# *RESIDUAL STRESS MEASUREMENTS BY X-RAY DIFFRACTION: CRITICAL EVALUATION OF ERROR SOURCES*

**Rogério Machado[1]**,

Alexei Kuznetsov[1],

Carlos Alberto Achete[1, 2]

Thomas Hirsch[3]

1 Divisão de Metrologia de Materiais (DIMAT), Inmetro, CEP 25250-020, Xerém, Duque de Caxias, RJ, Brazil

2 Programa de Engenharia Metalúrgica e de Materiais (PEMM), Universidade Federal do Rio de Janeiro, Cx. Postal 68505, CEP 21945-970, Rio de Janeiro, RJ, Brazil

3 Institut fuer Werkstofftechnik, Bremen, Germany

# MOTIVATION

***NACIONAL ROUND ROBIN FOR RESIDUAL STRESS MEASUREMENTS***

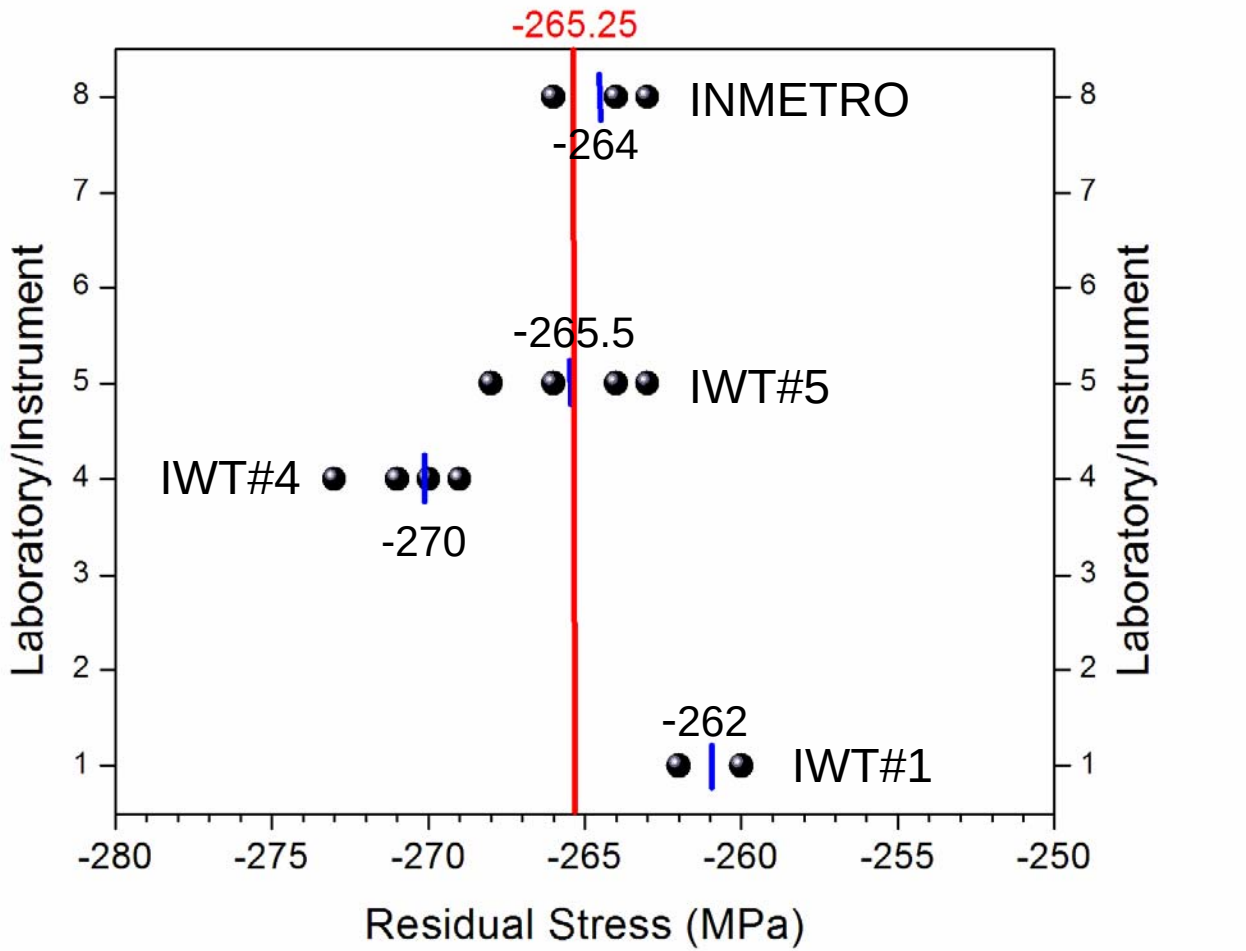
# PRELIMINARY STUDY

***Sample(s) choice***

***Definition of the method of data processing***

***Definition of experimental parameters***
***(Evaluation of influence of experimental conditions)***

## INTERCOMPARISON IWT-INMETRO (NOV/2007) [Sample A0b]
## [5 analysis per instrument]



# Sample properties (partially defined by sample preparation methods)

***ELASTIC CONSTANTS (+/- 5% RELATIVE)***

*AISI 1070, 1/2S2 = 5.7 $10^{-6}$ MPa; S1 = -1.235 $10^{-6}$ MPa*

***SAMPLE SHAPE (PLANE, CILINDER, …)***

*Plates of 20mm X 25mm (s11 direction)*

***MICROSTRUCTURE***

*Homogeneous tempered martensite with small carbides*

***PRESENCE OF STRESS/STRAIN GRADIENTS***

*A little gradient at surface showed by Cu radiation measurements*

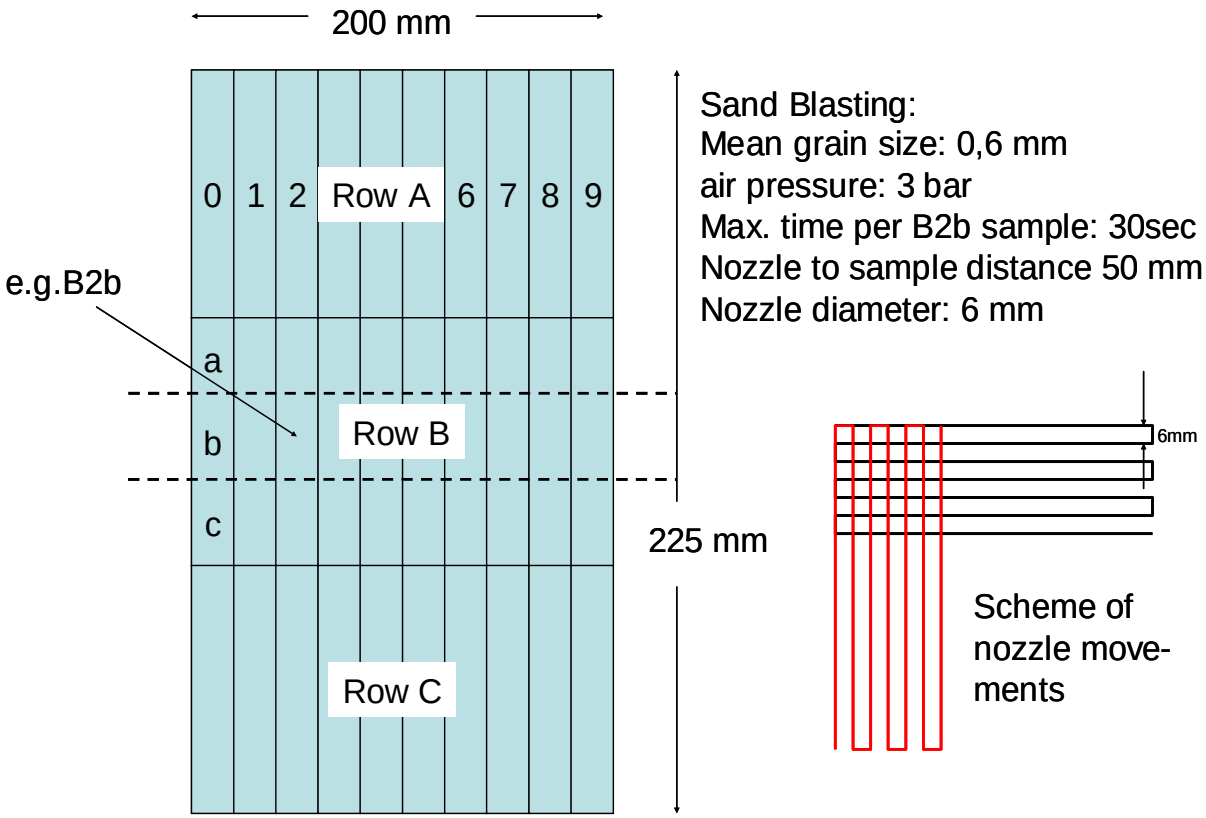
***PRESENCE OF CRYSTALLOGRAPHIC TEXTURE***

*Quasi ideally random texture*

# Sample properties (partially defined by sample preparation methods)

**Chemical Composition** C=0,73, Mn=0,64, P=0,010, S=0,001, Si 0,19, Cr=0,18, V=0,01 Ni=0,02 Mo=0,030 Al=0,041

**Average hardness** of 270 HV0,5. **Microstructure:** homogeneous tempered martensite with small carbides.

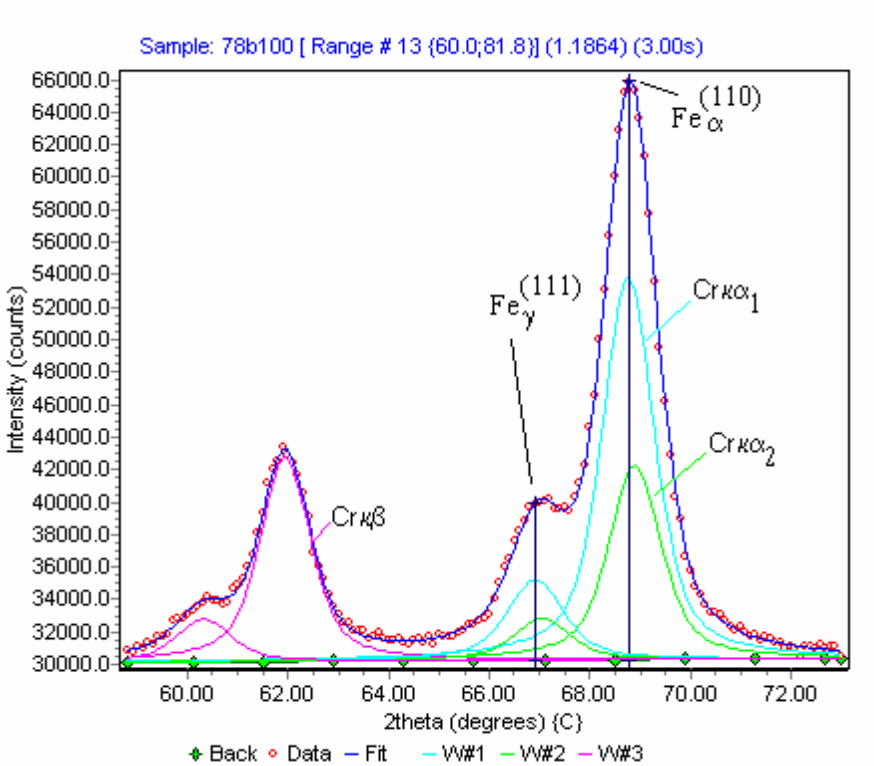



MANUFACTURED AT IWT/BREMEM/GERMANY (Dr. Thomas Hirsch)

# Method of data processing

## NUMERICAL METHOD FOR PEAK POSITION DETERMINATION

*Numerical fitting of Ka1 and Ka2 (and Kb) with symmetrical Pseudo Voight function.*
*No convolutions applied. Linear Background Subtraction.*



# Method of data processing

## ALGORITHM OF ANALYSIS (SOFTWARE)

**Based on traditional least-square fit of d x sen²PSi curves to experimental data (isotropic case)**

$$\varepsilon_{\psi\varphi}^{hkl} = \varepsilon_{00}^{hkl} + \frac{1}{2} S_2^{hkl} \sigma_\varphi \sin^2\psi + \frac{1}{2} S_2^{hkl} \tau_\varphi \sin(2\psi)$$

$$\varepsilon_{00}^{hkl} = \frac{d_{00}^{hkl} - d_{unstrained}^{hkl}}{d_{unstrained}^{hkl}} = S_1^{hkl}(\sigma_{11} + \sigma_{22} + \sigma_{33}) + \frac{1}{2} S_2^{hkl} \sigma_{33}$$

$$\sigma_\varphi = \sigma_{11}\cos^2\varphi + \sigma_{12}\mathrm{sen}(2\varphi) + \sigma_{22}\sin^2\varphi - \sigma_{33}$$

$$\tau_\varphi = \sigma_{31}\cos\varphi + \sigma_{23}\sin\varphi$$

$$\frac{1}{2} S_2^{hkl} = \left(\frac{1+\nu}{E}\right)^{hkl} = 5.710^{-6}\,MPa$$

$$S_1^{hkl} = -\left(\frac{\nu}{E}\right)^{hkl} = -1.23510^{-6}\,MPa$$

# Method of data processing

## ALGORITHM OF ANALYSIS (SOFTWARE)

### Additional assumption:

$$d_{00}^{hkl} \cong d_{unstrained}^{hkl} \longrightarrow \varepsilon_{\psi\varphi}^{hkl} = \left(\frac{1+\nu}{E}\right)^{hkl} \left(\sigma_\varphi \sin^2\psi + \tau_\varphi \sin\psi\right)$$

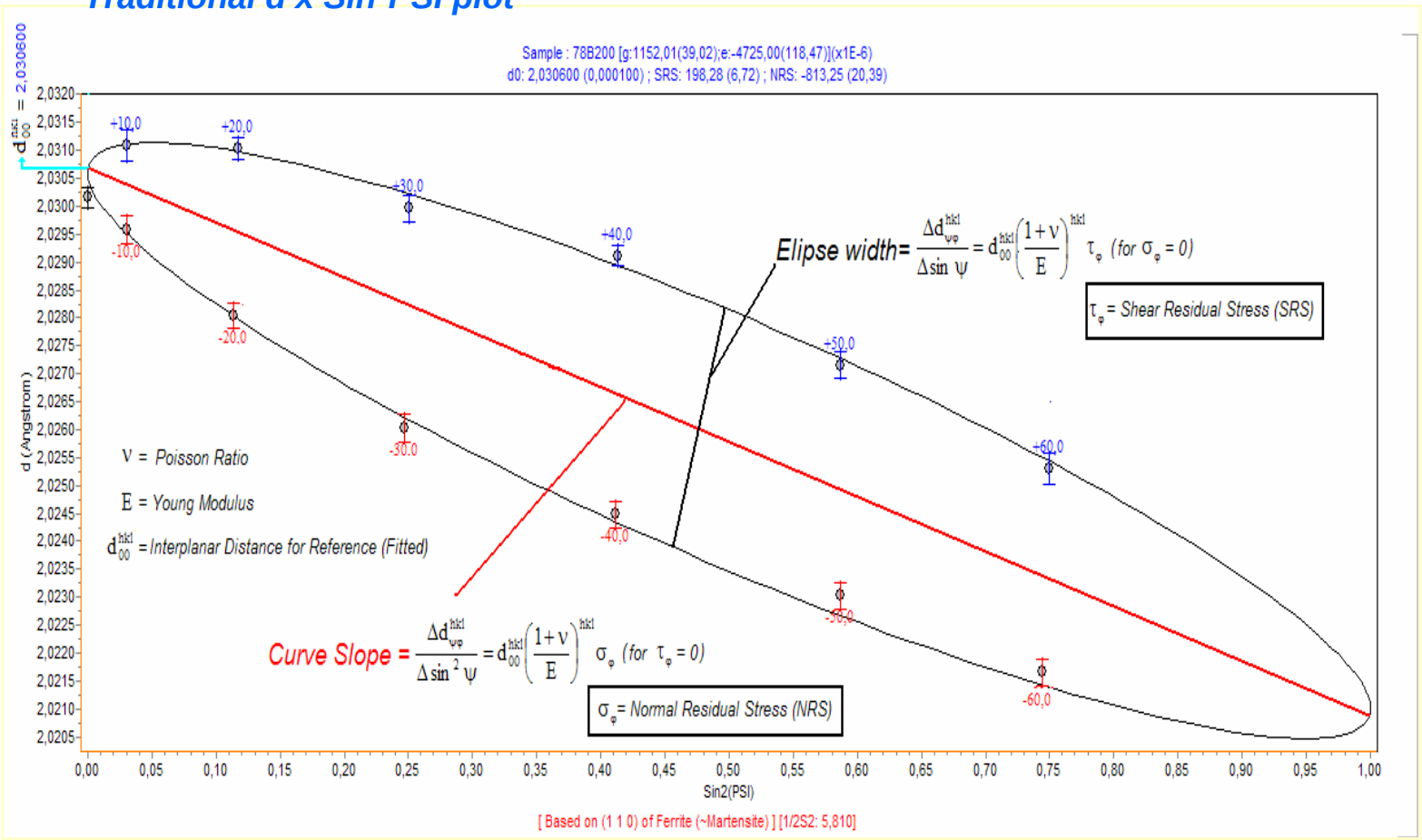
$$\downarrow$$

$$d_{\psi\varphi}^{hkl} = d_{00}^{hkl}\left(1 + \left(\frac{1+\nu}{E}\right)^{hkl} \left(\sigma_\varphi \sin^2\psi + \tau_\varphi \sin\psi\right)\right)$$

# Method of data processing

## ALGORITHM OF ANALYSIS (SOFTWARE)

### Traditional d x Sin²PSI plot



# *Experimental conditions*

***COUNTING TIME (STEP TIME/SCAN VELOCITY)***

***NUMBER OF PSI TILTS AND PSI $_{MAX}$***

***2THETA RANGE USED FOR BACKGROUND SUBTRACTION***

***GEOMETRY (PSI,OMEGA,LINE FOCUS, POINT FOCUS,…)***

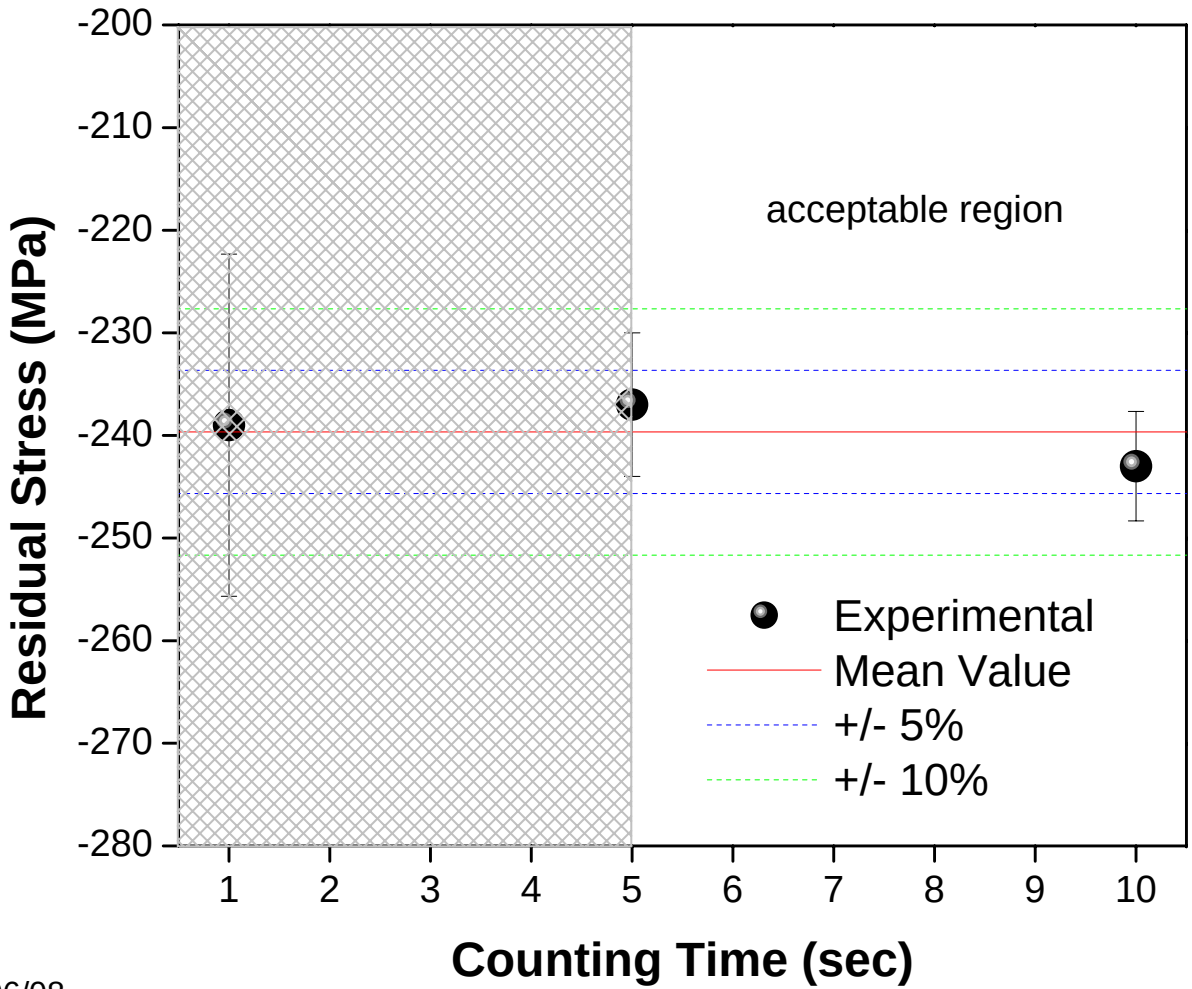
***X-RAY ENERGY (ABSORPTION EFFECTS)***

***OPERATOR REPRODUCIBILITY***

***INSTRUMENTAL MISALIGNMENTS (SERIOUS SOURCE OF ERROR)***

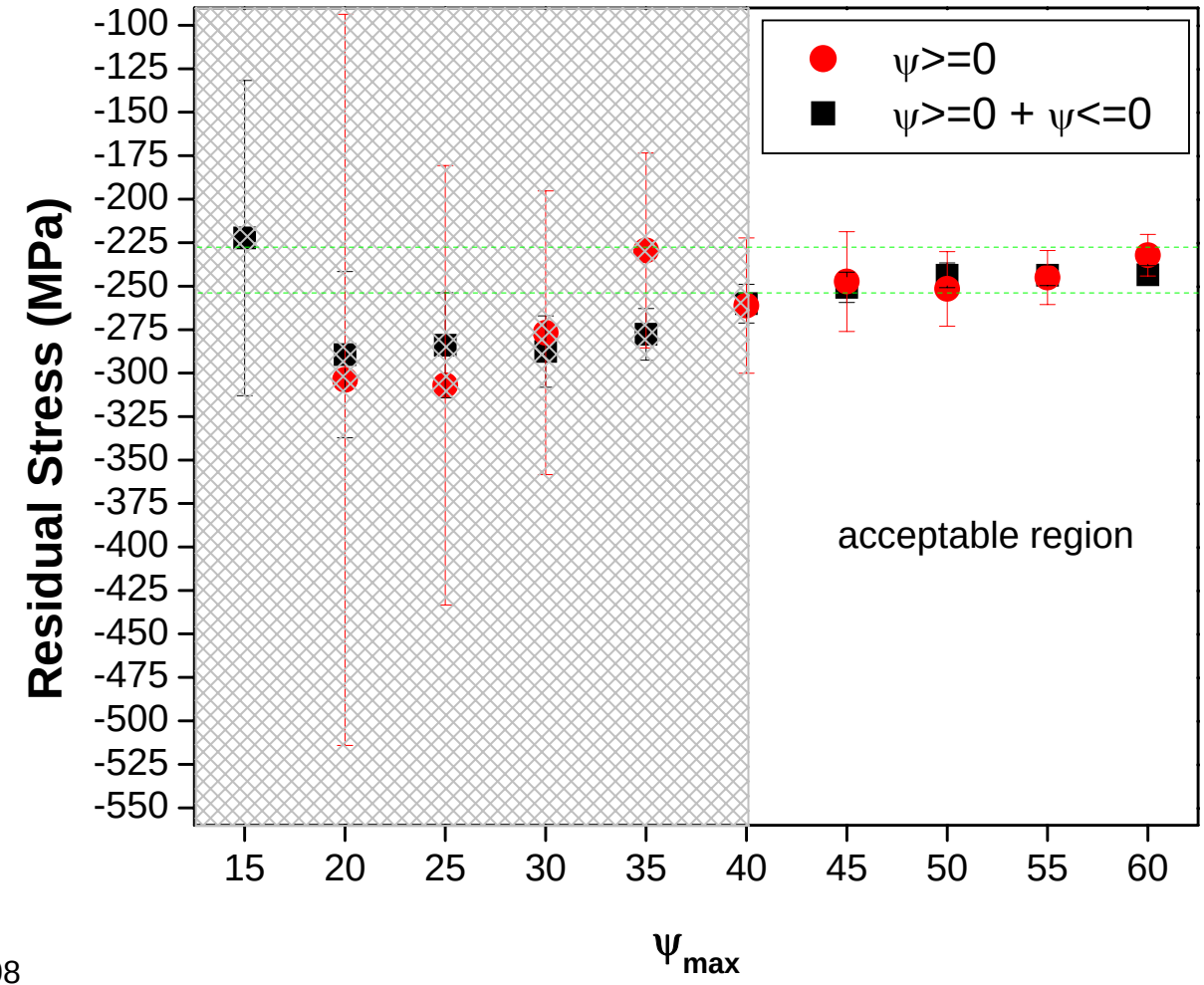
# *Experimental conditions*

## Effect of Counting Time



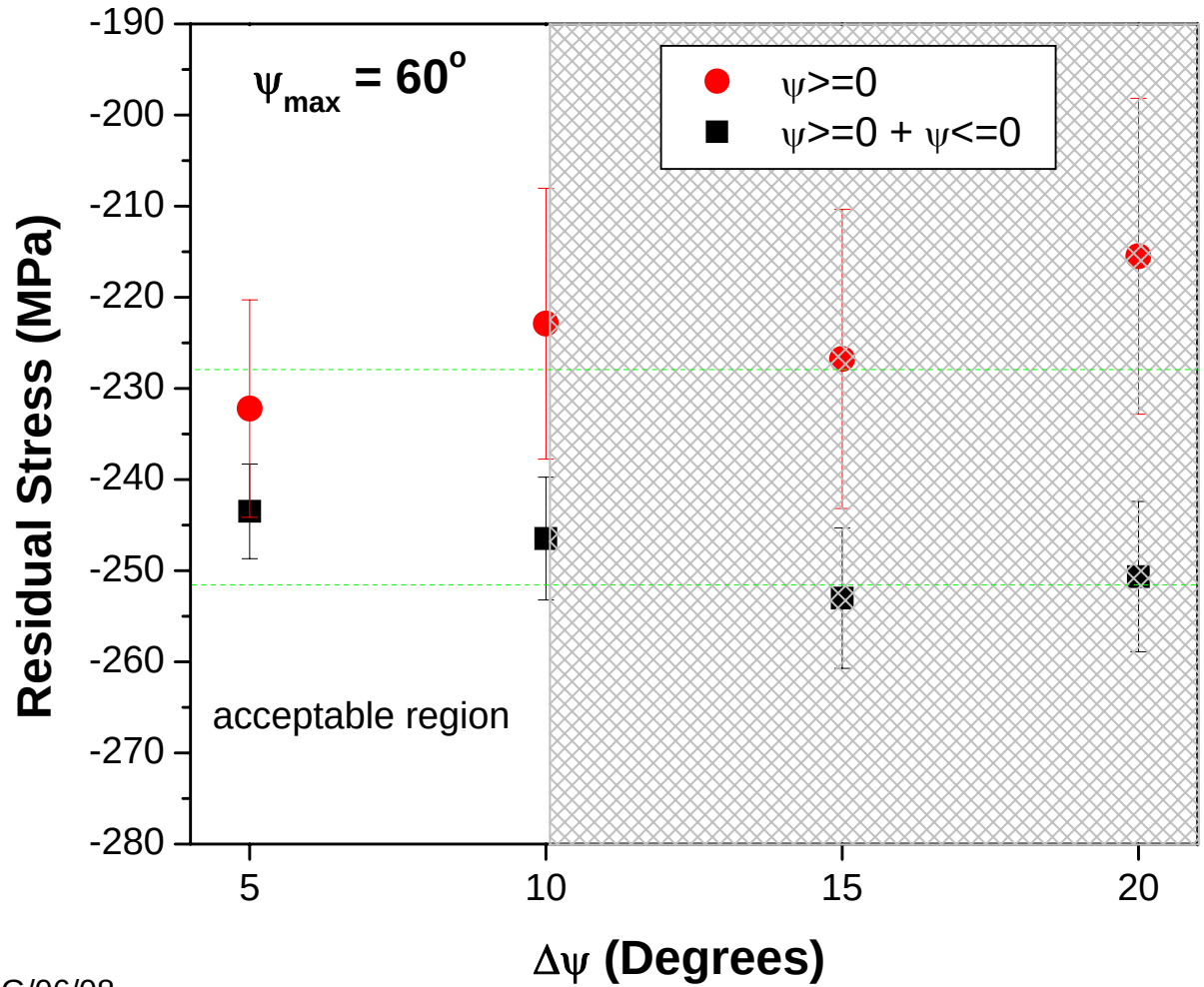
# Experimental conditions

## The Choice of PSI $_{MAX}$



# Experimental conditions

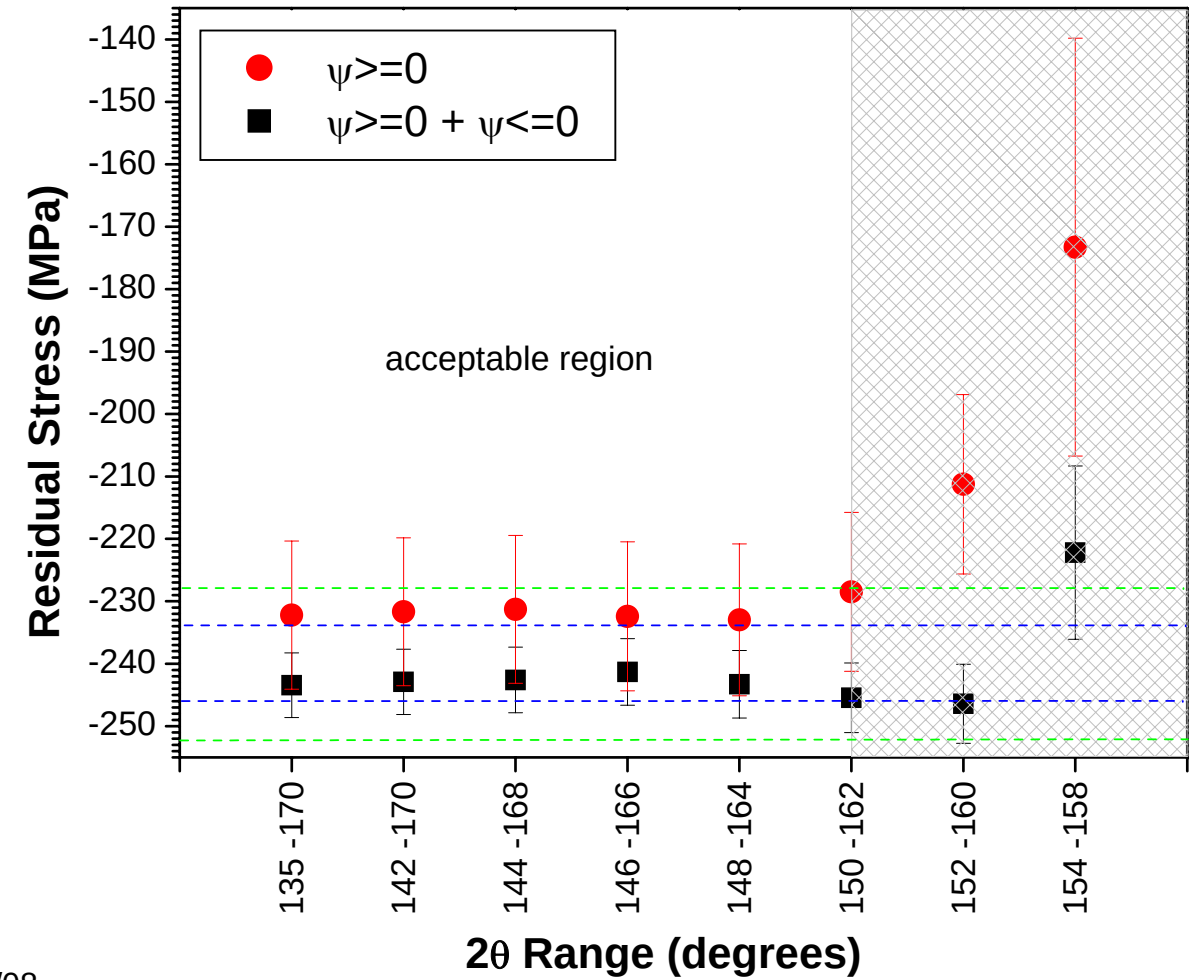
## Number of PSI Tilts



# Experimental conditions

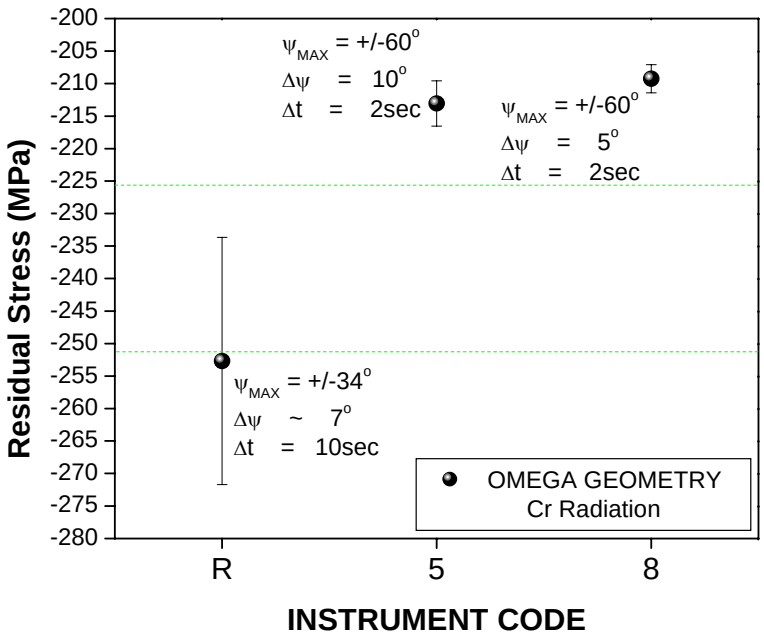
***Cr: Fe (211), 2Theta ≅ 156⁰***

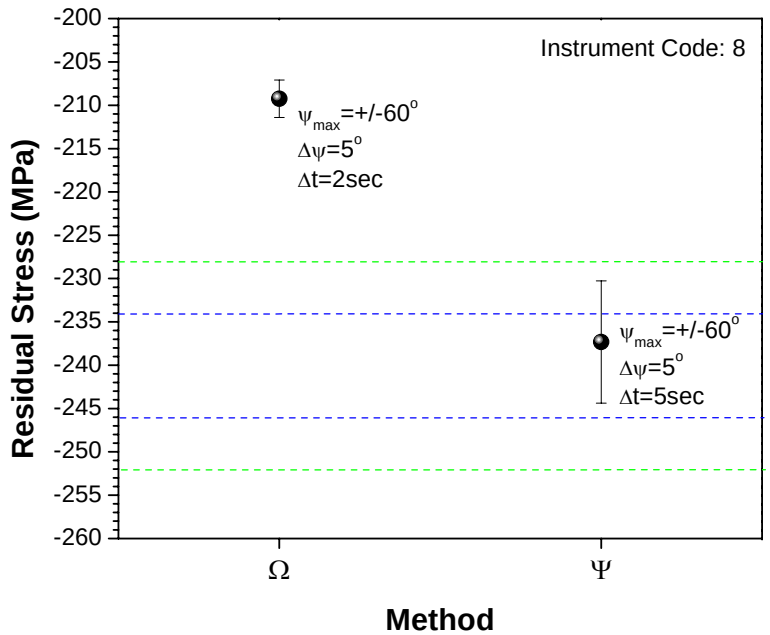
***2Theta Ranges***



# Experimental conditions

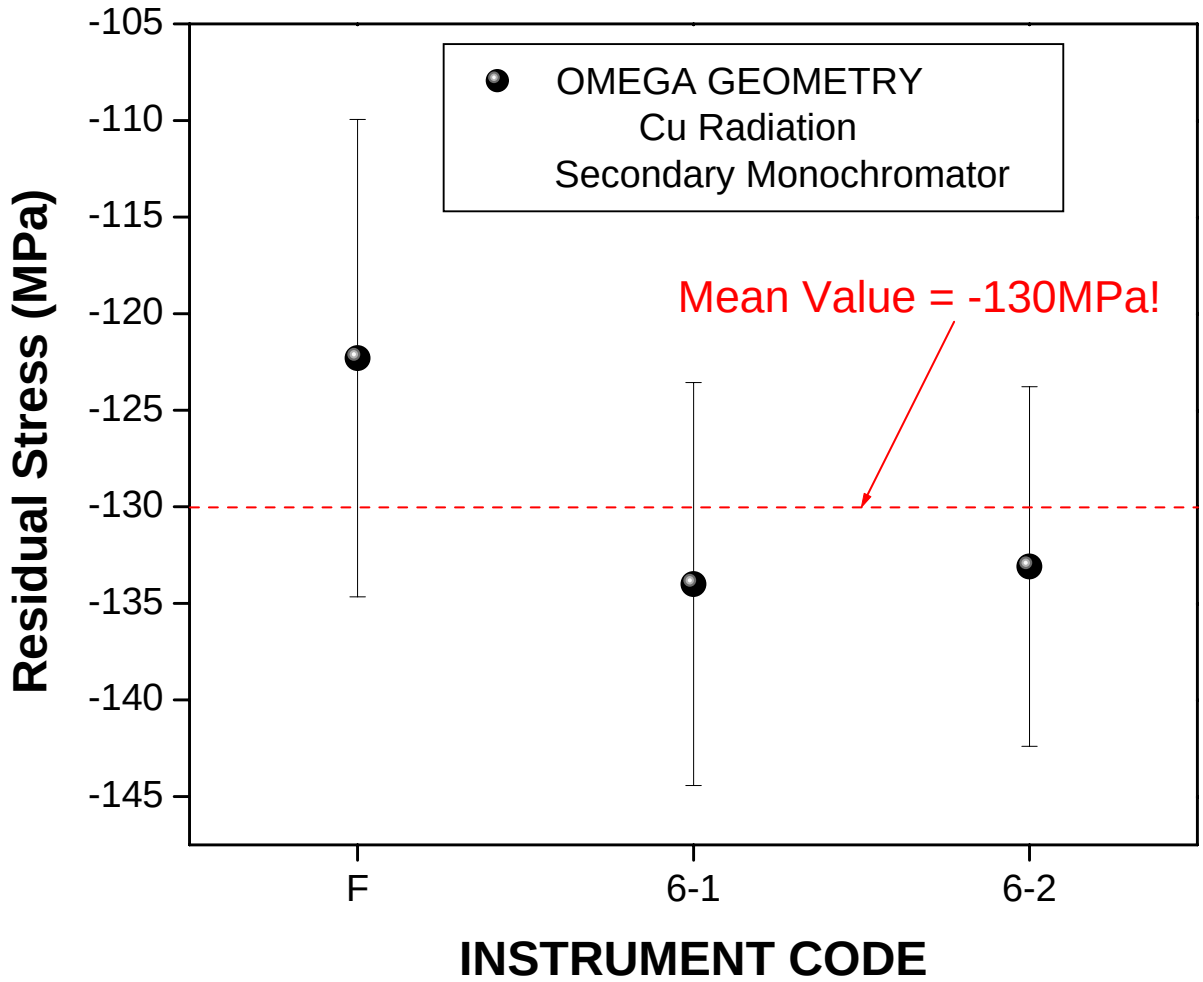
## Geometries



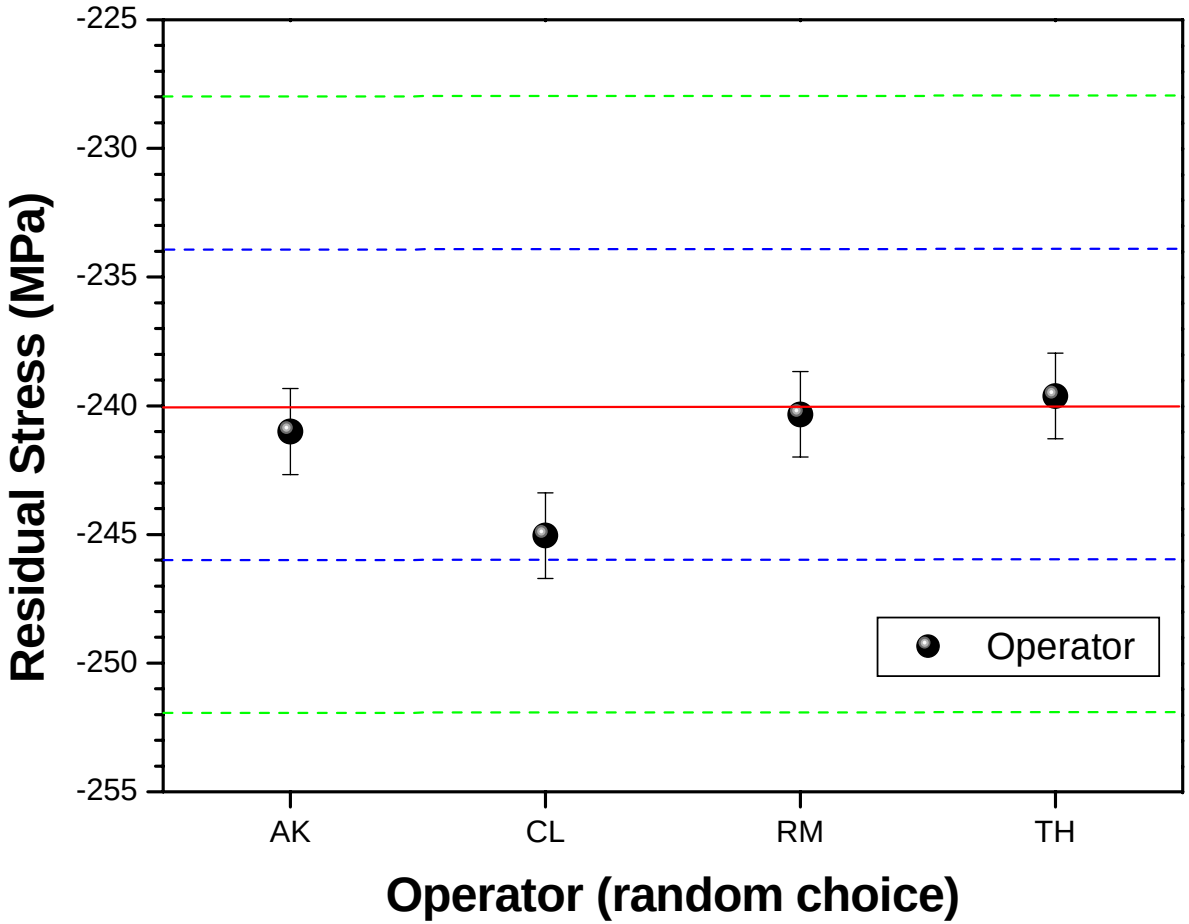
# *Experimental conditions*

***Cu Radiation***



# Experimental conditions

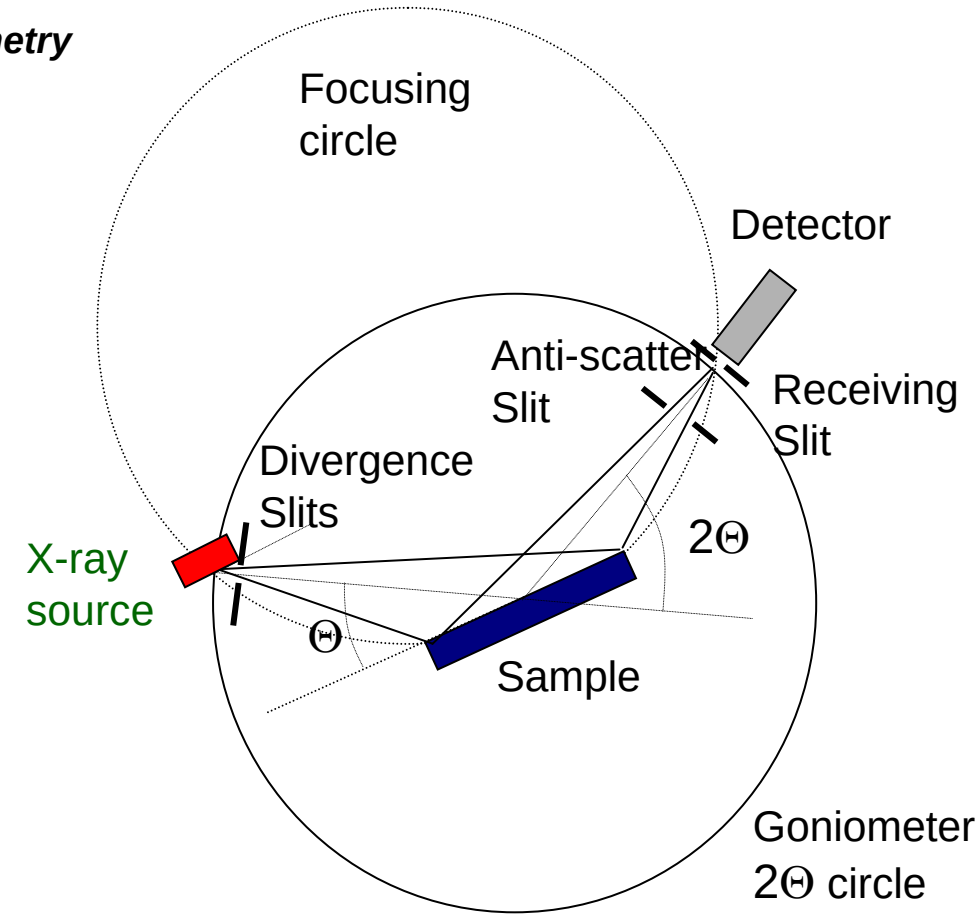
***Operator***



# *Experimental conditions*

## *Instrument Misalignments*

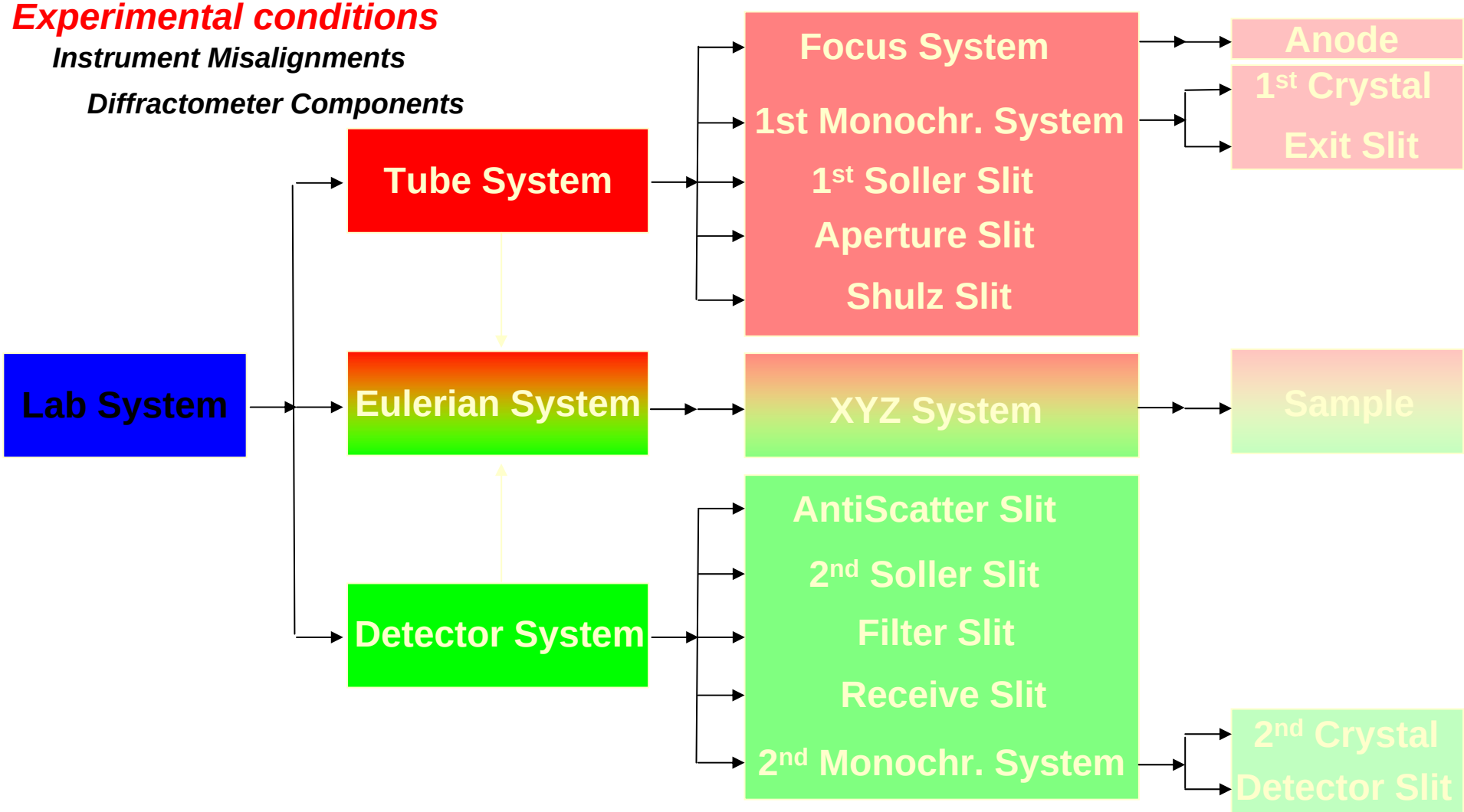
### *Diffractometer Geometry*



# *Experimental conditions*

***Instrument Misalignments***

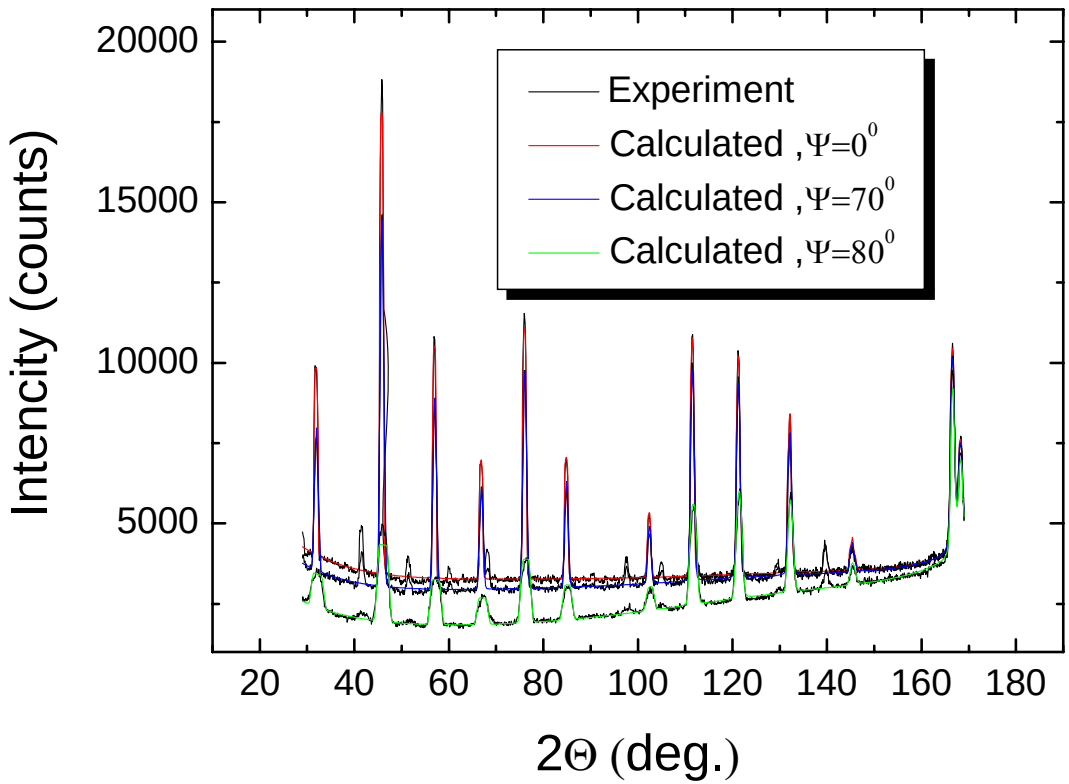
***Diffractometer Components***



# Experimental conditions

## Instrument Misalignments

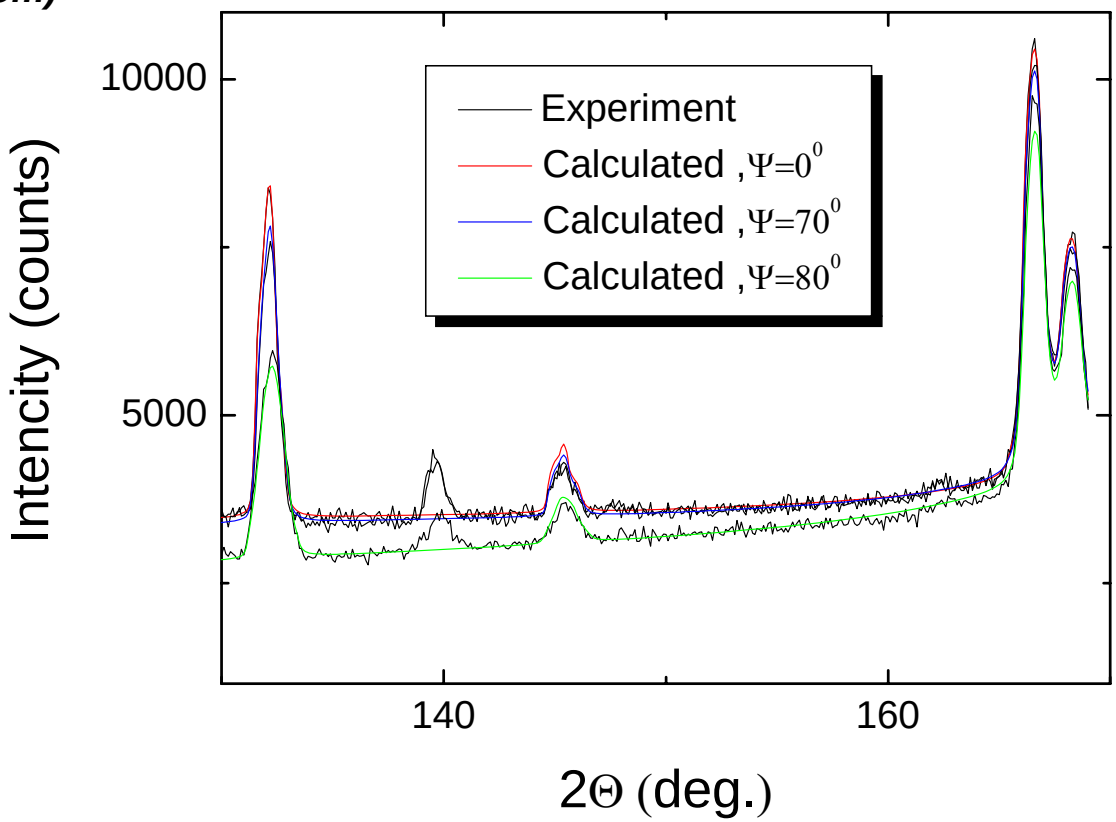
### $LaB_6$ Case



# *Experimental conditions*

## *Instrument Misalignments*

### *$LaB_6$ Case (zoom)*



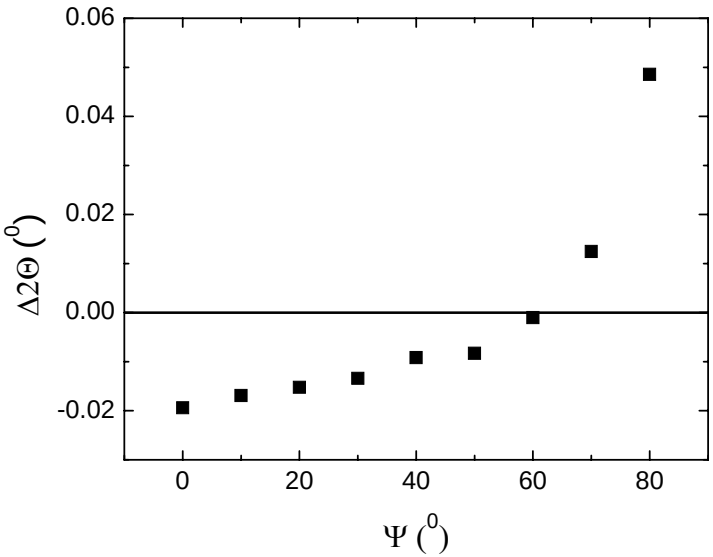

**Zero peaks shift means ideal alignment**

# *Experimental conditions*

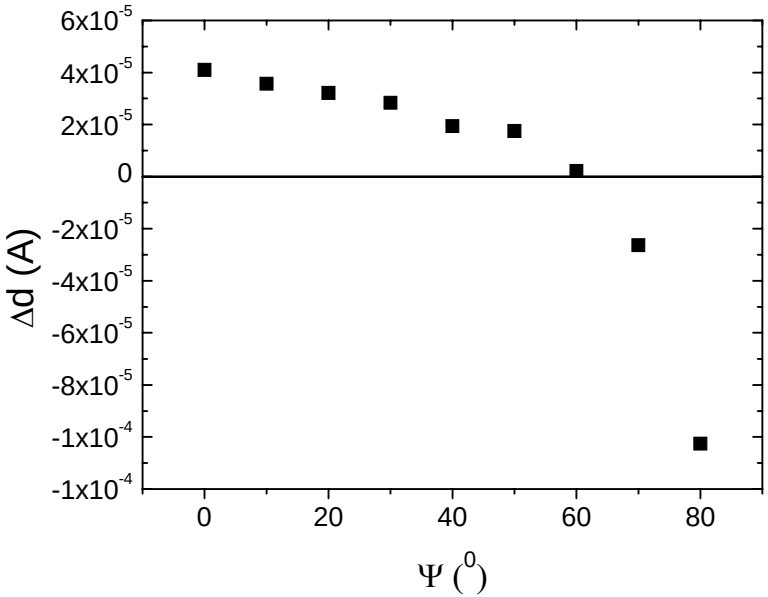
***Instrument Misalignments***

***$LaB_6$ sample displacement***

## Hypothetical 2Θ shift Fe (211)




## Hypothetical d-spacing shift Fe (211)




***Criterion for well aligned diffractometer: $|\Delta d_{max}|$ less than ~ $10^{-4}$A***

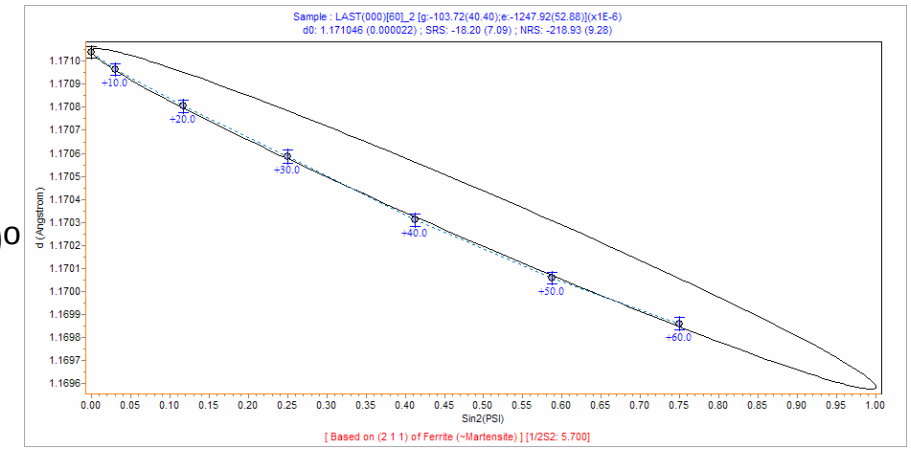
## *Influence on Final Results (example)*

PSI>=0

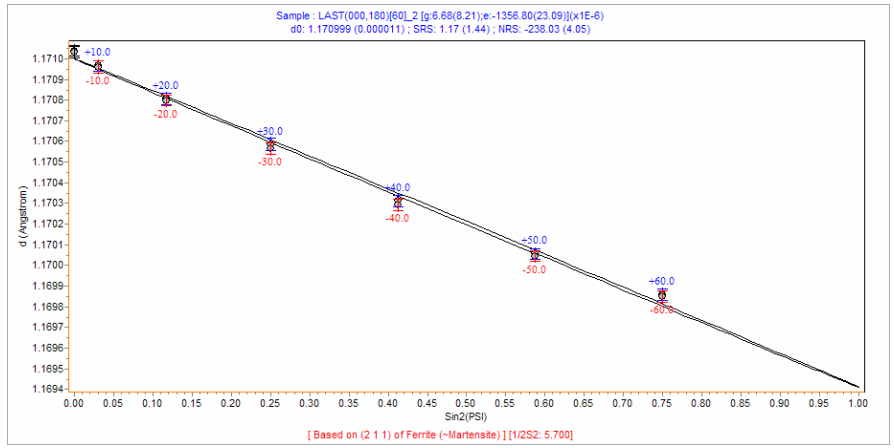
PSI<=0 + PSI>=0

60º

-219

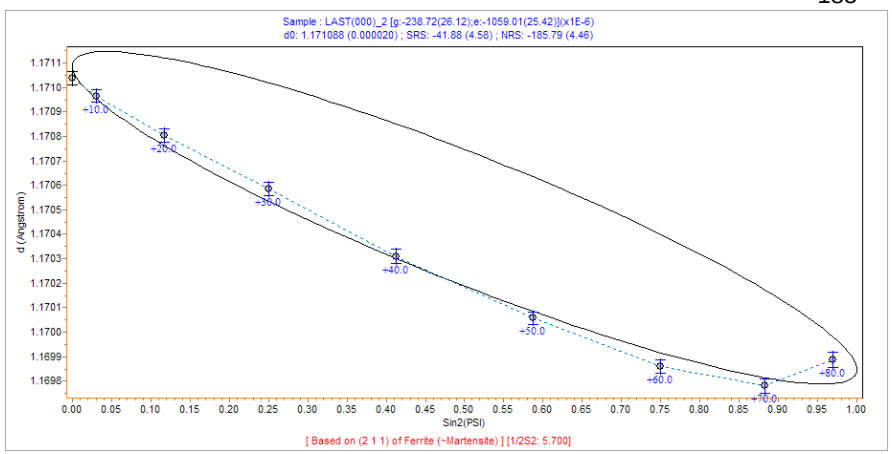



-238

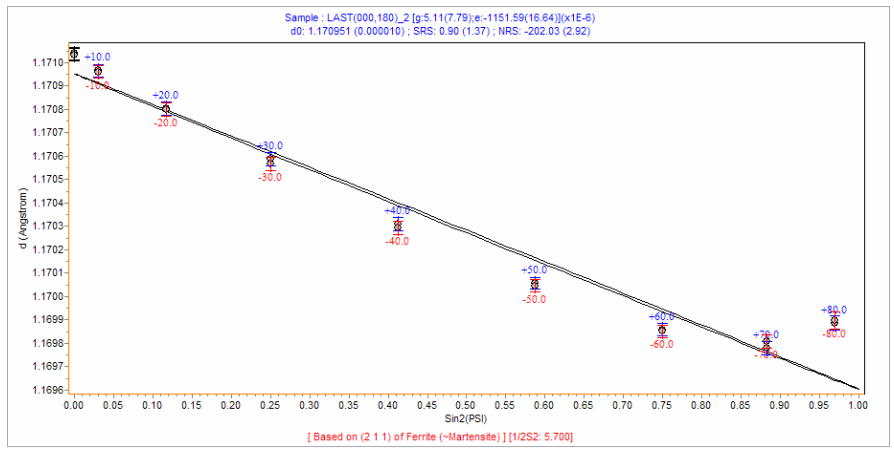



80º

-185




-202



## Conclusions

1.The selected material is suitable for national round robin (homogeneity, surface state).

2.Scattering of the results in the acceptable range can be expected in PSI geometry for wide range of experimental conditions.

3.Comparison of the data processing method with the method based on fundamental parameter approach to peak fitting (TOPAS) showed small differences in residual stress values.