Effect of internal stress on peak position: problems and opportunities

HCP materials

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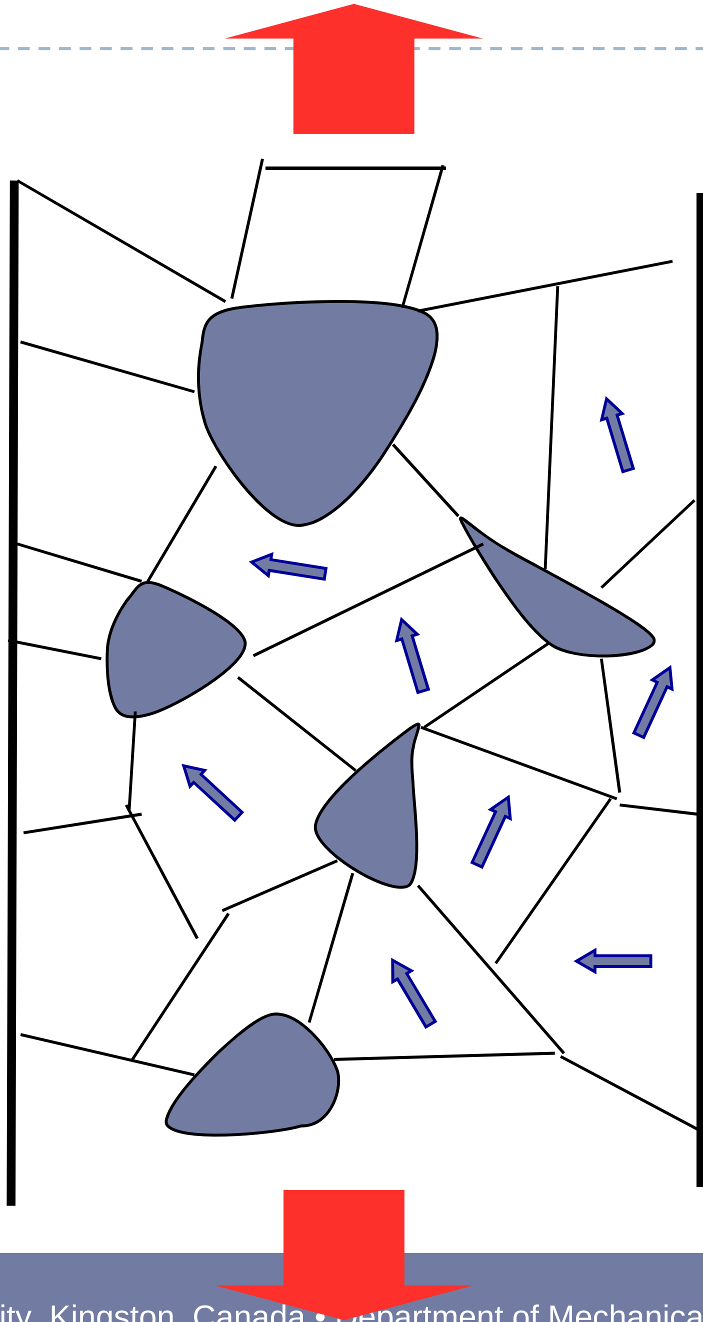
APD IV

Outline

- Interphase and intergranular stresses
- Effect on Rietveld refinement
- Opportunities to determine crystal properties
  - Impact of high energy neutrons on structure
  - Use of internal stresses to determine operating deformation modes
- Conclusions

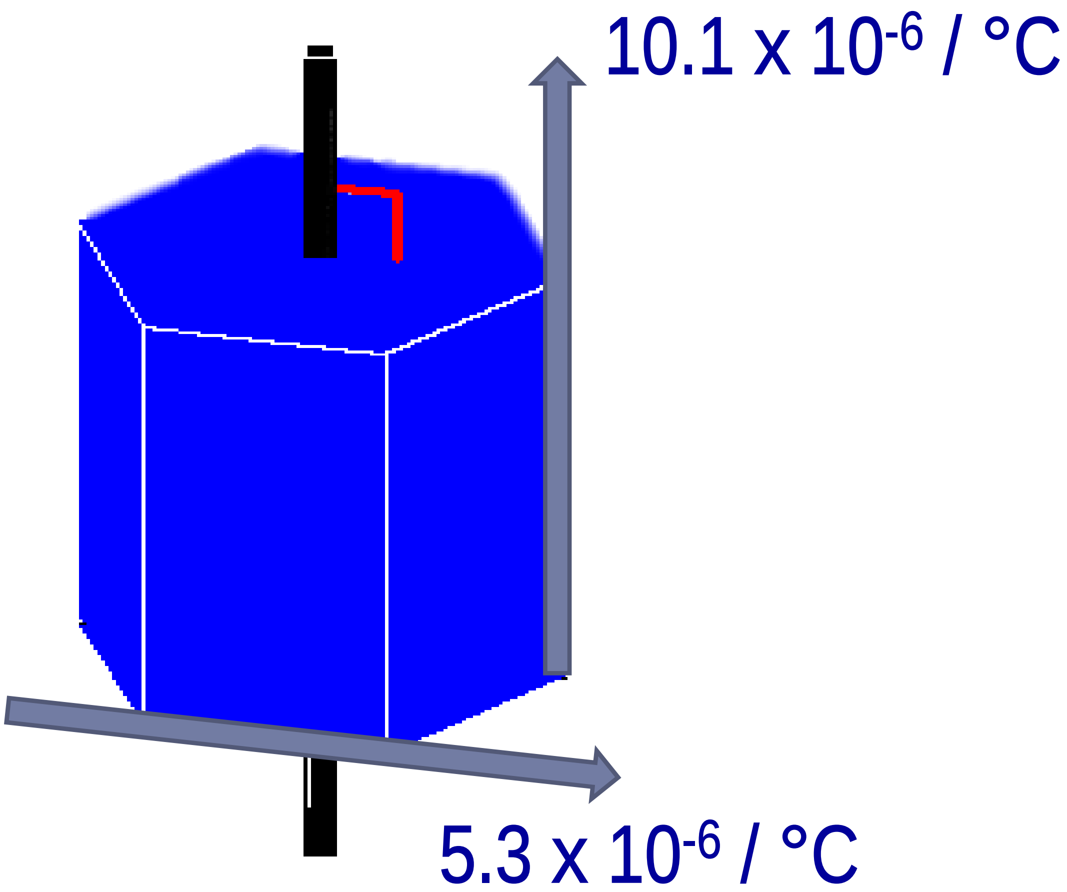
Constraints on phases and grains

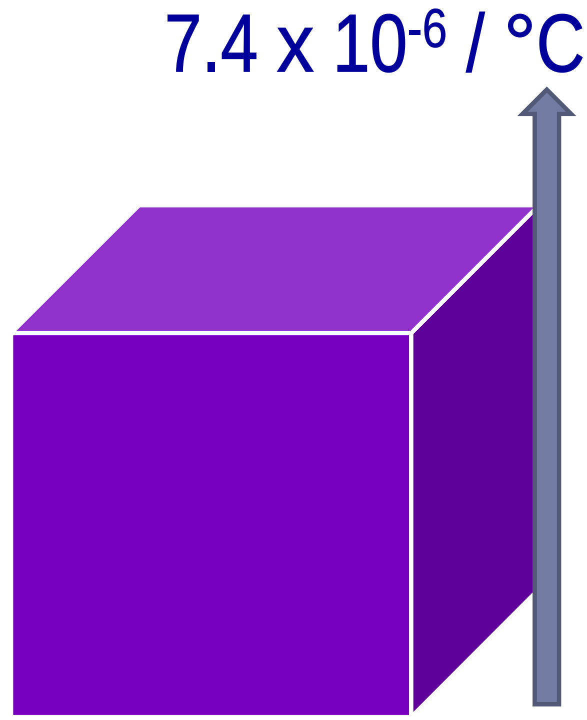


What is impact of interactions between phases and different grain orientations on stress / deformation?

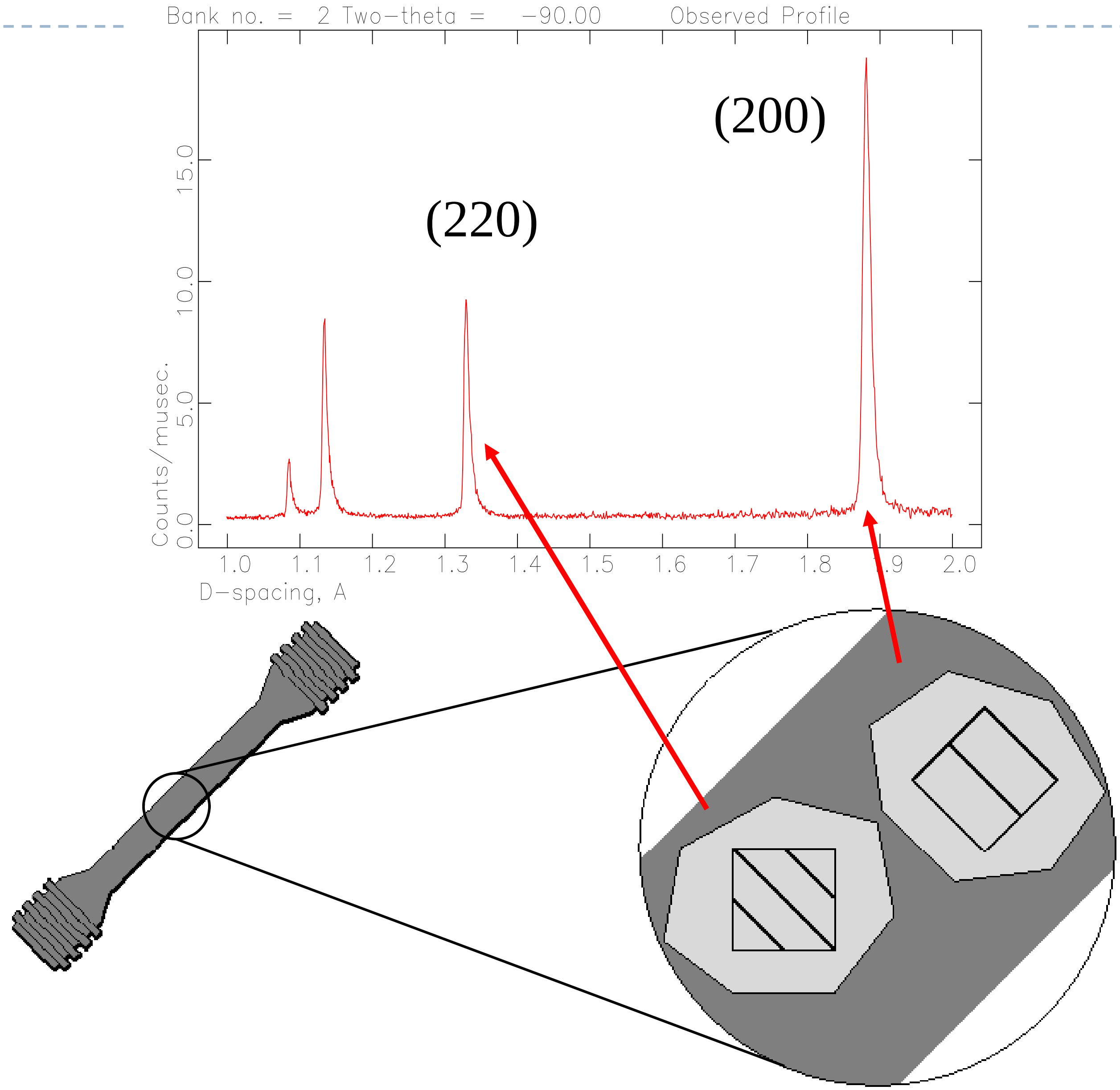
Thermal anisotropy

- e.g. Zirconium-Niobium alloy; two phase HCP and BCC





Diffraction measurement of elastic strain



$$\lambda = 2d\sin\theta$$

$$\varepsilon = \Delta d / d$$

Diffraction peaks measured *in specific direction in sample*

(cubic example shown)

Relationship d-*hk.l* and a & c

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

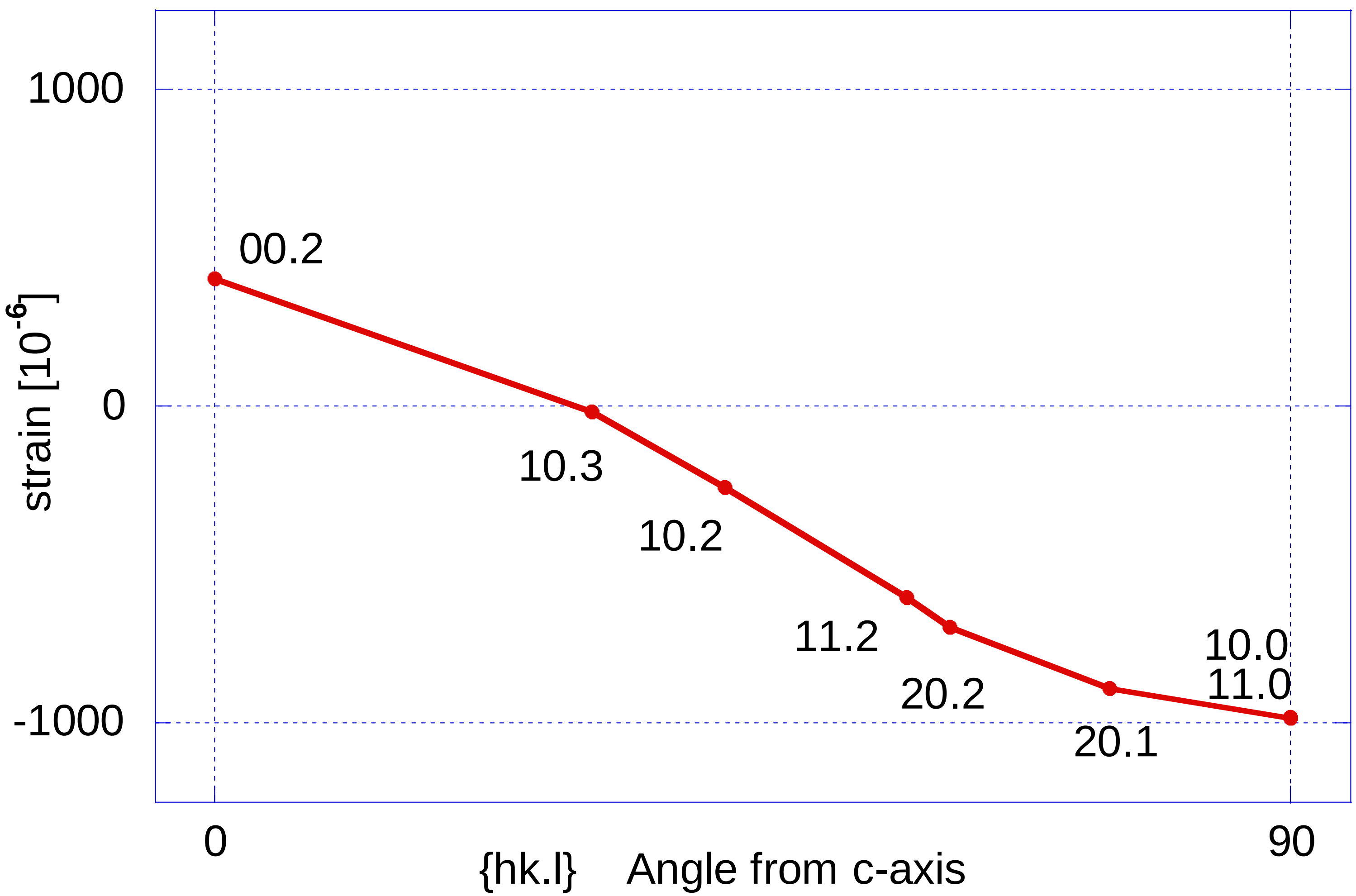
cubic

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2}$$

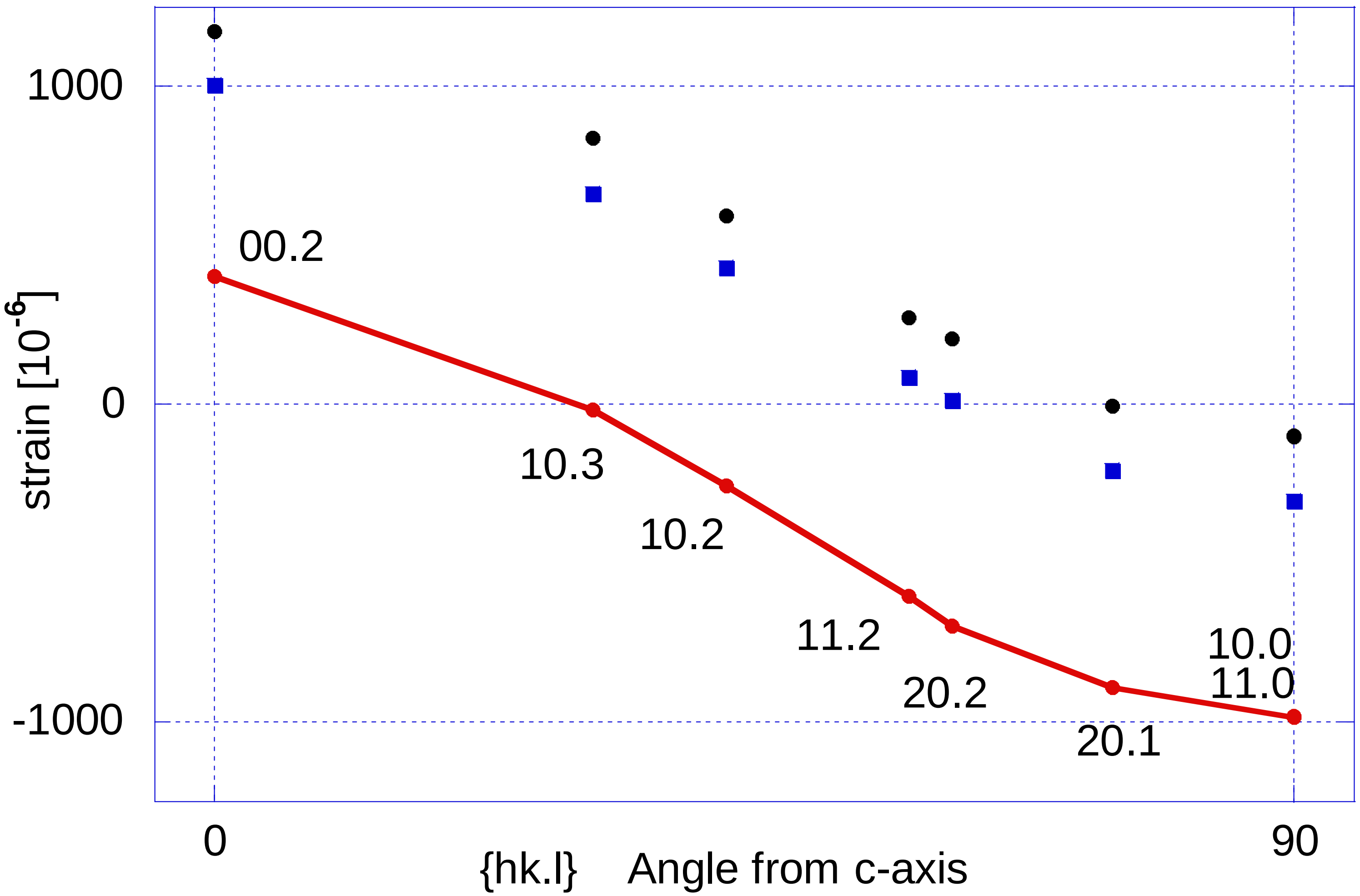
hexagonal

Will hold for single crystal, a single-crystal powder, and a “stress-free” polycrystal

Deviation from ideal relation due to: thermal stresses



Deviation from ideal relation due to: thermal stresses – effect of texture

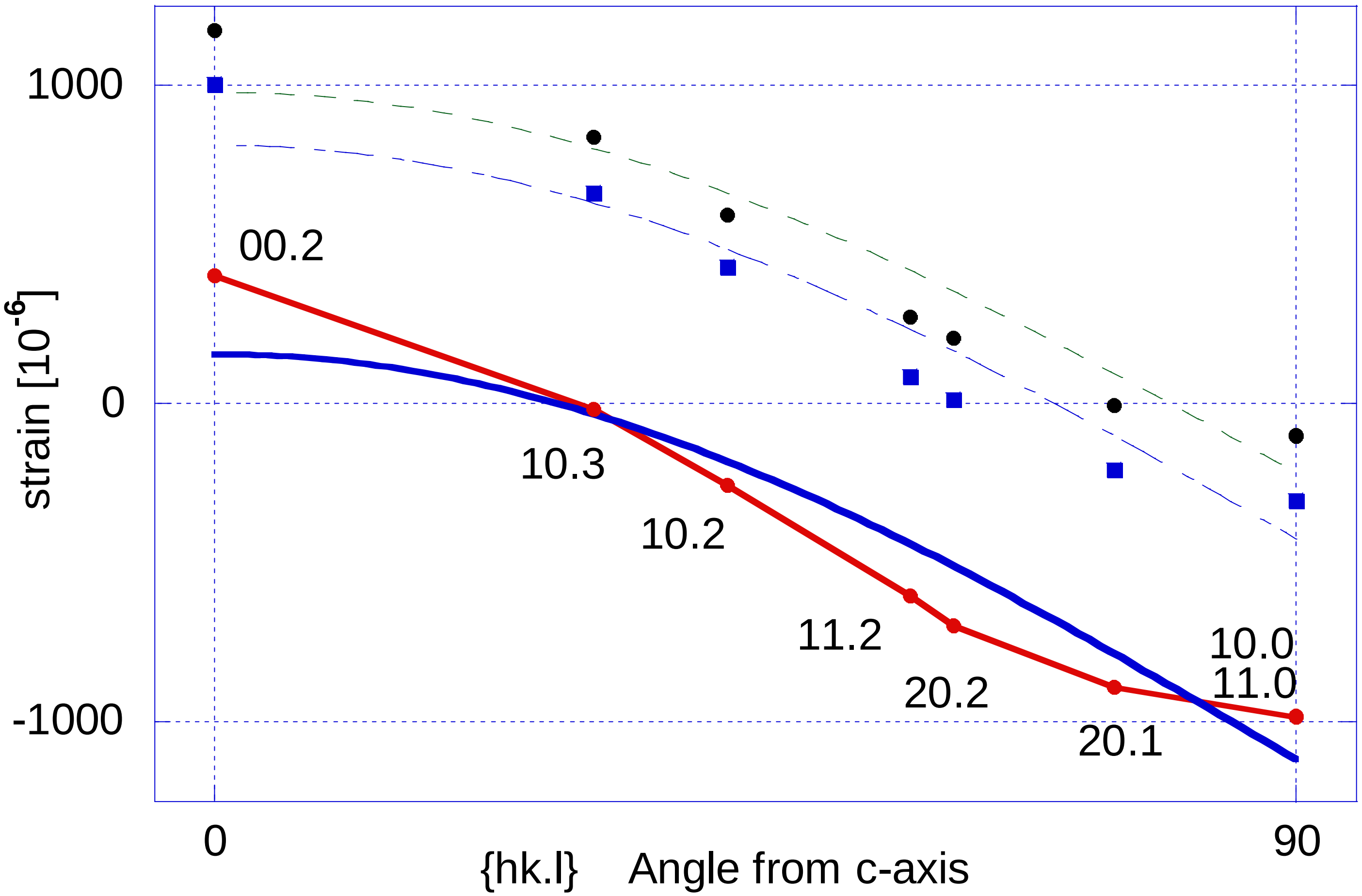


How to handle in Rietveld refinement?

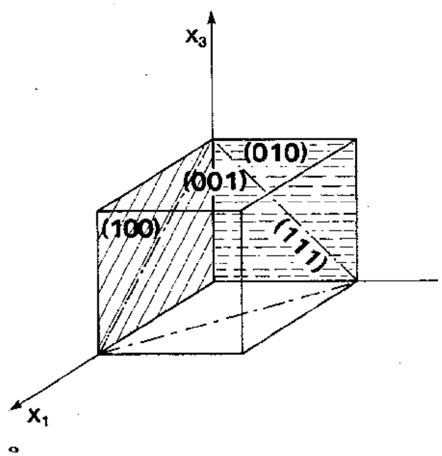
e.g. GSAS:

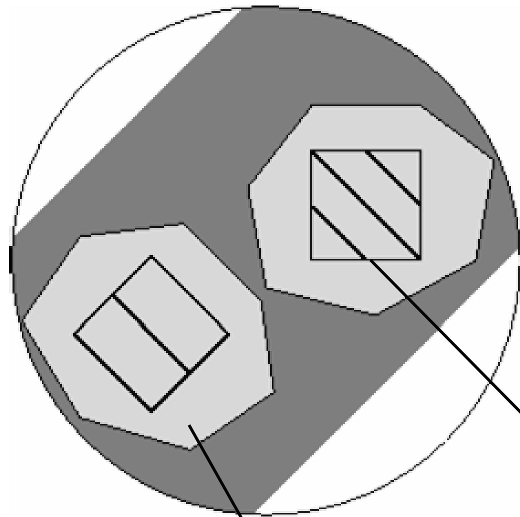
“RSTR” – isotropic strain parameter,

“RSTA” – assumes cosine variation with angle from c-axis

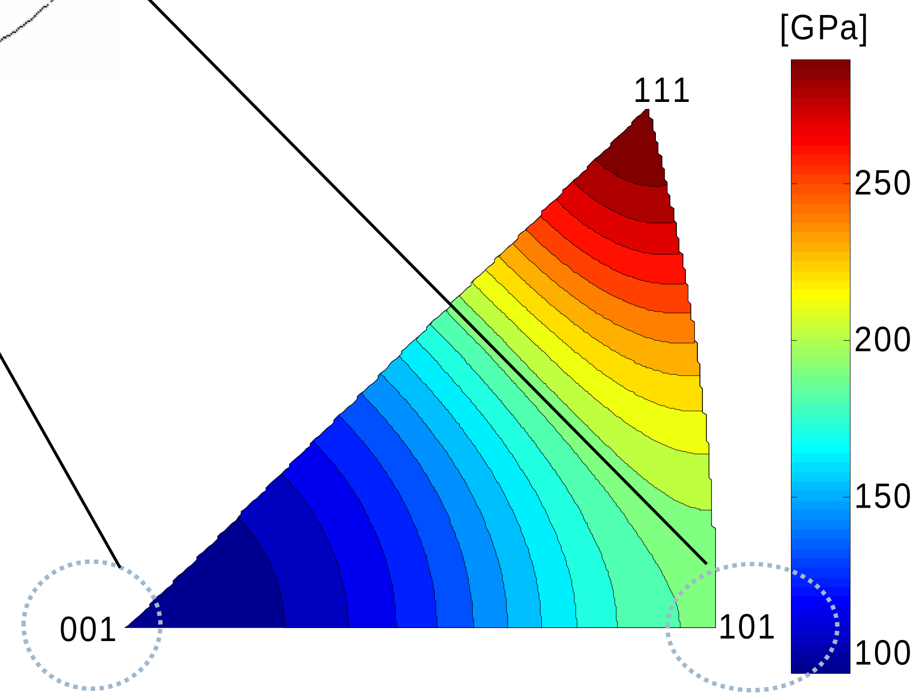


Elastic anisotropy

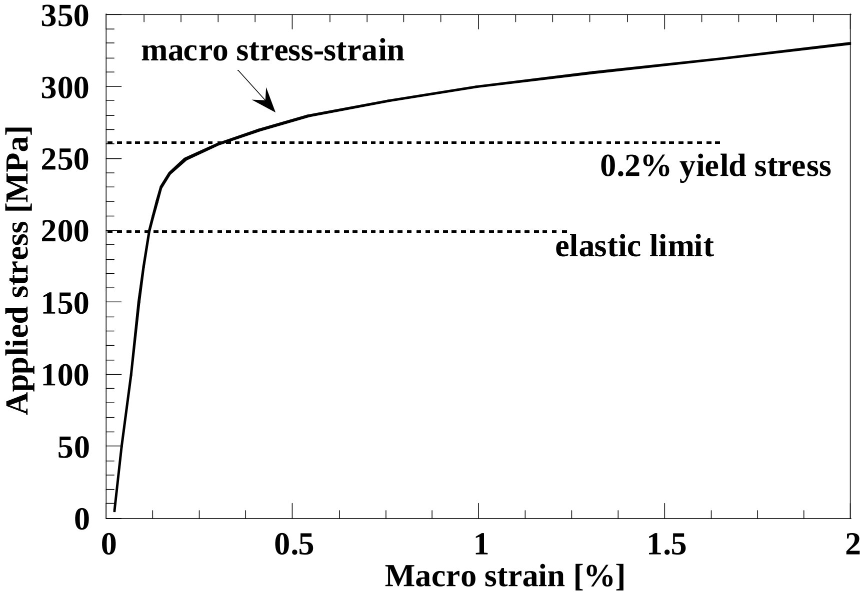




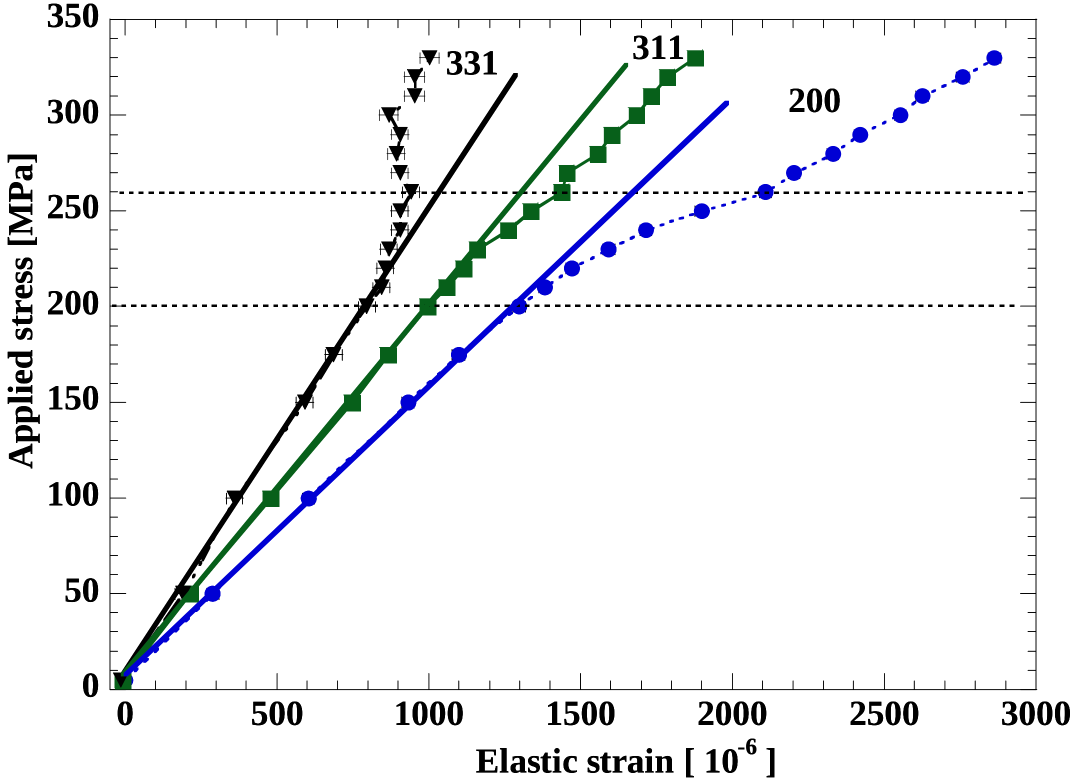
Elastic anisotropy; different stiffnesses in different crystallographic directions



Plastic anisotropy

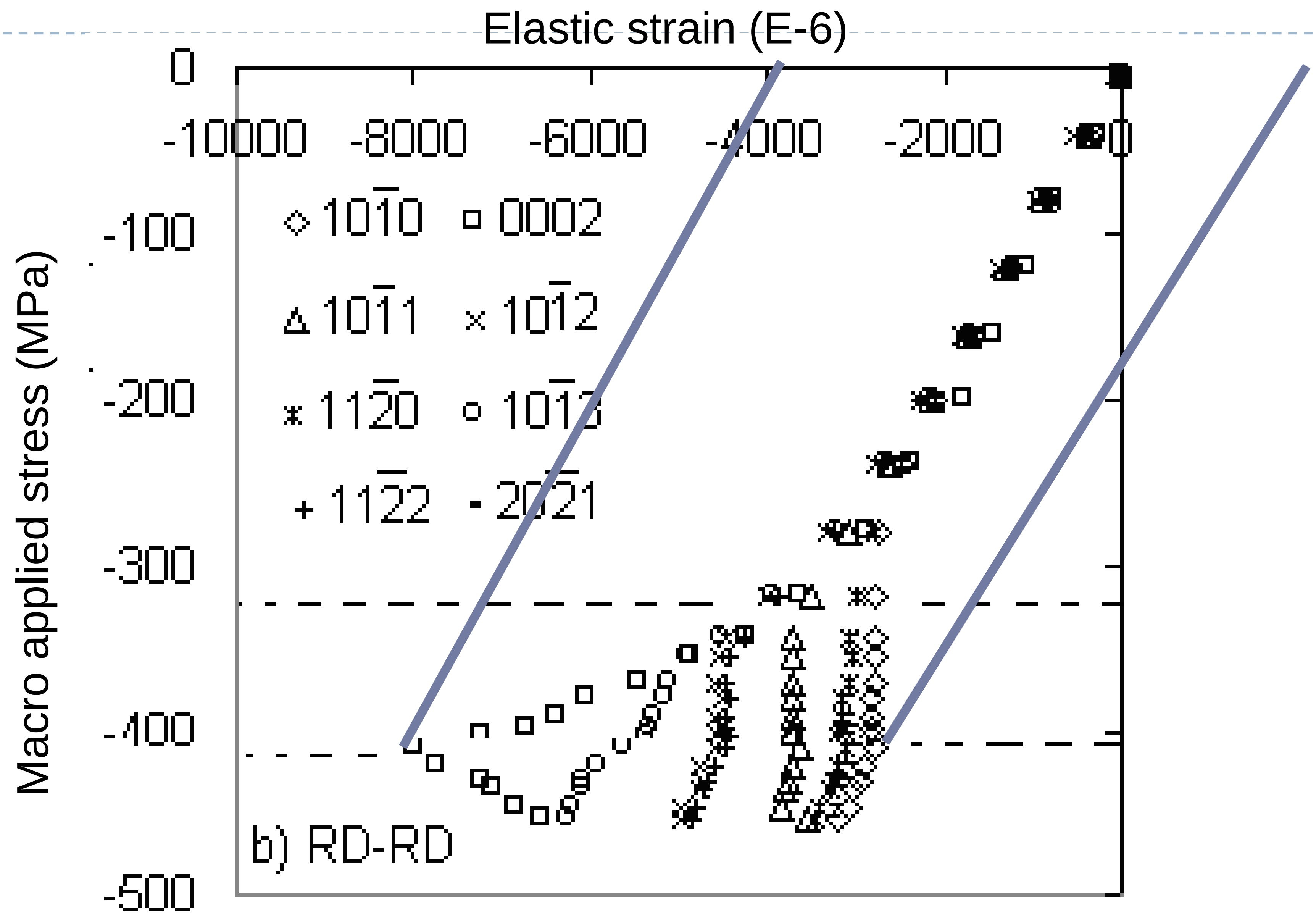


Plastic anisotropy;
introduces non-linearity

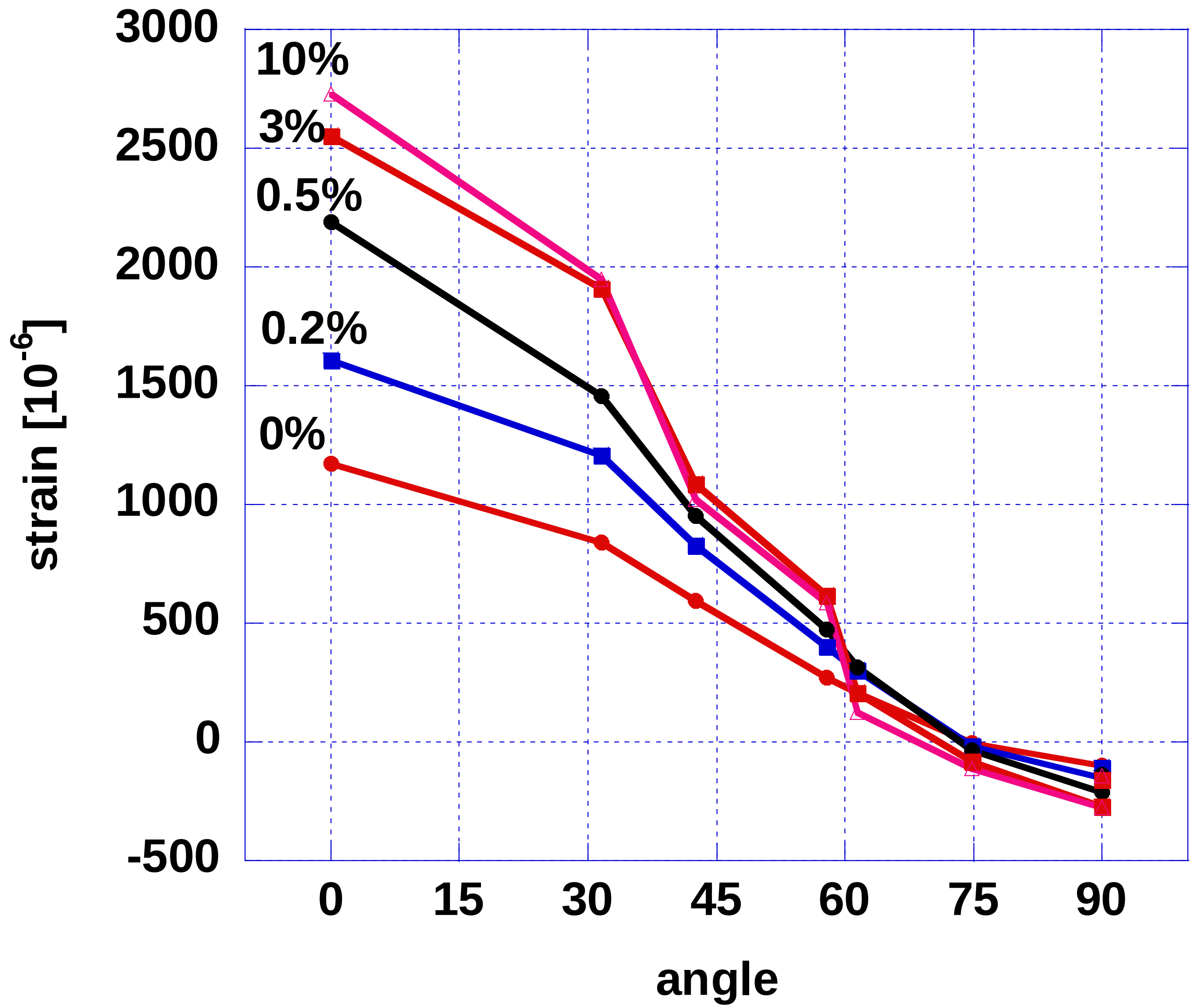


fcc structure
{111}<110> slip

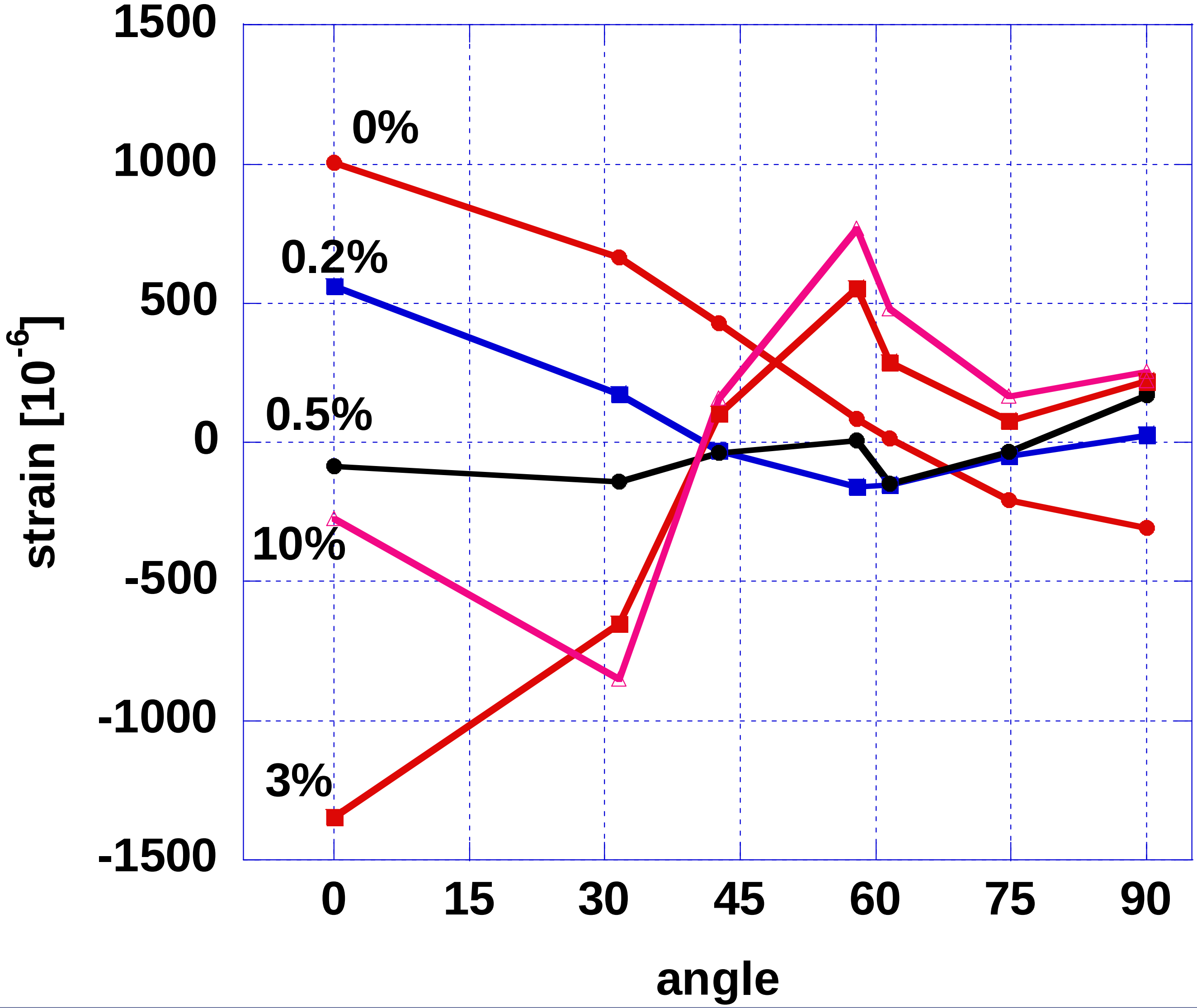
Examples of intergranular strains in Zr



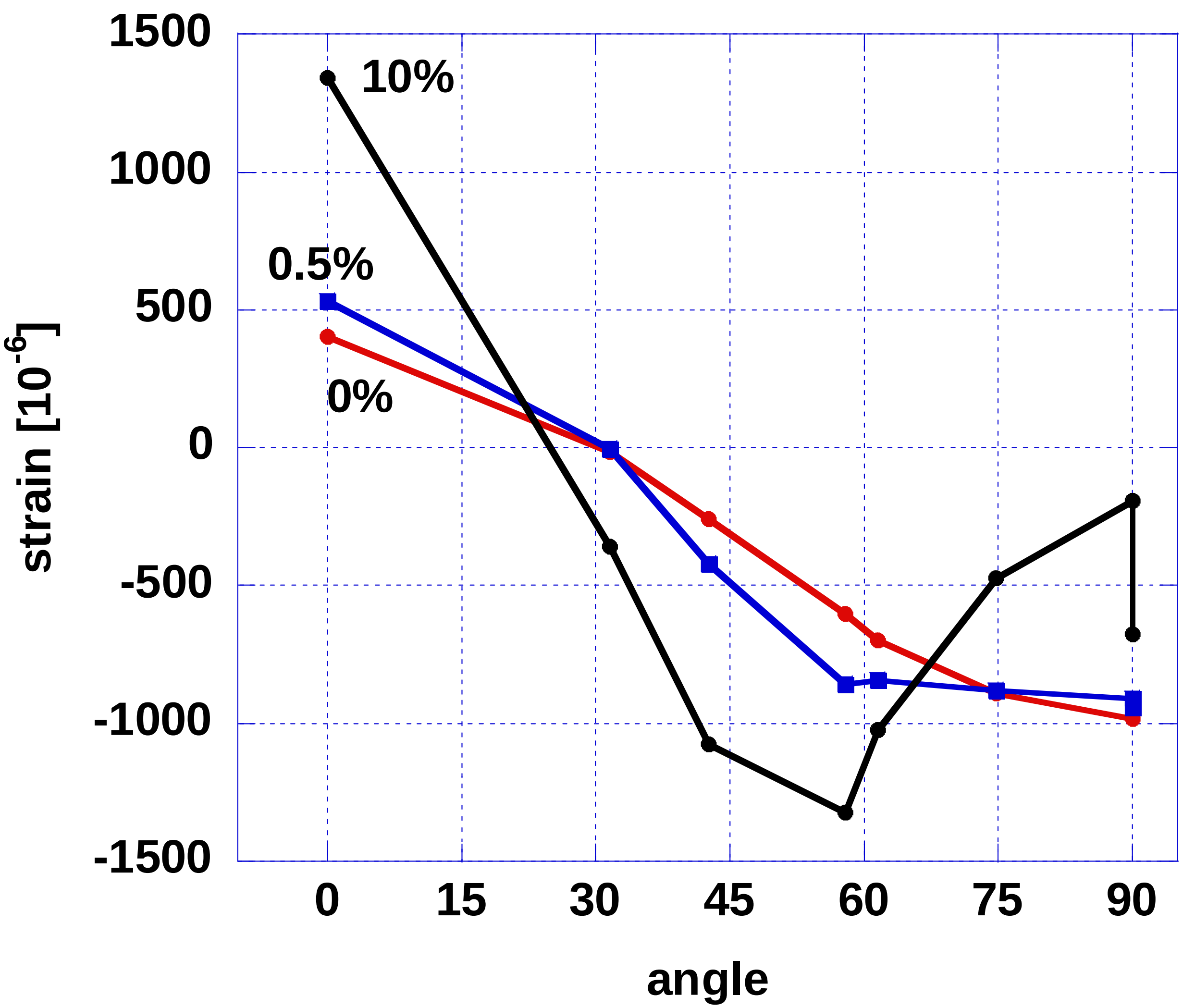
Deviation from ideal relation due to: plasticity induced stresses



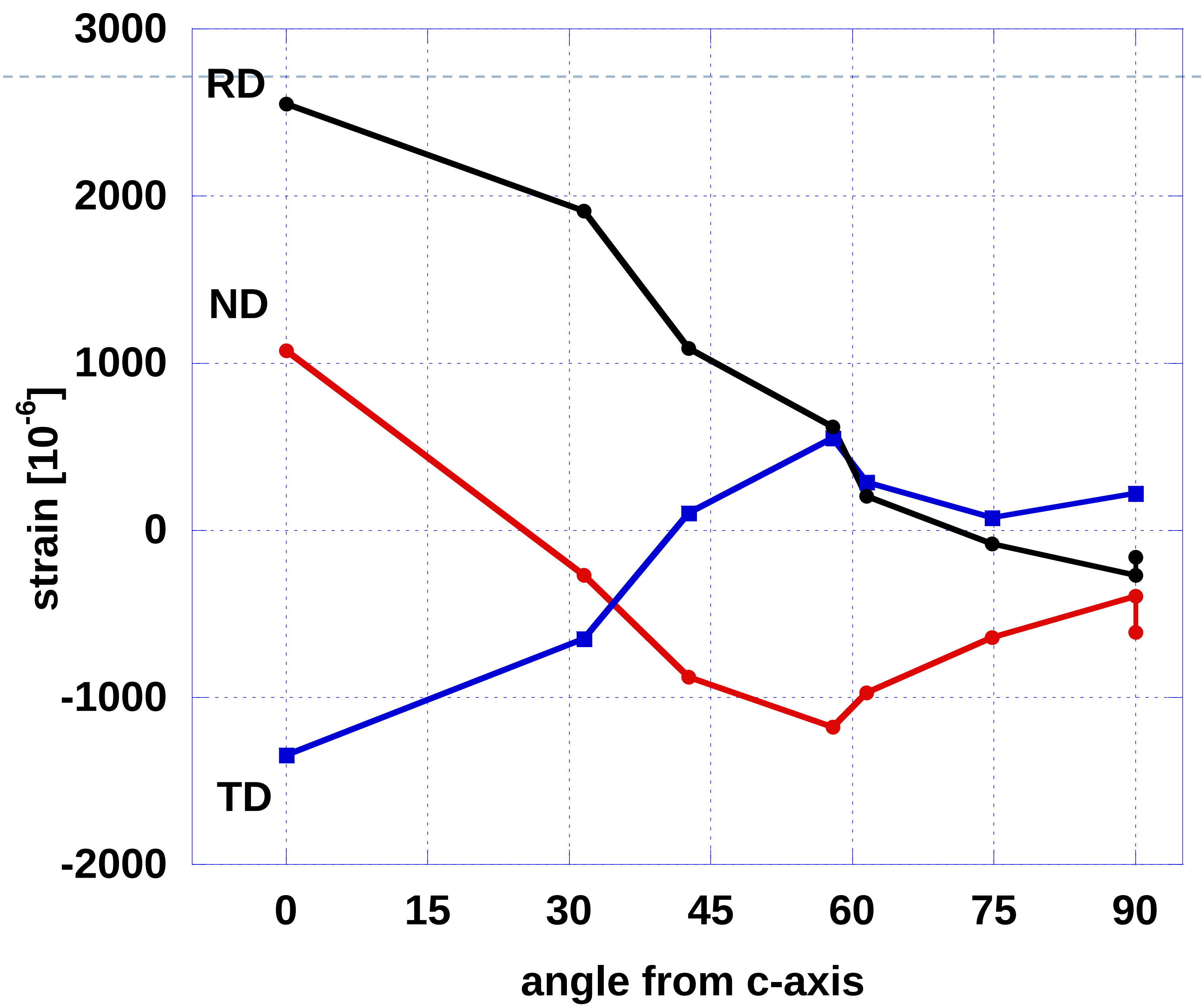
Deviation from ideal relation due to: plasticity induced stresses – response depends on texture



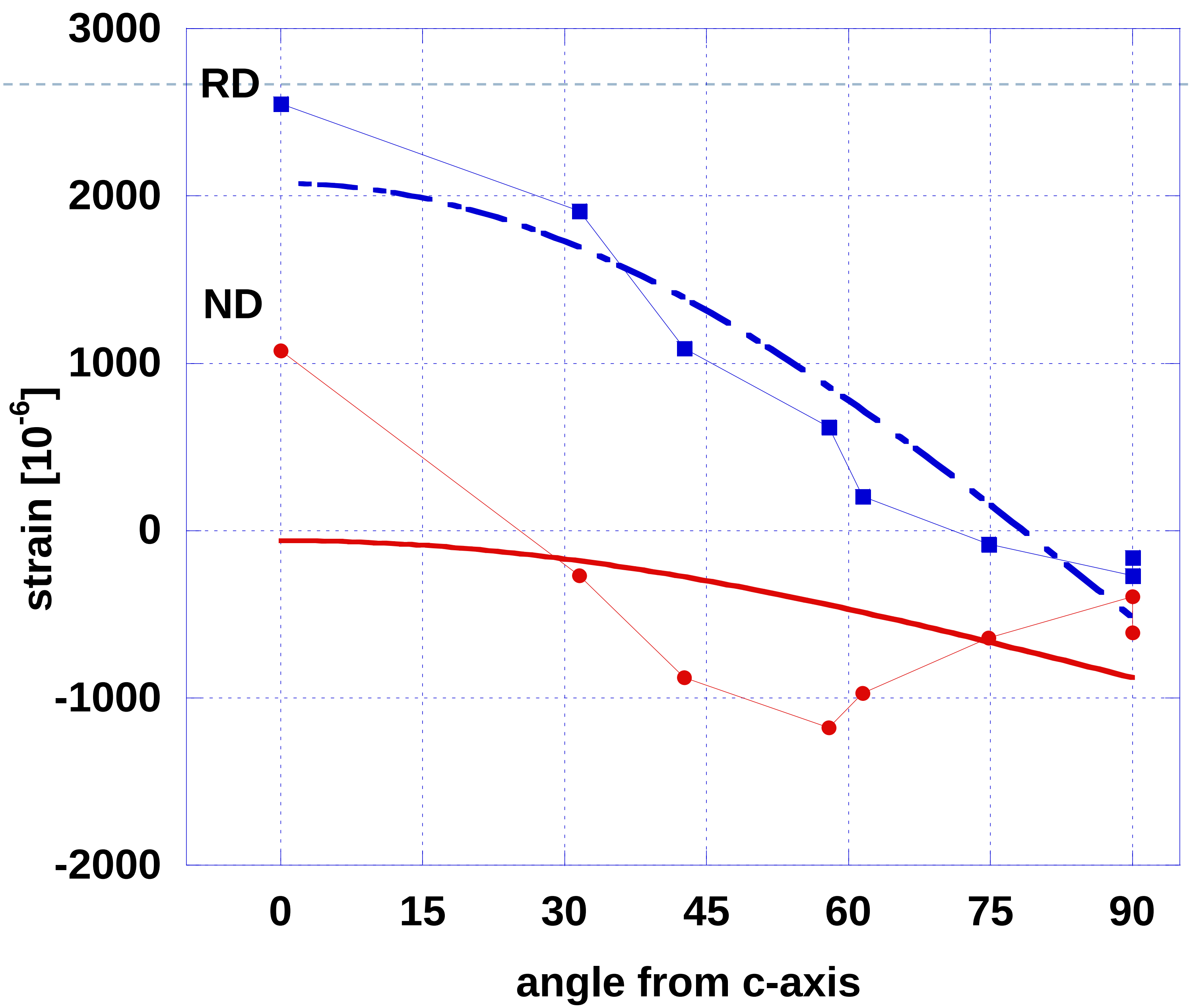
Deviation from ideal relation due to: plasticity induced stresses – response depends on texture



After 3% strain



After 3% strain



How to handle in a Rietveld refinement?

Most extreme peaks, typically strain of ~0.001

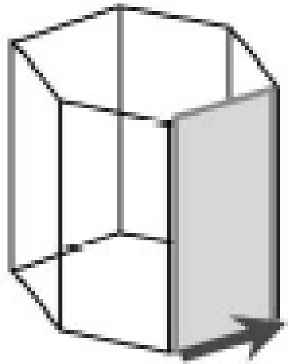
Response differs too far from relation to allow accurate peak width and/or peak intensity fit while maintaining peak positions

e.g. pseudo-anisotropic broadening

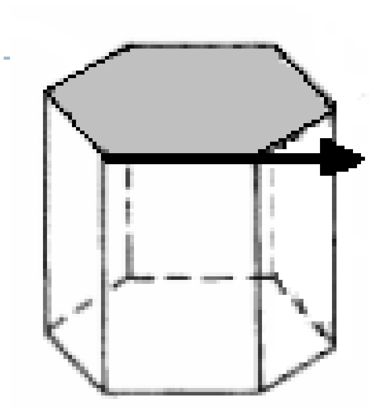
Solution

1) Allow peak positions to vary freely?
2) Incorporate models of plasticity?

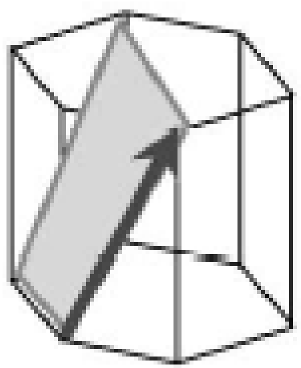
Deformation modes observed in single crystal hcp Zr



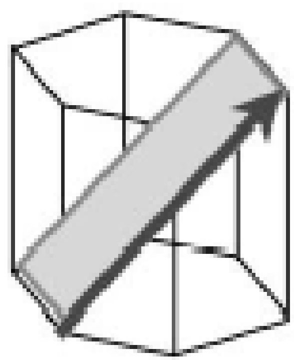
(1010) <1120>
prismatic slip



(0001) < 1120 >
basal slip



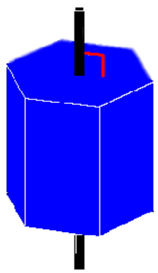
(1011) <1123>
pyramidal slip

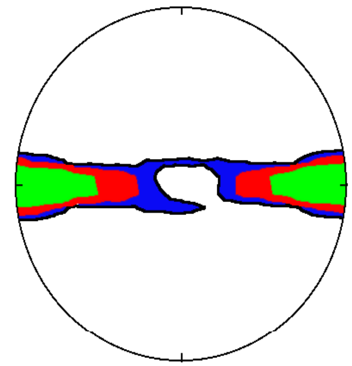


(1012) <1011>
tensile twin

Understanding strains – models of polycrystalline plasticity

Single crystal properties



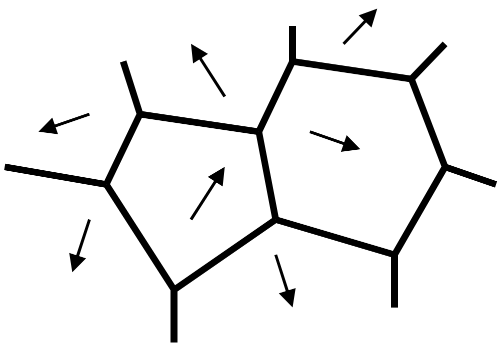


+

Crystallographic texture (ODF)

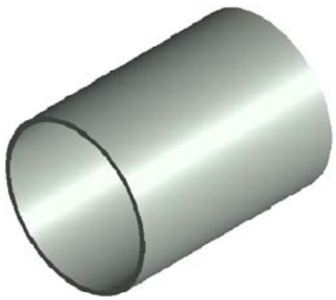
+

Crystal interactions w' matrix

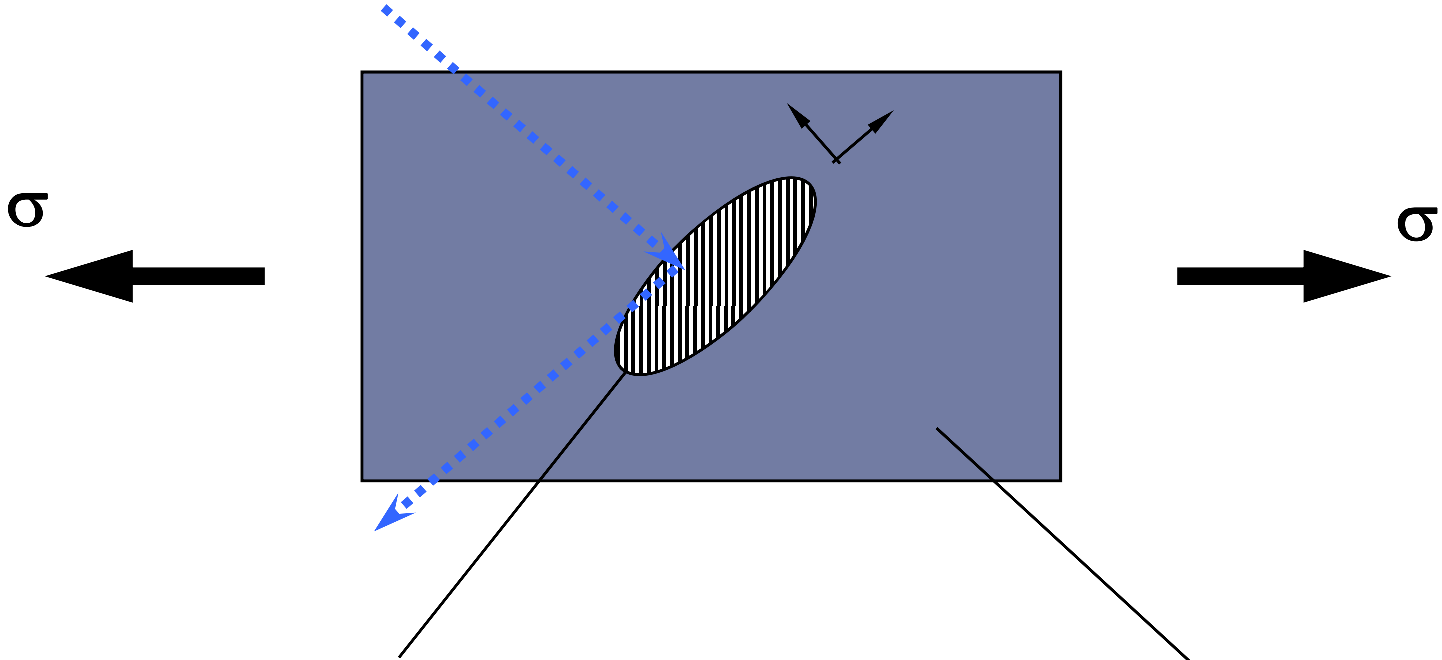


↓

Macroscopic & polycrystal properties



e.g. self-consistent elasto-plastic



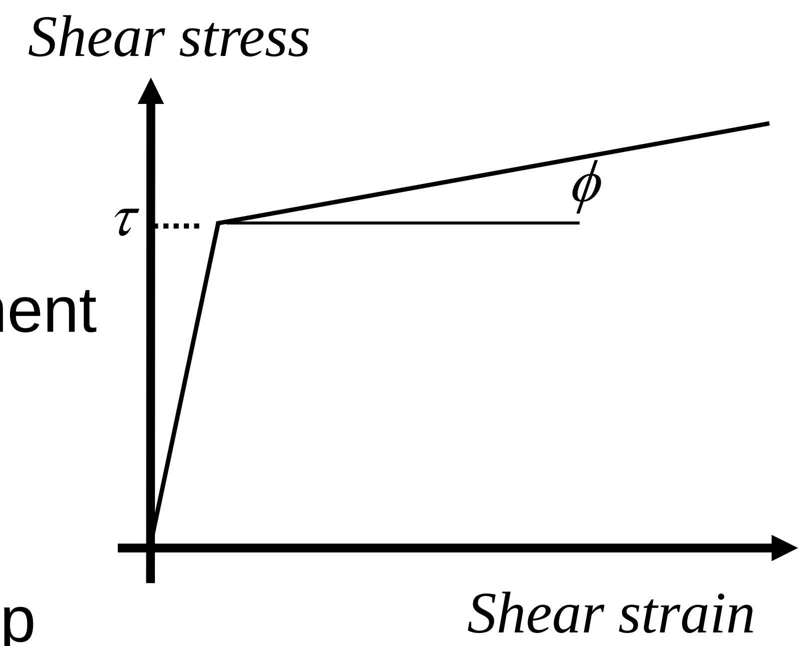
Single crystallite
• elastic and plastic anisotropy
• relate crystallite strain / stress state to macro applied strain / stress.

Matrix
• homogenous average of all the crystallites
• iterate to solution

Model: self-consistent elasto-plastic

Keep track of each grain, its stress state and its plastic strain.

Fixed parameters:
- Single crystal elastic constants
- Texture ($\Rightarrow$ grain population)
- Plastic slip directions / planes

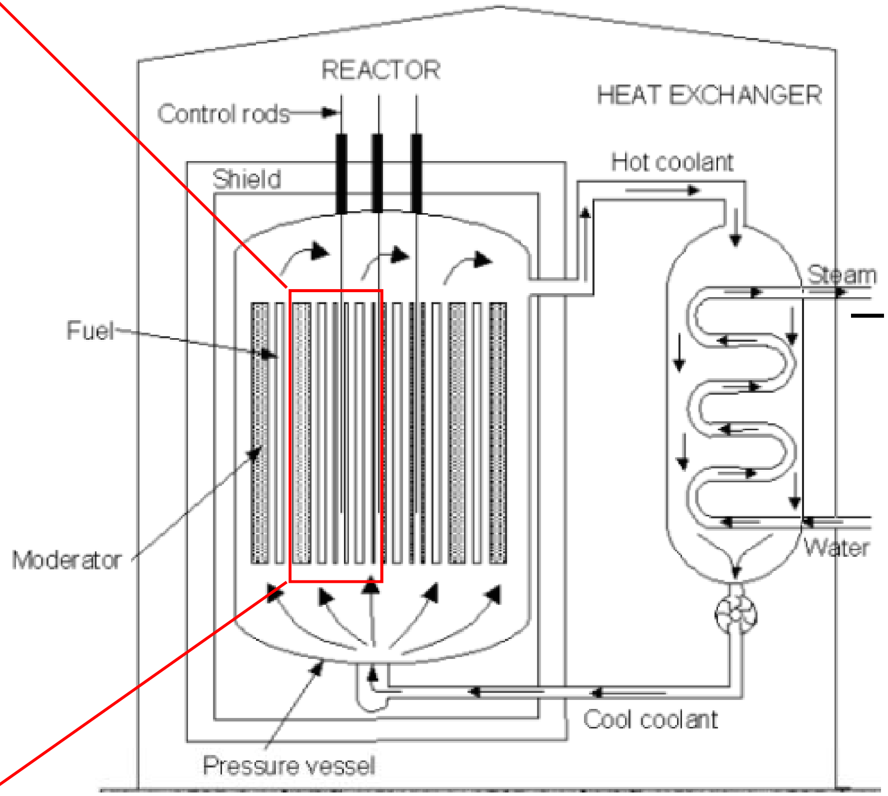


Fitted parameters, to get best agreement with experiment
- Critical resolved shear stress, τ
- Hardening gradient ϕ

i.e. only 2 fitting parameters for a slip system

(Perhaps more complex plastic law)

Use of zirconium



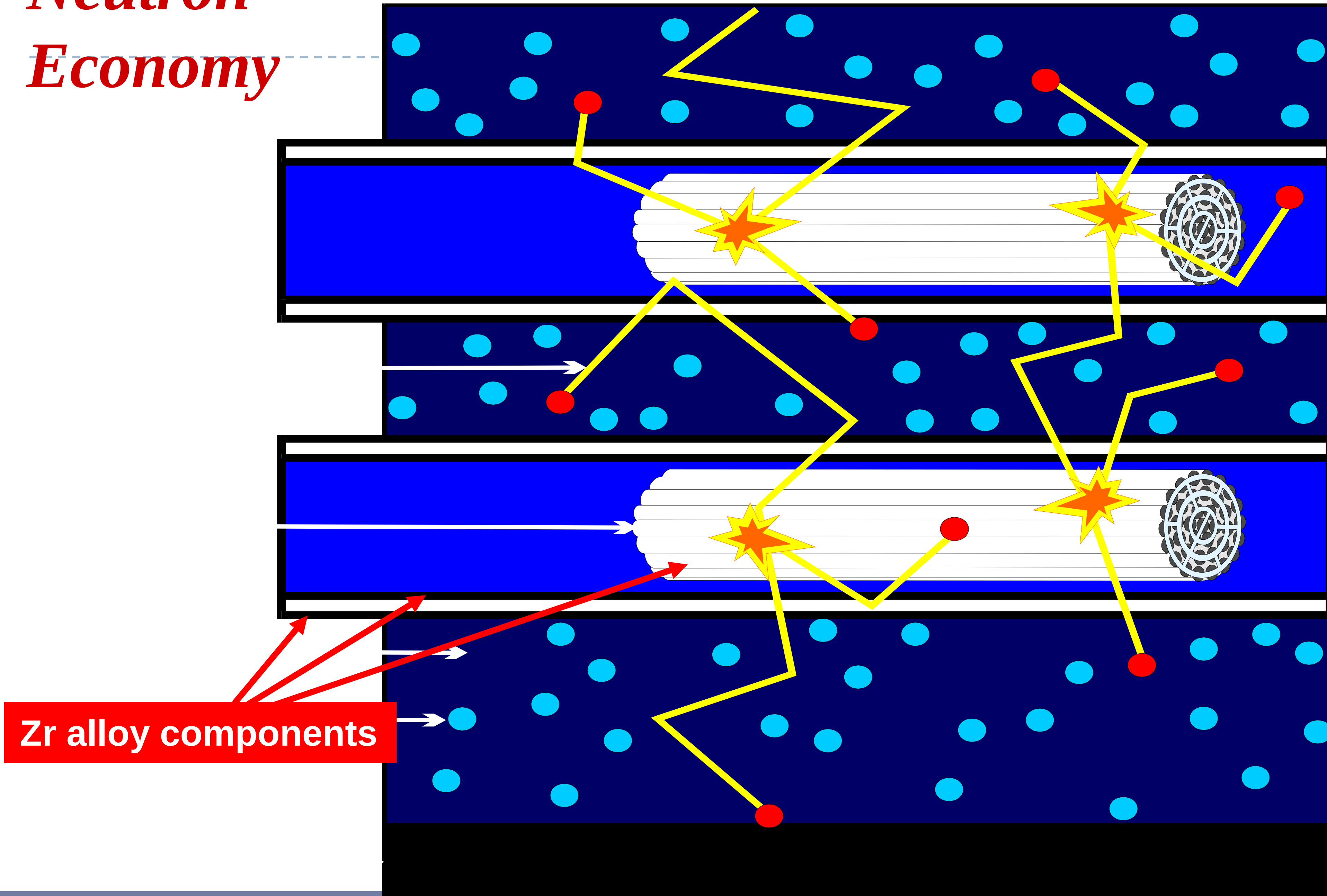
Reactors worldwide

- Zircaloy Fuel Cladding;
  - Pressurized or unpressurized H_2O coolant
  - Temperatures range from ambient to >300ºC
- PHWR;
  - Zr-2.5Nb* pressure tube contains coolant
  - Zircaloy-2 ‘calandria’ tube, separates hot pressure tube from moderator
- Research reactors;
  - Reflector vessel walls

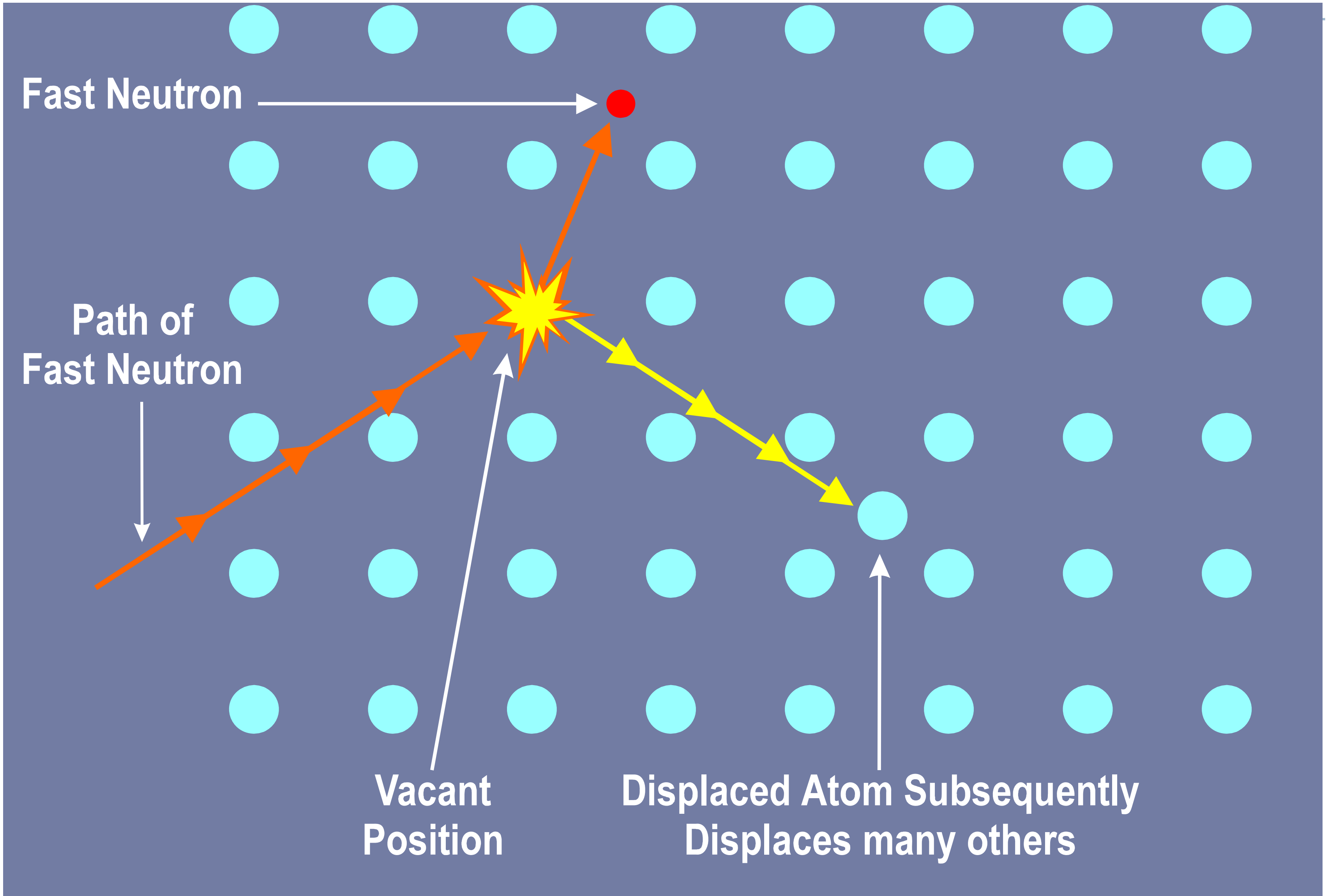
Metallurgy somewhat analogous to titanium

* 2 phase alloy, c.f. Ti-6Al-4V

Neutron Economy



Effect of neutron irradiation



On average, each atom is displaced from its position about once a year.

Residual structure after decay of thermal spike

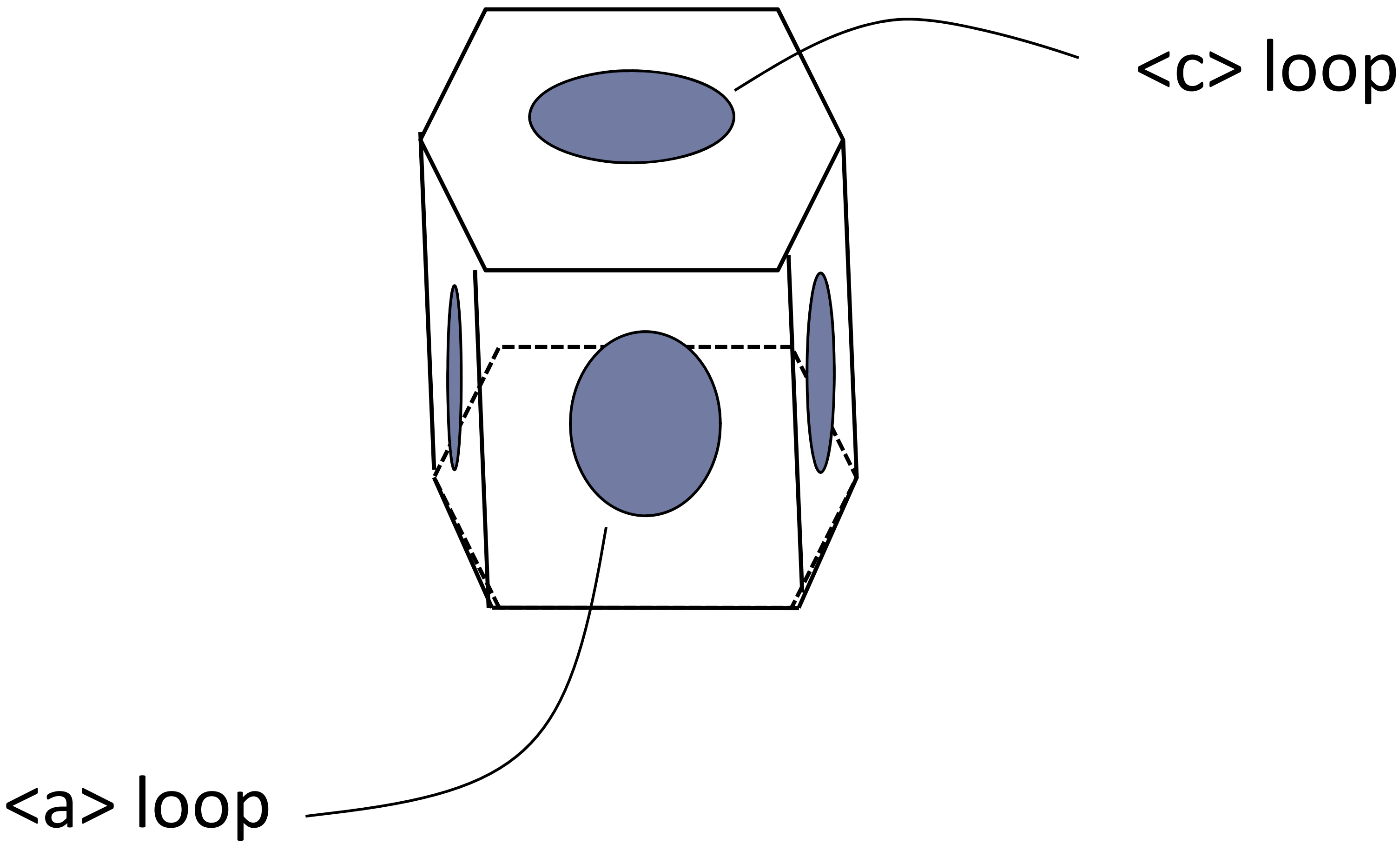
- Vacancy clusters
- Interstitial clusters
- Individual vacancies (freely migrating)
- Individual SIAs (freely migrating)

Subsequent Evolution

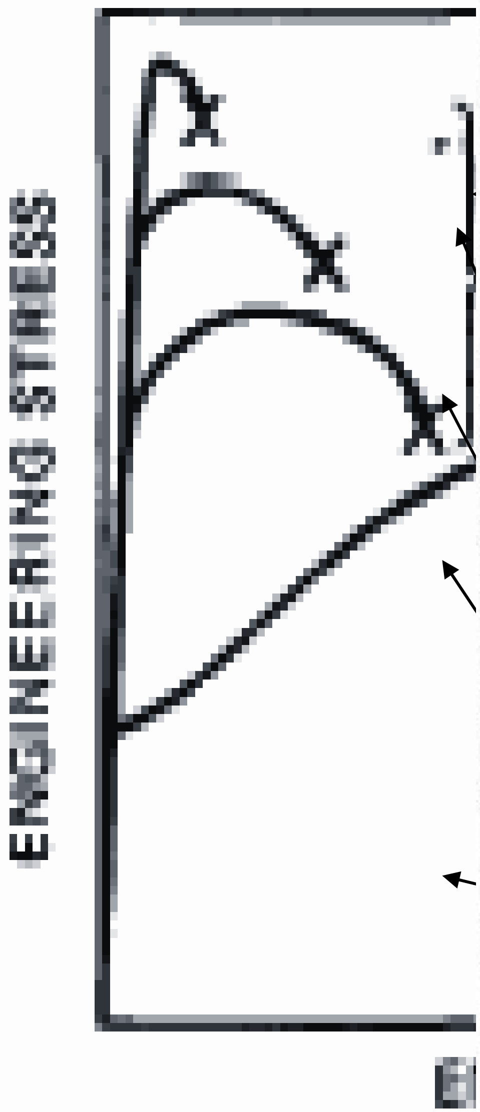
- Vacancies and interstitials migrate through the lattice and the microstructure evolves
- Usually a dense dislocation "loop" structure generated over first few dpa

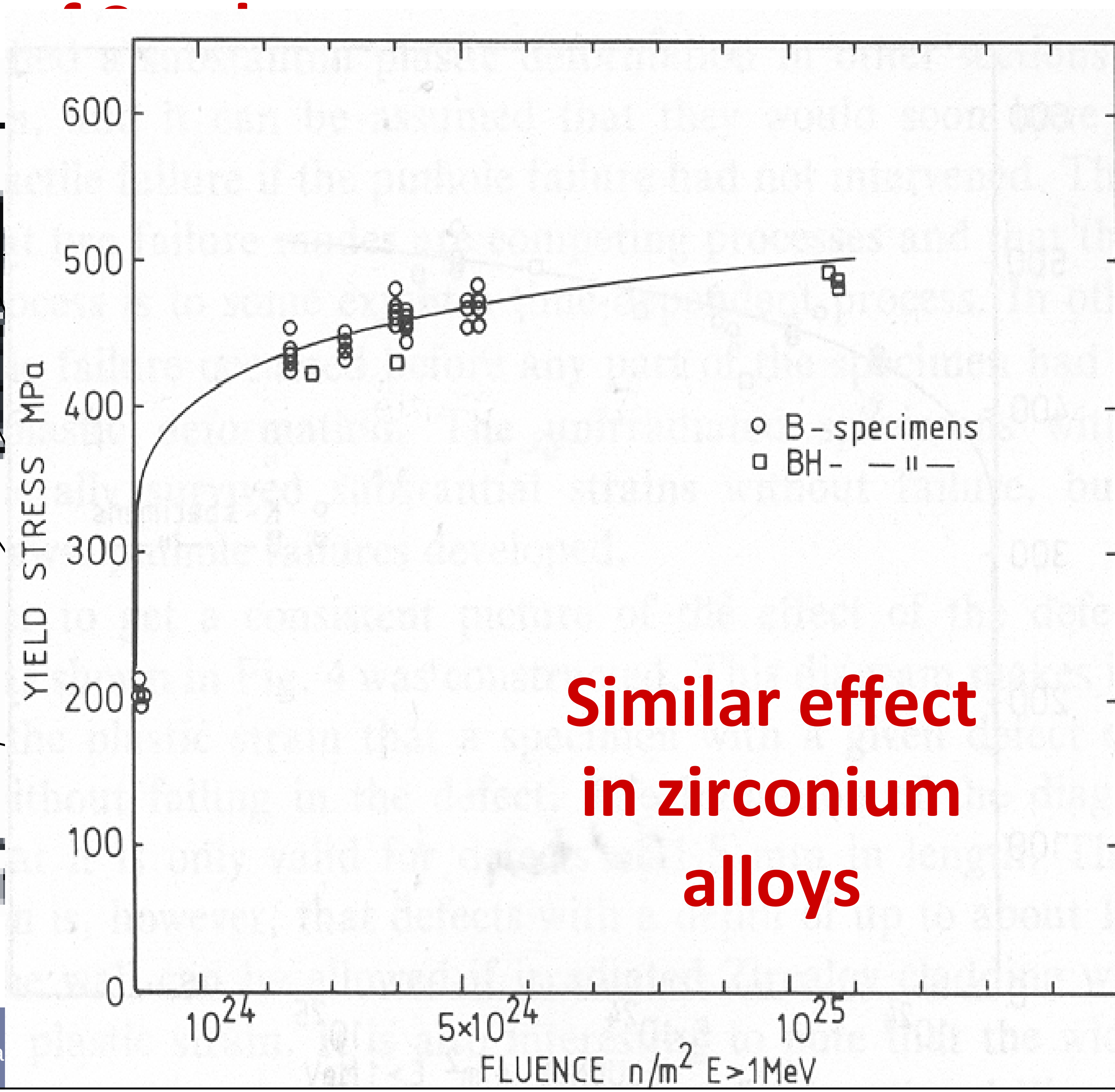
Dislocation loops

Differences in migration rates / anisotropy lead to different densities of <a> and <c> loop dislocations

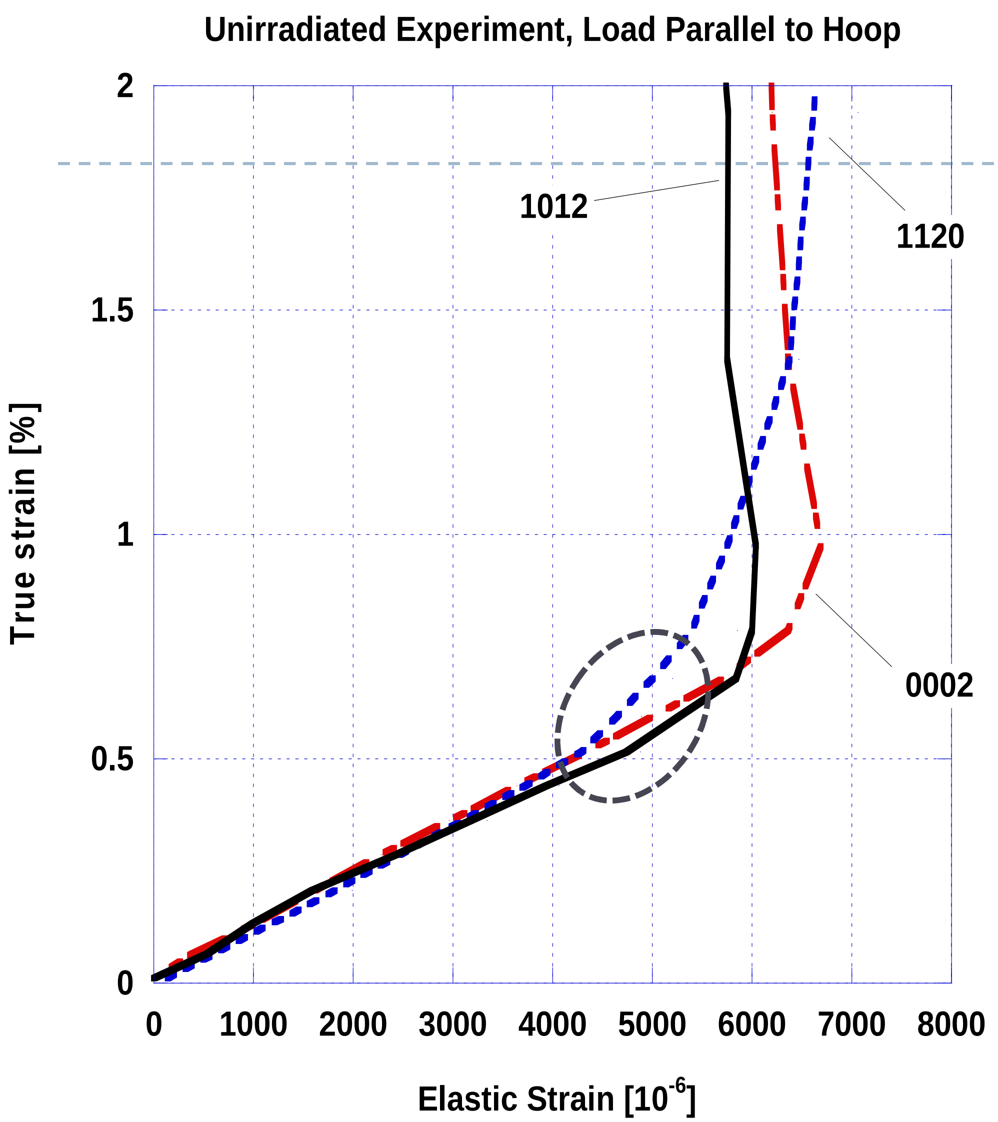


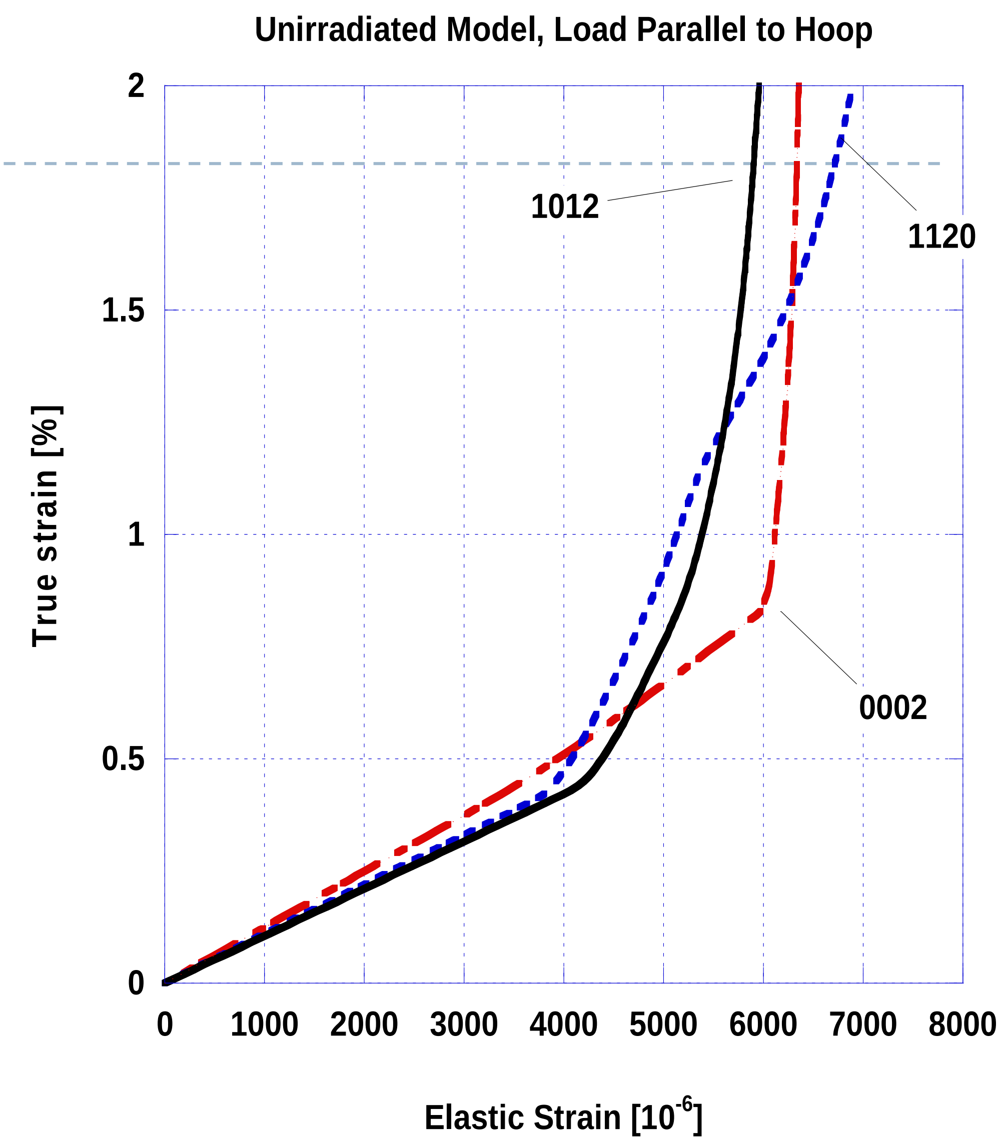
Hardening





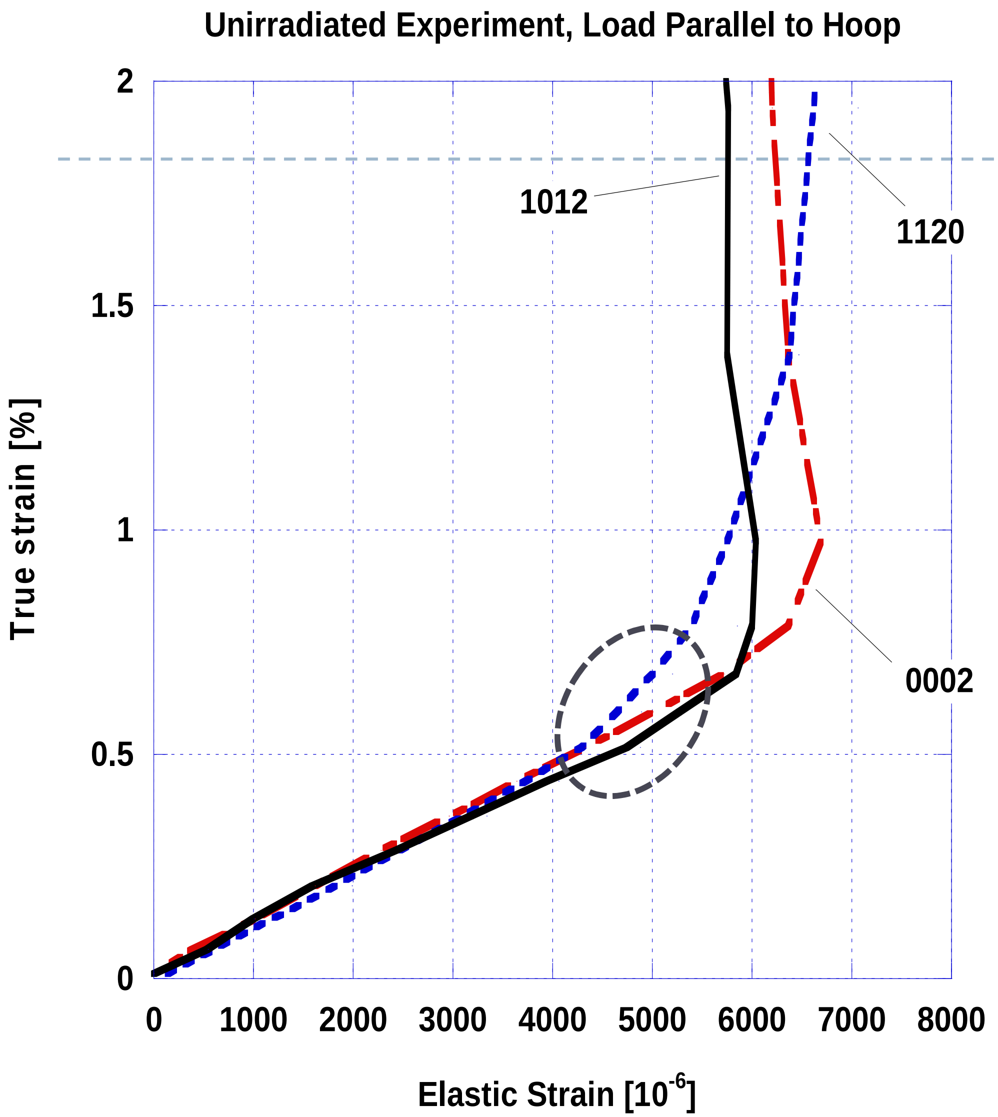
Similar effect in zirconium alloys



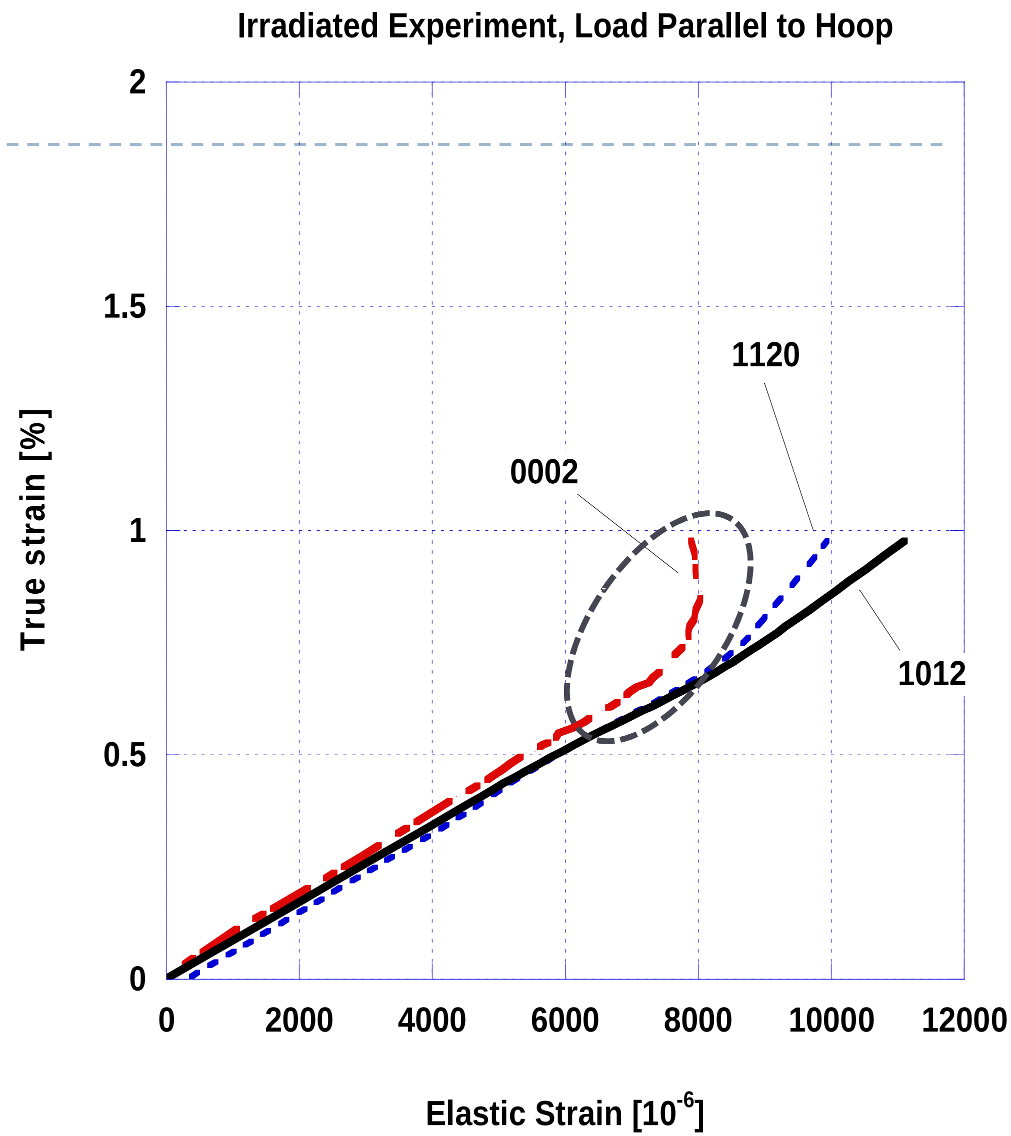


Experiment Model

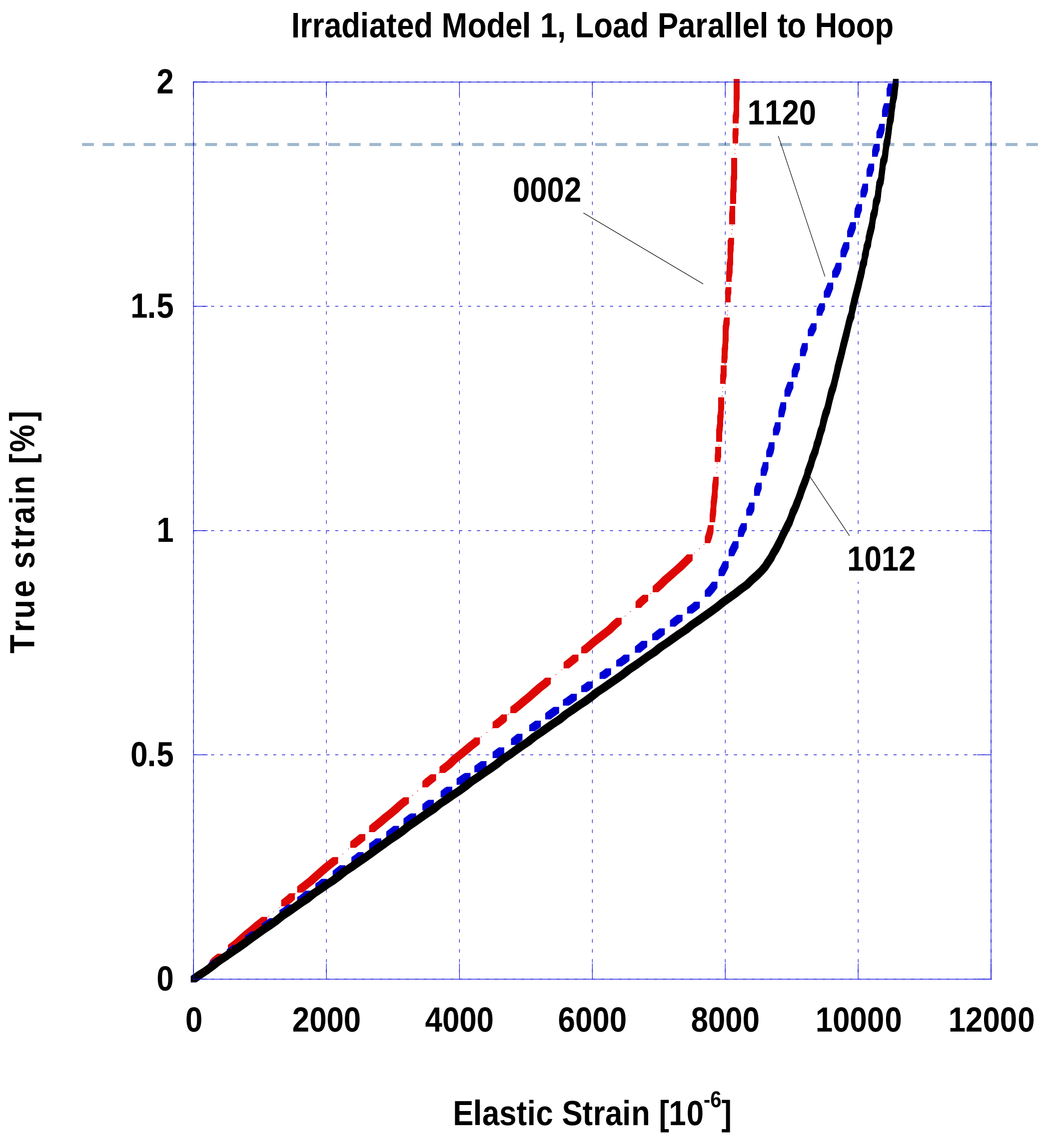
Very low hardening – plot vs macroscopic strain



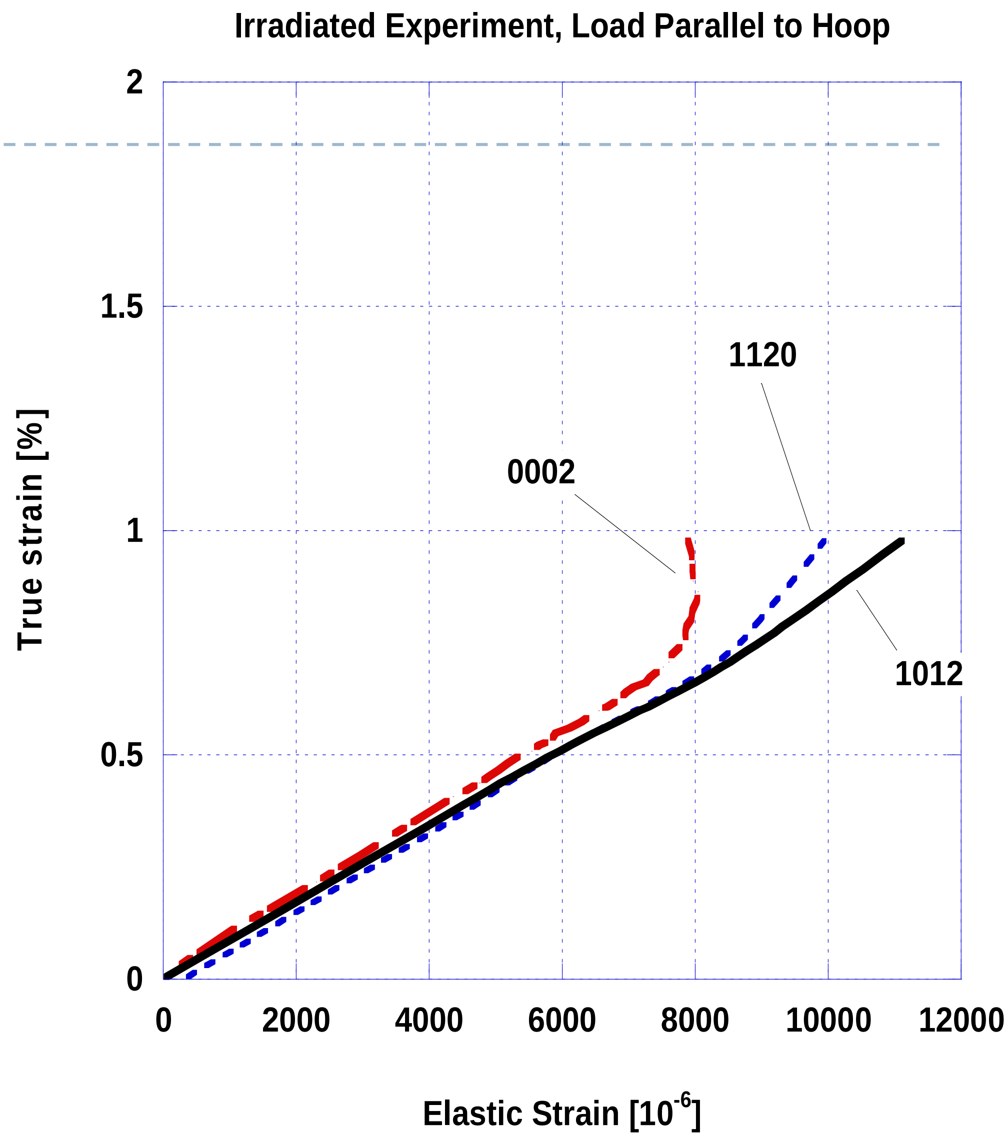
Unirrad Experiment



Irrad experiment



Irrad Model

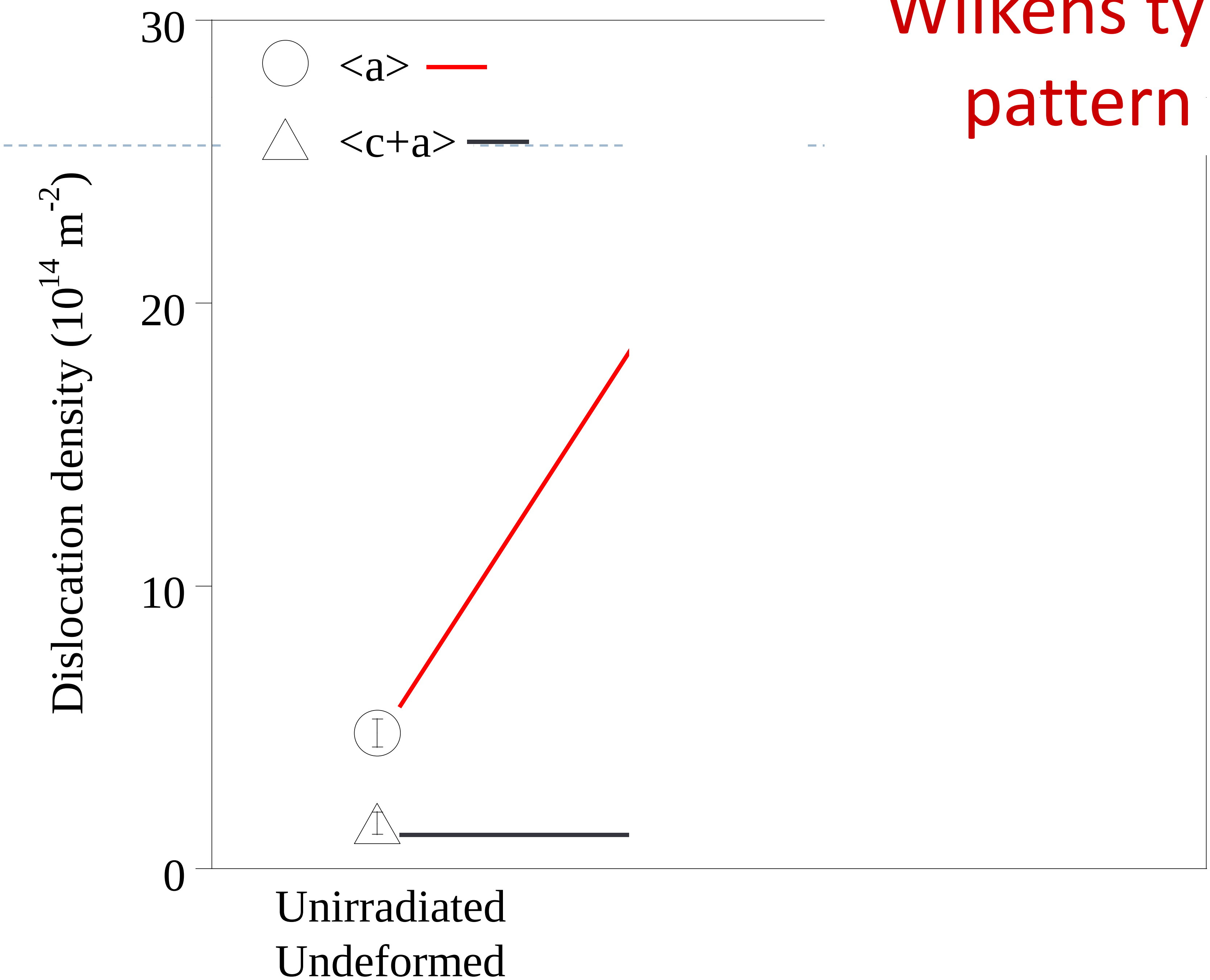


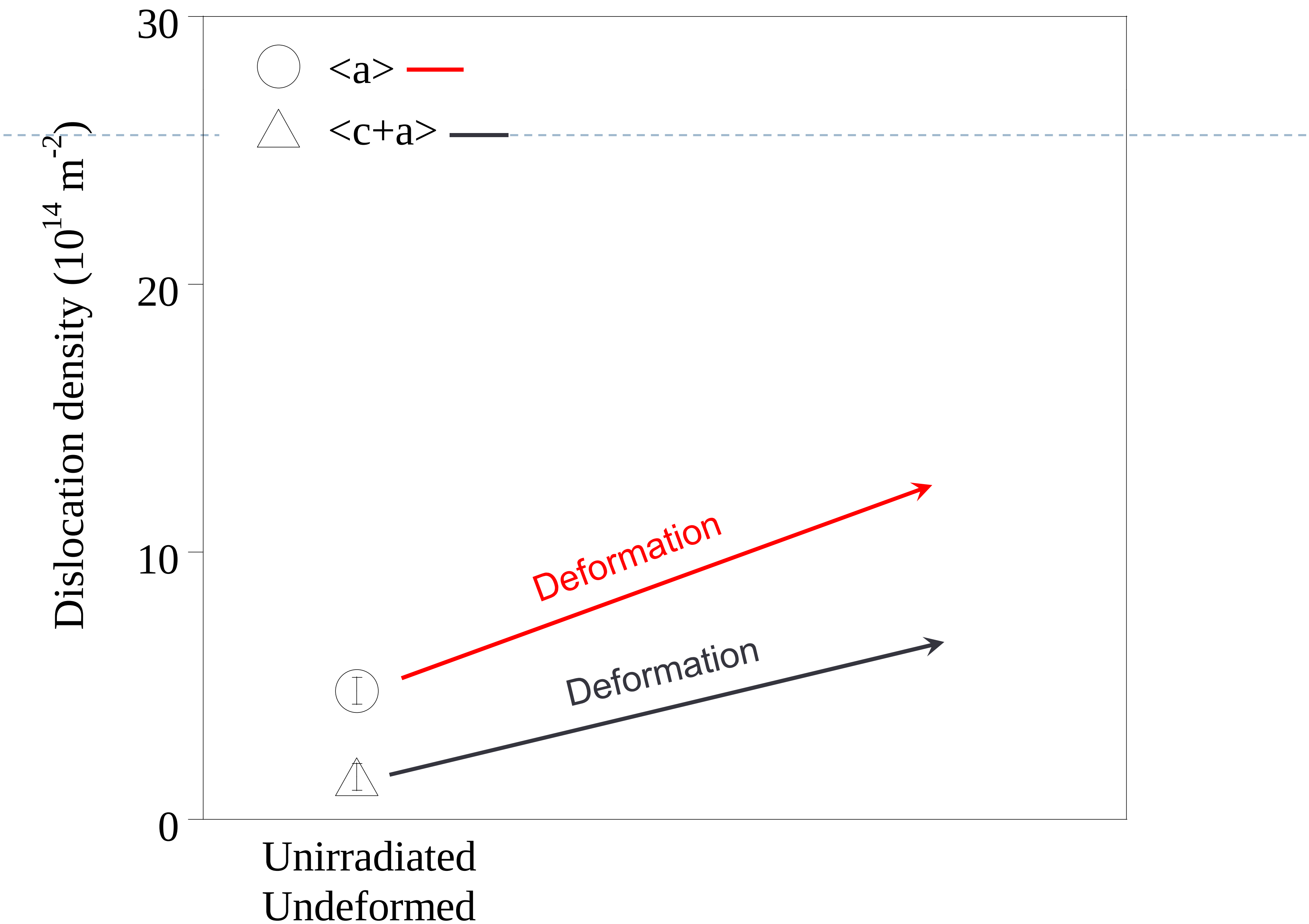
Irrad experiment

Impact of irradiation on model (fit) CRSS

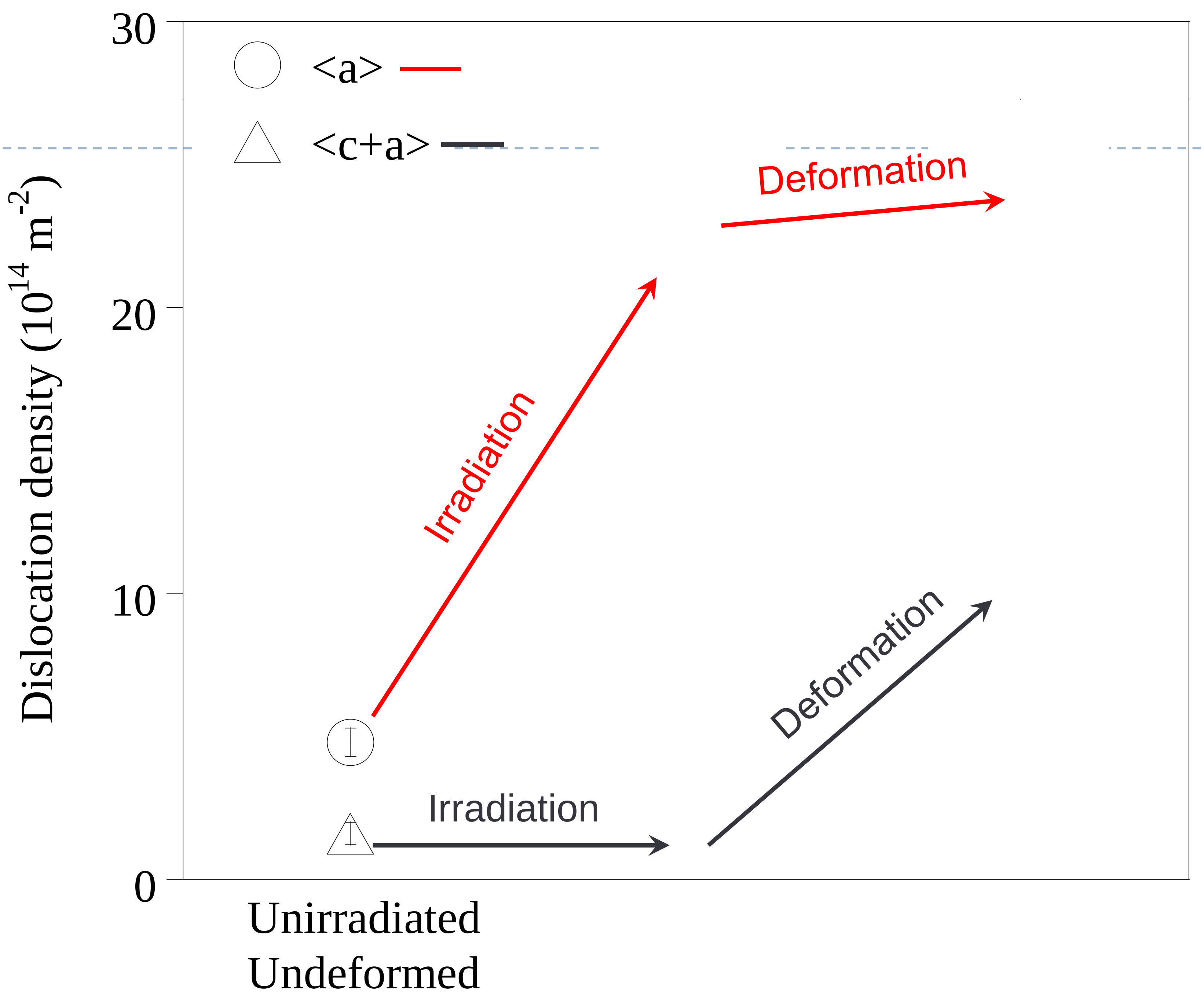
Mode	Unirradiated [MPa]	Irradiated [MPa]	Increase [MPa]
Prism <a>	160	370	210 (x2.3)
Basal <a>	170	370	200 (x2.2)
Tensile twin	450	*n/a*	*n/a*
<c+a>	460	500	0 (x1.1)
Macroscopic (experimental 0.2% yield)	*530 / 740*	*820 / 950*	*(x1.5 / x1.3)*

Wilkens type full pattern LPA





Dislocation density (10^{14} m^{-2})
30
20
10
0
<a>
<c+a>
Deformation
Deformation
Unirradiated
Undeformed


30
20
10
0
Dislocation density (10^{14} m^{-2})
<a>
<c+a>
Deformation
Irradiation
Deformation
Irradiation
Unirradiated
Undeformed

Conclusion

- Internal stresses change location of peaks enough that we must account for them in Rietveld refinement
- Can study these changes to determine crystal
  - Irradiation changed response (load sharing) of differently (crystographically) oriented grains.
  - Effect of irradiation on critical resolved shear stress in extruded Zr2.5Nb was determined
- Can be correlated with dislocation populations measured by peak width analysis (typically ex situ)

Acknowledgements

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  - LANSCE (Bjorn Clausen)
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- Levente Balogh - LANSCE
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