Markus Aicheler, Ruhr-University Bochum

CLIC Material Fatigue Study:

Thermal Fatigue Behavior versus Grain Orientation

Outline of the talk

- Motivation
- Observed material and tempers
- The 4 fatigue experiments
- Results and Discussion EBSD-Observation

Motivation

1. Observe influence of crystallographic orientation on fatigue behavior
2. Show similarities between the different fatigue tests

   => Show that the fatigue tests are coherent and relevant for CLIC structure!
3. Develop ideas how a good material against pulsed surface heating should look like

Observation material

C10100 (OFE Copper)

- Reference material
- Well known
- Results comparable to other researchers
- Supplementary fatigue data needed (CuZr already well tested by Samuli)

40% cold worked

- as received
- Round bar cold rolled Ø 40 mm and Ø 100 mm
- Yield Strength: $R_{p0.2}$ = 316 MPa
- Ultimate tensile strength: R_m = 323 MPa
- Average grain size: Ø 110 um

Brazed

- Heat treatment in vacuum furnace:

300 K/h -> 795 °C; 60 min hold

100 K/h -> 825 °C; 6 min hold

Natural cooling in vacuum

- Yield Strength: $R_{p0.2}$ ≈ 72 MPa
- Ultimate tensile strength: R_m = 270 MPa
- Average grain size: Ø 400 um

2h@1000 °C

- Heat treatment in vacuum furnace:

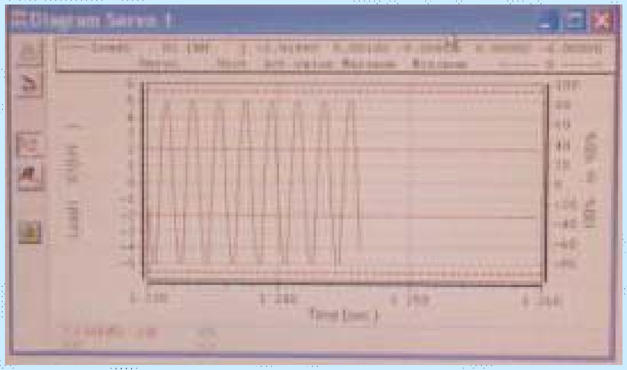
300 K/h -> 1000 °C; 120 min hold

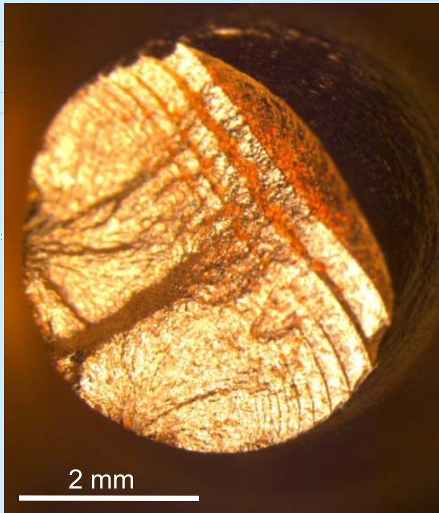
Natural cooling in vacuum

- Yield Strength: $R_{p0.2}$ ≈ 72 MPa
- Ultimate tensile strength: R_m = 257 MPa
- Average grain size: Ø 1400 um

Conventional fatigue test

- Mechanical fatigue; R = -1 (R = σ_{max}/ σ_{min})
- UTS electro-mechanical universal-test machine
- Repetition rate 0.5 Hz
- Load +/-220 MPa; stress controlled
- Sample shape conform ISO 12106
- 3-5 samples for one data point
- Damage criterion: rupture



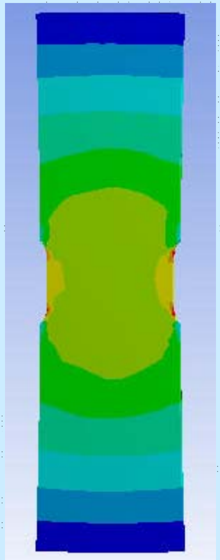


Ultrasound swinger test

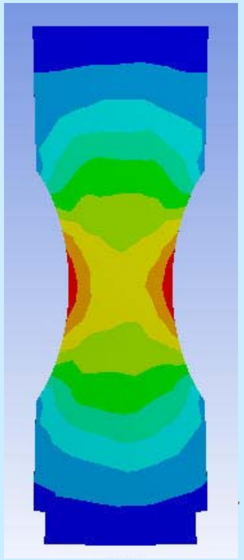
- Mechanical fatigue; R = -1 ($R = \sigma_{max} / \sigma_{min}$)
- Piezo electric resonant attenuator
- Repetition rate 24 kHz
- Load +/-70 MPa; strain controlled
- 3-5 samples for one data point
- Damage criterion: roughness or crack

New sample shape:

- easier surface observation & replica
- one defined gage length
- easier to machine & electro polish
- comparison to results of other researchers

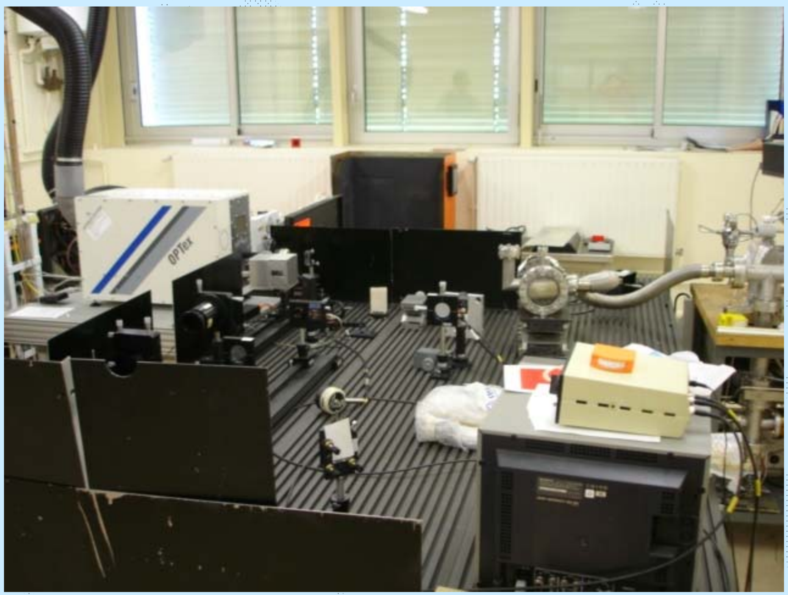


Former shape

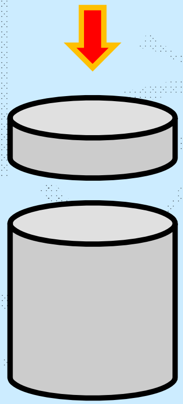


New shape

Laser fatigue test



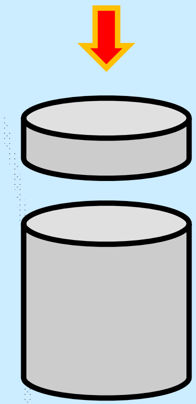
- Thermal fatigue in vacuum
- OPTEX Excimer Laser; λ = 248 nm
- Repetition rate 200 Hz
- 5 x 10^4 shots @ 0.3 J/cm^2 ≈ ΔT 250 °C
- Round disc diameter 40 mm
- 25 discrete spots per disc
- Damage criterion: Roughness

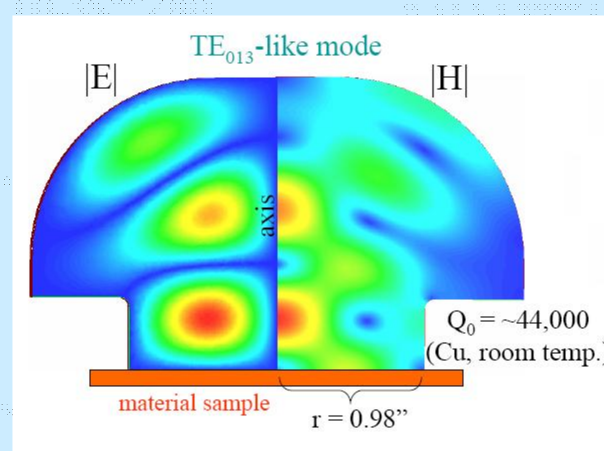


SLAC RF heating (Stanford)

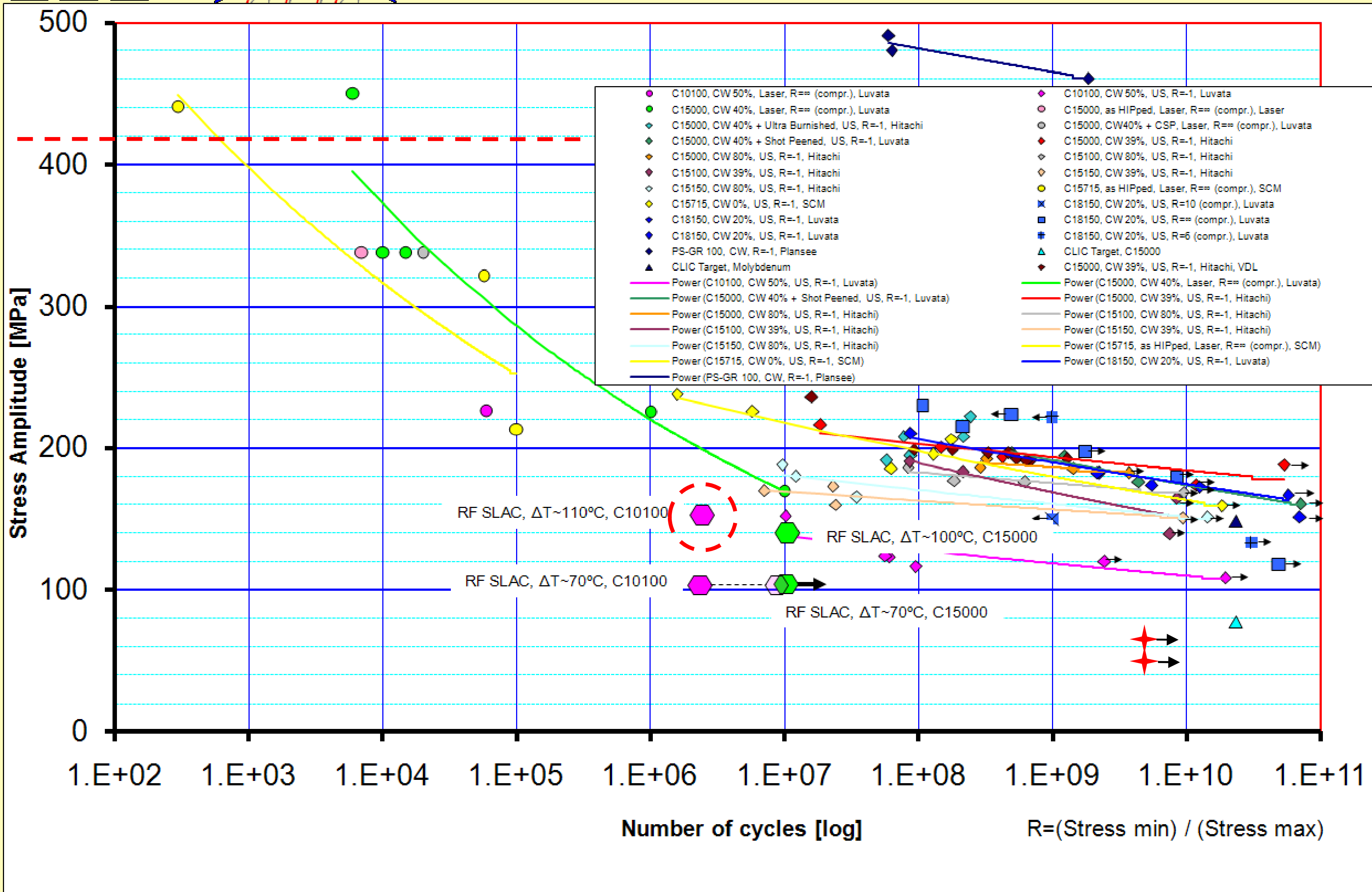
Photos: Sami Tantawi
Presentation 23 Jan. 2008

- Thermal fatigue due to RF heating
- Mushroom cavity @ 11,4 GHz
- Repetition rate 50 Hz
- 2 x 10^6 Pulses; ΔT_{max} 110 °C
- Round disc diameter 100 mm
- Continuous radial distribution of ΔT
- Damage criterion ?





Wöhler curves of the test results (Stress vs. N)



Observation techniques used: EBSD

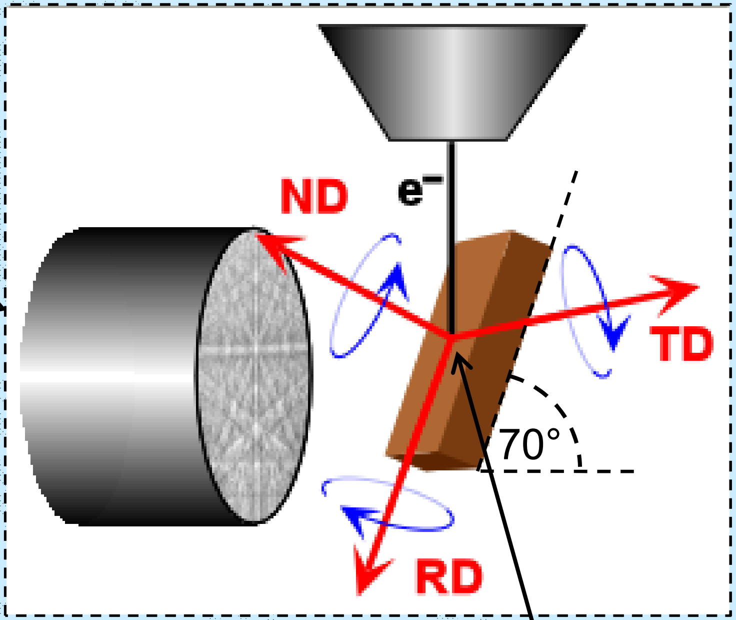
Electron Back Scattered Diffraction

SEM: Leo 1530 VP

EBSD unit: EDAX TSL

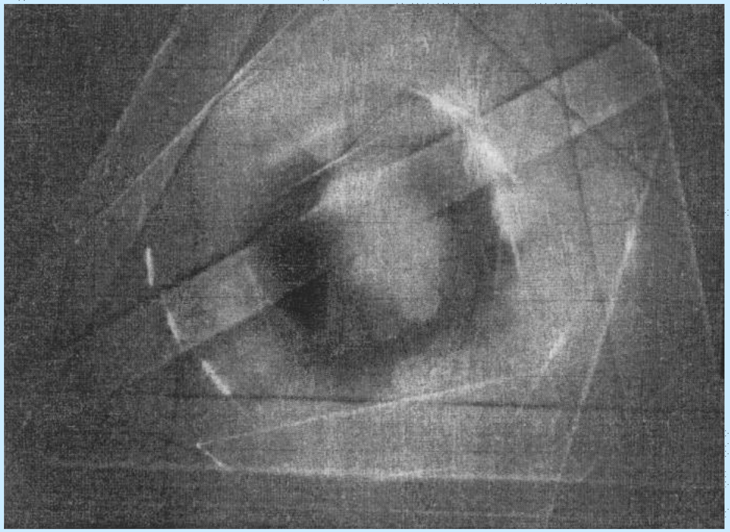
Phosphoric screen and digital camera

Electron Microscope column



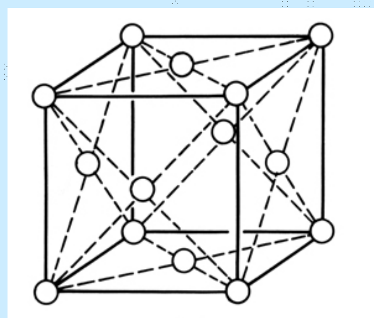
"Bragg Reflection"

Kikuchi pattern



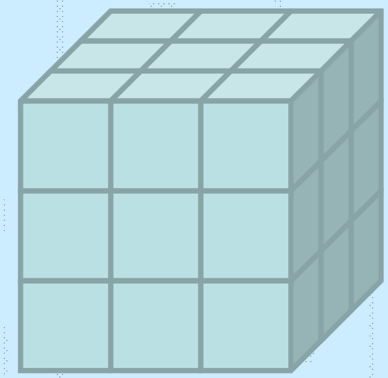
Crystallography

A cube with atoms on its corners and its faces (so called face centered cubic FCC)

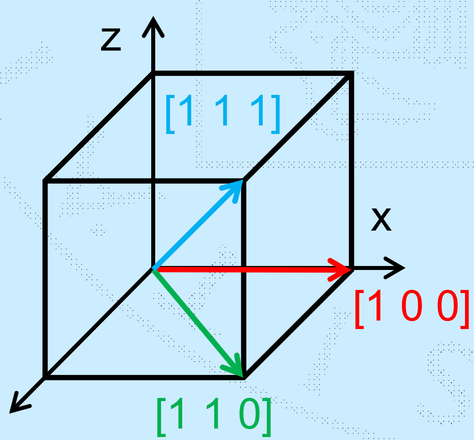


(elementary cell)

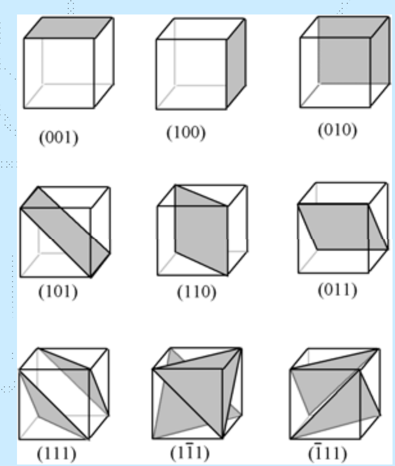
Plenty of these elementary cells



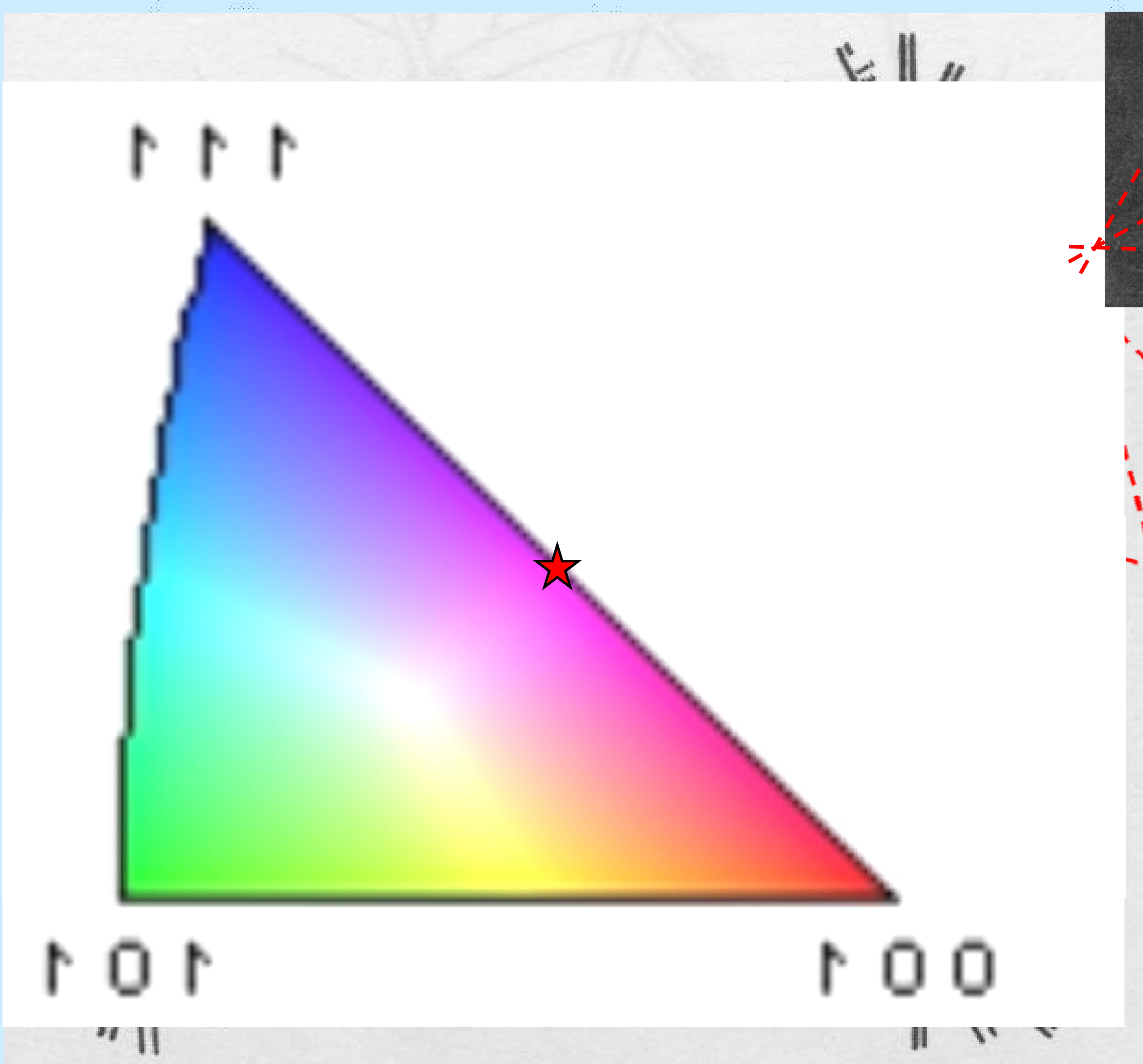
Crystallographic description of directions:

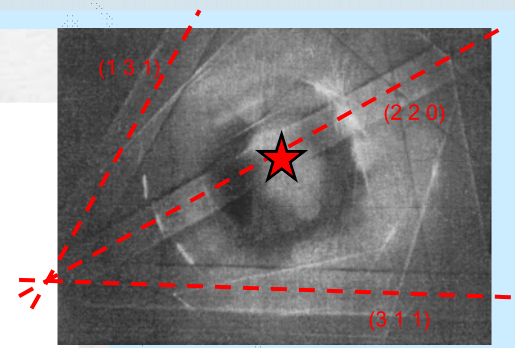


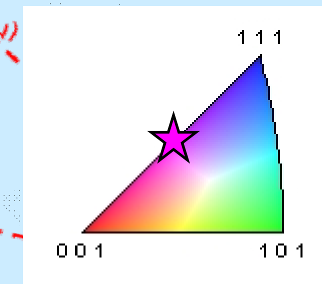
Crystallographic description of planes: Normal vector of planes!

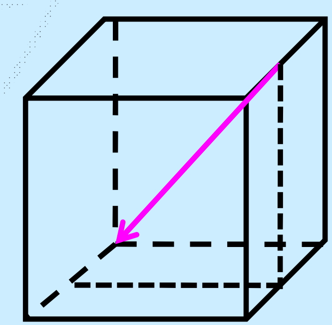


Kikuchi line indexing









EBSD

Scan:

Each point one Kikuchi pattern

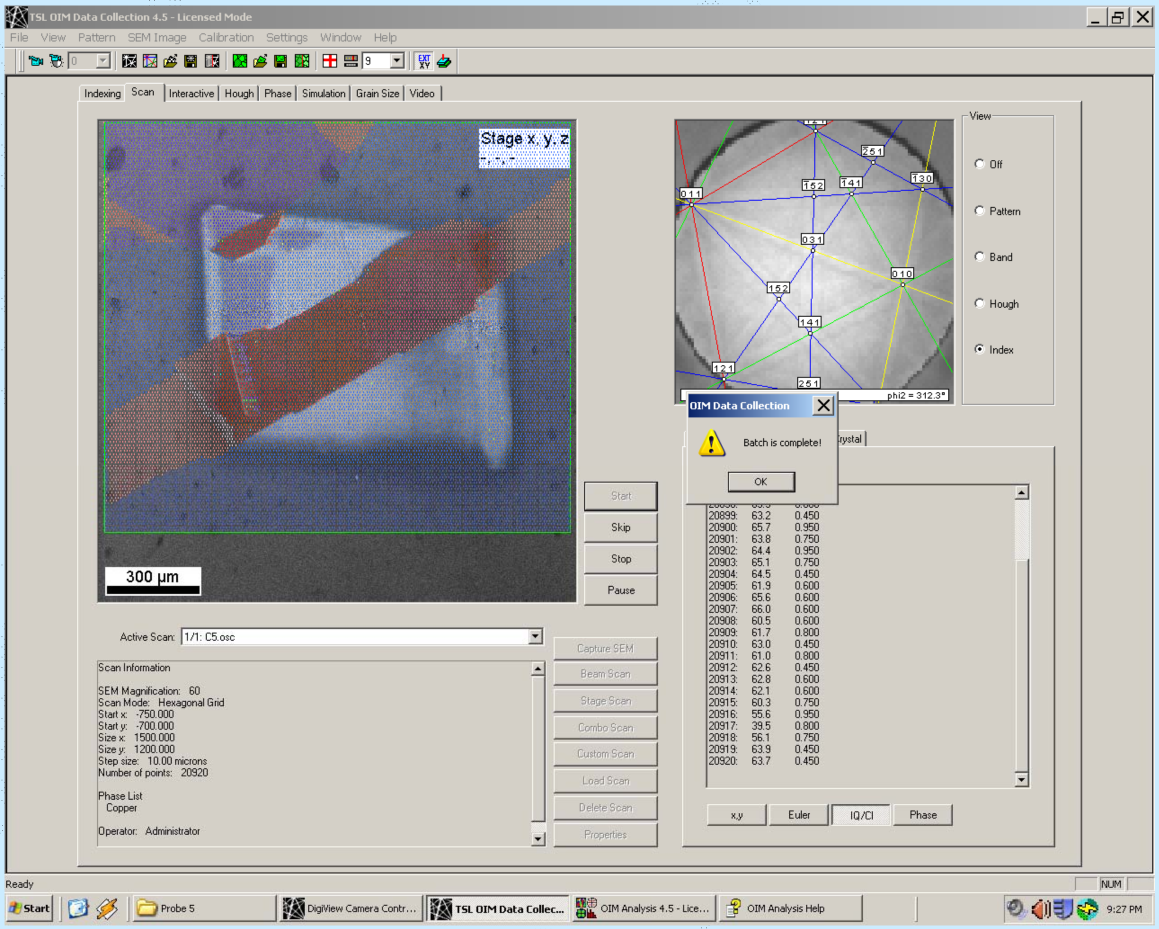
-> each point complete orientation information

=> Mapping of orientations

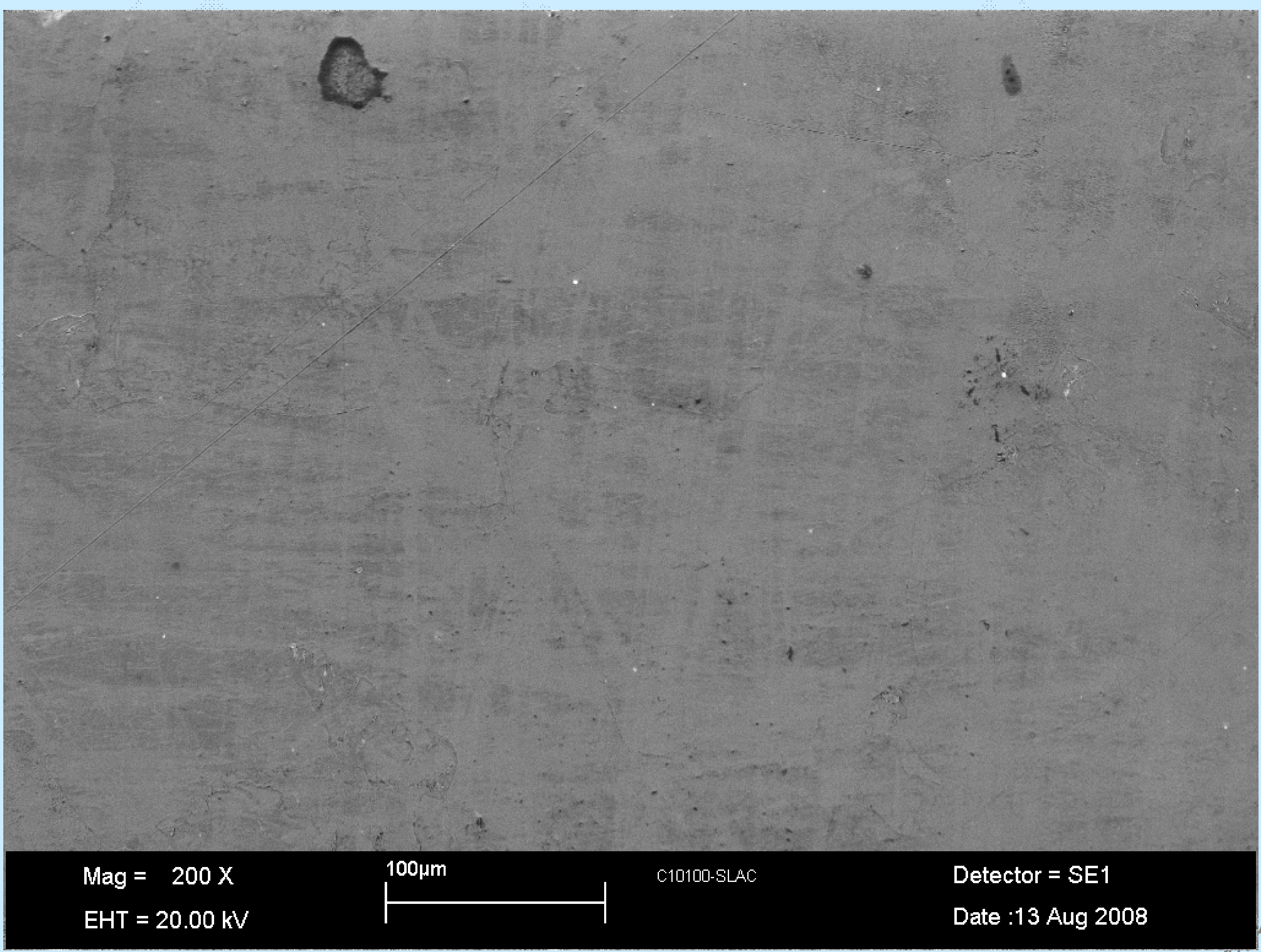
Scan rate: 75 points/s

Ordinary used for:

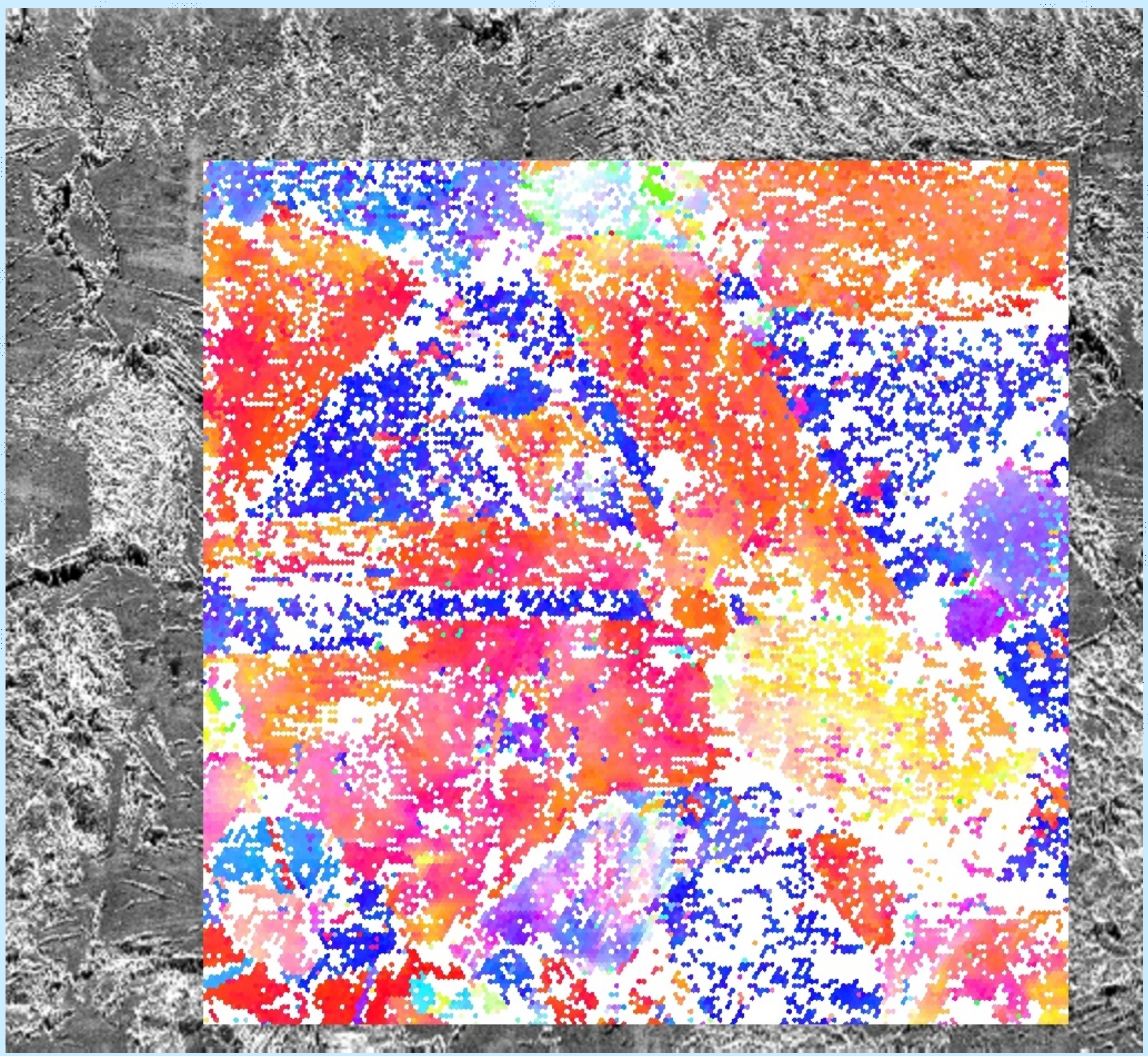
- Texture analysis
- Orientation of samples (like X-Ray diffraction but faster)
- Identification of different phases (like TEM but lower resolution/magnification)
- Possibility to connect with quantitative EDX scans

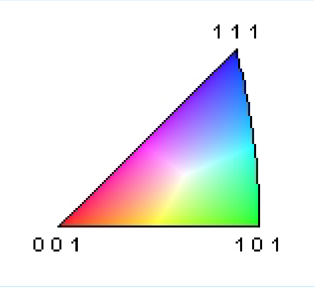


SLAC Virgin Surface

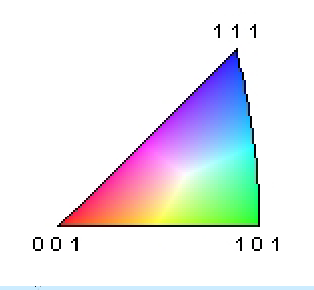


SLAC_Slash5





C10100_2h@1000_EP_Probe5_C5



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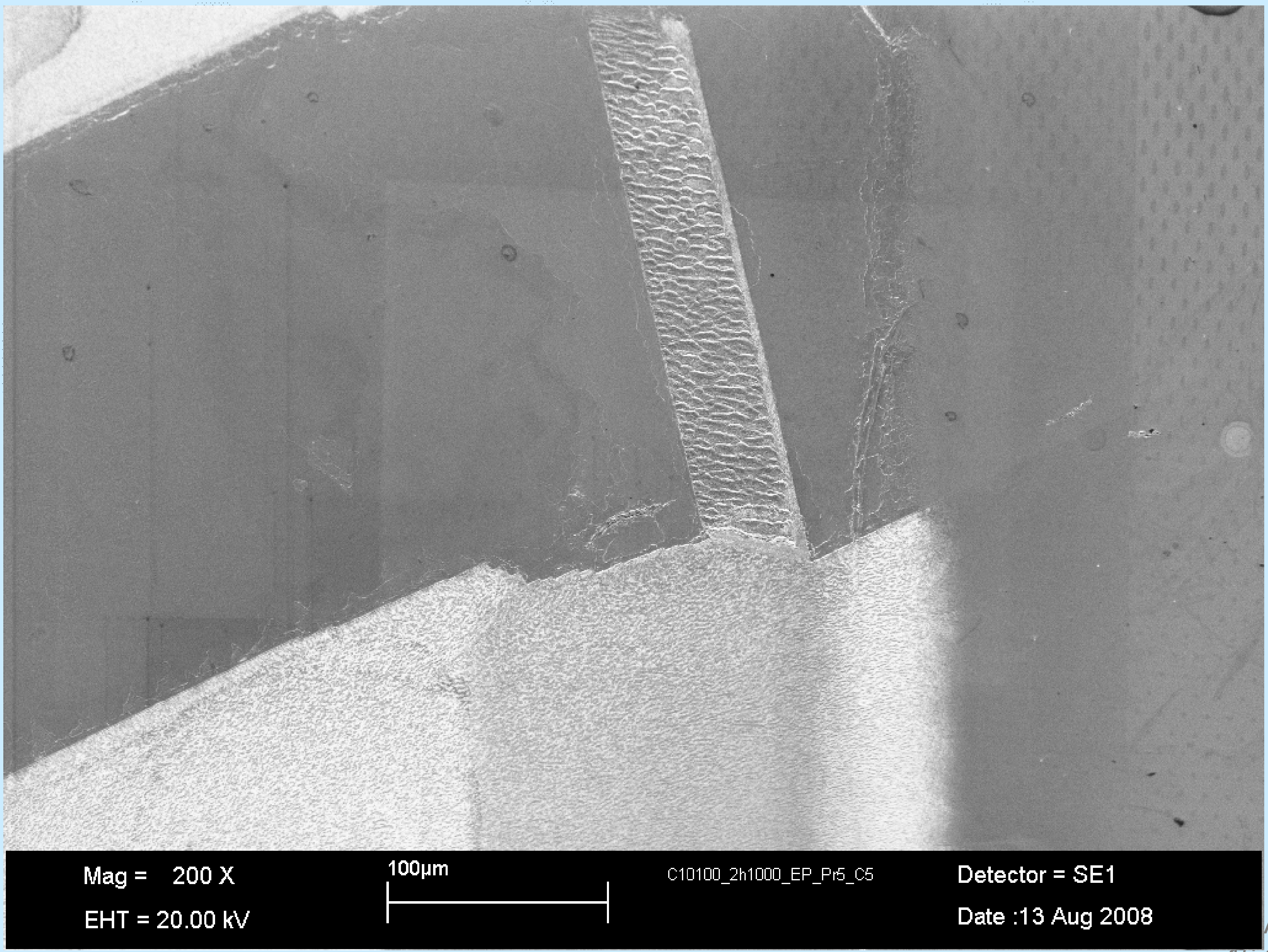

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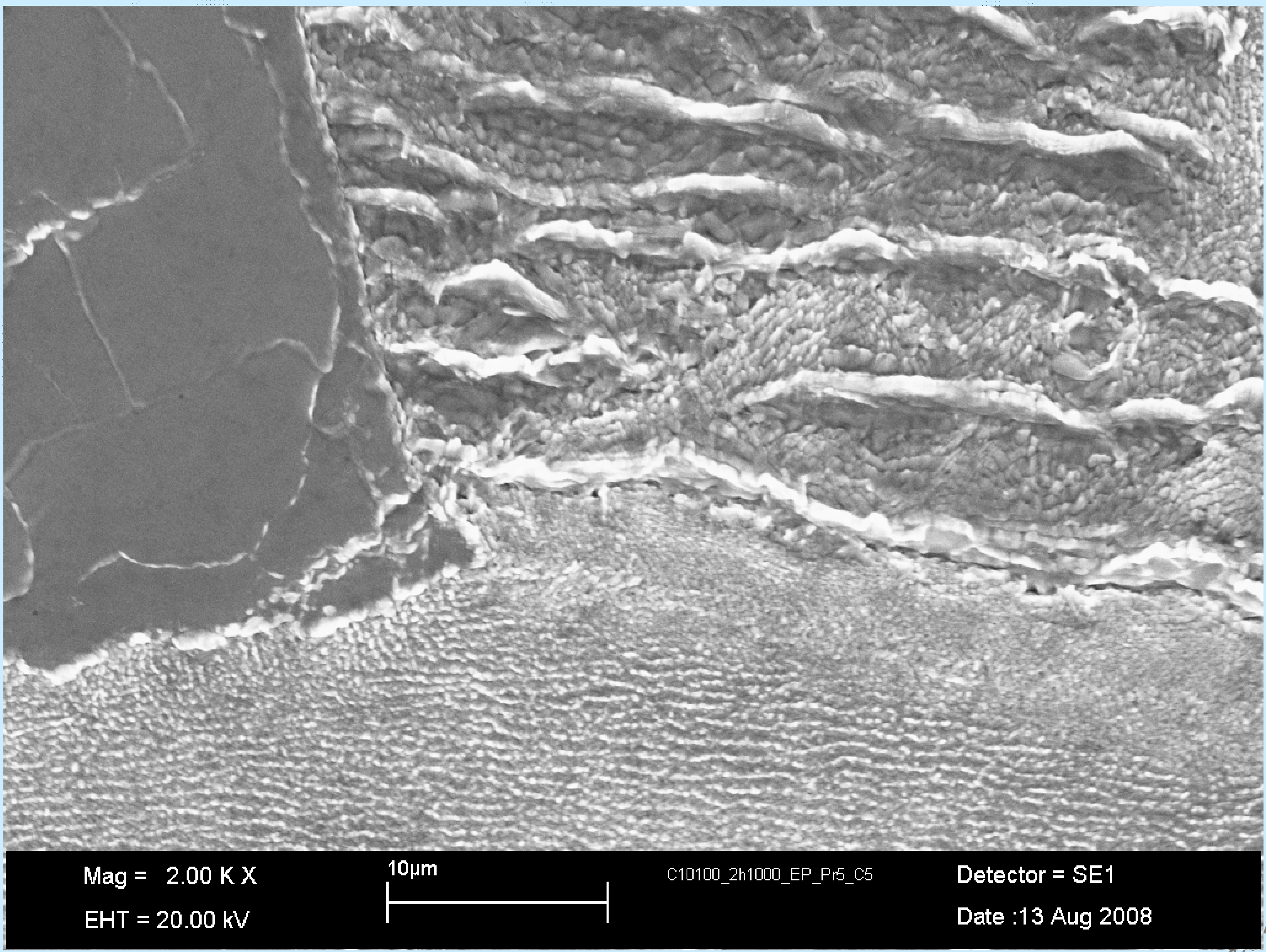

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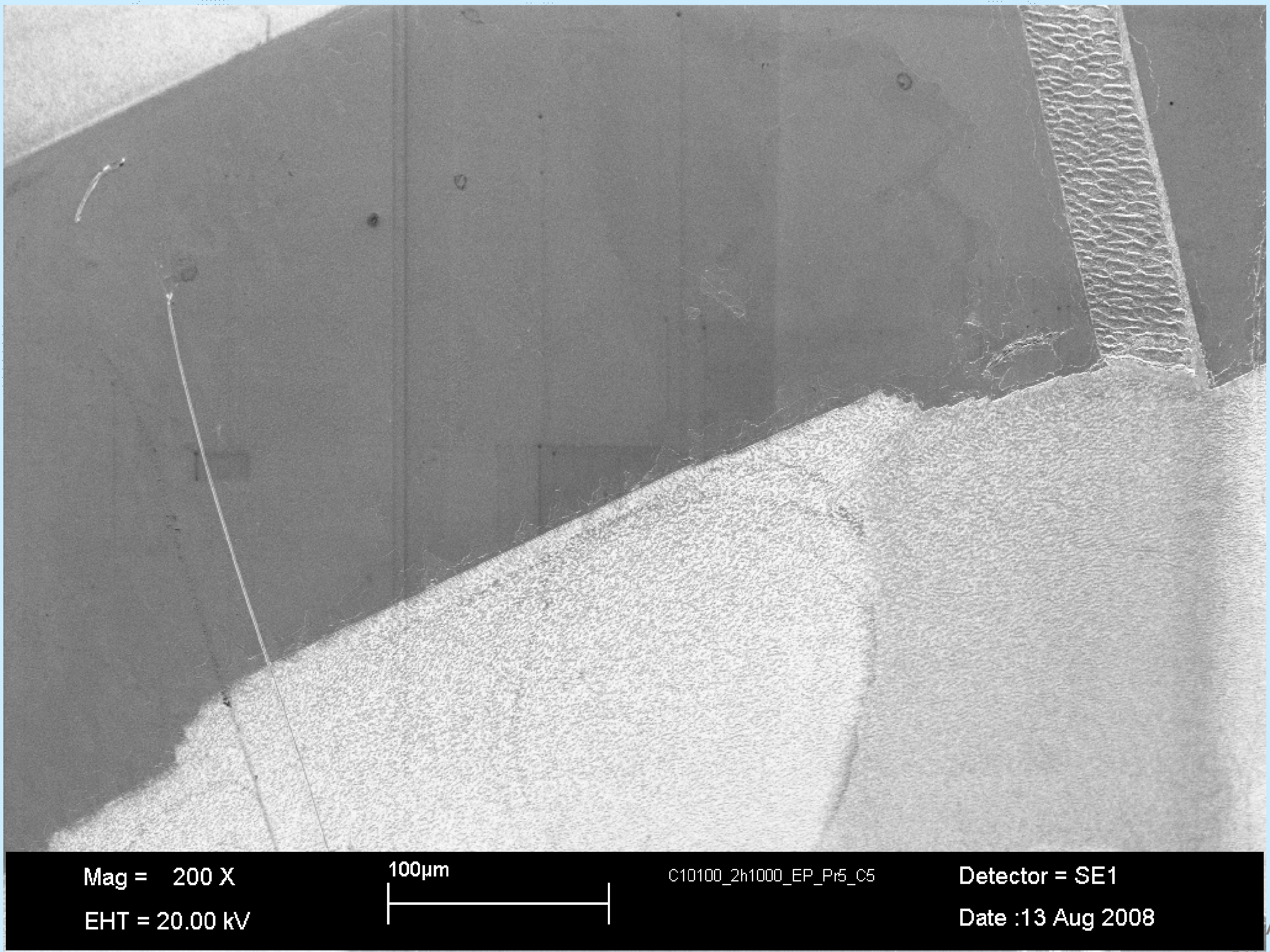

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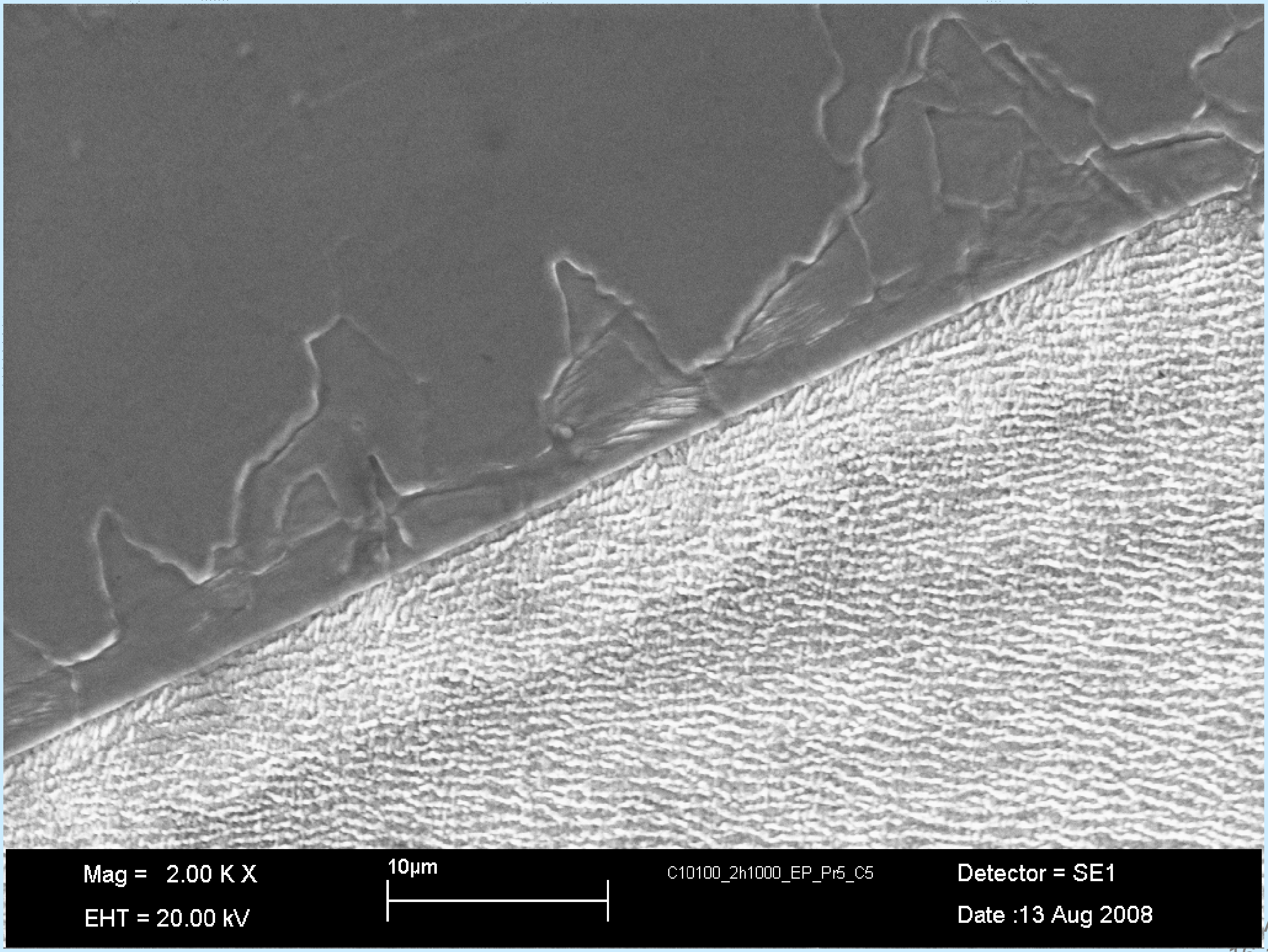
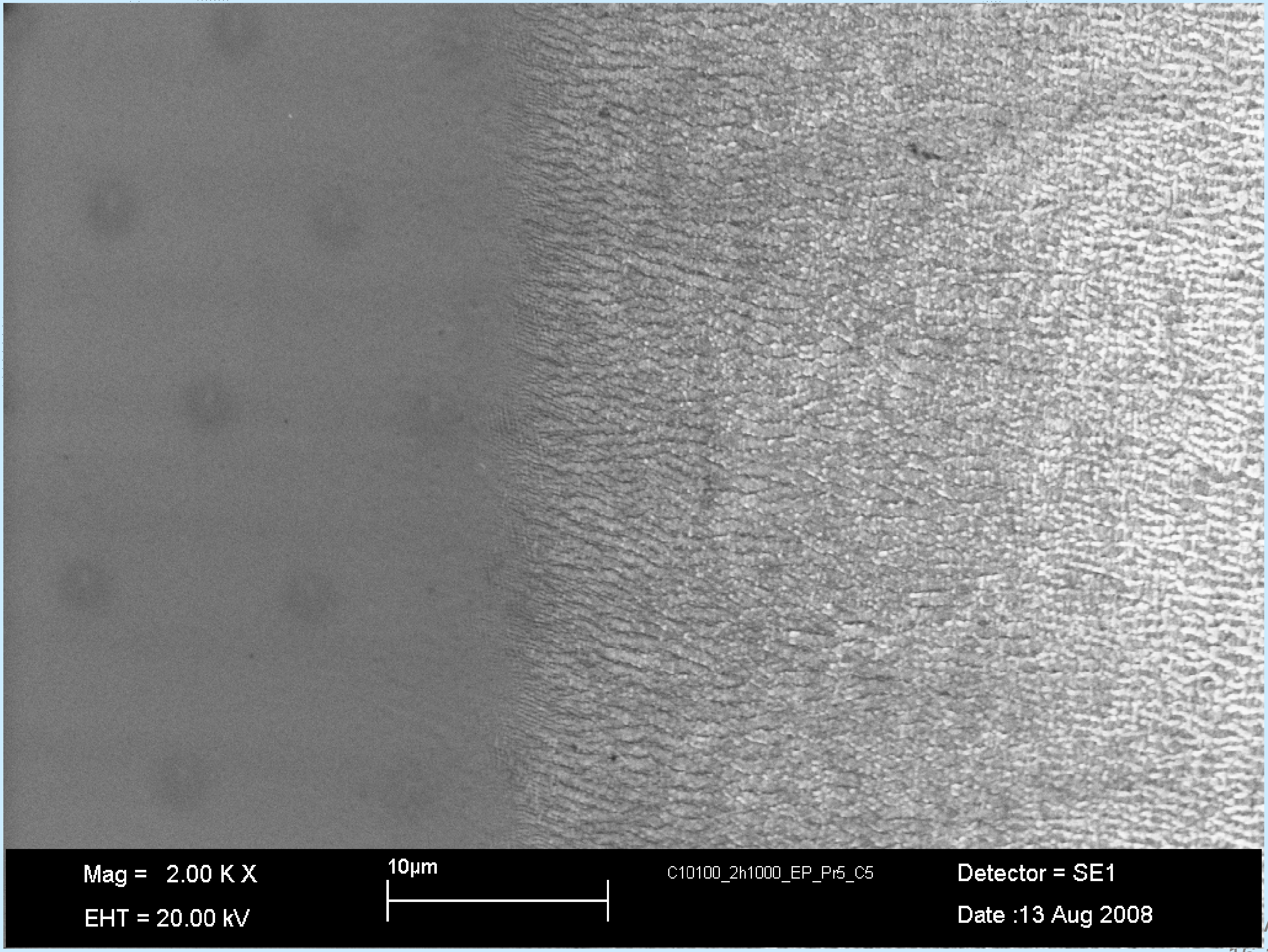
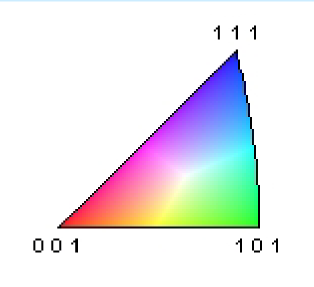


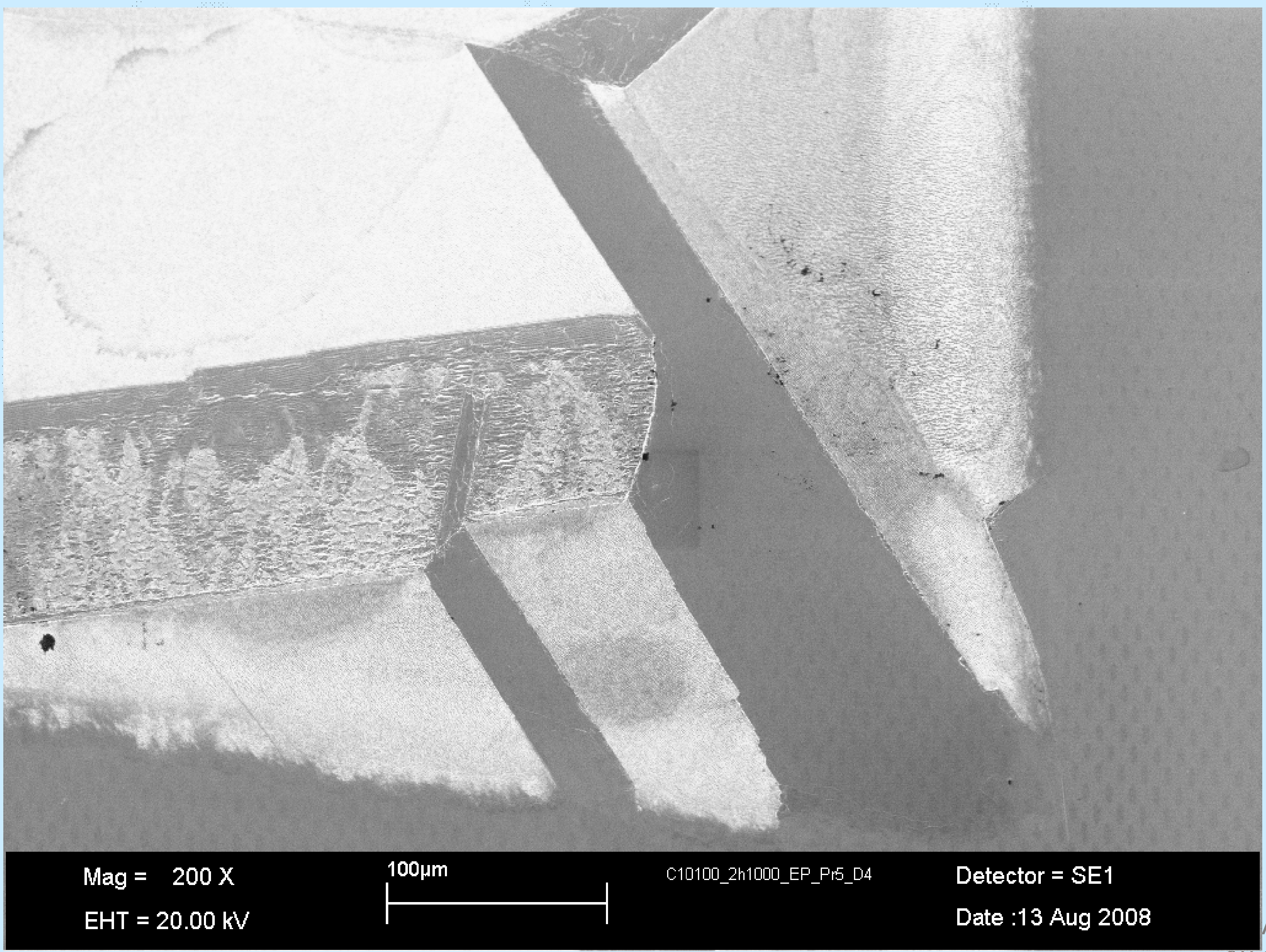
C10100_2h@1000_EP_Probe5_C5 Virgin Surface



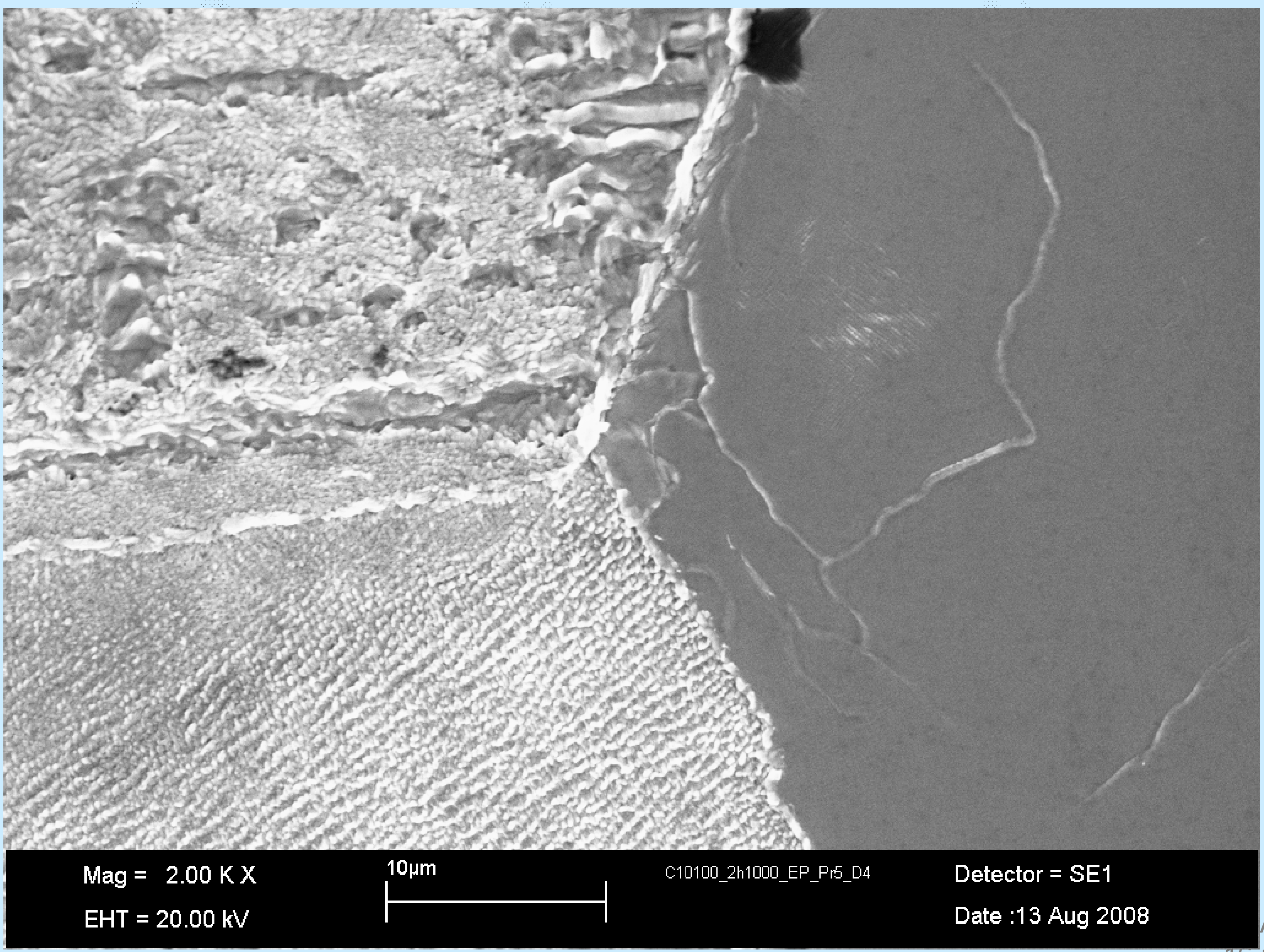
C10100_2h@1000_EP_Probe5_D4



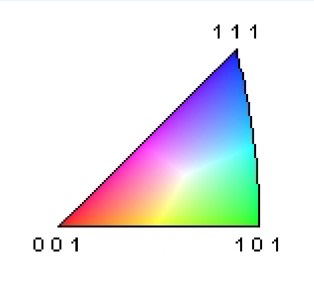
C10100_2h@1000_EP_Probe5_D4



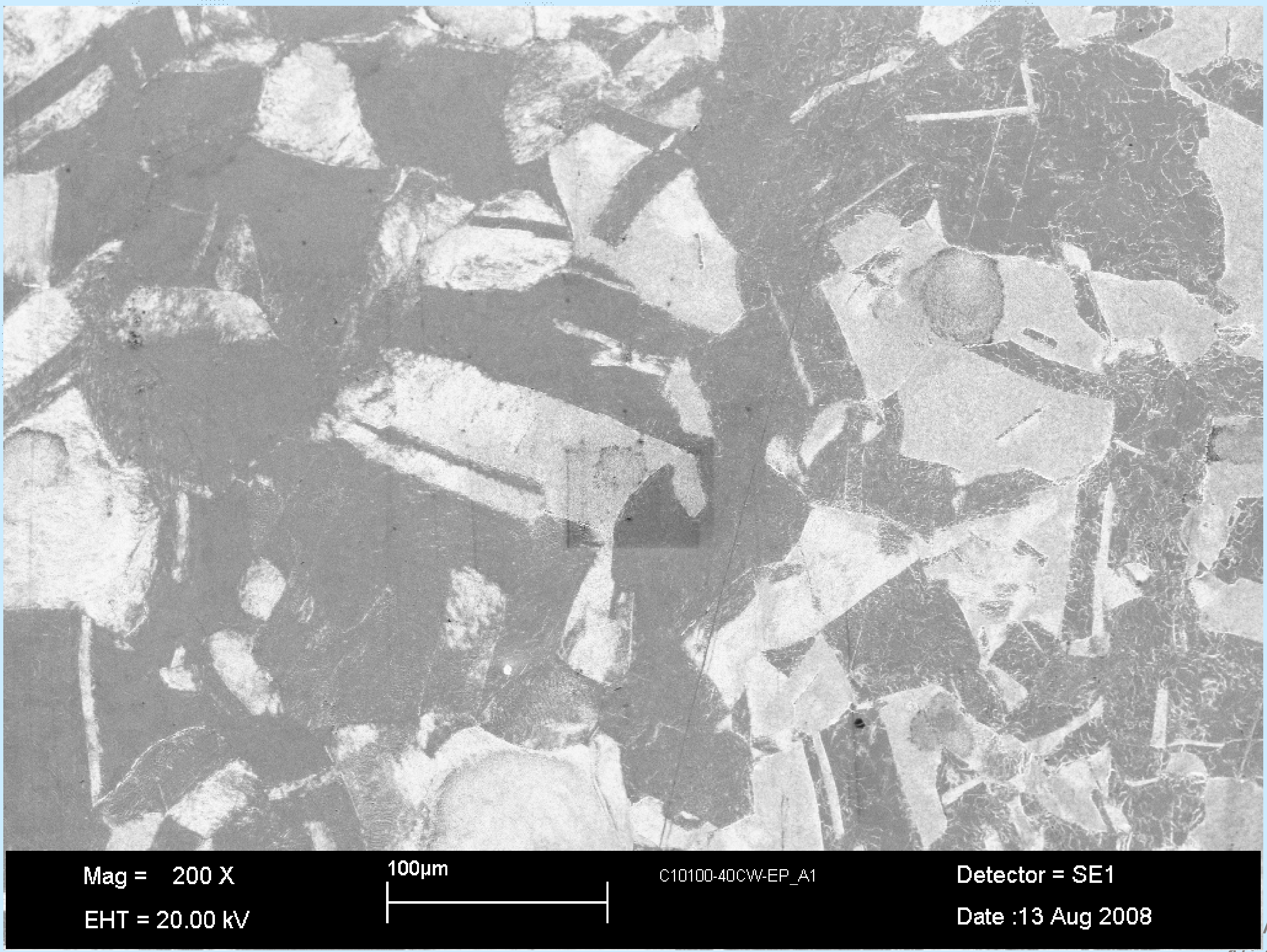
C10100_2h@1000_EP_Probe5_D4



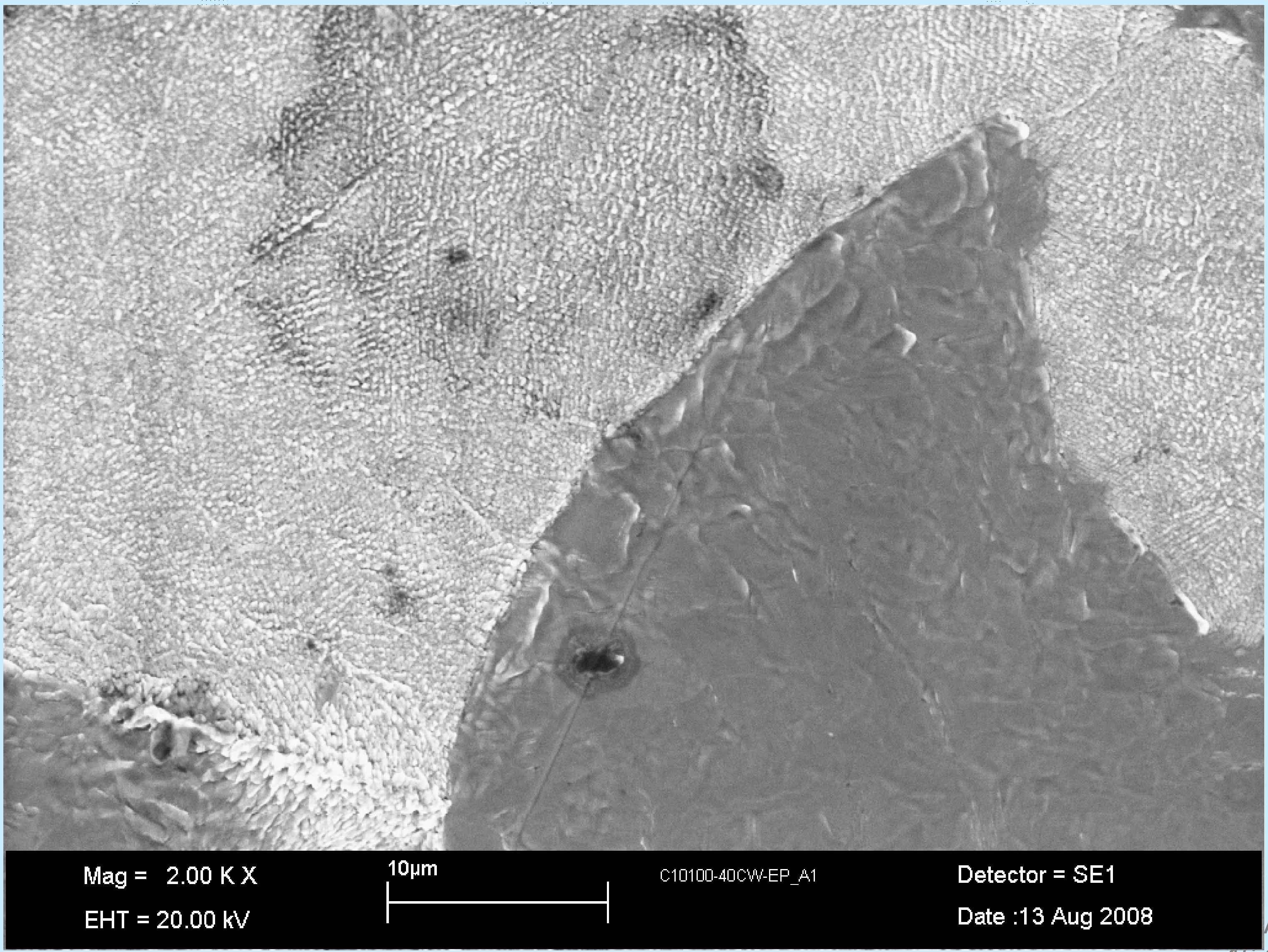
C10100-40%CW-EP_A1



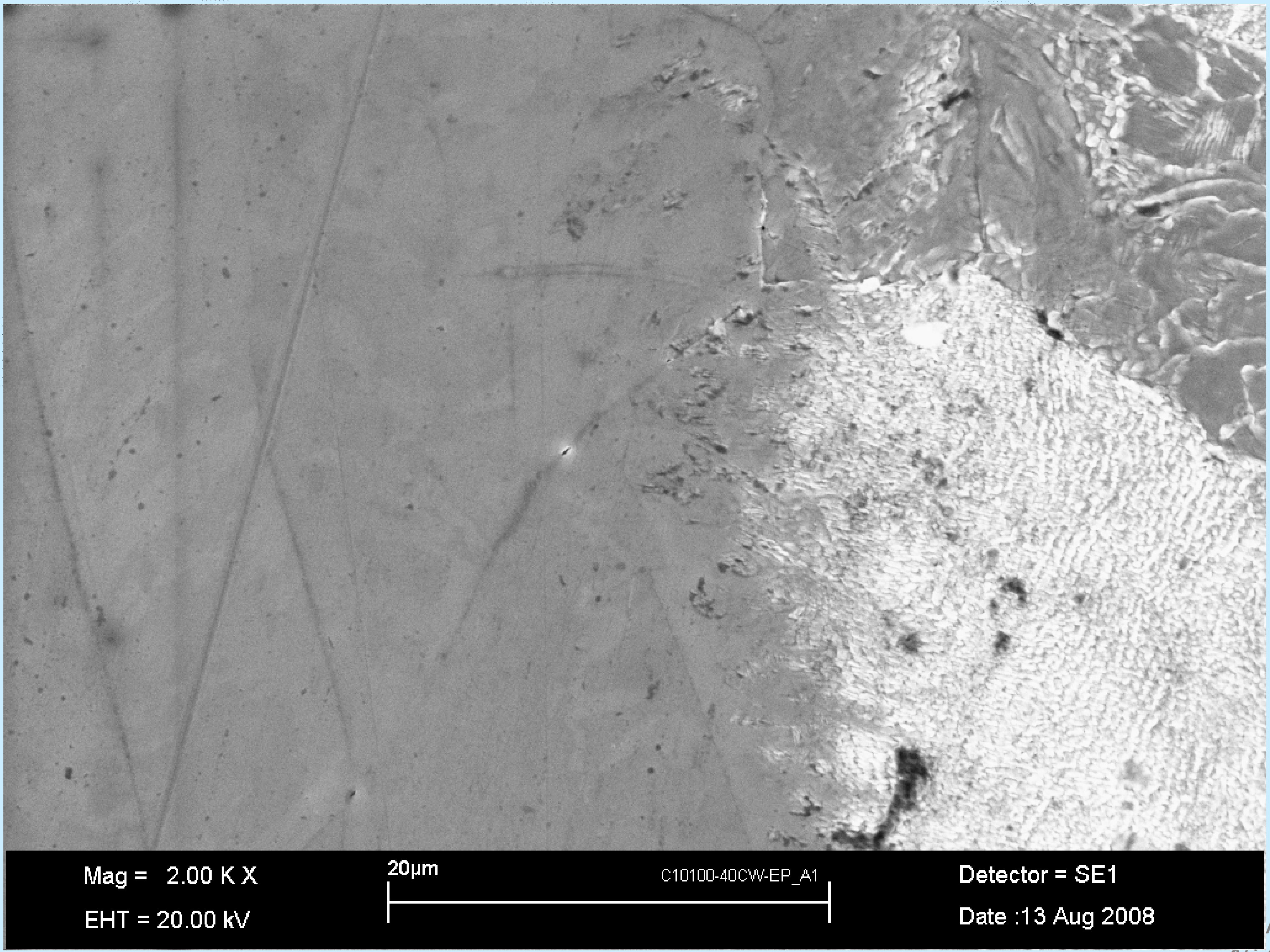
C10100-40%CW-EP



C10100-40%CW-EP



C10100-40%CW-EP Virgin Surface

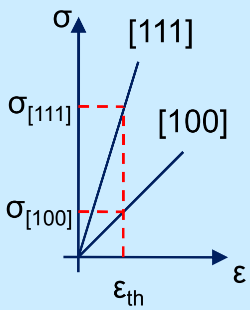


Discussion EBSD-Observation Results

- [1 1 1] (blue) direction high developed and [1 0 0] (red) direction less developed fatigue features

-> evidence of influence of orientation on fatigue behaviour

-> One reason: Isotropic thermal expansion causes due to anisotropic module different stresses ($\sigma_{[111]}/\sigma_{[100]}$ = 2.3 !!!)



=> fatigue samples of different orientation

=> quantify surface fatigue damage <-> orientation

- Orientation <–> fatigue behaviour same trend in RF-fatigue as well as in Laser fatigue!

-> additional hint for equality of fatigue techniques

=> proof the same behaviour for the ultrasound-device

- [1 1 1] (blue) direction dominating after extreme heat treatment

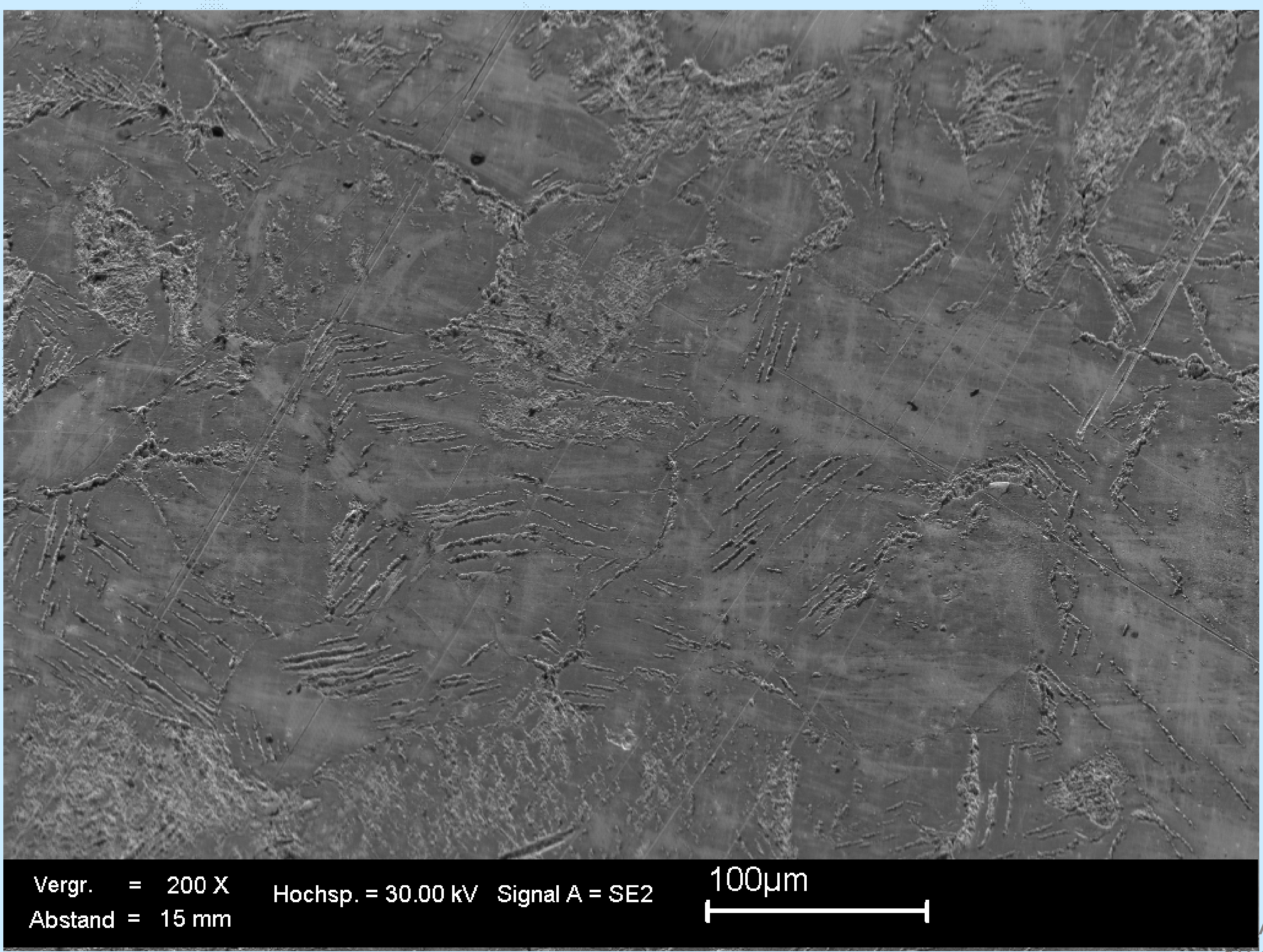
-> preferred and less preferred orientations

-> texture already exists in raw material

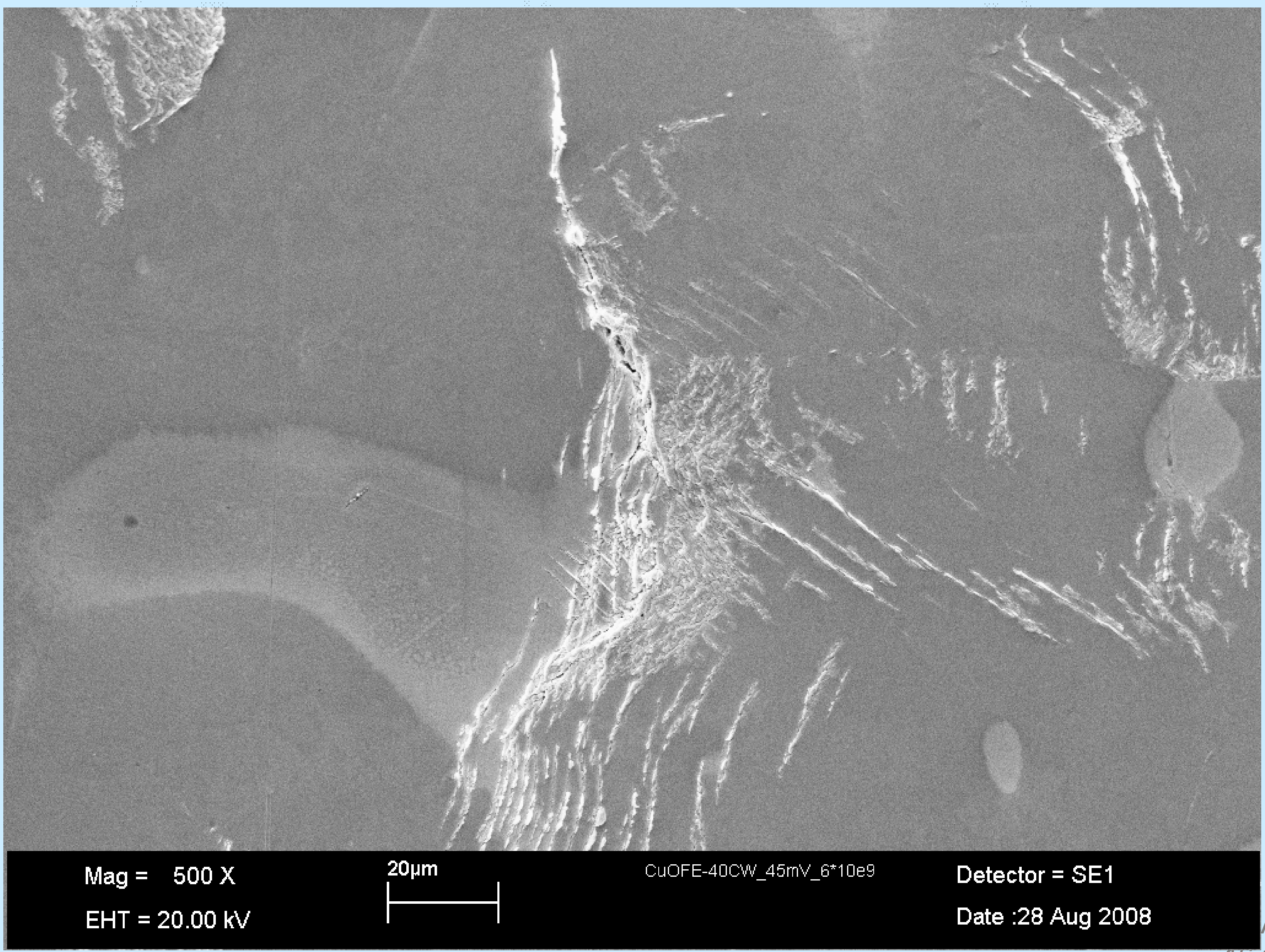
-> further texture development through thermal treatment

=> Need for further observation upon the influence of textures

Recent Picture: SLAC sample



Recent Picture: Ultra sound sample



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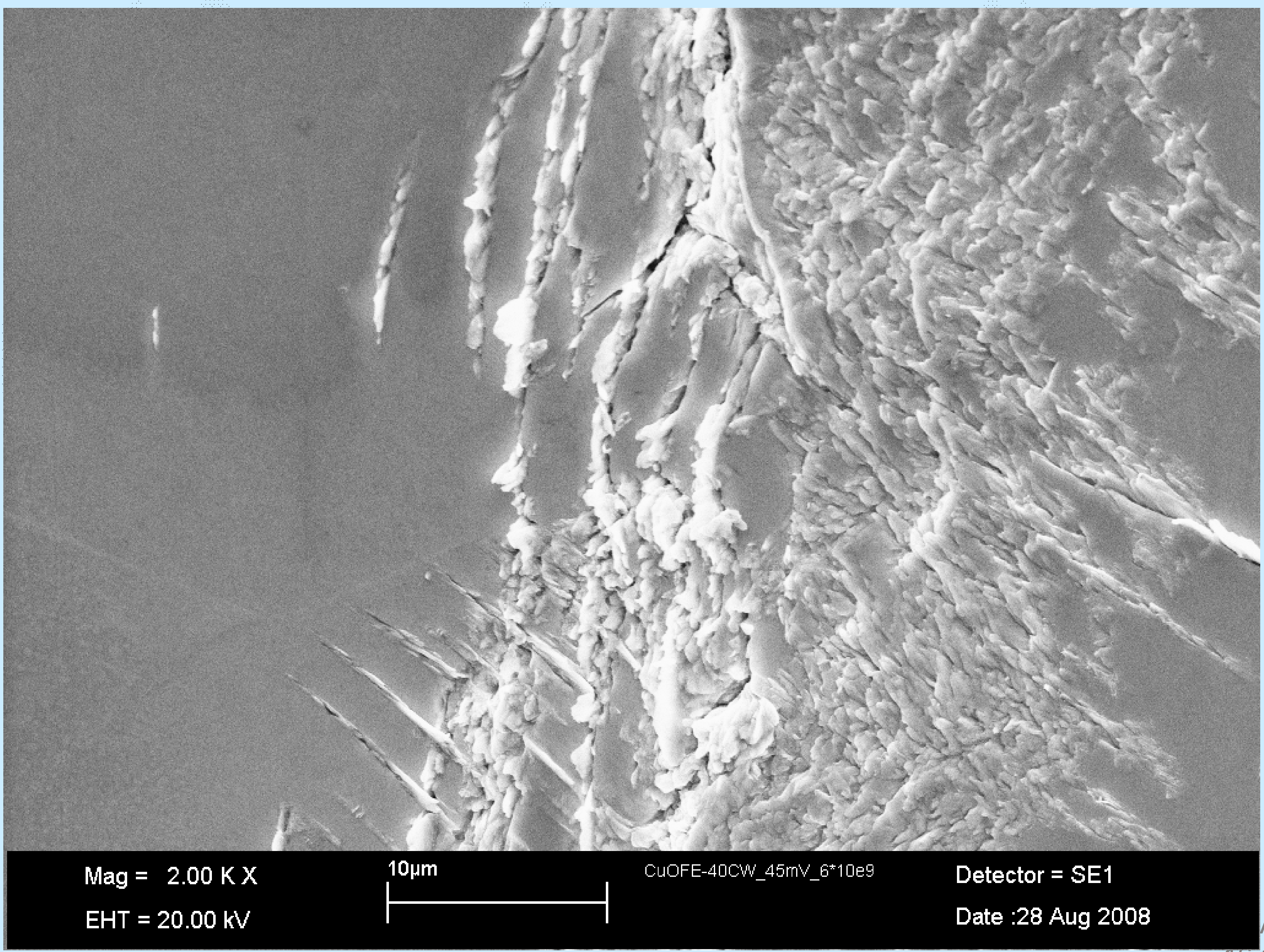


Thanks for the attention!

Observation techniques used: EBSD

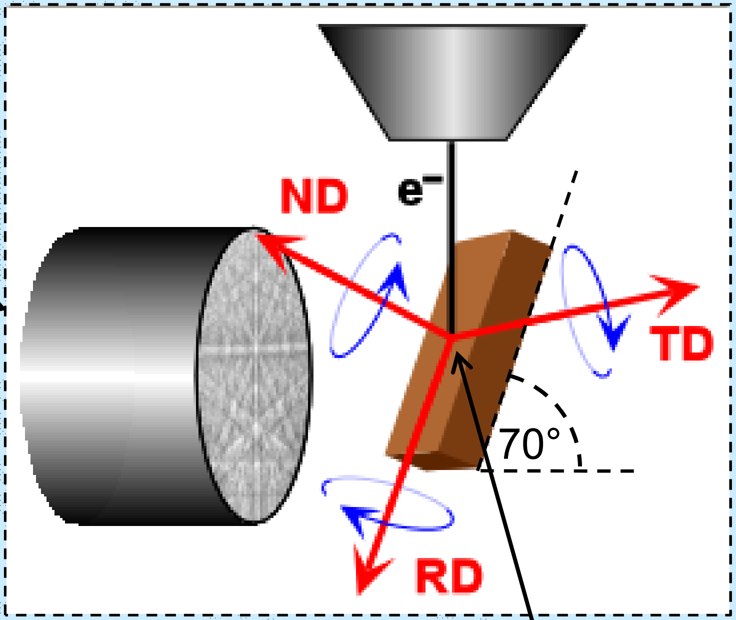
Electron Back Scattered Diffraction

SEM: Leo 1530 VP

EBSD unit: EDAX TSL

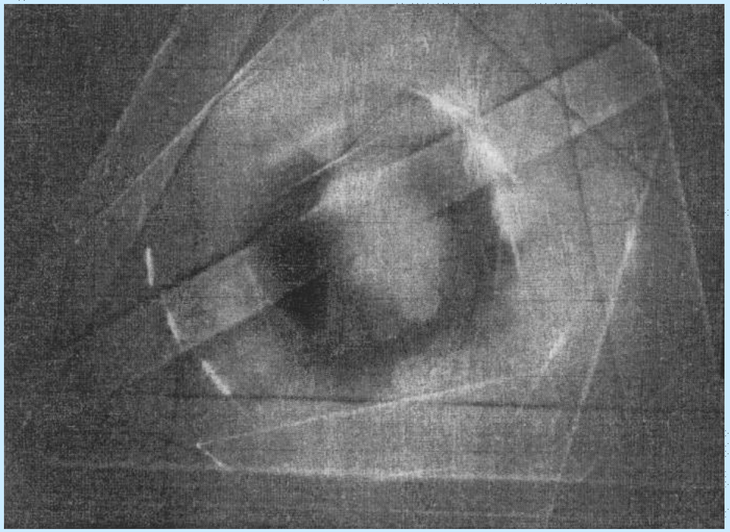
Electron Microscope column

Phosphoric screen and digital camera



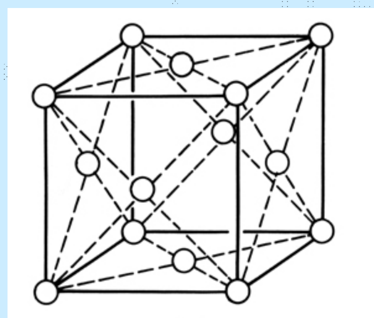
"Bragg Reflection"

Kikuchi pattern

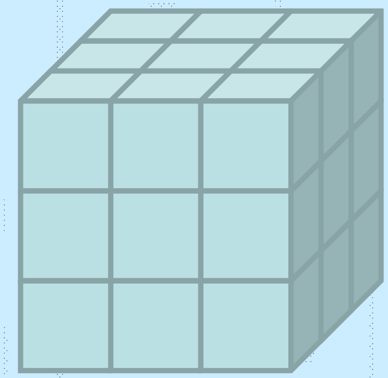


Basics for EBSD: Crystallography

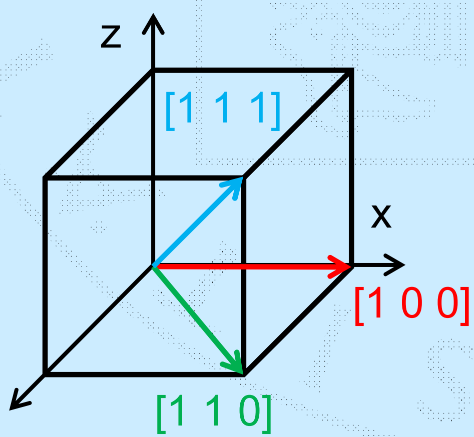
A cube with atoms on is corners and his faces (so called face centered cubic FCC)



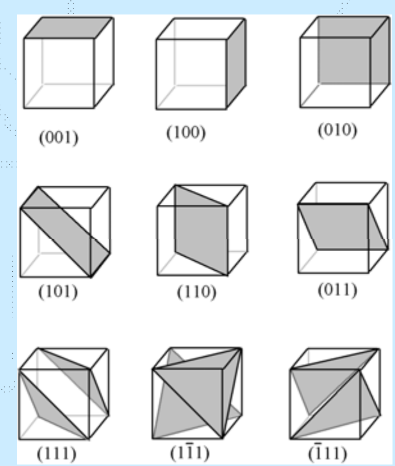
Plenty of these elementary cells



Crystallographic description of directions:



Crystallographic description of planes: Normal of planes!

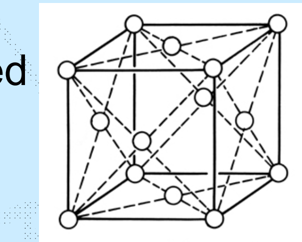


Basics for EBSD: Bragg Reflection

I_0: intensity of initial ray

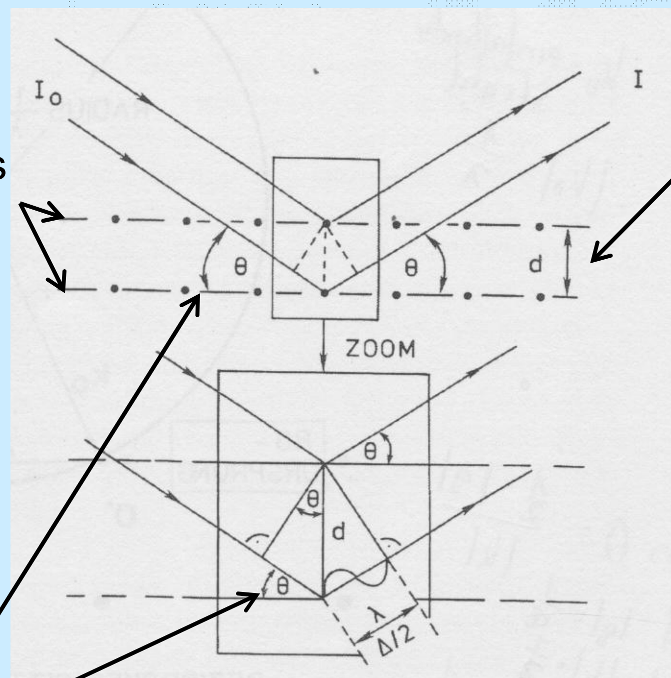
I: intensity of outgoing ray

Face centered cubic (FCC) crystal



Atomic planes of crystal

d: distance between crystal planes



Each Atom radiates as a “point like” source

(“**Elastic** Scatter”)

Only constructive interference if path difference equals wavelength! => intensity maximum.

$n \cdot \lambda = 2 \cdot d \cdot \sin\Theta$

Bragg equation

Θ: “Bragg angle”

λ: Wavelength

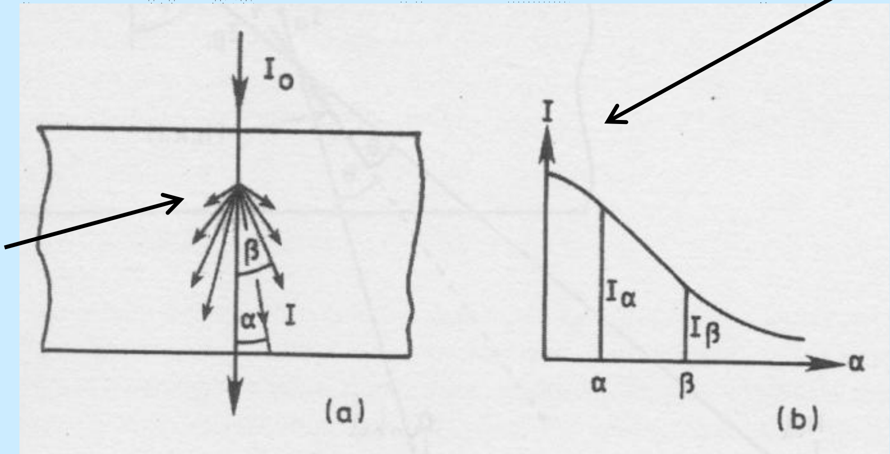
=> For known n, λ and θ one can calculate d

Basics for EBSD: Inelastic scatter in thick crystal

I_0: intensity of initial ray

Maximum intensity in initial direction (α = 0°, decreasing with increasing angle

Thick crystal: => more and more **inelastic** scatter in every direction!



α, β: diffraction angles

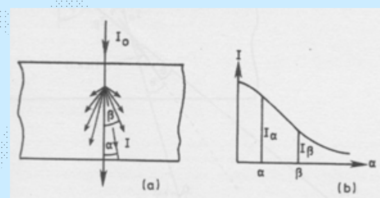
I: intensity of outgoing ray

Changes in λ due to energy dissipation negligible

=> new I_0 ray with initial λ but different direction!

Basics for EBSD: Kikuchi line construction

Facts:



1) $I_\alpha > I_\beta$

2) Every crystal plane produces interference **line** (perpendicular to drawing plane) due to ray cones of I_α and I_β

3) crystal plane (almost perpendicular in the crystal) gets hit from both sides

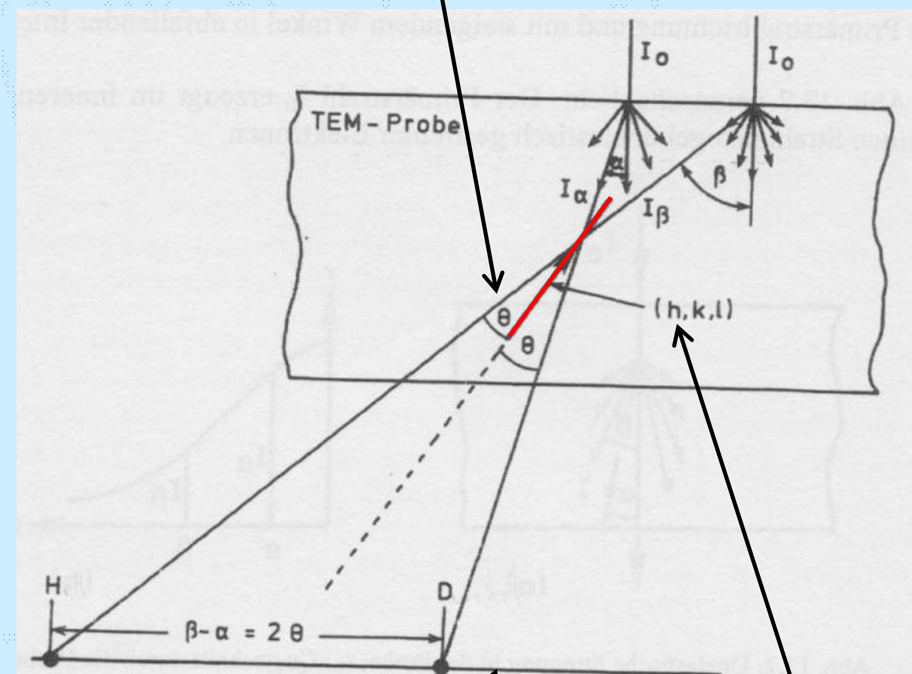
$$f = \frac{\text{scattered intensity}}{\text{primary intensity}}$$

In H: Background + $\underbrace{(f \cdot I_\alpha - f \cdot I_\beta)}_{> 0}$ => brighter

In D: Background + $\underbrace{(f \cdot I_\beta - f \cdot I_\alpha)}_{< 0}$ => darker

Θ: "Bragg angle"

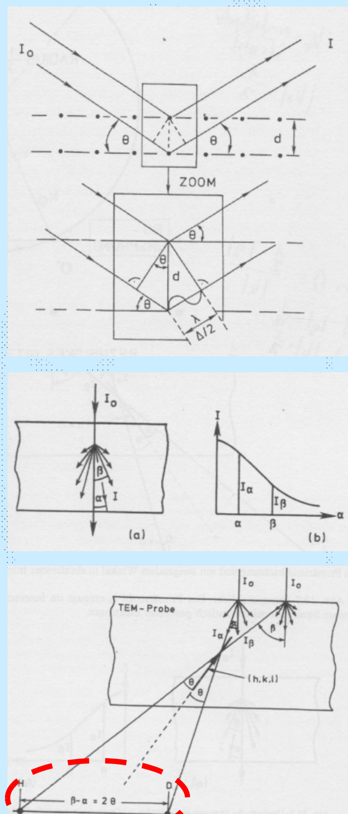
I_α ; I_β: intensities of new initial rays

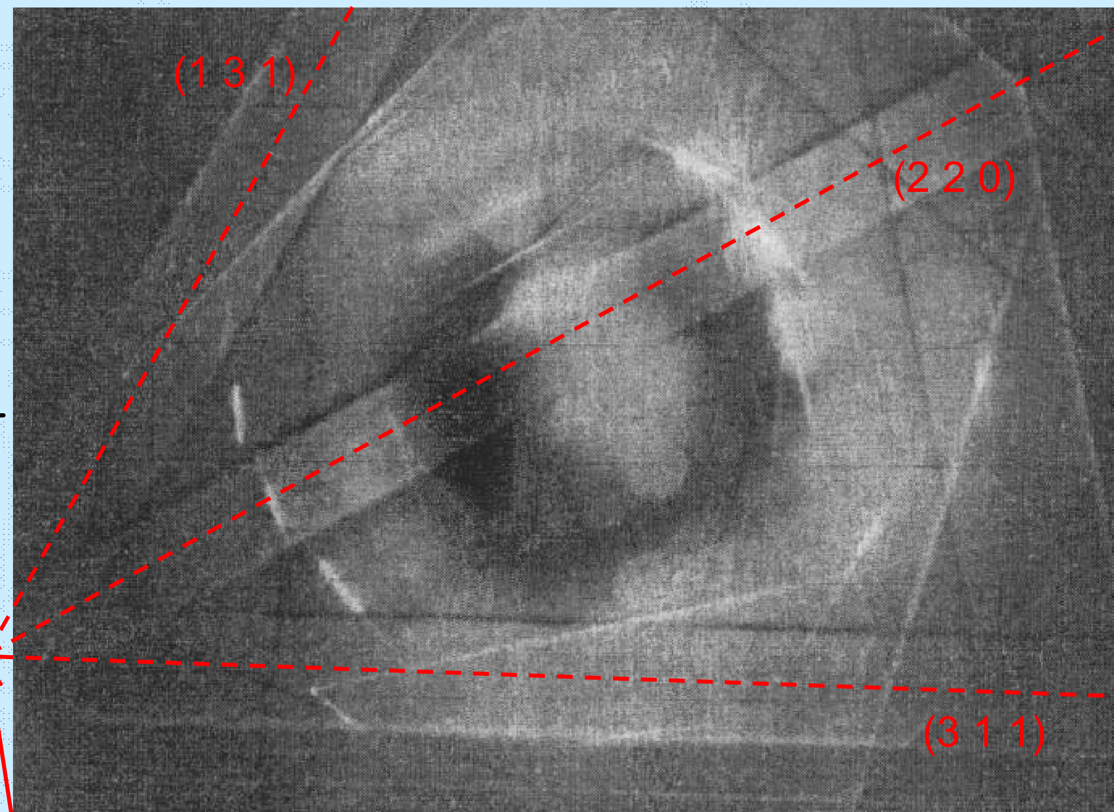


Screen

(h,k,l): crystal plane

Basics for EBSD: Kikuchi line

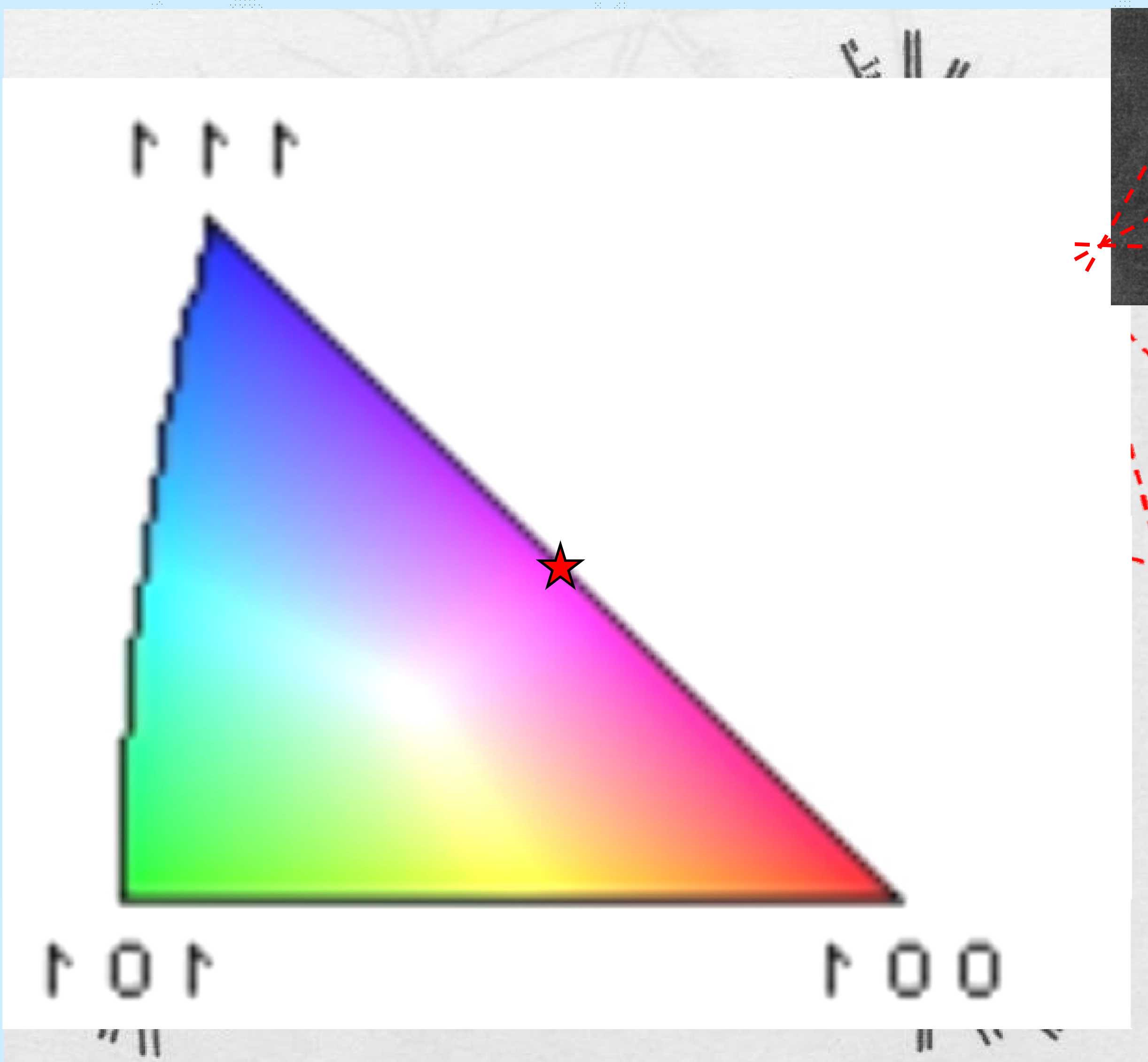


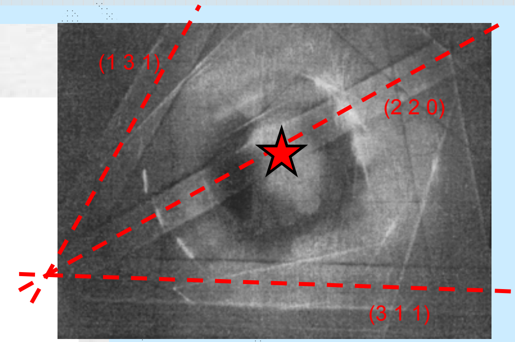


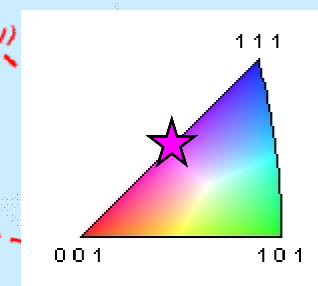
Zone axis [-1 1 -2]

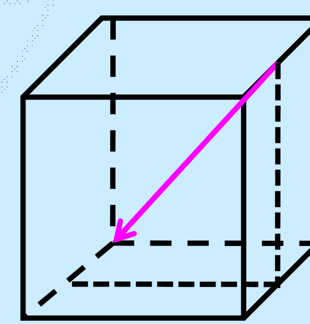
Distance => Bragg equation
=> planes space d!
=> plane identification

Basics for EBSD: Kikuchi line indexing









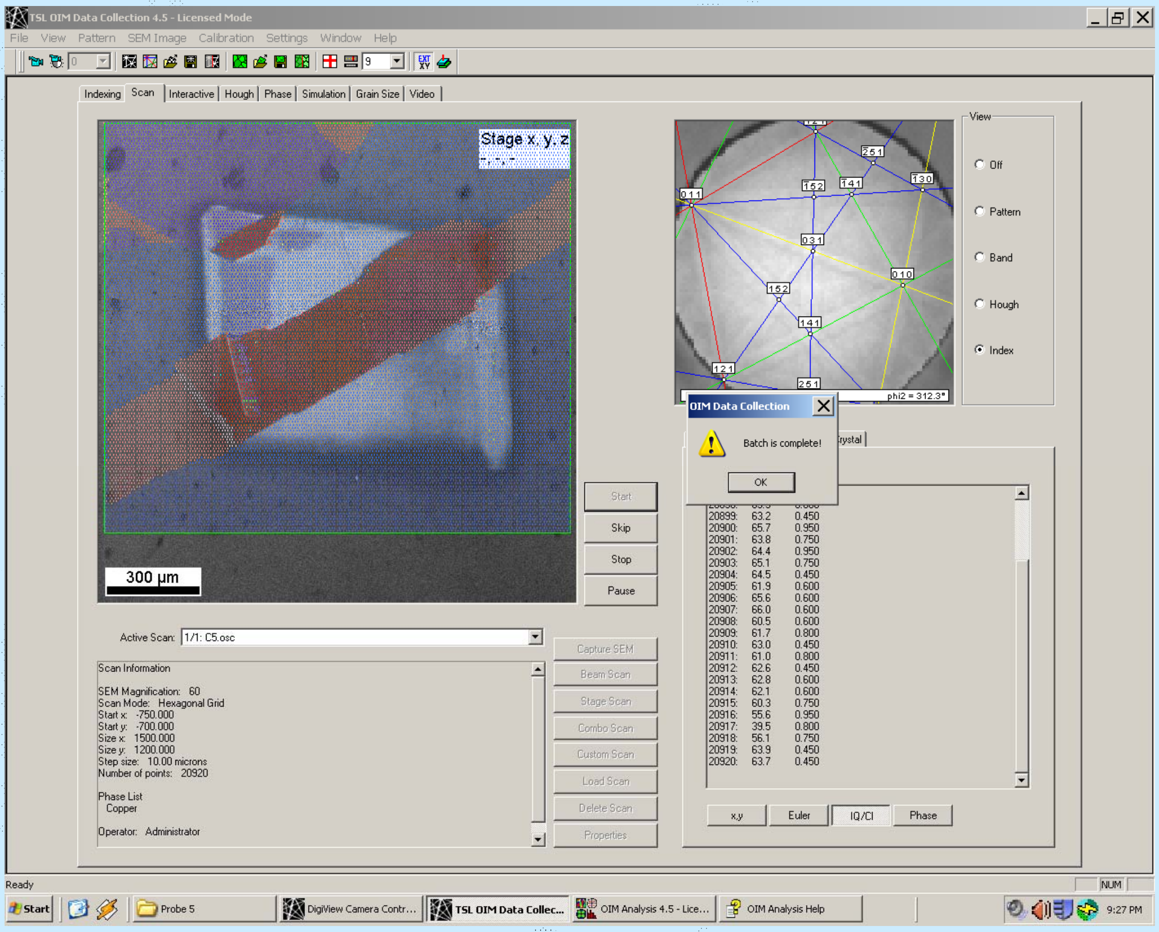
Basics for EBSD: EBSD-Software

Scan:

Each point one Kikuchi pattern

-> each point complete orientation information

=> Mapping of orientations



Scan rate: 75 points/s

Ordinary used for:

- Texture analysis
- Orientation of samples (like X-Ray diffraction but faster)
- Identification of different phases (like TEM but lower resolution/magnification)
- Possibility to connect with quantitative EDX scans