



AGH UNIVERSITY OF SCIENCE  
AND TECHNOLOGY

# Badanie mechanizmów odkształcenia sprężysto-plastycznego materiałów polikrystalicznych z wykorzystaniem dyfrakcji neutronów

*mgr inż. Przemysław Kot*

*Promotor: prof. dr hab. inż. Andrzej Baczmański*

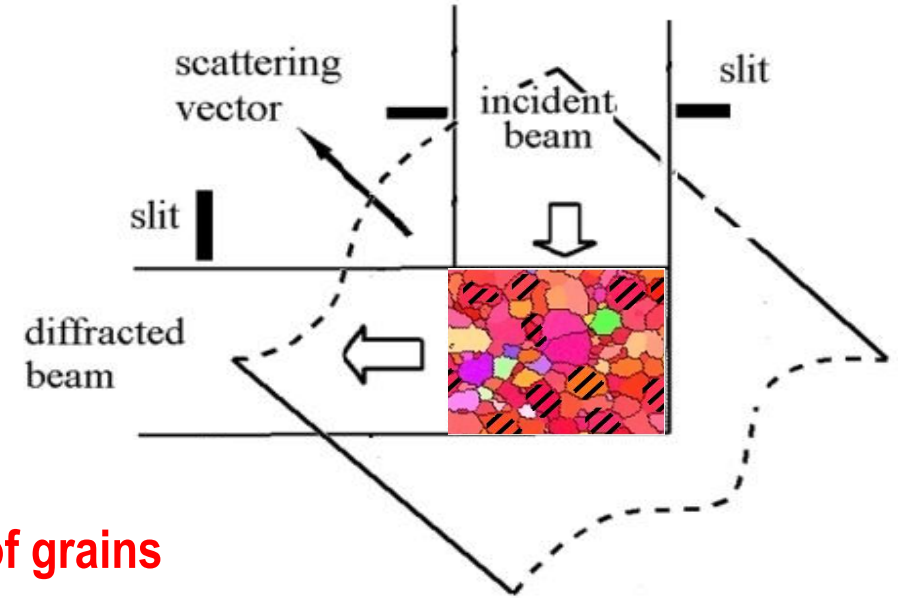
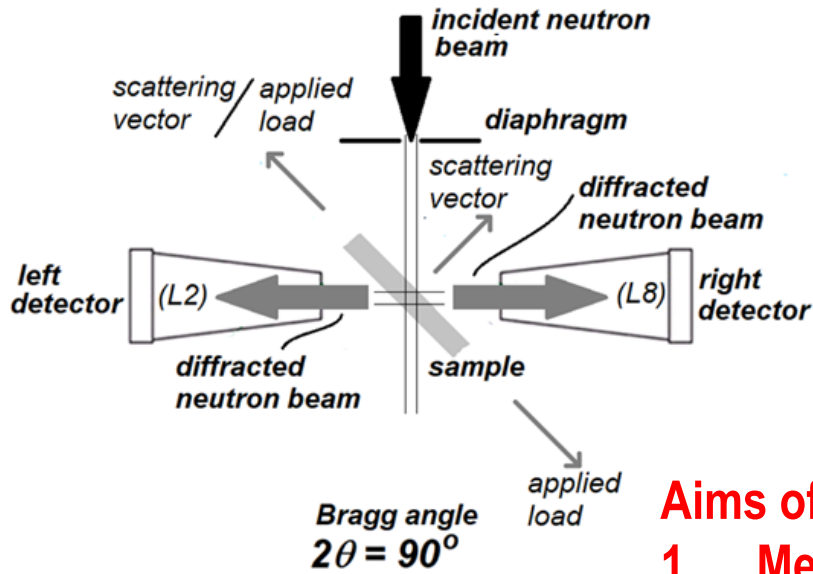
*Promotor pomocniczy: dr inż. Marcin Wroński*

# Plan

1. **Prezentacja aparatury której używamy w Dubnej**
2. **Pomiary naprężeń w kompozycie Al/SiC**
3. **Metoda grup krystalitów z użyciem dyspersji kątowej (HK9 , Řež, Praga) - lokalizacja naprężeń.**
4. **Magnez AZ31 – Próba ściskania (EPSILON, FLNP, Dubna): naprężenia podczas bliźniakowania**
5. **Ewolucja naprężeń ścinających na systemach poślizgów**
6. **Podsumowanie**

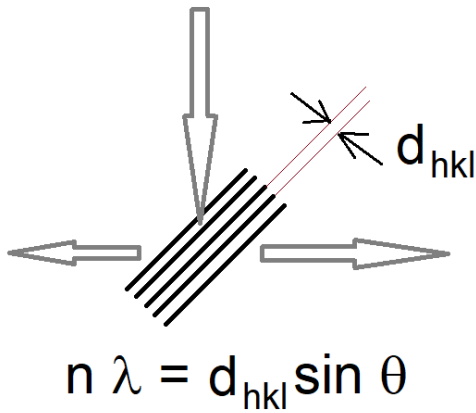
# Stress/strain measurement using diffraction

In situ diffraction measurement under applied loads



**Aims of study:**

1. Mechanical behaviour of grains
2. Stress localization
3. Critical resolved shear stresses

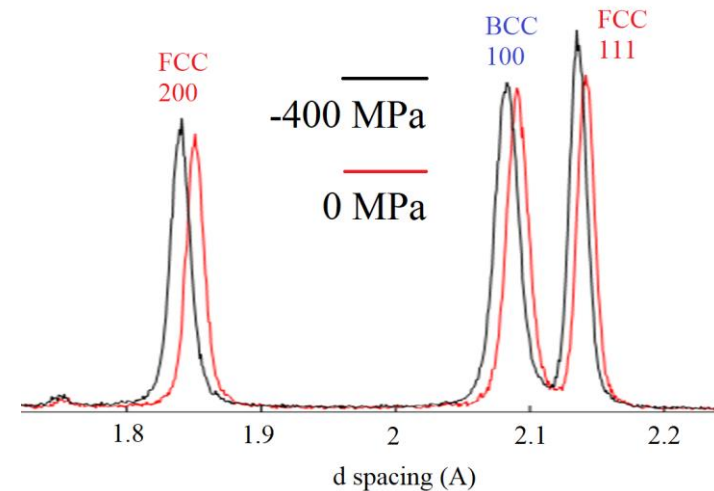


**Time of Flight**

$$d_{hkl} = \frac{hT}{2mL \sin \theta}$$

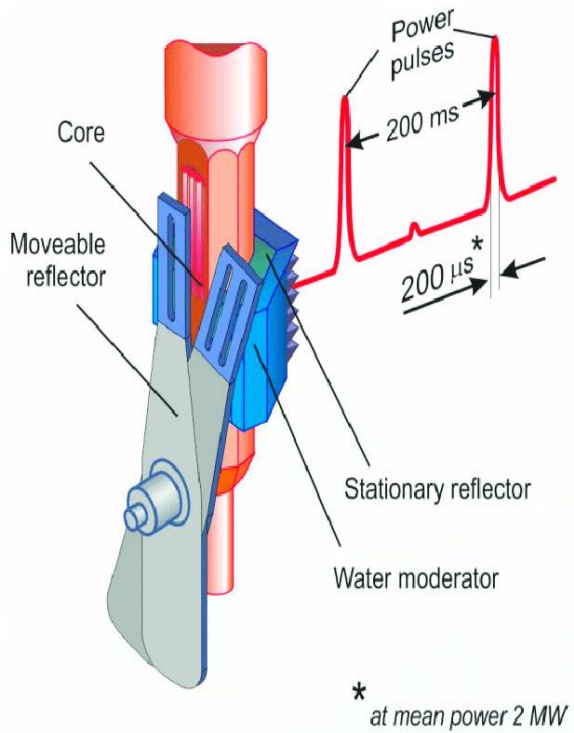
Relative lattice strain for loads:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^{initial}}{d_{hkl}^{initial}}$$

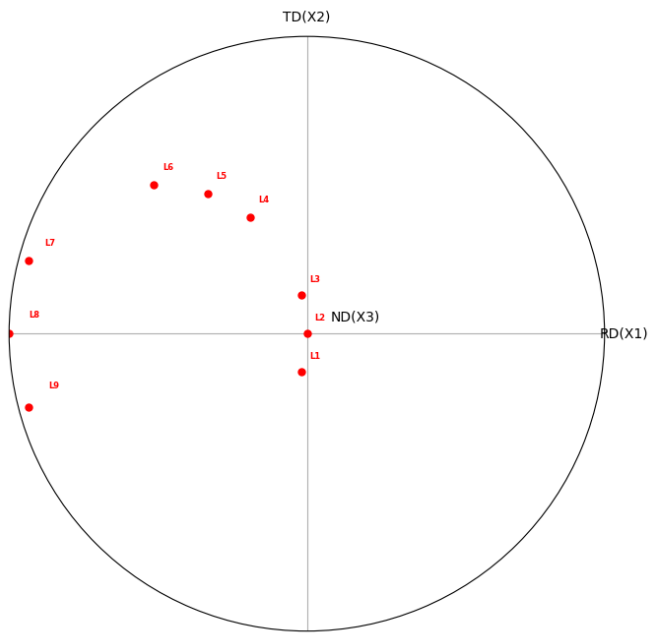
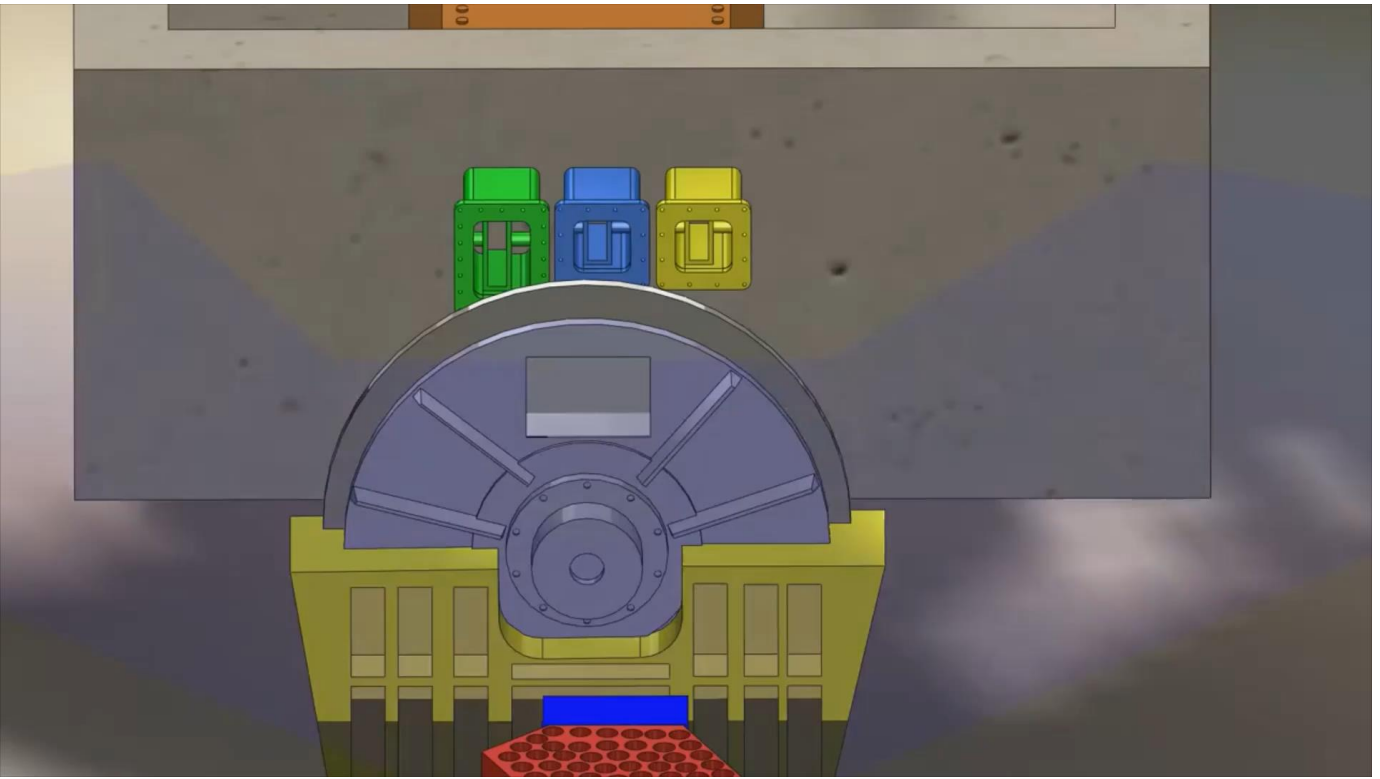
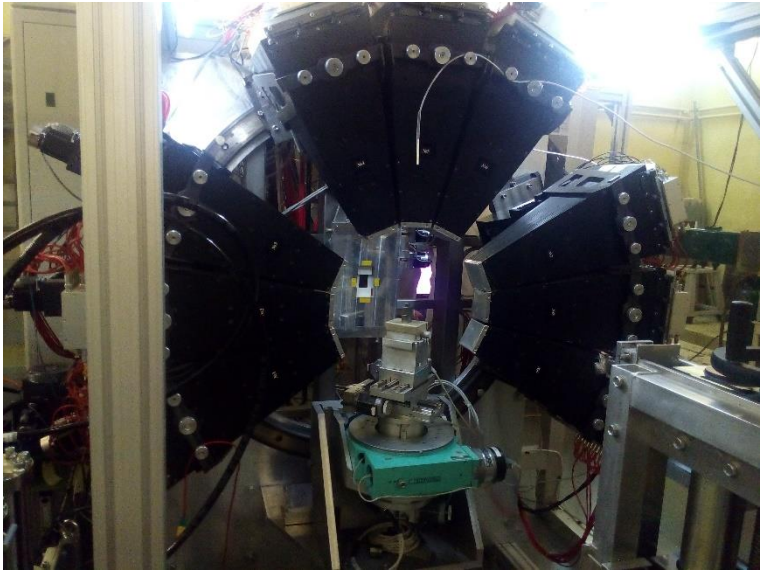


# IBR-2 impulse reactor

Joint Institut for Nuclear Research (Dubna, Russia)

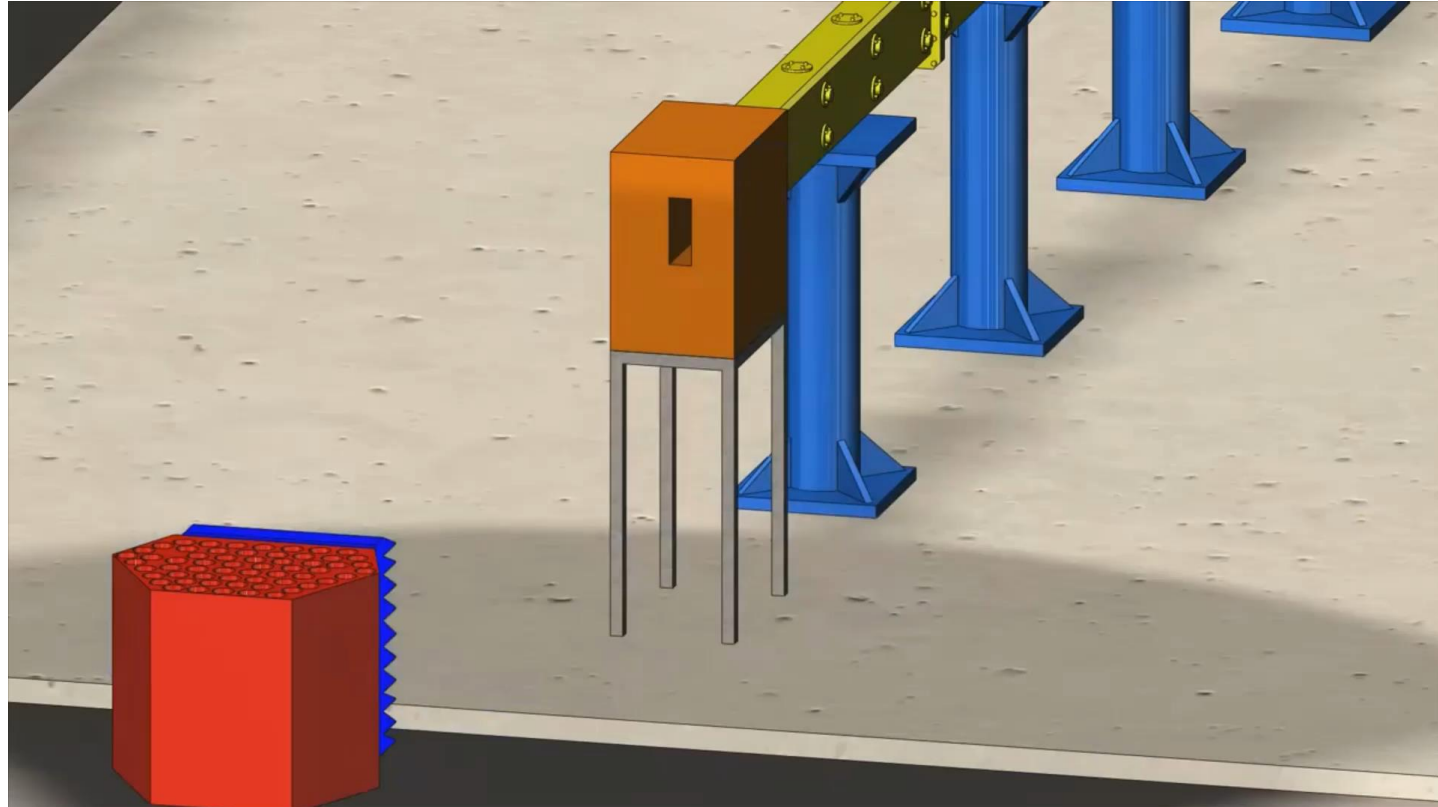
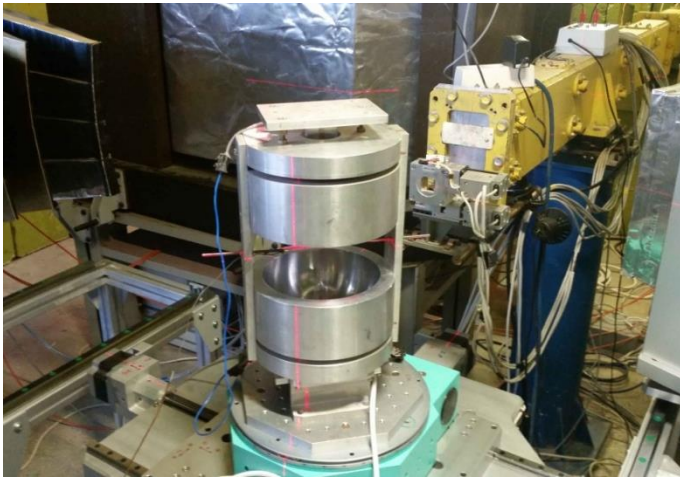


# EPSILON-MSD diffractometer





# FSD diffractometer





# Al/SiC<sub>p</sub> metal matrix composite

# Al/SiC<sub>p</sub>

metal matrix composite (83% Al2124 and 17% SiC):

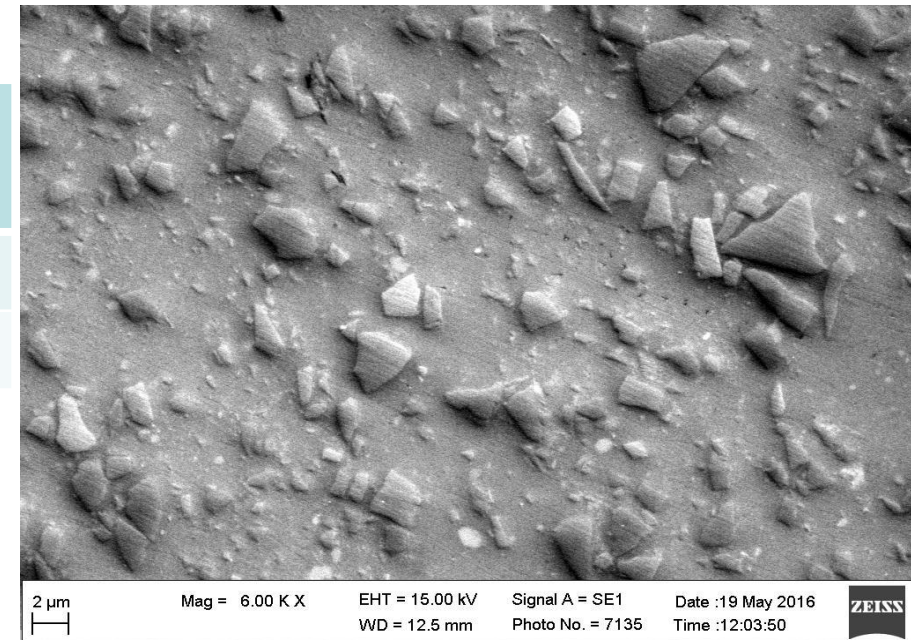
- significant difference between elastic constants,
- Al plastic, SiC elastic.

Al	Cu	Mg	Mn
93,3 – 94,3%	4,0 – 4,4%	1,3 – 1,6%	0,4 – 0,7%

GPa	Young's modulus	Poisson's ratio	Plastic deformation
SiC	450	0.21	No
Al	70	0.33	Yes

Single crystal elastic constants:

- SiC (6H - hexagonal),
- Al cubic



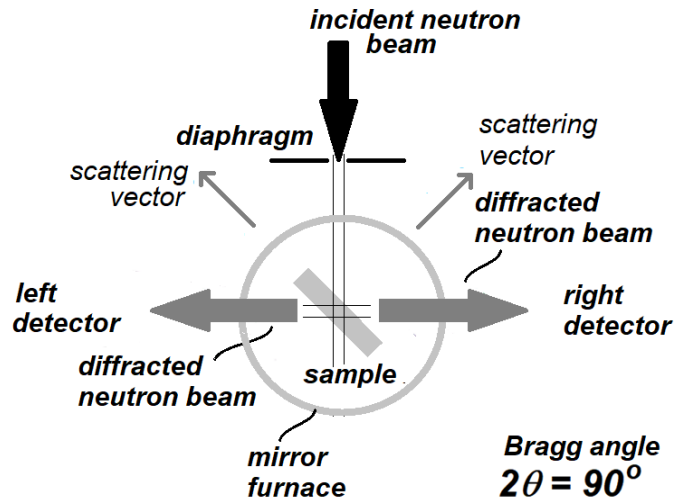
**SiC - 0.7 $\mu$ m**

E. Gadalińska, A. Baczmański, S. Wroński, P. Kot, M. Wroński, M. Wróbel, Ch. Scheffzük, G. Bokuchava, K. Wierzbanowski, Metals and Materials International, 25 (2019) pp 657–668.

P. Kot, A. Baczmański, E. Gadalińska, S. Wroński, M. Wroński, M. Wróbel, G. Bokuchava, Ch. Scheffzük, K. Wierzbanowski, Journal of Materials Science & Technology, 36 (2020) 176–189

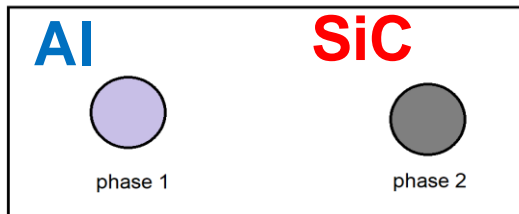


# Al/SiC (T6) in-situ cooling test

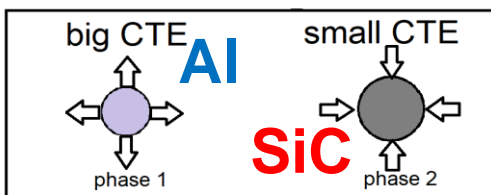


## Fourier Stress Diffractometer FSD - diffractometer

mirror furnace

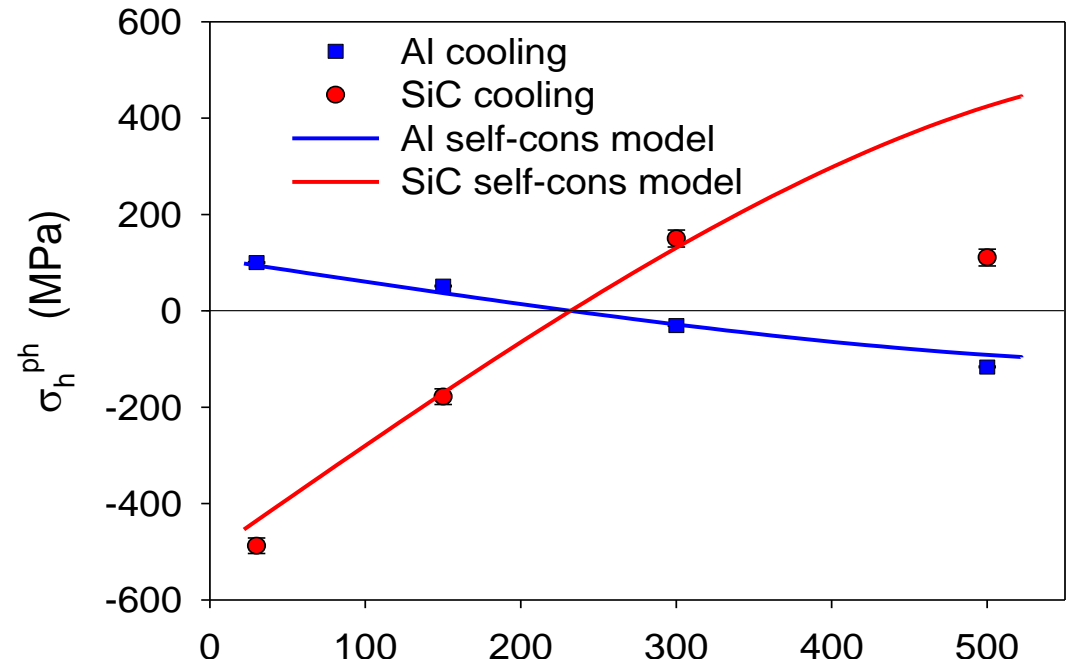


cooling



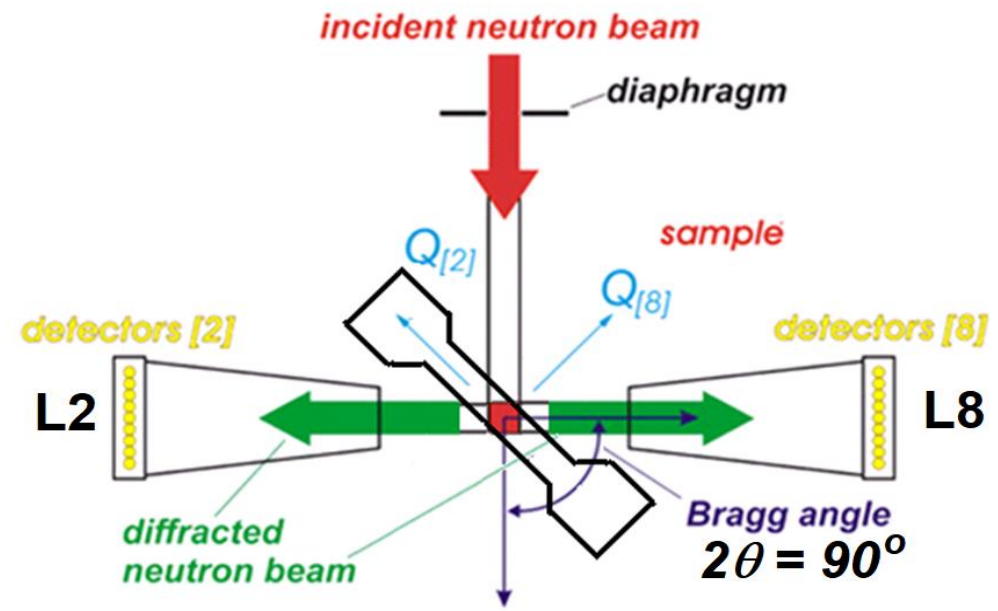
$$\varepsilon_{hkl} = \frac{d_{hkl}^{Al/SiC} - d_{hkl}^{Al,SiC}}{d_{hkl}^{Al,SiC}}$$

Phase	Young modulus (GPa)	Poisson ratio	Measured mean CTE (K <sup>-1</sup> ) for the range: 22 °C – 500°C
6H-SiC	460	0.21	3.3 · 10 <sup>-6</sup>
Al	68	0.33	27.5 · 10 <sup>-6</sup>



← **cooling**

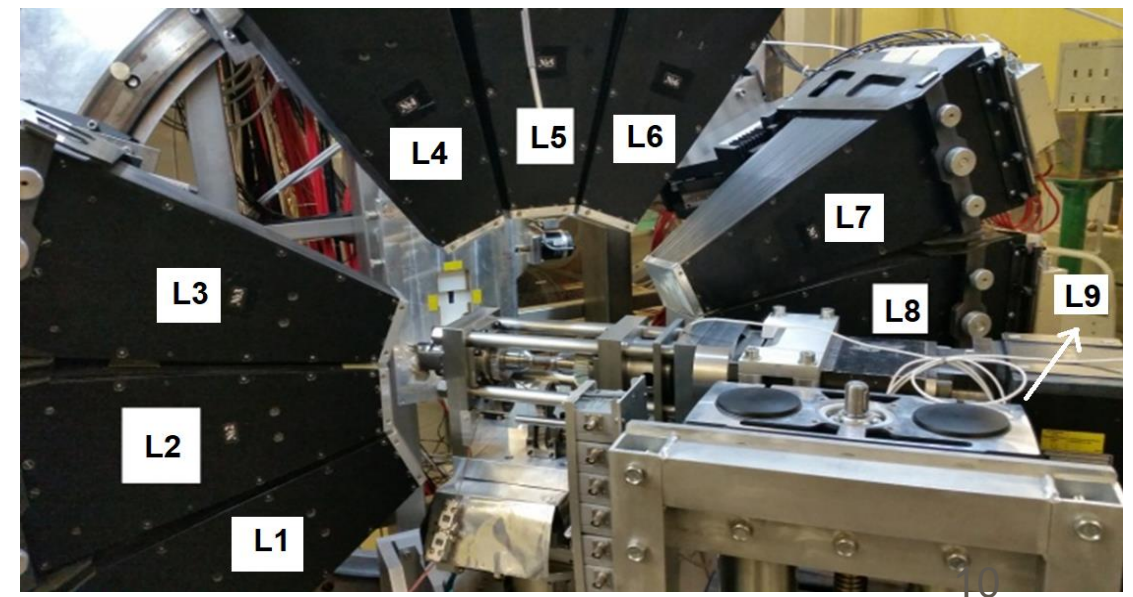
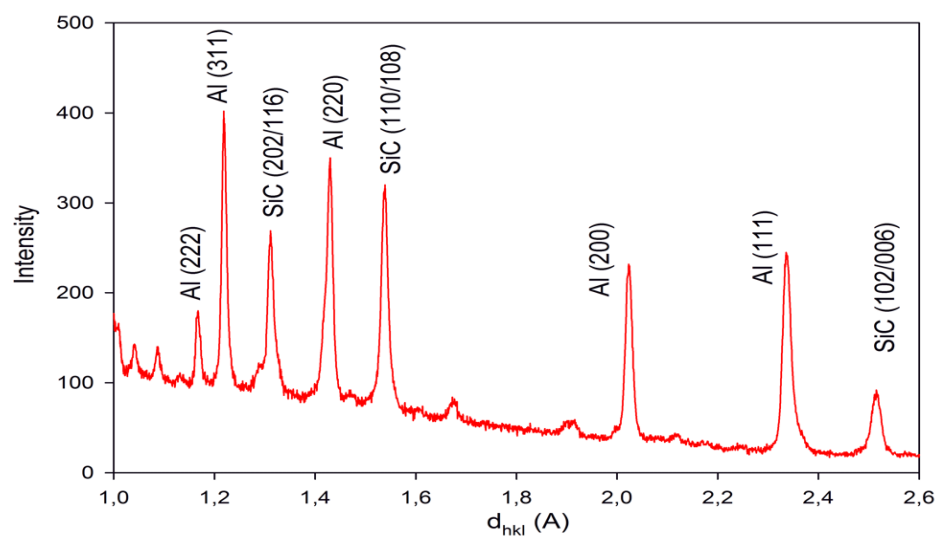
„in situ” applied load



$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^{initial}}{d_{hkl}^{initial}}$$

Time of Flight:  $d_{hkl} = \frac{hT}{2mL \sin \theta}$

EPSILON - MDS diffractometer



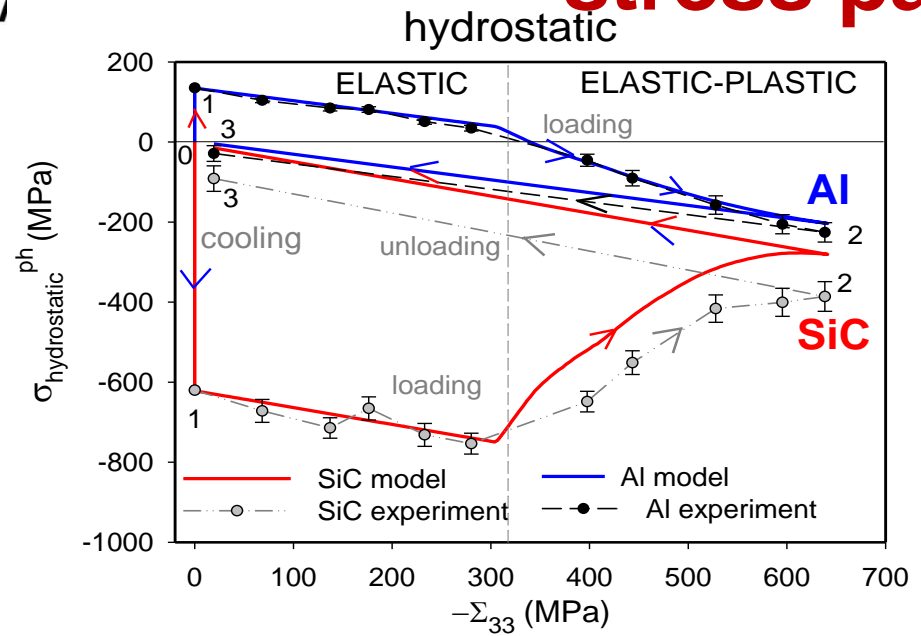
# Al/SiC– Metal Matrix Composite (T6)

## stress partitioning (tensile)

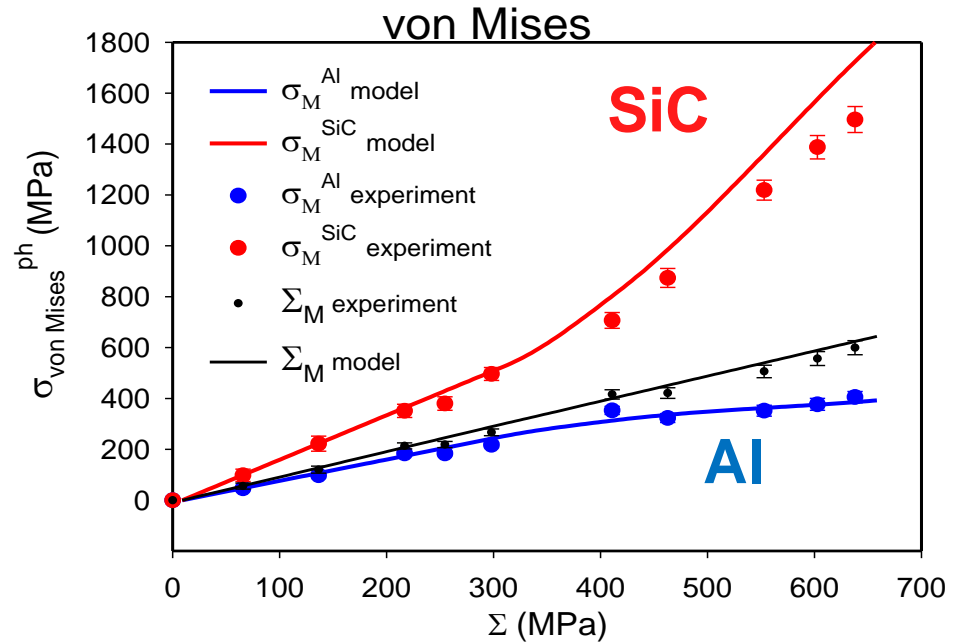
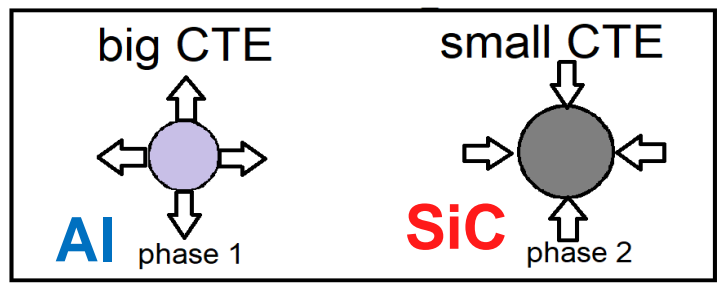
$\tau_{Al}$ (MPa)	$H_{Al}$ (MPa)
120	50

$$\sigma_h^{ph} = (\sigma_{11}^{ph} + \sigma_{22}^{ph} + \sigma_{33}^{ph})/3$$

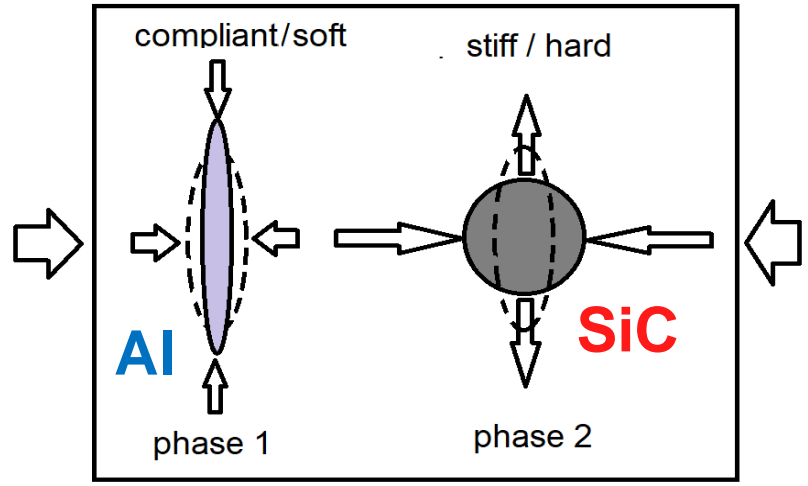
Slip system  $\langle 110 \rangle \{ 111 \}$



cooling



COMPRESSION



$$\sigma_M^{ph} = \sqrt{[(\sigma_{11}^{ph} - \sigma_{22}^{ph})^2 + (\sigma_{22}^{ph} - \sigma_{33}^{ph})^2 + (\sigma_{33}^{ph} - \sigma_{11}^{ph})^2]/2}$$



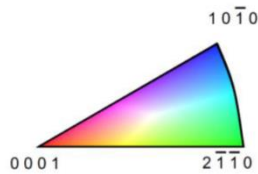
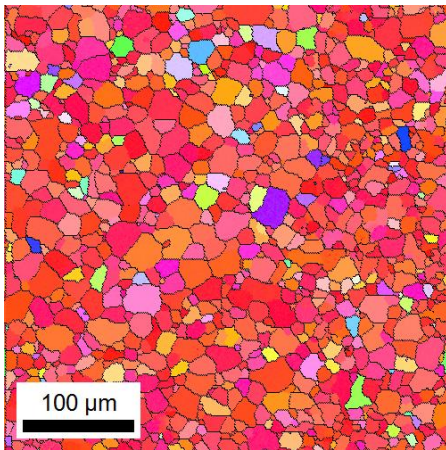
# AZ 31 magnesium alloy



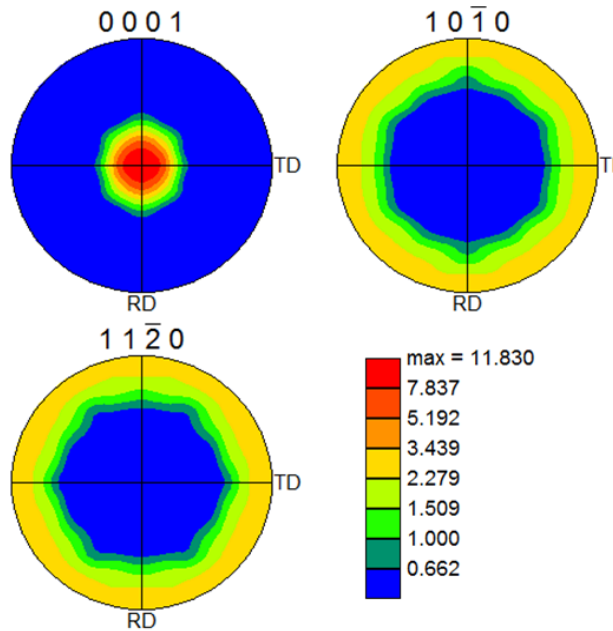
# Material characterization Mg alloy (AZ31) – hot rolled

	Al	Zn	Mn	Cu	Mg
wt%	2,5-3,5	0,7-1,3	0,2-1,0	0,05	94,15-96,55

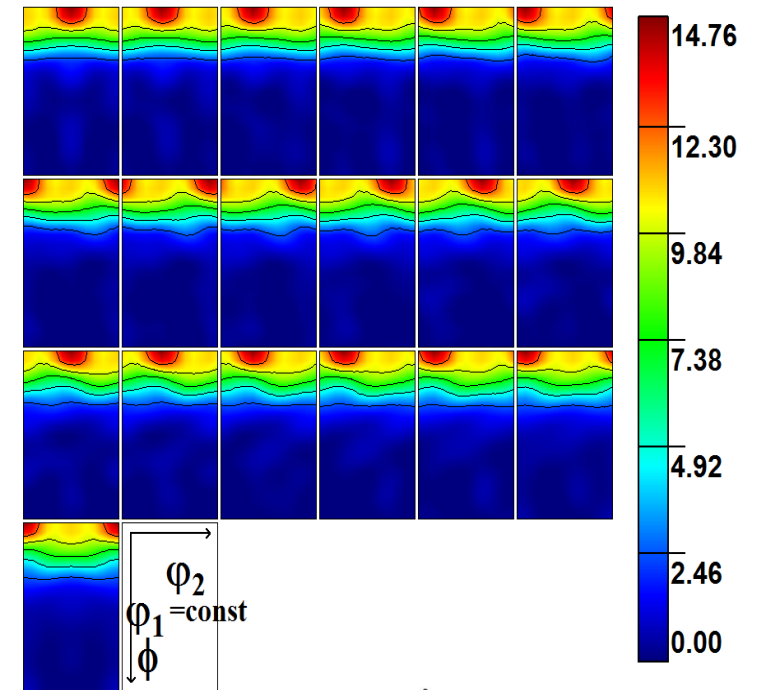
## EBSD



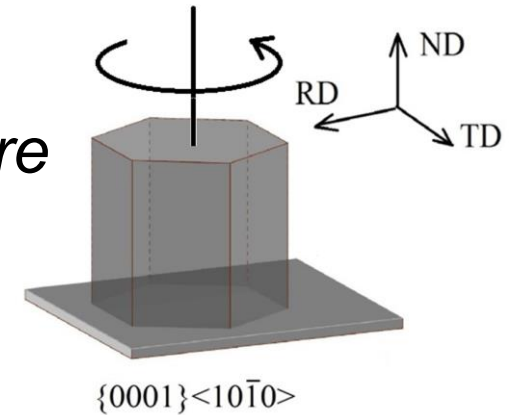
Orientation map



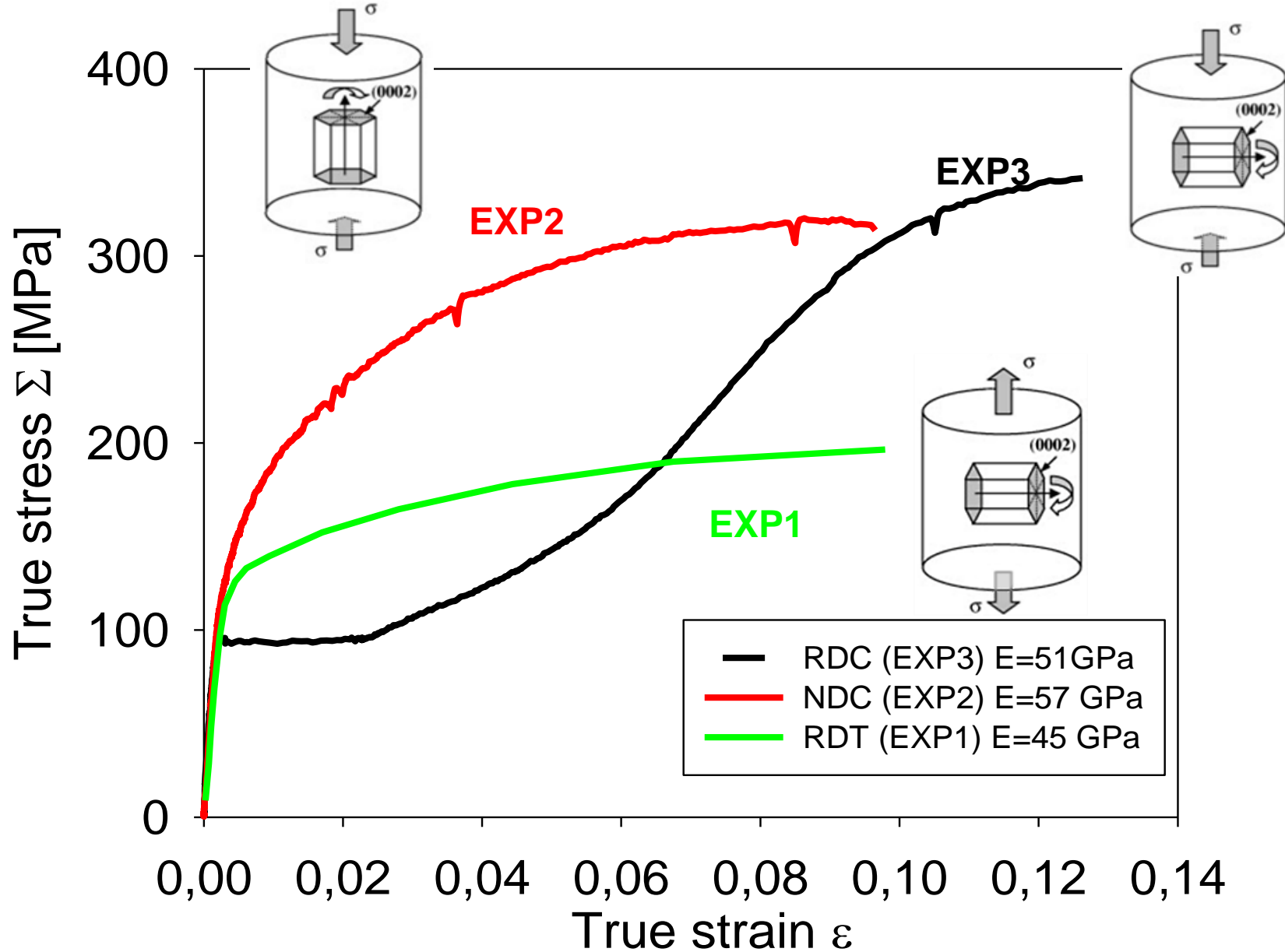
Crystallographic texture



Fibre texture

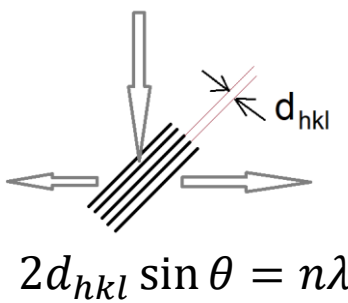


# Mg alloy (AZ31) – anisotropy



# Experiment – HK9, Nuclear Physics Institute, Řež, (Czech Republic) – tensile RD (Exp. 1)

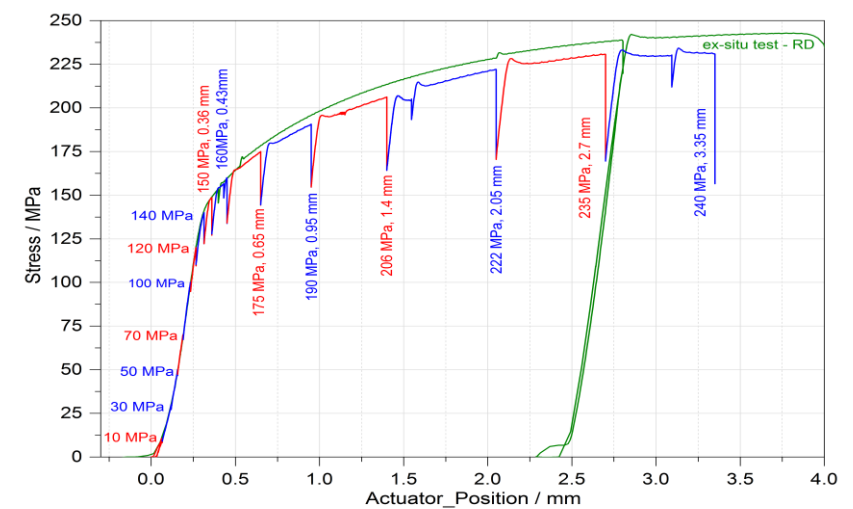
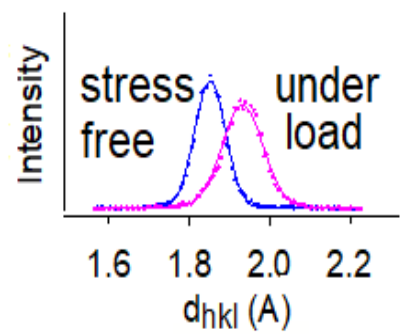
## Sample



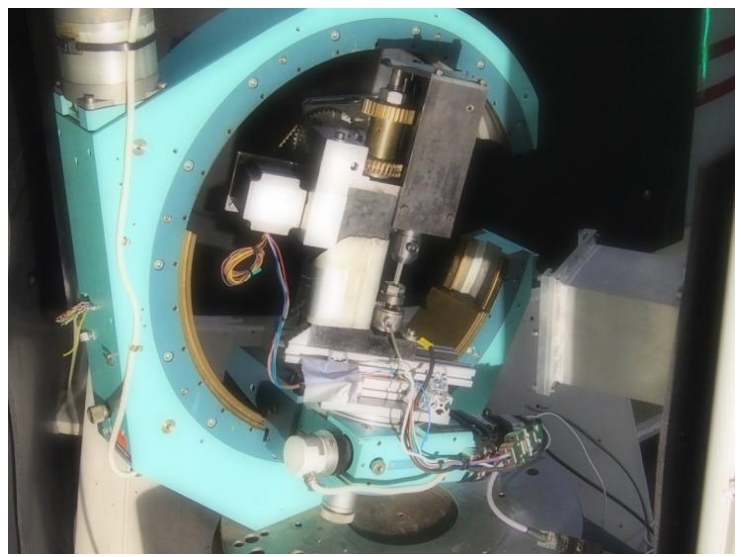
Relative lattice strain for loads:

$$\epsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^{initial}}{d_{hkl}^{initial}}$$

## Experiment – in situ tensile test in RD

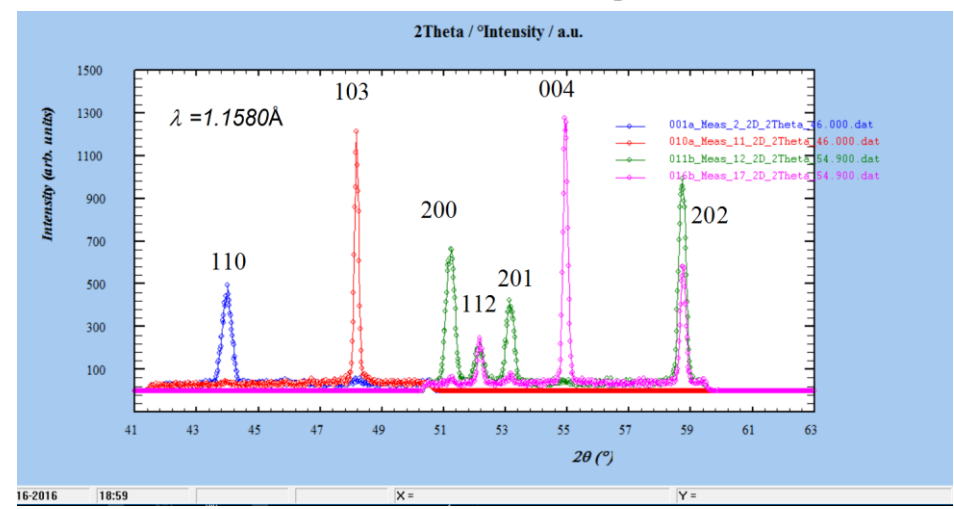
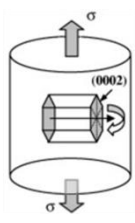


## Eulerian cradle



## Monochromatic wavelength: 1.158 Å

EXP1

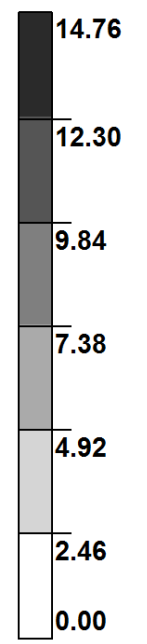
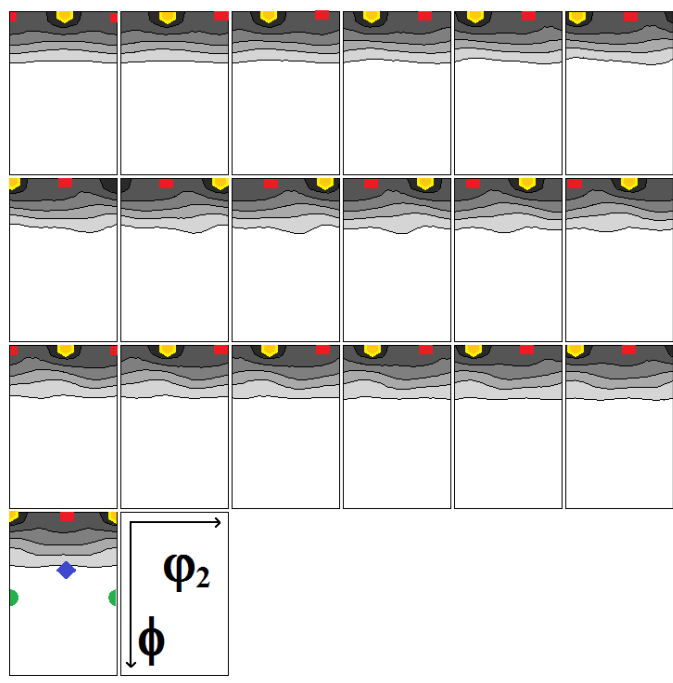
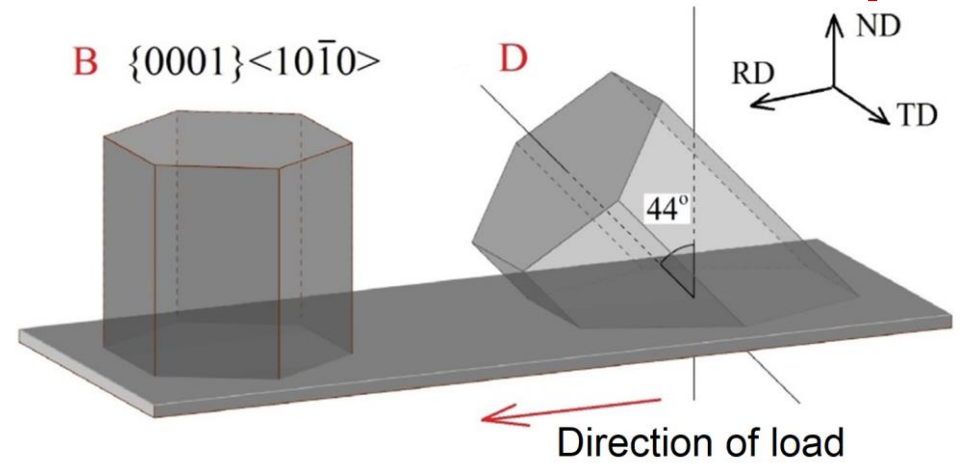
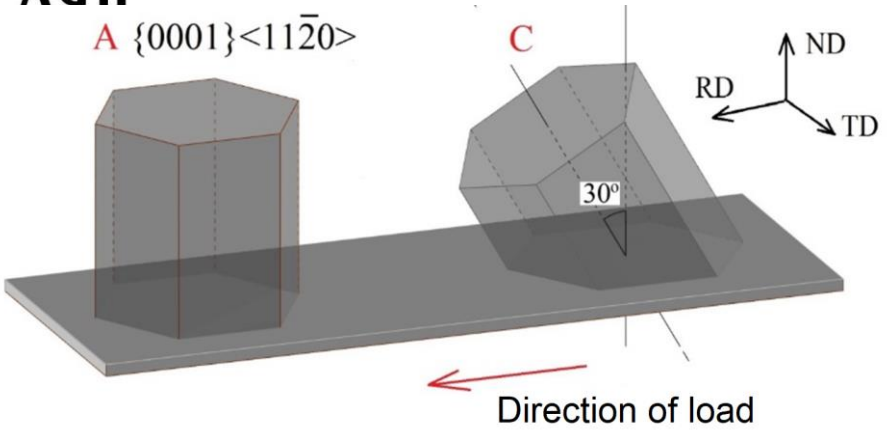




# Crystallite group method (neutrons)

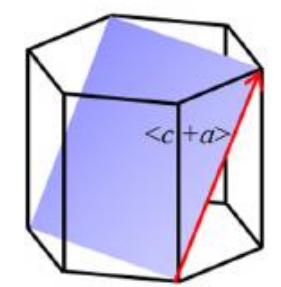
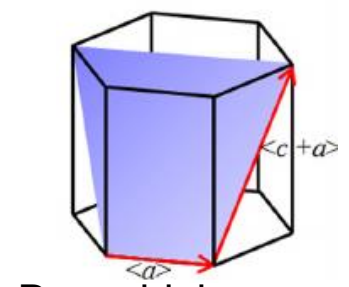
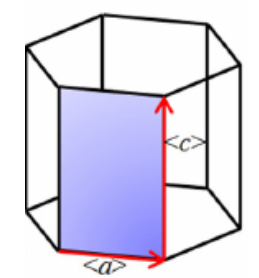
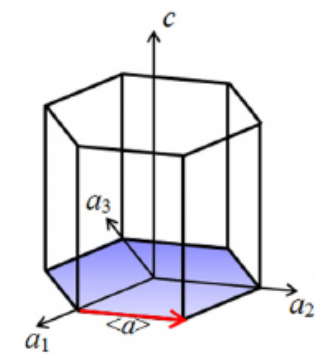
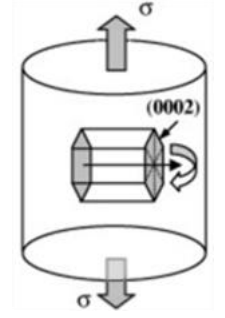


# Lattice orientations – tensile RD (Exp. 1)

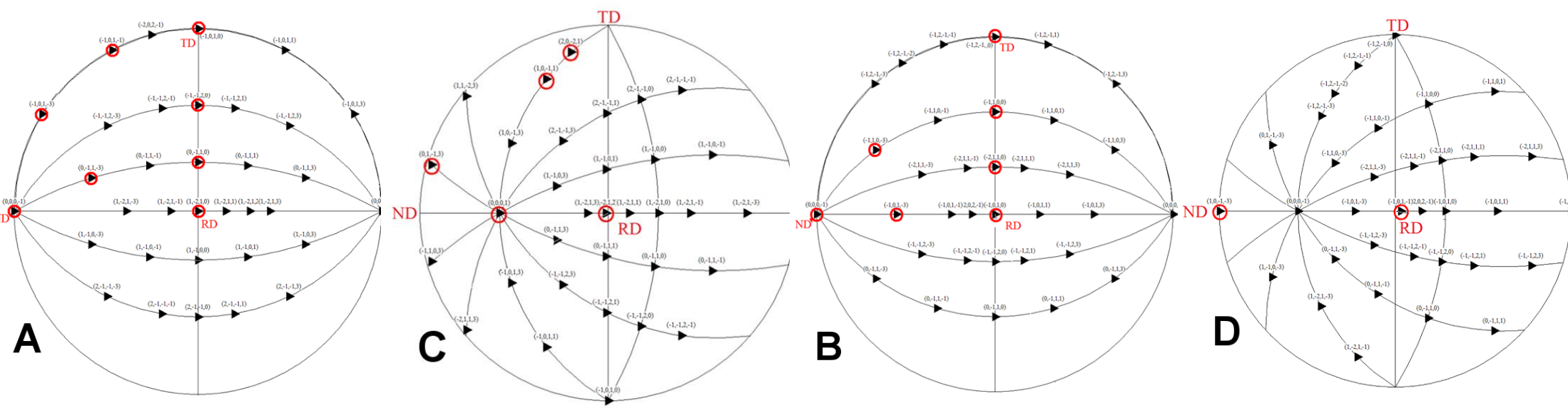
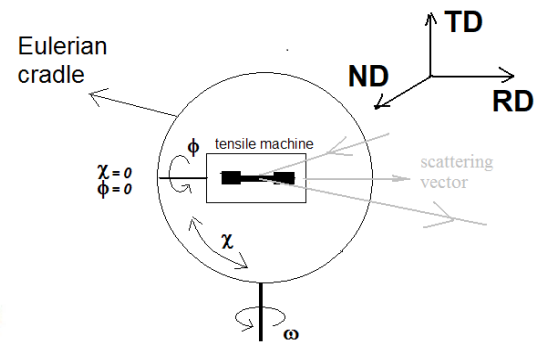
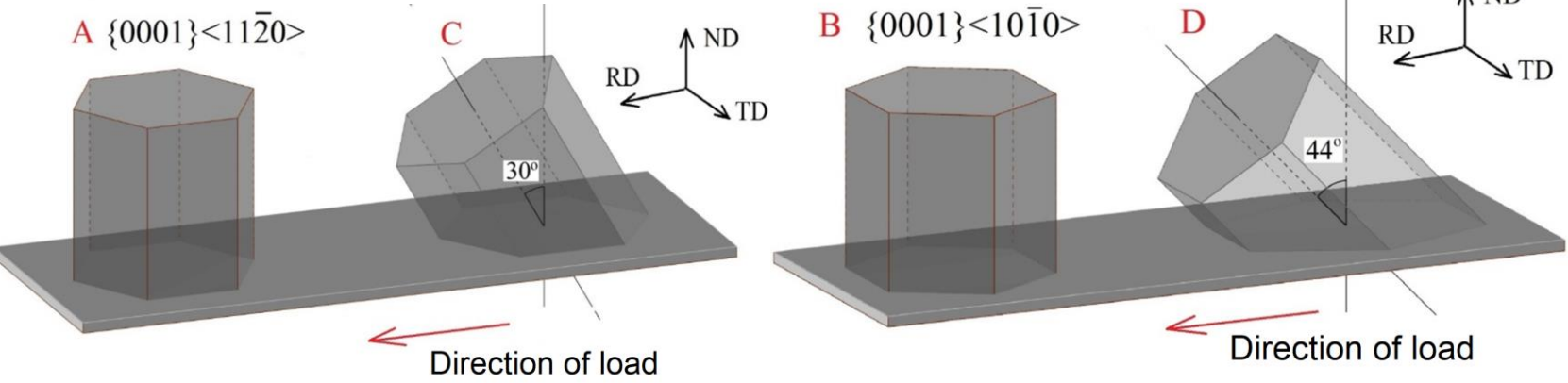


- A  $\{0001\}\langle 11\bar{2}0 \rangle$
- B  $\{0001\}\langle 10\bar{1}0 \rangle$
- ◆ C
- D

## EXP1



# ...crystal orientations-tensile RD (Exp. 1)

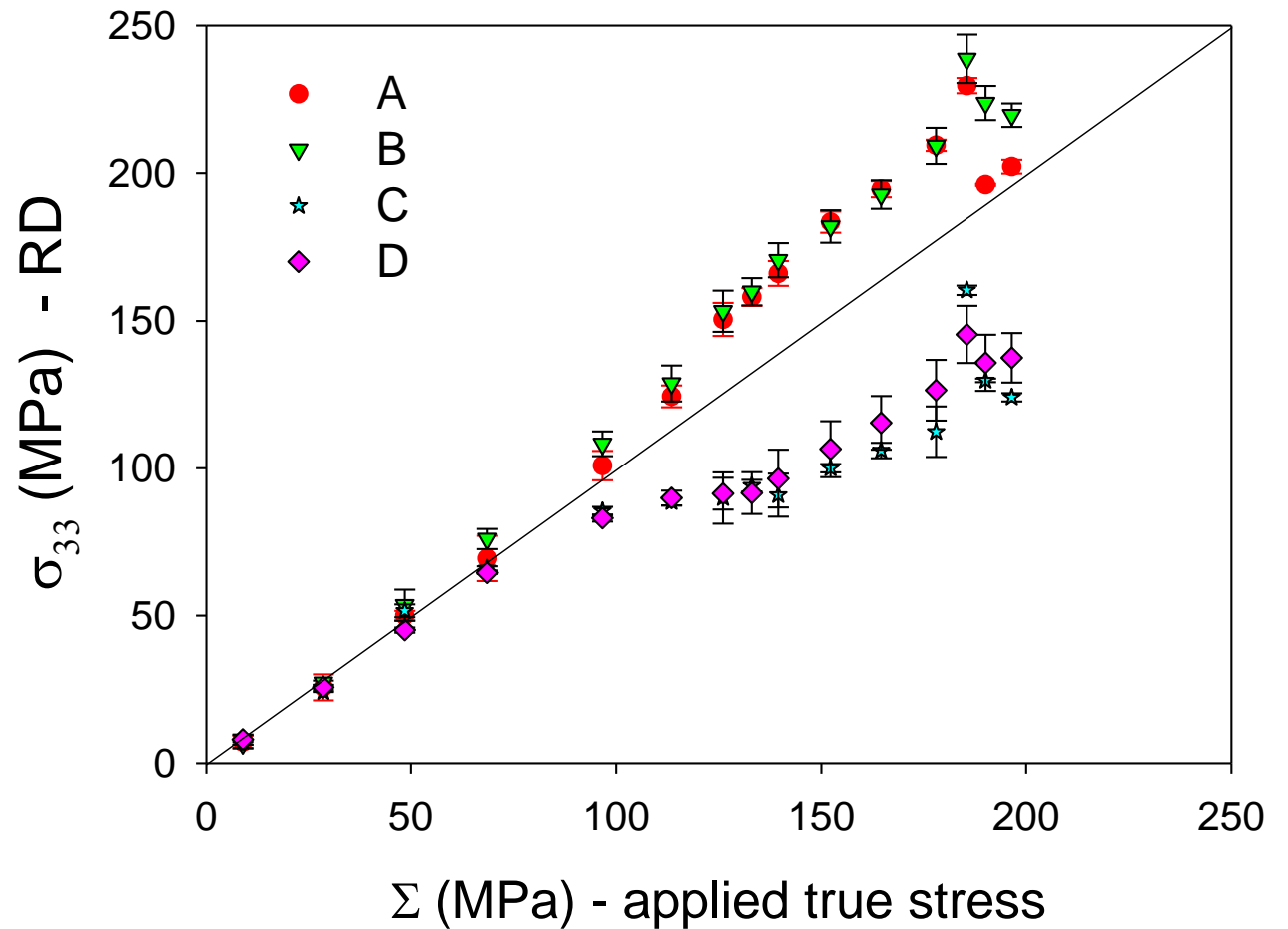
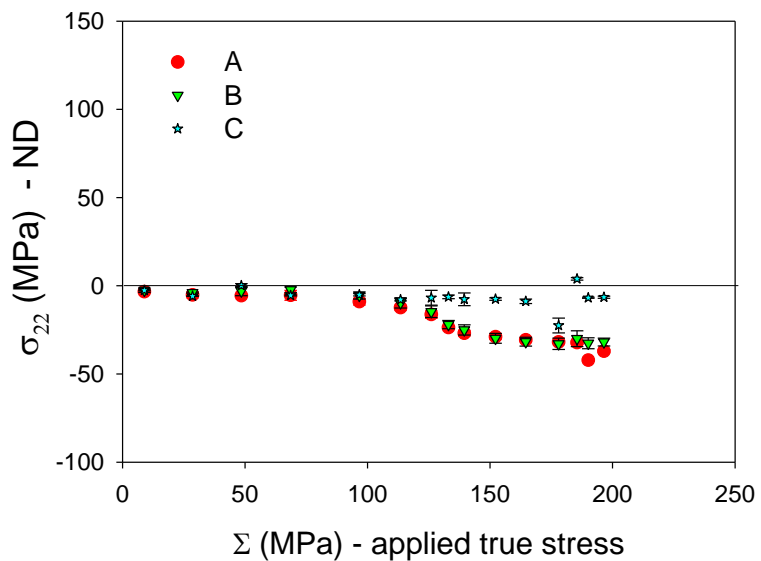
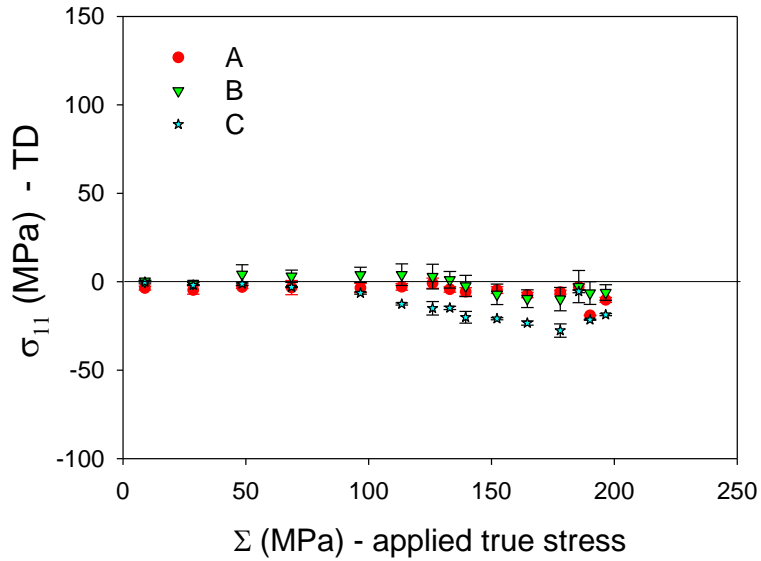


Least squares fitting

$$\langle \varepsilon(\varphi, \psi) \rangle_{\{hkl\}} = \gamma_{3i} \gamma_{3j} S_{ijkl}^g \sigma_{kl}^g$$

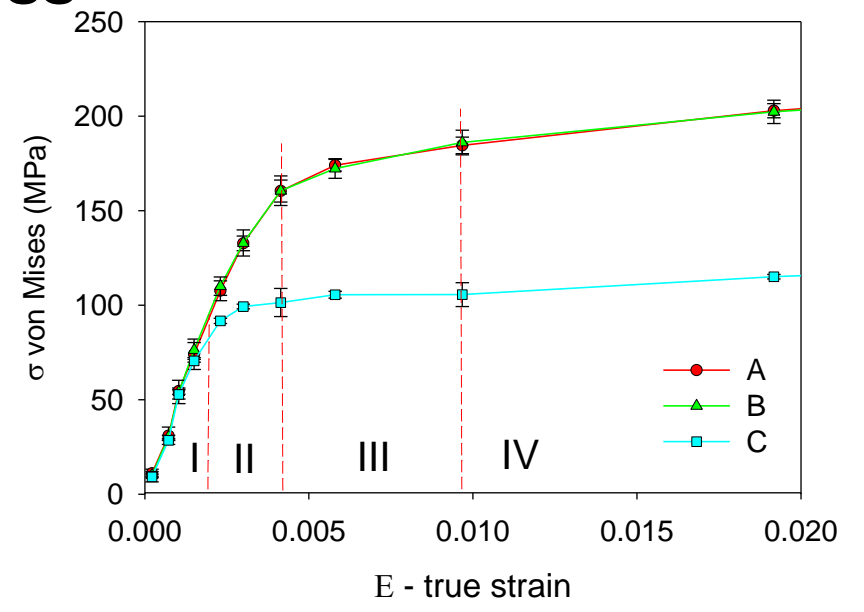
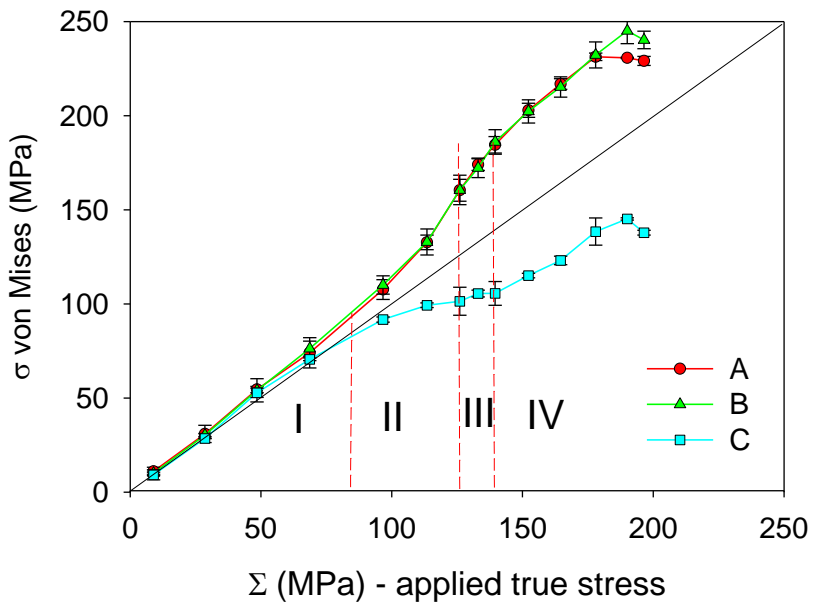
/ measured lattice strains      / direction cosines      / single crystal elastic constants  
 \ stress for group of crystals

# Determination of stress tensor for different loads-tensile RD (Exp. 1)

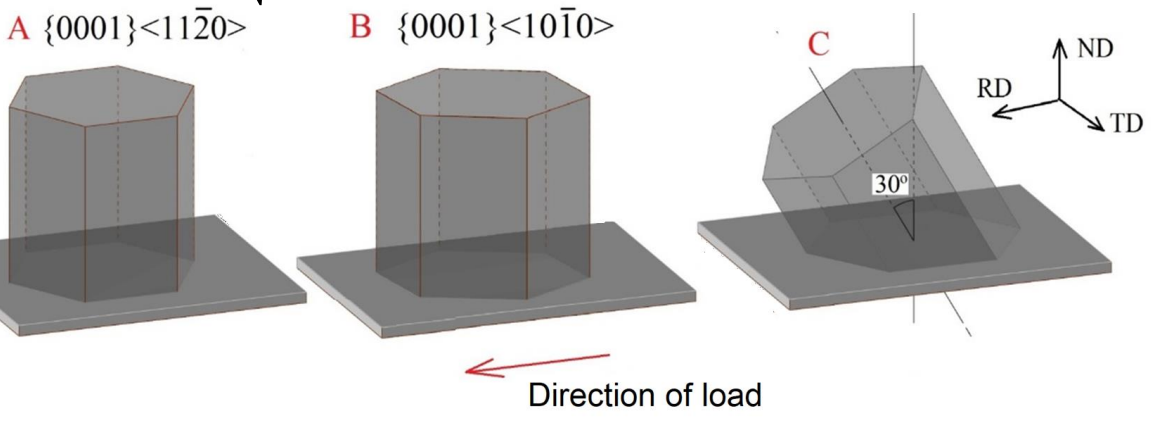


# Determined von Mises stress-tensile RD (Exp.1)

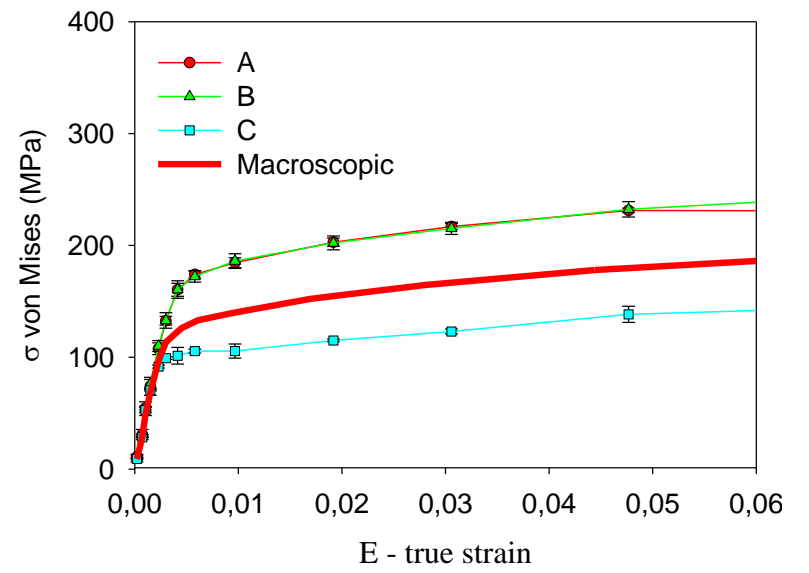
## von Mises stress



$$\sigma_{vonMises} = \sqrt{\frac{(\sigma_{11} - \sigma_{22})^2 + (\sigma_{11} - \sigma_{33})^2 + (\sigma_{22} - \sigma_{33})^2}{2}}$$



## Macroscopic stress

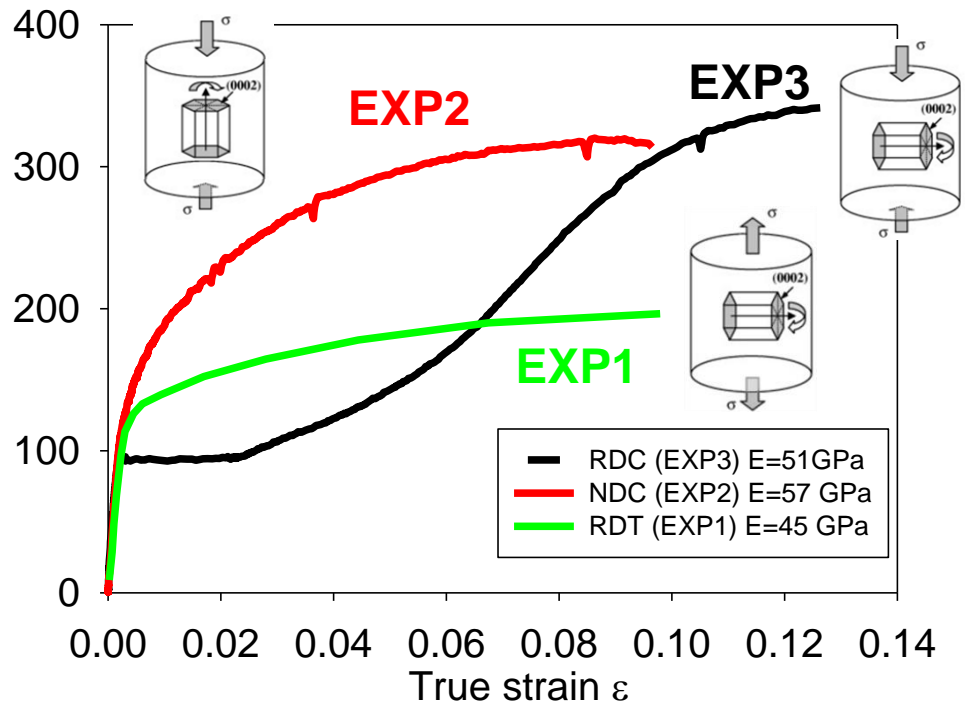
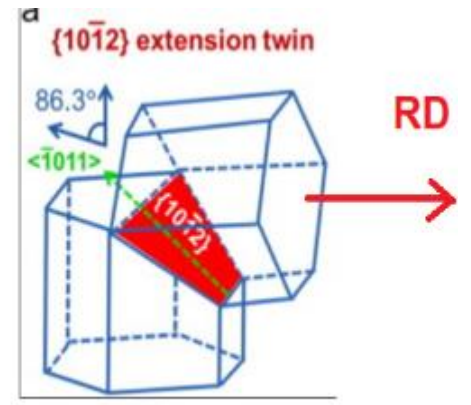
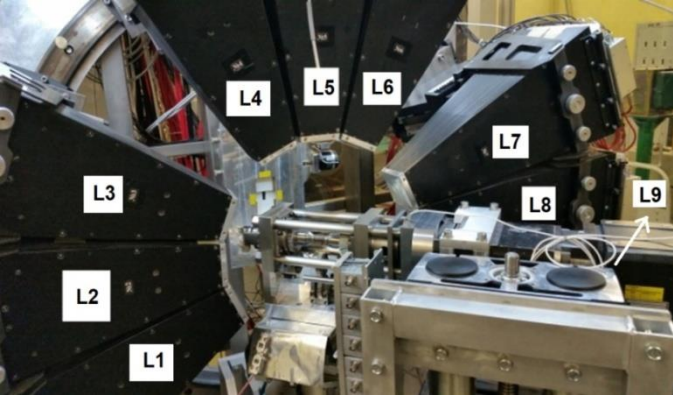




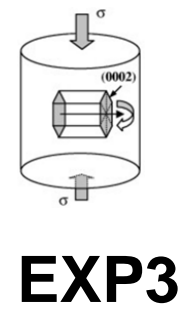
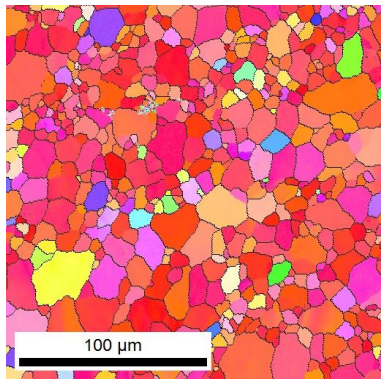
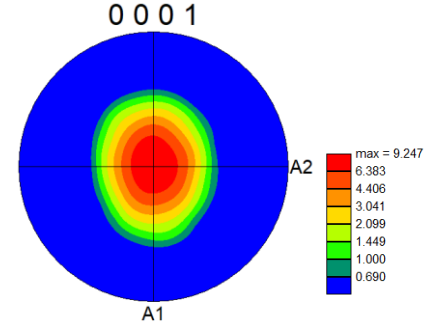


# **Compression test (EPSILON, FLNP, Dubna): stresses during twinning**

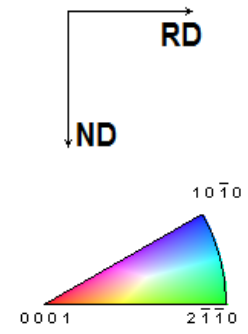
## Compression test in ND (Exp. 2) and RD (Exp. 3)



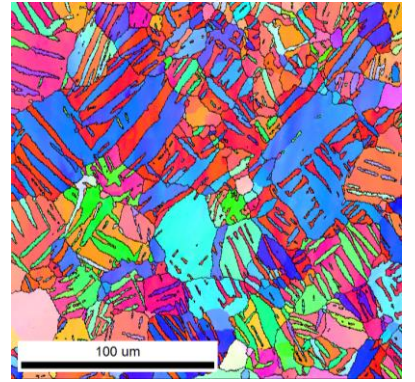
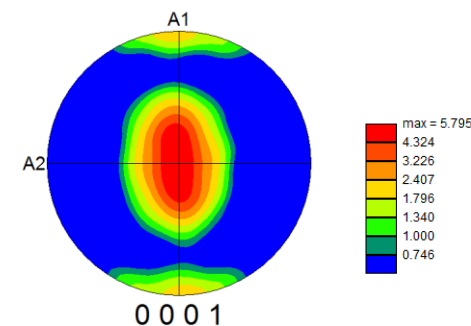
Initial state



EXP3



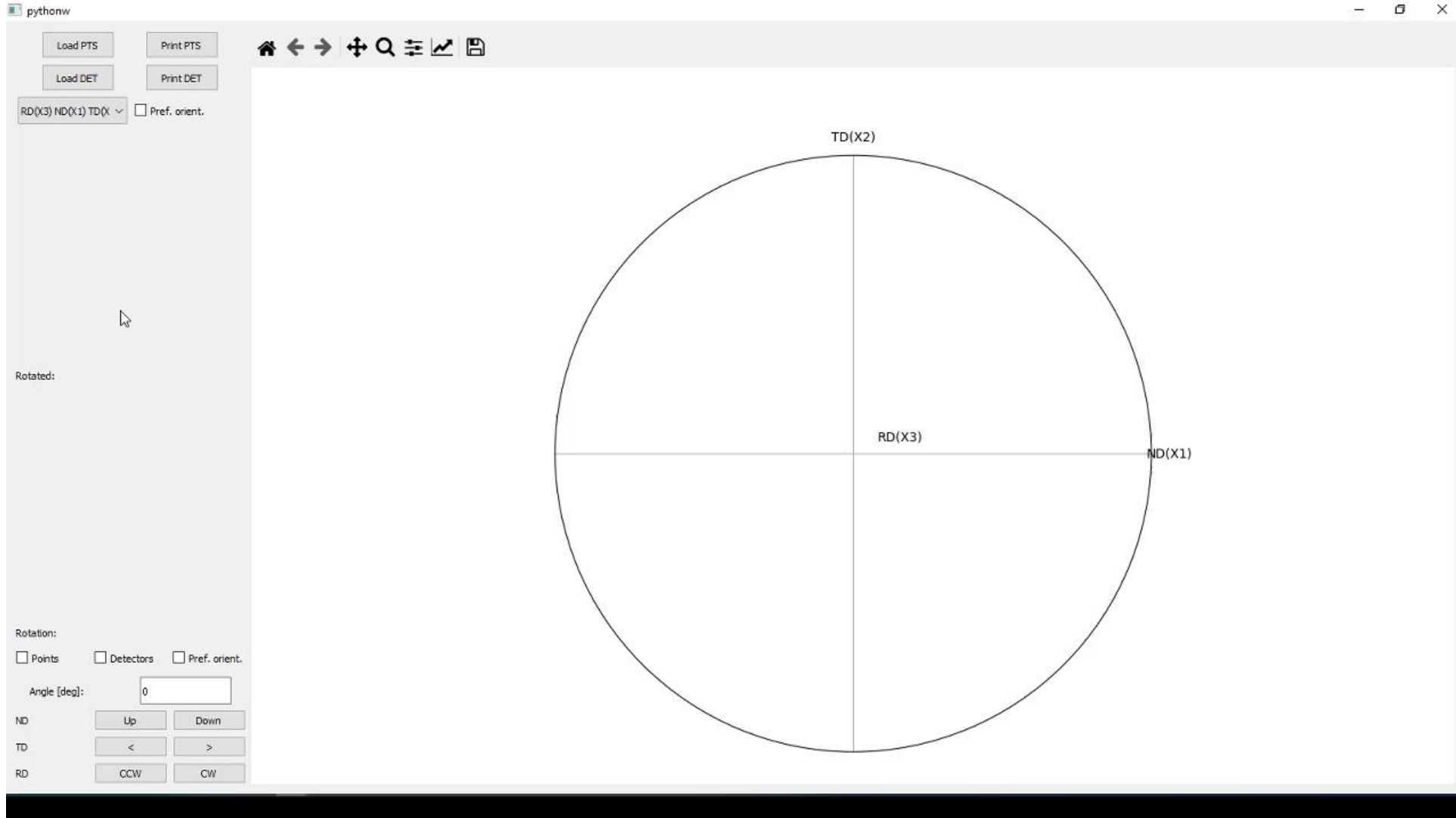
$\epsilon = 3\%$





# Crystallite group method – TOF in Dubna

# Search for orientations



pythonw

Load PTS Print PTS

Load DET Print DET

RD(X3) ND(X1) TD(X)  Pref. orient.

Rotated:

Rotation:

Points  Detectors  Pref. orient.

Angle [deg]:

ND Up Down

TD < >

RD CCW CW

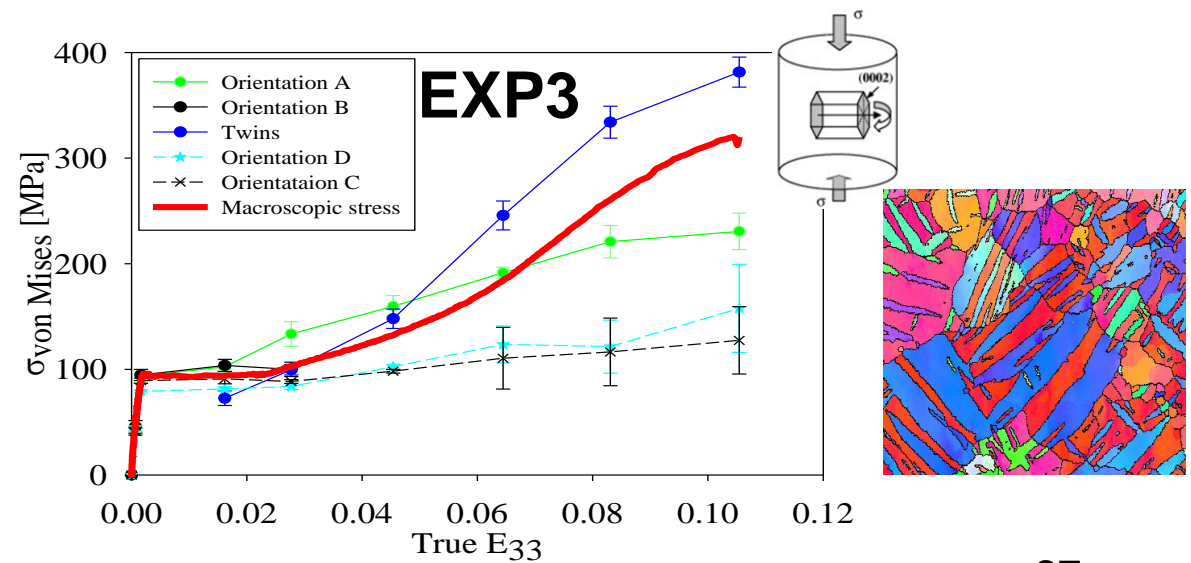
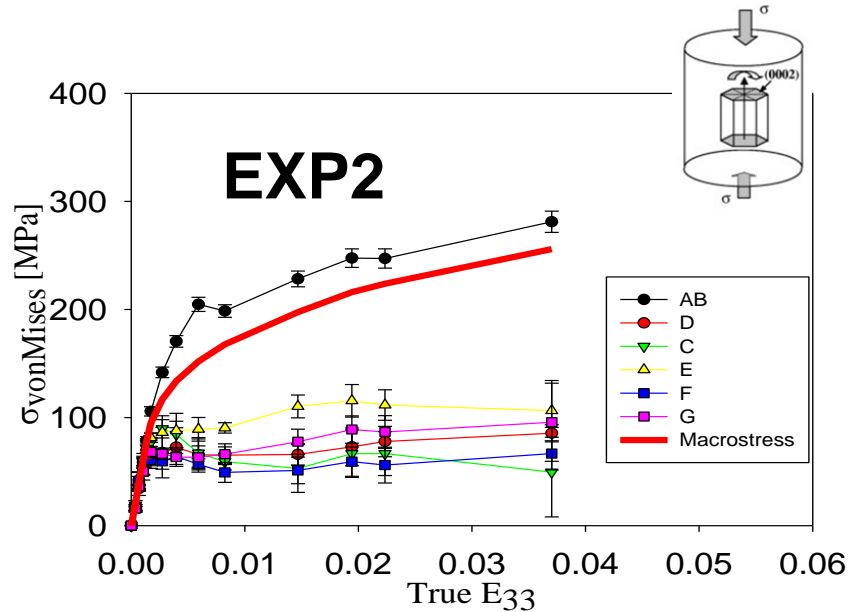
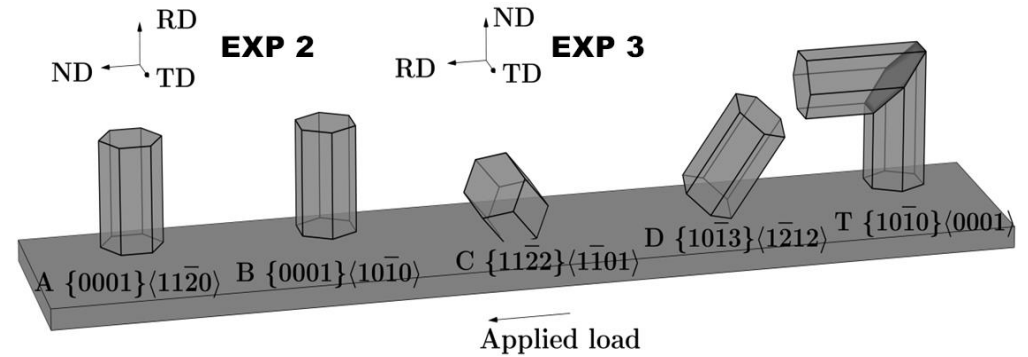
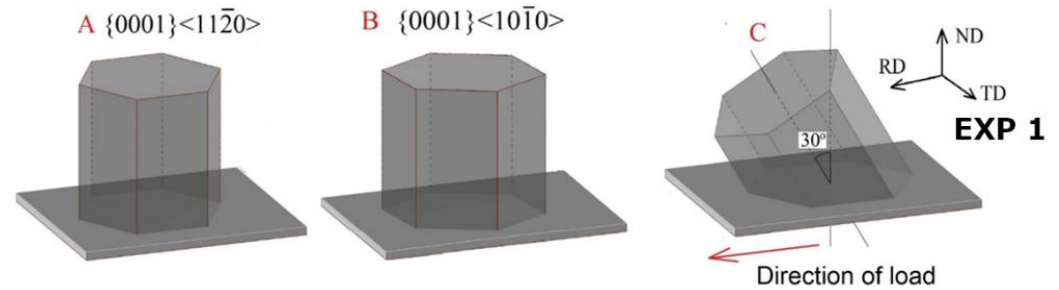
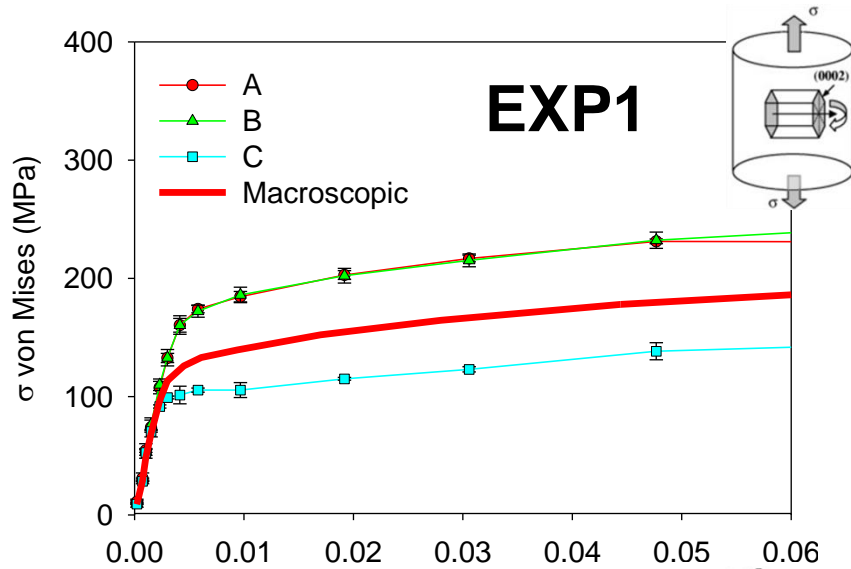
TD(X2)

RD(X3)

ND(X1)



# Von Mises Stresses

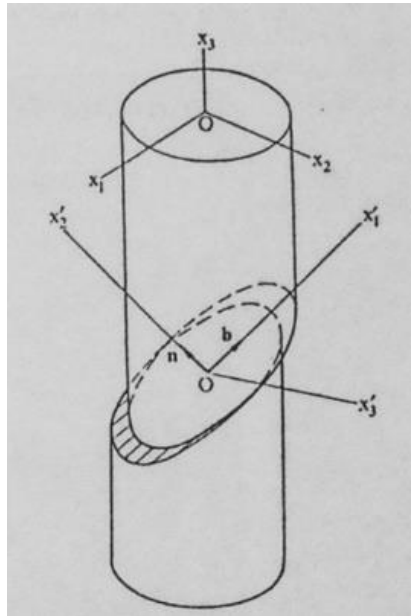


# Determination of critical resolved shear stress (CRSS)

# Calculation of Critical Resolved Shear Stresses (CRSS)

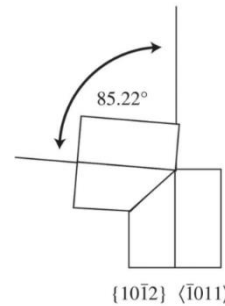
Tensile twinning

$$\tau = \sum_{i,j} m_i n_j \sigma_{ij}$$

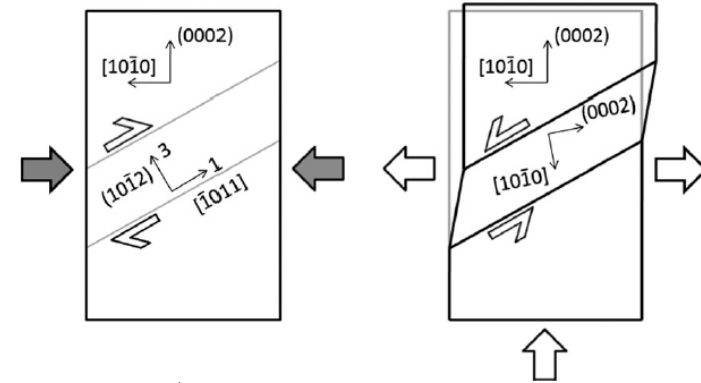


resolved shear stress:

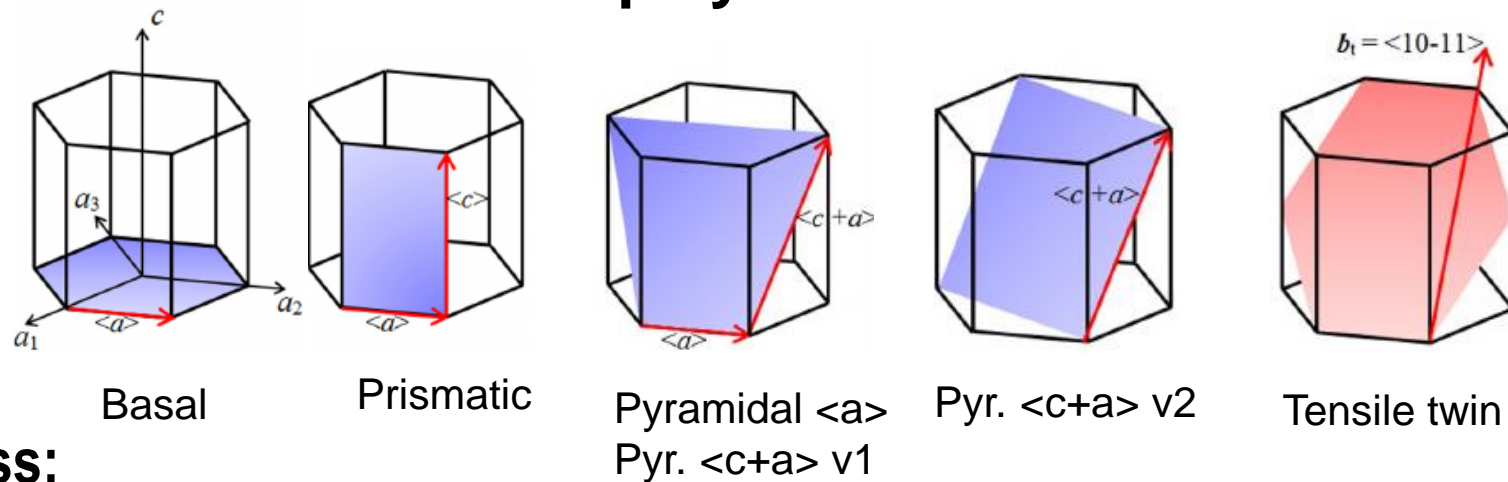
$$\tau = \sum_{i,j} m_i n_j \sigma_{ij}$$



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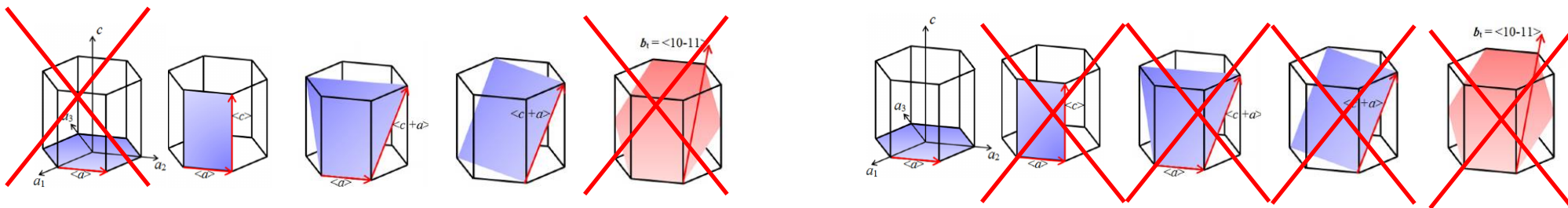
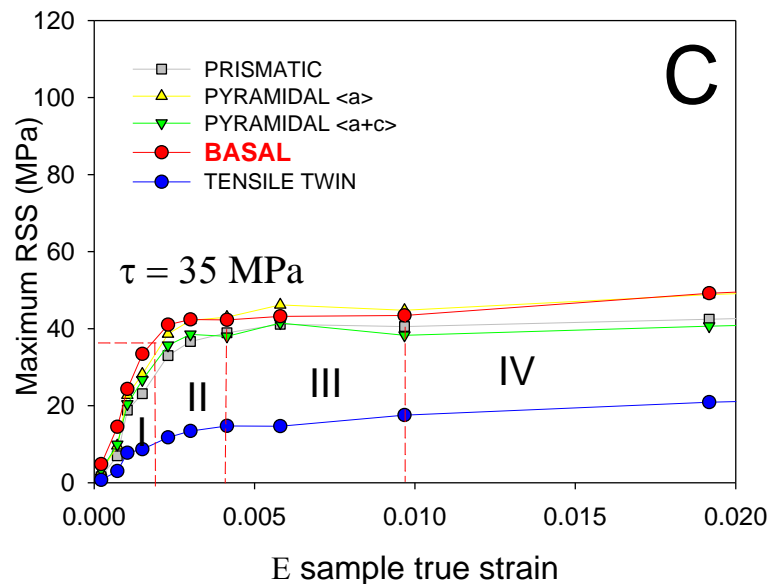
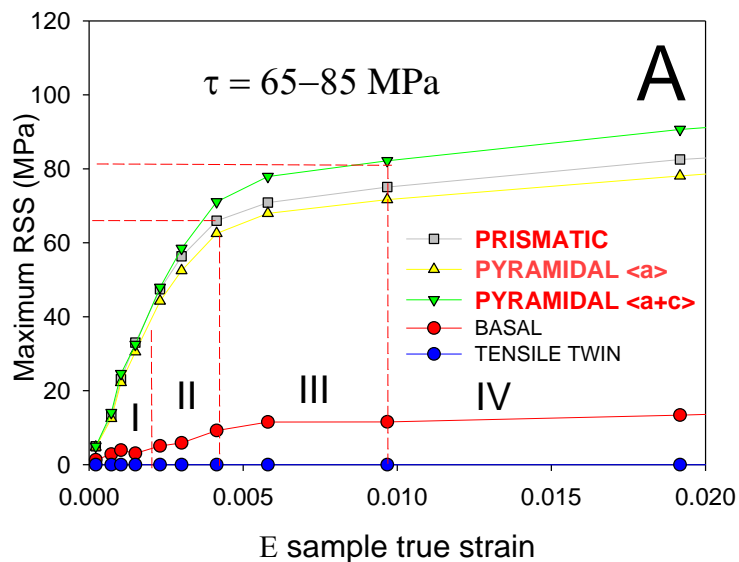
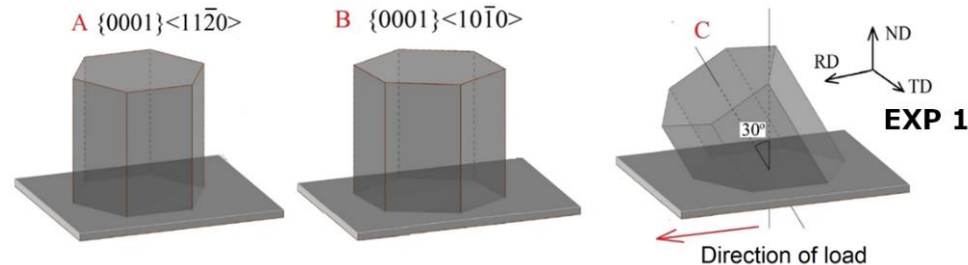
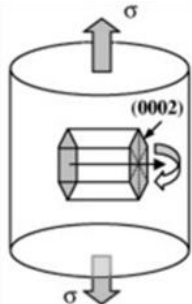


Slip systems



Selection of maximum resolved shear stress.

# Resolved shear stress – Exp. 1



CRSS: Basal ~35 MPa  
Non-basal > 65 MPa

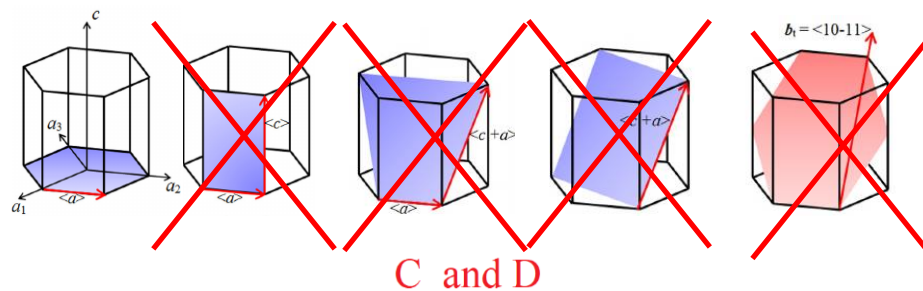
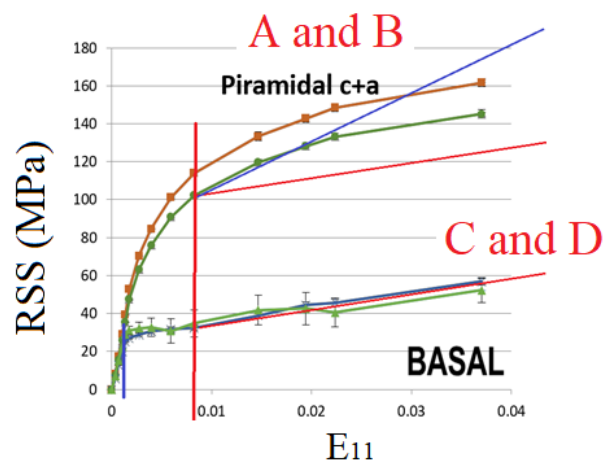
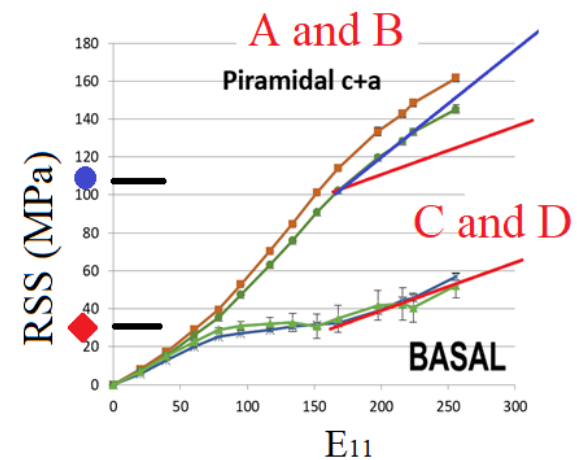
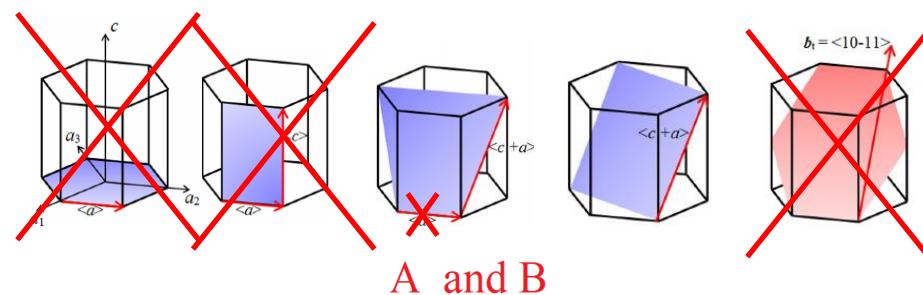
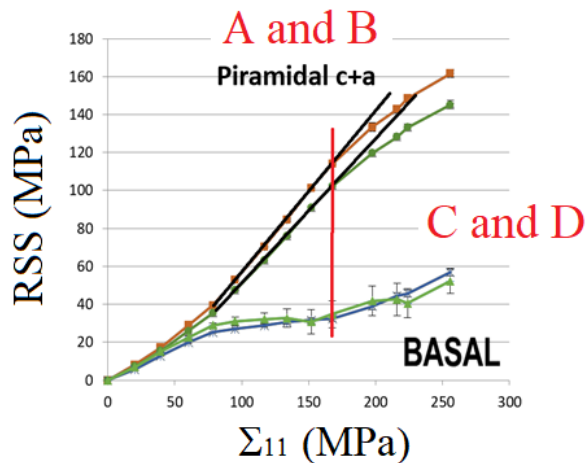
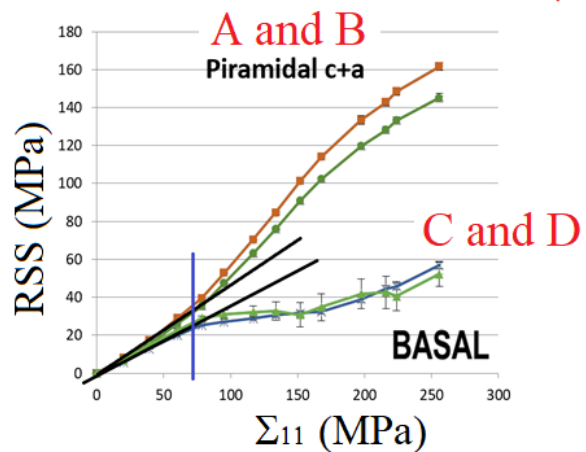
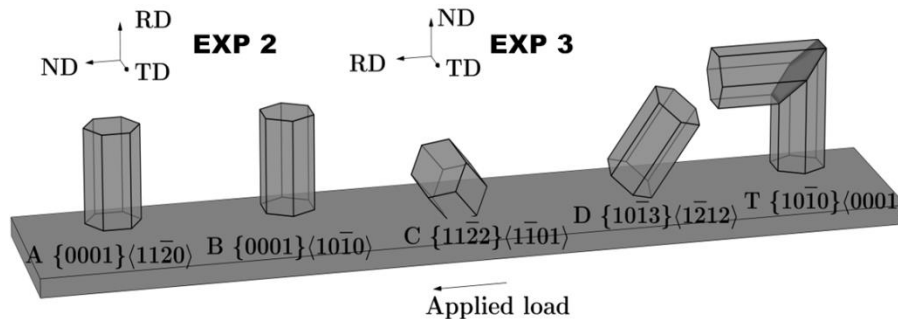
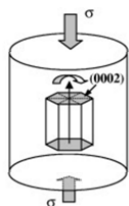


## CRSS (MPa) from Exp.1

Slip system	Neutron Diffraction - direct method CRSS [MPa]
<b>Basal B</b> $\{0001\}\langle 11\bar{2}0 \rangle$	35
<b>Prismatic P</b> $\{10\bar{1}0\}\langle 11\bar{2}0 \rangle$	65 - 85
<b>Pyramidal <math>\pi\langle a \rangle</math></b> $\{10\bar{1}1\}\langle 11\bar{2}0 \rangle$	
<b>Pyramidal <math>\pi_1 \langle c+a \rangle</math></b> $\{10\bar{1}1\}\langle 11\bar{2}3 \rangle$	

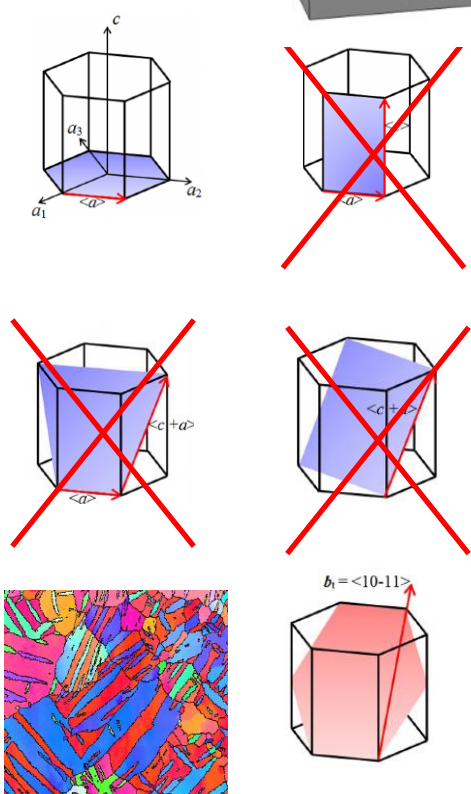
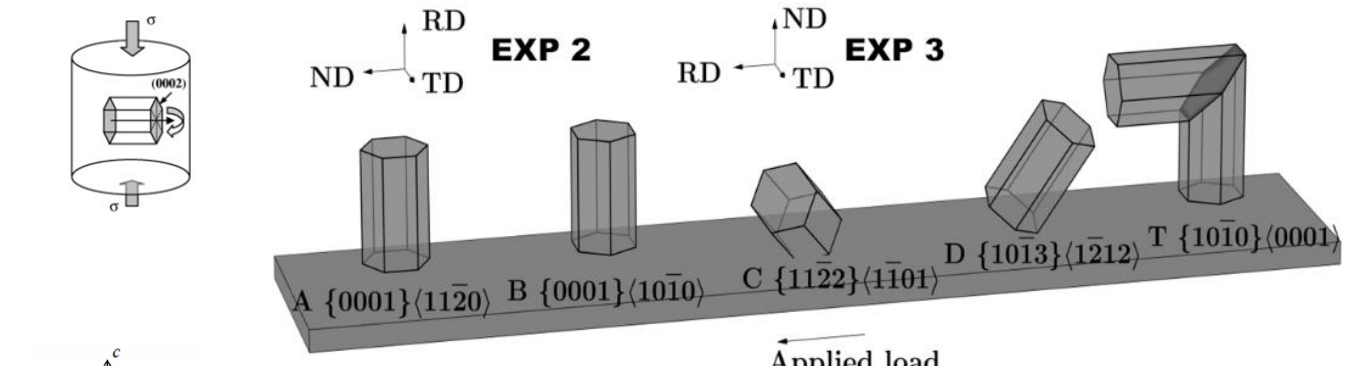
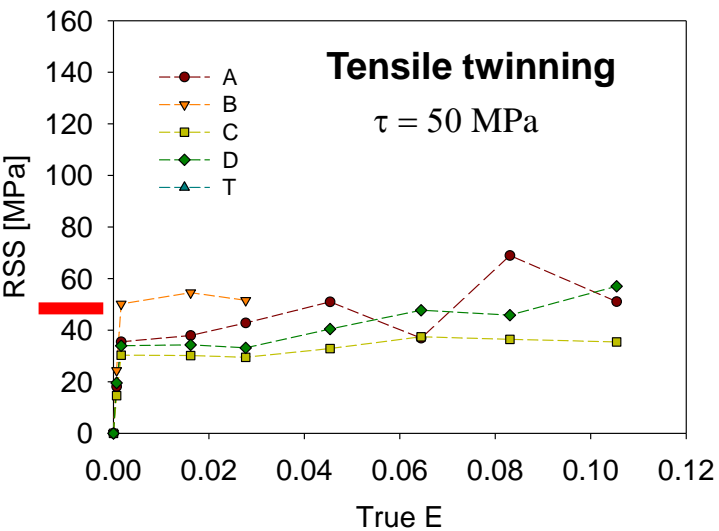
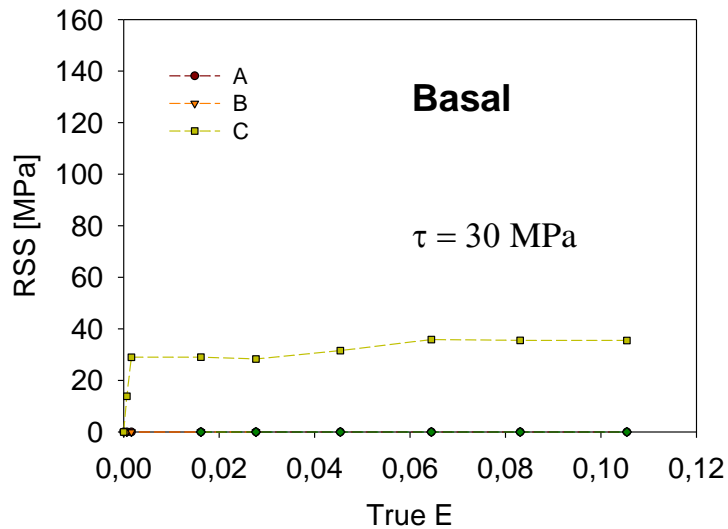
# Resolved shear stress – Exp. 2

EXP2



CRSS: ● Piramidal ~110 MPa  
 ◆ Basal ~30 MPa

# Resolved shear stress – EXP 3

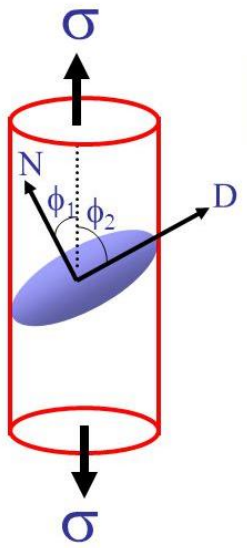


Slip system	CRSS [MPa]
<b>Basal B</b> {0001}<11-20>	~ 30
Prismatic P {10-10}<11-20>	65-85 (?)
Pyramidal <a> {10-11}<11-20>	65-85 (?)
<b>Pyramidal &lt;c+a&gt;</b> {10-11}<11-23>	~ 110
<b>Tensile Twin</b> {10-12}<10-11>	~ 55

\* Direct diffraction measurement of critical resolved shear stresses and stress localisation in magnesium alloy, A. Baczmański, P. Kot, S. Wroński, M. Wróbel, M. Wroński, J. Pilch, M. Muzyka, K. Wierzbowski, Y. Zhao, L. Le Joncour, M. François, B. Panicaud, *Materials Science & Engineering A*, 2021 vol. 801

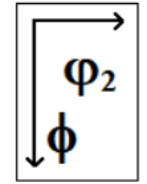
The role of basal slip in the generation of intergranular stresses in magnesium alloy studied using X-ray diffraction and modelling, A. Baczmański, M. Wroński, P. Kot, S. Wroński, A. Łabaza, K. Wierzbowski, A. Ludwik, M. Marciszko-Wiąckowska, *Materials & Design* 2021 202 ISSN: 0264-1275

# Next measurement – PRELUDIUM 20 (wniosek)

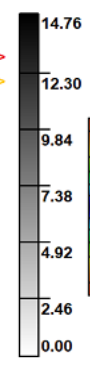
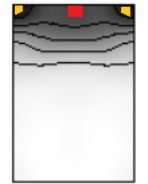


$$\tau = \sigma \cos \phi_1 \cos \phi_2$$

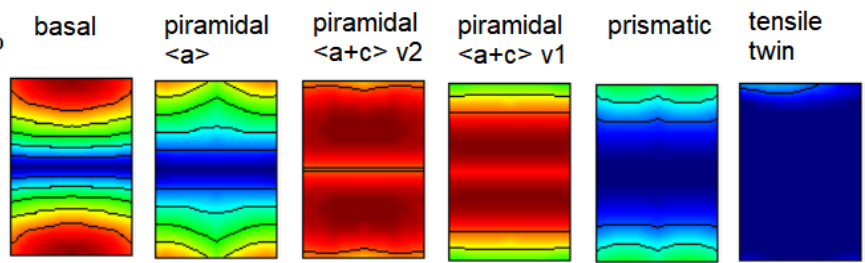
$\phi_1 = 90^\circ$



ODF  
 ■ A {0001} <1120>  
 ■ B {0001} <1010>

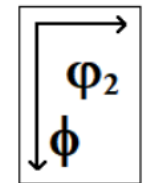


Schmid factor

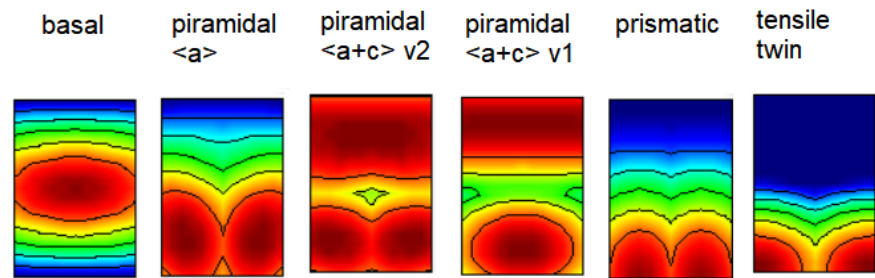
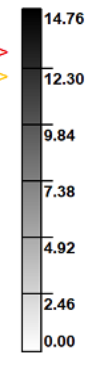


Schmid factor

$\phi_1 = 90^\circ$



ODF  
 ■ A {0001} <1120>  
 ■ B {0001} <1010>





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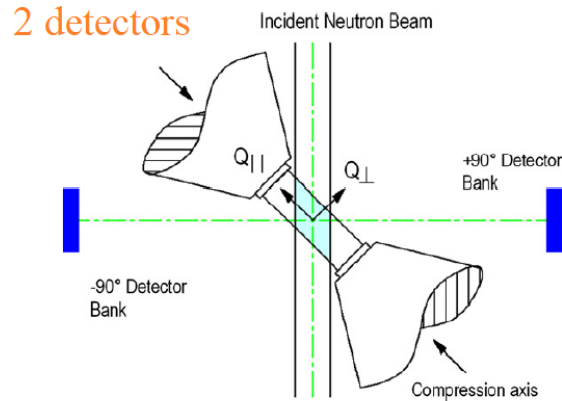
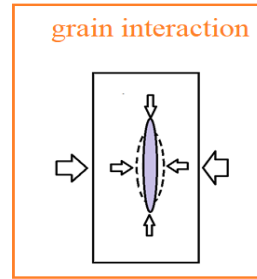


Fig. 3. Schematic of the in situ compression set-up of the SMARTS instrument.



Voce law - dislocation hardening

$$\hat{\tau}^\alpha = \tau_0^\alpha + (\tau_1^\alpha + h_1^\alpha \gamma_{ac}) \left( 1 - \exp \left( -\frac{h_0^\alpha}{\tau_1^\alpha} \gamma_{ac} \right) \right)$$

- Grain interaction depend on model – assumption
- Hardening - optimisation

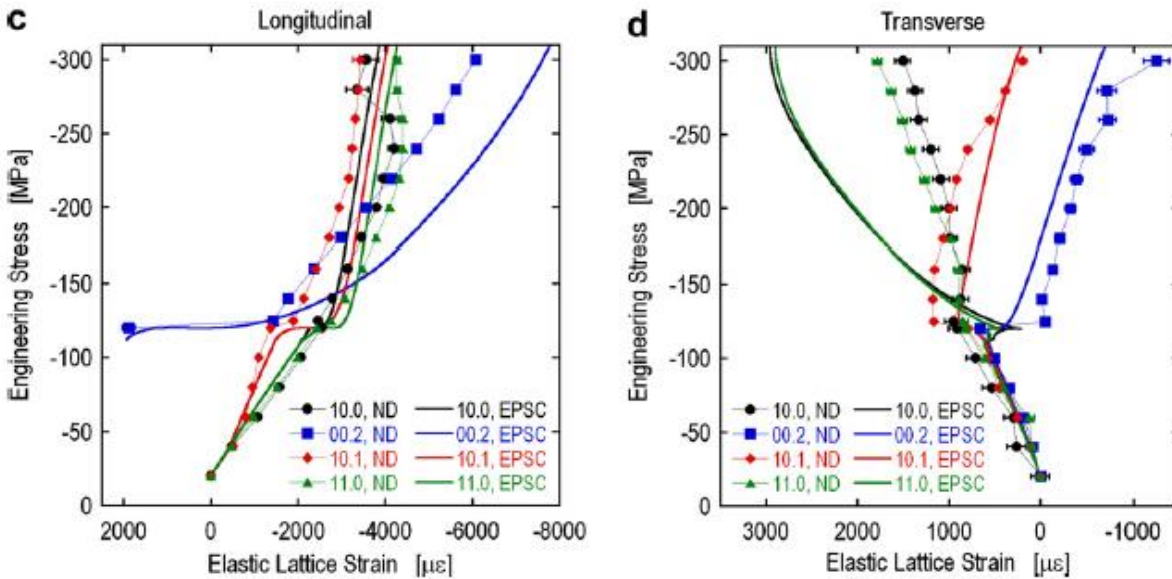


Fig. 8. Measured (line and symbol) and predicted (thick line) elastic lattice strains for the 'Continuity' assumption: (a) parallel to the loading axis; (b) perpendicular to the loading axis; and for the 'FIF' assumption: (c) parallel to the loading axis; and (d) perpendicular to the loading axis.

Table 1  
CRSS and hardening parameters used for the two assumptions

Assumption	Initial twin fraction	Deformation system	$\tau_0$ (MPa)	$\tau_1$ (MPa)	$\theta_0$ (MPa)	$\theta_0$ (MPa)
Continuity	N/A	Basal	12	20	40	0
		Prism	60	20	40	0
		Pyramidal	100	117	2500	0
		Tensile twin	54	0	0	0
Fixed initial fraction	3%	Basal	12	20	240	0
		Prism	60	20	240	0
		Pyramidal	100	117	2000	0
		Tensile twin	60	0	0	0

**Table 1**

List of material constants for various self-consistent models.

Model	Mode	$\tau_0$	$\tau_1$	$h_0$	$h_1$	Latent	$A^{th1}$	$A^{th2}$
Affine	Basal	9	1	5000	25	4	0.72	0
	Prismatic	79	40	590	50	4		
	Pyramidal	100	100	5000	0	2		
	Tensile twin	47	0	0	0	4		
Secant	Basal	13	4	5000	30	4	0.82	0
	Prismatic	73	35	400	60	4		
	Pyramidal	110	83	2500	0	2		
	Tensile twin	31	0	0	0	4		
$m^{eff}$ ( $m^{eff} = 0.1$ )	Basal	17	6	3800	100	4	0.81	0
	Prismatic	77	33	650	50	4		
	Pyramidal	148	35	9600	0	2		
	Tensile twin	33	0	0	0	4		
Tangent	Basal	21	5	3000	140	4	0.81	0
	Prismatic	90	15	580		4		
	Pyramidal	145	30	9600	70	2		
	Tensile twin	38	0	0	0	4		

Q. Chen et al. *Materials Science & Engineering A* 774 (2020) 138912

Assessment in predictability of visco-plastic self-consistent model with a minimum parameter approach: Numerical investigation of plastic deformation behavior of AZ31 magnesium alloy for various loading conditions

## Model + diffraction

## Direct method

H. Wang et al./International Journal of Solids and Structures 47 (2010) 2905–2917

**Table 1**  
List of material constants for various self-consistent models.

Model	Mode	$\tau_0$
Affine	Basal	9
	Prismatic	79
	Pyramidal	100
	Tensile twin	47
Secant	Basal	13
	Prismatic	73
	Pyramidal	110
	Tensile twin	31
$m^{eff}$ ( $m^{eff} = 0.1$ )	Basal	17
	Prismatic	77
	Pyramidal	148
	Tensile twin	33
Tangent	Basal	21
	Prismatic	90
	Pyramidal	145
	Tensile twin	38

Slip system	CRSS [MPa]
<b>Basal B</b> $\{0001\}\langle 11\bar{2}0 \rangle$	~ 30
Prismatic P $\{10\bar{1}0\}\langle 11\bar{2}0 \rangle$	65-85 (?)
Pyramidal <a> $\{10\bar{1}1\}\langle 11\bar{2}0 \rangle$	65-85 (?)
<b>Pyramidal &lt;c+a&gt;</b> $\{10\bar{1}1\}\langle 11\bar{2}3 \rangle$	~ 110
<b>Tensile Twin</b> $\{10\bar{1}2\}\langle \bar{1}011 \rangle$	~ 50

# Podsumowanie

- 1. Dla materiałów fazowych nasza metoda pozwala określić co dzieje się w każdej z faz materiału, jak te fazy oddziałują (Al/SiC – relaksacja naprężeń termicznych).**
- 2. Dla materiałów stekstrowanych możemy opisać lokalizację naprężeń na grupach kryształitów i opisać aktywowanie poślizgów i bliźniakowania**
- 3. W przypadku badanego stopu magnezu występują 3 typy ziaren – miękkie, twarde i ulegające bliźniakowaniu (zależy to od sekwencji uruchamianych poślizgów i bliźniakowania)**
- 4. Wyniki badań są jednoznaczne – nie potrzebujemy dodatkowych założeń używanych w modelu. Możliwa jest weryfikacja modeli**

#### **Podziękowania:**

Praca została częściowo dofinansowana z grantów Narodowego Centrum Nauki nr UMO-2017/25/B/ST8/00134 i UMO-2015/19 / D / ST8 / 00818.  
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Eksperymenty z dyfrakcją neutronów były współfinansowane przez Pełnomocnika Rządu RP w ZIBJ w Dubnej w ramach Projektu nr 75/28/2020.





**Dziękuję za uwagę.**