Akademia Górniczo-Hutnicza
im. Stanisława Staszica w Krakowie

Laboratorium Badań Mikroskopowych WFiIS

dr hab. inż. Sebastian Wroński

Wydział Fizyki i Informatyki Stosowanej

Laboratorium Badań Mikroskopowych WFiIS

Jeol JSM 6440LV

STEMserviceSEM
Serwis aparatury firmy JEOL od 1991r.

Principal Specifications

Resolution	HV mode	3.0 nm (30kV), 8.0 nm (3kV), 15 nm (1kV)
	LV mode	4.0 nm (30kV)
Magnification		×8 to ×300,000 (at 11kV or higher)
		×5 to ×300,000 (at 10kV or lower)
Preset magnifications		5 steps, user selectable
User operation recipe		Optics, Specimen stage, Image mode,
		LV pressure[*1], Standard recipe
Image mode		Secondary electron image,
		Composition[*1], Topography[*1], Shadowed[*1]
Accelerating voltage		0.3 kV to 30 kV
Filament		Factory pre-centered filament
Electron gun		Fully automated, manual override
Condenser lens		Zoom condenser lens
Objective lens		Super conical objective lens
Objective lens apertures		3 stages, XY fine adjustable
Stigmator memory		Built in
Electrical image shift		±50μm (WD=10mm)
Auto functions		Focus, brightness, contrast, stigmator
Specimen stage		Large eucentric type,
		X: 125mm, Y: 100mm, Z: 5mm to 80mm,
		Tilt: −10° to +90°, Rotation: 360°
Motor control		5 axes computer controlled
Navigator/Reference		2 images
Specimen exchange		Through the front door
Maximum specimen		200mm diameter
LV Pressure[*1]		1 to 270 Pa

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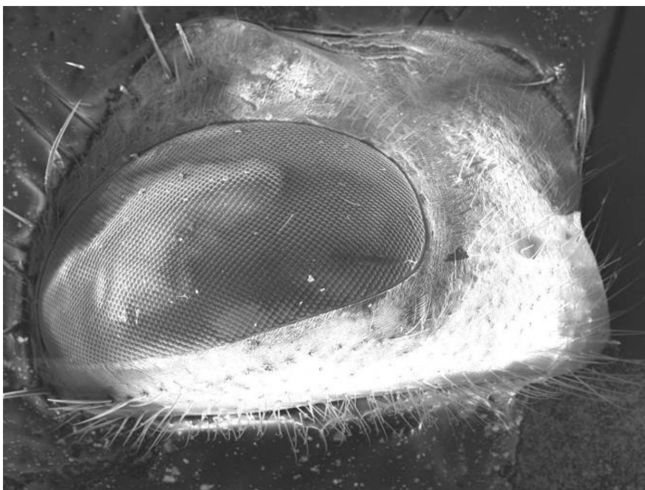
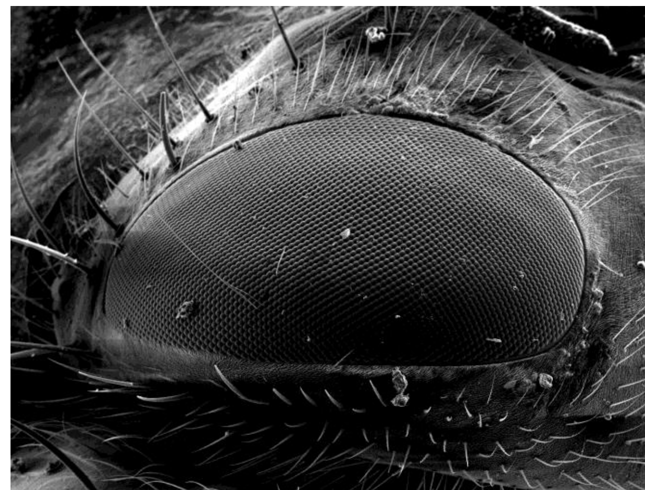
Napylarka BAL-TEC SCD050 umożliwiająca nanoszenie cienkich warstw (filmów) zarówno metalicznych, jak i węglowych na preparaty przygotowane do badań SEM i EDS.





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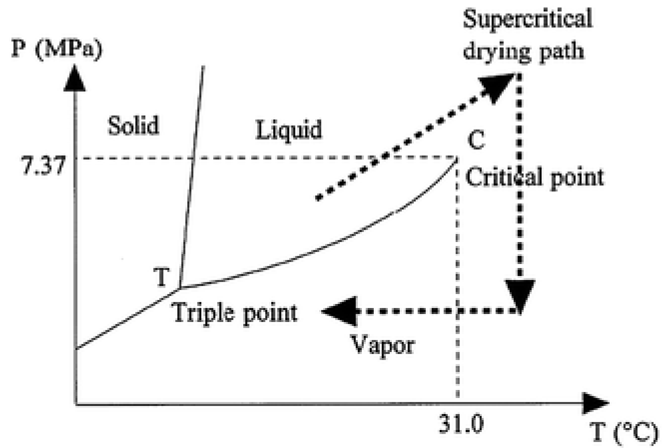
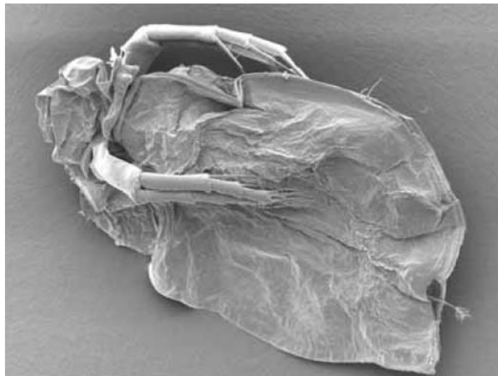
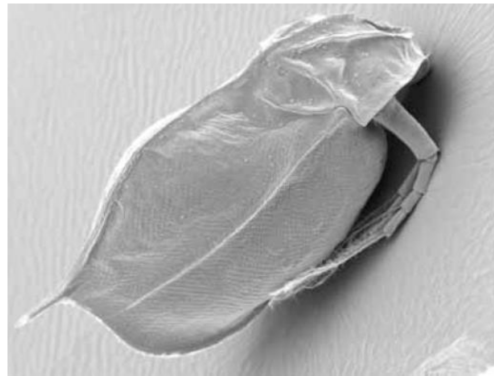
Suszarka BAL-TEC CPD030 zautomatyzowana suszarka próbek w punkcie krytycznym.

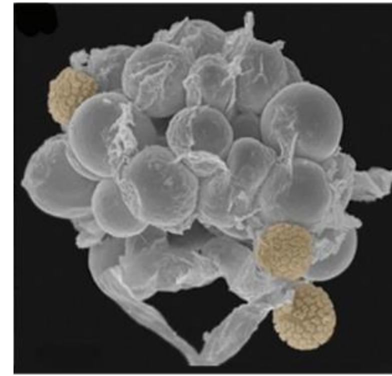


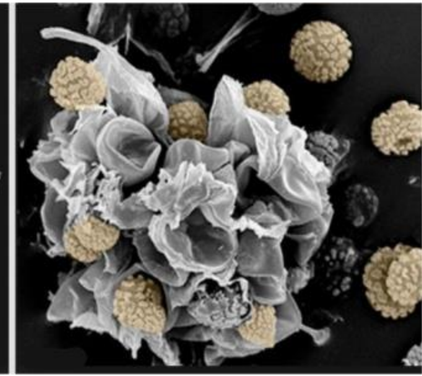


Air dried sample (Water flea)



Critical point dried sample (Water flea)





https://www.leica-microsystems.com/science-lab/brief-introduction-to-critical-point-drying/

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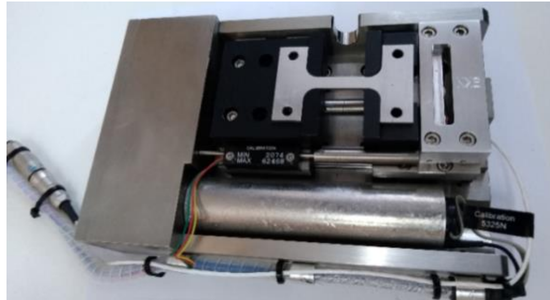
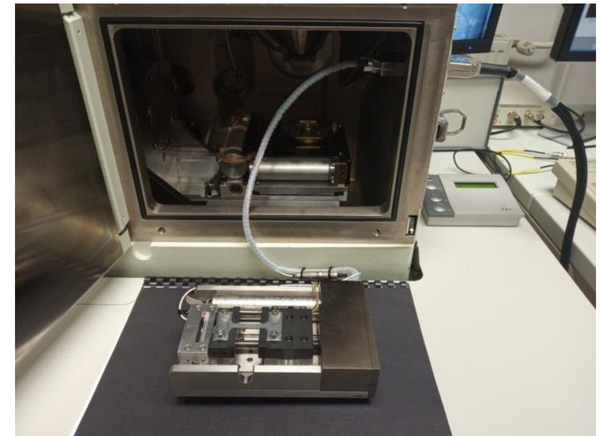
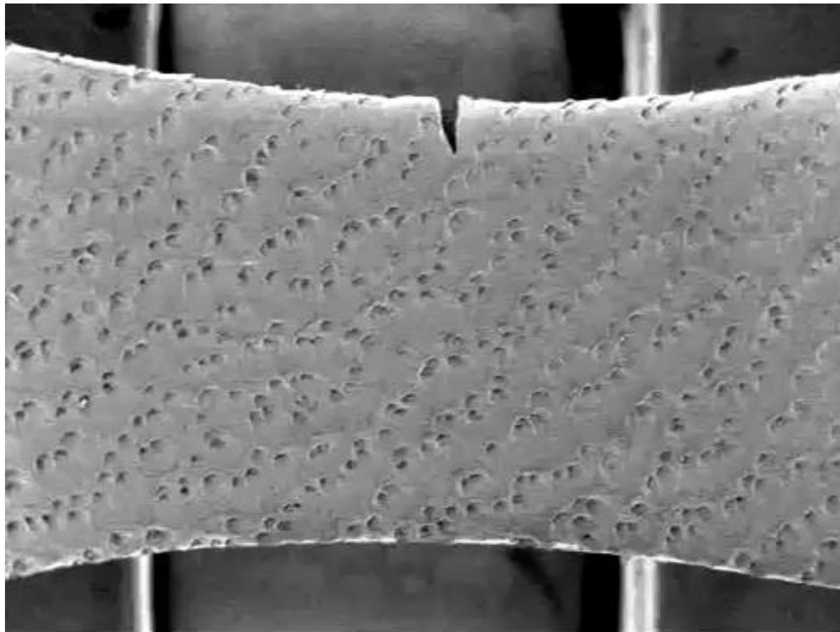
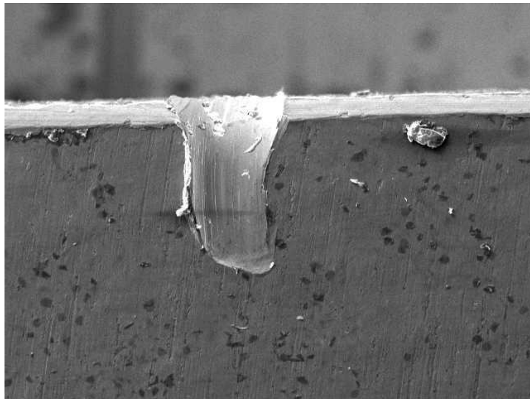
IN-SITU Tensile and compression stage

DEBEN





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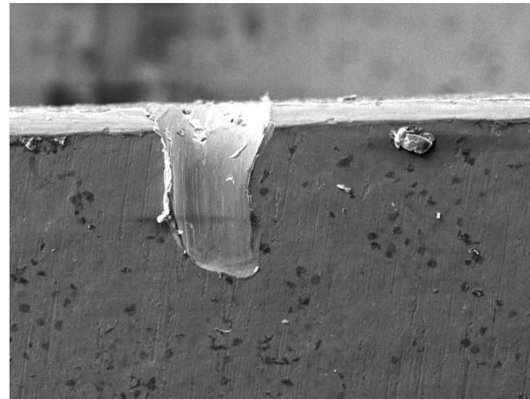
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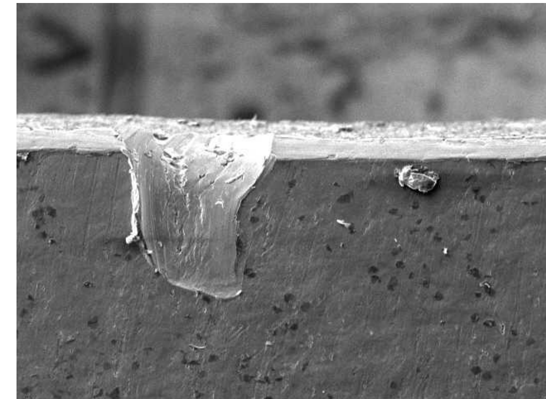
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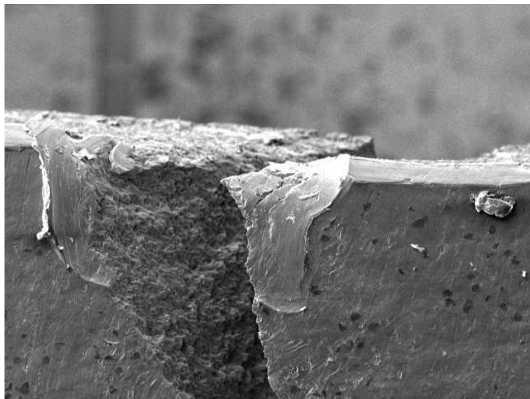
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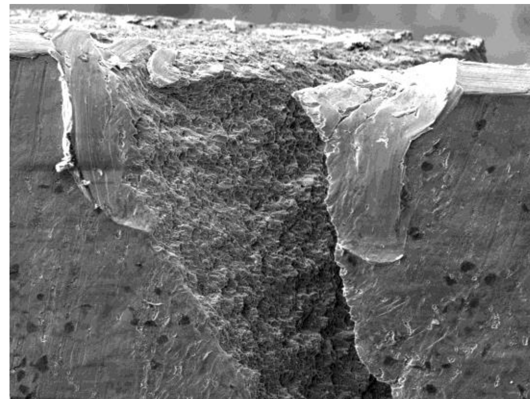
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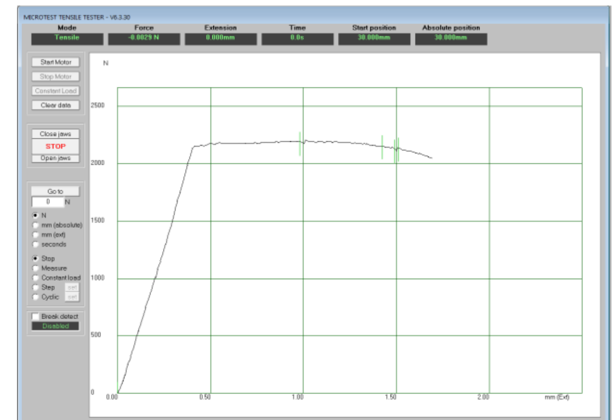
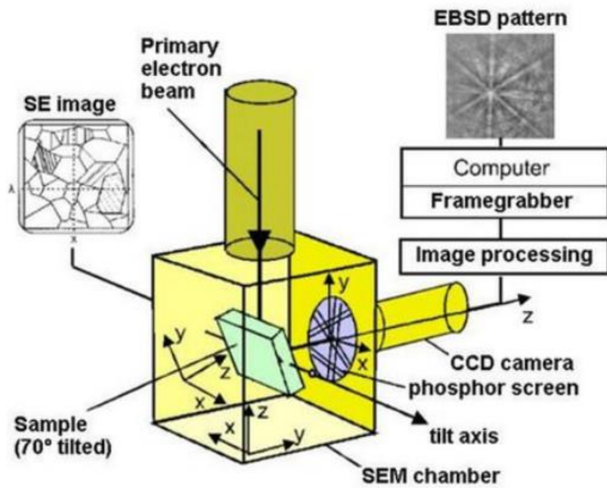
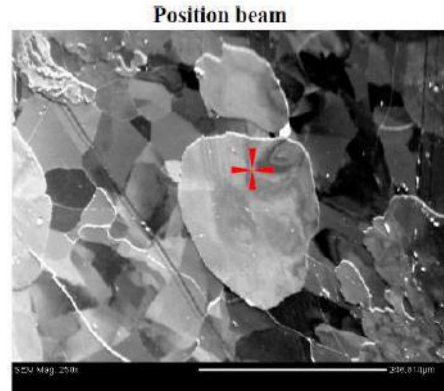
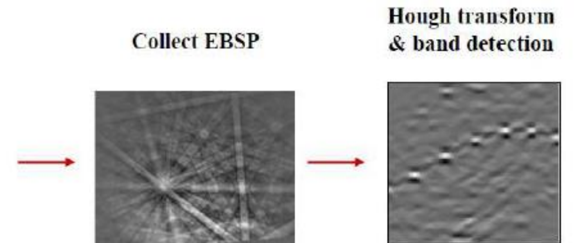


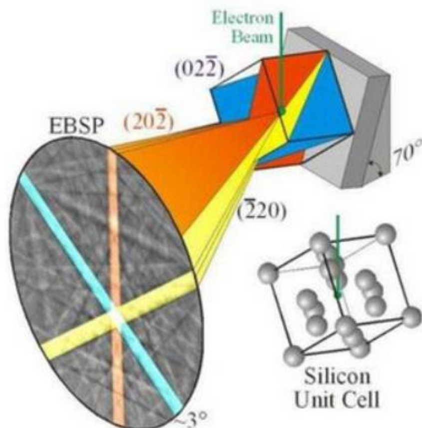
EBSD - Electron backscatter diffraction

Dyfrakcja wstecznie rozproszonych elektronów (EBSD)







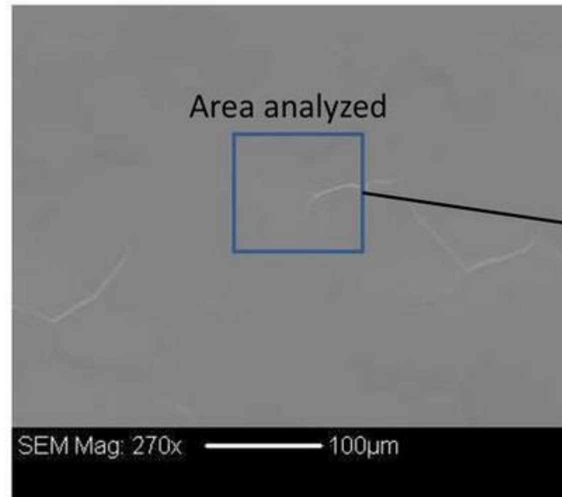


Document Oxford Instruments

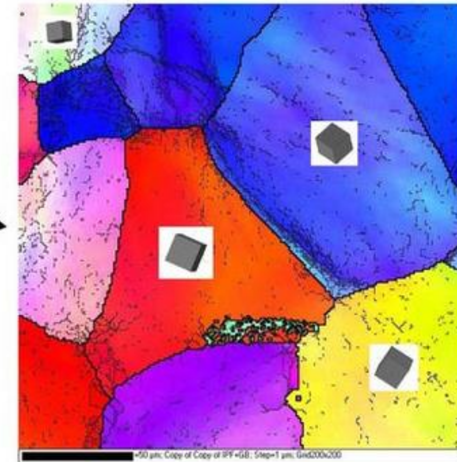
Pierwszy obraz EBSD opublikowano w 1928 roku!

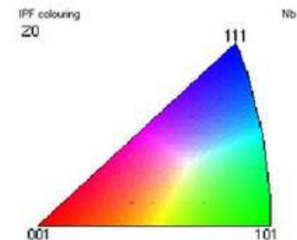
EBSD - Electron backscatter diffraction

SEM image



Grain boundaries shown for high (>10 deg) and low (>1 deg) angles



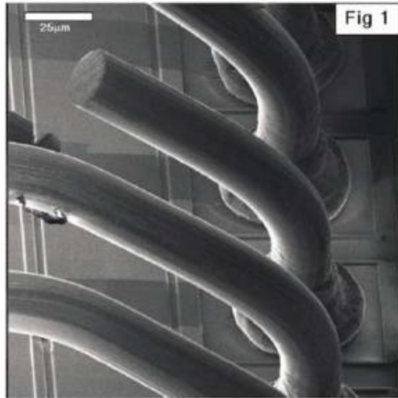


Możliwości :

Niedoskonałości sieci krystalicznej (dyslokacje)
Figury biegunowe/ Funkcja rozkładu orientacji
Rozkład dezorientacji
Rozkłady własności mikrostrukturalnych

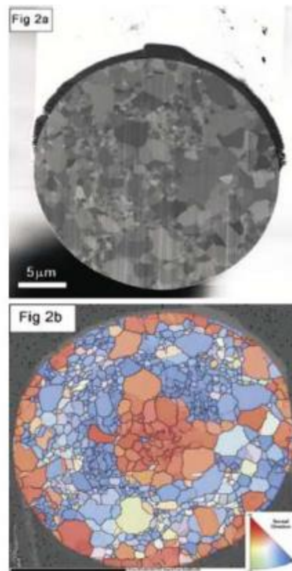
- Rozmiary i kształty ziaren
- Charakterystyki granic
- Niejednorodności w obrębie ziaren
- typy bliźniaków

EBSD - Electron backscatter diffraction

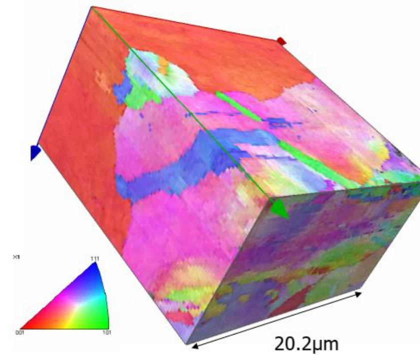


Gold wires, 25 µm diameter

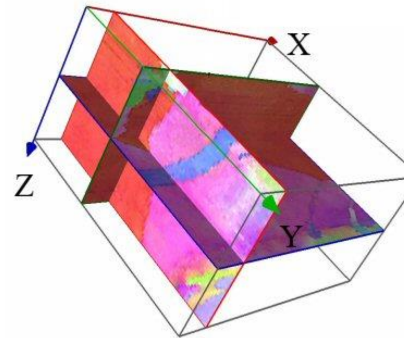
<100> fiber texture in red
<111> fiber texture in blue

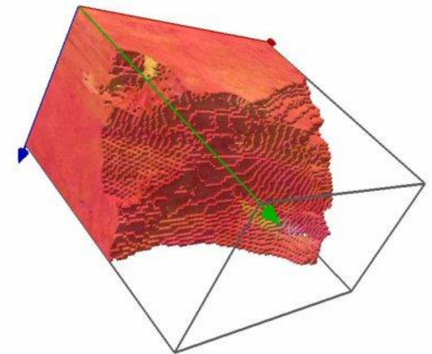


Grains size 0.72 µm



Sample: Copper
51 layers
0.2µm resolution in X, Y and Z
Number of voxels: 101x91x51
EBSD speed: 200^pic/s, 50 sec. per layer
FIB speed: about 45 minutes per layer
Total EBSD time: about 1 hour
Total FIB time: about 38 hours





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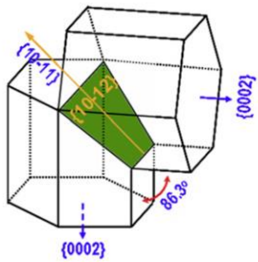
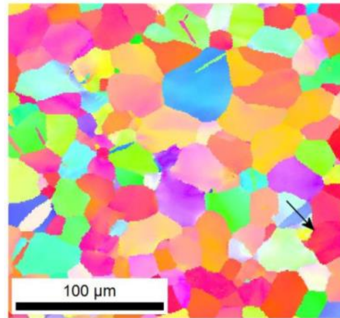
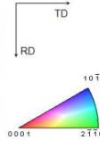
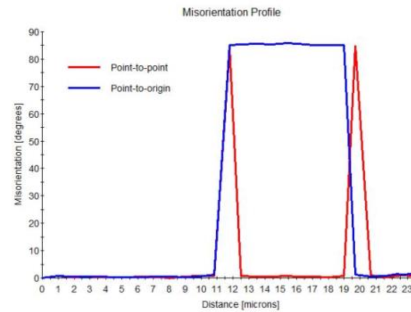
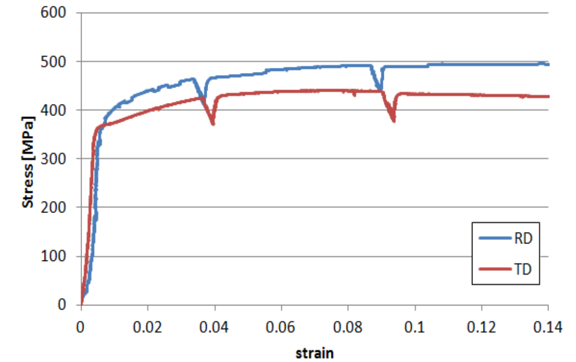


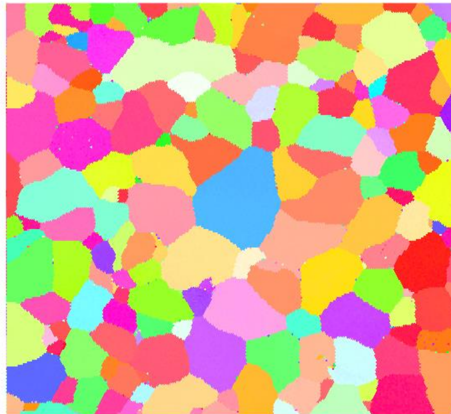




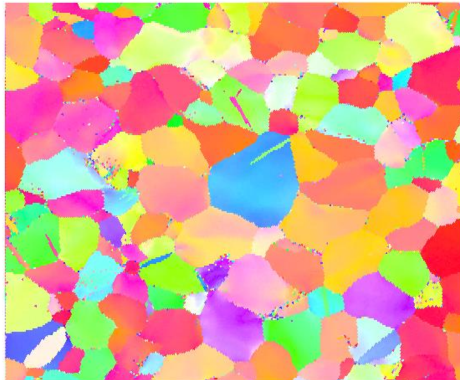




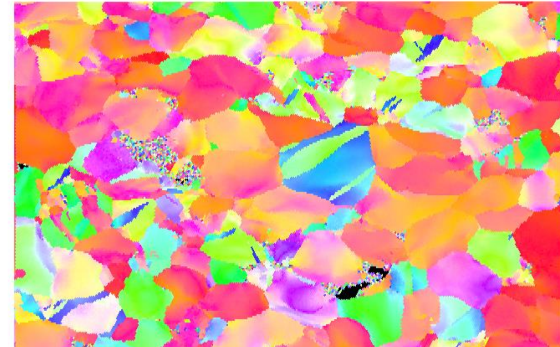
Strain = 0

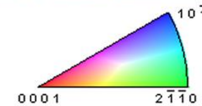


Strain = 5%



Strain = 10%



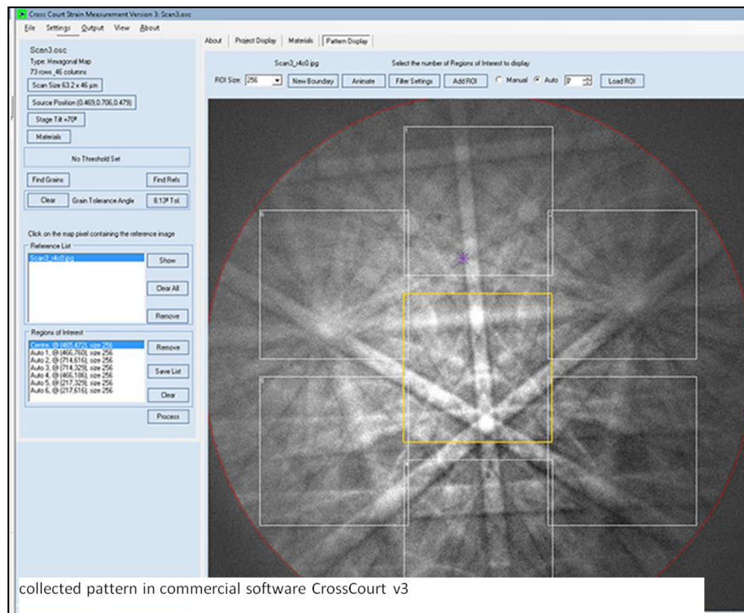


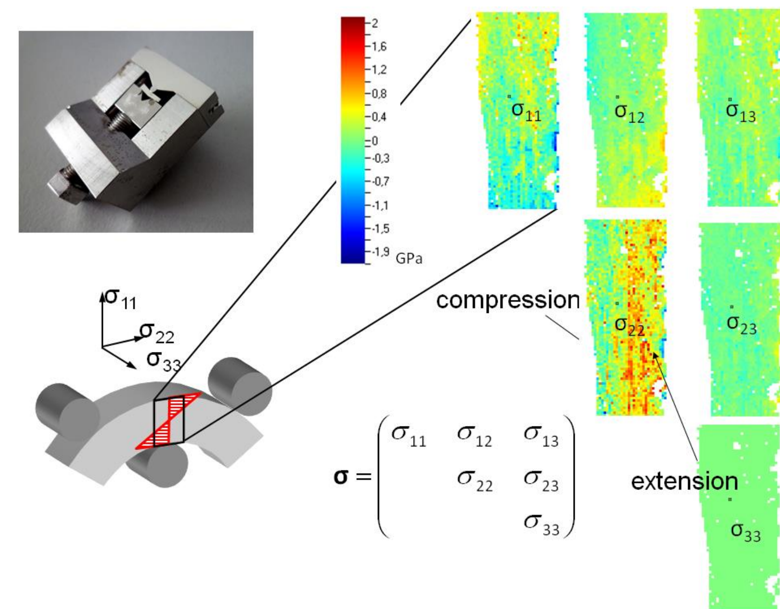
100 μm

The conventional analysis of EBSD patterns using a so-called Hough transform, allows measuring the orientation of a crystal with a precision of only about 0.5°.

Cross-correlation technique

In this technique a pattern obtained from a selected position on the sample is compared to a pattern from a reference point which is supposed to be free of elastic strain (or of known elastic strain) and which is very close in orientation to that of the measured position.





A.J. Wilkinson, G. Meaden, and D.J. Dingley. High resolution mapping of strains and rotations using electron backscatter diffraction. Mat. Sci. Tech., 22:1271–1278, 2006

https://www.mpie.de/3095346/Techniques_XR_EBSD

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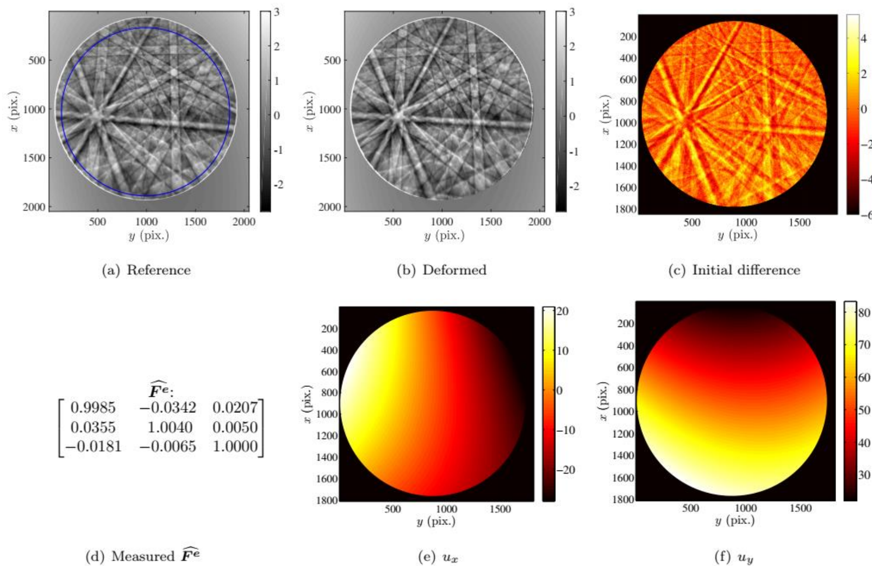
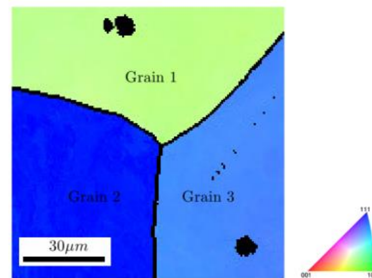
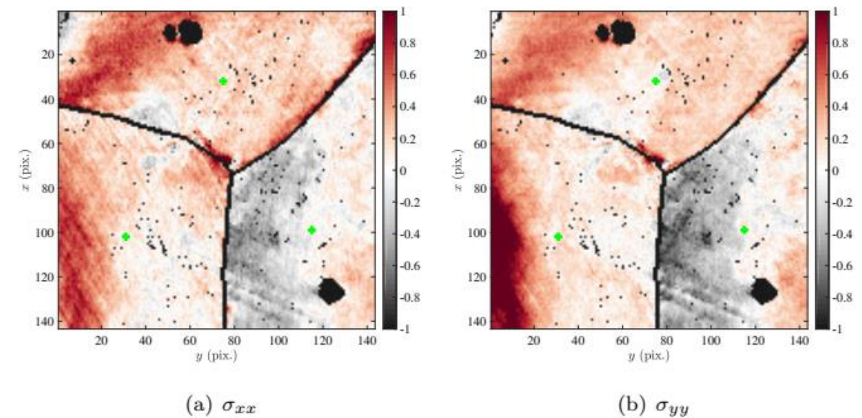
Qiwei Shi, Stéphane Roux, Félix Latourte, François Hild. Estimation of Elastic Strain by Integrated Image Correlation on Electron Diffraction Patterns. Ultramicroscopy, Elsevier, 2019, 199, pp.16-33.10.1016/j.ultramic.2019.02.001.

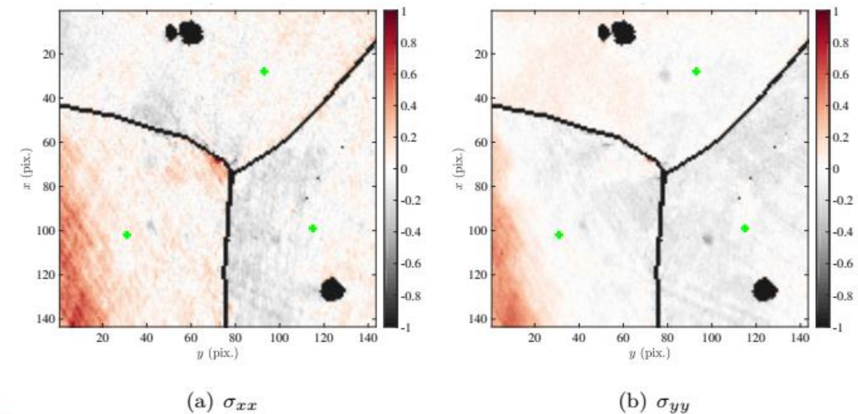




CrossCourt



ADDICTED



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a) Kossel Technique

Electron Probe

E C D A B

Electron Probe

Detector

D A B

$\lambda_{Specimen}$

Interplanar Spacing $d_{(hkl)i}$

Excitation Volume inside of Grain A

b) Pseudo Kossel Technique

Electron Probe

Solid Angle ω

E C A D B

Electron Probe

Target Holder

Target

λ_{Target}

Detector

Interplanar Spacing $d_{(hkl)i}$

Diffraction Volume inside of several Grains

c) EBSD Technique (Kikuchi)

$\lambda_{Electron\ beam}$

D E A B C

70°

Electron Probe

)*

Detector

A

Interplanar Spacing $d_{(hkl)i}$

)* Detectable

Diffraction Volume inside of Grain A

S Däbritz, E Langer, W Hauffe, Kossel and pseudo Kossel CCD pattern in comparison with electron backscattering diffraction diagrams, Applied Surface Science 179(1-4):38-44 (2001)

AGH

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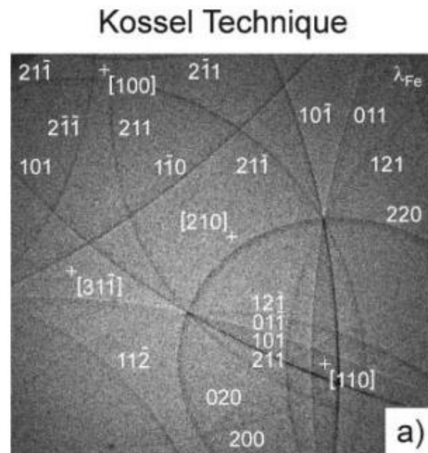
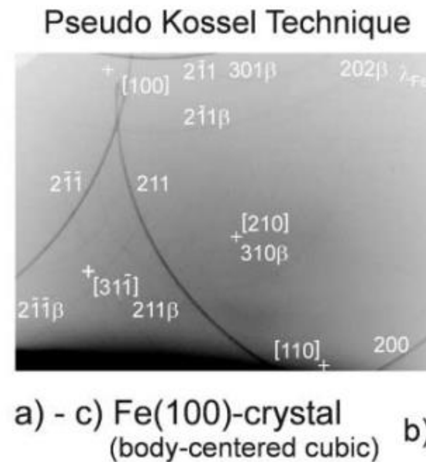
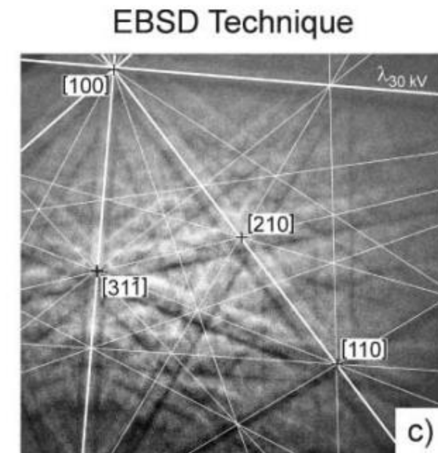
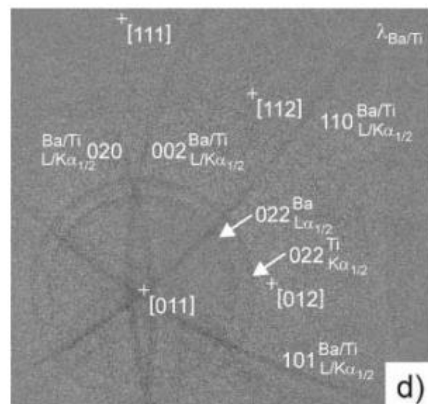
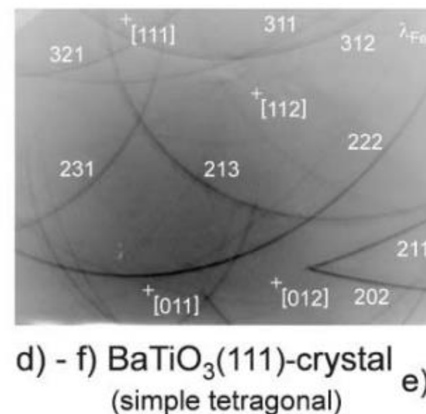




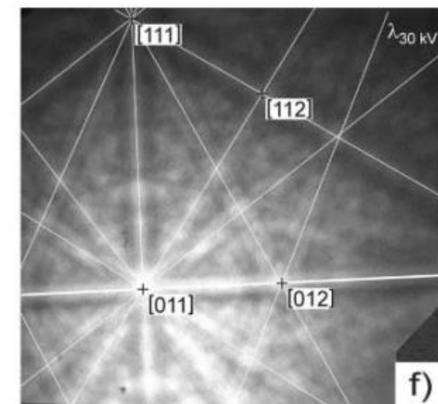
a) - c) Fe(100)-crystal
(body-centered cubic)







d) - f) $BaTiO_3$(111)-crystal
(simple tetragonal)



S Däbritz, E Langer, W Hauffe, Kossel and pseudo Kossel CCD pattern in comparison with electron backscattering diffraction diagrams, Applied Surface Science 179(1-4):38-44 (2001)

Component	LSI	DBI	EBSD
Generation depth	1–10 μm	1–100 μm	A few 10 nm
Lateral resolution	Only a few μm	μm to mm	>50 nm
Absolute crystallographic orientation	0.2°/0.4°	0.5°	About 1°
Relative crystallographic orientation	0.15°/0.3°	0.3°	0.5°
Precision of lattice constant, Δa	10^{-5} nm/10^{-4} nm	10^{-4} nm/10^{-3} nm	Only inexact

Comparison and application ranges of the methods LSI, DBI, and EBSD[a]

Parameters and application areas	LSI	DBI	EBSD
Proof of the single-crystal state in the microarea	+++	+++	+++
Investigation of polycrystals	+++	+++	+++
Determination of the crystal structure	+++	++	+
Determination of symmetry parameters	+++	++	
Measurements of a single grain	+++	++	++
Precision determination of lattice constants	* * *	++	
Determination of crystallographic orientations	+++	++	+++
Contributions to the texture analysis	+++	++	* * *
Mapping			+++
Detectability, location and determination of crystal defects	+	* * *	
Estimation of dislocation densities between 10^6 and 10^{10} cm^{-2}	++	+	
Proof of mechanical tensions and deformations	+++	+	
Determination of the crystal stoichiometry	++	+	
Determination of the expansion coefficient	+++	+	
Determination of the chemical concentration of elements	++		
Contribution to the sintering process	++	+	
New formation of phases and phase change in the high- and low-temperature range	++	+	
Distinction between polar directions of non-centrosymmetrical crystals	+++		
Measurements of residual stresses in micron regions	+++		

[a] * * *: main application range; +++: very good; ++: good; +: possible.

LSI – Kossel Technique
DBI – Pseudo Kossel Technique
EBSD - Electron backscatter diffraction

S Däbritz, E Langer, W Hauffe, Kossel and pseudo Kossel CCD pattern in comparison with electron backscattering diffraction diagrams, Applied Surface Science 179(1-4):38-44 (2001)

QImaging® Retiga-4000R

ccd sensor	
Light-Sensitive Pixels	4.19 million; 2048 x 2048
Binning Modes	2x2, 4x4, 8x8
ROI (Region of Interest)	From 1x1 pixels up to full resolution, continuously variable in single-pixel increments
Exposure/Integration Control	10µs to 17.9min in 1µs increments
Sensor Type	Kodak® KAI-4021 progressive-scan interline CCD (monochrome or color)
Pixel Size	7.4µm x 7.4µm
Linear Full Well	40,000e- (1x1); 80,000e- (2x2)
Read Noise	12e- @ 20MHz
Dark Current	1.64e-/pix/s
Cooling Type	Peltier thermoelectric cooling to 25˚C below ambient
Digital Output	12 bits
Readout Frequency	20, 10, 5MHz
Frame Rate	4fps full resolution @ 12 bits (125fps maximum with binning and ROI functions)

KAI–4021 CCD Image Sensor

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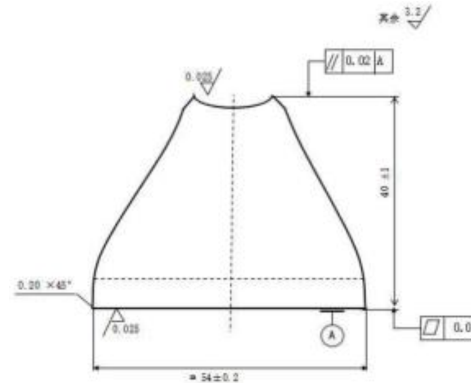
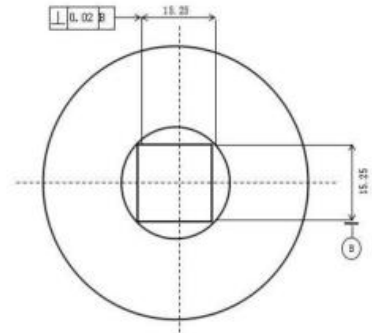
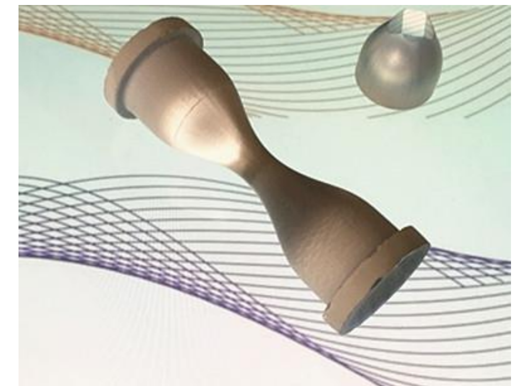
fiber optic taper

Measurement	Specified and Tolerance
Input diameter	Ø54±0.2mm
Height of taper	40±1mm
Output	15.25x15.25(±0.2)mm
Magnification Ratio	2.55:1±3%
Single fiber diameter	6μm with EMA
Numerical aperture	1.0 (small end)
Core/clad ratio	70/30
Total distortion	≤4% of effective area
Frame run out	≤0.5mm
Shear distortion	≤250μm
Barrel/pin cushion	≤4%(effective area)
Surface flatness	N=4







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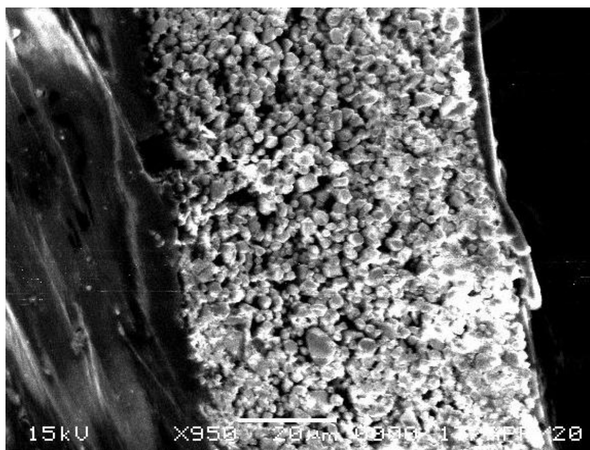
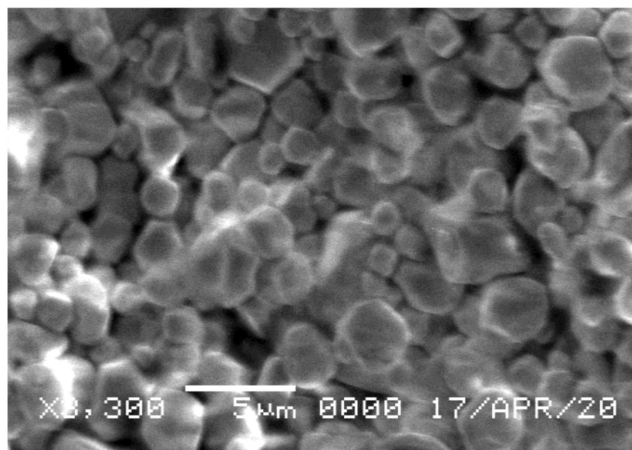
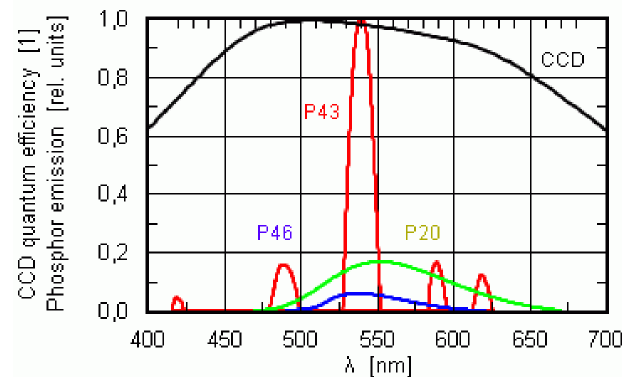
Scyntylator - MAMORAY™ HDR-C Plus Gd2O2S:Tb





P43 Phosphor screen

composition	Gd_2O_2S:Tb
efficiency	up to 600 ph / e- @ 10 keV
light emission	green (max. @ 545 nm)
decay time	1 ms from 90% to 10%
	1.6 ms from 10% to 1%



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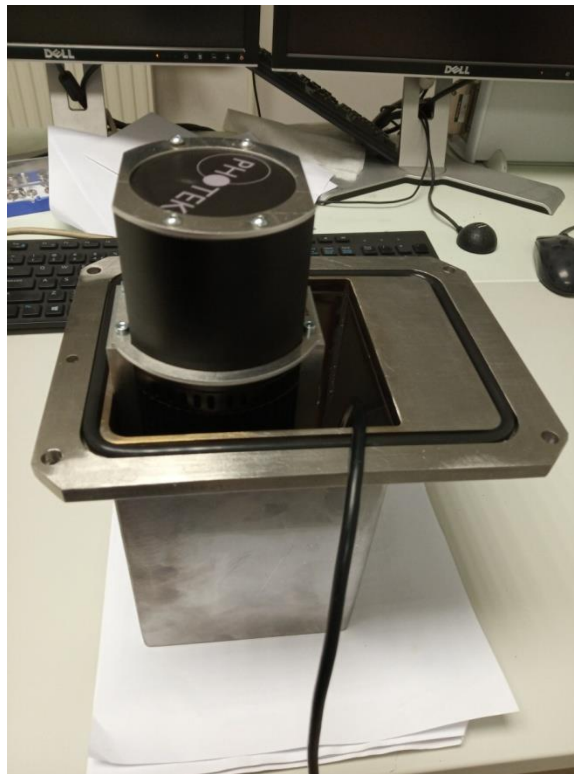

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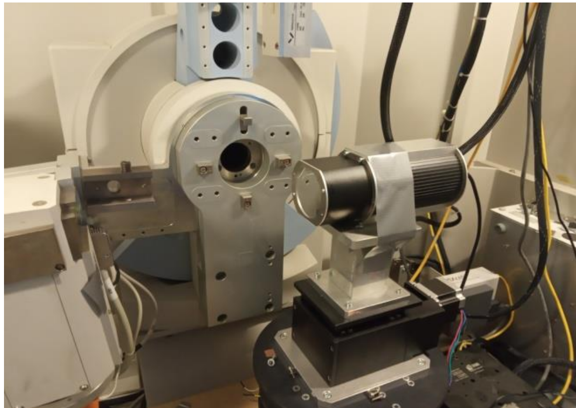

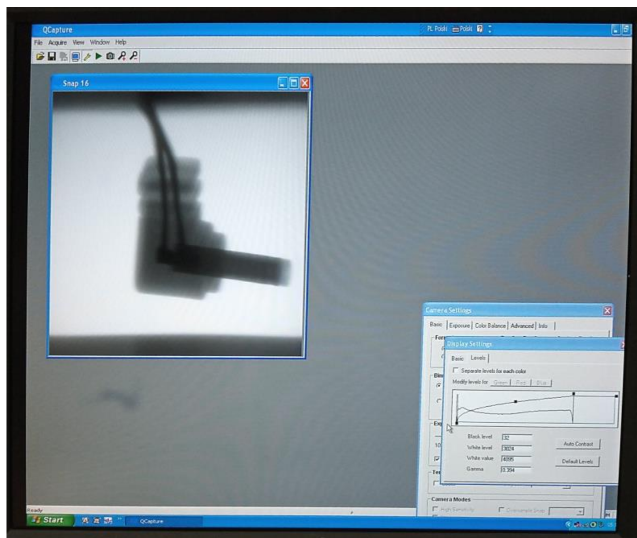

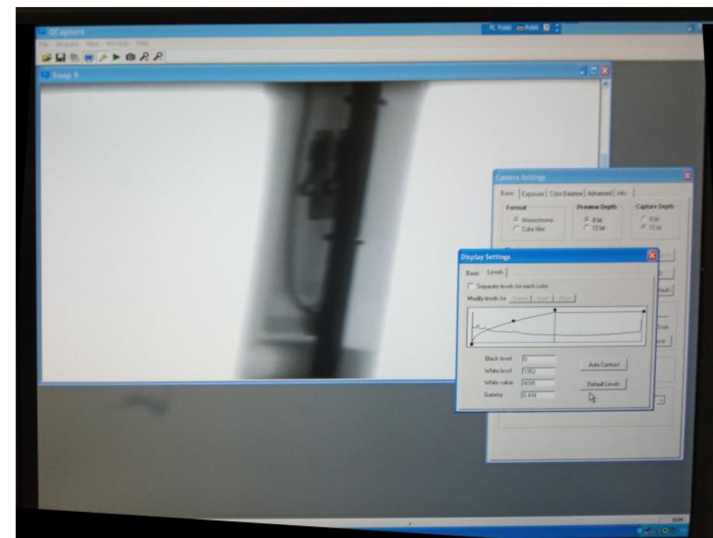

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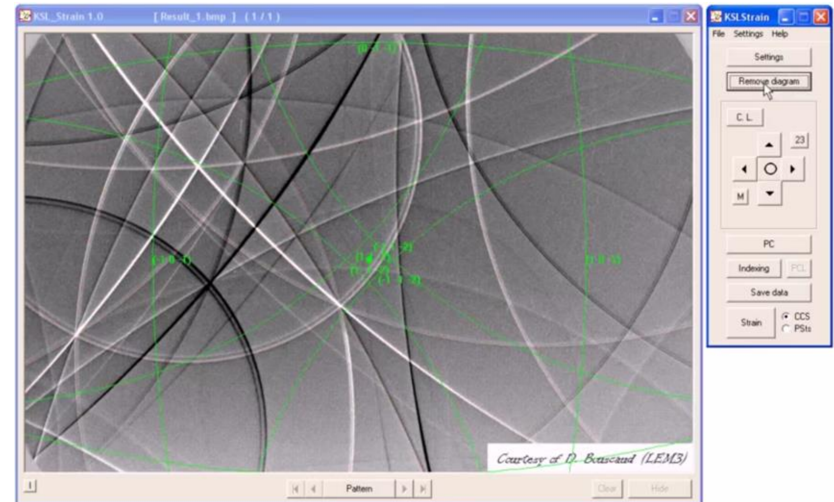
KSL*Strain* - a program for analysis of
Kossel diffraction patterns

Instytut Metalurgii i Inżynierii Materiałowej
im. Aleksandra Krupkowskiego **Polskiej Akademii Nauk**

Dr hab. Adam Morawiec, Prof. PAN



A. Morawiec, A program for refinement of lattice parameters and strain determination using Kossel diffraction patterns, J. Appl. Cryst. (2016). 49, 322-329

Dziękuję za uwagę