

# Slip-Modes, Slip-Systems and Dislocation Densities in Individual Grains of Polycrystalline CoTi and CoZr

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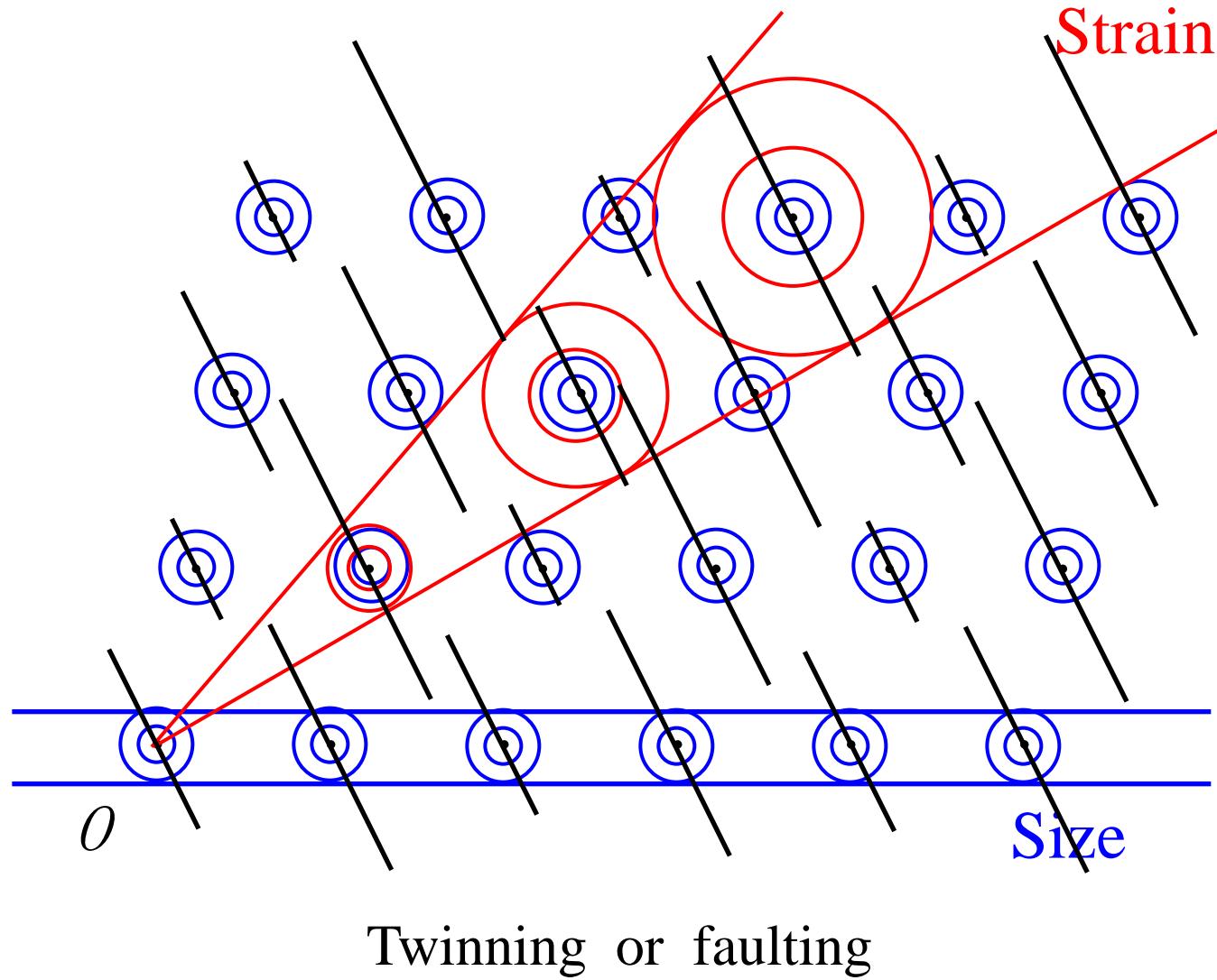
Tamás Ungár

Department of Materials Physics, Eötvös University Budapest, Hungary

Seminar Lecture, 13 February, 2015  
Indian Institute of Science, Bangalore, India

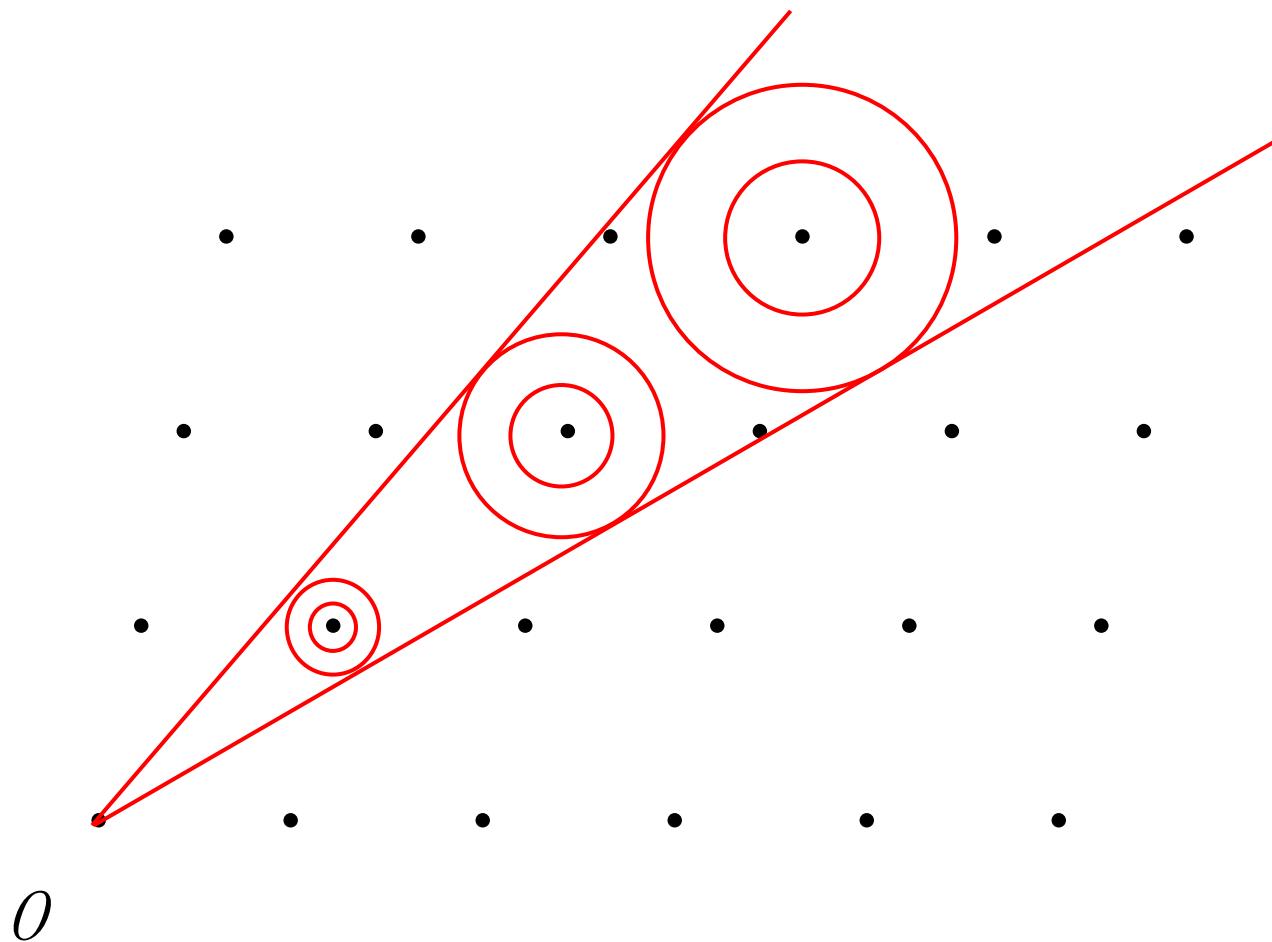
# Schematics of line-broadening

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# The manifestation of strain-broadening:

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broadening **increases** with the **distance** from the  
**origin** of reciprocal space

# the dislocation theorem of strain broadening

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- Stokes & Wilson: A. J. C., Proc. Camb. Phil. Soc., 38 (1942) 313  
Krivoglaz & Ryaboshapka: Phy.Met.Metallov., 15 (1963) 18-31.  
Wilkens: phys.stat.sol., 2 (1962) 692-712.  
Gaál: Acta Phys. Acad. Sci. Hung. 33 (1973) 411-418.  
Groma, Ungár, Wilkens: J. Appl. Cryst. 21 (1988) 47-53.  
Kužel, Klimanek, J. Appl. Cryst., 22 (1989) 299-307.  
Groma: Phys. Rev. B, 57 (1998) 7535–42.  
Barabash: Mater. Sci. Eng. A, 309 (2001) pp. 49–54.

# The hierarchy of non-size-type lattice defects

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Krivoglaz: the spatial dependence of strain:  $\varepsilon(r)$

**0** dimensional: point defects  
point-defect-type, e.g. precipitates  
inclusions

$$\varepsilon(\mathbf{r}) \sim 1/r^2$$

---

**1** dimensional: **dislocations**  
non-equilibrium triple-junctions  
linear-type defects

$$\varepsilon(\mathbf{r}) \sim 1/r$$

---

**2** dimensional: planar defects, e.g. stacking faults  
twin boundaries  
grain boundaries  
domain boundaries

$$\varepsilon(\mathbf{r}) \sim \text{constant}$$

**crystal-space**

*versus*

**reciprocal-space**

**short** distance  
[ m ]



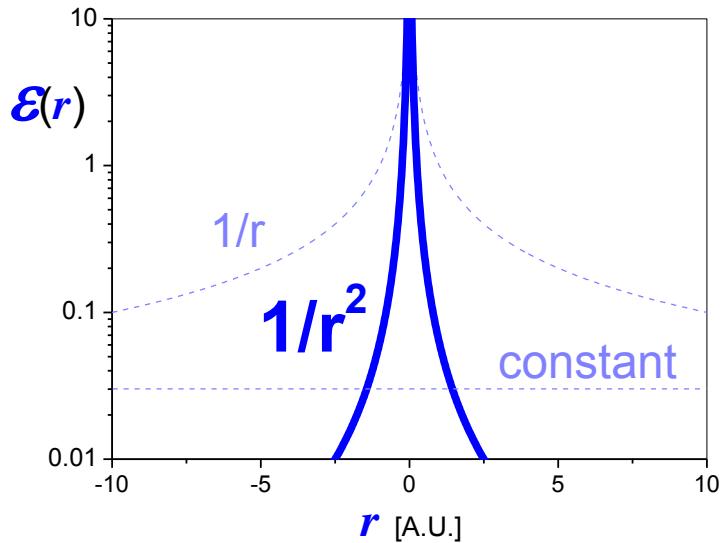
**long** distance  
[ 1/m ]

**long** distance  
[ m ]



**short** distance  
[ 1/m ]

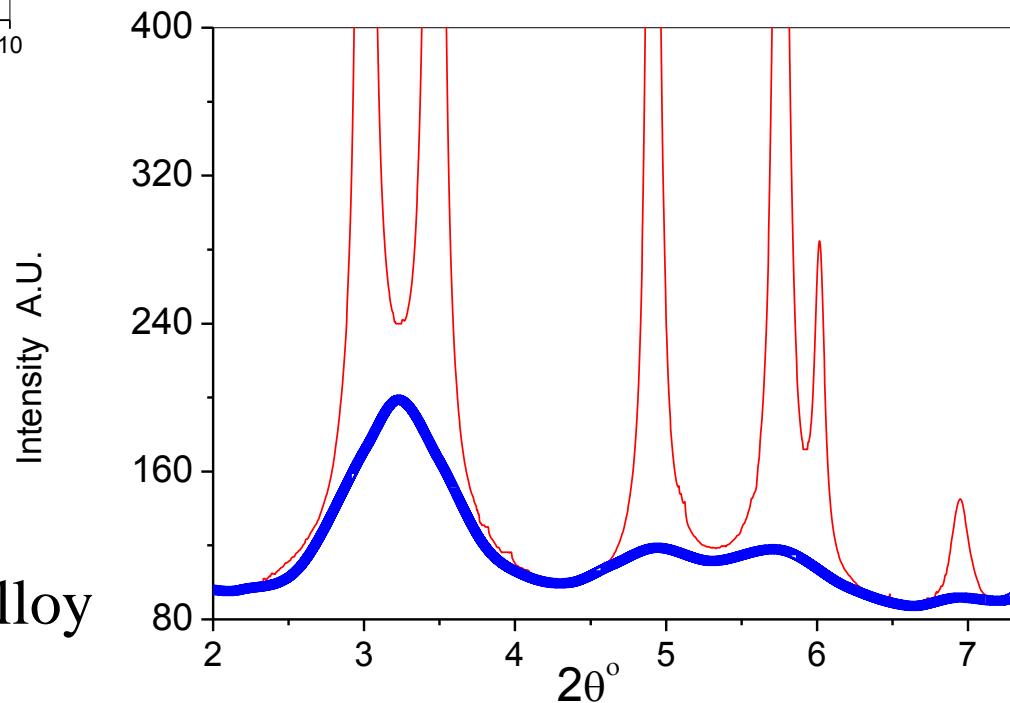
# 0-dimensional: point defects



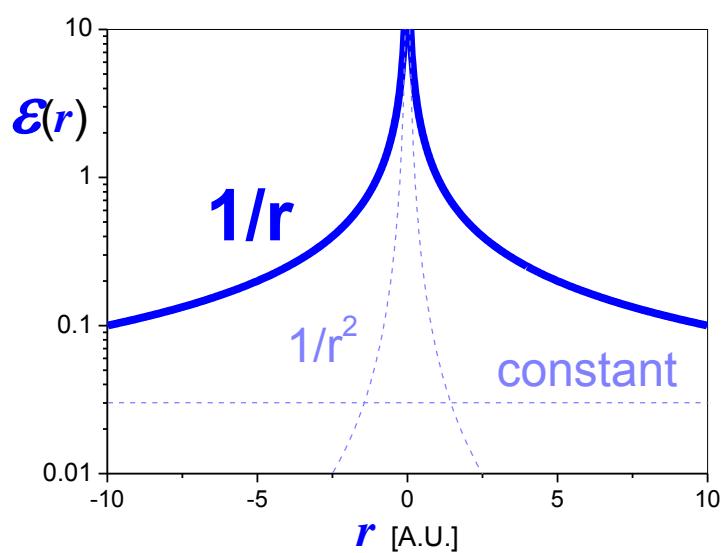
crystal-space

diffraction pattern  
Huang-scattering

Nanocrystalline Ni-18Fe alloy  
L.Li et al. ActaMater.57(2009)4988-5000



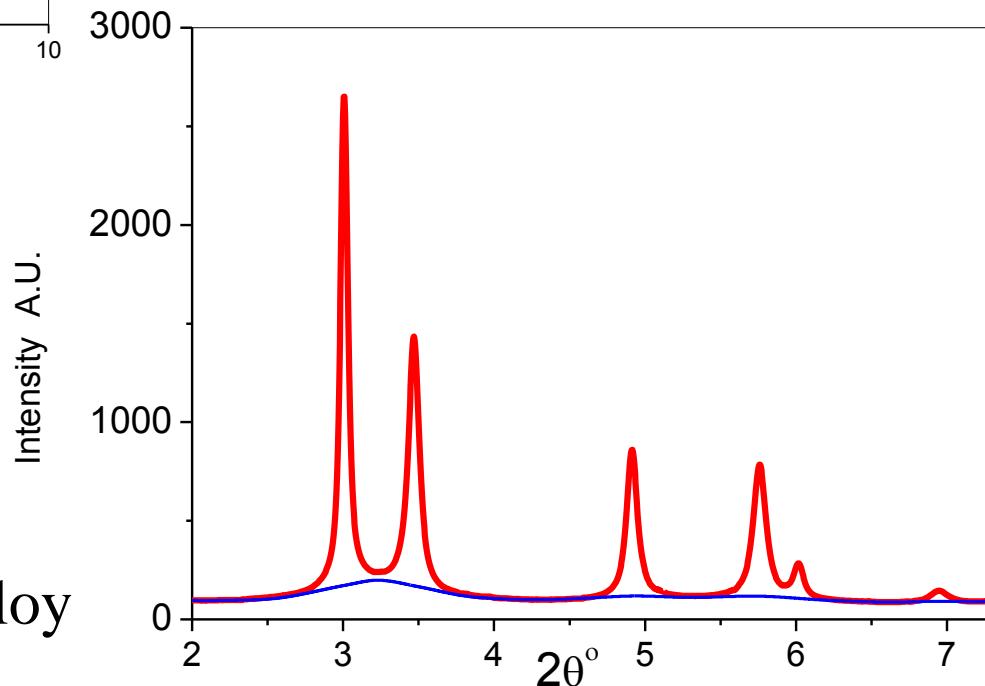
# 1-dimensional: linear defects: *dislocations*



crystal-space

*line-broadening*

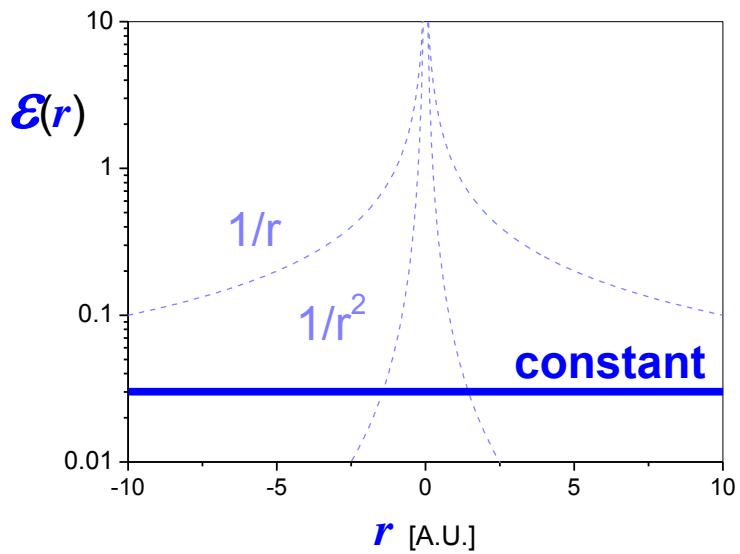
diffraction pattern



Nanocrystalline Ni-18Fe alloy

L.Li et al. ActaMater.57(2009)4988-5000

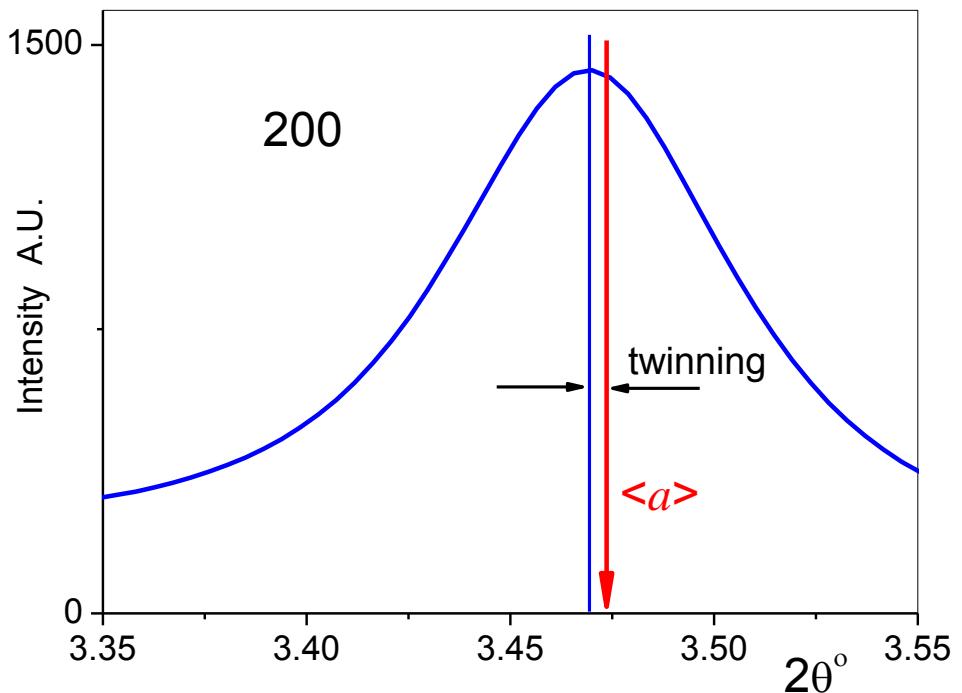
# 1-dimensional: planar defects: *twinning*



crystal-space

*stacking faults*

diffraction pattern  
*shift + broadening*



Nanocrystalline Ni-18Fe alloy

L.Li et al. ActaMater.57(2009)4988-5000

**0** dimensional: point defects  
point-defect-type, e.g. precipitates  
inclusions

$$\varepsilon(r) \sim 1/r^2$$

**1** dimensional: **dislocations**  
non-equilibrium triple-junctions  
linear-type defects

$$\varepsilon(r) \sim 1/r$$

**2 di**

**lattice defects**

**which cause strain broadening**

domain boundaries

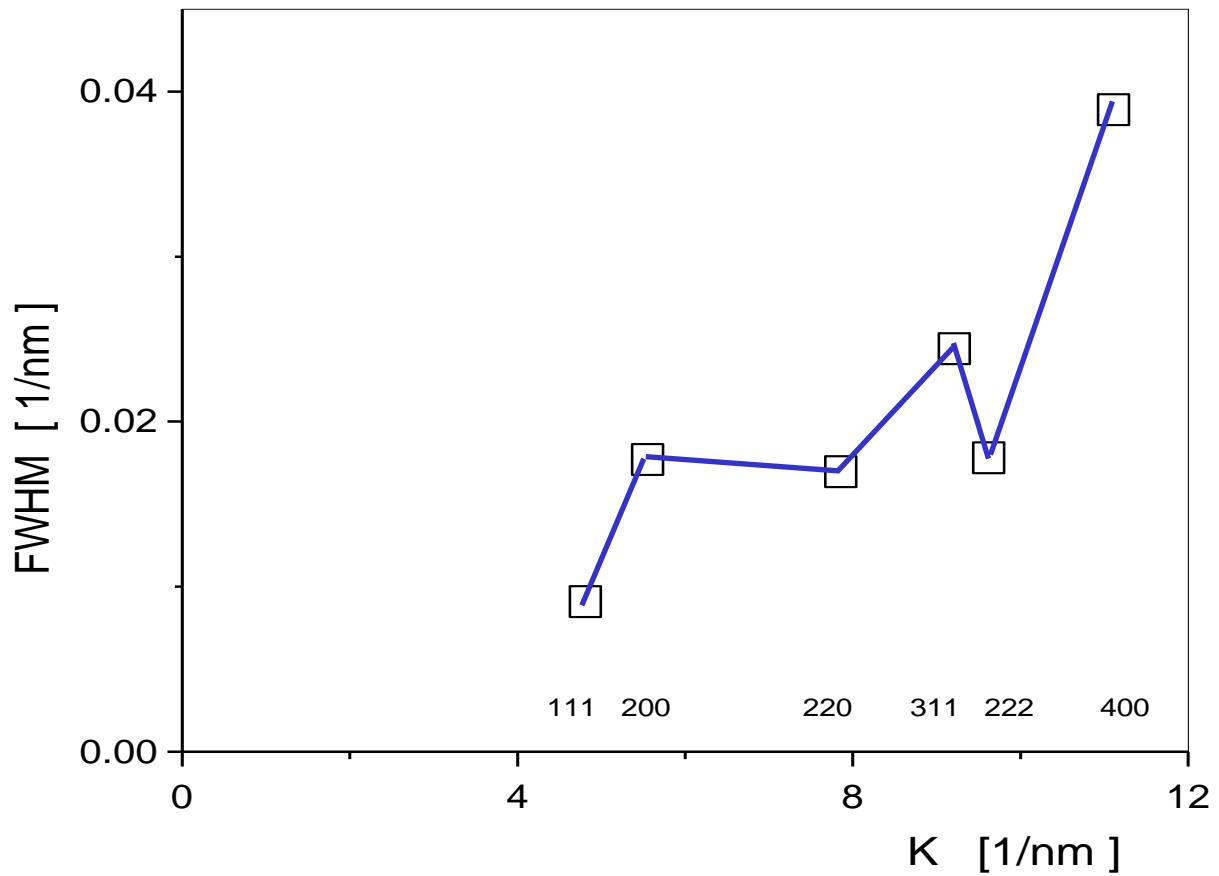
$$\varepsilon(r) \sim \text{constant}$$

*hkl* dependence of line-broadening:  
strain anisotropy

# Line-broadening is $hkl$ dependent

Copper deformed by Equal Channel Angular Pressing (ECAP)

*Williamson-Hall plot*

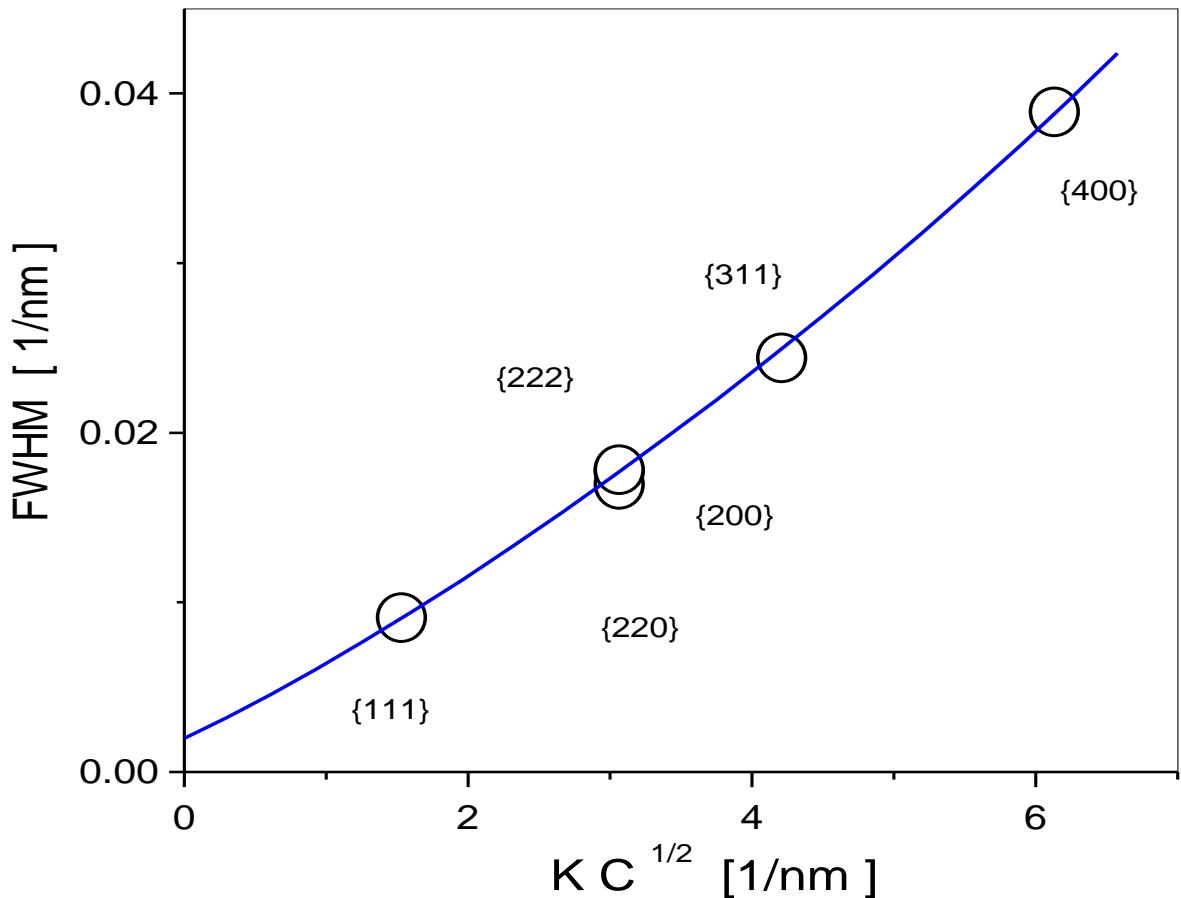


Strain anisotropy

# Line-broadening is $hkl$ dependent

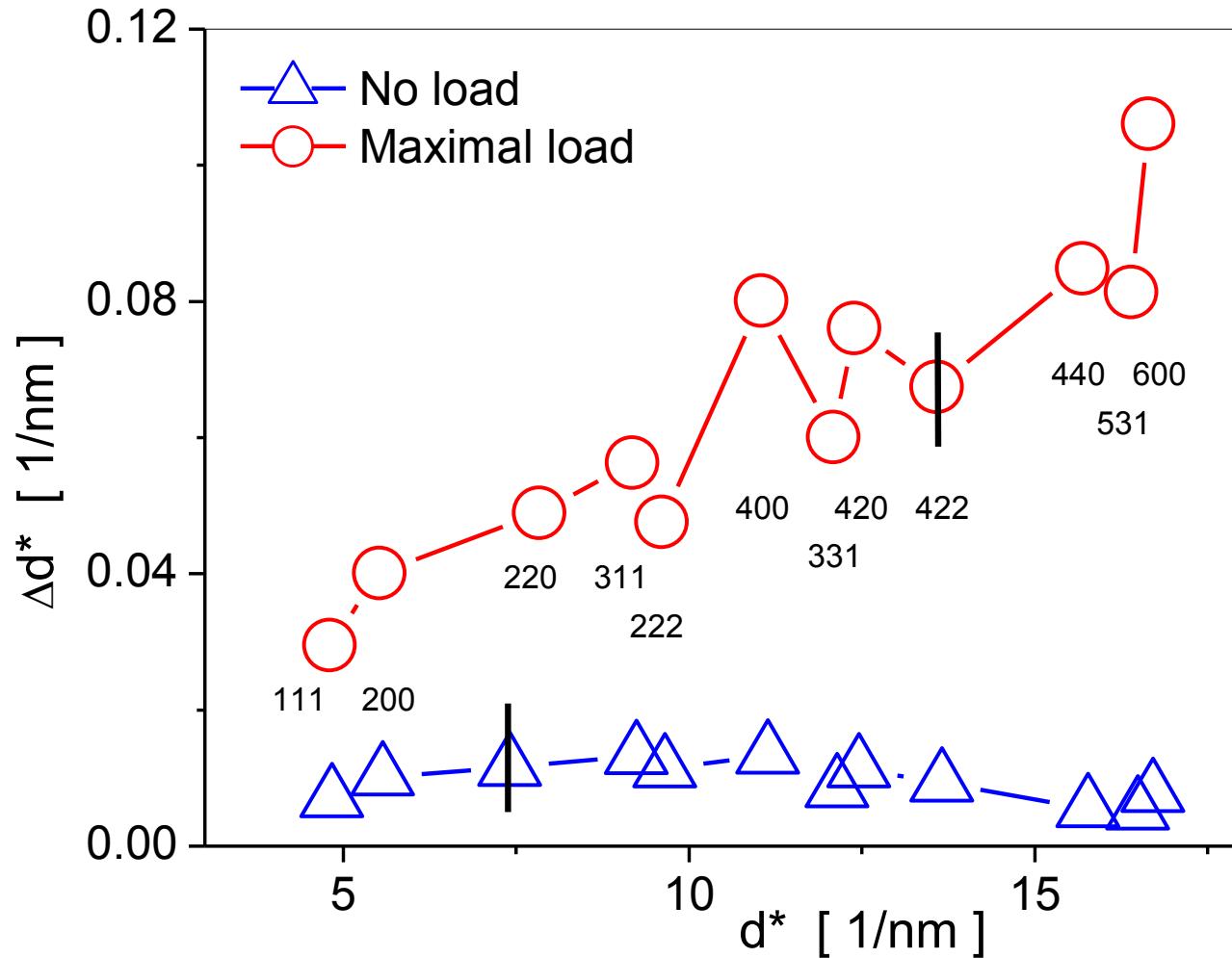
Copper deformed by Equal Channel Angular Pressing (ECAP)

modified Williamson-Hall plot



# Line-broadening is $hkl$ dependent

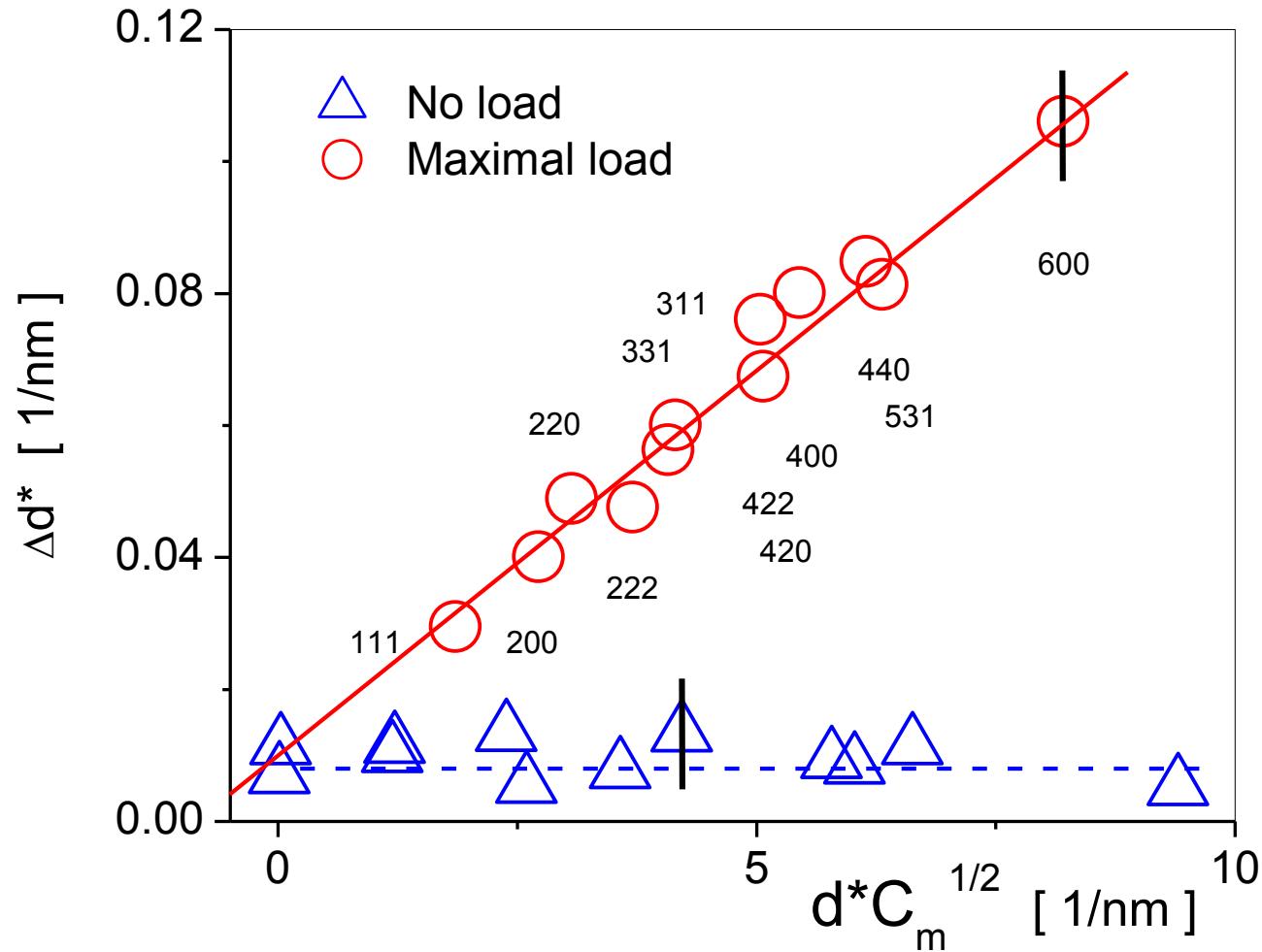
in-situ plastically strained 316-stainless steel



# Dislocation-model of strain-anisotropy

*modified* Williamson-Hall plot

$C_{hkl}$ : contrast of dislocations



line-broadening + strain-anisotropy + peak-shift:

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X-rays and/or neutrons:

dislocation-***density***

- + dislocation ***character***: edge/screw
  - + dislocation ***type***:  $\langle a \rangle$ ,  $\langle c \rangle$  or  $\langle c+a \rangle$
- 

synchrotron:

- + dislocation-***density***
- + + ***slip-systems*** & ***slip-modes***  
on the ***grain-level*** in ***polycrystals***

Slip-Modes, Slip-Systems and Dislocation Densities  
in  
individual grains of polycrystalline aggregates  
of  
plastically deformed **CoTi** and **CoZr** alloys

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**T.Ungár, G.Ribárik, Gy.Zilahi, R.Mulay, U.Lienert, L.Balogh, S.Agnew,**

*Acta Materialia, 71 (2014) 264–282*

# Slip-Modes in B2

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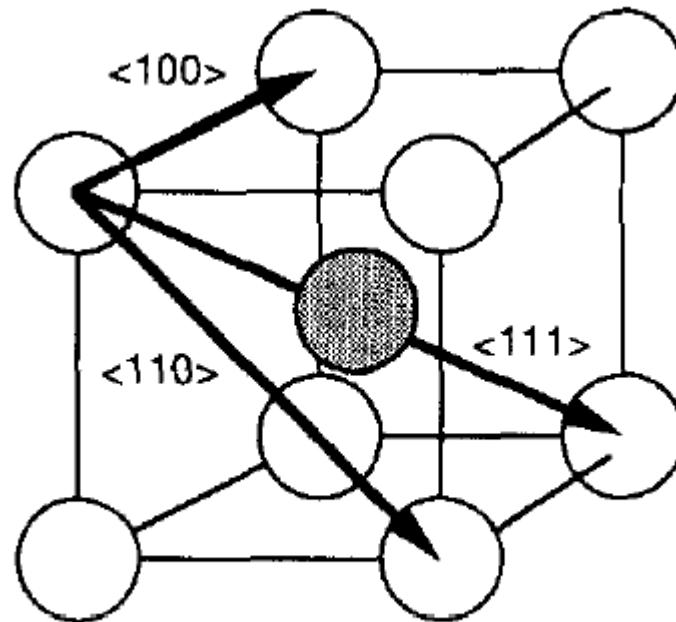


Fig. 1. B2 structure, showing the slip vectors.

earlier TEM investigations:

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single crystals:

**only soft-mode**

with  $<100>$  type Burgers vectors

**can yield**

e.g. Takasugi T Tsurisaki K, Izumi O, Ono S. *Philos Mag A* 1990;61(5):785-800.

polycrystals:

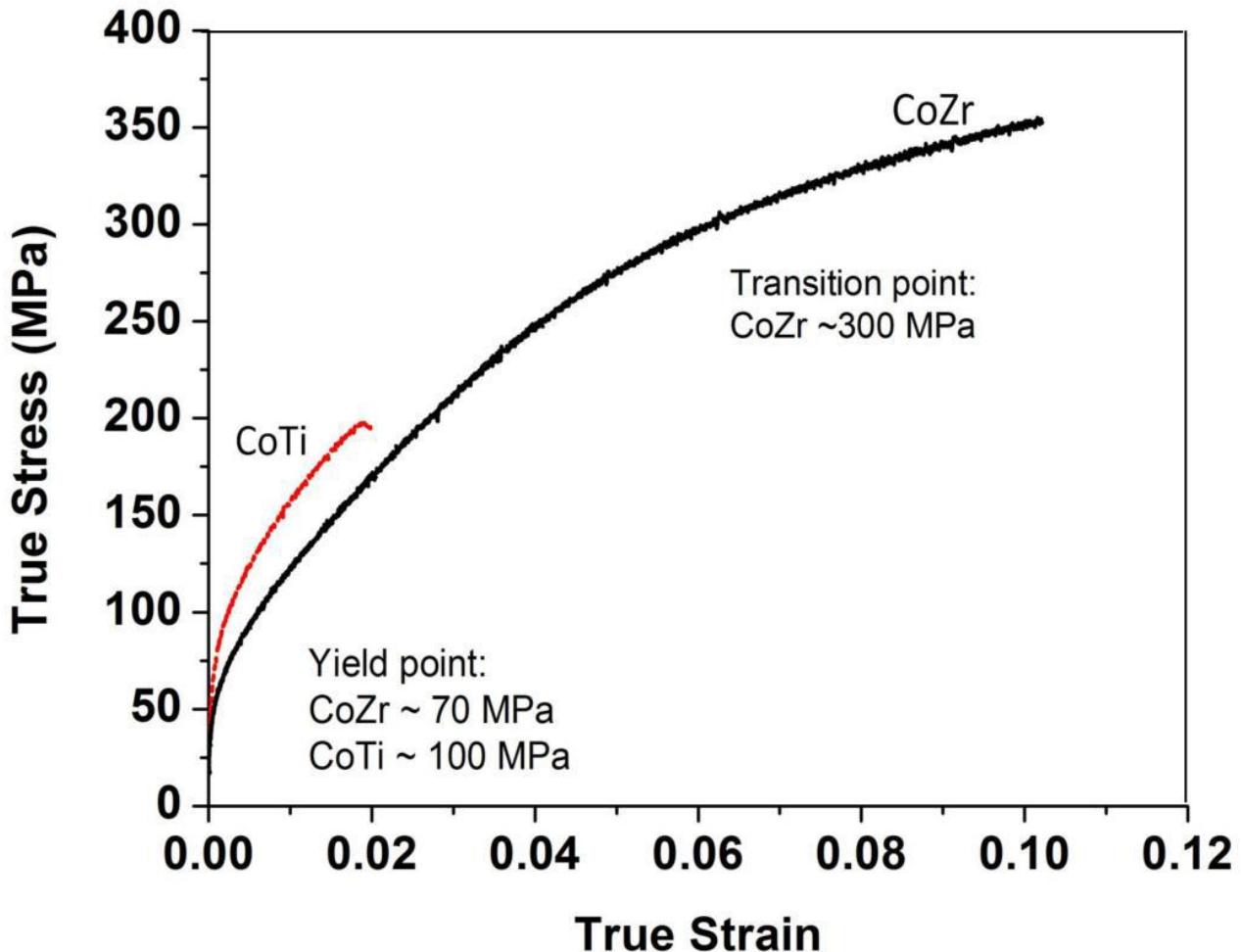
**hard-mode**

with  $<110>$  and/or  $<111>$  Burgers vectors

**are also present**

e.g. Wollmershauser JA, Neil CJ, Agnew SR. *Metall Mater Trans 41A*;2010:1217-9.

# stress-strain curve of *polycrystalline* CoTi and CoZr

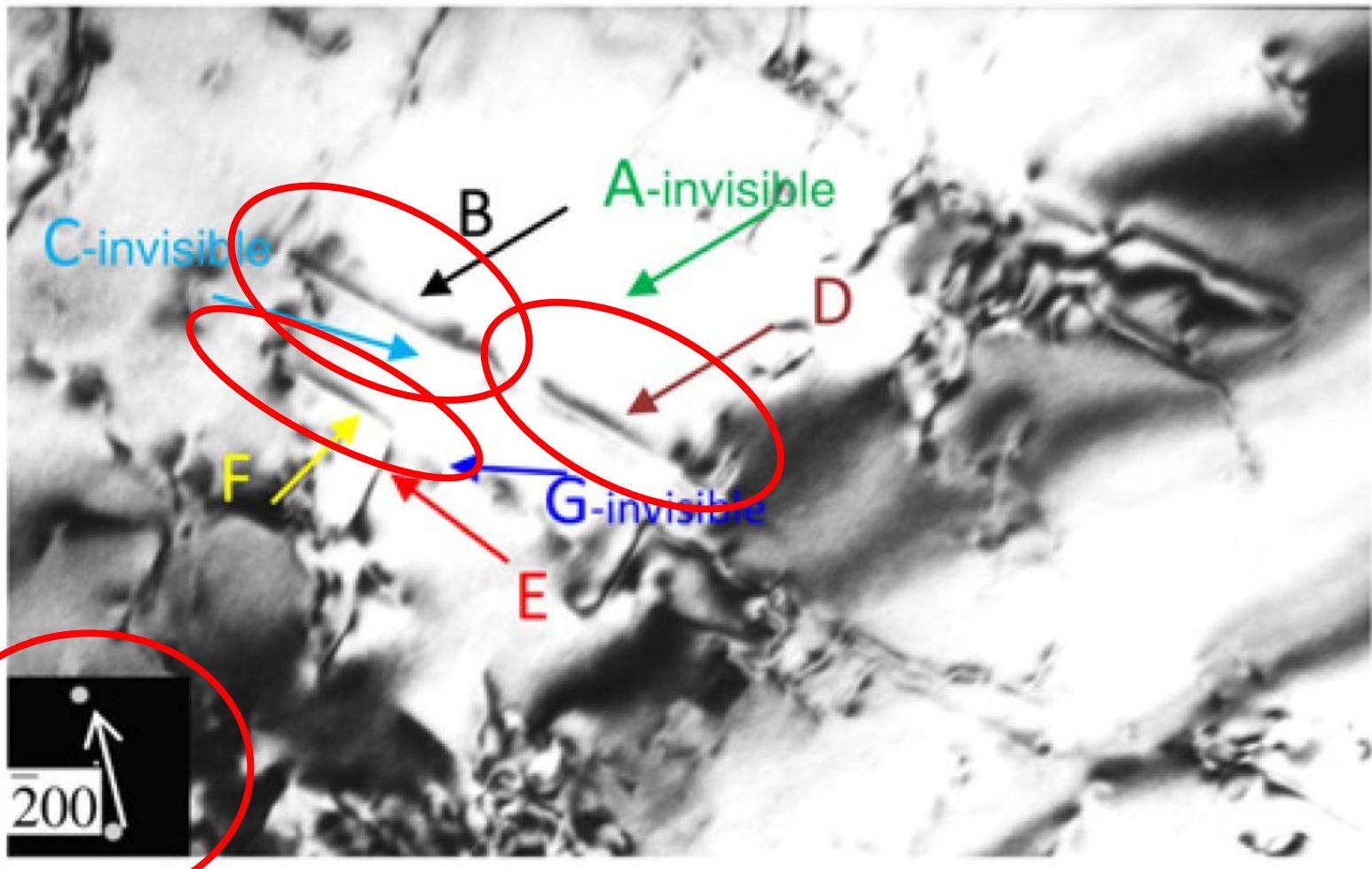


# Hard-mode dislocations in CoTi:

B, D and F

(9% strain)

(e)



the **contrast of dislocations** depends on:

the **relative orientation** between

(1) the **diffraction vector**

(2) the **Burgers vector**

(3) the **line vector**

(4) and the **elastic constants** of the material

in line broadening of X-ray or neutron diffraction

the contrast appears in

the mean-square-strain

$$\langle \varepsilon_{L,g}^2 \rangle \cong \frac{\rho \cdot C \cdot b^2}{4\pi} f(L/R_e)$$

---

**C** is the contrast factor of dislocations

$$C = C(g, b, l, c_{ij})$$

if  $C$  could be measured  
for many different  $\mathbf{g}$  vectors  
there could be a chance to determine:  $\mathbf{b}$  and  $\mathbf{l}$

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powder diffraction is NOT suitable  
because of the **averaging** over the **orientations** of the  $\mathbf{g}$  vectors

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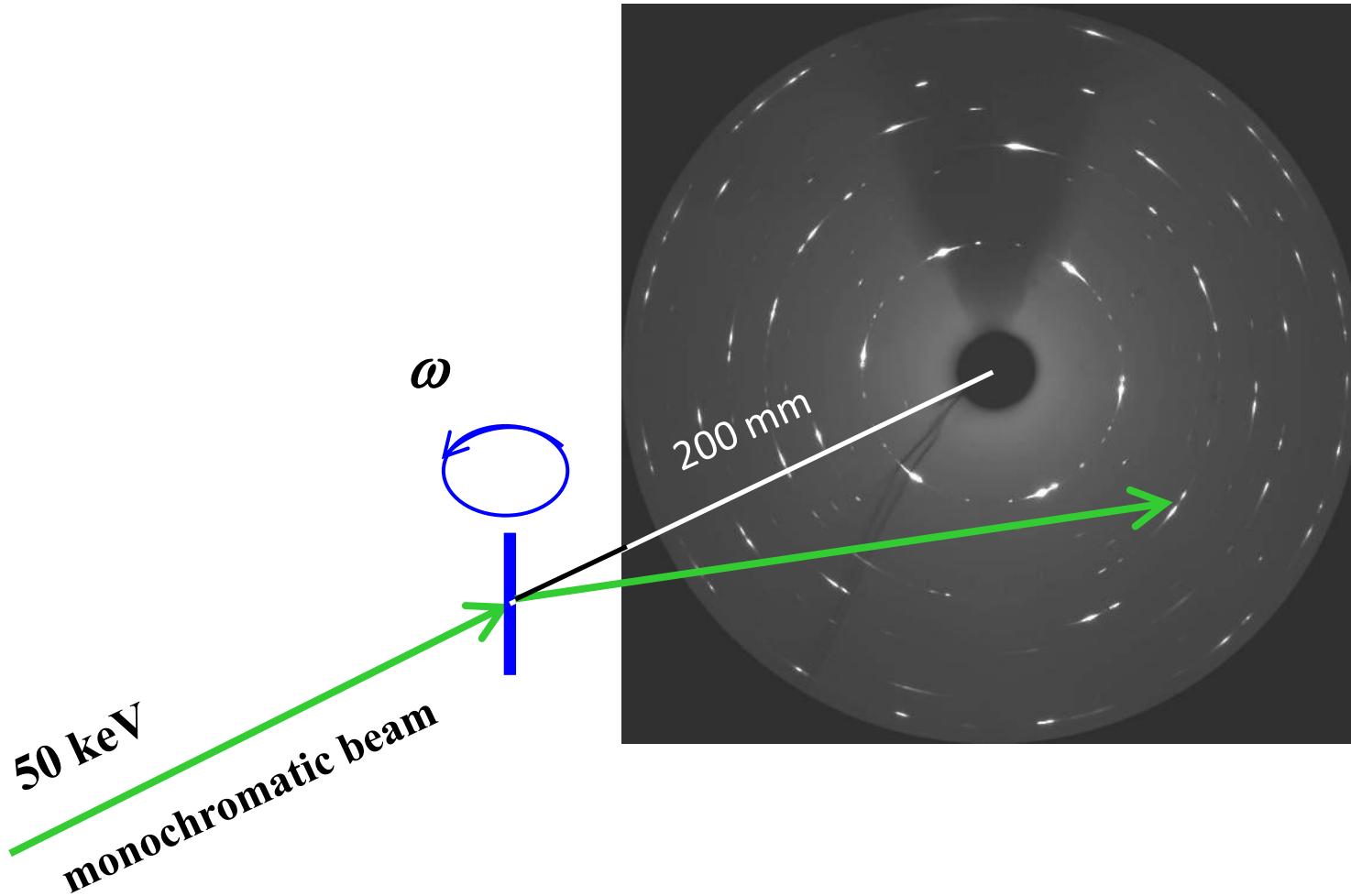
many different  $\mathbf{g}$  vectors:  
needs single-crystal diffraction

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in a **polycrystal**: **single-grain diffraction**

# diffraction of single grains in a polycrystalline specimen

Lauridsen EM, Schmidt S, Suterb RM, Poulsen HF. *J Appl Cryst* 2001;34:744-750.

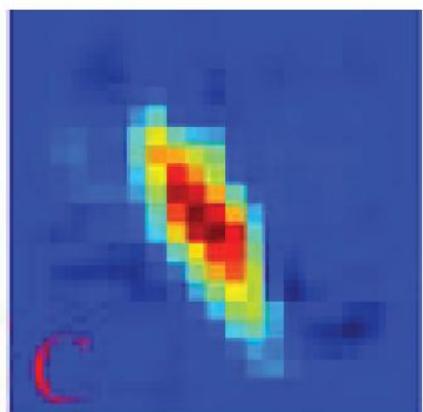
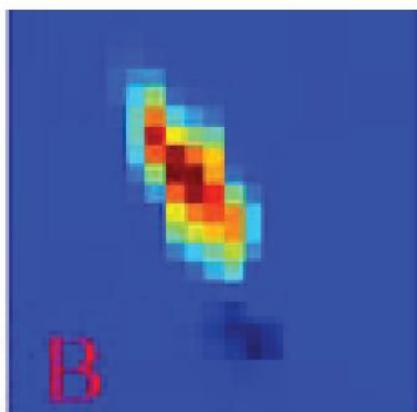


# Multigrain crystallography

HO Sorensen, S Schmidt, JP Wright, GBM Vaughan, S Techert, EF Garman, J Oddershede, J Davaasambu, KS Paithankar, C Gundlach, HF Poulsen, *Z.Kristallographie*, 227 (2012) 63-78.

Rotation angle,  $\omega$ , of slice :

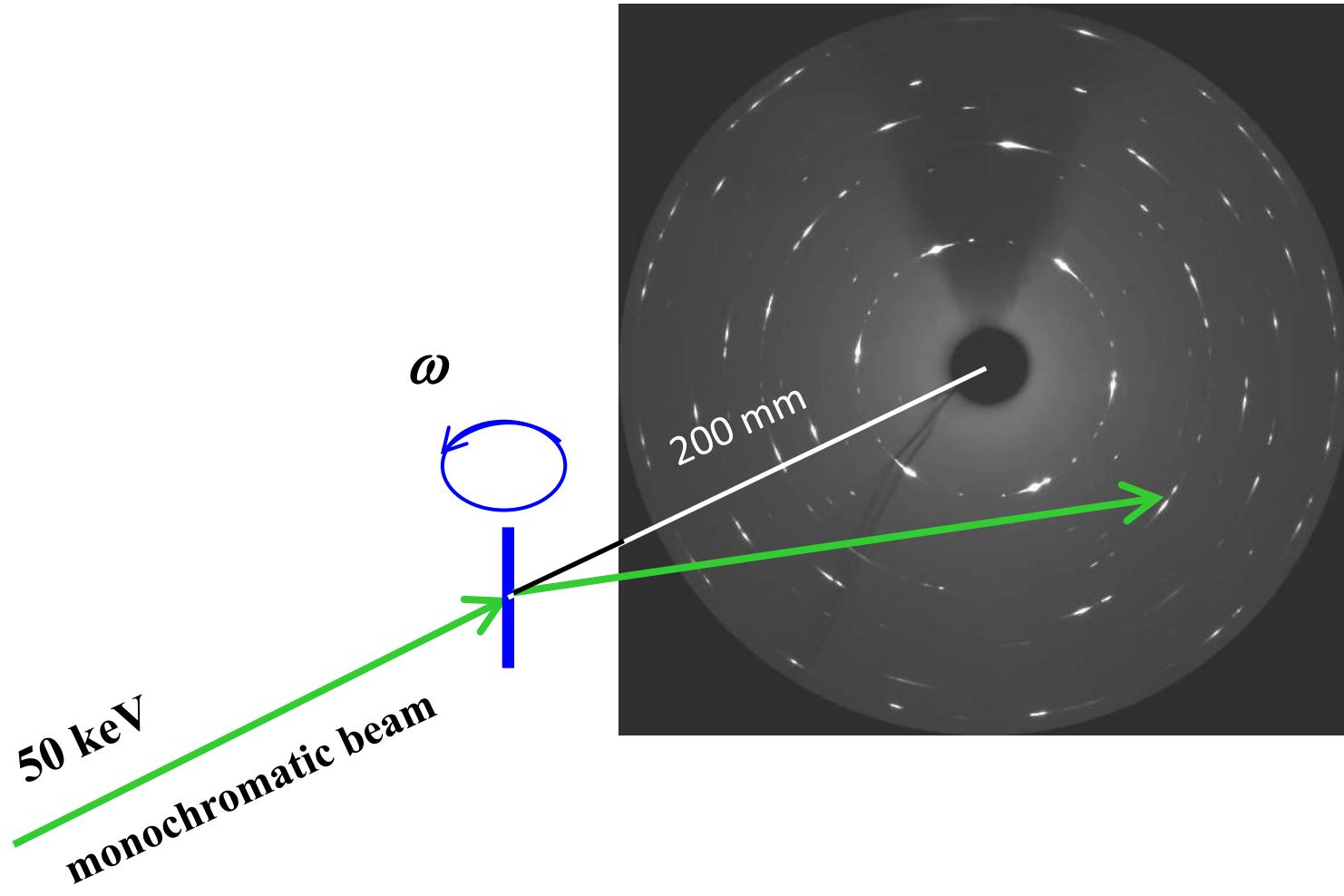
**10.65° 10.95° 11.25° 11.55° 11.85° 12.15° 12.45°**



distance sample to detector:  
~5 to ~38 cm  
provides only a  
limited angular resolution

# diffraction of single grains in a polycrystalline specimen

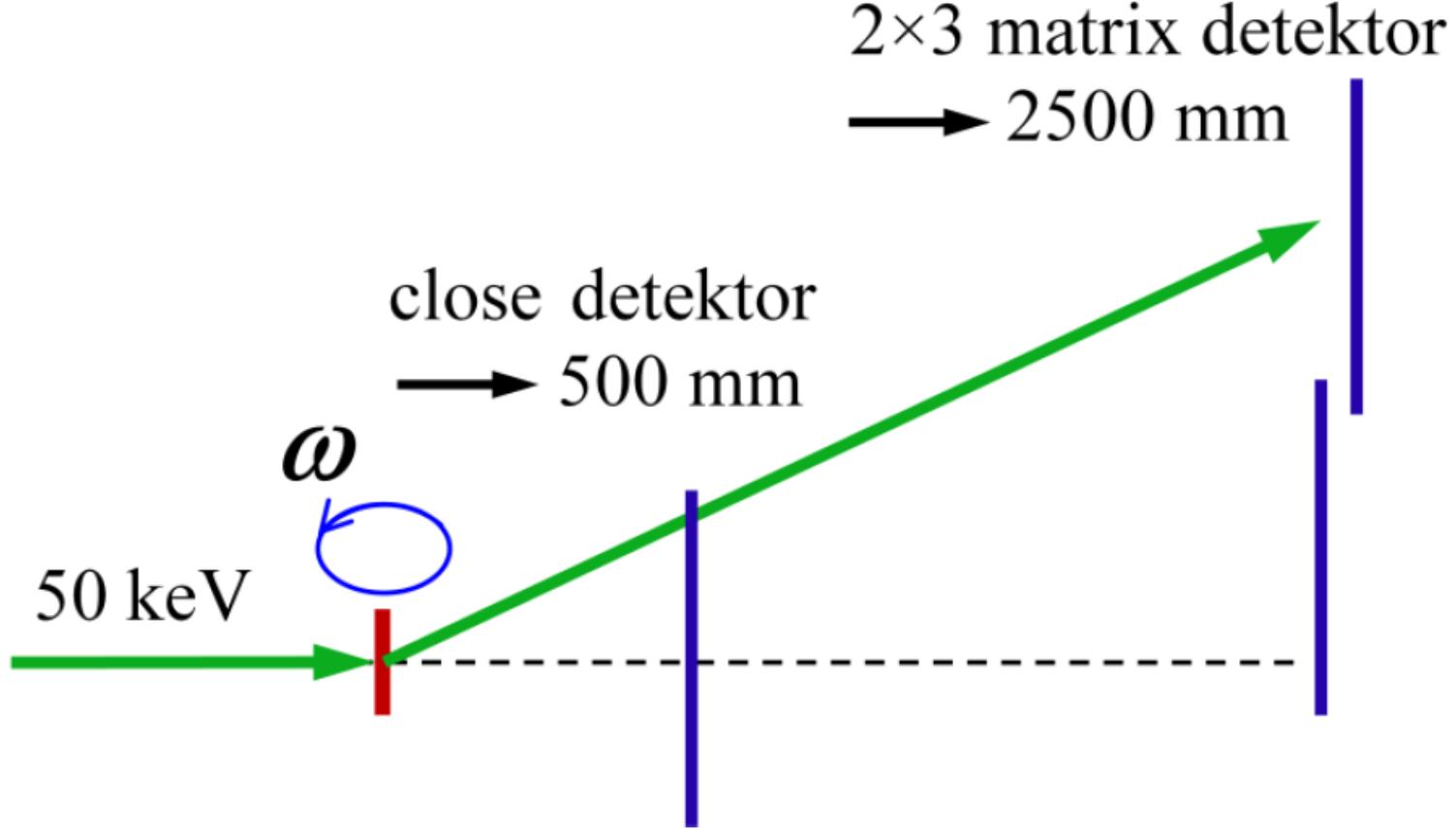
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angular resolution has to be increased

## close and far detector positions

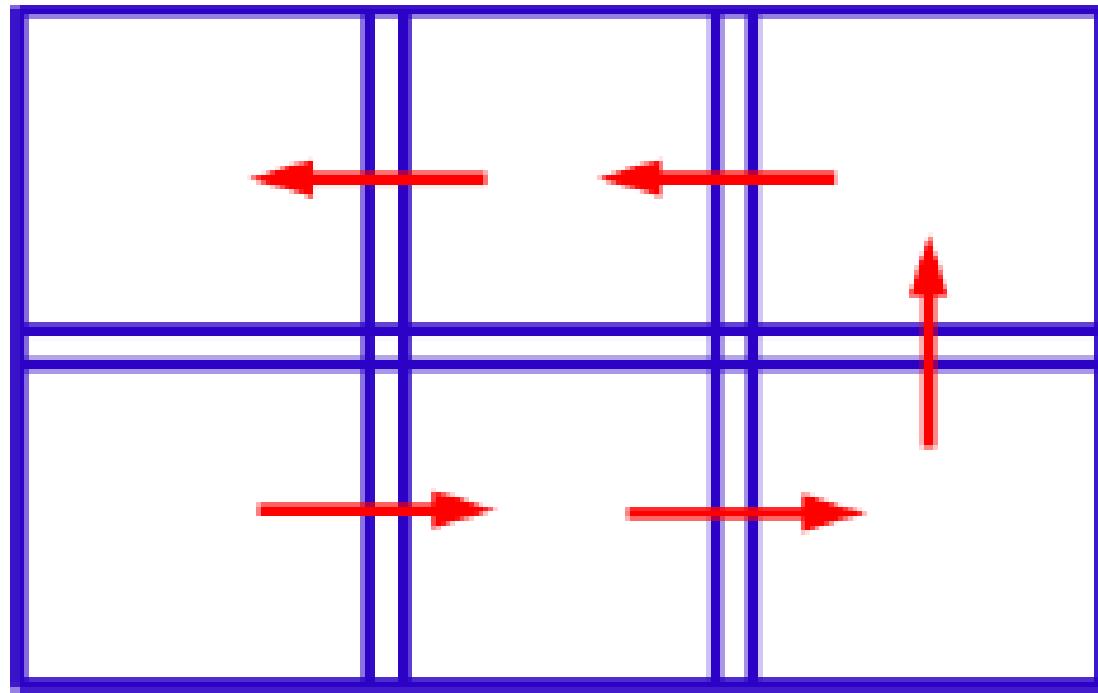
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## far detector positions

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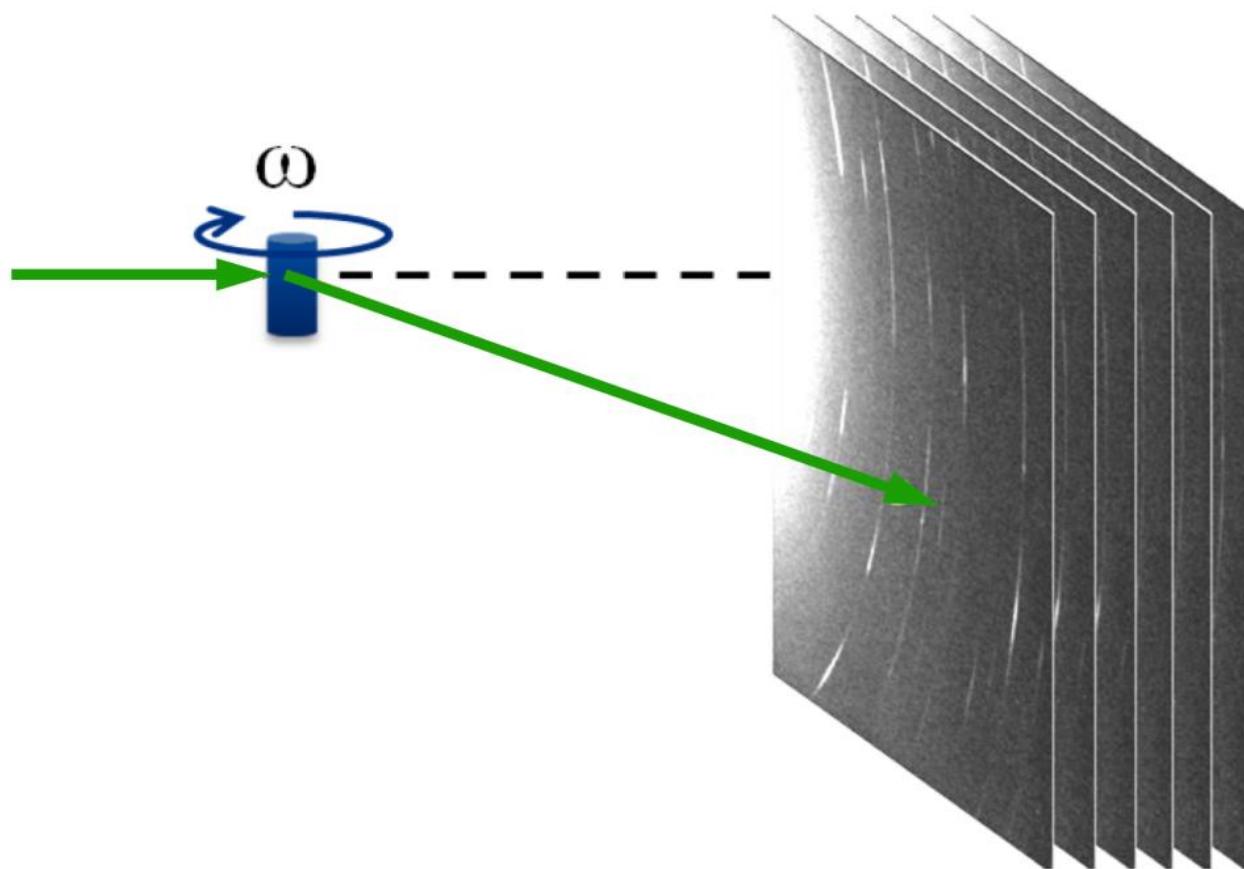
far-detector is moved  
into different positions



## far detector positions

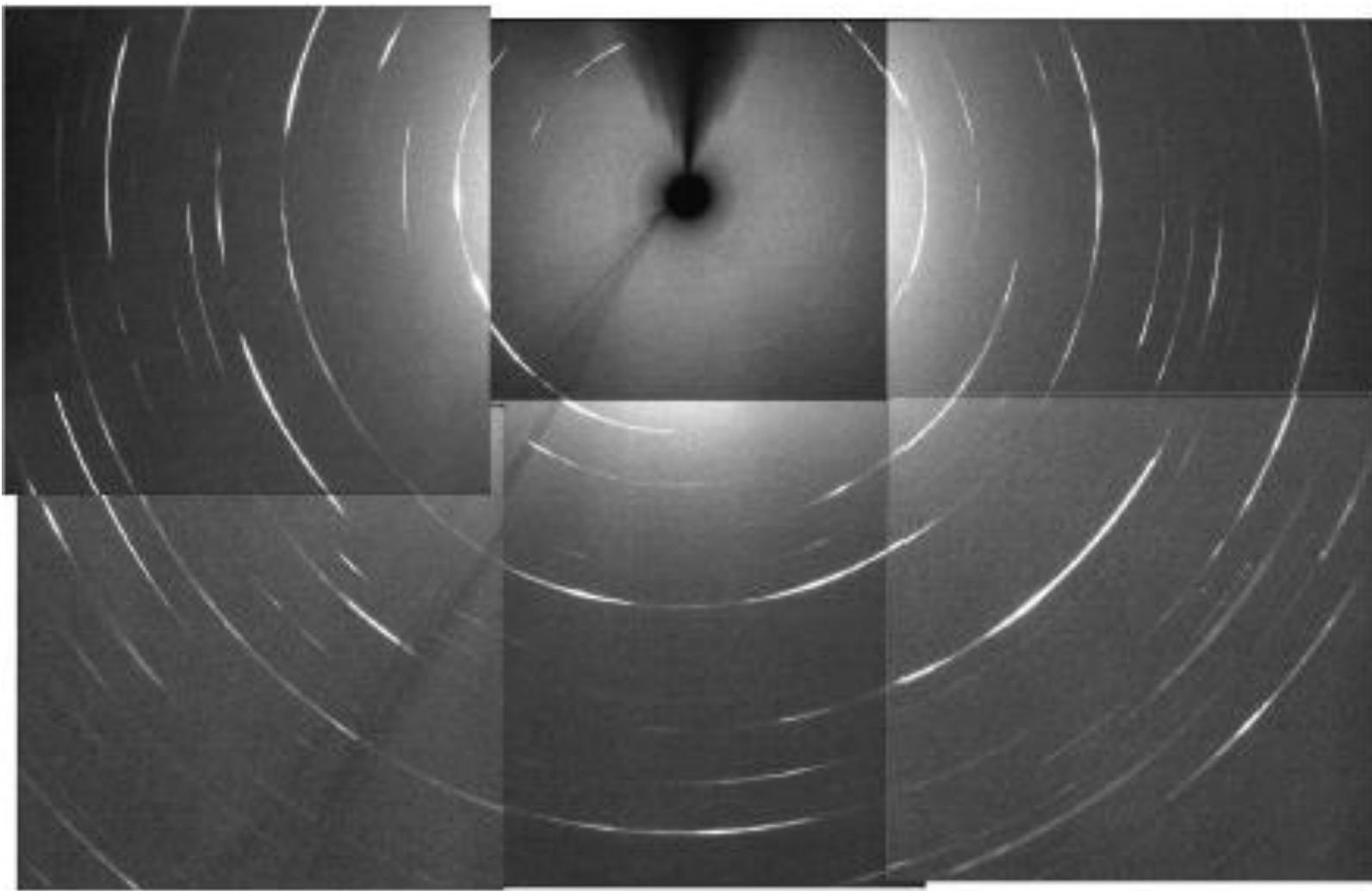
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$\omega$  “frames” in one far-detector position



## far detector positions

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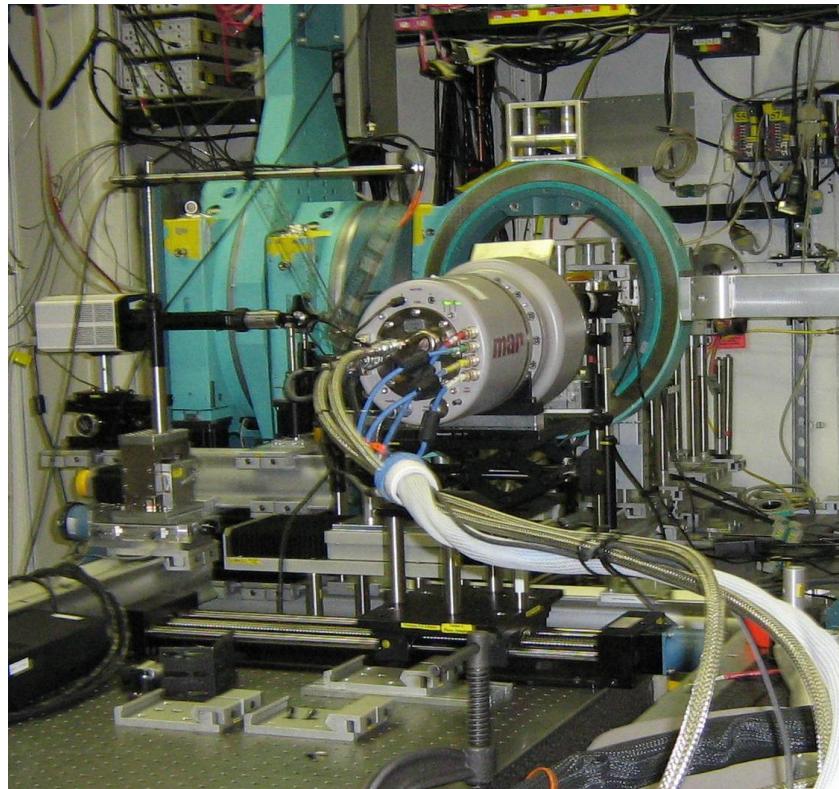


montage of the far-detector image: CoZr

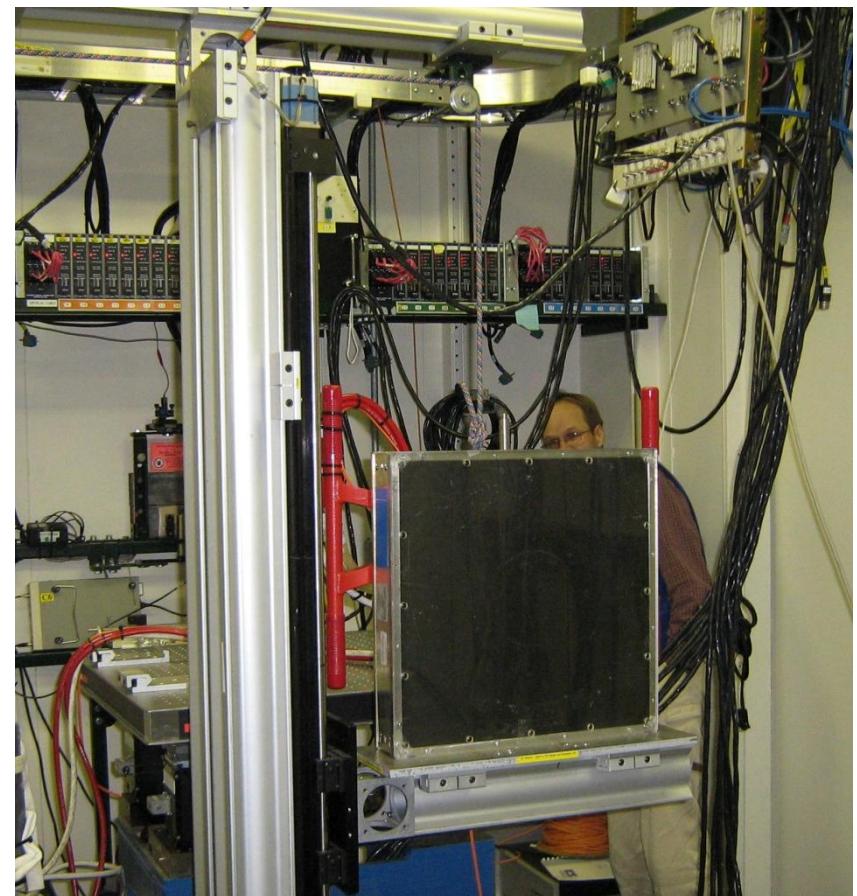
# APS synchrotron: 1ID

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close detector

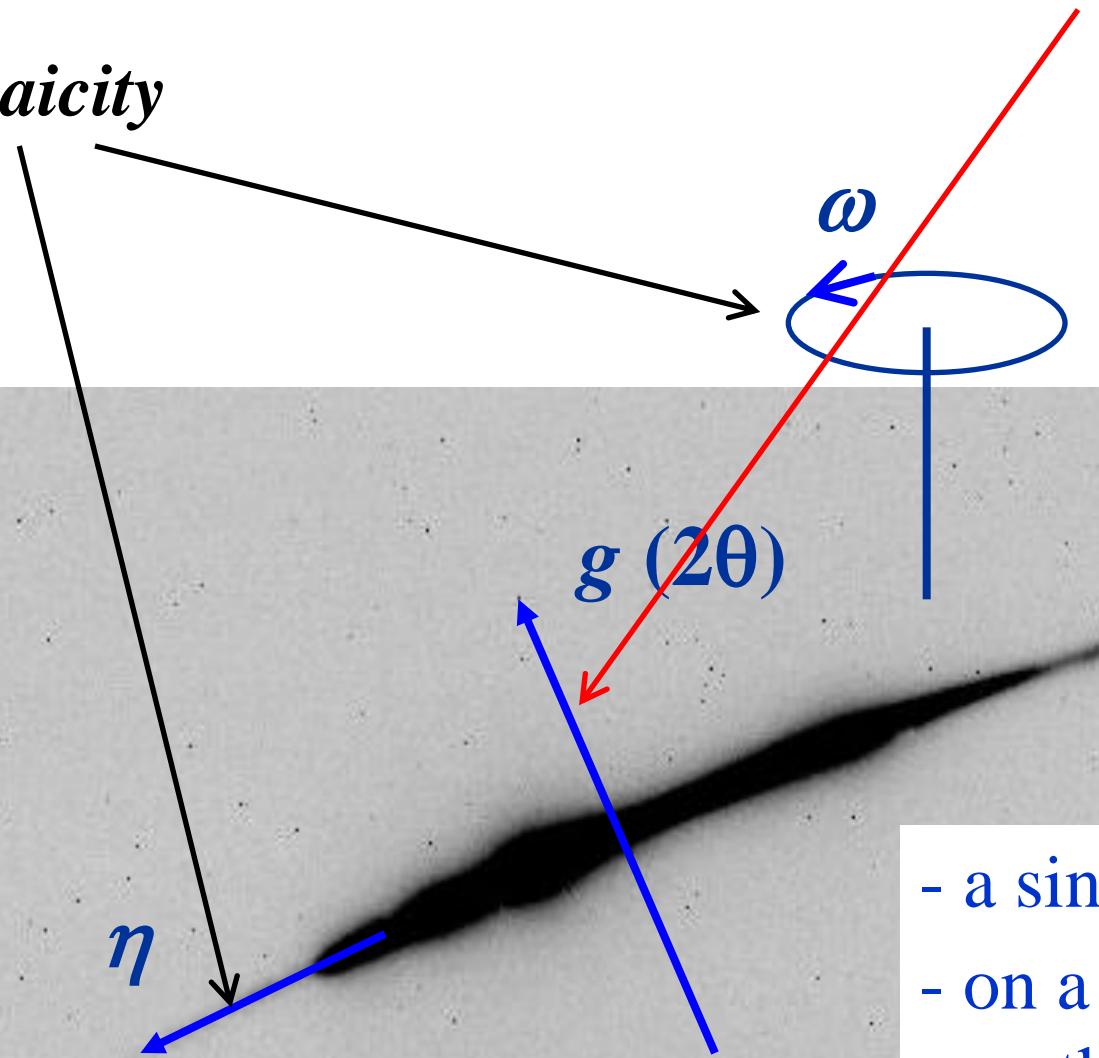


far detector



*change in lattice spacing  
“line-profile”*

*Mozaicity*



- a single reflection
- on a single  $\omega$  “frame”
- on the “far” detector

*Intensity distribution in reciprocal space:*

$$I(\omega, \eta, 2\theta)$$

*Line profile*

$$I(2\theta)$$

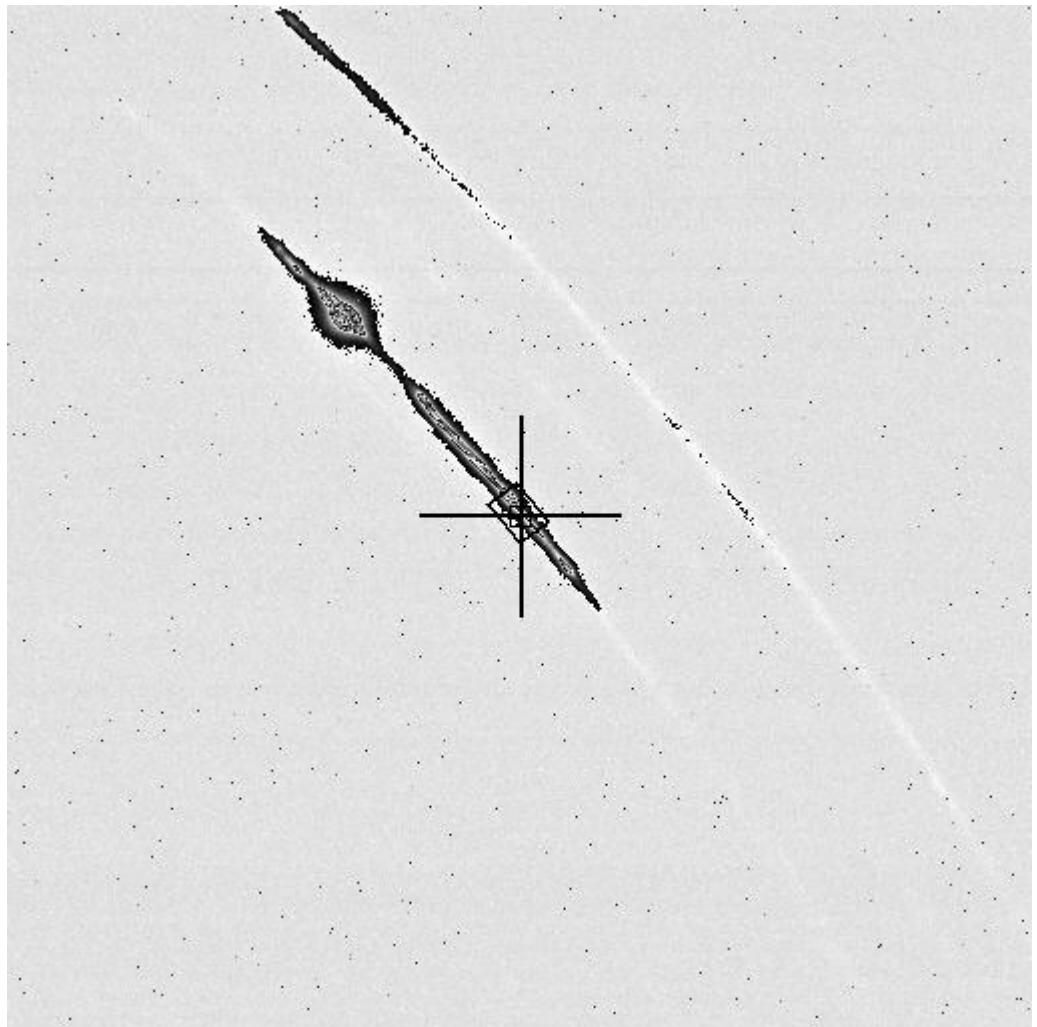
*integration over  $\omega$  and  $\eta$ :*

$$I(2\theta) = \iint I(\omega, \eta, 2\theta) d\omega d\eta$$

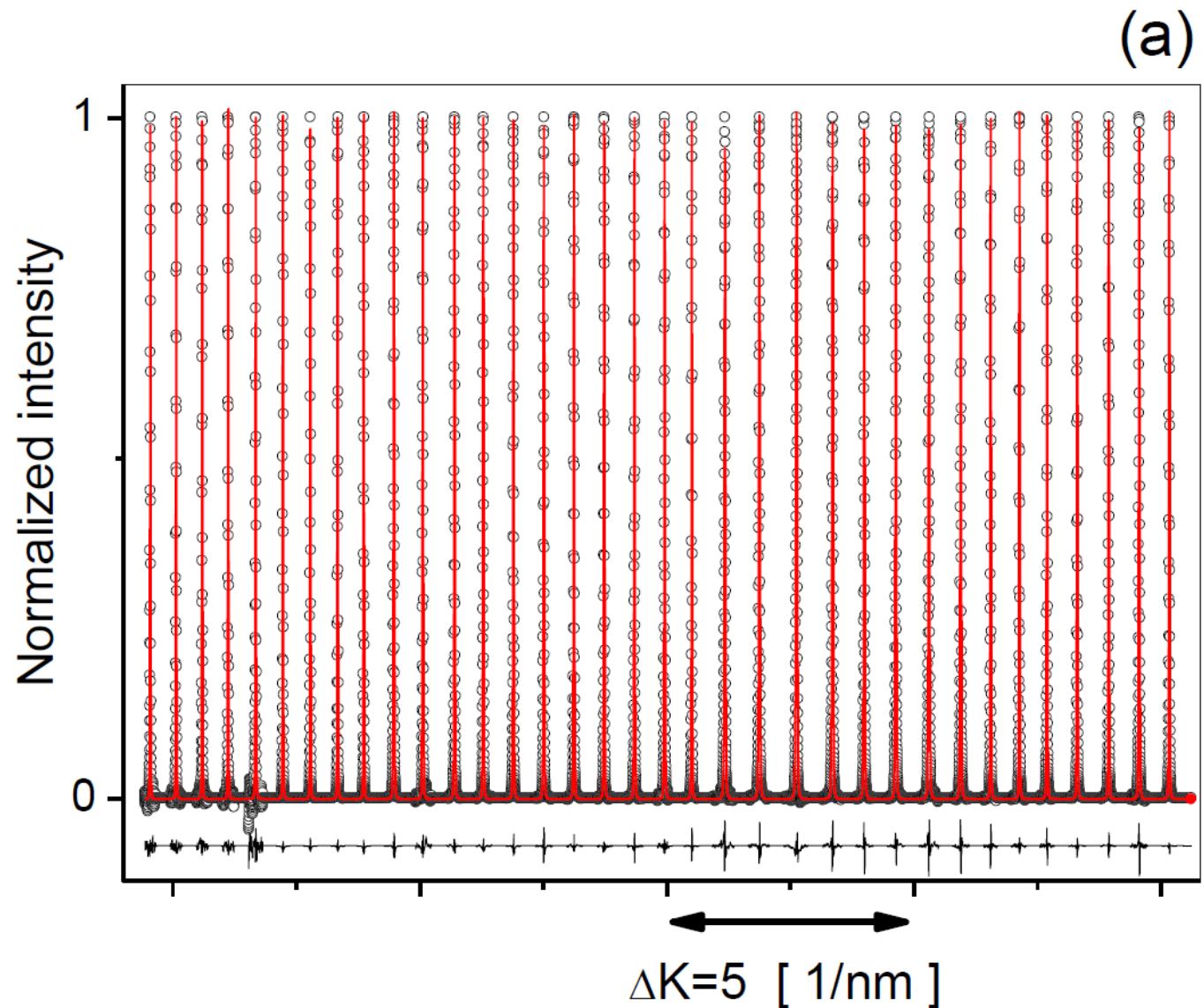
# *Searching for integration volumes*

## integration procedure:

× : position of absolute maximum of a peak  
box: region in  $\eta$  and  $2\theta$

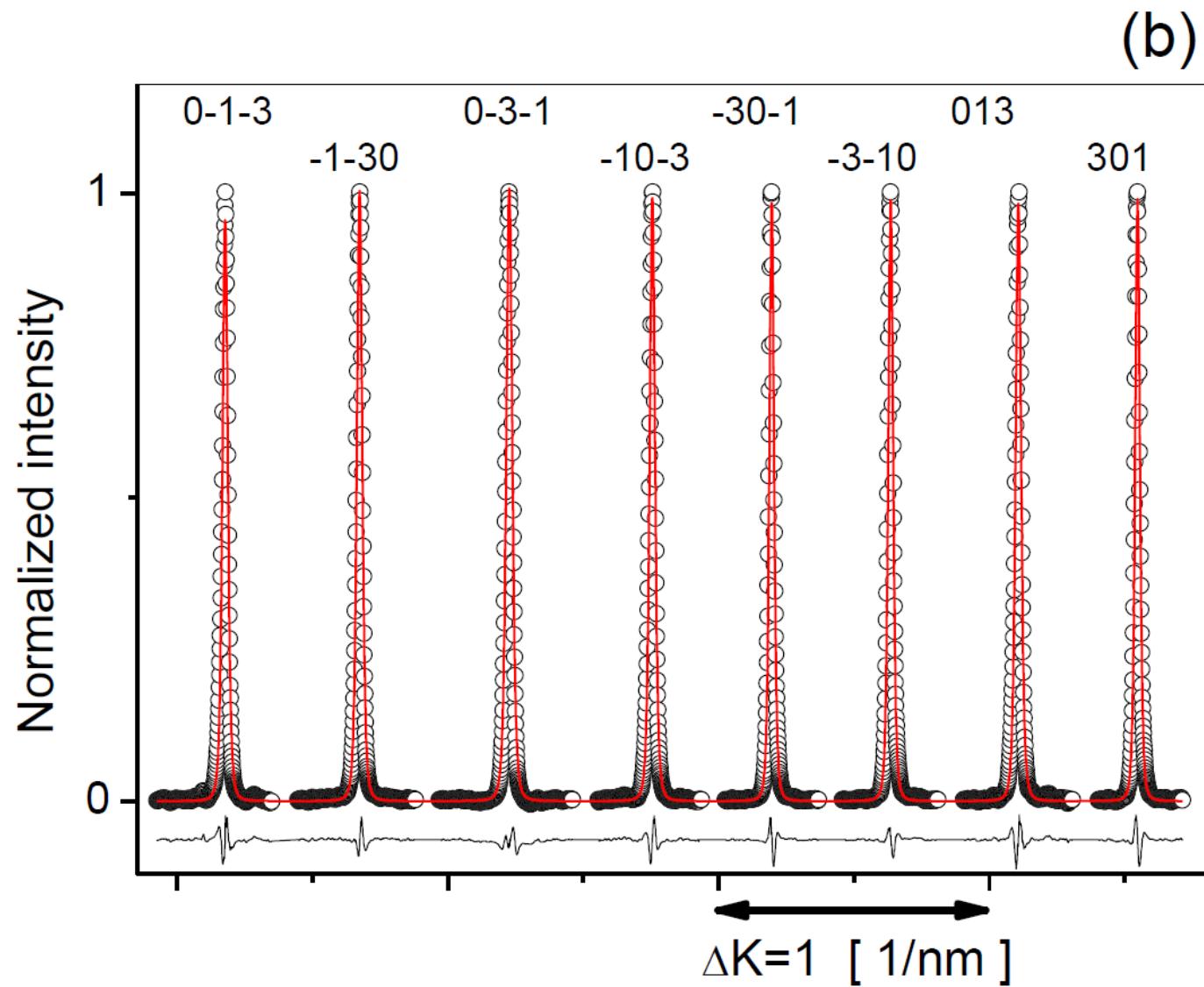


35 peaks corresponding to one of the grains in CoTi



only the 310 type peaks

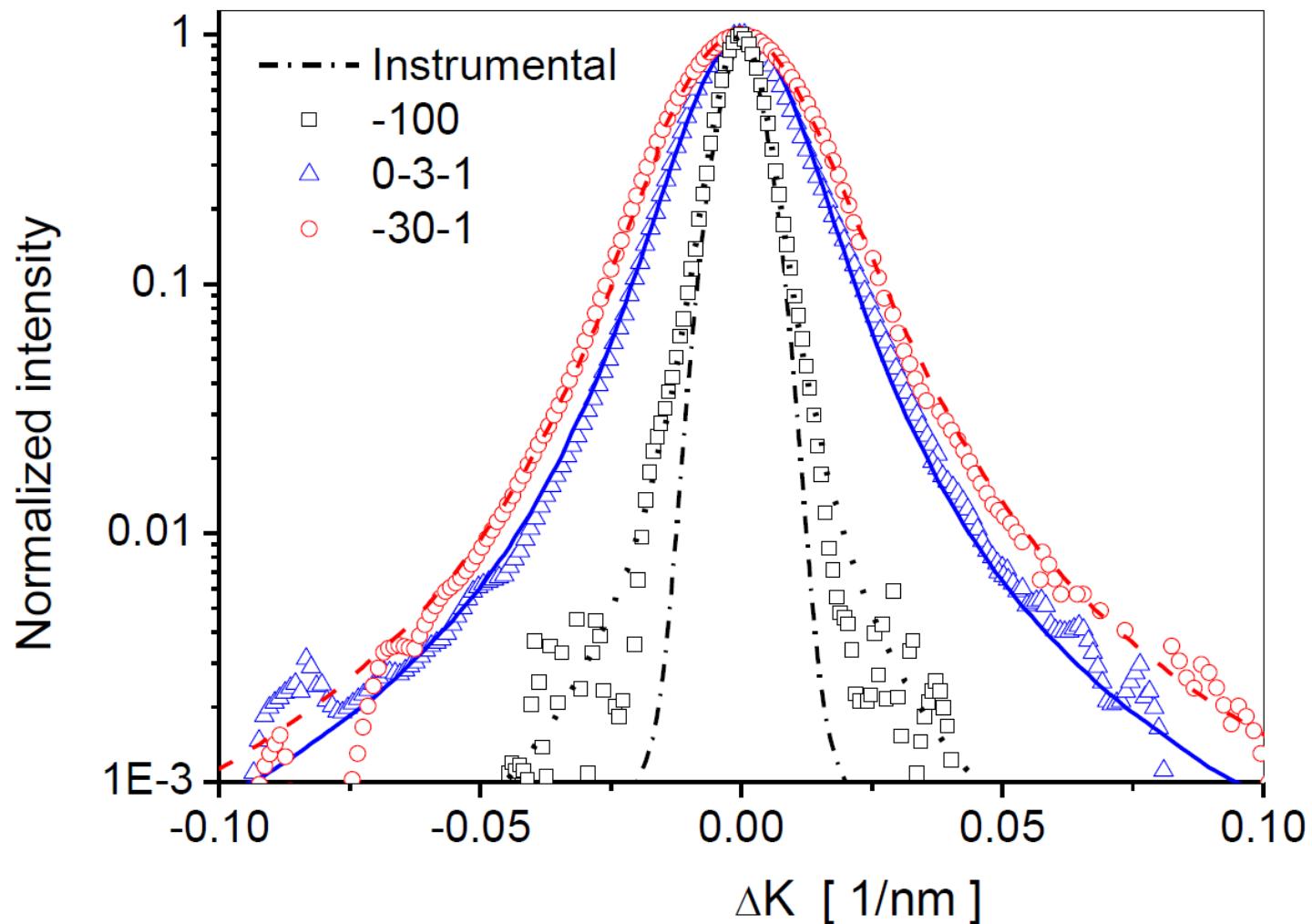
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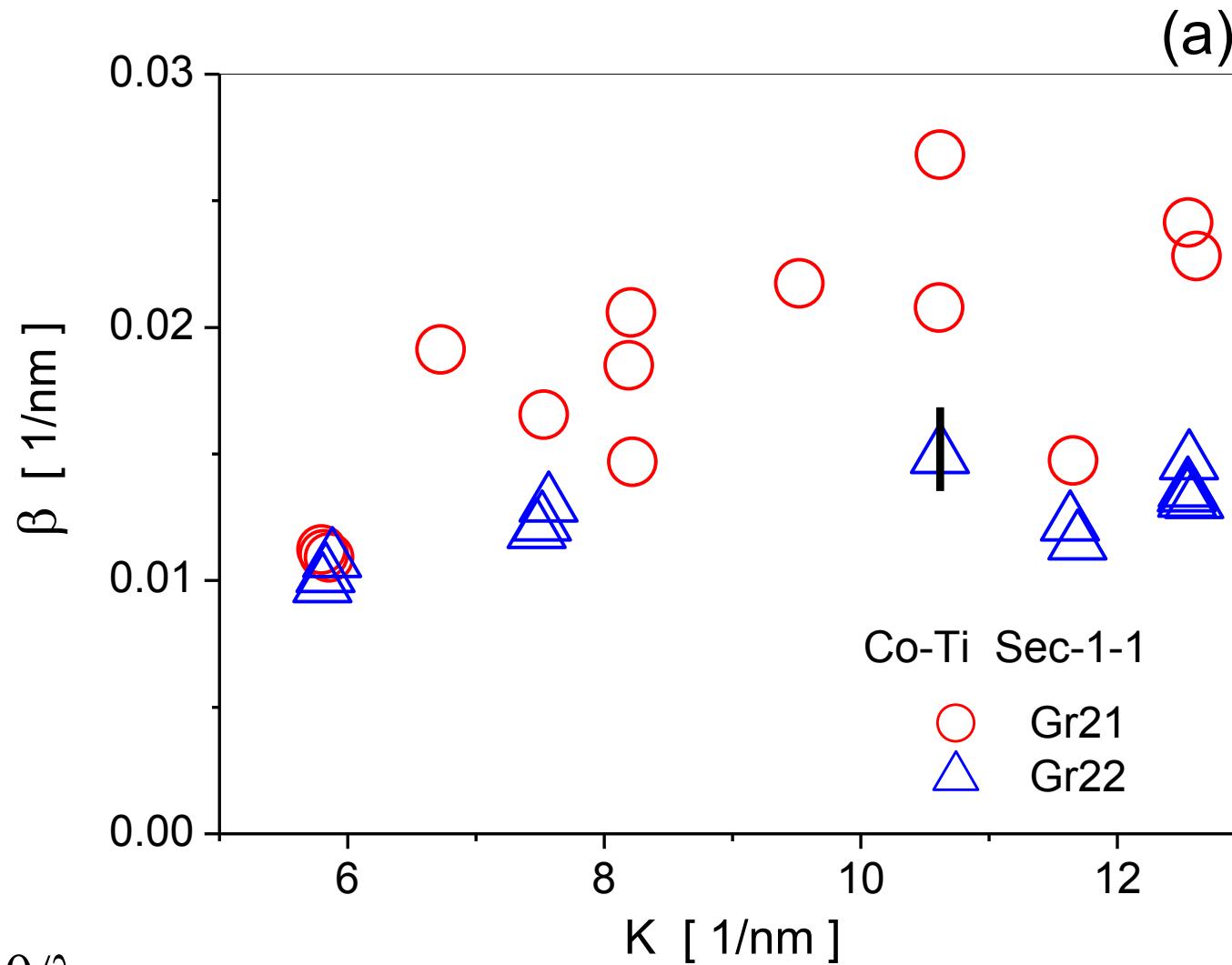
the narrowest and two broad peaks

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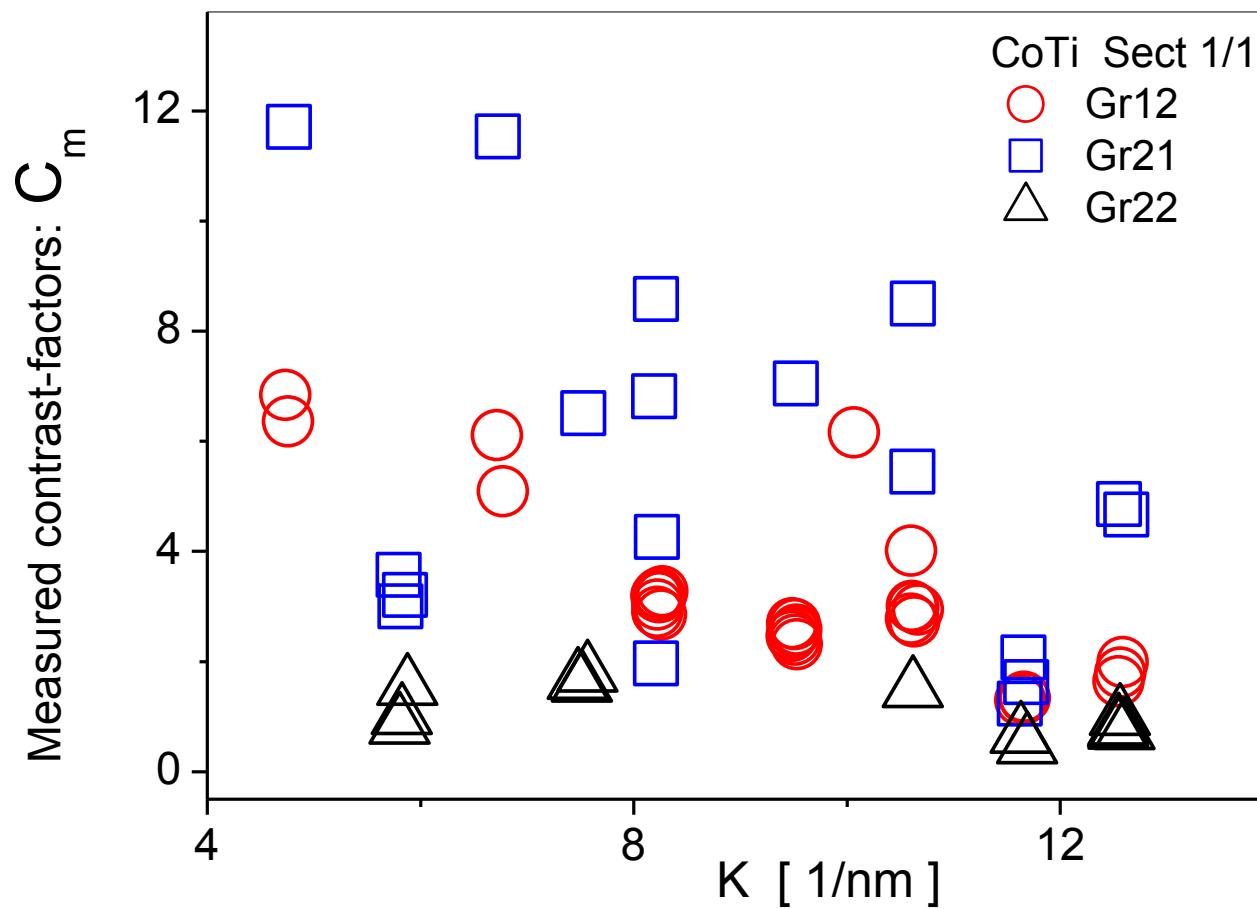
(c)



# Williamson-Hall plots for two grains in CoTi

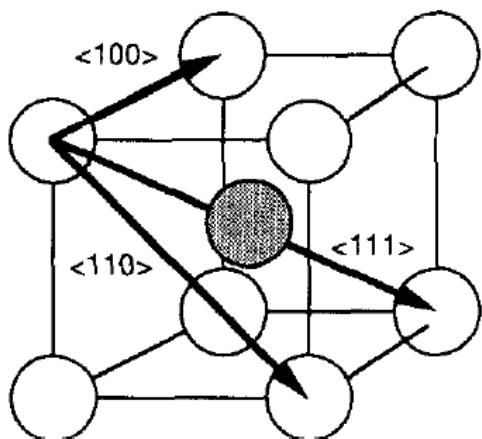


# the measured contrast factors for three grains in CoTi



$$K = 2\sin\theta/\lambda$$

## possible dislocations in B2: CoTi and CoZr



the **number** of measured contrast factors is  
usually **less than 73**  
typically **between 12 and 35**

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Monte-Carlo type procedure:

starting with **12 randomly selected slip-systems**

**cross-check** criterion:

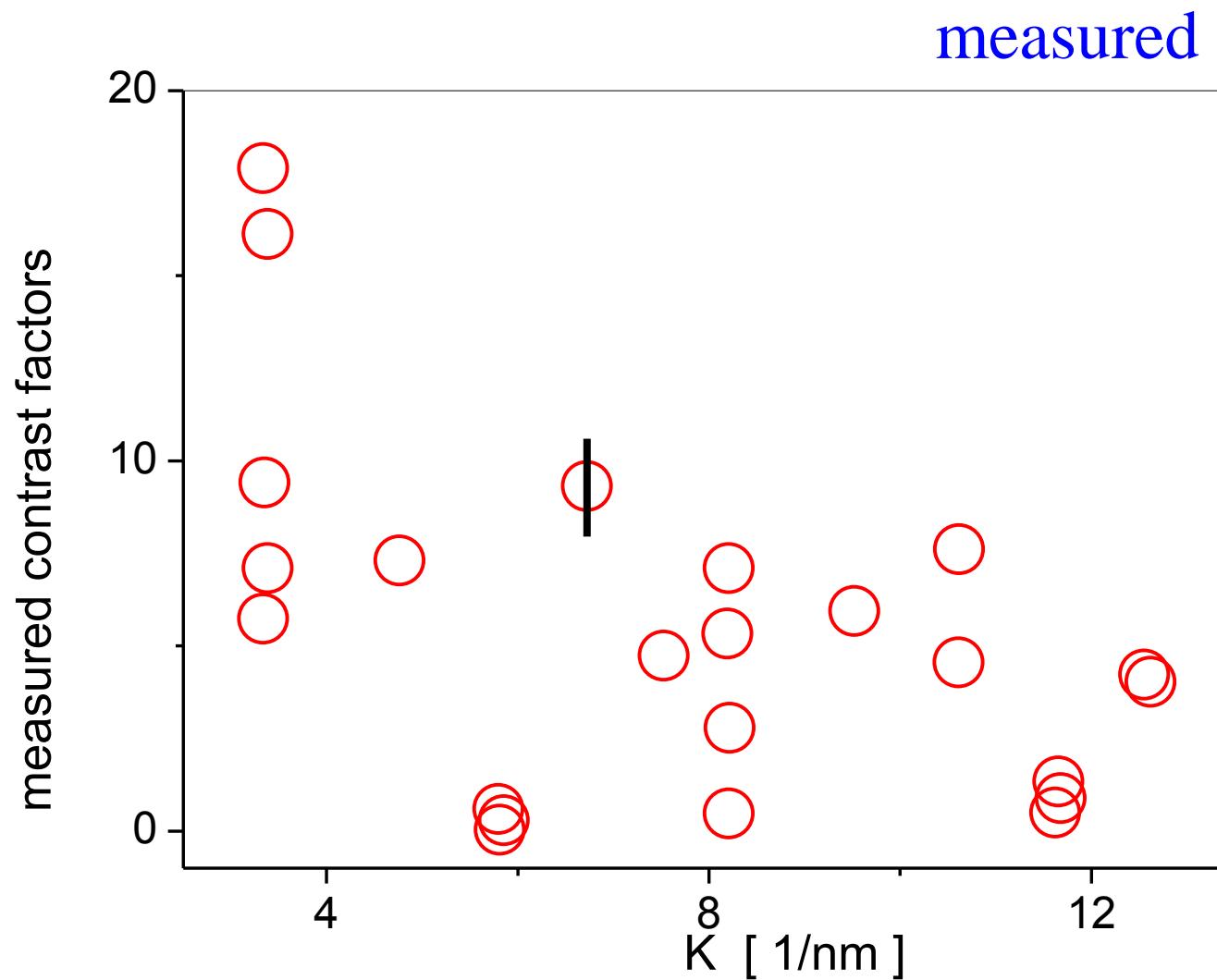
**maximum 11 slip-systems**  
**with substantial fraction**

---

typical **number** of prevailing slip systems: **3 to 6**

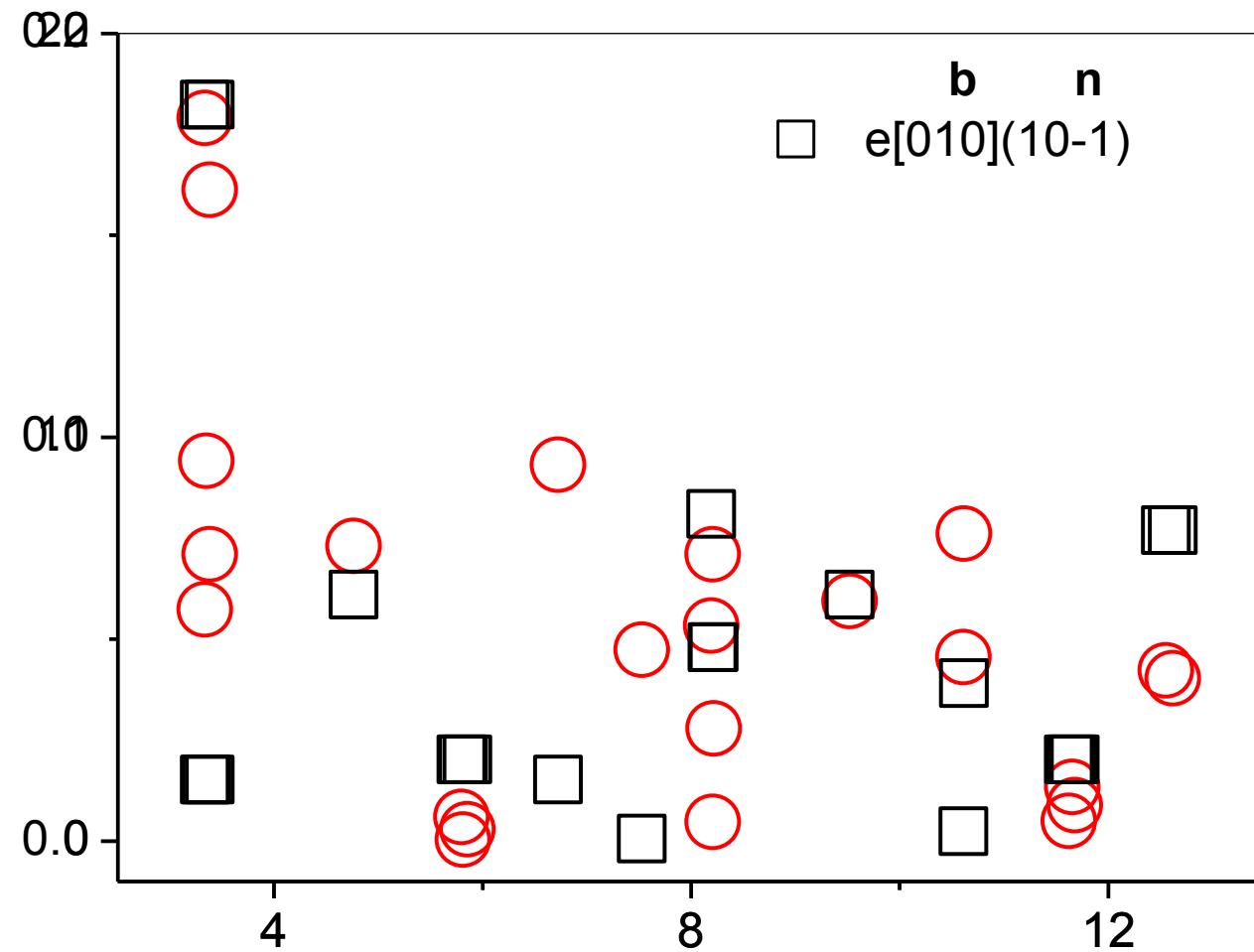
the measured and calculated contrasts in a grain of CoTi

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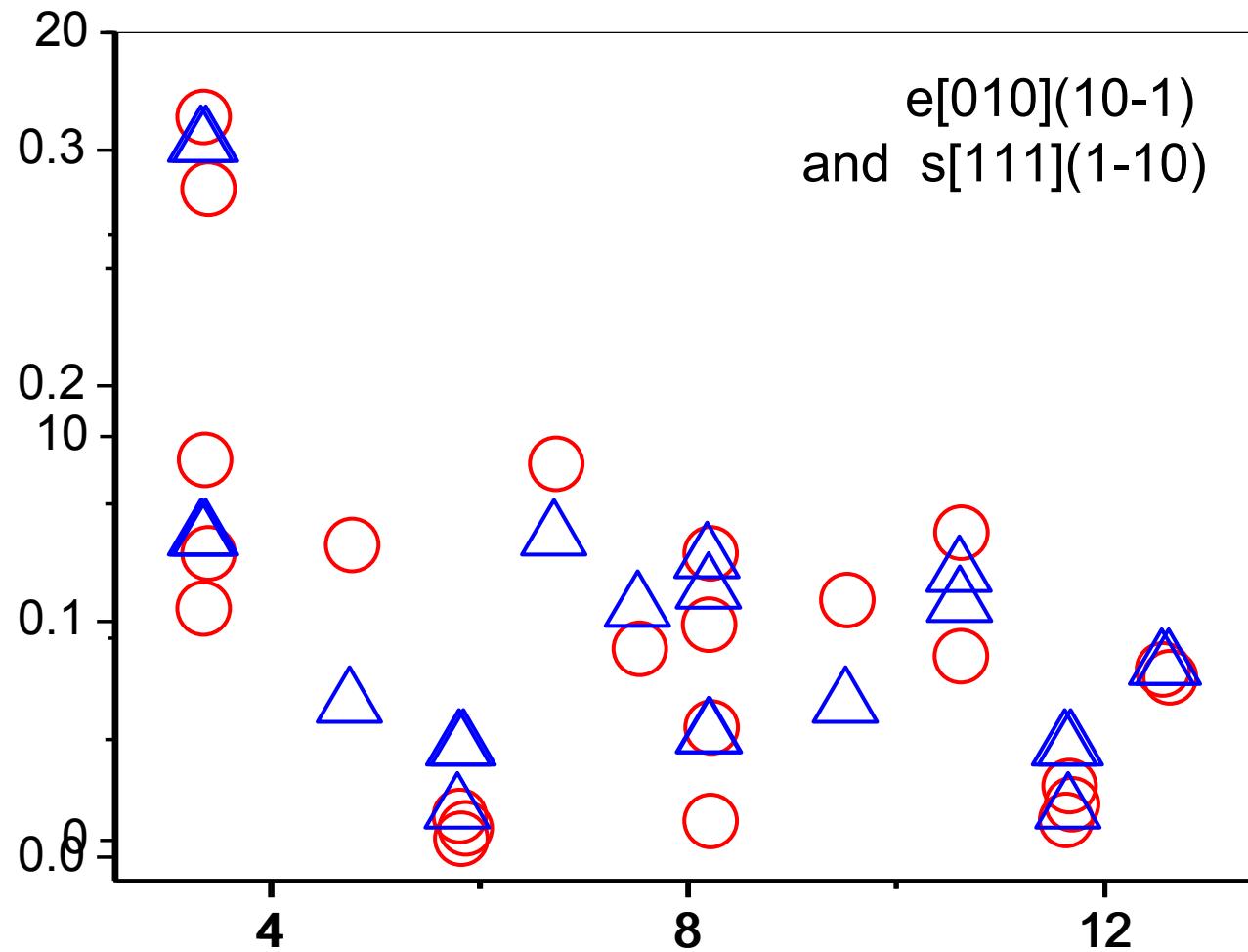
one dislocation: soft-mode dislocation:  $f(\rho)=0.51$

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two dislocations: soft-mode dislocation:  $f(\rho)=0.51$   
hard-mode dislocation:  $f(\rho)=0.26$

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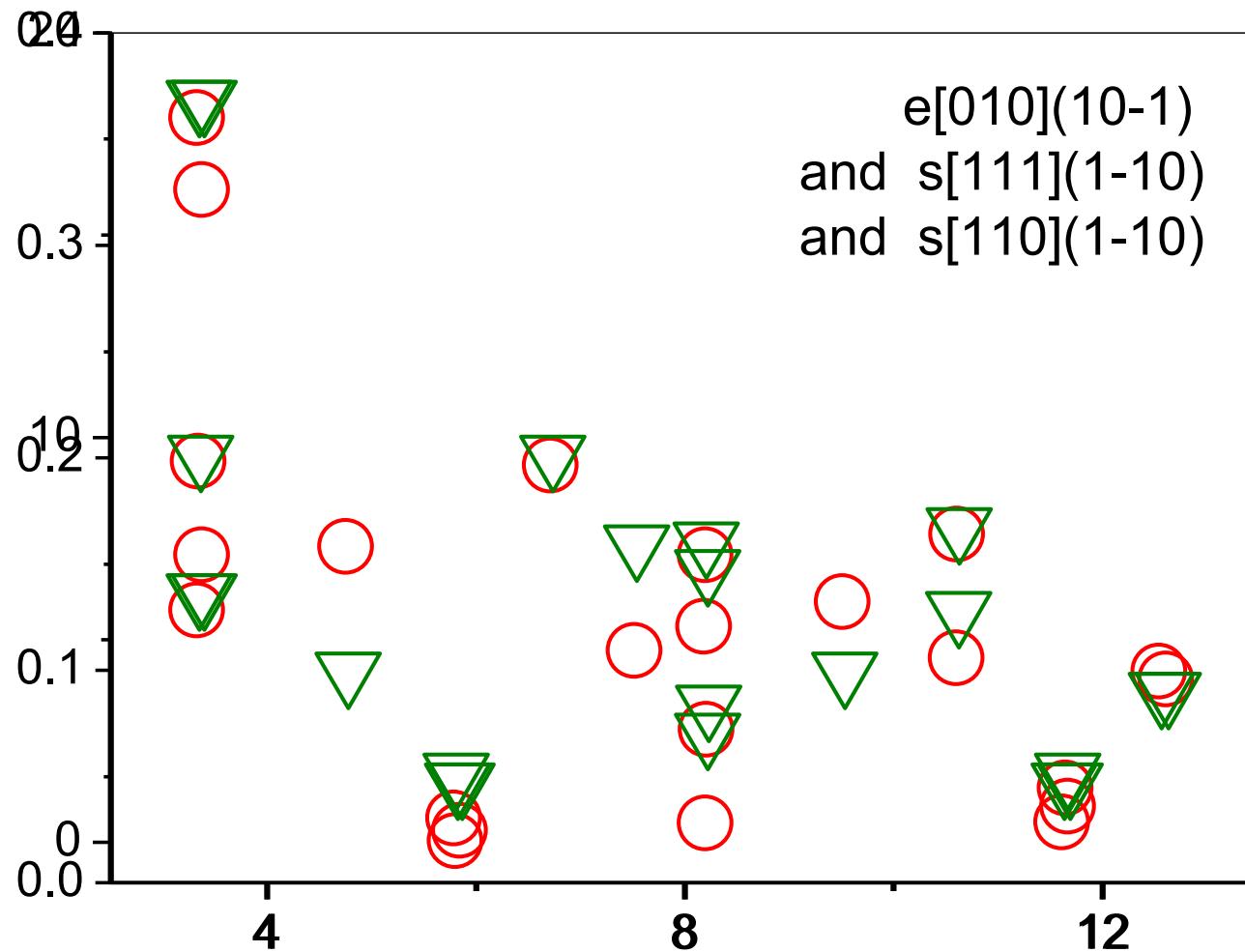


three dislocations:

soft-mode dislocation:  $f(\rho)=0.51$

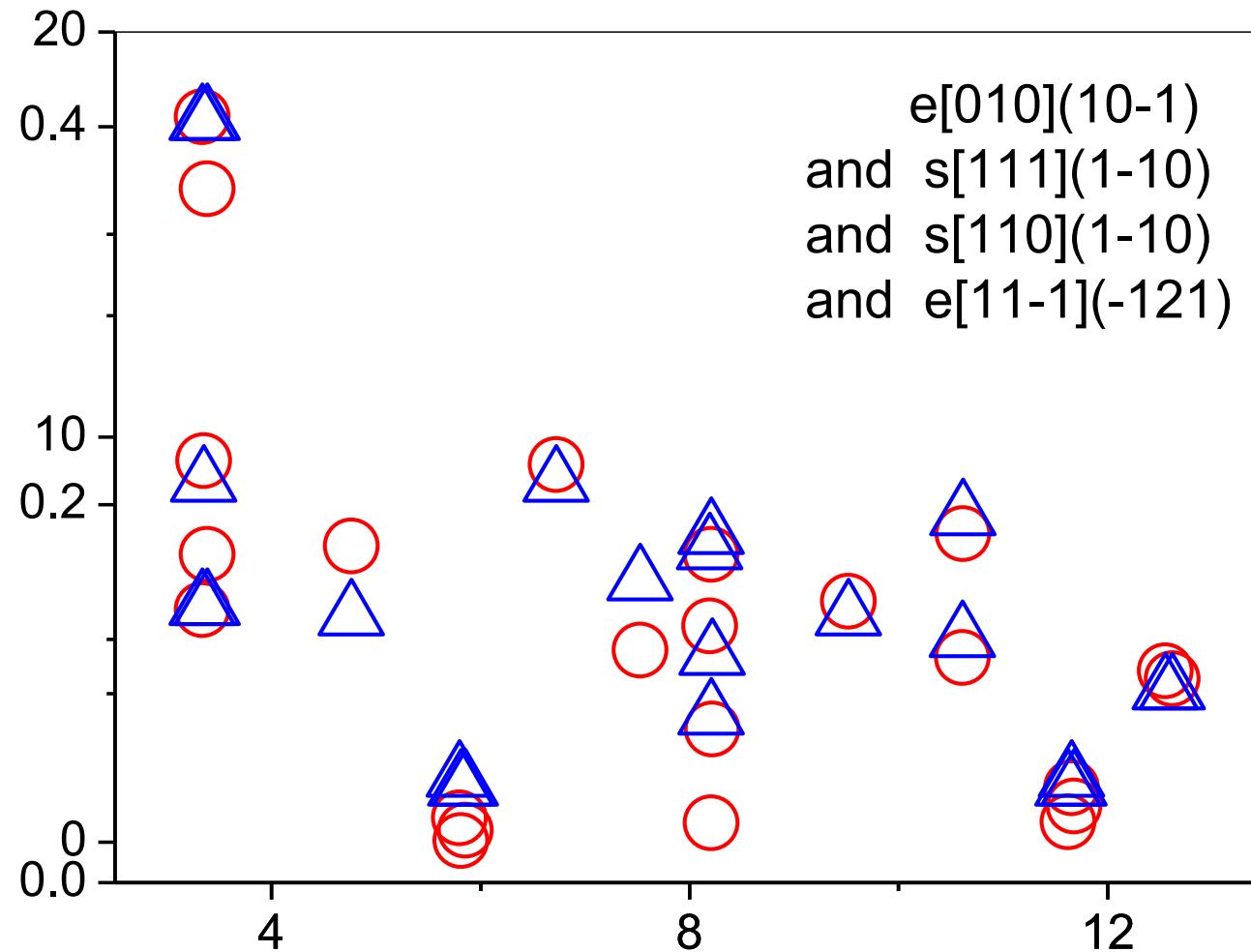
hard-mode dislocation:  $f(\rho)=0.26$

hard-mode dislocation:  $f(\rho)=0.18$



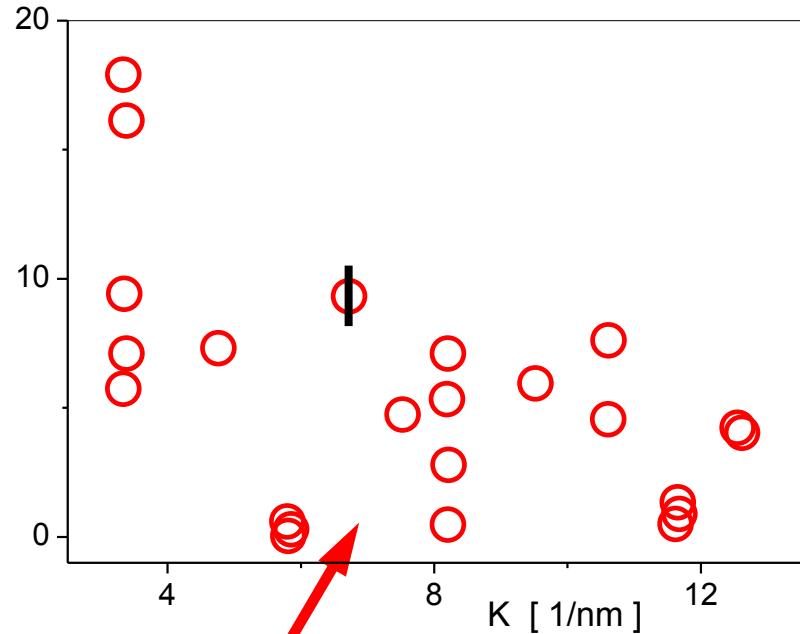
four dislocations:

soft-mode dislocation:  $f(\rho)=0.51$   
hard-mode dislocation:  $f(\rho)=0.26$   
hard-mode dislocation:  $f(\rho)=0.18$   
hard-mode dislocation:  $f(\rho)=0.05$



# the measured and calculated contrasts in a grain of CoTi

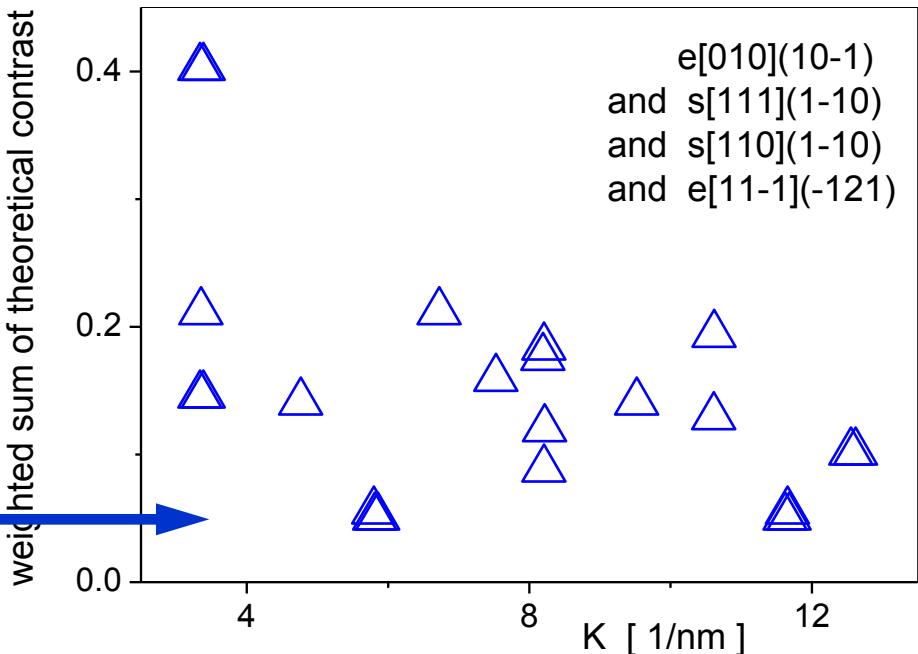
measured contrast factors



the lock

the keys

soft-mode dislocation:  $f(\rho)=0.51$   
hard-mode dislocation:  $f(\rho)=0.26$   
hard-mode dislocation:  $f(\rho)=0.18$   
hard-mode dislocation:  $f(\rho)=0.05$

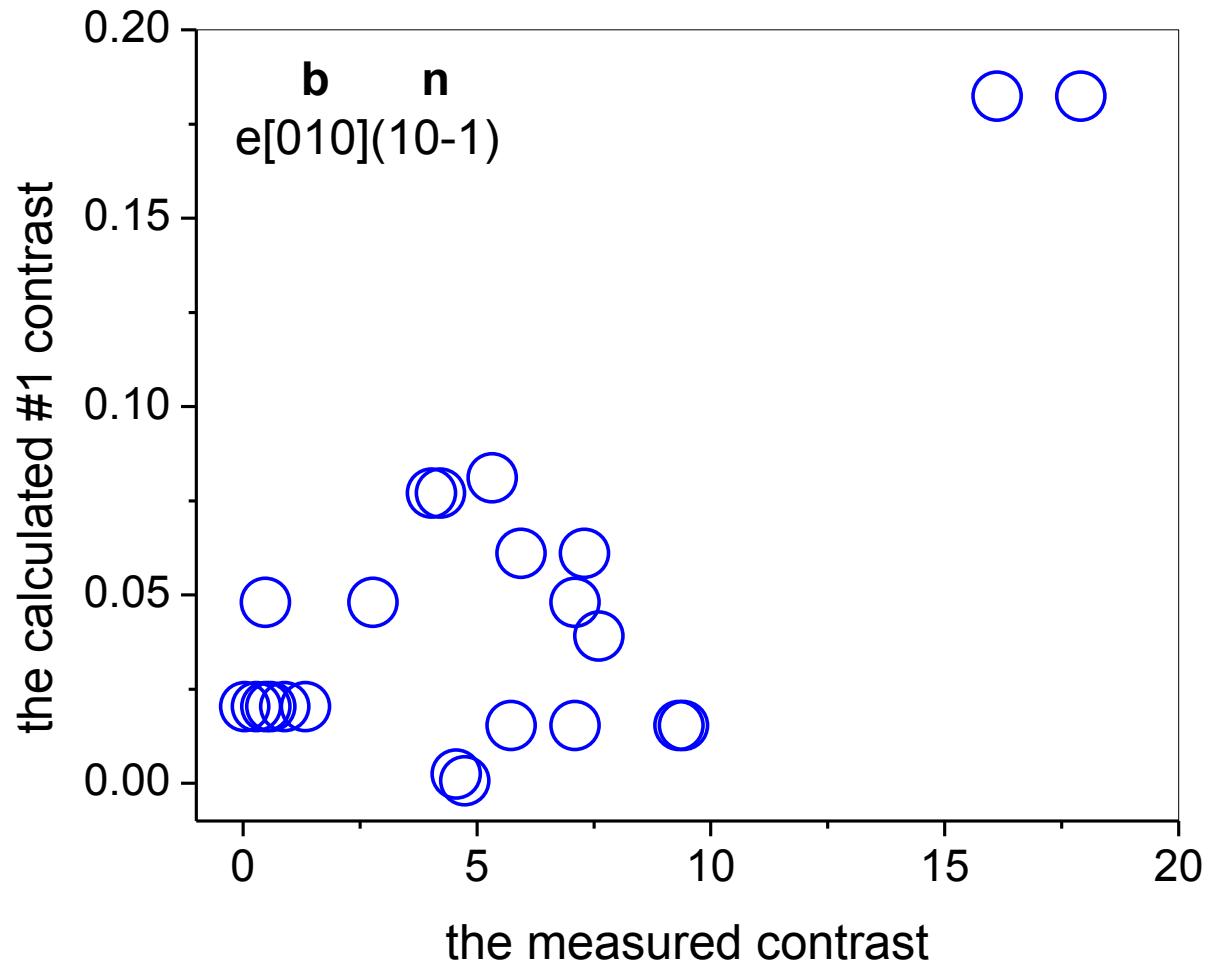


alternative illustration  
of matching  
the measured and calculated contrasts

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the measured and calculated contrasts in a grain of CoTi

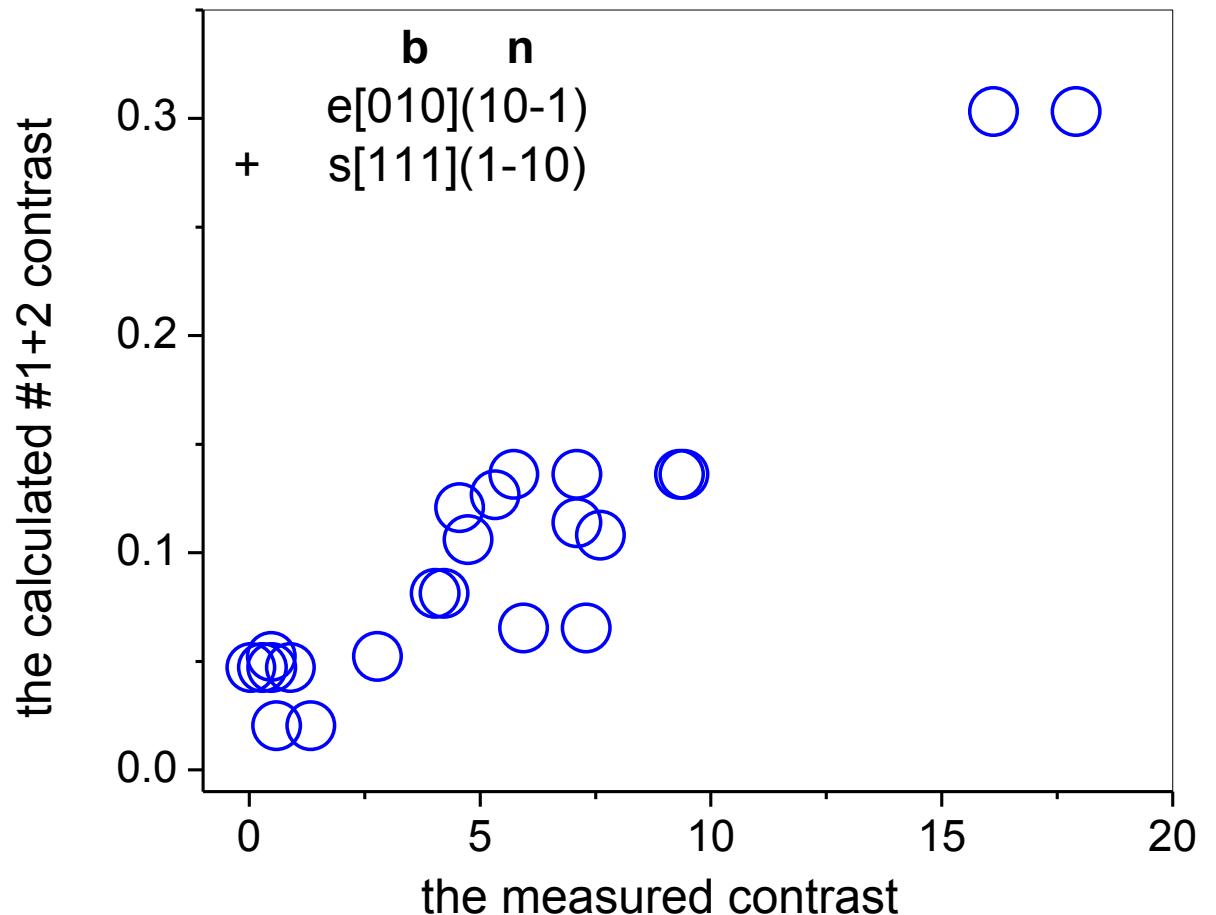
## #1 soft-mode dislocation: $f(\rho)=0.51$



# the measured and calculated contrasts in a grain of CoTi

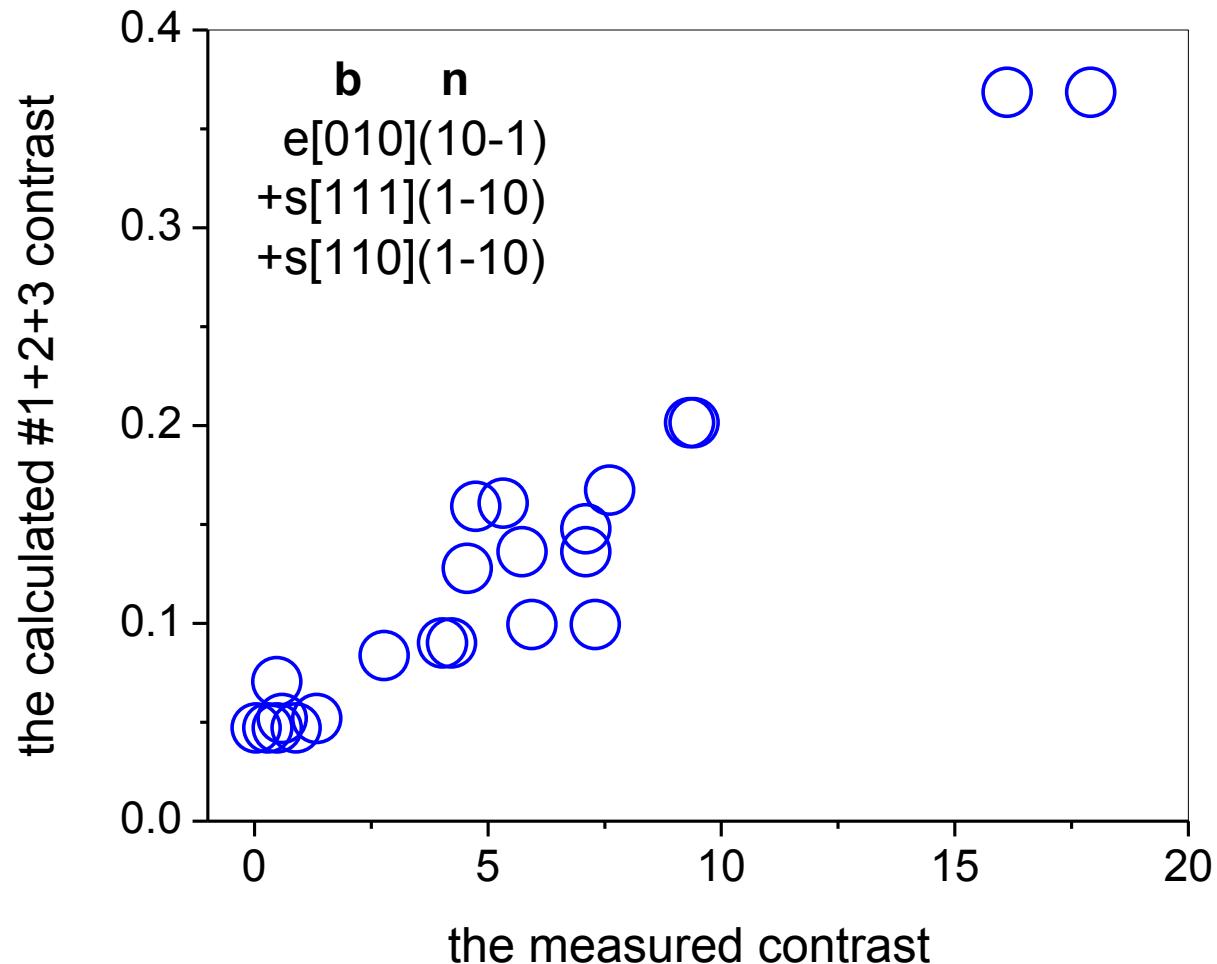
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- #1 soft-mode dislocation:  $f(\rho)=0.51$
- #2 hard-mode dislocation:  $f(\rho)=0.26$



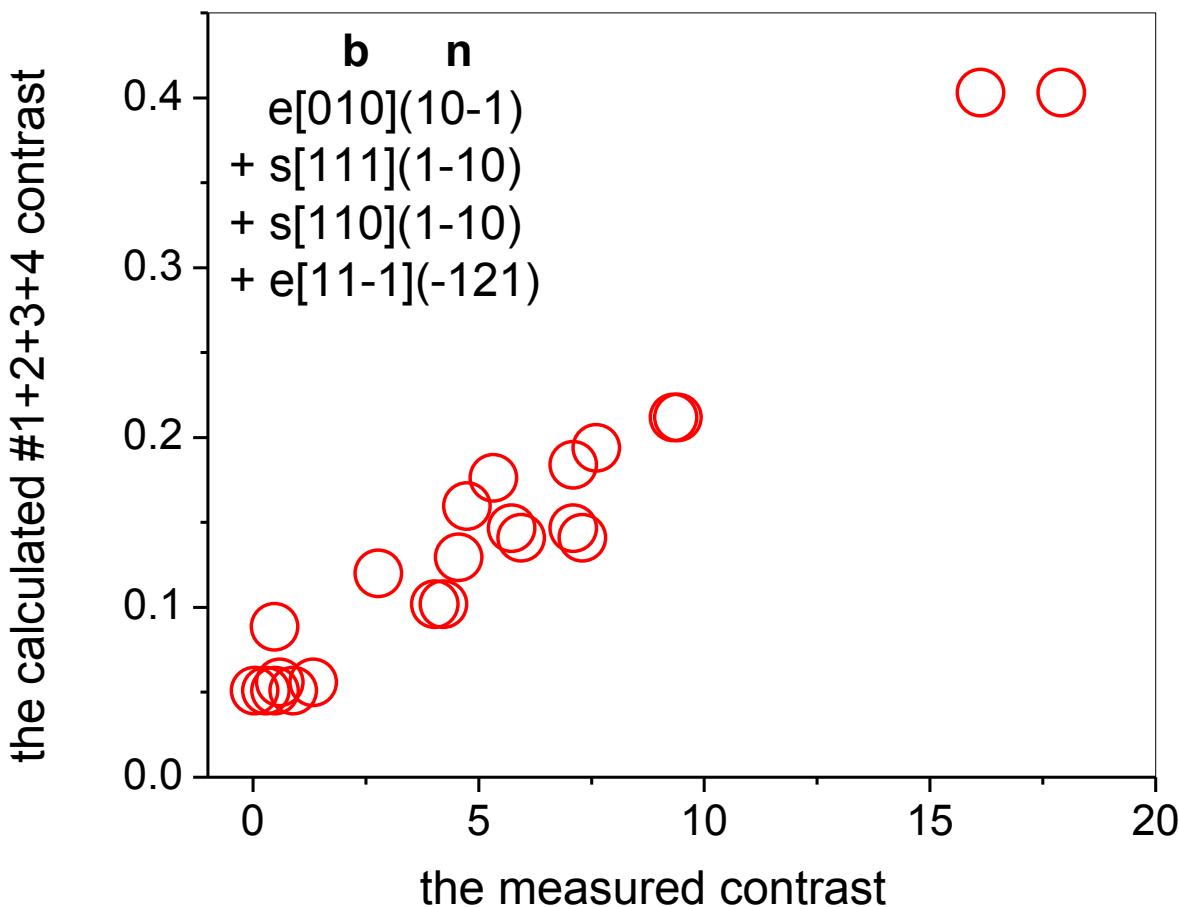
# the measured and calculated contrasts in a grain of CoTi

- #1 soft-mode dislocation:  $f(\rho)=0.51$
- #2 hard-mode dislocation:  $f(\rho)=0.26$
- #3 hard-mode dislocation:  $f(\rho)=0.18$



# the measured and calculated contrasts in a grain of CoTi

- #1 soft-mode dislocation:  $f(\rho)=0.51$
- #2 hard-mode dislocation:  $f(\rho)=0.26$
- #3 hard-mode dislocation:  $f(\rho)=0.18$
- #4 hard-mode dislocation:  $f(\rho)=0.05$



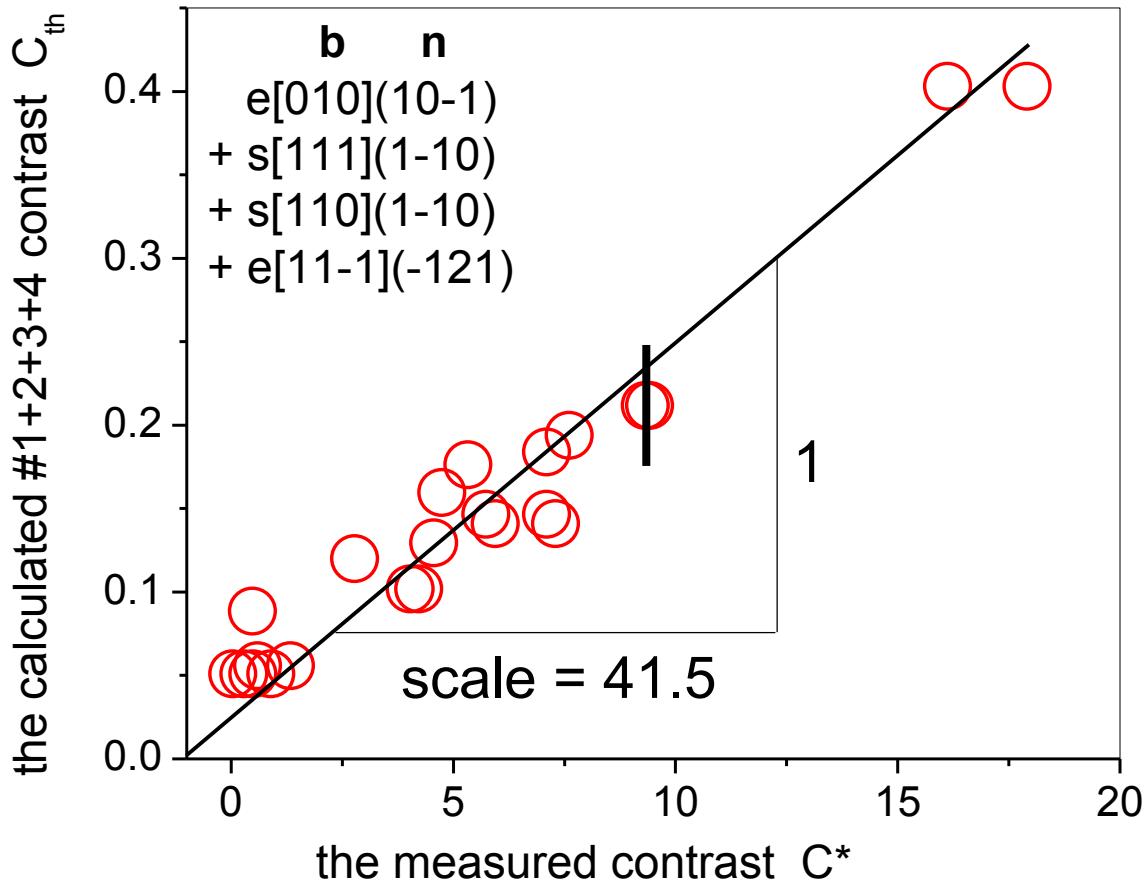
# the measured and calculated contrasts in a grain of CoTi

#1 soft-mode dislocation:  $f(\rho)=0.51$

#2 hard-mode dislocation:  $f(\rho)=0.26$

#3 hard-mode dislocation:  $f(\rho)=0.18$

#4 hard-mode dislocation:  $f(\rho)=0.05$

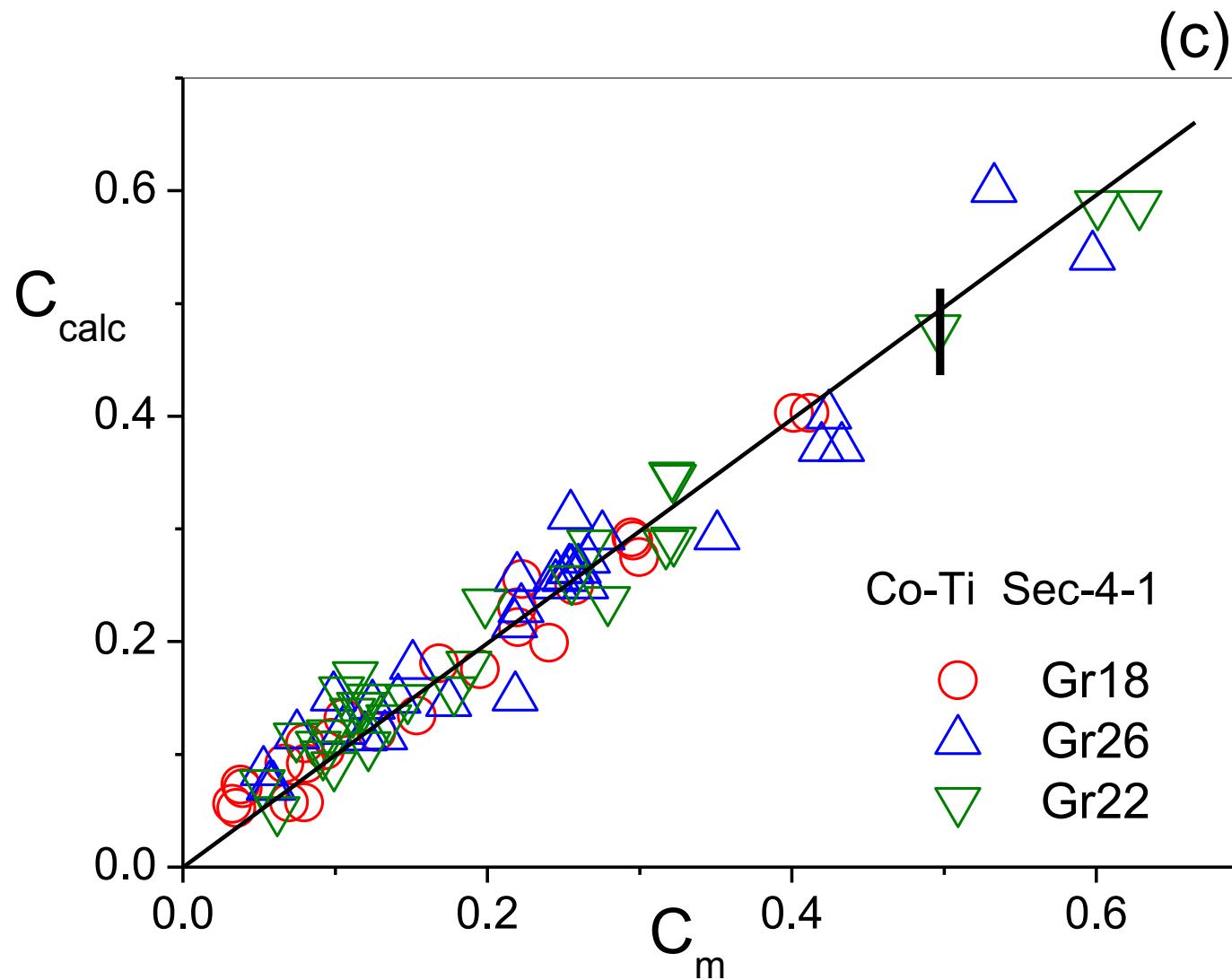


$$\rho C_{th} = \rho^* C^*$$

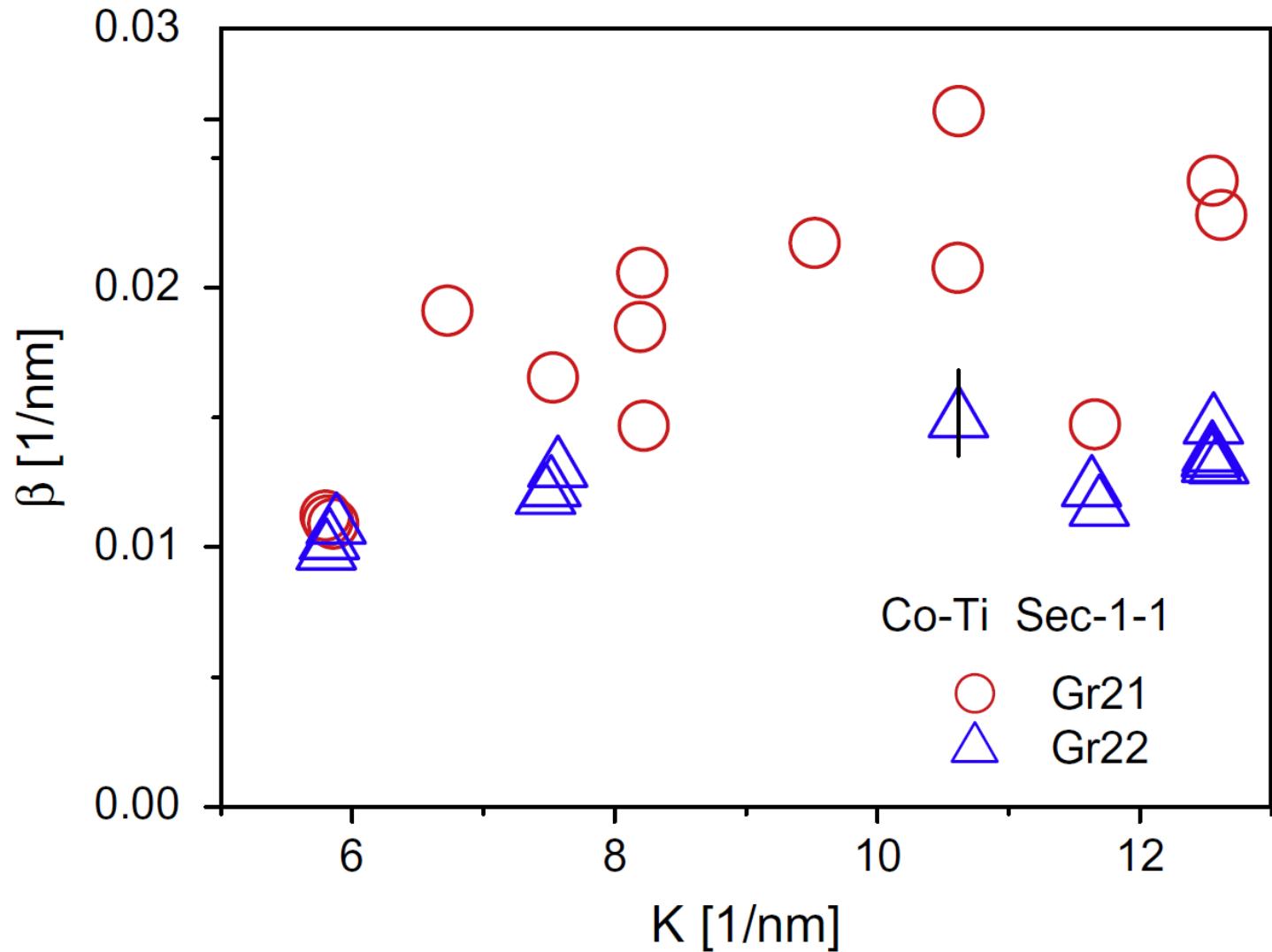
$$\rho = \frac{\rho^* C^*}{C_{th}}, \text{ scale} = \frac{C^*}{C_{th}}$$

the measured and calculated contrast in three grains of CoTi

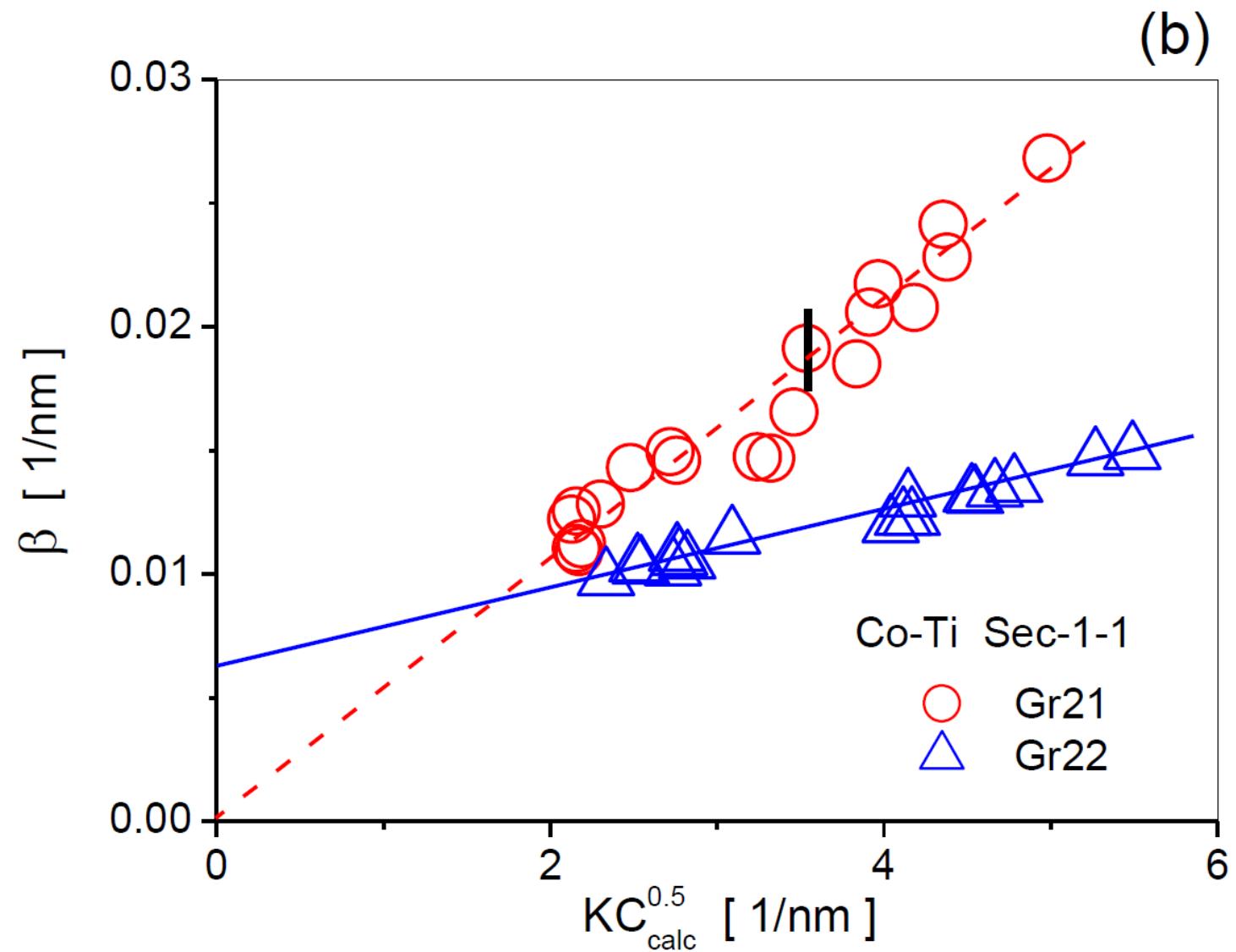
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# Williamson-Hall plots for two grains in CoTi

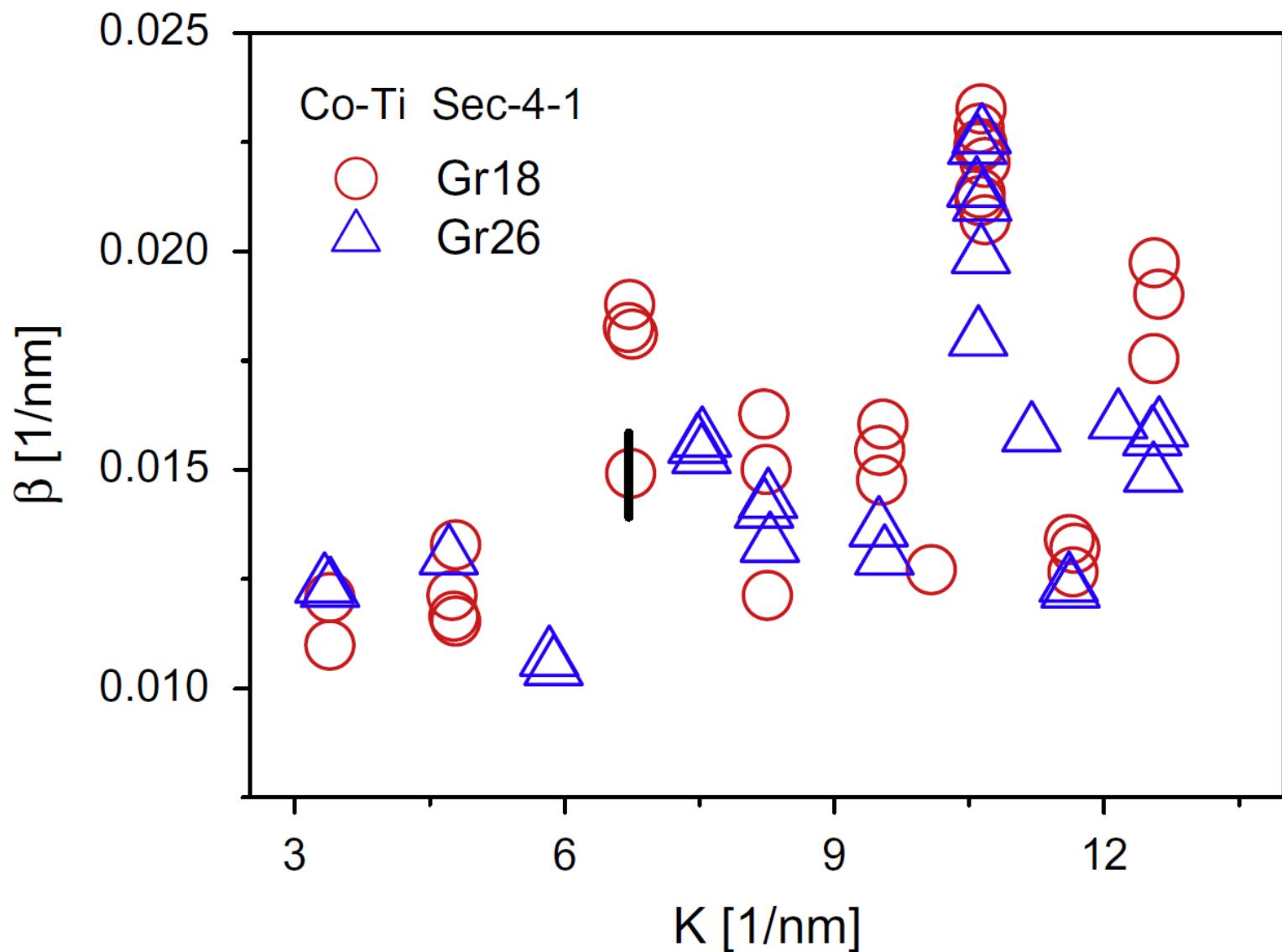


# *modified* Williamson-Hall plots for two grains in CoTi

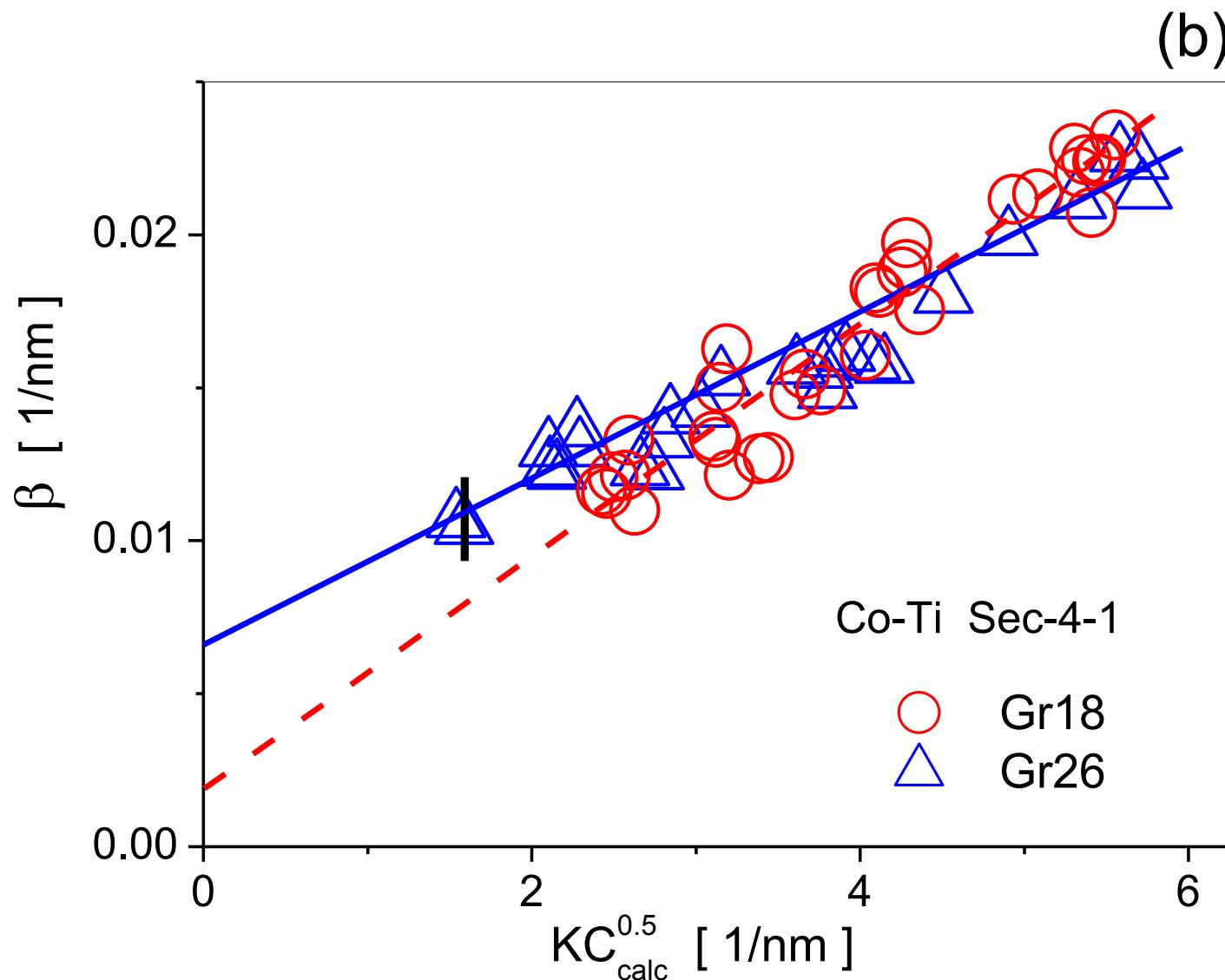


# Williamson-Hall plots for two grains in CoTi

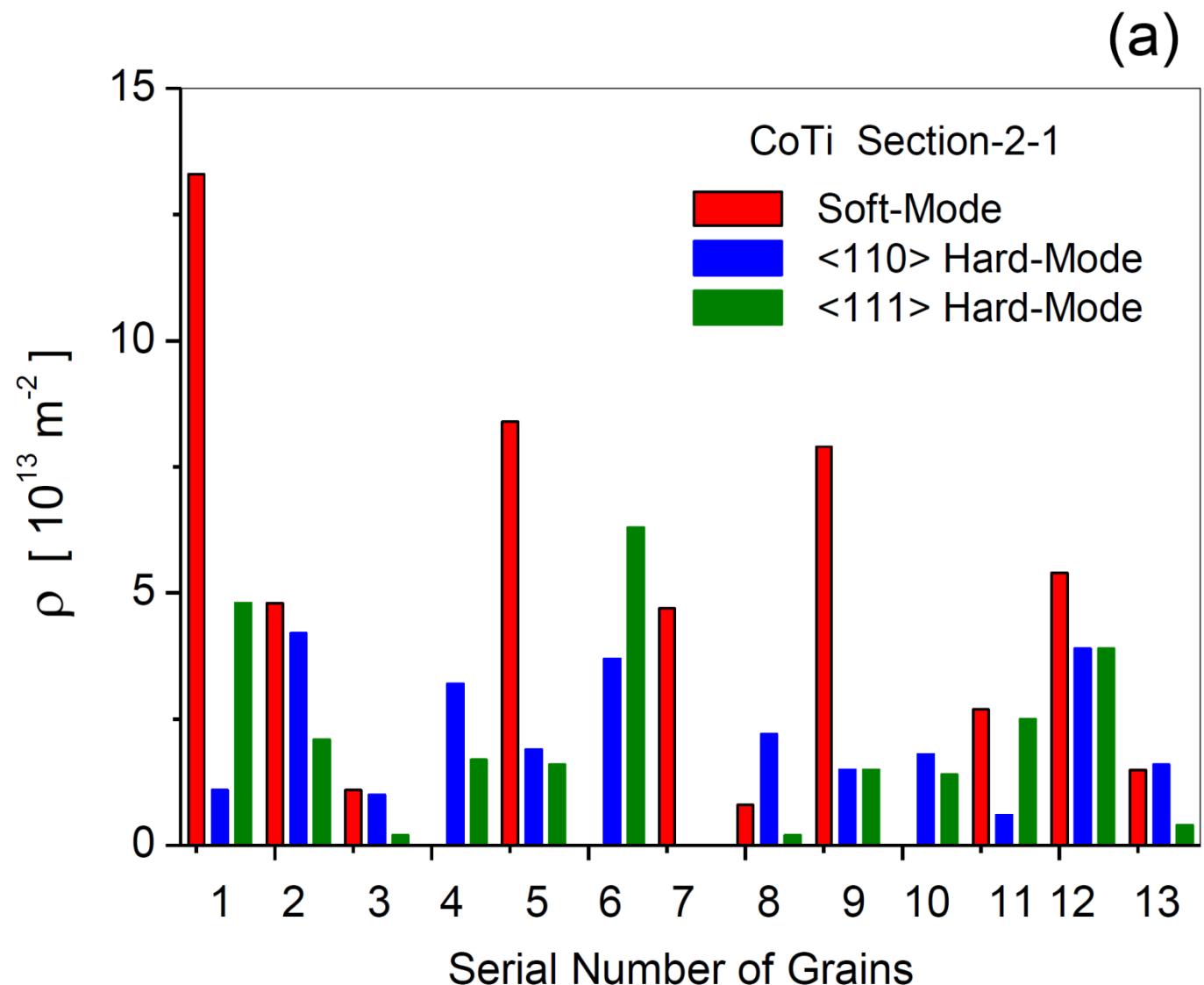
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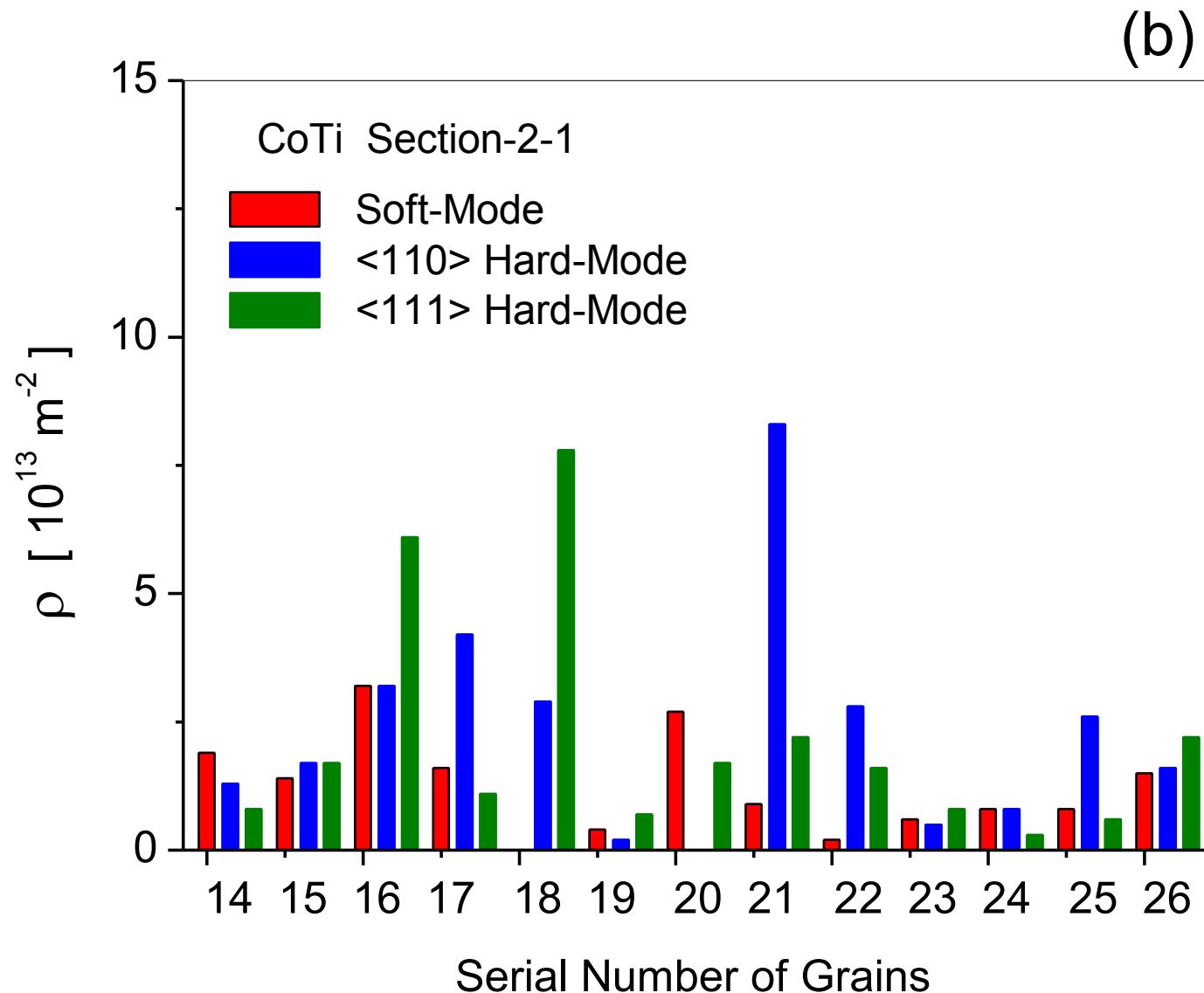
# *modified* Williamson-Hall plots for two grains in CoTi



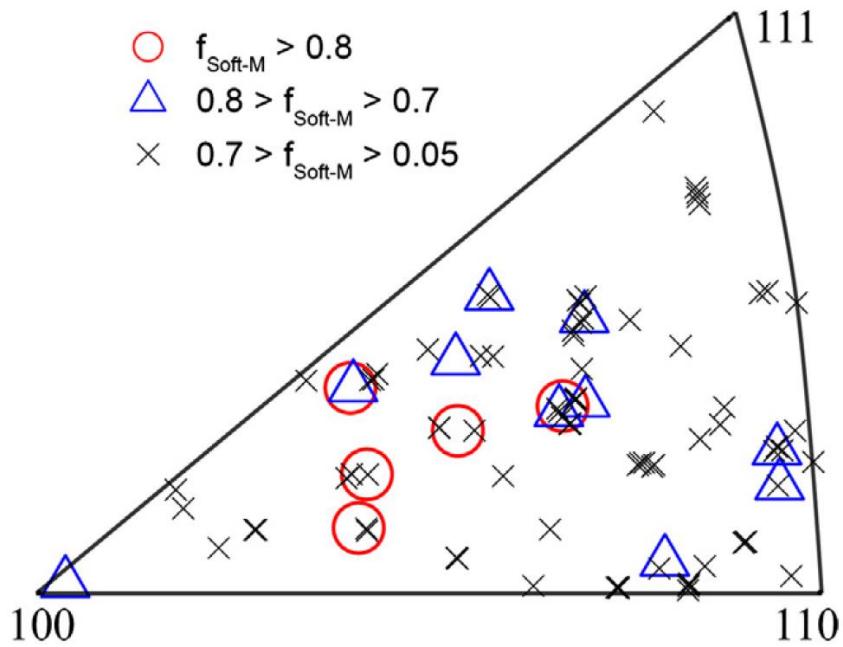
# dislocation densities in 1 to 13 of 123 investigated grains in CoTi



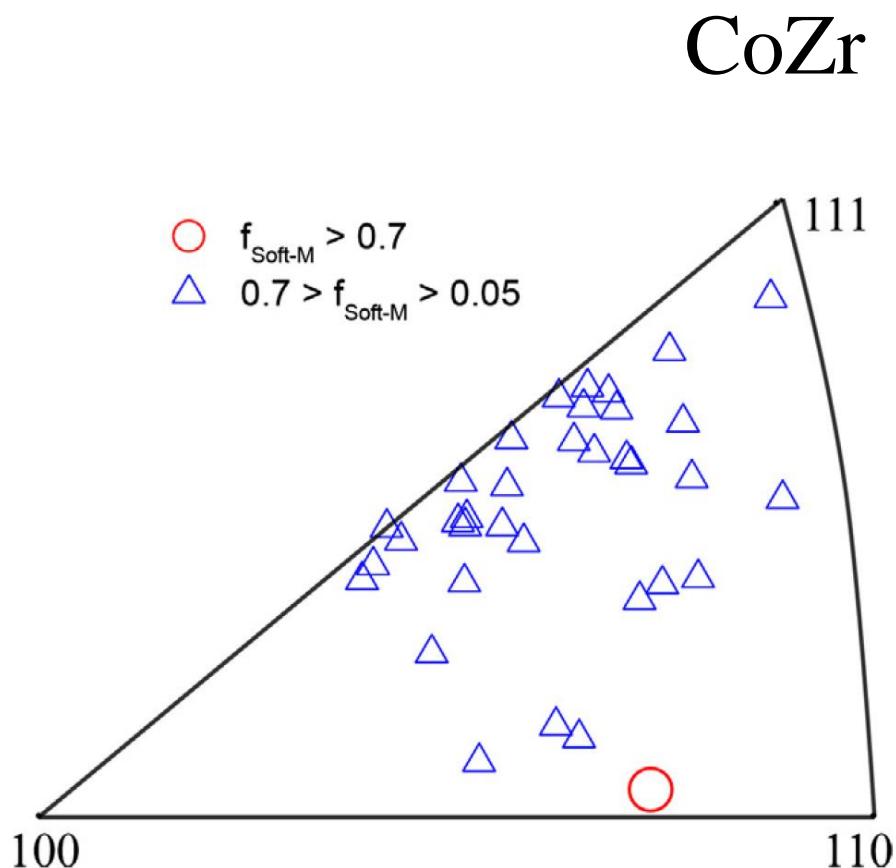
# dislocation densities in 13 to 26 of 123 investigated grains in CoTi



# correlation with Schmid's law ?

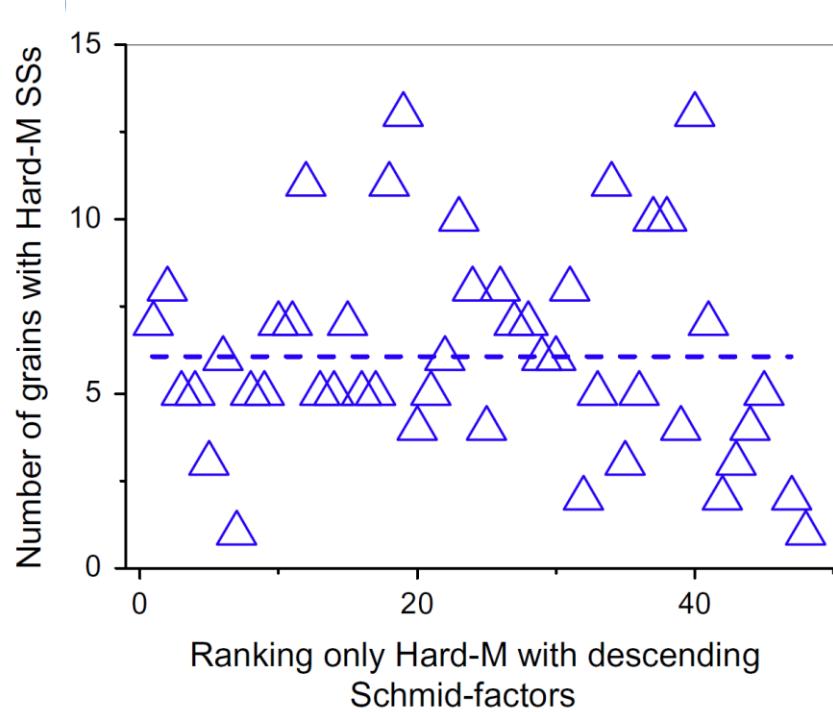
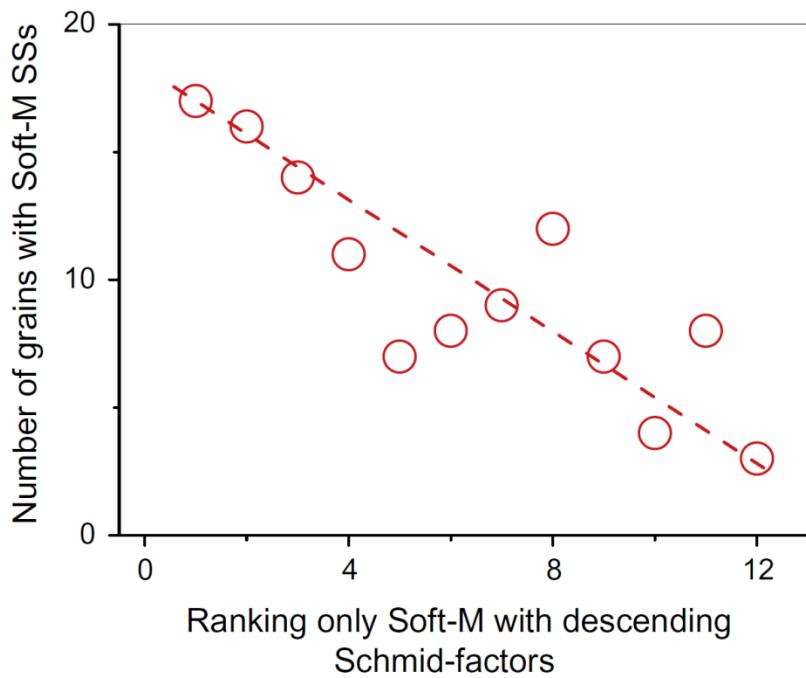


CoTi



CoZr

# correlation with Schmid law ?



hard-modes make an essential contribution  
to plasticity when the two alloys are polycrystalline

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good correlation with previous TEM results

much better statistics

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more can be done ....

*Thank you*