Friedel-pair based indexing method for 3DXRD

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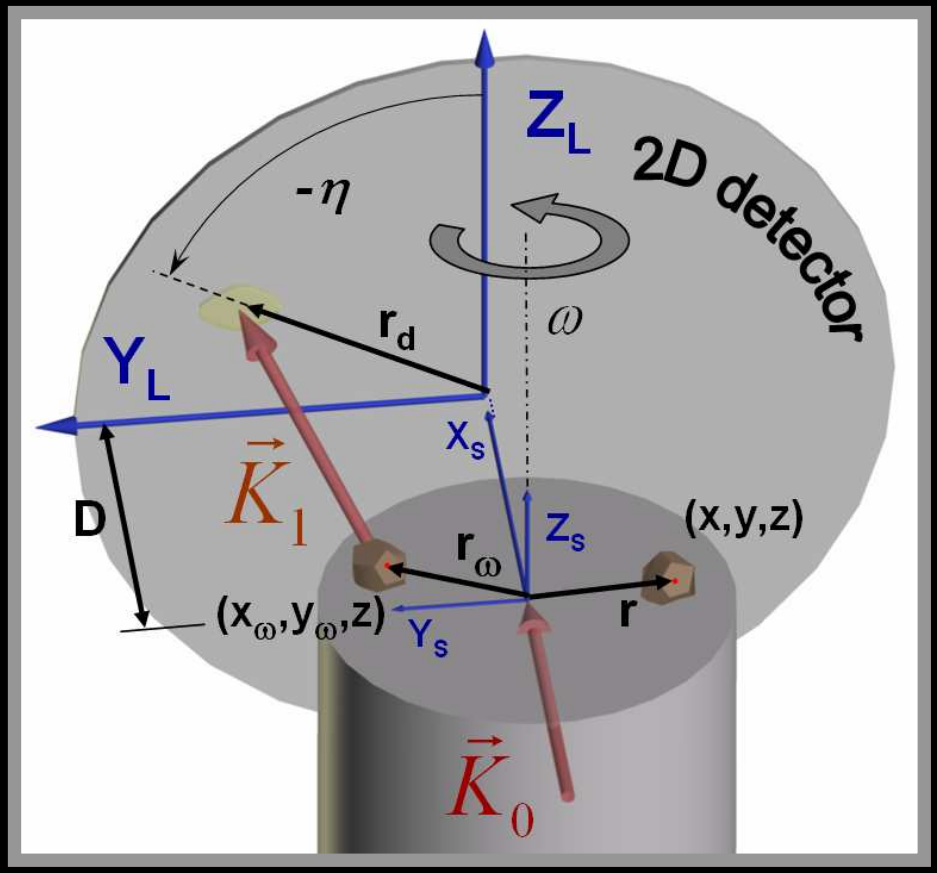
Motivation

- Investigation of grain dynamics by conventional methods like TEM and EBSD is not possible due to their destructive nature or is limited to grains at the free surface
- Development of an accurate indexing method for the orientation, position and strain of single grains in a polycrystalline sample
- Nondestructive **3DXRD** is applied to study orientation changes of bulk grains during tensile straining of a copper sample

Experimental setup



General equation predicting spot position of single grains

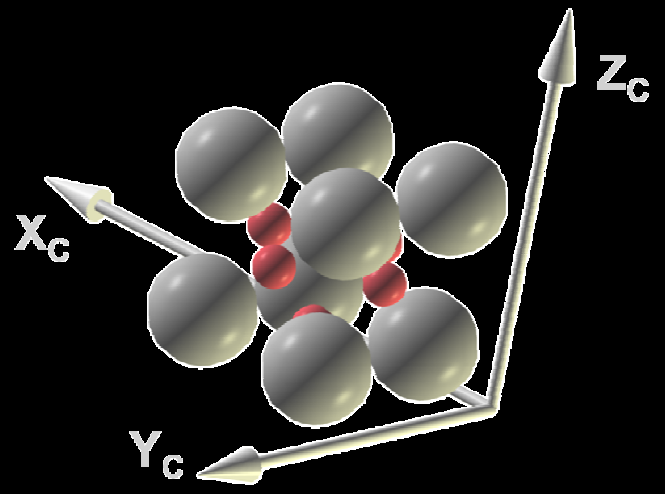
$$\mathbf{r}_d = \mathbf{r}_\omega + \mathbf{e}_1 t - D\mathbf{e}_0$$

Diffractometer equations

$$\begin{pmatrix} D \\ u \\ v \end{pmatrix} = \begin{pmatrix} x\cos\omega - y\sin\omega \\ x\sin\omega + y\cos\omega \\ z \end{pmatrix} + \left[\begin{pmatrix} 1 \\ 0 \\ 0 \end{pmatrix} + 2\sin\theta\, R \begin{pmatrix} \gamma_h \\ \gamma_k \\ \gamma_l \end{pmatrix} \right] t$$

where:

$$R = \Omega_z S U^{-1}$$



For a given crystal

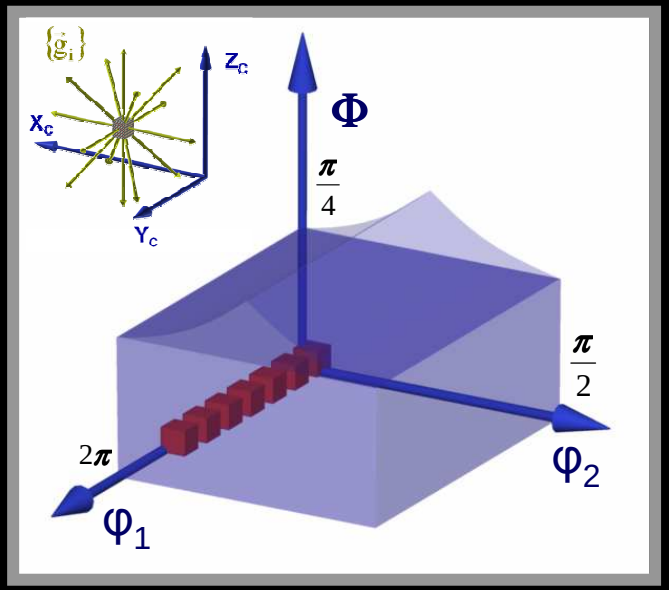
$\gamma_h \gamma_k \gamma_l$ – lattice plane normal in CCS

Ω_z – sample rotation matrix in LCS

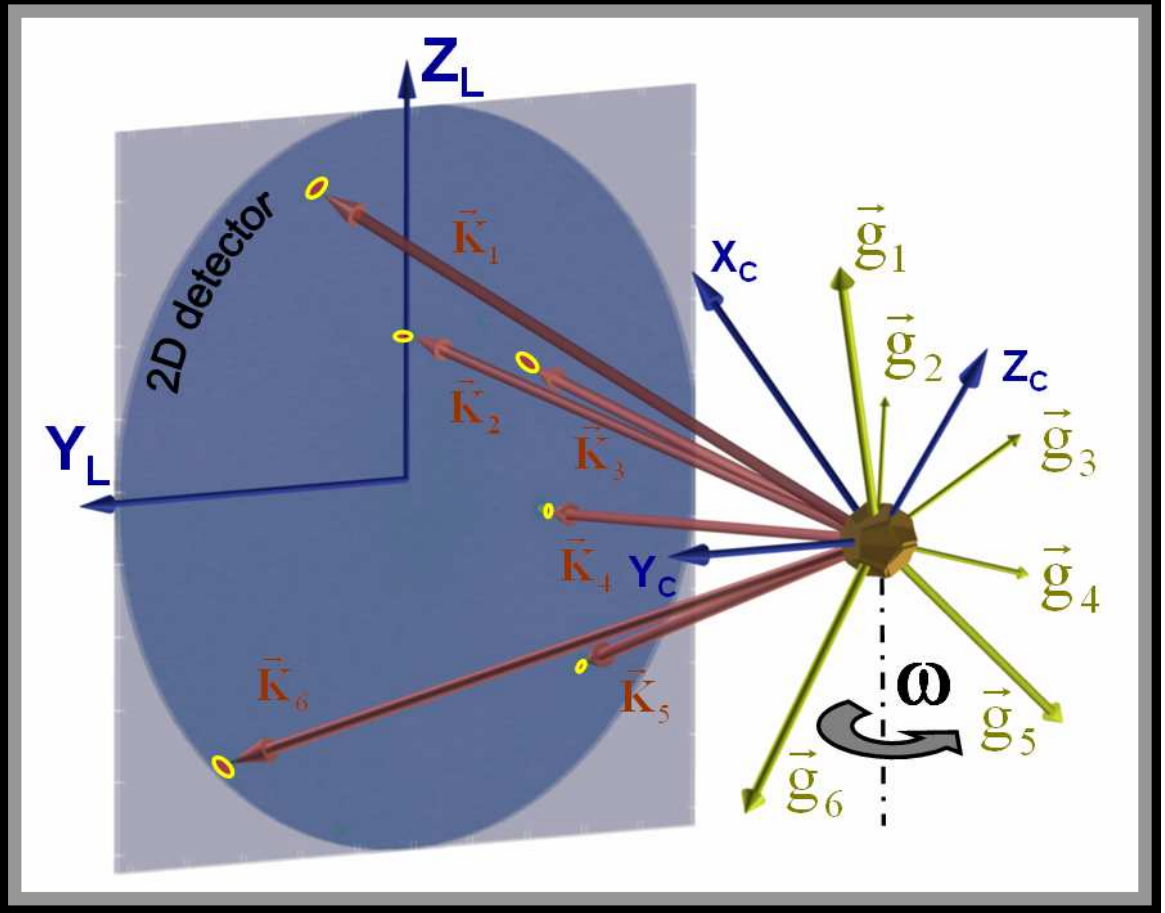
S – relates the SCS to the LCS

U – orientation matrix of the grain in SCS

Existing Indexing Method



$$(\varphi_1, \Phi, \varphi_2) \Rightarrow U_1$$



$$\mathbf{g}_{S\_sim} = \mathbf{U}_1^{-1}\mathbf{g}^c \quad \Leftrightarrow \quad \mathbf{g}_{S\_exp} = \mathbf{S}^{-1}\boldsymbol{\Omega}_z^{-1}\mathbf{g}_\omega$$

Indexing Methods

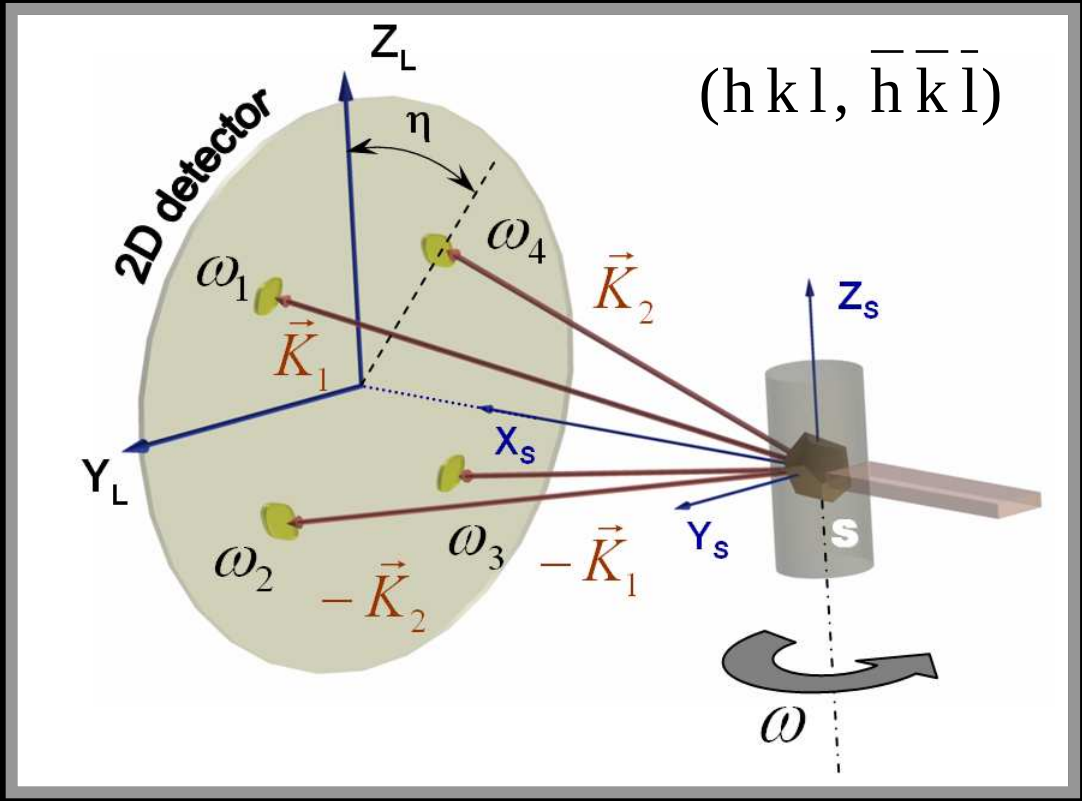
Old method features :

- the whole FZ of the orientation space has to be searched through to find the right U
- small ω range is sufficient to obtain U

New method features:

- candidate matrix U_{h00} is obtained from experiment
- evaluation is based on Friedel pairs

New Method, Based on Friedel Pairs



Advantages:

- Symmetry properties enable a clear separation of grain orientation effects from grain position
- Reduced number of unknowns which have to be simultaneously fitted

Disadvantages:

- Relatively large ω interval has to be measured

$\Delta\omega_{min} = 90°\text{-}2\,\theta$

$$\omega_{2,3} \cong \omega_{1,4} \pm 180° \;; \eta_{3,4} \cong \eta_{1,2} \pm 180°$$

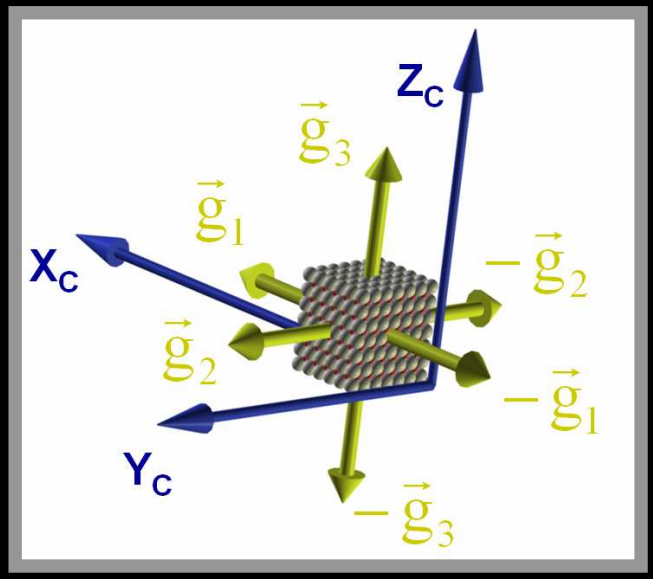
Candidate U

$$\mathbf{U}_{h00} = \begin{bmatrix} e_{g_{h00}} \\ e_{g_{0h0}} \\ e_{g_{h00}} \times e_{g_{0h0}} \end{bmatrix}$$

$$\mathbf{g}_{S\_exp} \Leftrightarrow \mathbf{S}^{-1}\boldsymbol{\Omega}_z^{-1}\mathbf{U}_{h00}\mathbf{g}_{hkl}$$

$$\left(\mathbf{g}_{S\_exp}\right)_i = \mathbf{U}*^{-1}\,\mathbf{g}^{c}$$

Completeness criteria



- at least two perpendicular *h00* type reflections per grain are required
- Direct approach avoids scanning of the orientation space (fast)

Grain orientation estimation

- Using Friedel pairs the orientation of the grain can be expressed as function of grain diffraction vectors g^c

$$\frac{v - v_F}{u + u_F} = \frac{R_{31}\gamma_h + R_{32}\gamma_k + R_{33}\gamma_l}{R_{21}\gamma_h + R_{22}\gamma_k + R_{23}\gamma_l}$$

$$\chi^2(\varphi_1, \Phi, \varphi_2) = \sum_{i=1}^{N} \left(\frac{v - v_F}{u + u_F} - \frac{R_{31}\gamma_h + R_{32}\gamma_k + R_{33}\gamma_l}{R_{21}\gamma_h + R_{22}\gamma_k + R_{23}\gamma_l} \right)_i^2$$

Grain position estimation

- Using Friedel pairs the CM position of the grain can be expressed as function of detector coordinates (*u, v*)

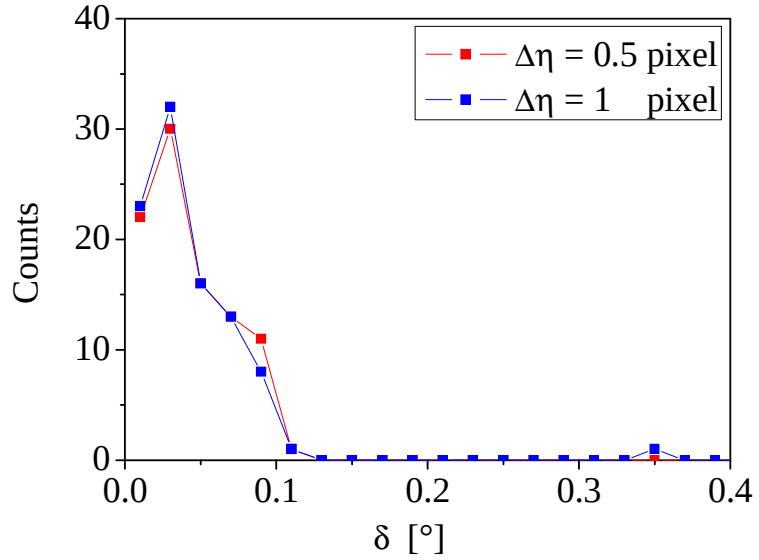
$$u - u_F = x\left(2\sin\omega - \frac{u+u_F}{D}\cos\omega\right) + y\left(2\cos\omega + \frac{u+u_F}{D}\sin\omega\right)$$

$$v + v_F = 2z - x\frac{v-v_F}{D}\cos\omega + y\frac{v-v_F}{D}\sin\omega$$

$$\chi^2(x,y,z) = \sum_{i=1}^{N}\left(u - u_F - f(x,y)\right)_i^2 + \sum_{i=1}^{N}\left(v + v_F - f(x,y,z)\right)_i^2$$

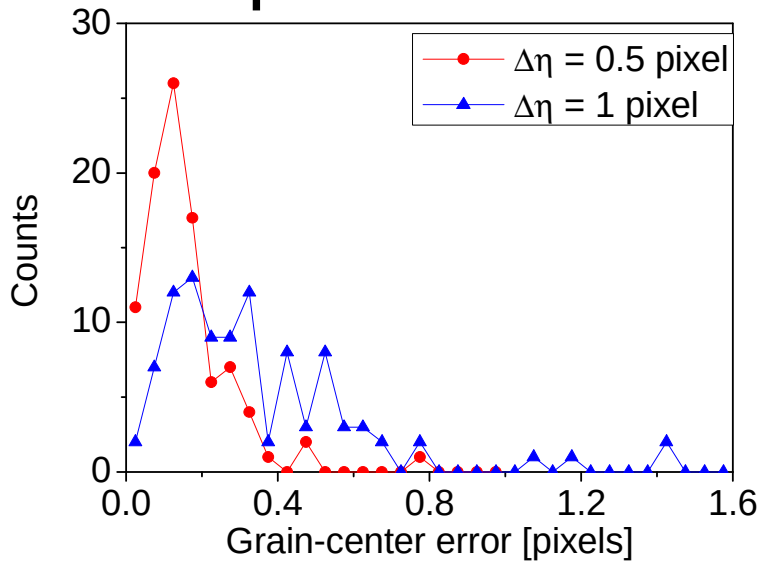
Results - Simulations

Grain orientation estimation



$$\delta = f(\Delta\omega, \Delta\eta)$$

Grain position estimation

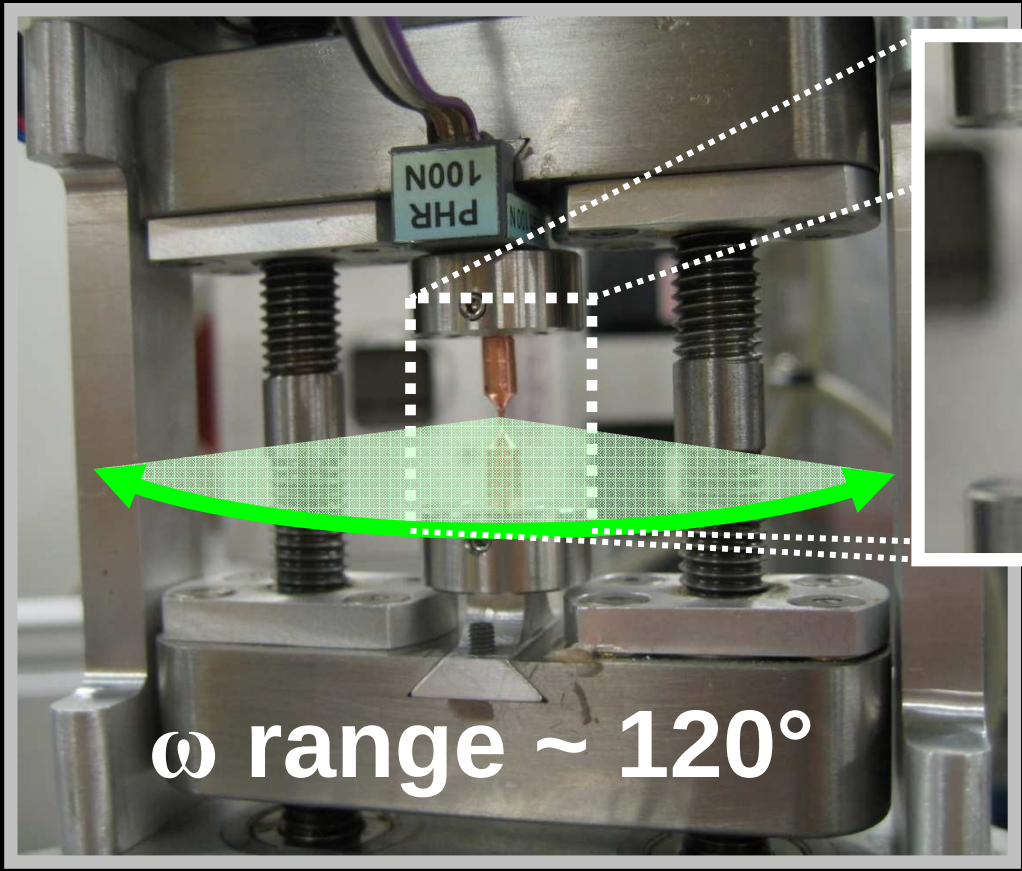


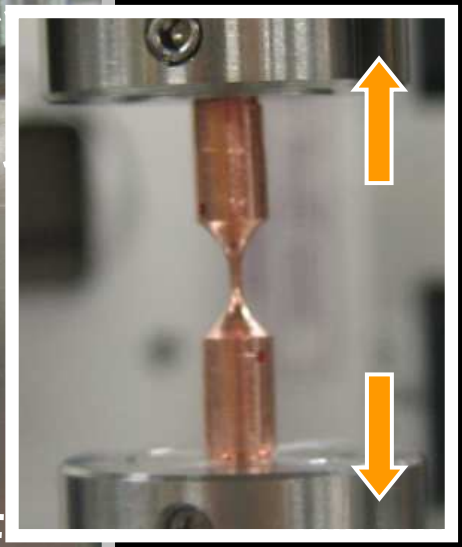
Cu, 70 keV
sample diameter: 600µm
grain number: 100
pixel size: 100 µm ❯ MAR345 (HASYLAB)

$\omega \in \langle 0;120 \rangle \cup \langle 180;300 \rangle$ $\Delta\omega = 0.5°$

EXPERIMENTAL

Application to in-situ investigations





Applied strains:

0%

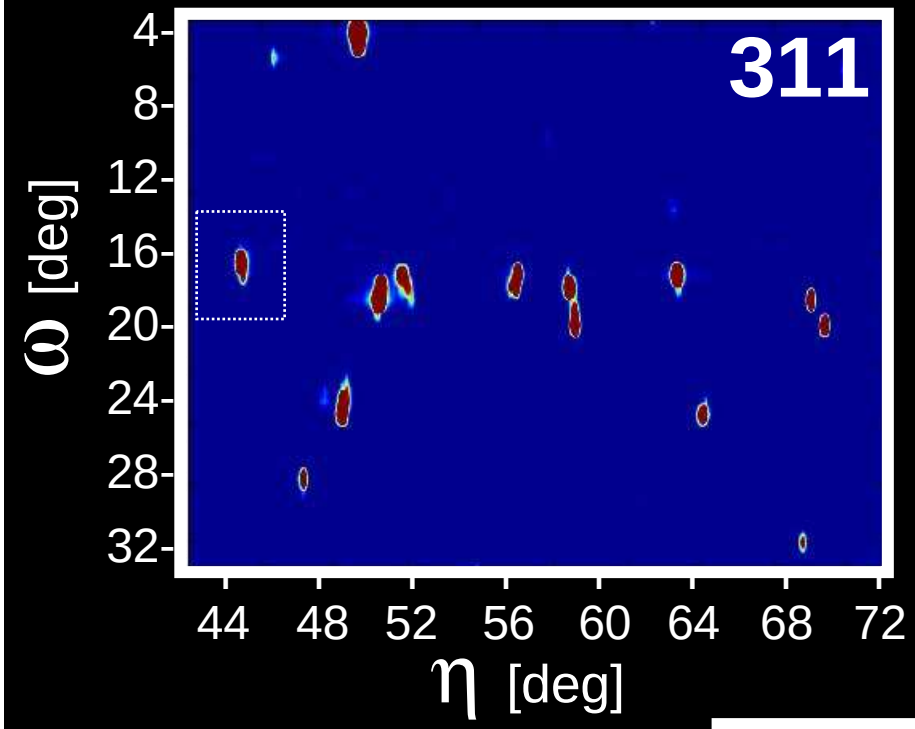
4%

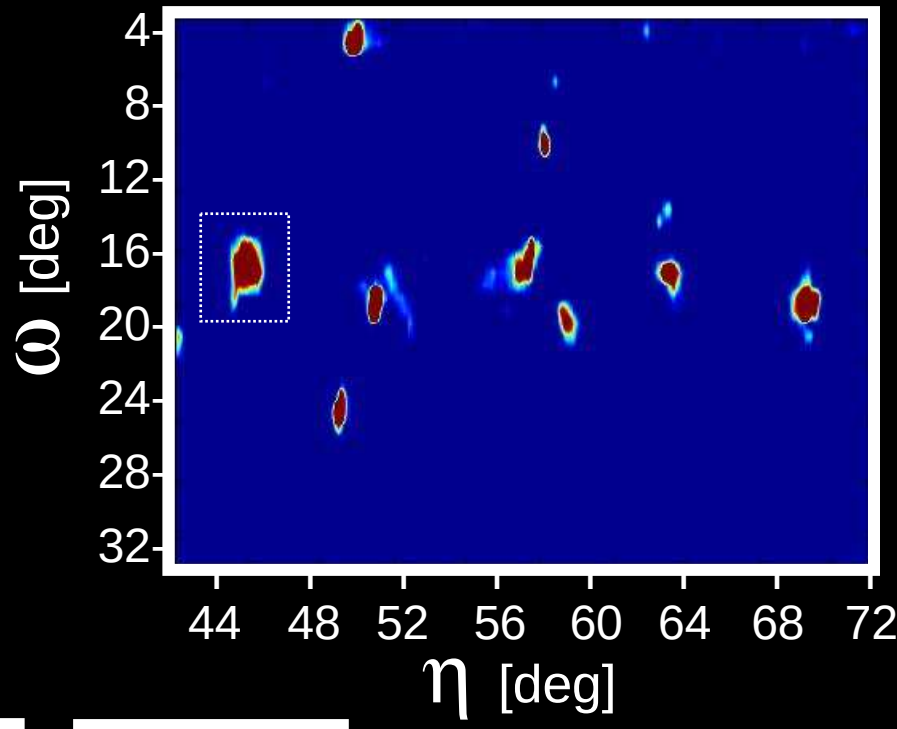
- Average grain size **~ 50 µm**
- Sample diameter **600 µm**
- Beam cross section **width 1 mm, height 100 µm**

Copper sample mounted in the rig

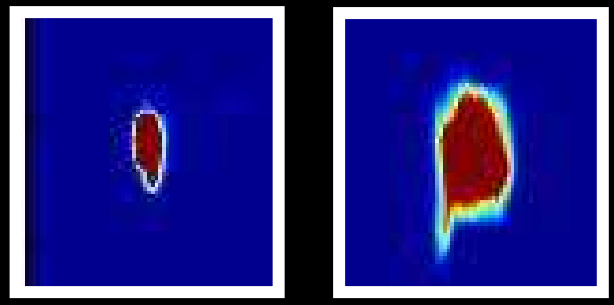
- $\omega \in \langle 0;120 \rangle \cup \langle 180;300 \rangle$ **Δω = 0,75°**

Indexing of Experimental Data





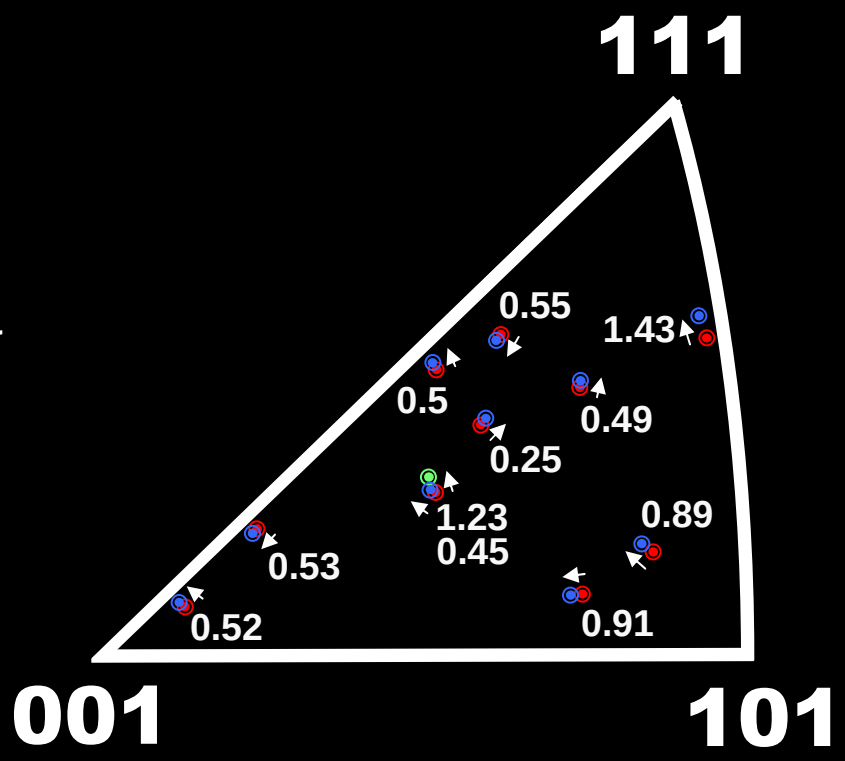
ε = 0%



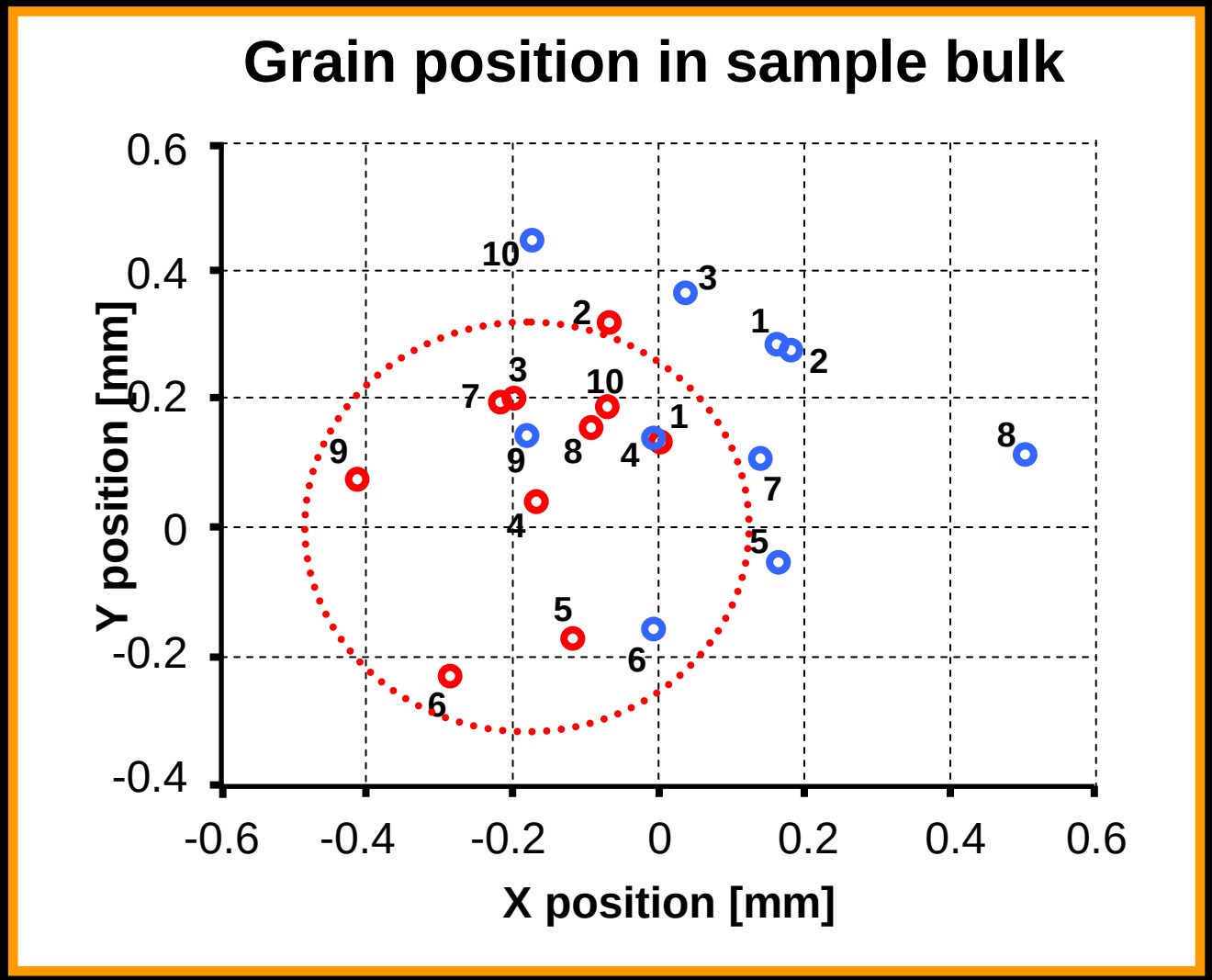
ε = 4 %

Results

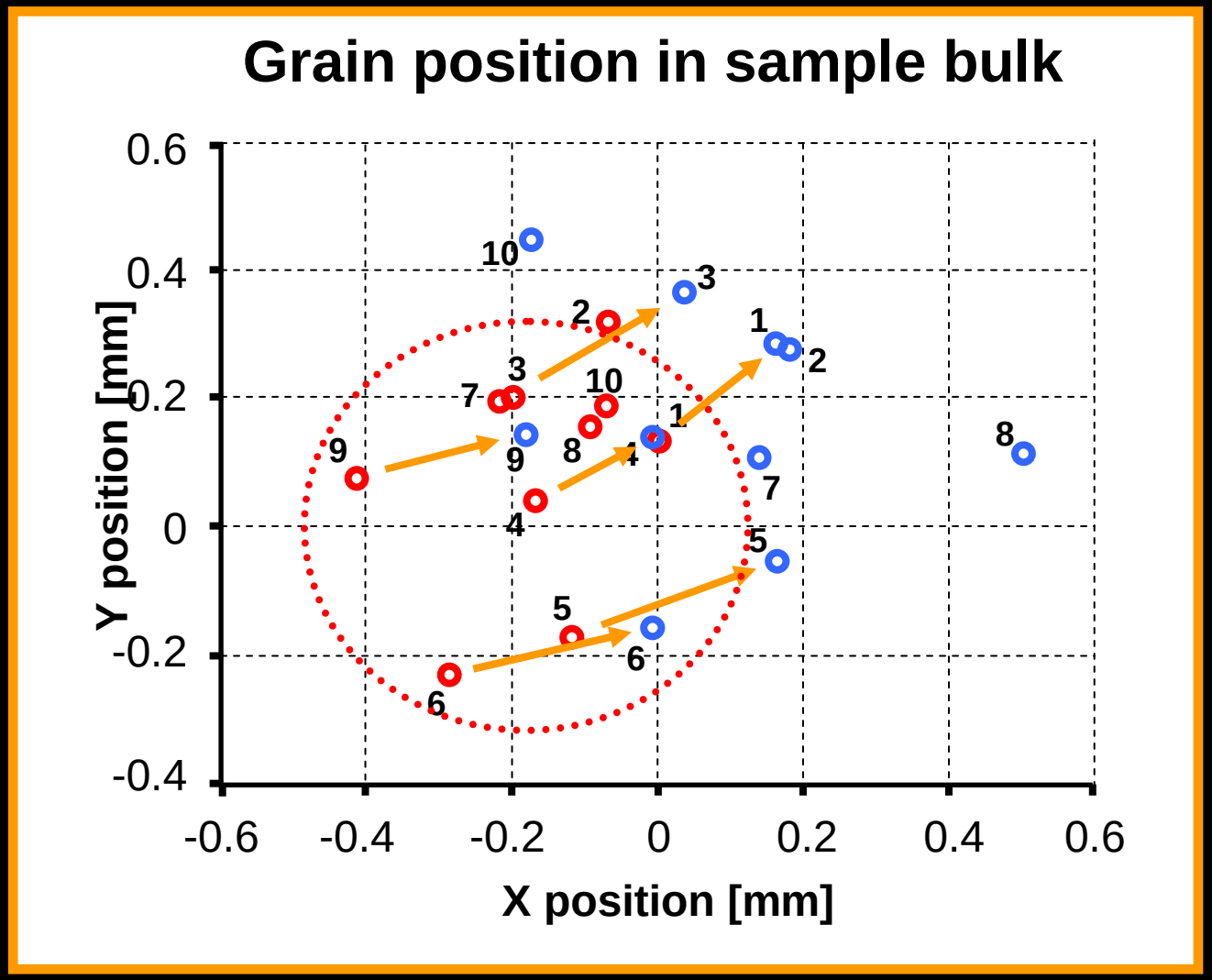
- Inverse pole figure of the tensile axis showing the orientation change of 10 different grains during plastic deformation of a Cu sample. The blue spots mark the final orientation after a true strain 4%



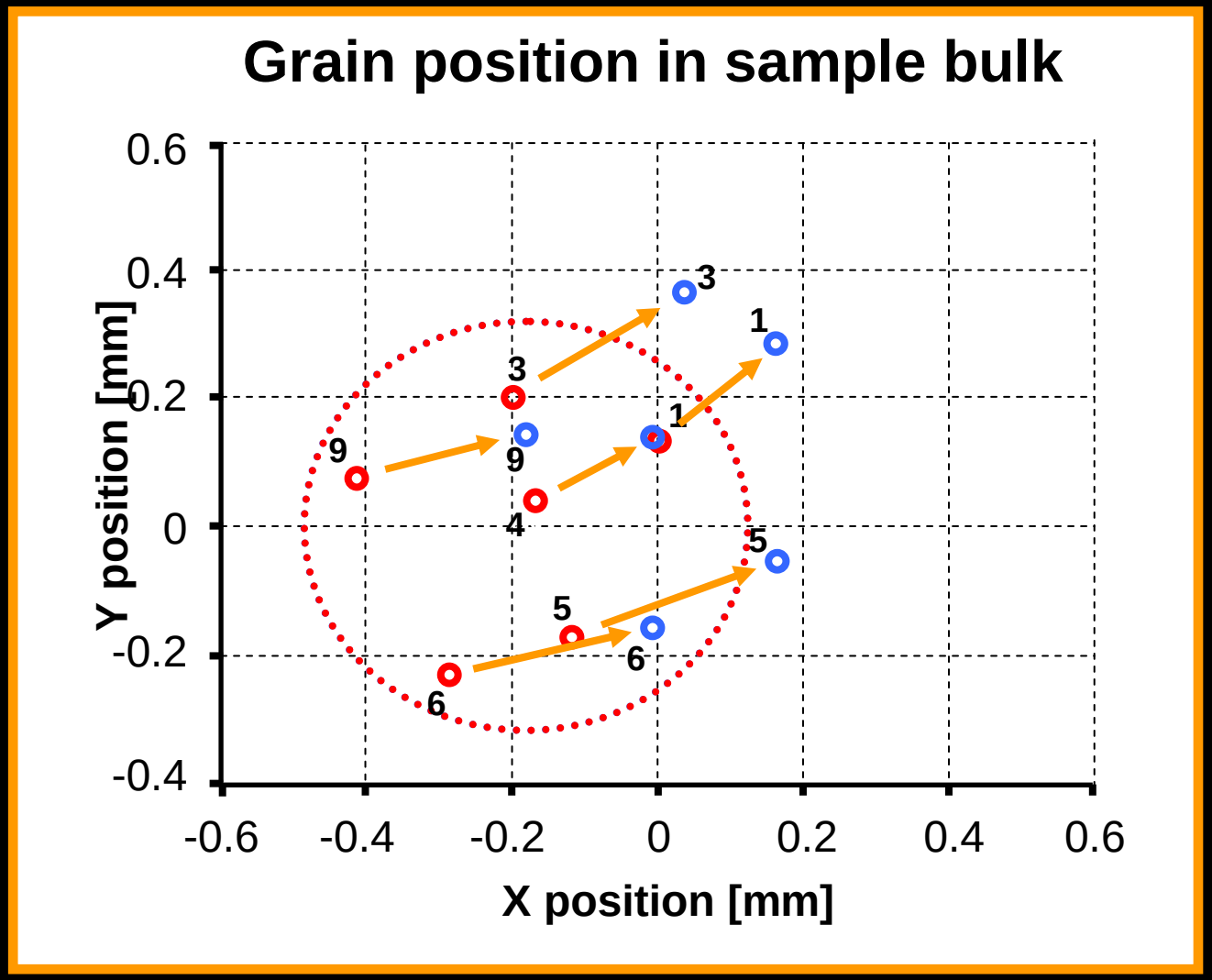
Results


Grain position in sample bulk
Y position [mm]
X position [mm]
0.6
0.4
0.2
0
-0.2
-0.4
-0.6
-0.4
-0.2
0
0.2
0.4
0.6

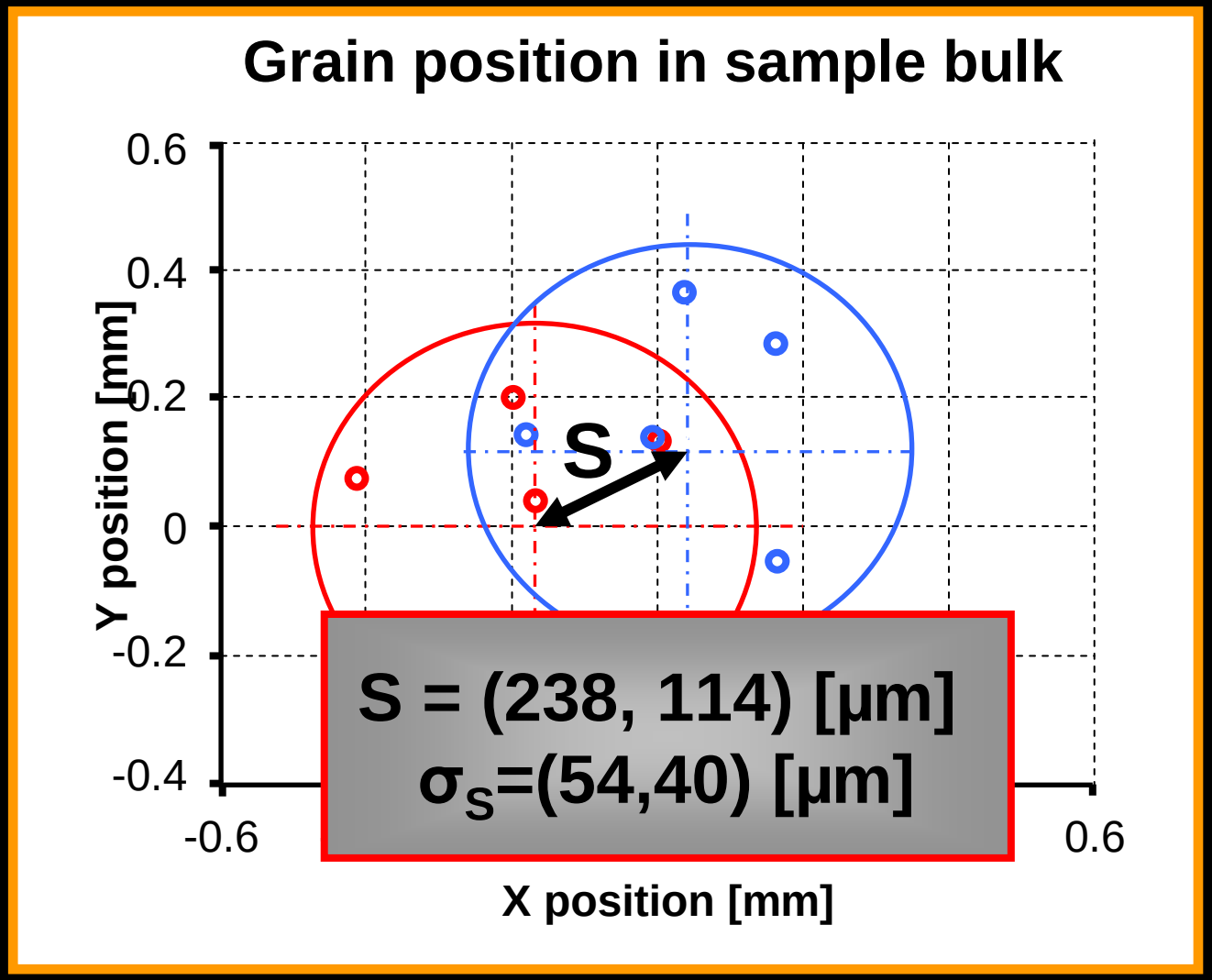
Results


Grain position in sample bulk
Y position [mm]
X position [mm]
0.6
0.4
0.2
0
-0.2
-0.4
-0.6
-0.4
-0.2
0
0.2
0.4
0.6
10
3
2
1
2
3
7
10
1
8
9
9
8
4
7
4
5
5
6
6

Results


Grain position in sample bulk
Y position [mm]
X position [mm]
0.6
0.4
0.2
0
-0.2
-0.4
-0.6
-0.4
-0.2
0
0.2
0.4
0.6
3
1
3
1
9
9
4
5
5
6
6

Results


Grain position in sample bulk
0.6
0.4
0.2
0
-0.2
-0.4
-0.6
0.6
Y position [mm]
X position [mm]
S
S = (238, 114) [µm]
σS=(54,40) [µm]

Conclusion

- Depending on the number of reflections the new indexing method predicts an accuracy for grain orientation of about 0.1° or better
- Grain positions can be evaluated with an absolute error of about 0.3 times the detector pixel size
- Orientation changes of grains during uniaxial plastic deformation (Cu) are similar to previous results obtained by Poulsen (*Acta Materialia 51 (2003) 3821*) in aluminium

THANK YOU FOR YOUR ATTENTION