$I^D(s) = \int \exp\{ - 2\pi^2 L^2 g^2 \langle \varepsilon_{\boldsymbol{g},L}{}^2 \rangle \} \exp(2\pi i L s)\, \mathrm{d}L$$

where:

$$\langle \varepsilon_{g,L}{}^2 \rangle = (b/2\pi)^2 \, \pi\rho\, C \;\; f(\eta)$$

Size profile: I^S

assuming **log-normal** size distribution:

$$f(x) = \frac{1}{(2\pi)^{1/2}\sigma}\frac{1}{x}\exp\left\{-\frac{[\log(x/m)]^2}{2\sigma^2}\right\}$$

m: median

σ: variance

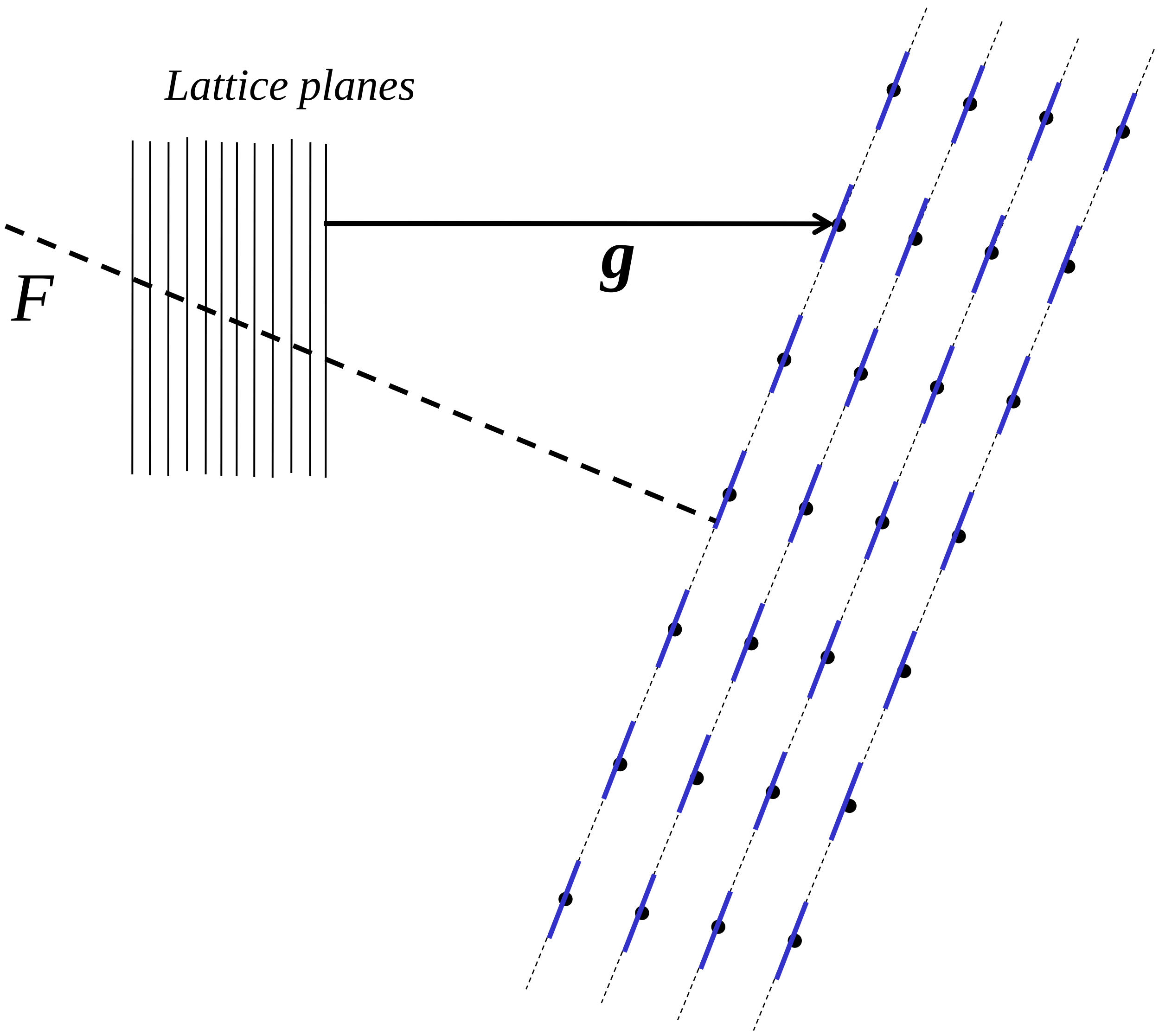
$$I^S(s) = \int_0^\infty \mu \frac{\sin^2(\mu\pi s)}{(\pi s)^2} \operatorname{erfc}\left[\frac{\log(\mu/m)}{2^{1/2}\sigma}\right] \mathrm{d}\mu$$

shape-anisotropy can be allowed for

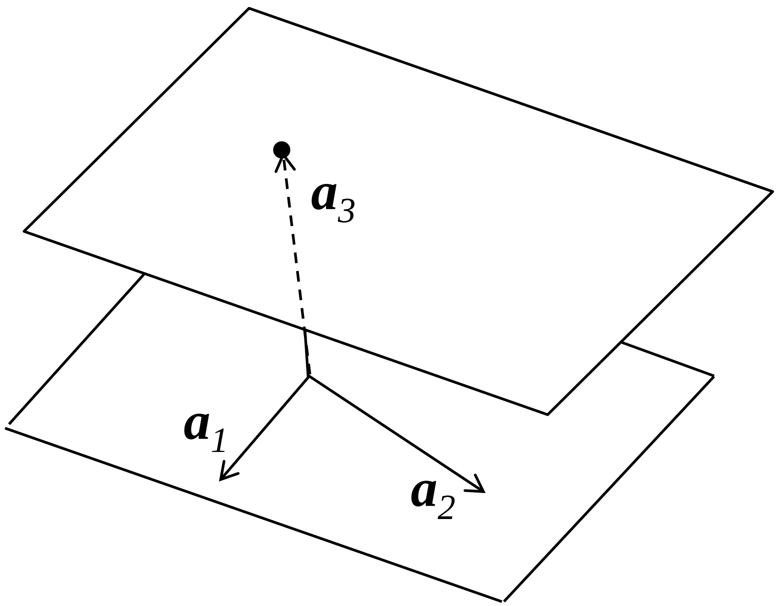
Profile functions for

faulting and ***twinning***

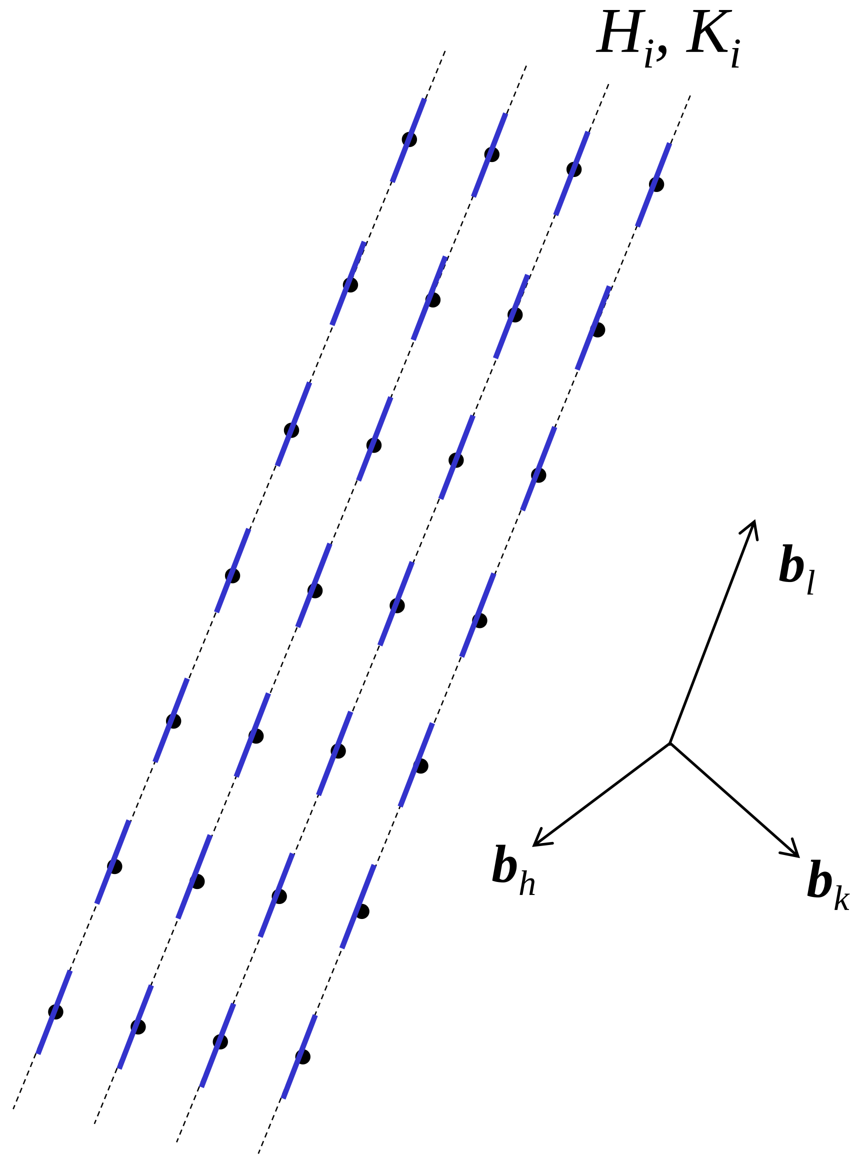
Line broadening from streaking


Lattice planes
F
g

Coordinate systems

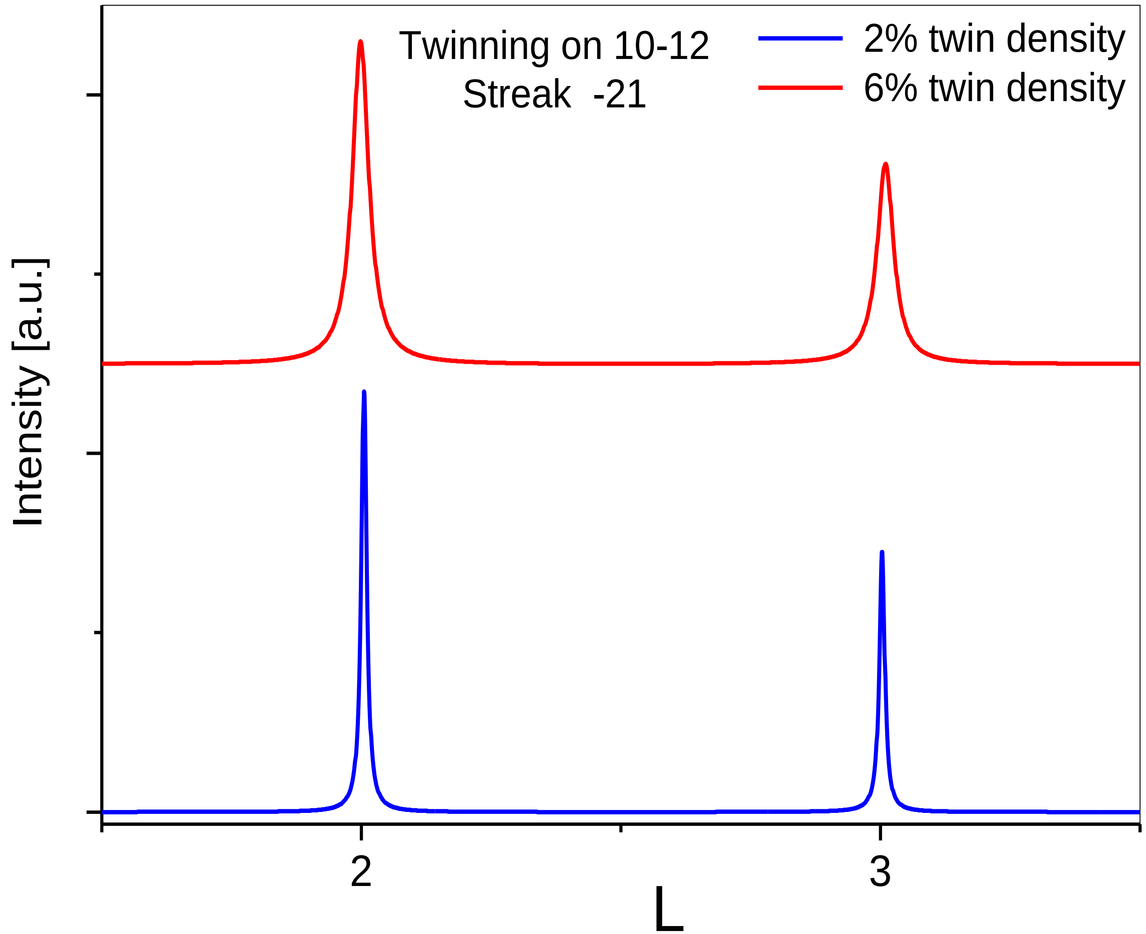


tricline



Intensity distribution along the $(H_i,K_i)=(-2\ 1)$ *streaks*

DIFFaX, **Treacy MMJ, Newsam JM, Deem MW**, Proc. Roy. Soc. London A, (1991) 433, 499-520

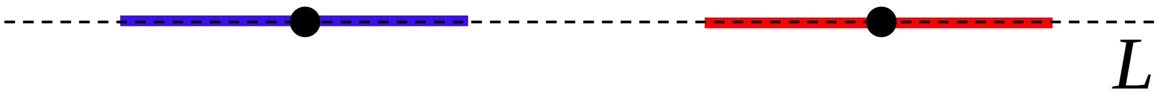


twinning on

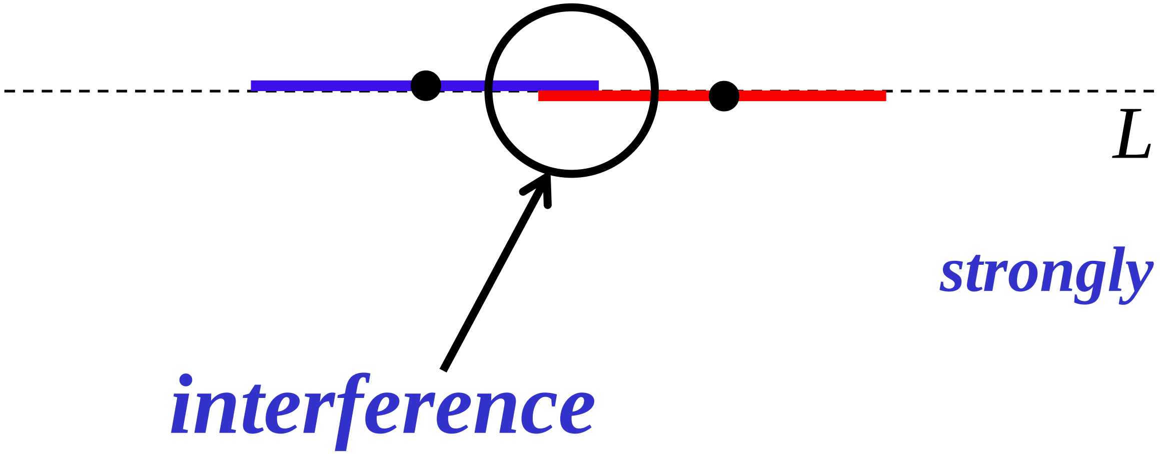
pyramidal planes

NOT line-profiles yet

Overlapping peaks along the ***same streak***: ***asymmetry***



almost symmetric peaks

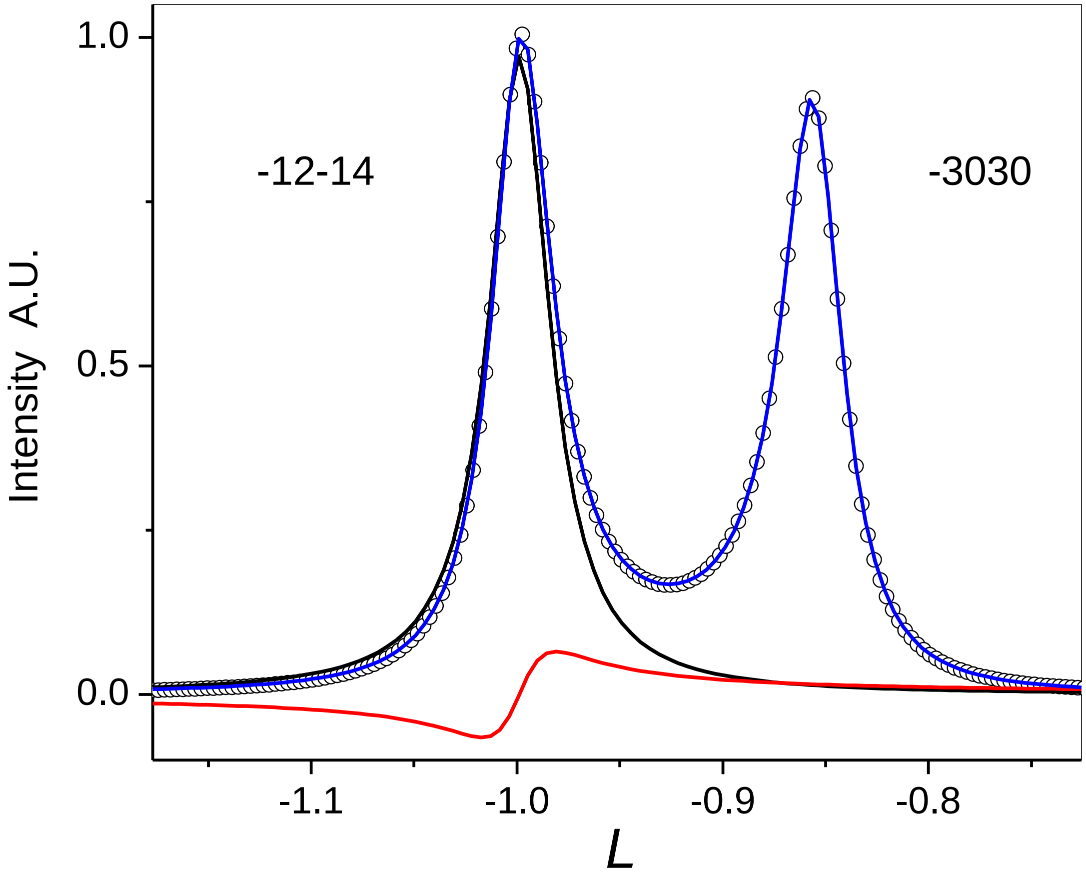


strongly asymmetric peaks

Ti: *pyramidal* twin plane: $11\bar{2}2$

{11.4} reflection: $\bar{1}2\bar{1}4$ sub-reflection (parent crystal)

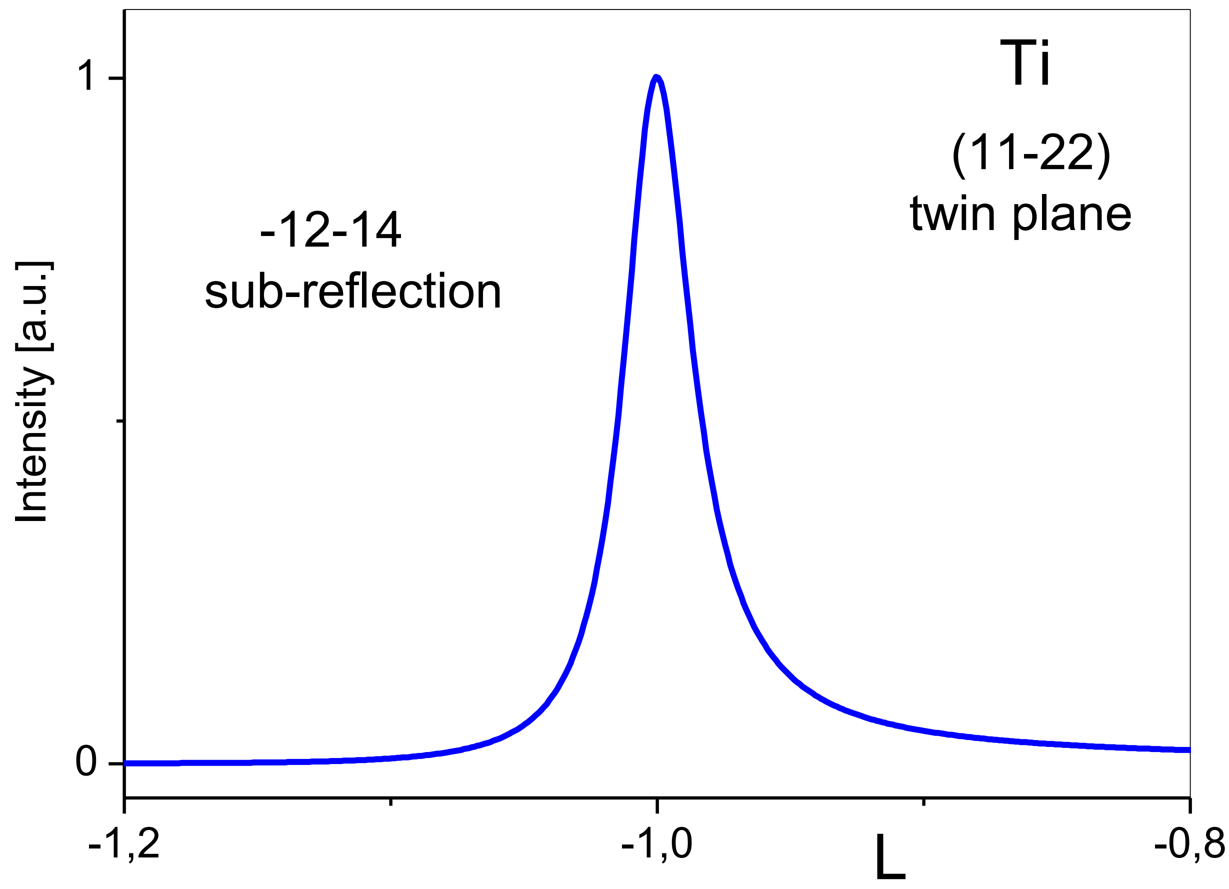
{31.0} reflection: $\bar{3}030$ sub-reflection (twin crystal)



$\beta = 6\ \%$

Ti: *pyramidal* twin plane: $11\bar{2}2$

only the $\bar{1}2\bar{1}4$ sub-reflection in the {11.4} reflection



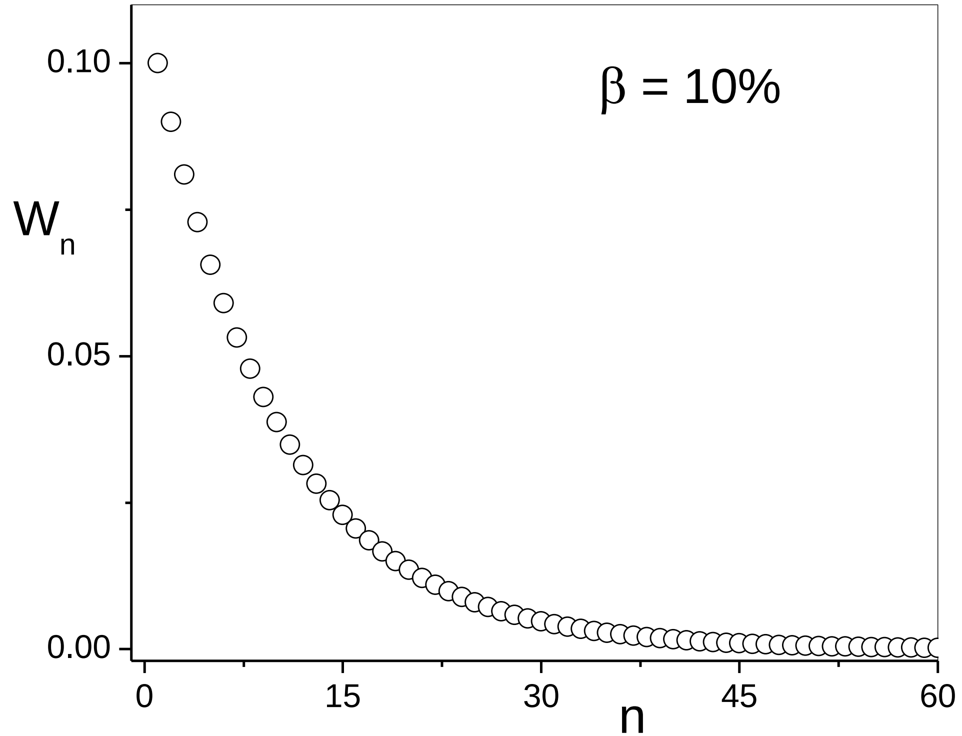
$\beta = 6\ \%$

Intensity distribution in the streaks

twinning probability *or* *twin boundary* frequency: β

fraction *of* twin lamellae *of* thickness *of* n *crystal-layers*: W_n

$$W_n = \beta(1-\beta)^{n-1}$$



Intensity distribution in the streaks: $I(L)$

$$I(L) = \frac{I_0}{1 + \frac{4(L-L_0)^2}{FWHM_L{}^2}} \left[1 + A_{asym} \cdot (L - L_0)\right]$$

where: $$FWHM_L = \frac{2\beta}{D_{tri}\sqrt{1-\beta}}$$

$I(L)$: *Lorentzian* type function

L. Balogh, G. Tichy and T. Ungár, *J. Appl. Cryst.* 42, (2009) 580-591.

It can been shown that:

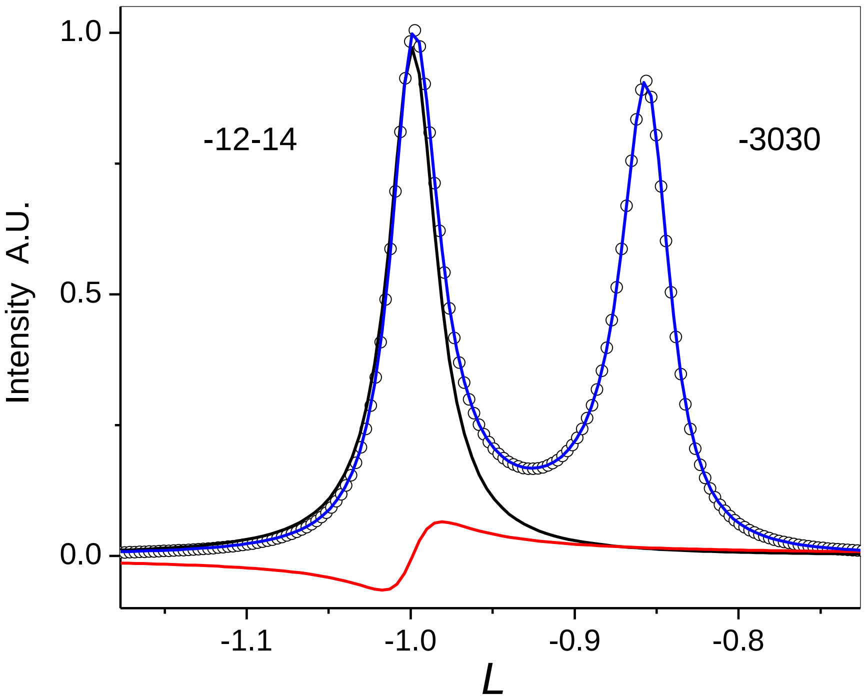
$$FWHM_L = \frac{2\beta}{D_{tri}\sqrt{1-\beta}}$$

is a ***universal*** relation for ***twinning***,

where $\mathbf{D_{tri} = 5.775}$

L. Balogh, G. Tichy and T. Ungár, *J. Appl. Cryst.* 42, (2009) 580-591.

Intensity distribution in the streaks: ***sub-profiles***



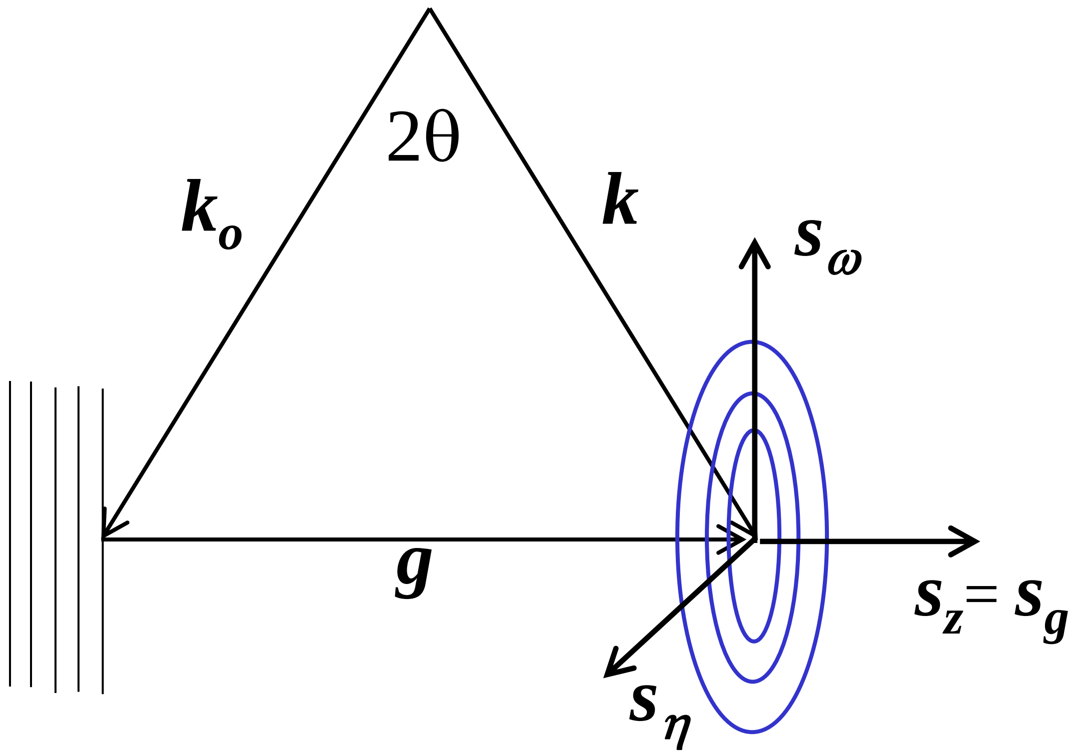
$$I(L) = \frac{I_0}{1 + \frac{4(L - L_0)^2}{FWHM_L^{\ 2}}}\left[1 + A_{asym} \cdot (L - L_0)\right]$$

I(L) : the sum of

a ***symmetrical*** **+** an ***antisymmetrical***

Lorentzian type function

Intensity distribution in reciprocal-space is 3 dimensional



$$I = I(s_\omega, s_\eta, s_g)$$

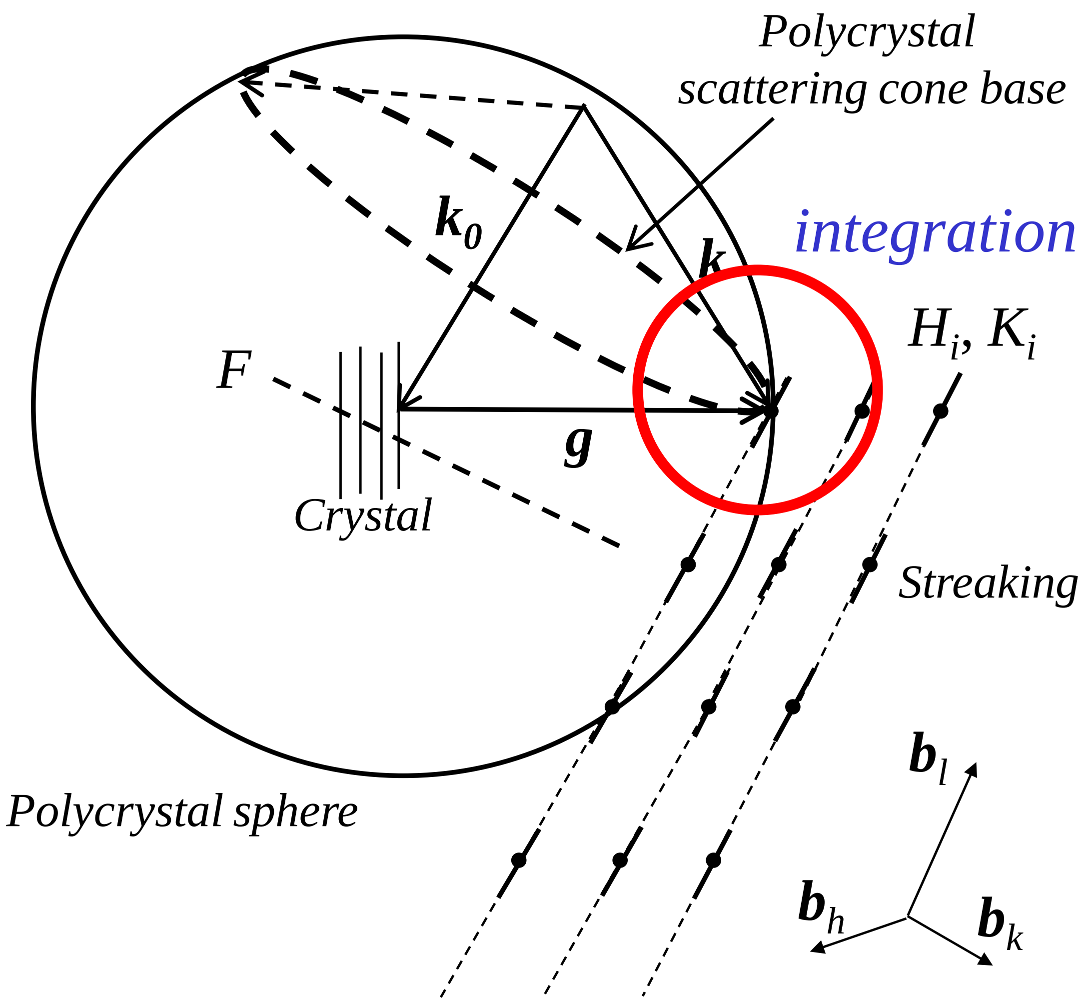
Line profile:

intensity distribution along the diffraction vector: ***g***

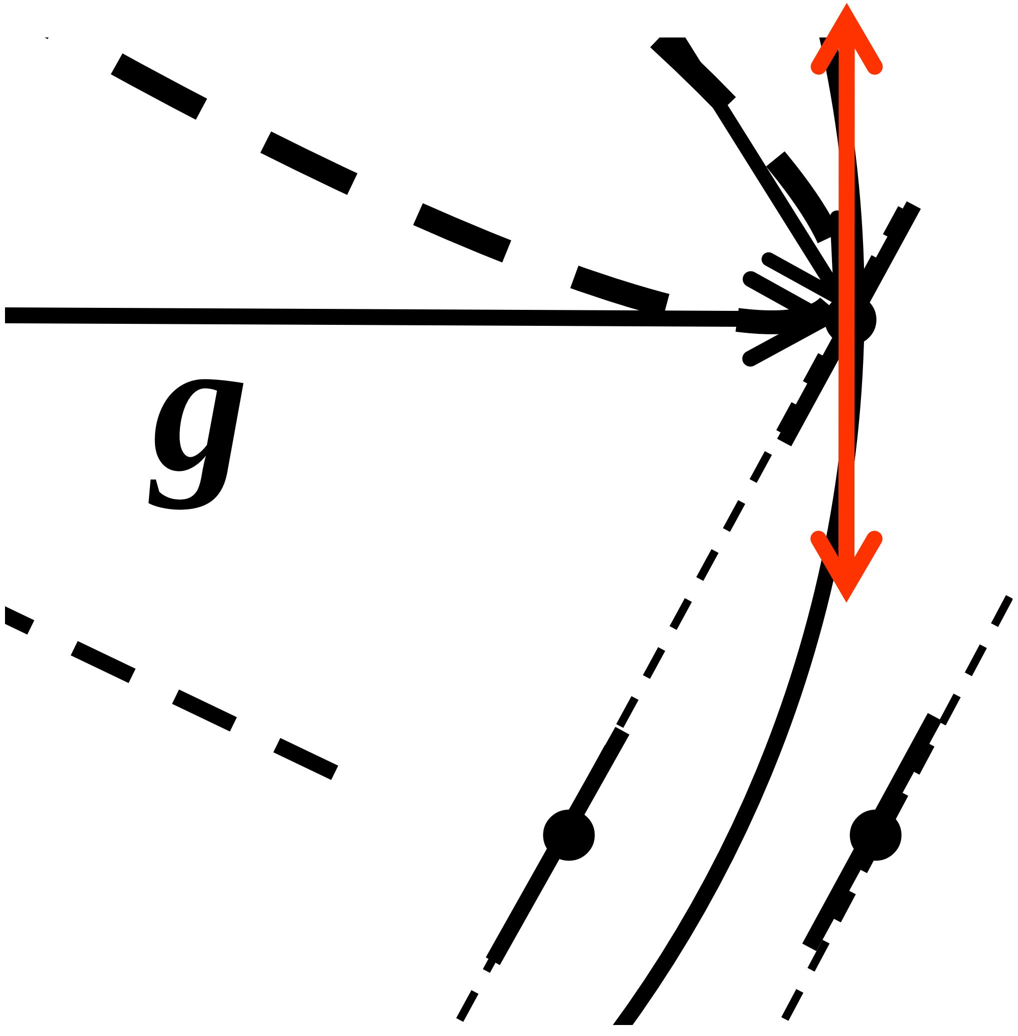
scattered radiation is integrated normal to the ***g vector***

$$I(s_g) = \iint I(s_\omega, s_\eta, s_g)\, ds_\omega\, ds_\eta$$

Correlation between streaking and line broadening



integration normal to the ***g* vector**



It can been shown that:

transformation between

L and ***g*** is ***linear***

to a ***very good*** approximation

L *into* ***g*** transformation for twinning on close-packed planes

$$FWHM_g(\beta) = \frac{1}{g}\left|\frac{h+k+l}{a^2}\right| FWHM_L(\beta)$$

$$FWHM_L = \frac{2\beta}{D_{tri}\sqrt{1-\beta}}$$

L. Velterop, et. al., *J. Appl. Cryst.* (2000). 33, 296-306
E. Estevez-Rams, et. al., *J. Appl. Cryst.* 34, (2001) 730
L. Balogh, G. Ribárik, T Ungár, J.Appl.Phys. 100 (2006) 023512

L *into* *g* transformation for *twinning* on *pyramidal planes* in *hcp* crystals

$$(10\bar{1}1):\quad FWHM(g) = \frac{2}{g}\left|\frac{4c^2h + 2c^2k - 3a^2l}{6a^2c^2}\right| FWHM_{\text{triclinic}}$$

$$(10\bar{1}2):\quad FWHM(g) = \frac{2}{g}\left|\frac{2c^2h + c^2k + 3a^2l}{3a^2c^2}\right| FWHM_{\text{triclinic}}$$

$$(11\bar{2}1):\quad FWHM(g) = \frac{2}{g}\left|\frac{2c^2h + 2c^2k + a^2l}{2a^2c^2}\right| FWHM_{\text{triclinic}}$$

$$(11\bar{2}2):\quad FWHM(g) = \frac{2}{g}\left|\frac{c^2h + c^2k + a^2l}{a^2c^2}\right| FWHM_{\text{triclinic}}$$

$$FWHM_{\text{triclinic}} = \frac{2\beta}{D_{tri}\sqrt{1-\beta}}$$

L. Balogh, G. Tichy and T. Ungár *J. Appl. Cryst. 42, (2009)* 580-591.

Ti: *pyramidal* twin plane: $11\bar{2}2$

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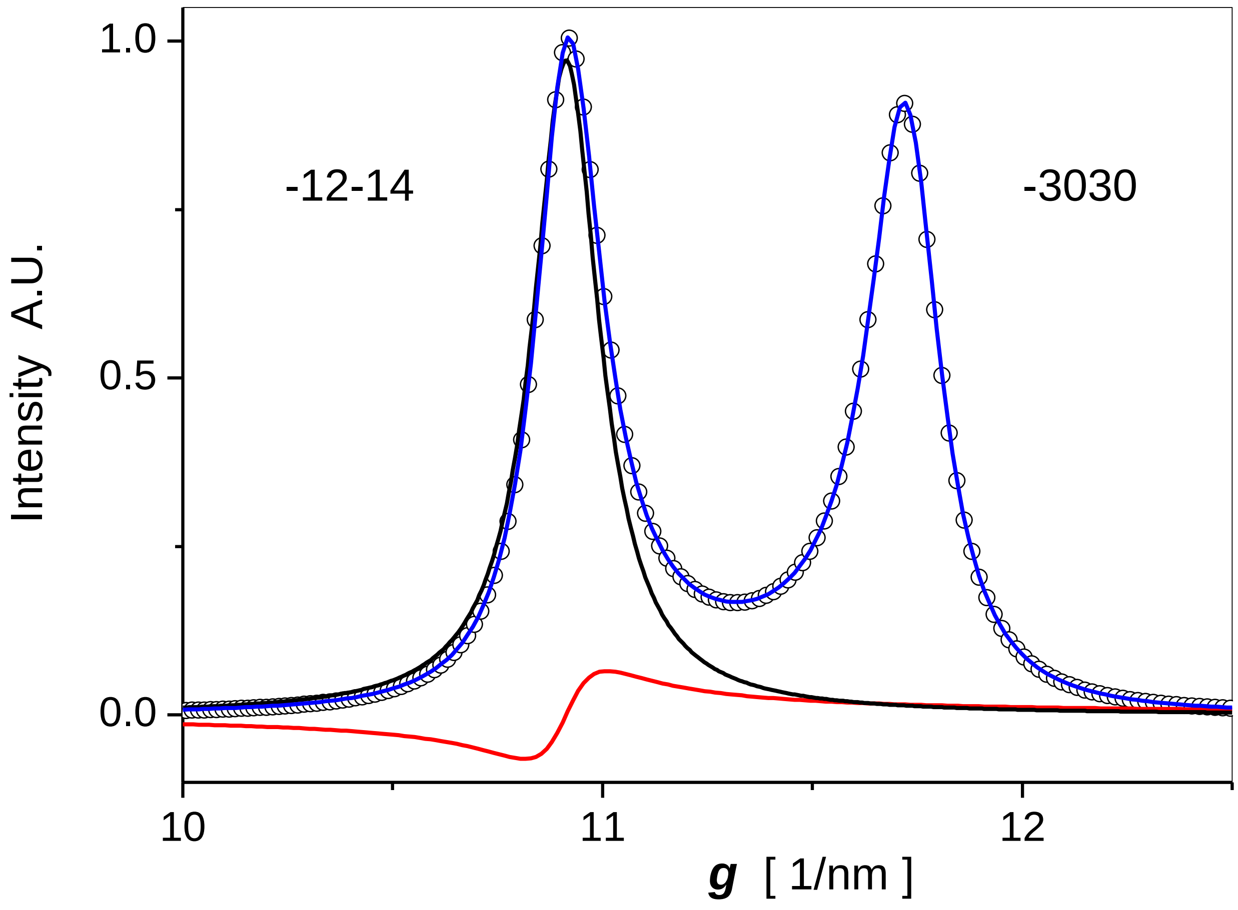


$\beta = 6\ \%$

Profile functions for twinning

along the ***streaks*** there is ***NO hkl dependence***,

up to the asymmetry

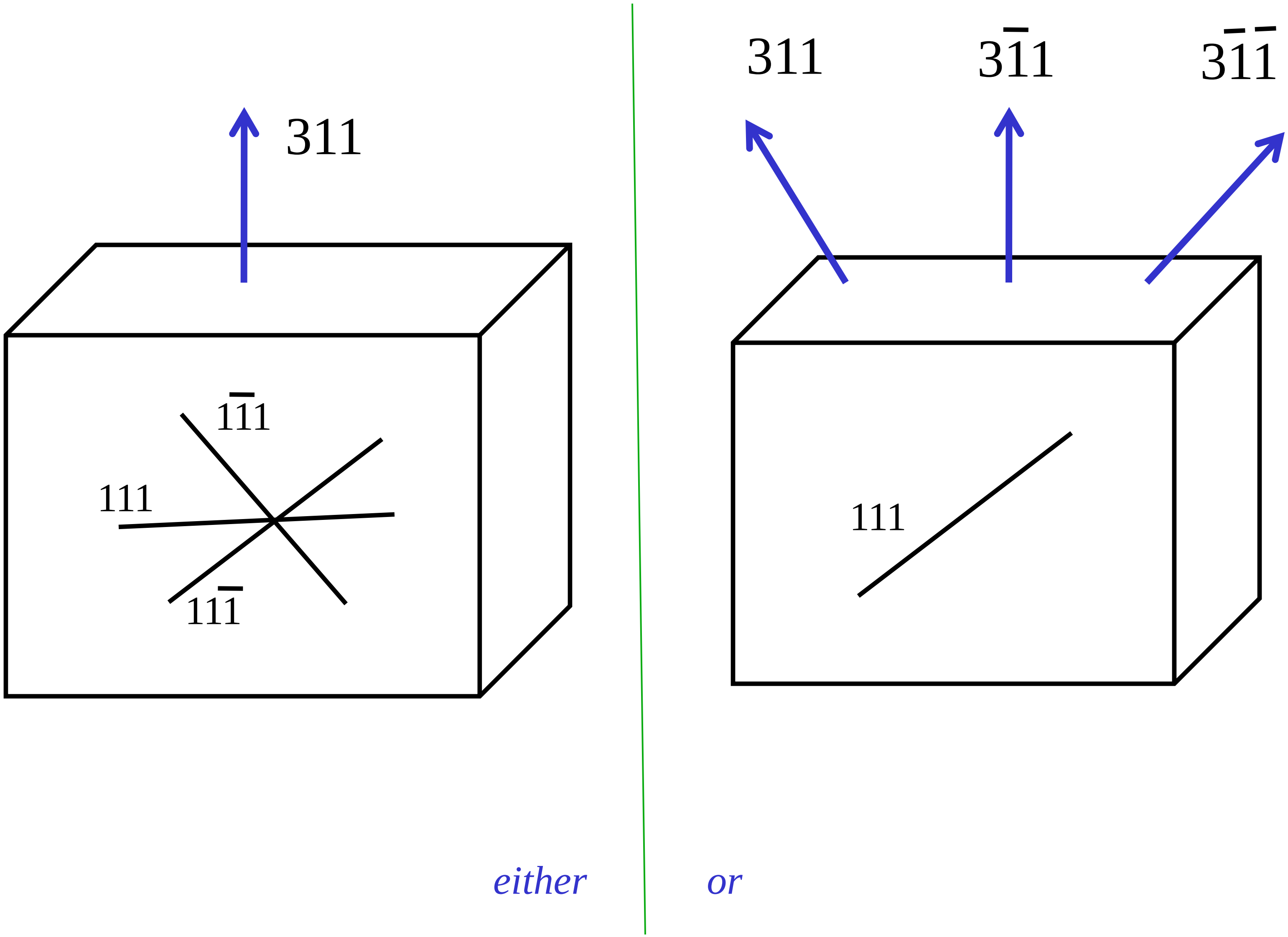
L *into* ***g*** transformation

produces the ***hkl dependence***

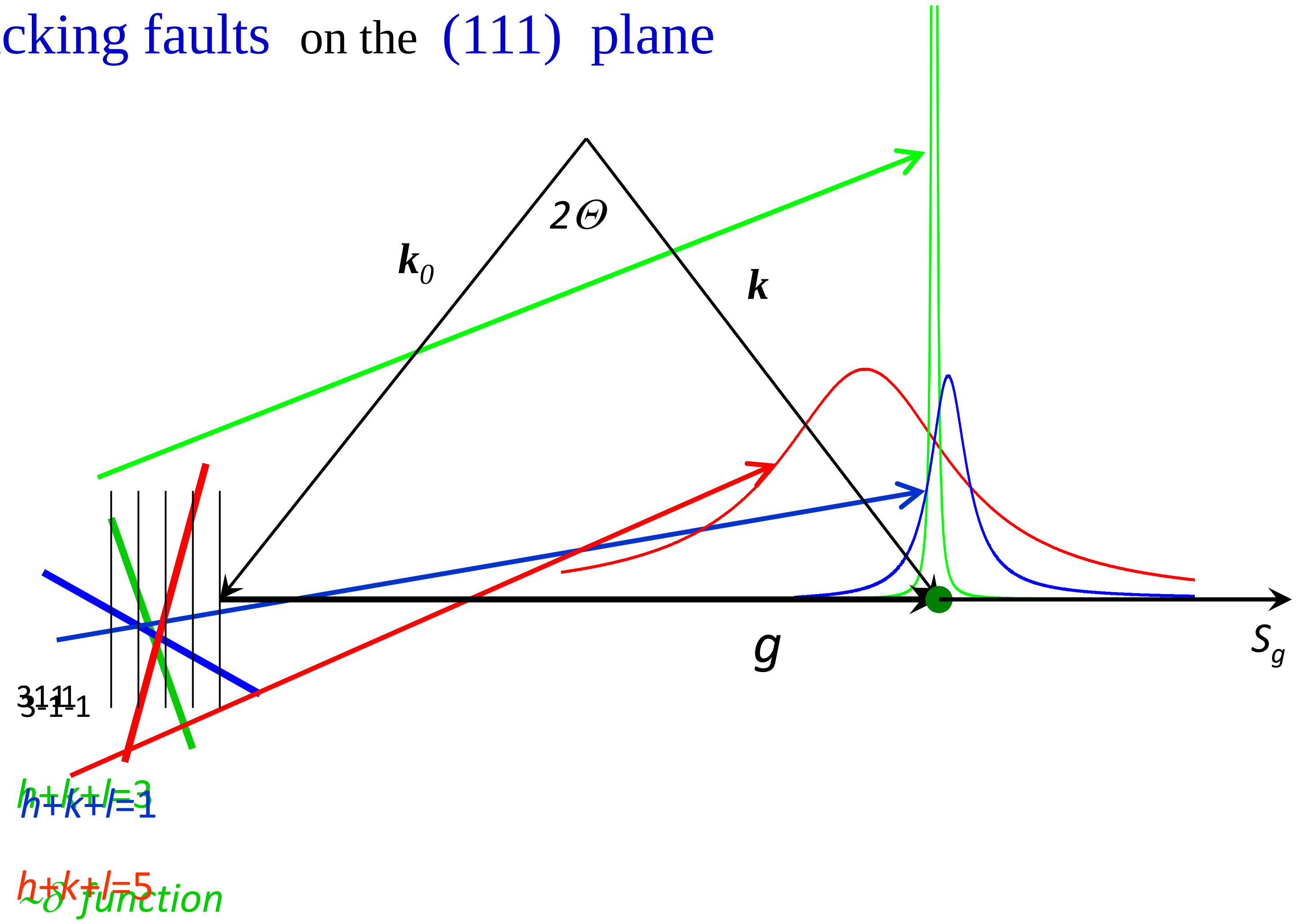
Resultant or measured profiles are

the ***sum*** of ***sub-profiles***

Summation of sub-profiles

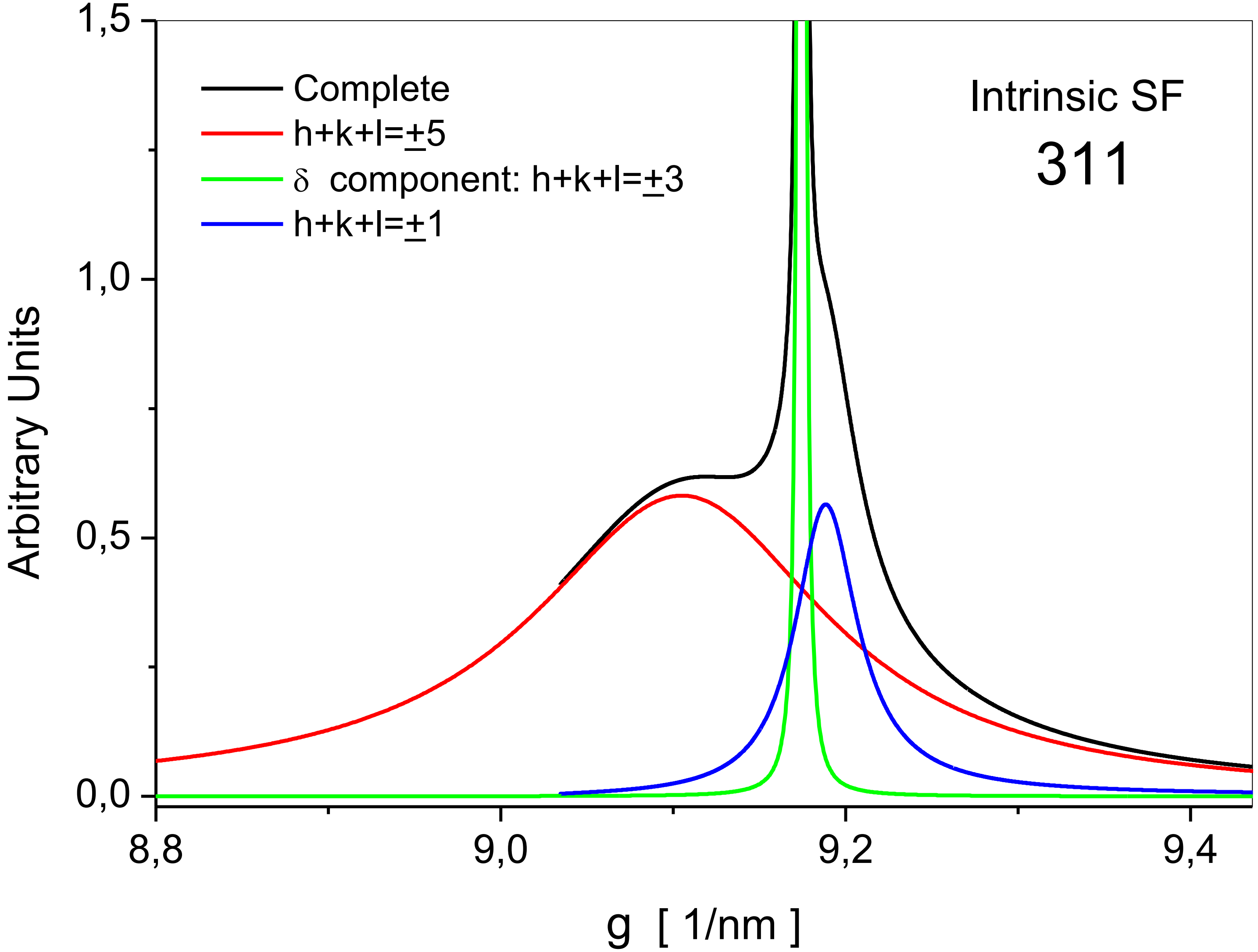


intrinsic stacking faults on the (111) plane



the ***311*** type sub-reflections in ***fcc*** crystals

Subreflections according to different *hkl* permutations



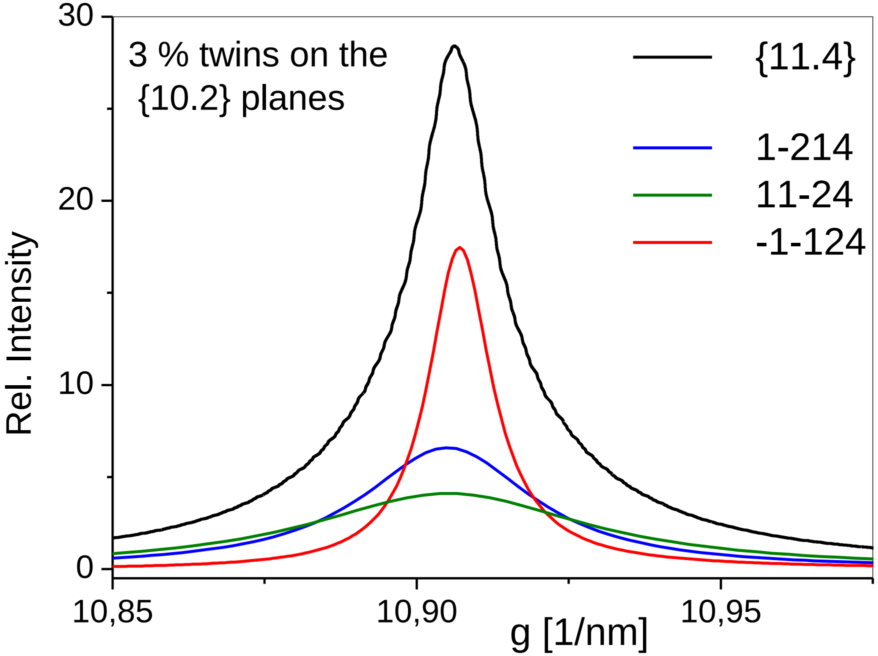
$\alpha = 4\ \%$

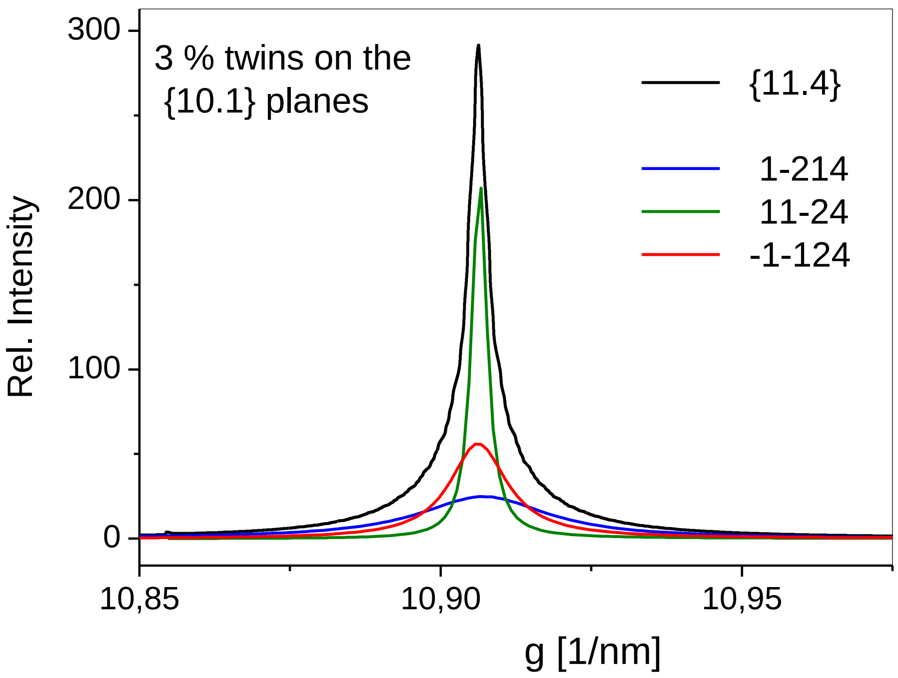
hkl dependence in the *I*(*g*) profiles: Ti

subreflections and {11.4} powder diffraction profiles

Twin planes: {10.2}

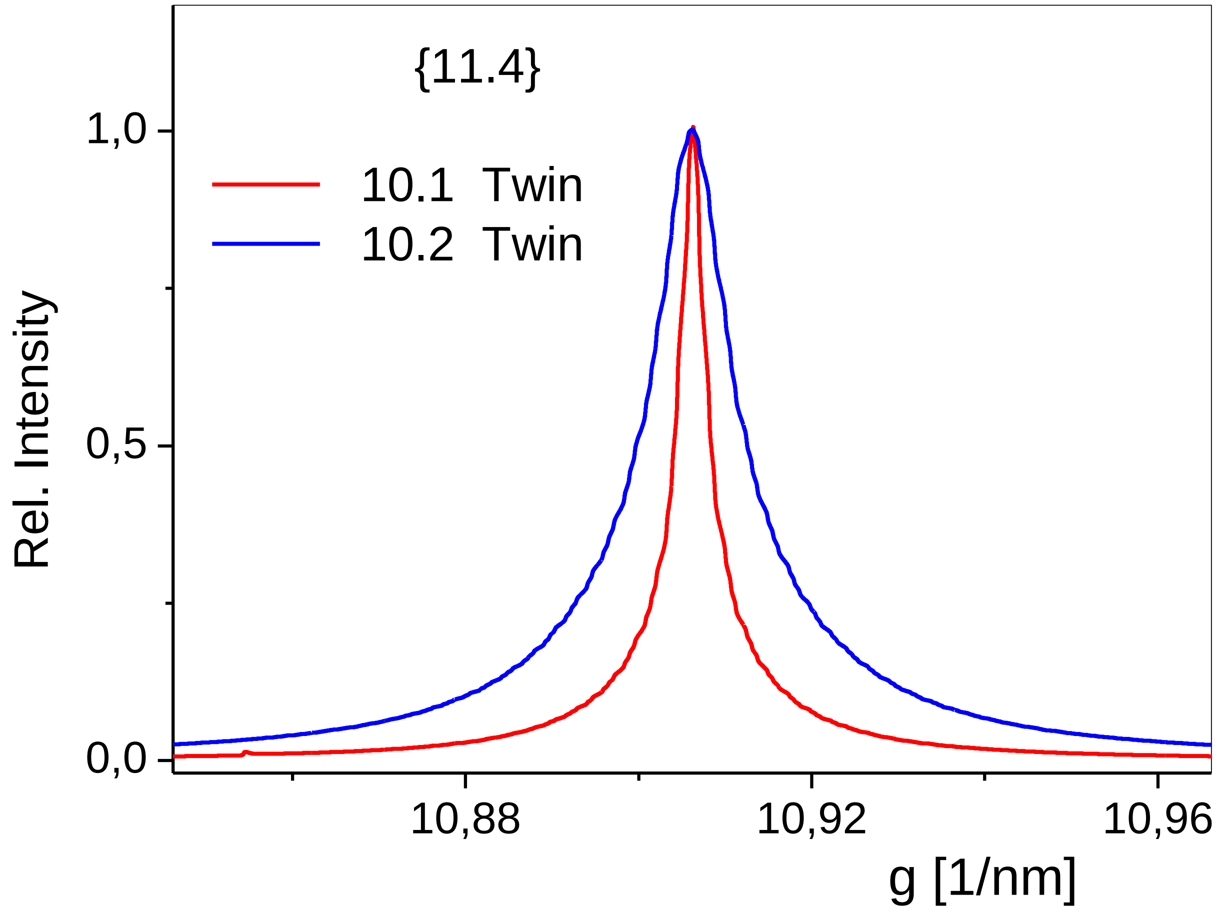
Twin planes: {10.1}





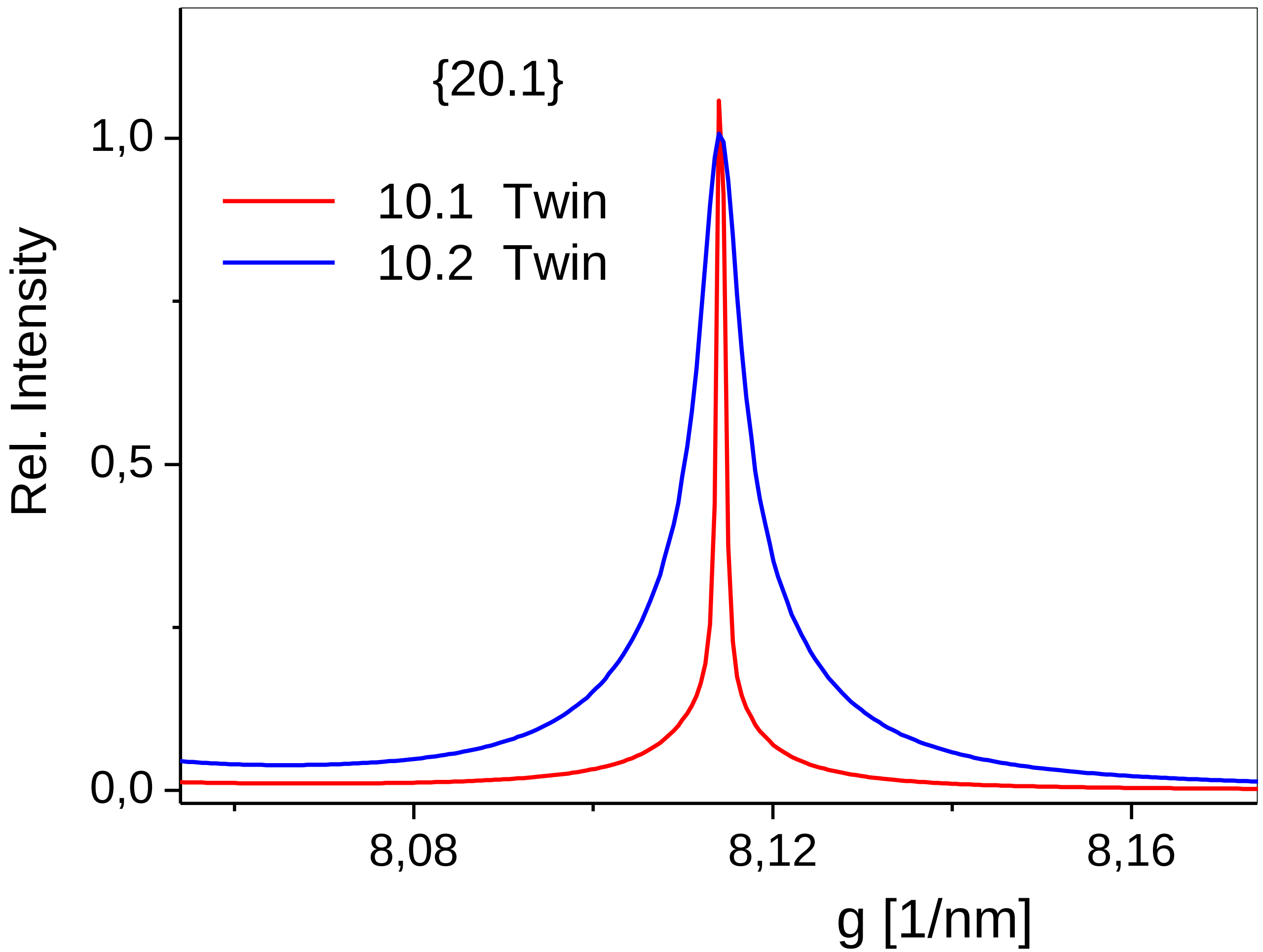
Two different twin planes: {10.1} and {10.2}

{11.4}, {20.1}, {21.0} reflection



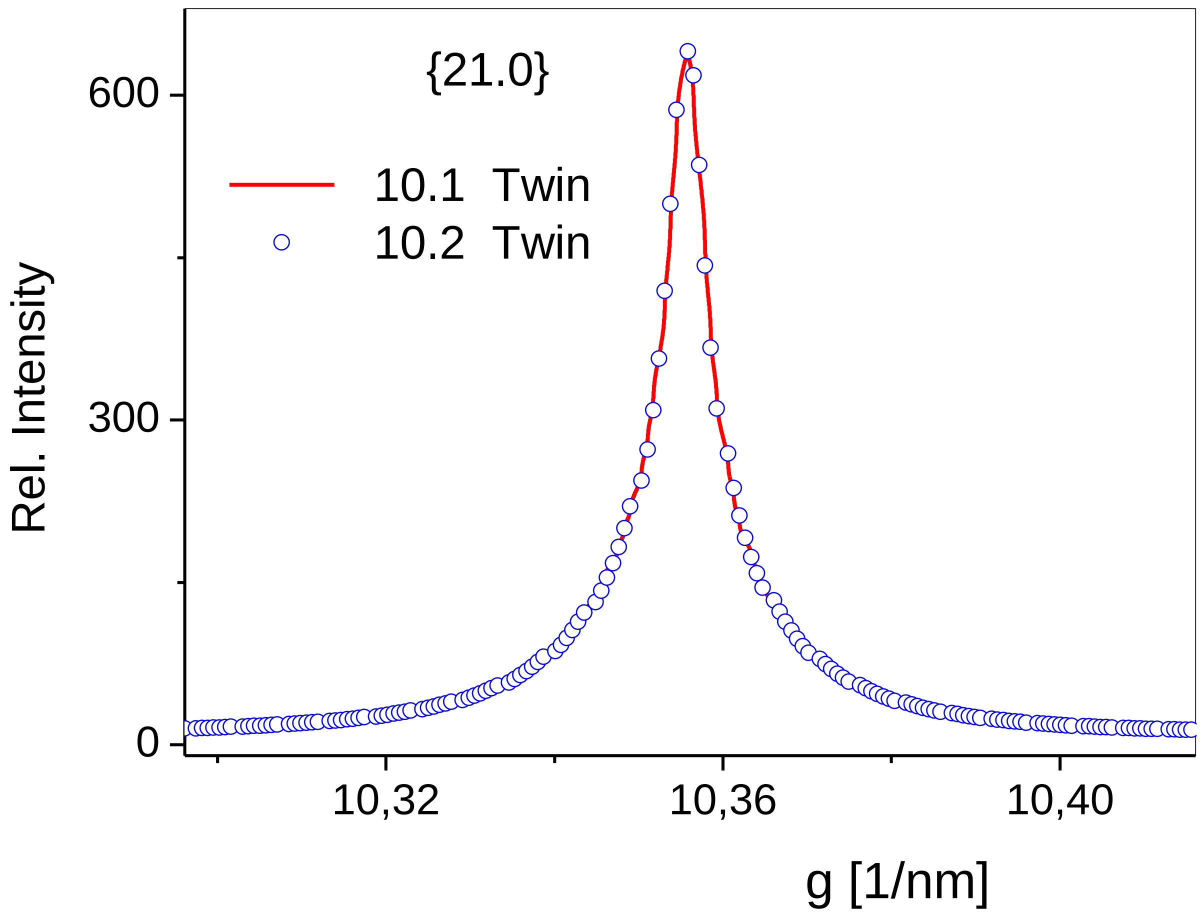
Two different twin planes: {10.1} and {10.2}

{11.4}, {20.1}, {21.0} reflection



Two different twin planes: {10.1} and {10.2}

{11.4}, {20.1}, {21.0} reflection



Philosophy of line profile analysis

$$I_{meas} \Leftrightarrow I_{size} * I_{strain} * I_{PF} * I_{instr} + BG$$

Measured pattern

Physically modeled

Experimentally determined

theoretical pattern: $I_{th}(g)$

Profile-functions for planar faults: $I_{hkl}^{PF}(g)$

$$I_{hkl}^{PF}(g) = w_\delta I_{hkl}^{\delta}(g) + \sum_{j=1}^{n} w_L^j I_{L,hkl}^{j}(g)$$

δ functions

sub-profiles

w: fractions, (~ permutations of *hkl*)

Sub-profiles:

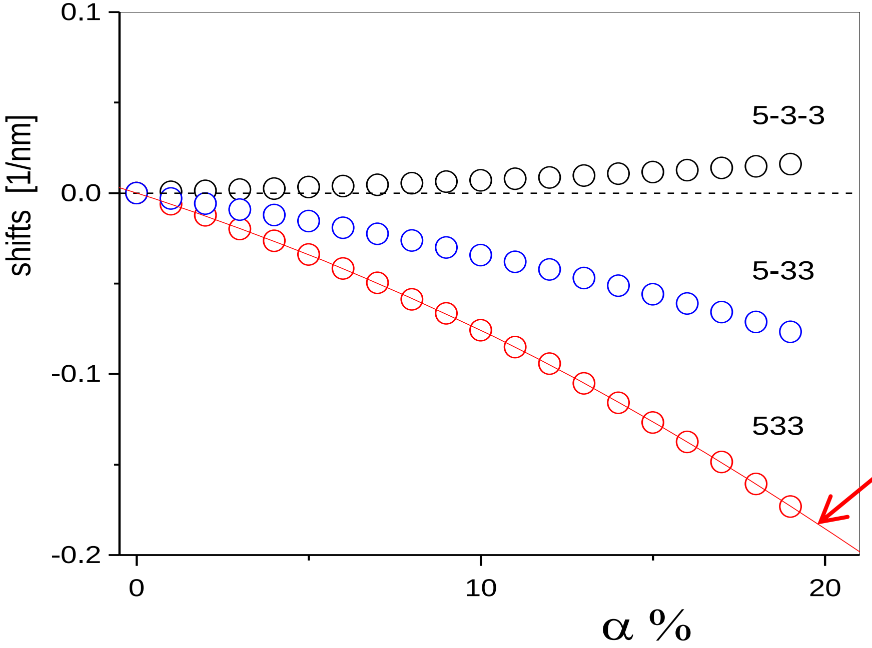
- symmetrical + antisymmetrical

  Lorentzian functions

- easy to parameterize

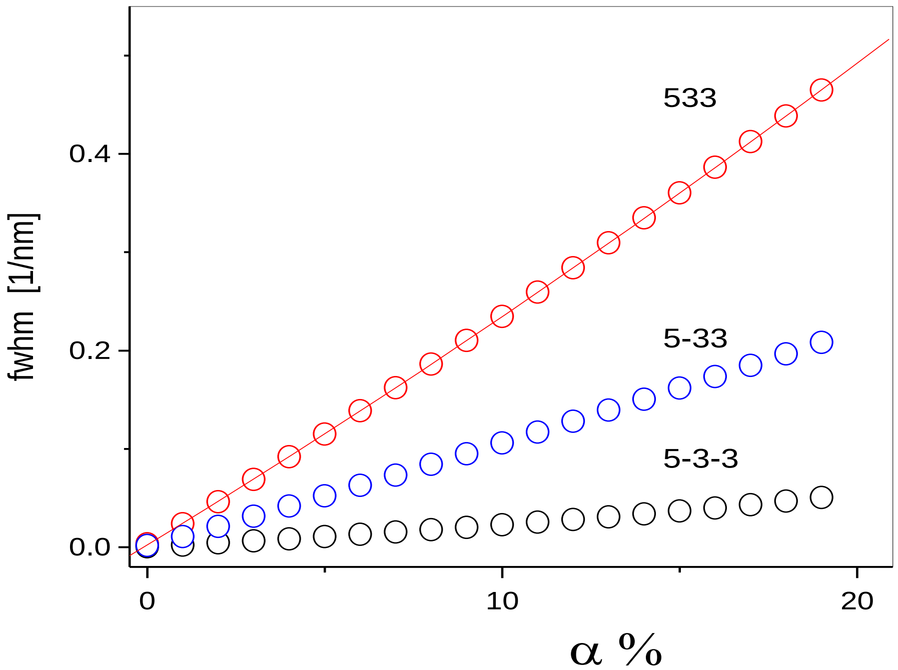
Sub-reflections of the {533} reflection for intrinsic SF

Shifts



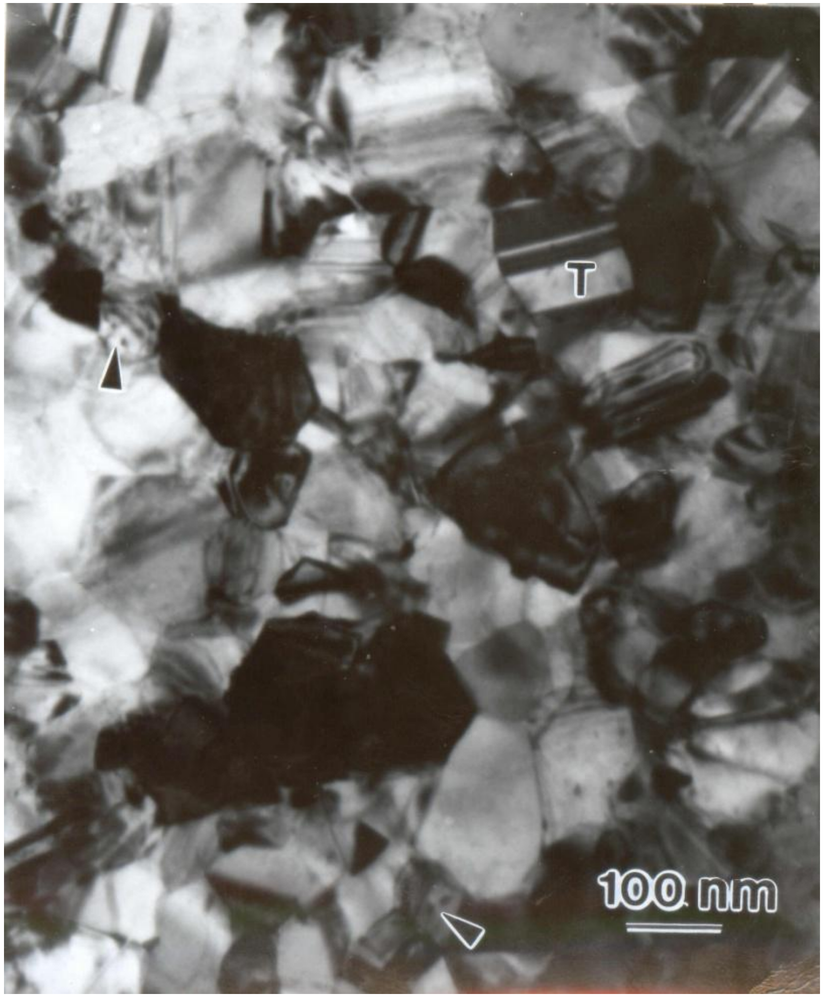
5th order polinomials

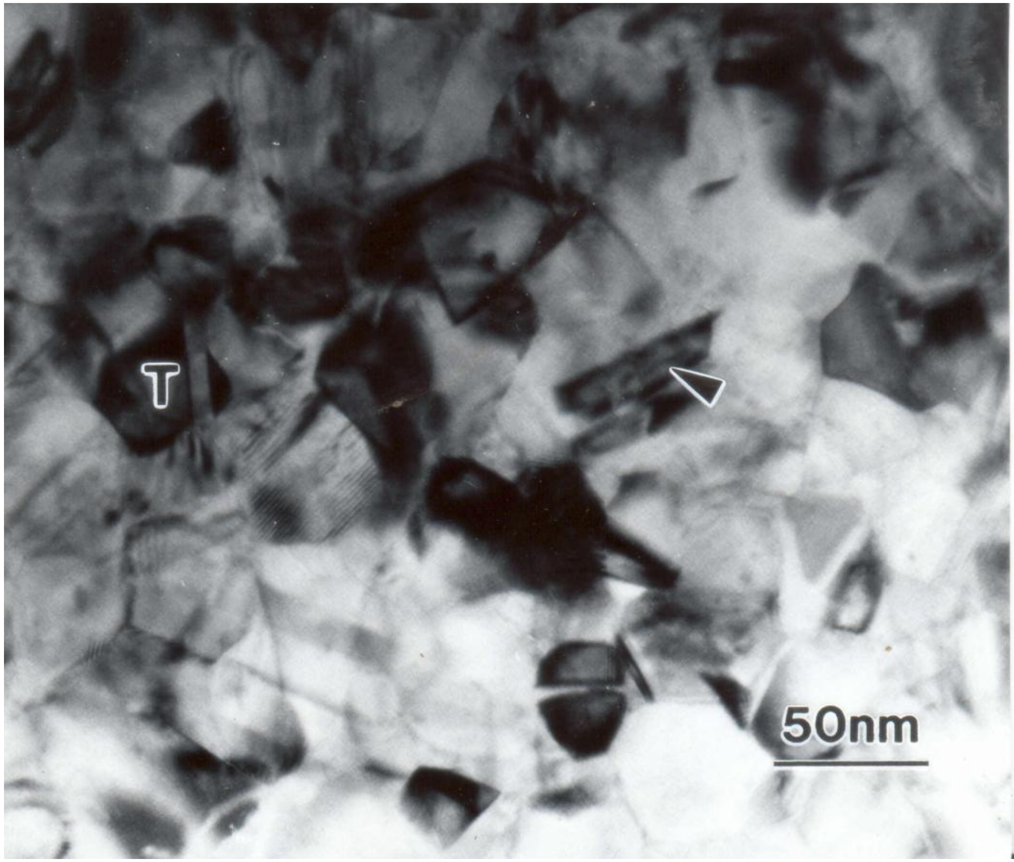
Breadths



Inert-Gas condensed nanocrystalline copper

G. Sanders, G. E. Fougere, L. J. Thompson, J. A. Eastman, J. R. Weertman, *Nanostruct. Mater.* 8, (1997) 243.

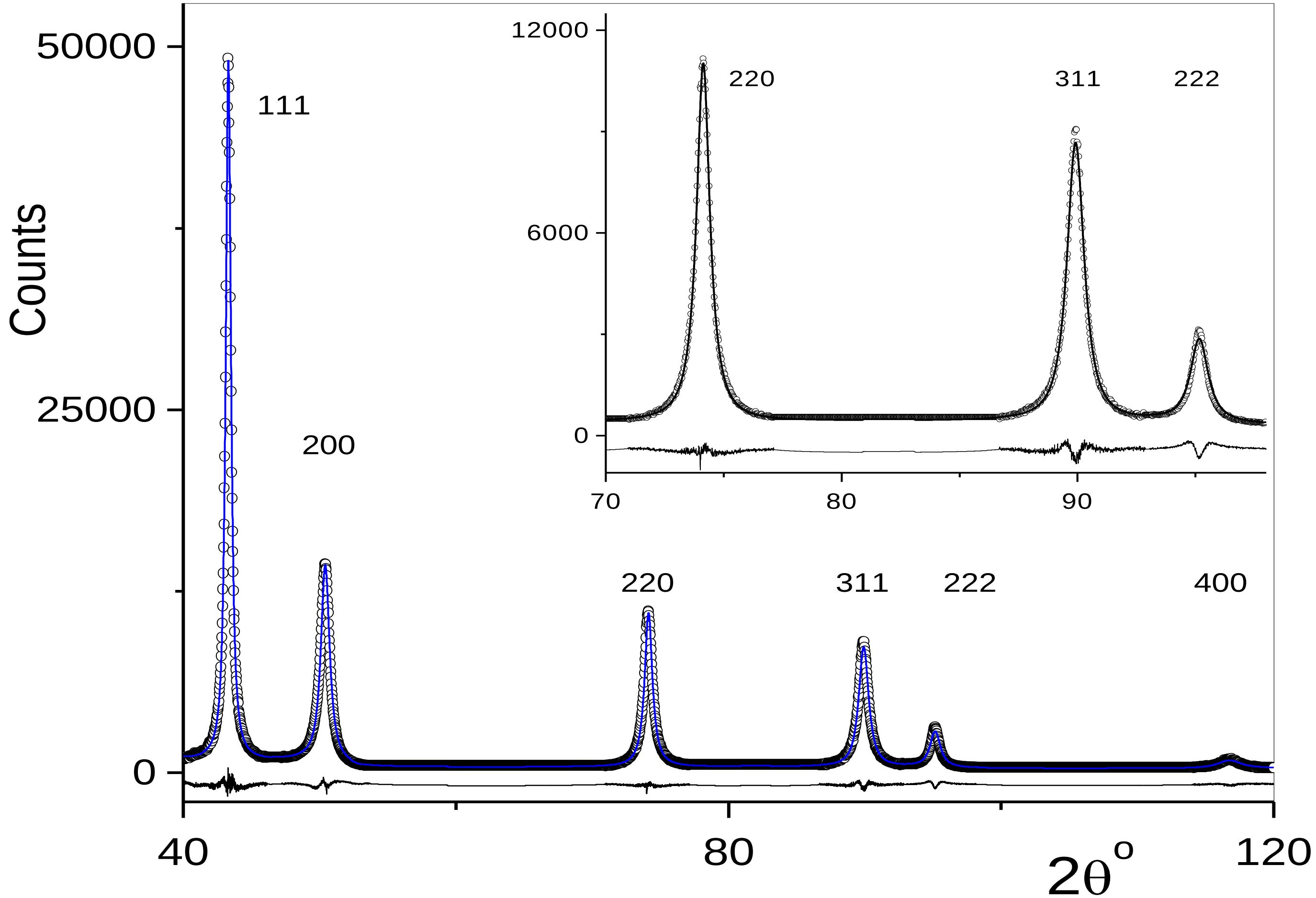




Inert-Gas condensed nanocrystalline copper

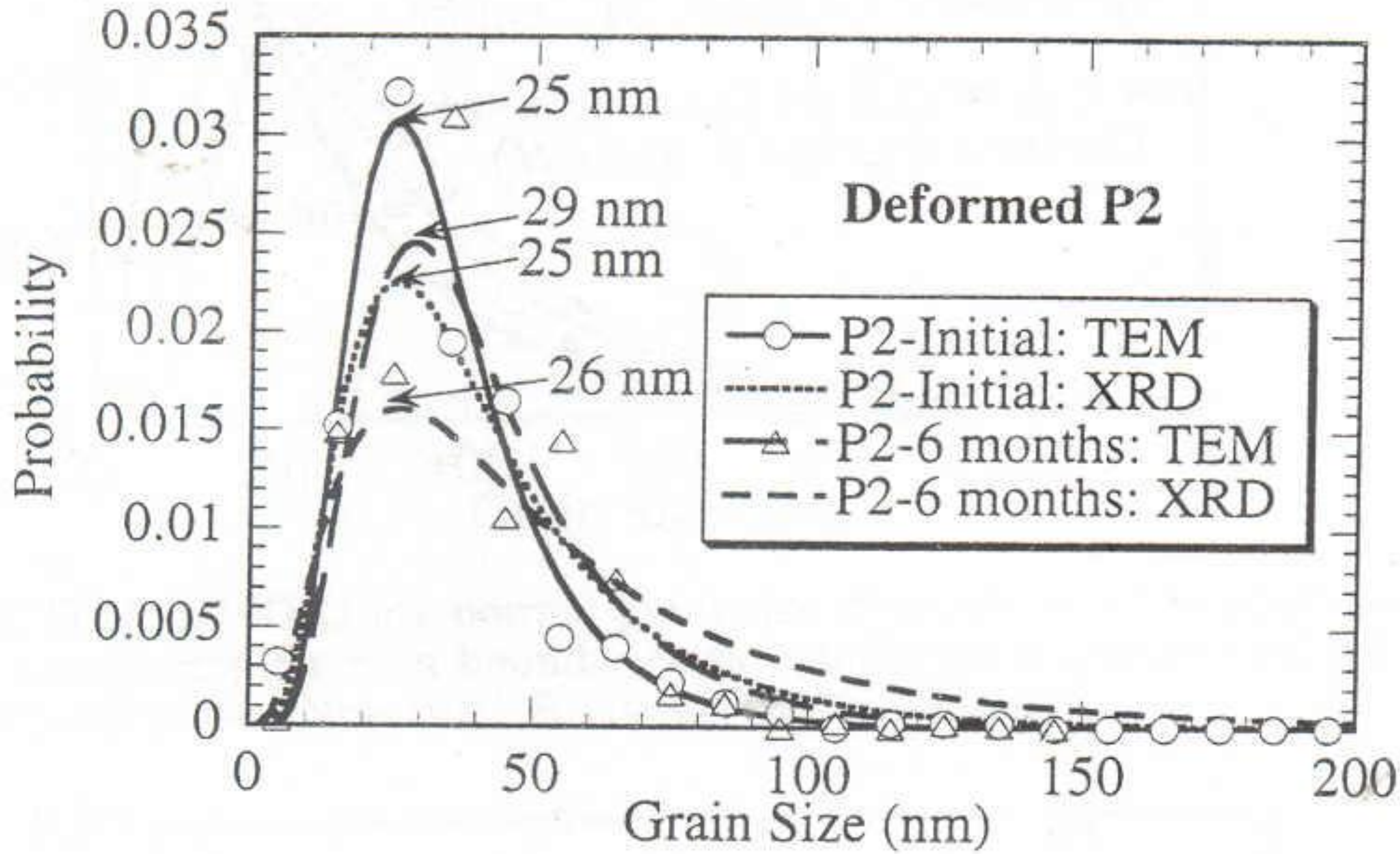
T.Ungár, S.Ott, P.G.Sanders, A.Borbély, J.R.Weertman, *Acta Materialia*, ***10***, *3693-3699 (1998)*

L. Balogh, G. Ribárik, T Ungár, *J.Appl.Phys.* ***100*** *(2006) 023512*



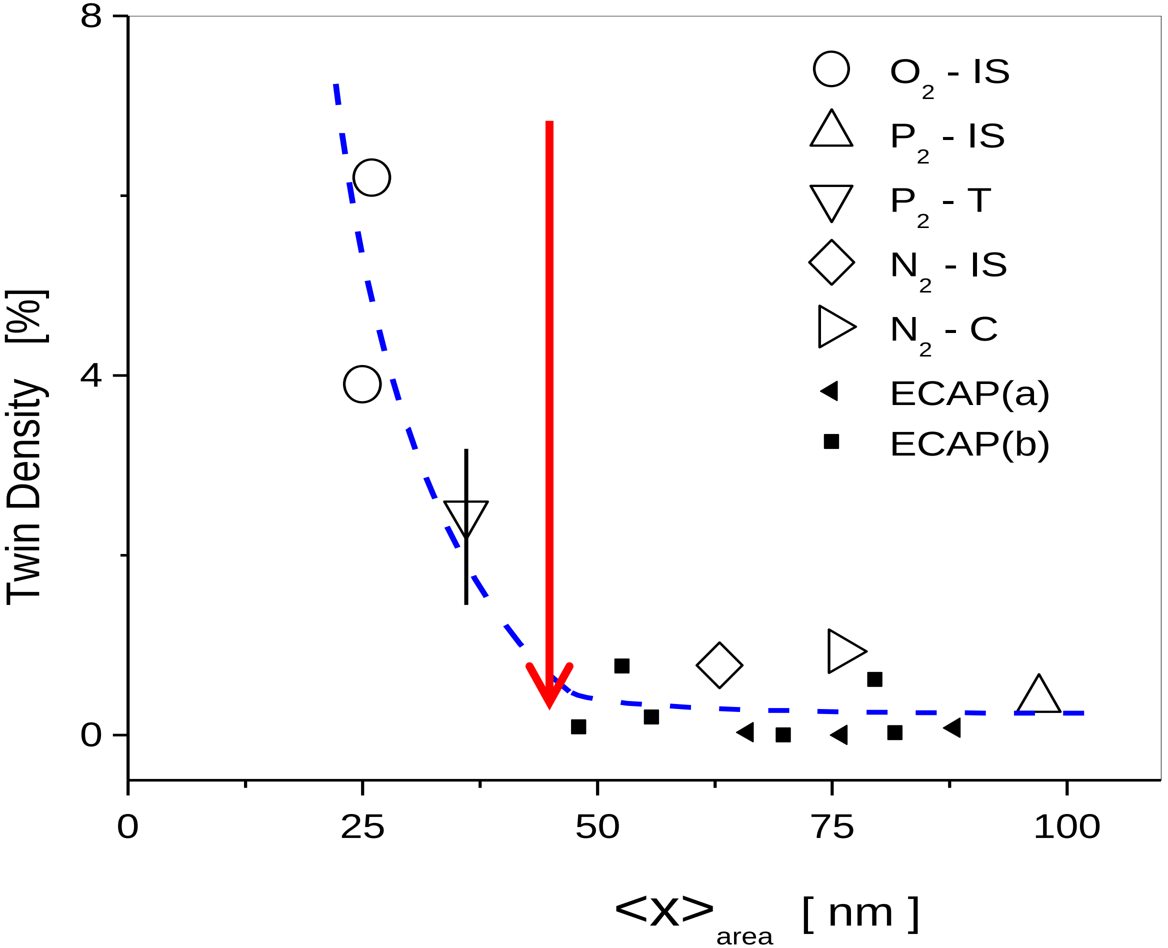
eCMWP

TEM and X-ray grain size



Twin density *vs*. crystallite size: in Cu

L. Balogh, G. Ribárik, T Ungár, *J.Appl.Phys*. 100 (2006) 023512



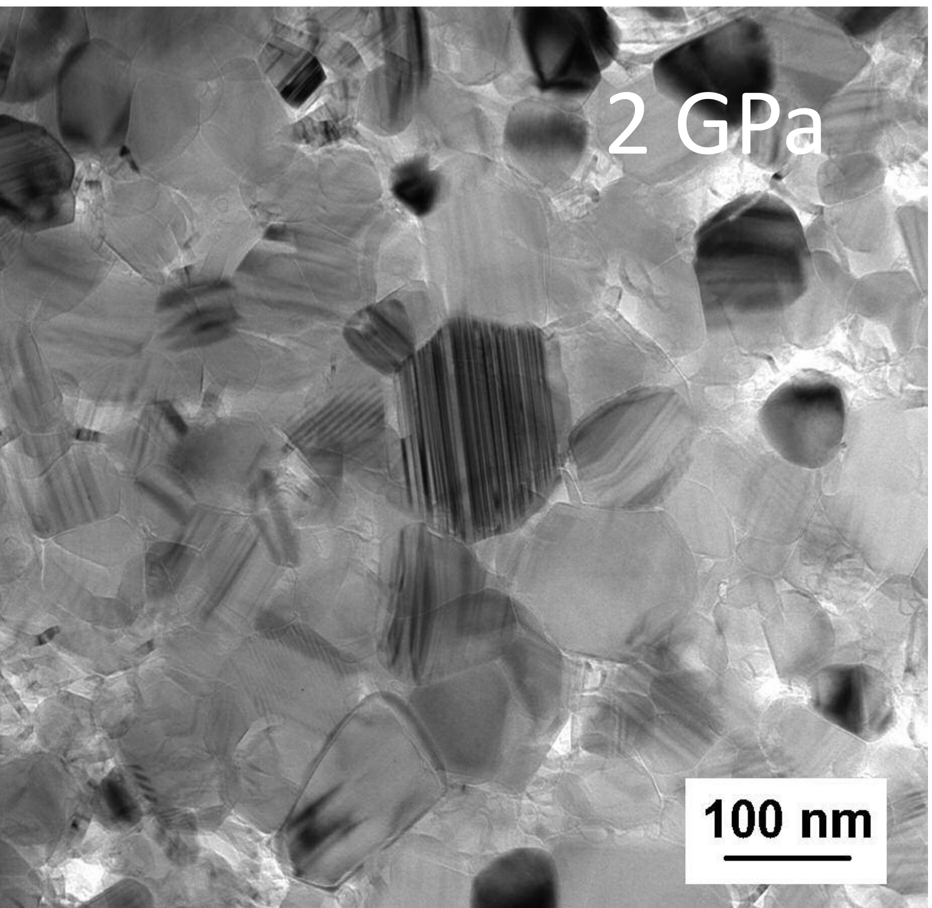
Twinning in Cu:

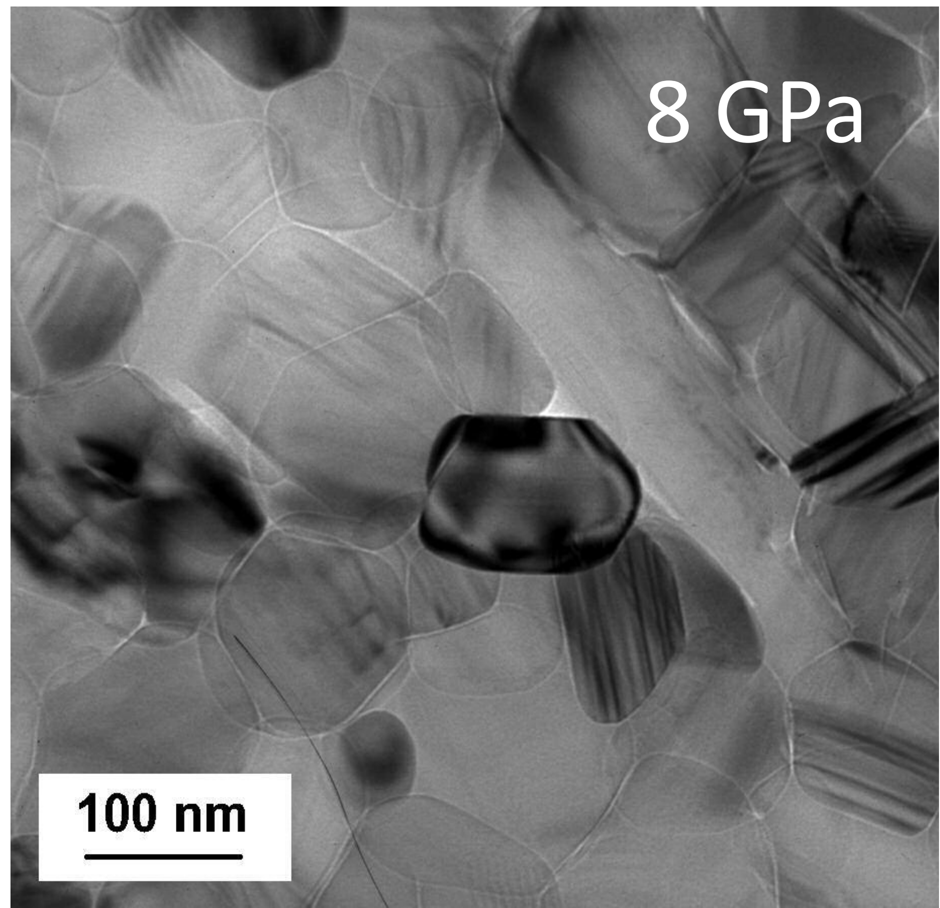
below ~ 40 nm

Sintering nanocrystalline SiC

J. Gubicza, S. Nauyoks, L. Balogh, J. Lábár, T. W. Zerda, T. Ungár, *J. Mater. Res.* **22** (2007) 1314

T_{sinter} 1800 °C

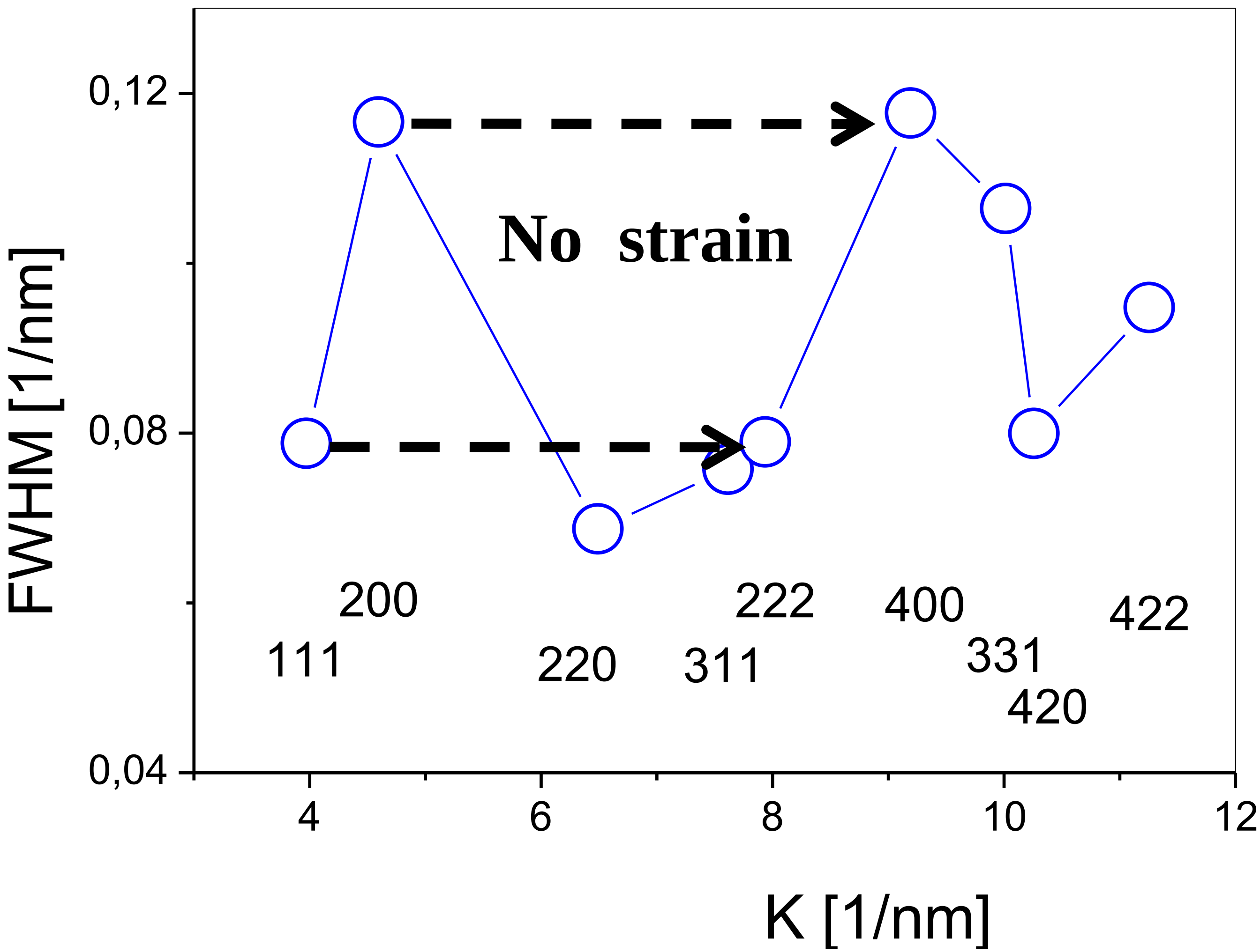




Sintering nanocrystalline SiC

J. Gubicza, S. Nauyoks, L. Balogh, J. Lábár, T. W. Zerda, T. Ungár, *J. Mater. Res.* **22** (2007) 1314

1800 ^{o}C at **2** GPa

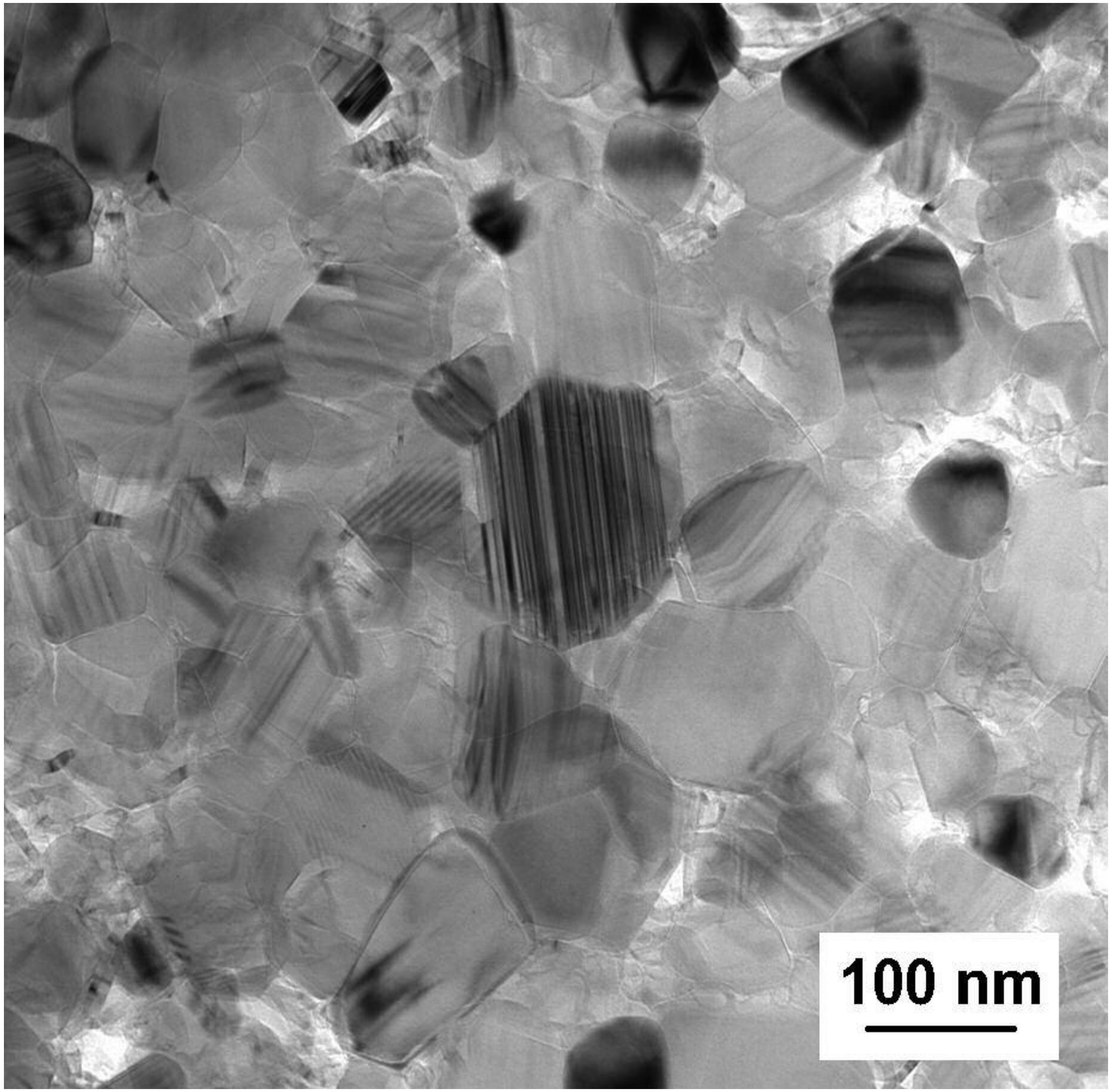


Sintering nanocrystalline SiC

J. Gubicza, S. Nauyoks, L. Balogh, J. Lábár, T. W. Zerda, T. Ungár, *J. Mater. Res.* **22** (2007) 1314

1800 °C at
2 GPa

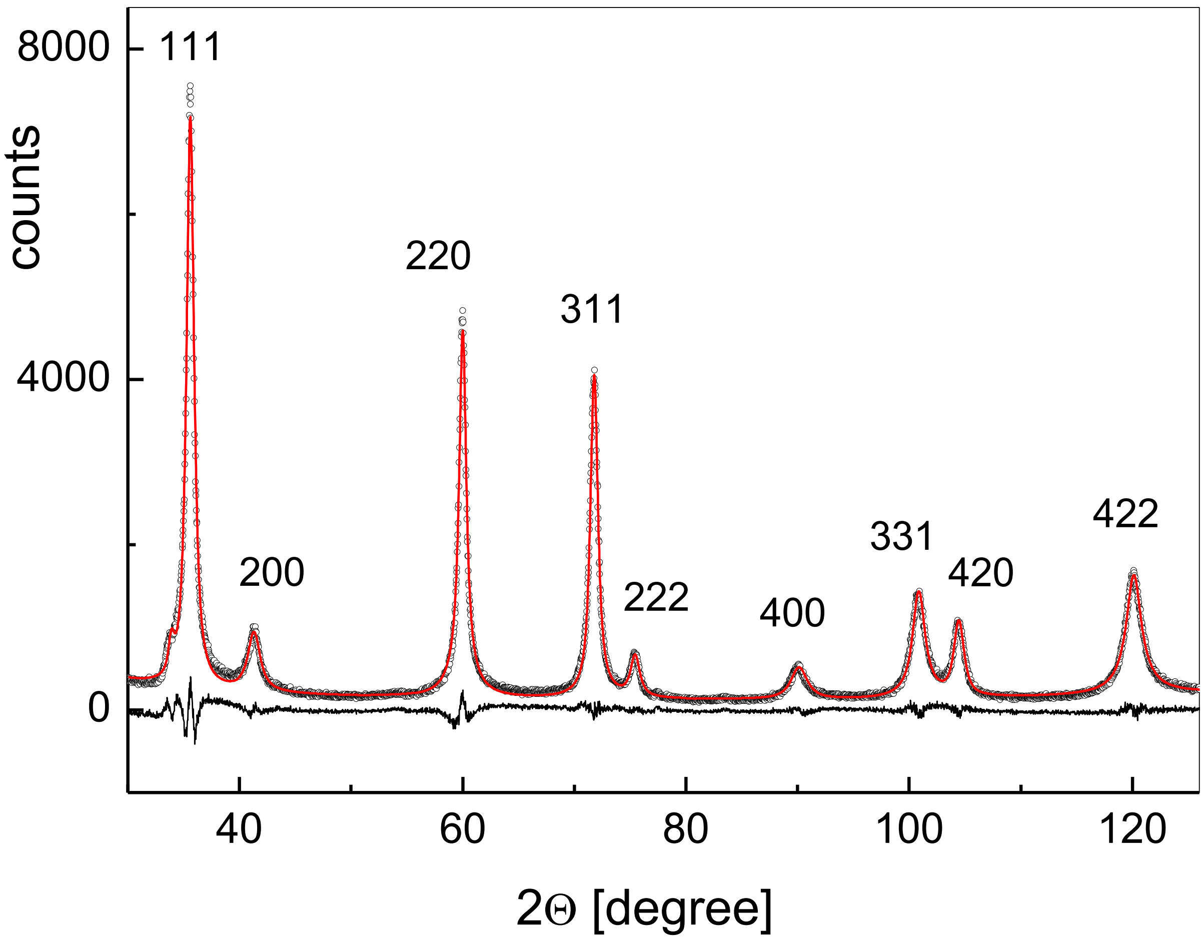
Equiaxed grains
with
twin boundaries



Sintering nanocrystalline SiC

J. Gubicza, S. Nauyoks, L. Balogh, J. Lábár, T. W. Zerda, T. Ungár, *J. Mater. Res.* **22** (2007) 1314

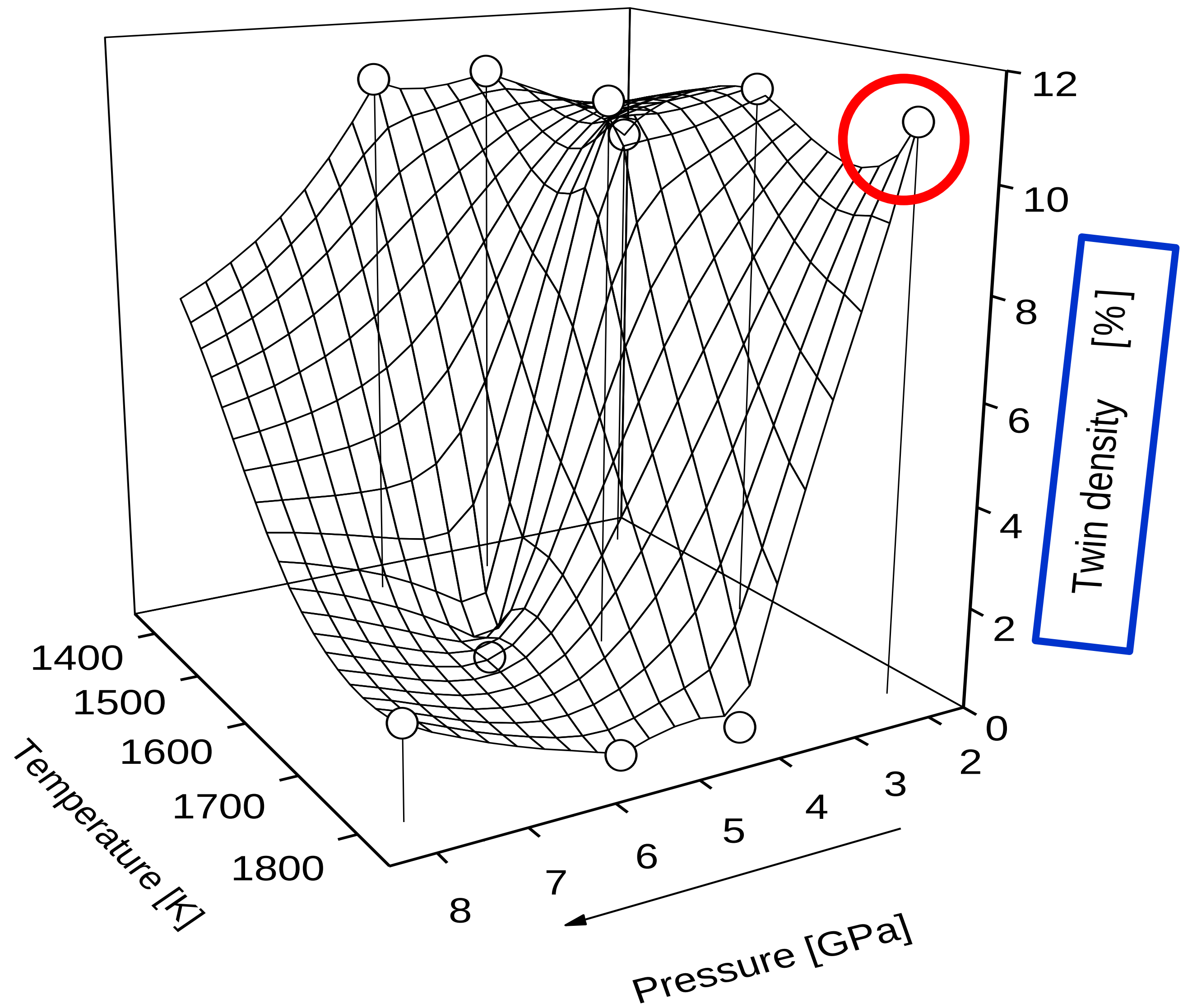
1800 °C at
2 GPa



eCMWP
procedure

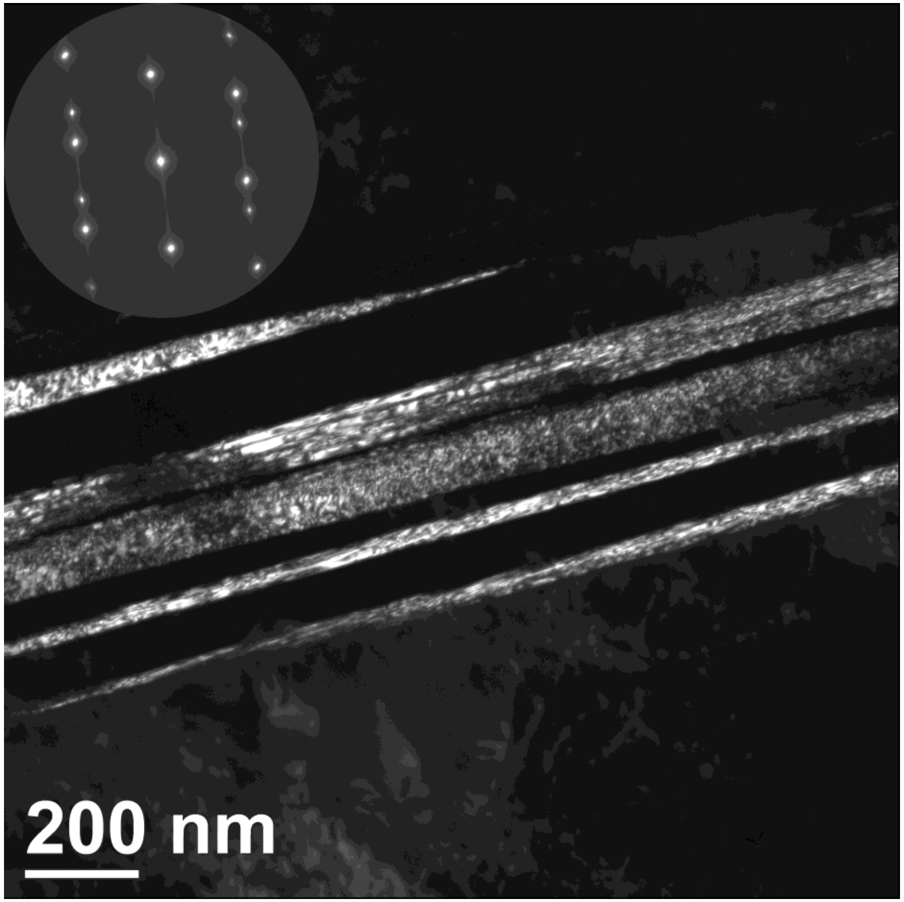
Sintering nanocrystalline SiC

J. Gubicza, S. Nauyoks, L. Balogh, J. Lábár, T. W. Zerda, T. Ungár, *J. Mater. Res.* **22** (2007) 1314

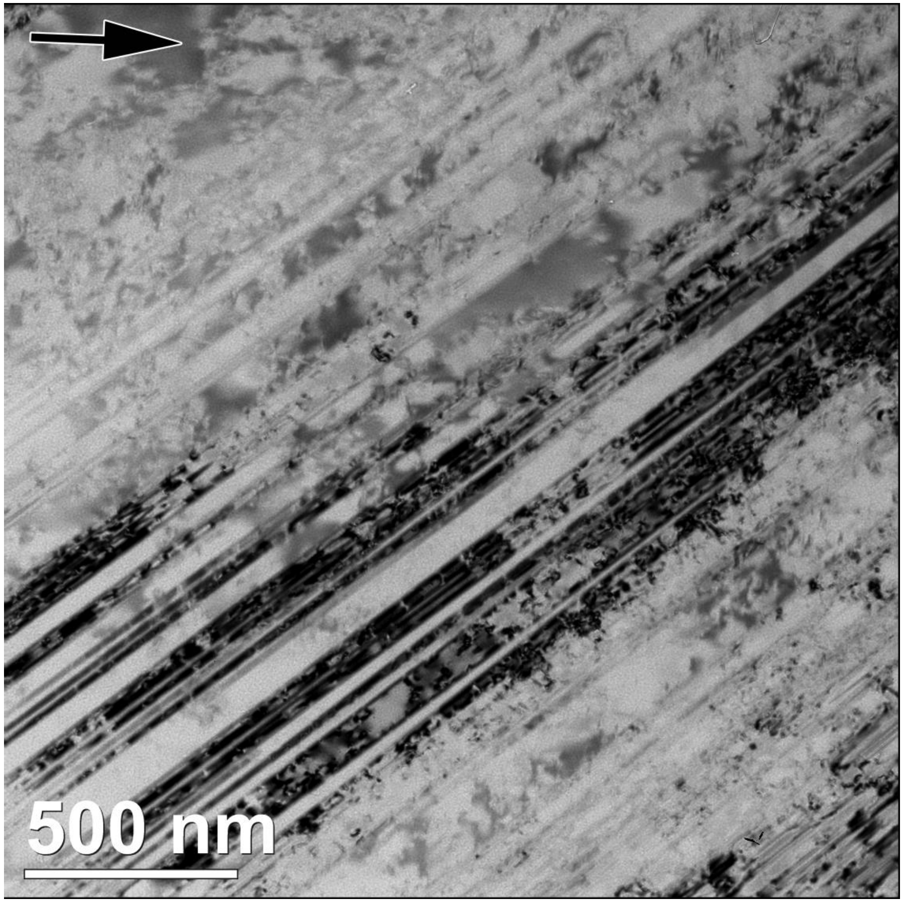


Twinning in tensile deformed TWIP steel

L. Balogh, D.W. Brown, T. Holden, in preparation



$\varepsilon_{eng} = 33\%$



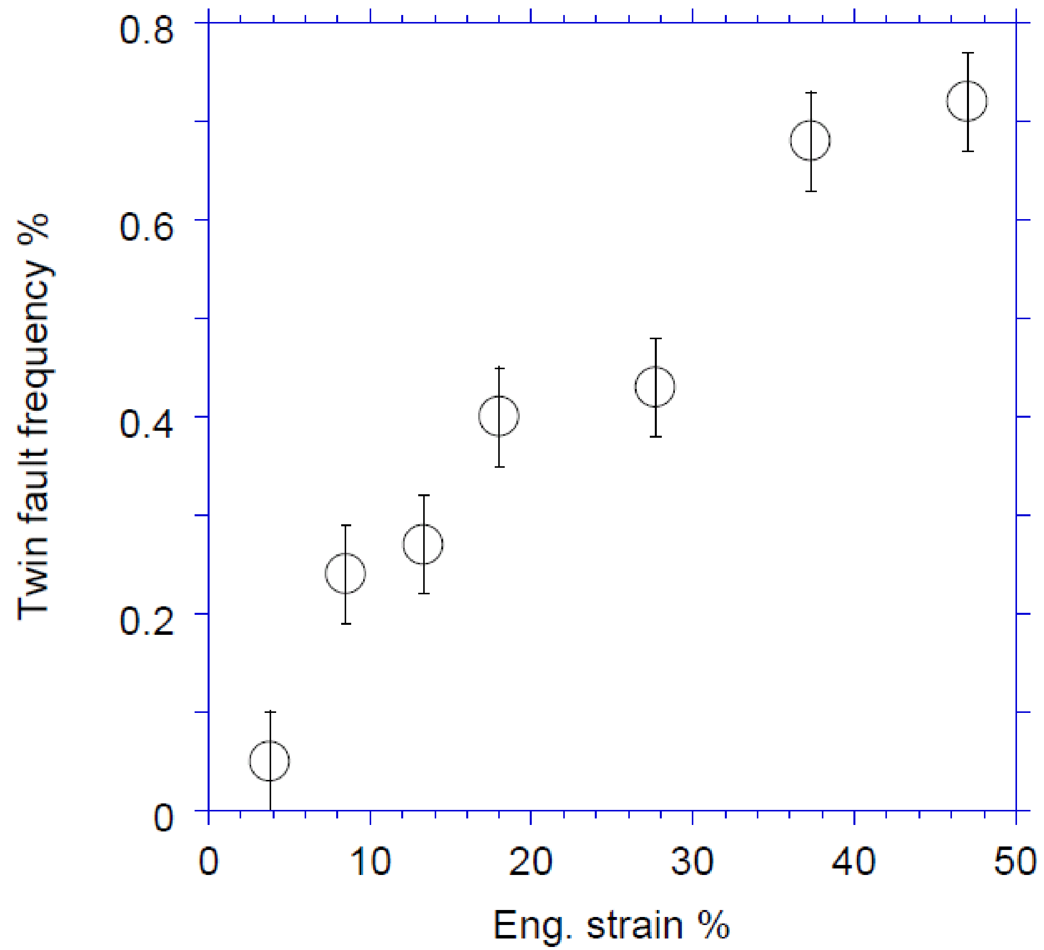
$\varepsilon_{eng} = 42\%$

Twinning in tensile deformed TWIP steel

L. Balogh, D.W. Brown, T. Holden, in preparation

eCMWP

procedure



TOF neutron diffraction
at SMARTS
Lujan Neutron Scatt. Cent.
Los Alamos Nat. Lab.

Access to the software package

- CMWP
- eCMWP
- ANIZC
- planar-defect parameter-files

http://www.renyi.hu/cmwp

http://metal.elte.hu/anizc/

http://metal.elte.hu/~levente/stacking

Thanks to my coworkers:

Levente Balogh

Gábor Ribárik

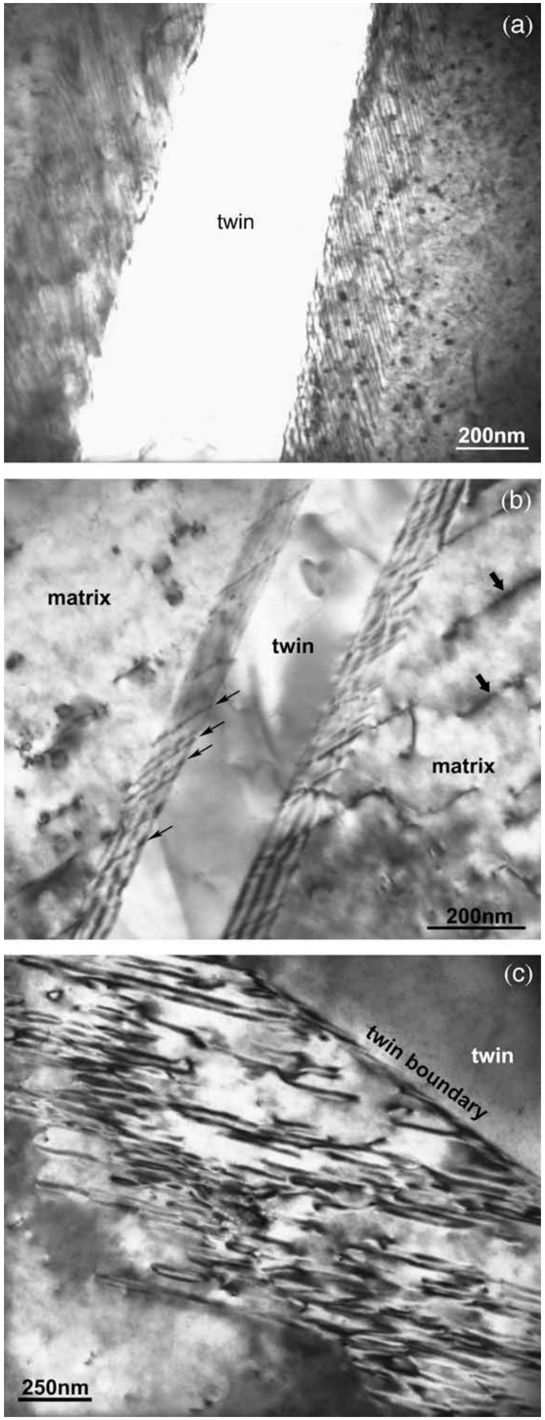
Géza Tichy

Thank you

for your

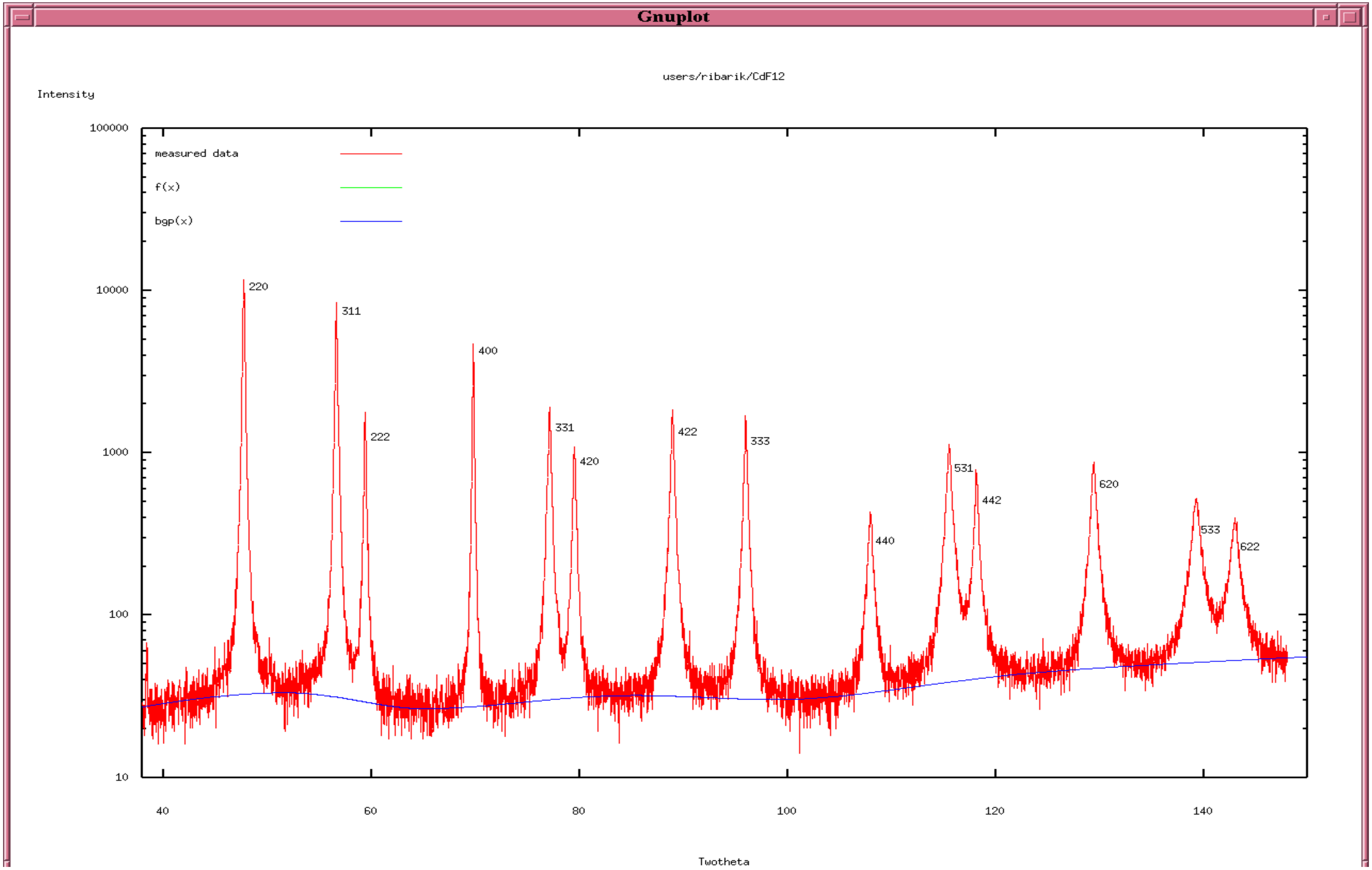
attention

Dislocations in the pyramidal twin boundaries in Mg
B. Li, E. Ma, AM 2004



CdF_2 ball milled for 12 min

G. Ribárik, N. Audebrand, H. Palancher, T. Ungár and D. Louër, *J. Appl. Cryst.* (2005). 38, 912–926



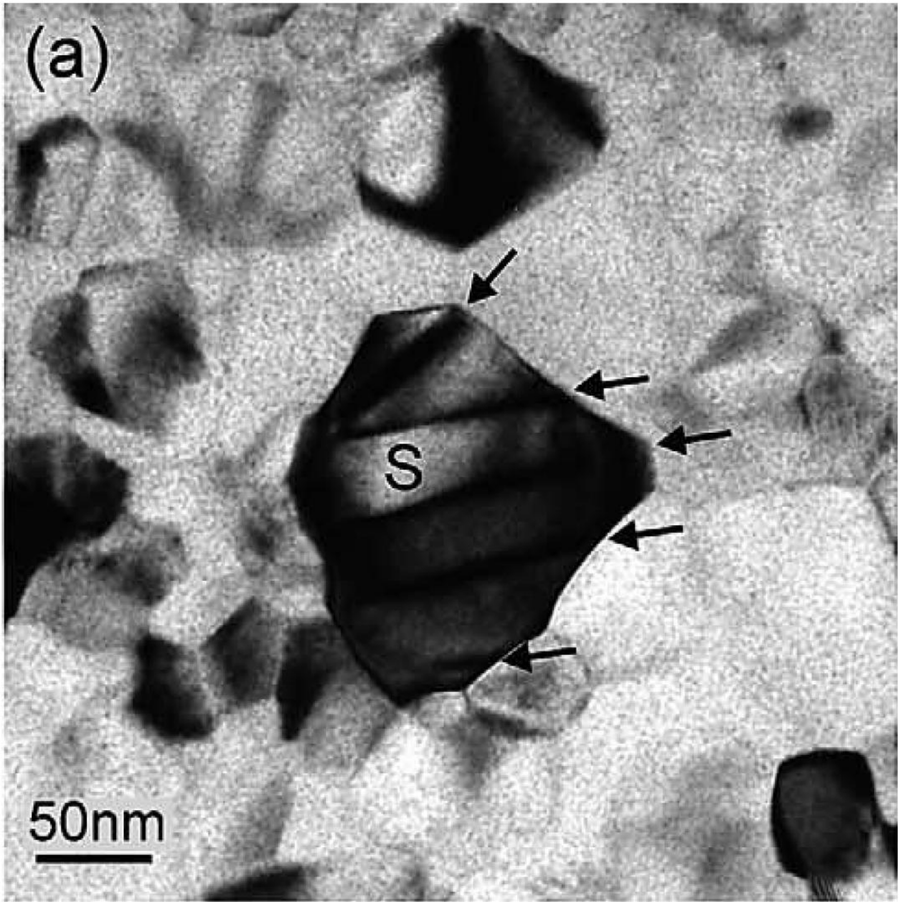
T. Waitz, V. Kazykhanov, H.P. Karnthaler:

Martensitic phase transformations in nanocrystalline **NiTi** studied by TEM

Acta Materialia 52 (2004) 137–147

Perfect matching along the twin plane

Bright field image of a **twinned grain**.



HRTEM image of a grain containing two **twin related variants**: RT1 & RT2