Using ECCI and EBSD to study the microstructure of WC-Co hardmetals

GN-MEBA 2020

https://nano.oxinst.com/library/fr/blog/

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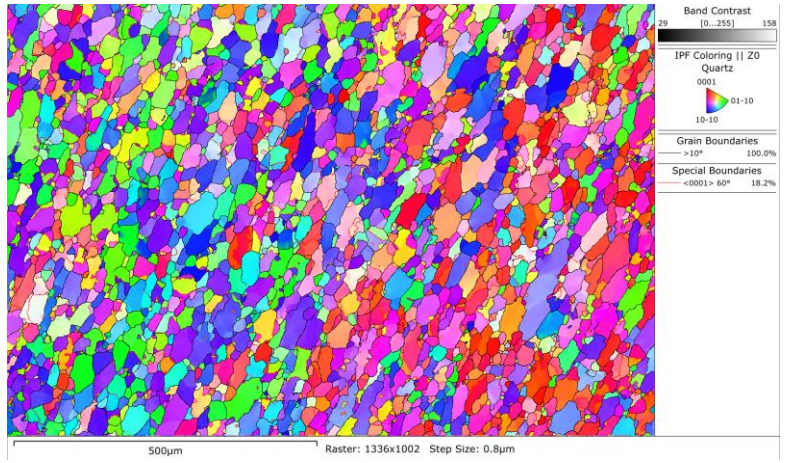
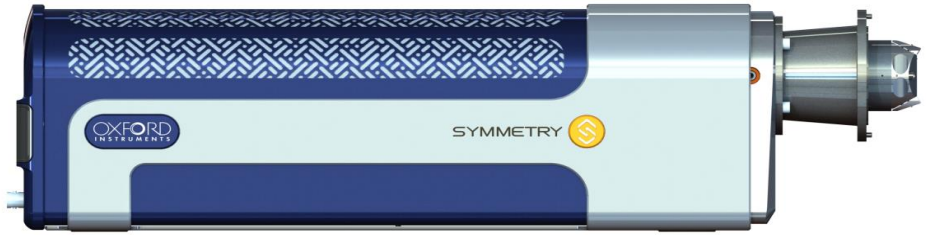
Highlights

- Symmetry CMOS detector and AZtecCrystal
- Introduction to EBSD and ECCI analysis
- What is tungsten carbide
- Why use EBSD and ECCI to study WC-Co?
  1. Subgrains in the WC parent grains
  2. Vertical dislocation density and crystal orientation?
  3. Identify dislocation type in the WC grains

Guide the development of new materials.





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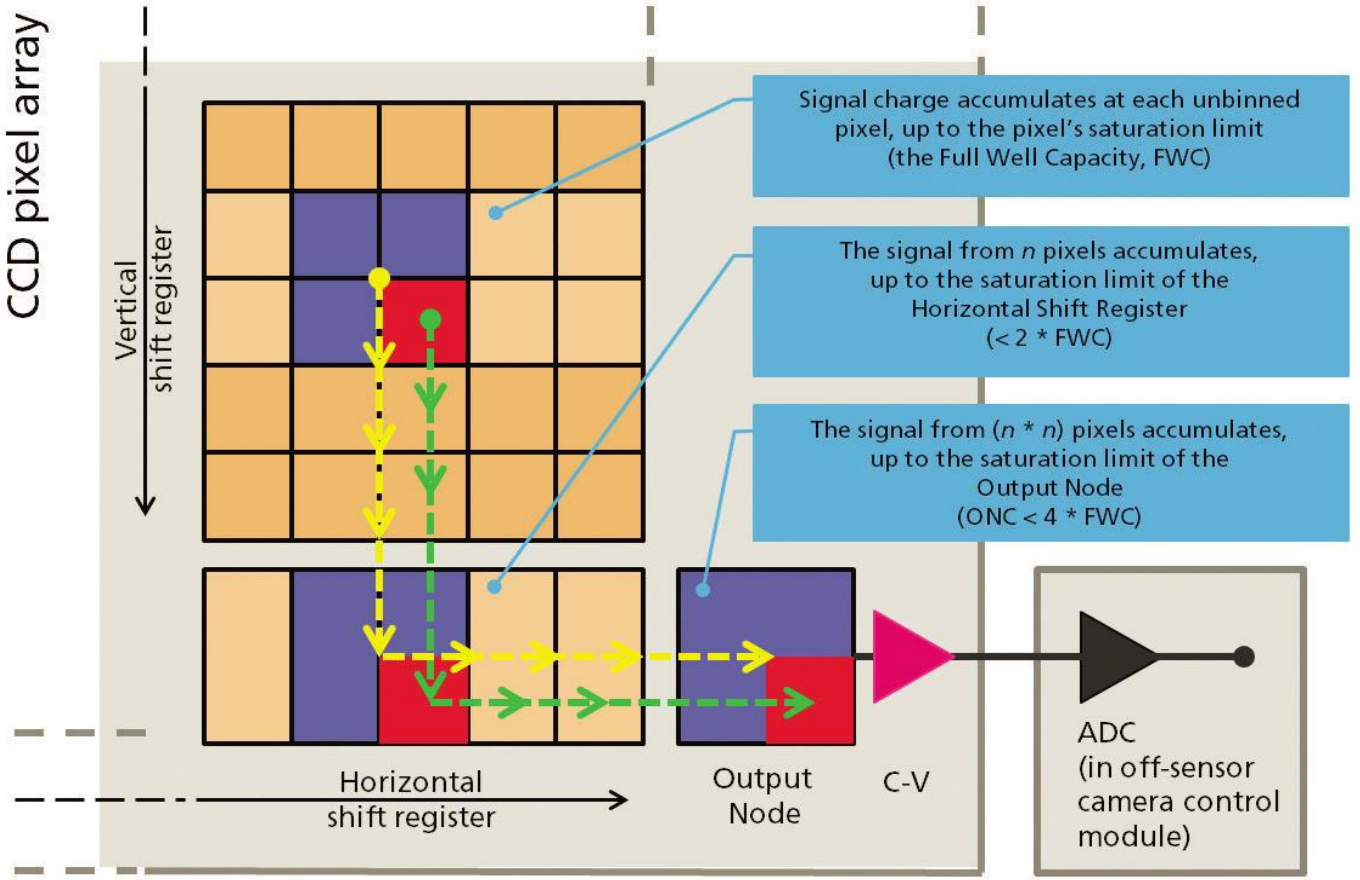
Symmetry CMOS EBSD

- Symmetry is a completely new design of EBSD detector
- Symmetry is the first (and only) truly 'all in one' detector solution for EBSD
  - Highest speed maximum acquisition speed (>4500pps) more the double that of previous generation of EBSD detectors
  - Highest sensitivity fast analysis at lower beam current; fast analysis at lower kV
  - Highest resolution sharpest EBSPs
- Symmetry offers the highest real time indexing speeds and the highest sensitivity and the highest resolution patterns all in a single detector
- Symmetry uses next generation CMOS image sensor technology coupled with high efficiency, high resolution optics to deliver break through performance across the entire EBSD application range

CMOS vs CCD

- In CCDs, charge is transferred, in a linear fashion from one pixel to another to a single readout node on the sensor
- The slowest element in the process chain is the analogue-to-digital conversion (ADC)
- The ADC is a 'bottleneck'
- It is not integrated onto the sensor, requiring a slow and noisy analogue connection between the sensor and a separate camera control module

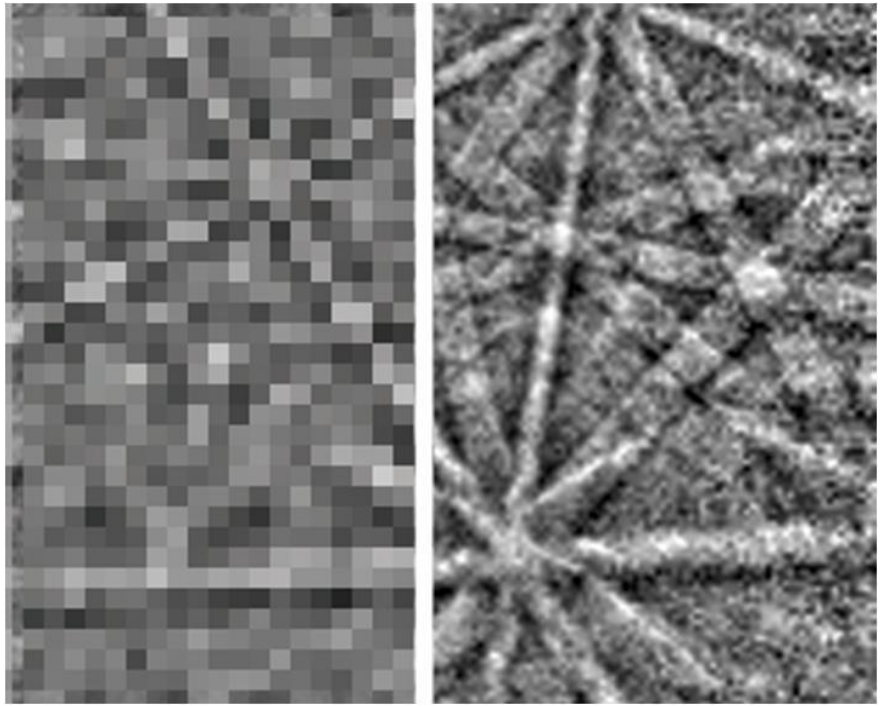


CMOS vs CCD

Symmetry does not need high pixel binning to achieve high speeds

- better quality data at twice the speed

CCD-based
1500 pps
40 x 30 pixels



Symmetry
> 4500 pps
156 x 88 pixels

Symmetry S2

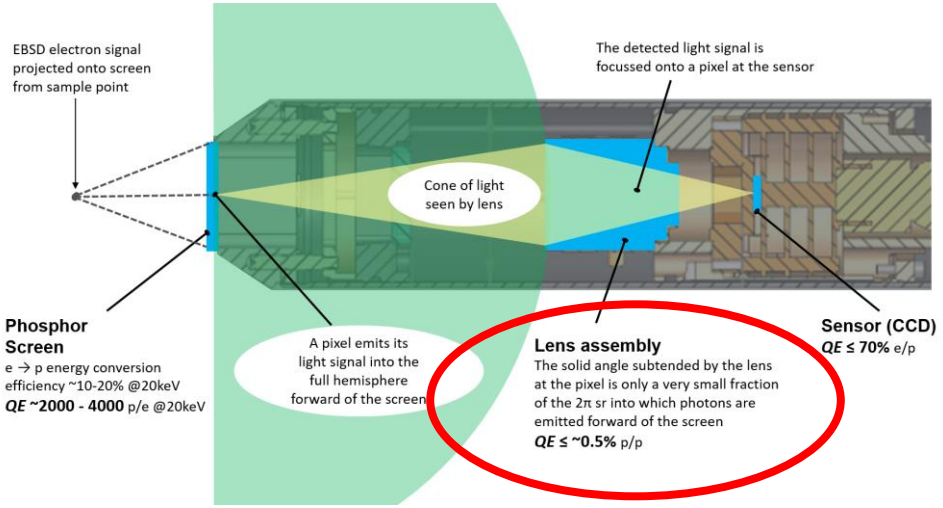
- The Symmetry S2 is a new, higher performance version of the market-leading Symmetry detector
- The S2 keeps the innovative design benefits of Symmetry:
  - Fibre optic lens giving optimum sensitivity
  - Full megapixel resolution – ideal for HR EBSD studies
  - Software controlled Elevation control
  - Proximity sensors for collision avoidance
  - Integrated forescatter detector system
- However, the S2 adds an increased maximum speed mode, capable of analysing > 4500 pps

Resolution	Speed
1244 x 1024	> 240 pps
622 x 512	> 800 pps
156 x 128	> 3000 pps
156 x 88	> 4500 pps

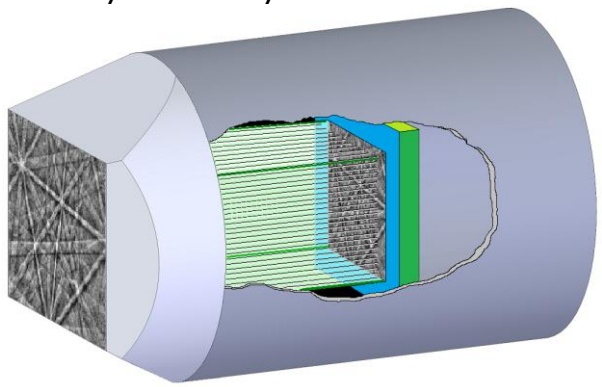
1. Fibre Optics Sensitivity

>99% of the light does not get to the sensor!!

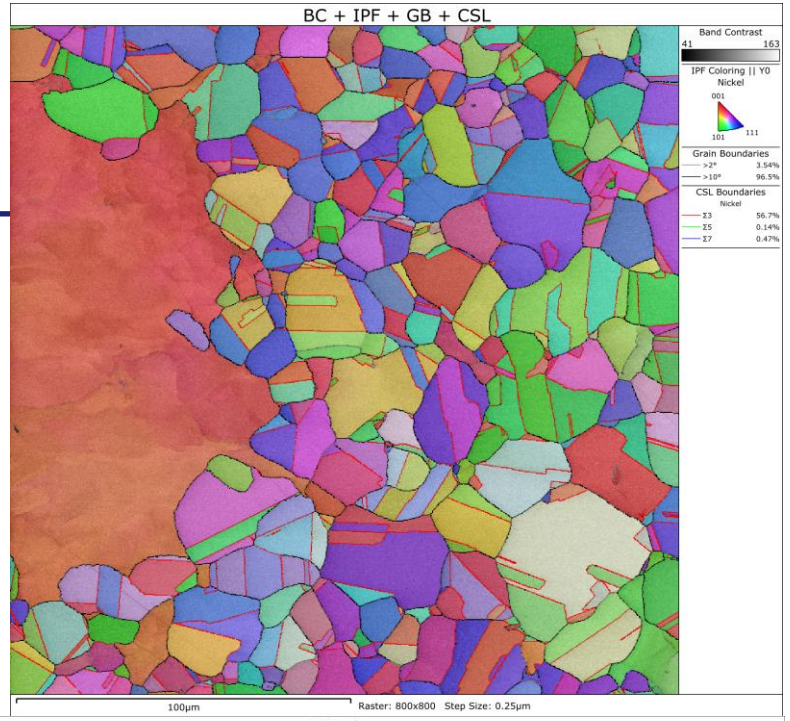
Traditional Lens Coupled EBSD Detector



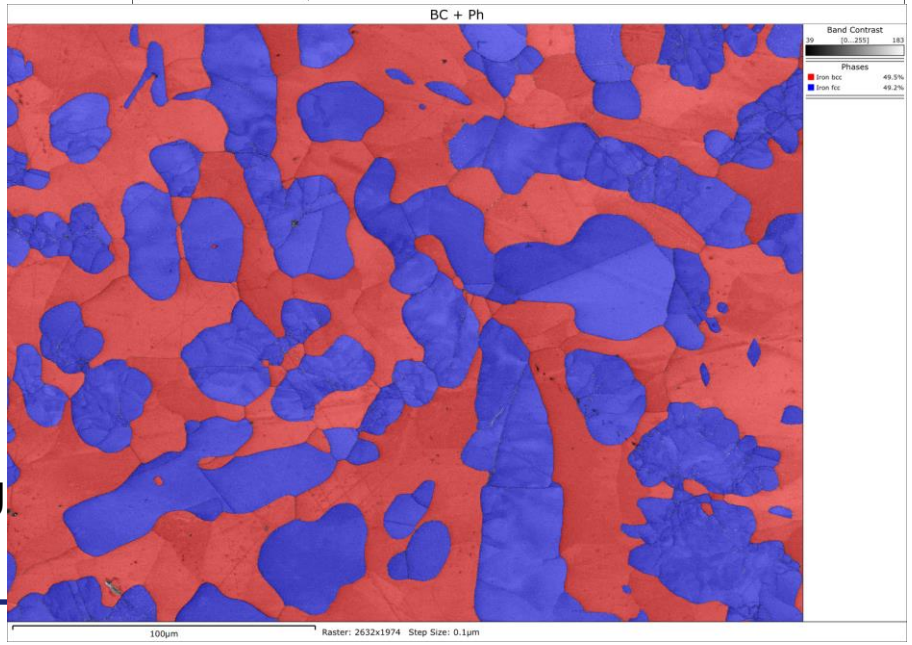
Symmetry S2 EBSD Detector



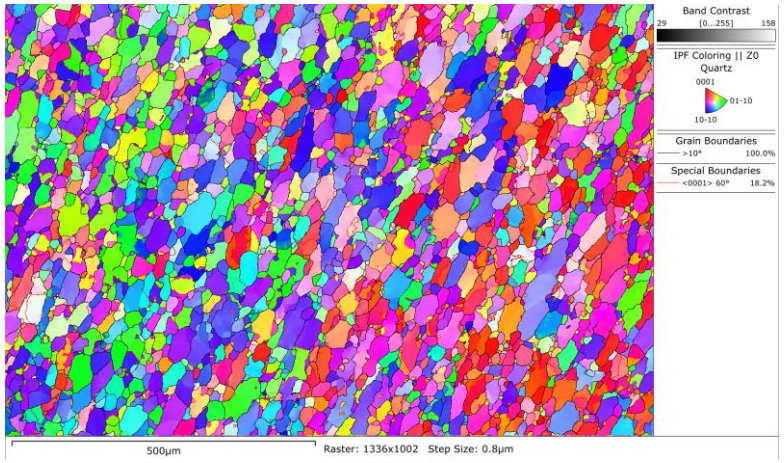
Ni superalloy
3020 pps
99% indexing
4.09 nA, 20 kV



Duplex steel
4728 pps
98.7% indexing
10 nA, 20 kV

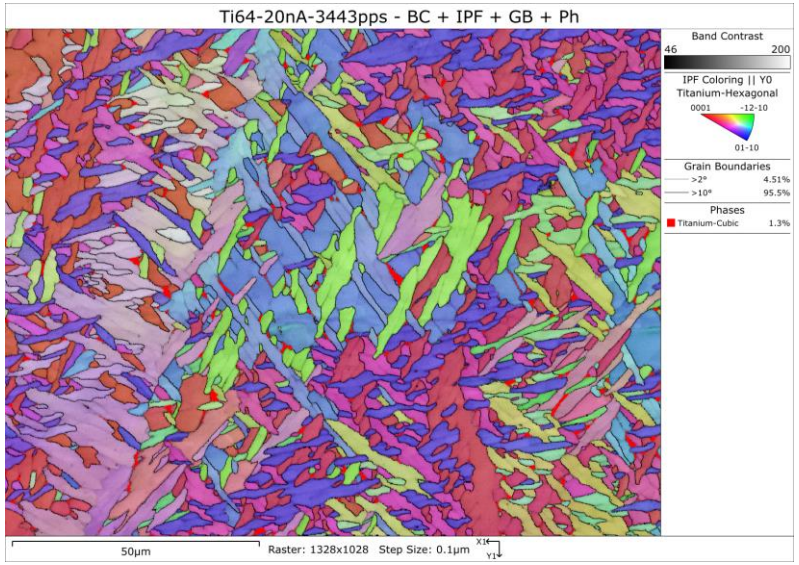


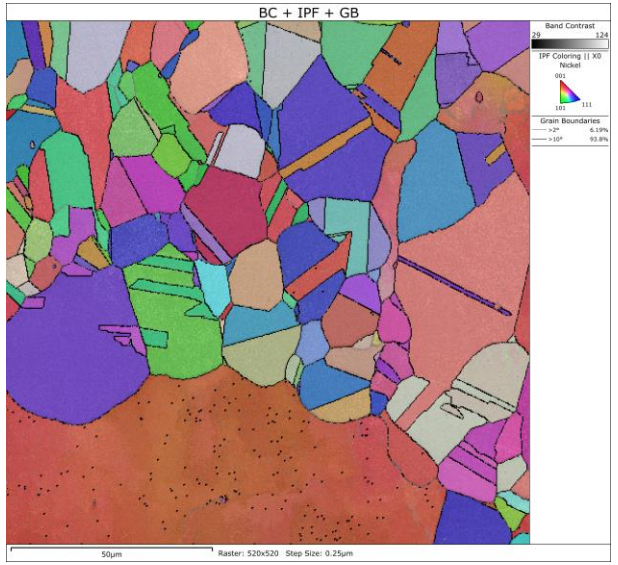
Fibre Optics Sensitivity



Quartz rock
1872 pps
~95% indexing
29 nA, 20kV

2 phase Ti64 alloy
3443 pps
98% indexing
20.4 nA, 20 kV





Ni superalloy
220 pps
99% indexing
4.97 nA, 5kV

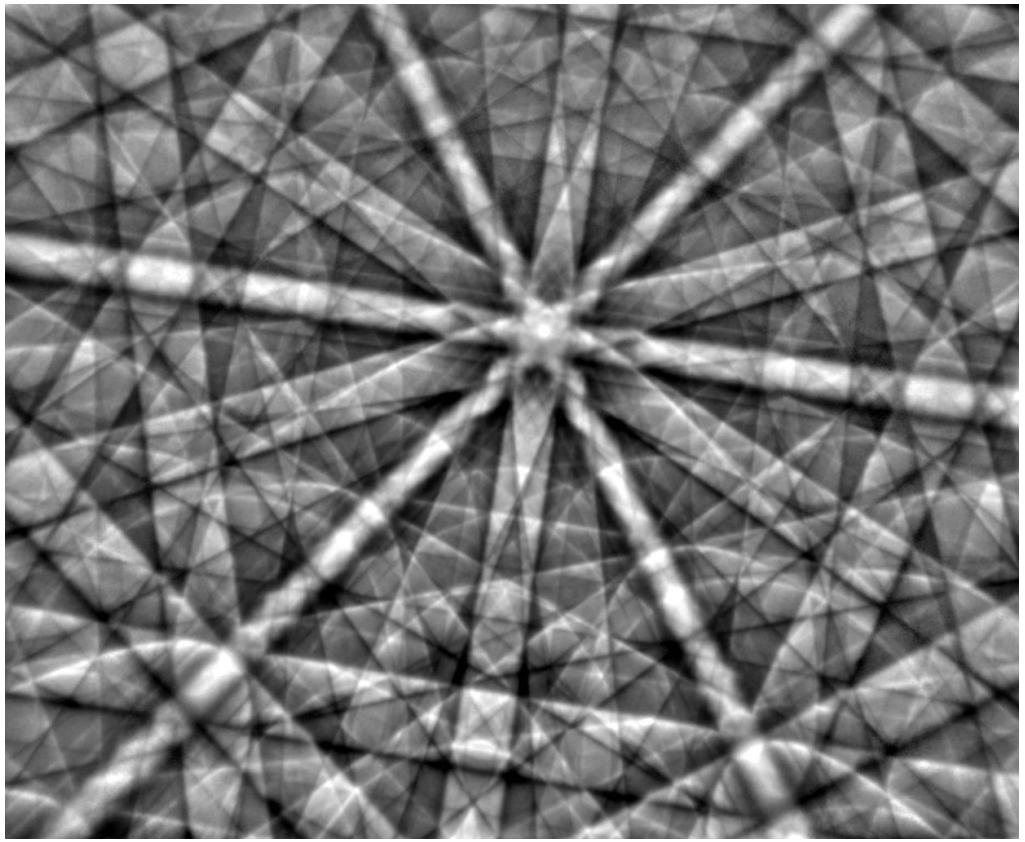
Speed comparison with lens-coupled CMOS

Performance at 20nA…

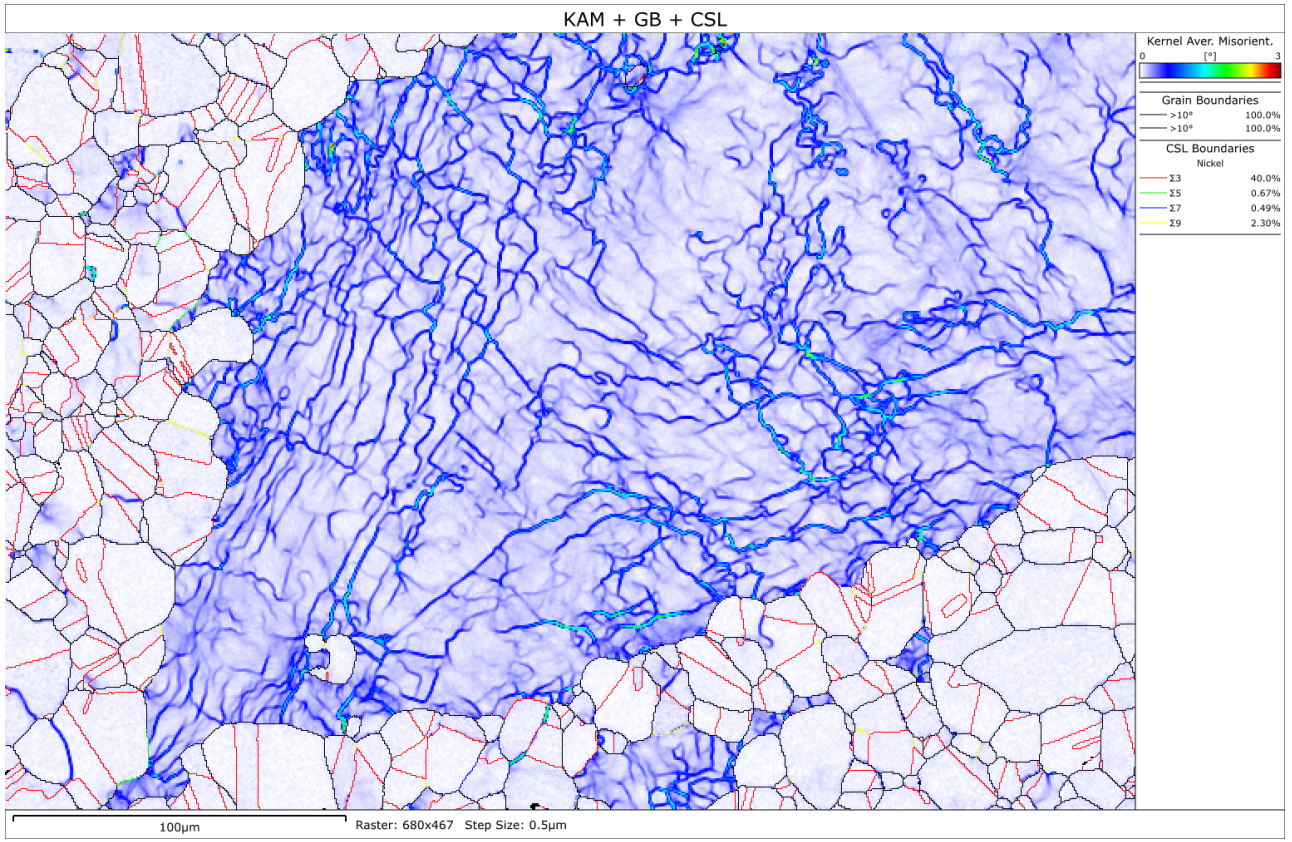
Material	Symmetry S2 Typical Speed @ 20nA	Lens Coupled CMOS Typical Speed @ 20nA	The Symmetry S2 is faster…
Simple Steels	>4500pps	>3400pps	1.3x
Light metals	>3000pps	>1500pps	2x
Deformed steels	>1500pps	>750pps	2x
Simple Geo	>1000pps	>500pps	2x
Complex Geo	>500pps	>200pps	2.5x

Symmetry S2's fibre-optics sensitivity ensures higher speeds for all sample types under standard operating conditions

2. High resolution EBSPs



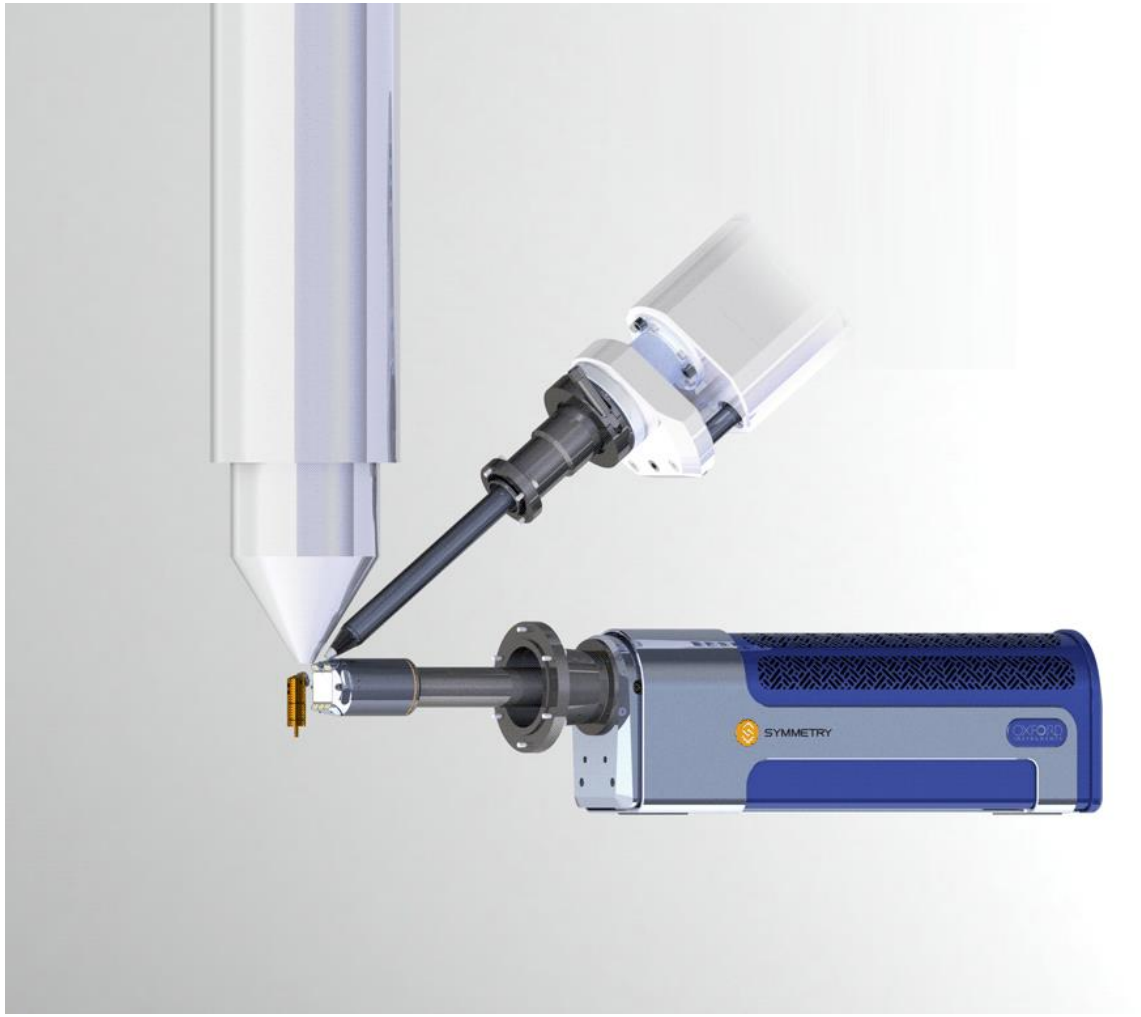
Full 1244 x 1024 pixel resolution EBSP from Ni



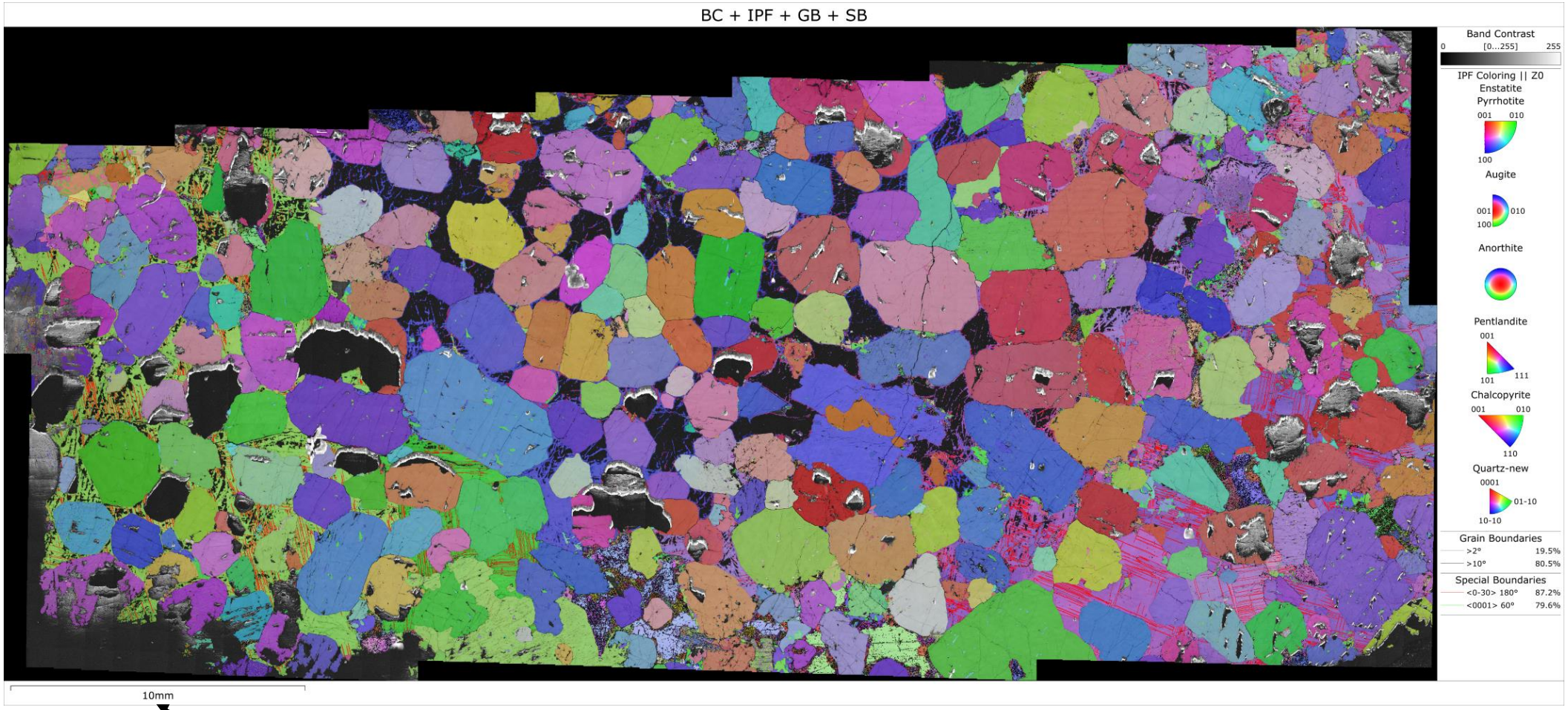
Kernel Average Misorientation map showing dislocation lines in a deformed Ni grain, collected using Refined Accuracy indexing

3. Detector Tilting

- A super-convenient feature
- Unique to Symmetry S2
- Ensures detector always in optimum position
- Ideal for:
  - Large samples (long WD)
  - Low accelerating voltages
  - TKD analyses (short WD)
- Controlled from SW Interface
- Autocalibration – instant indexing



Detector Tilting – enabling extra-large sample analysis



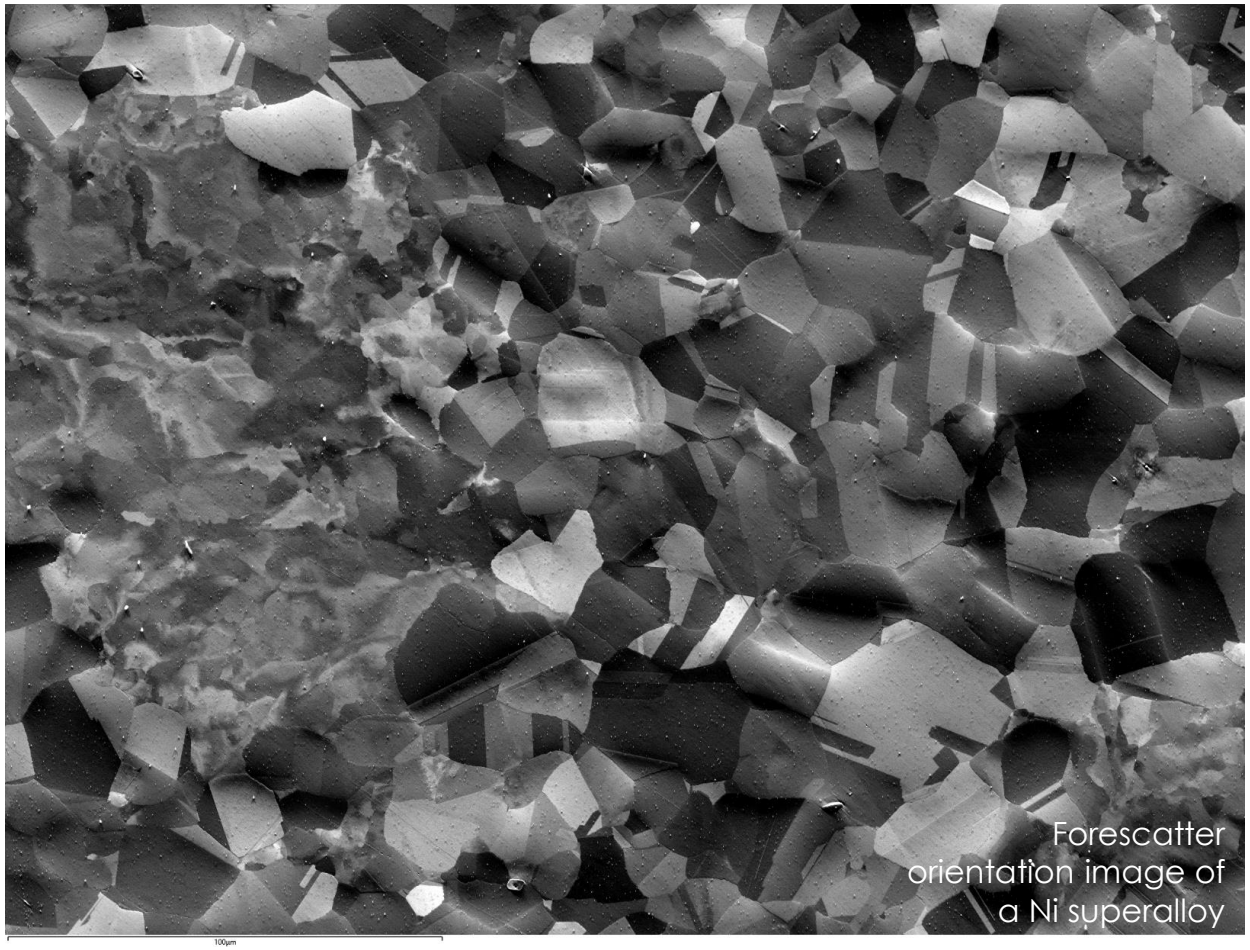
1 cm!

Orientation map of a large geological thin section (pyroxenite from the Bushveld Layered Intrusion – courtesy of Alexandra Stavropoulou)

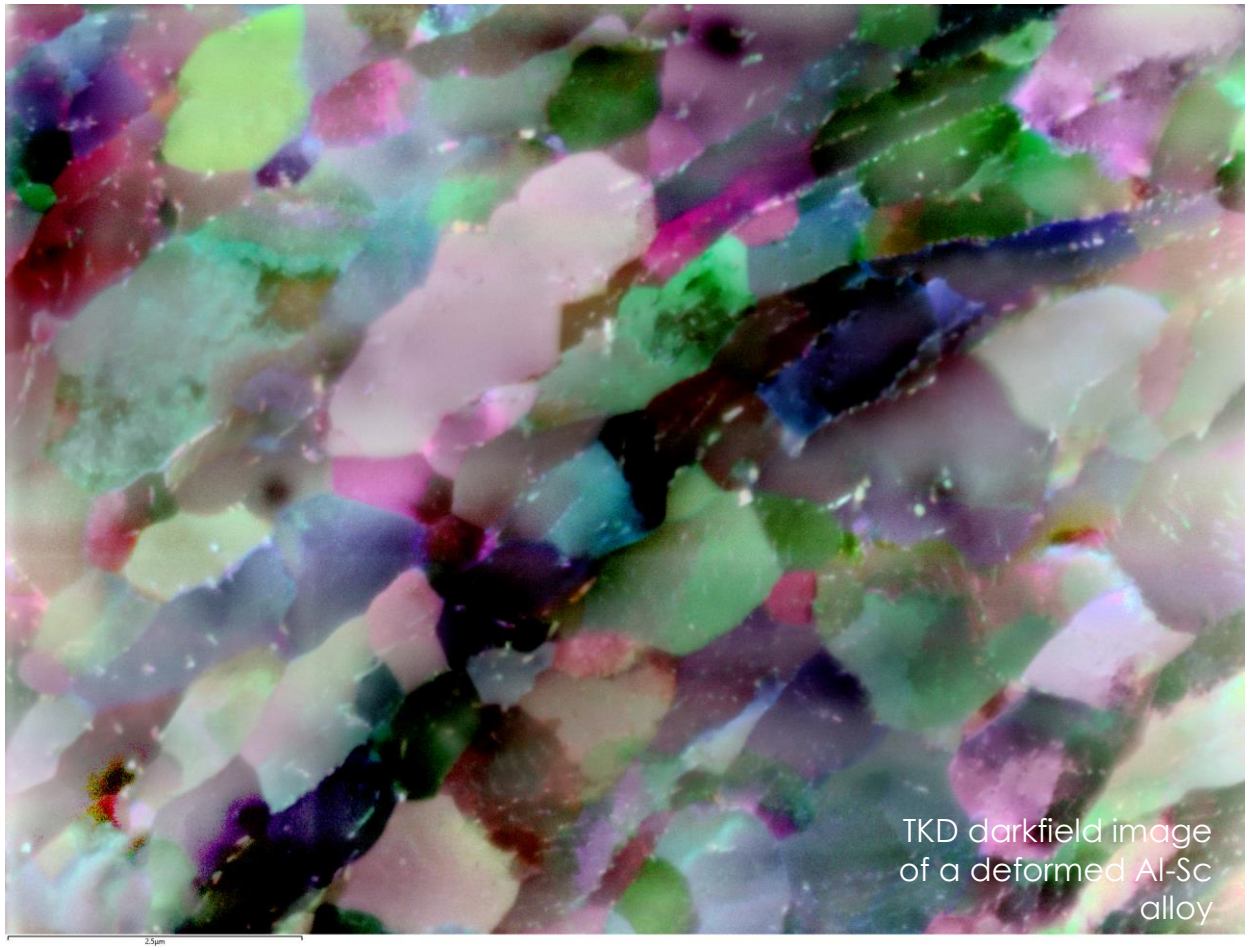
4. Proximity Sensor

Technology is patent pending

5. Integrated Forescatter diodes-Example FSD images



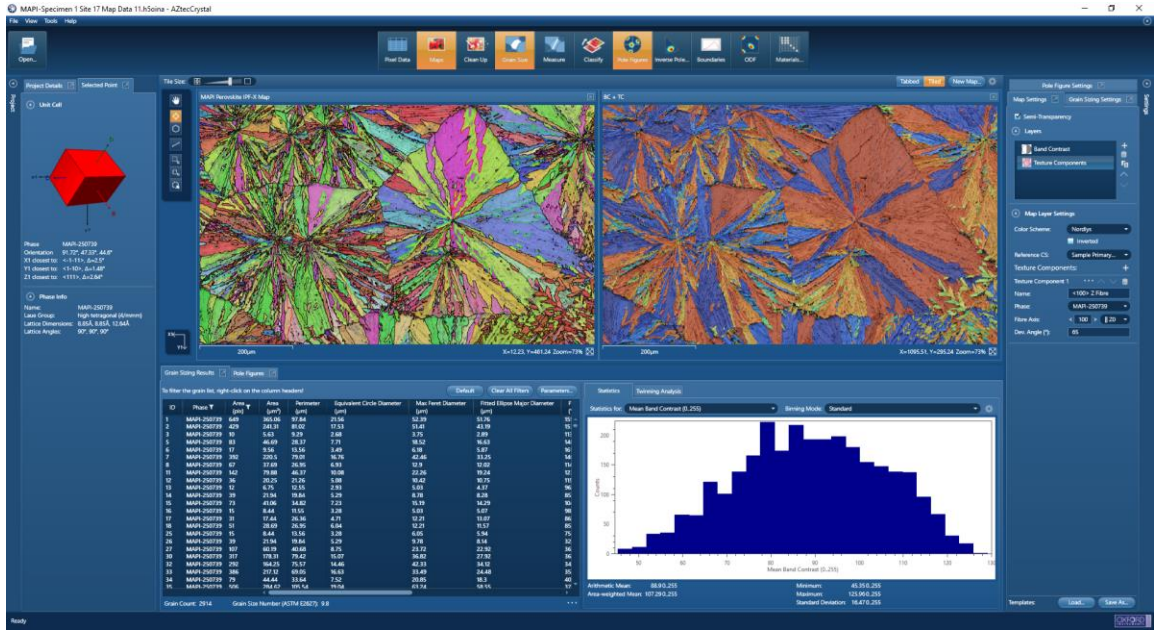
Forescatter orientation image of a Ni superalloy



TKD darkfield image of a deformed Al-Sc alloy

AZtecCrystal – OI's new EBSD data processing software

- First version launched in 2019 – replaces legacy Channel5 software
- Version 2.0 released in October 2020
- New interface
- New functionality
- Includes a new tool for microstructure classification

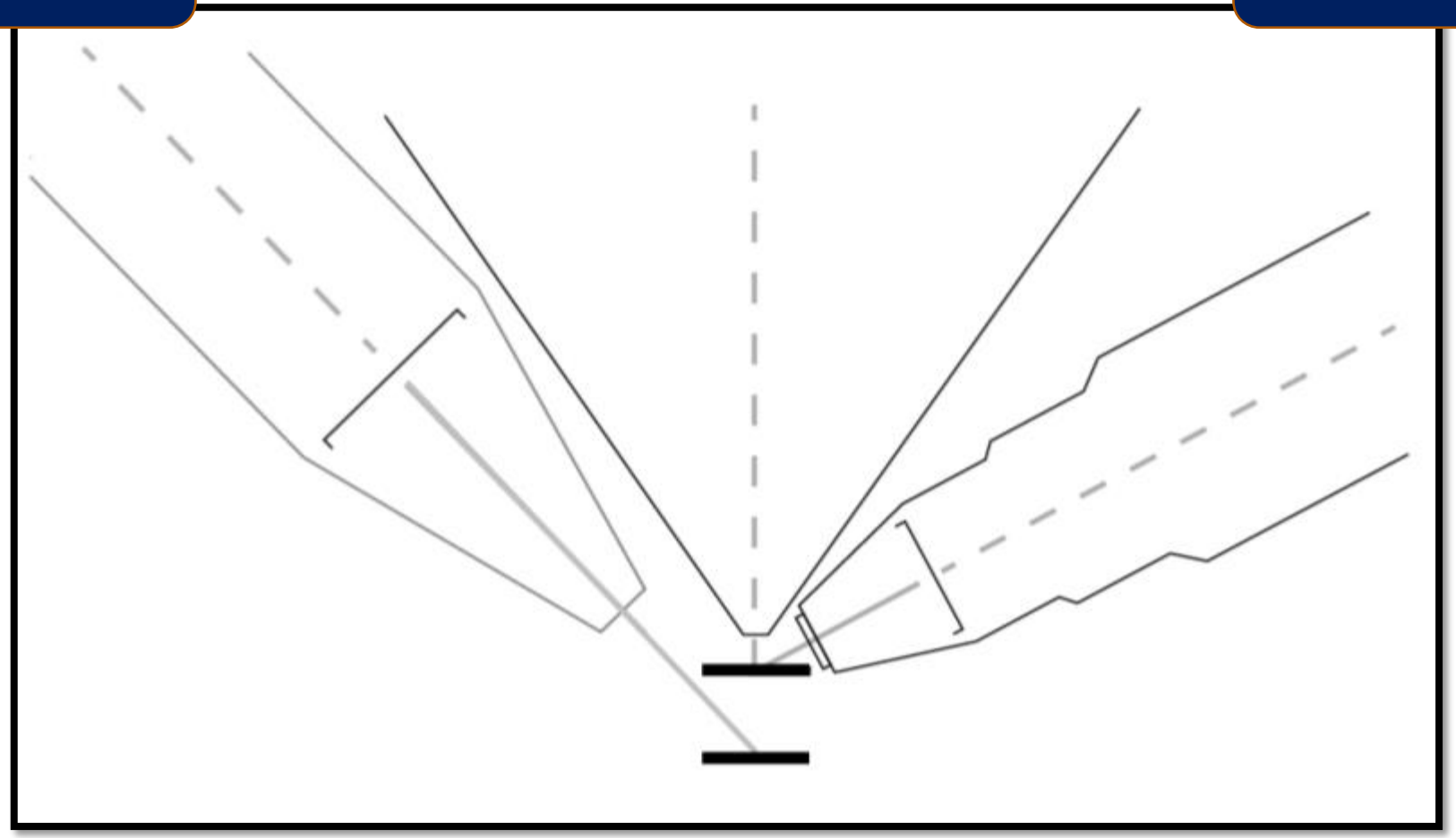


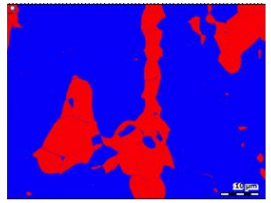
AZtec software-combining EDS and EBSD analysis

Ultim MAX

Ultim EXTREME

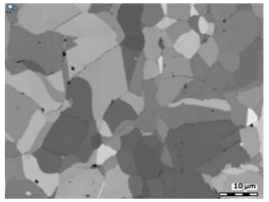
RELATE



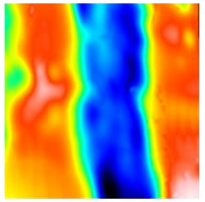


EBSD phase map

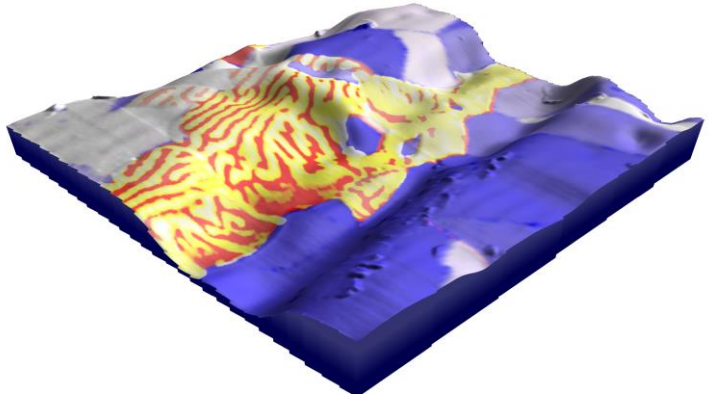
MFM



EBSD grain boundary

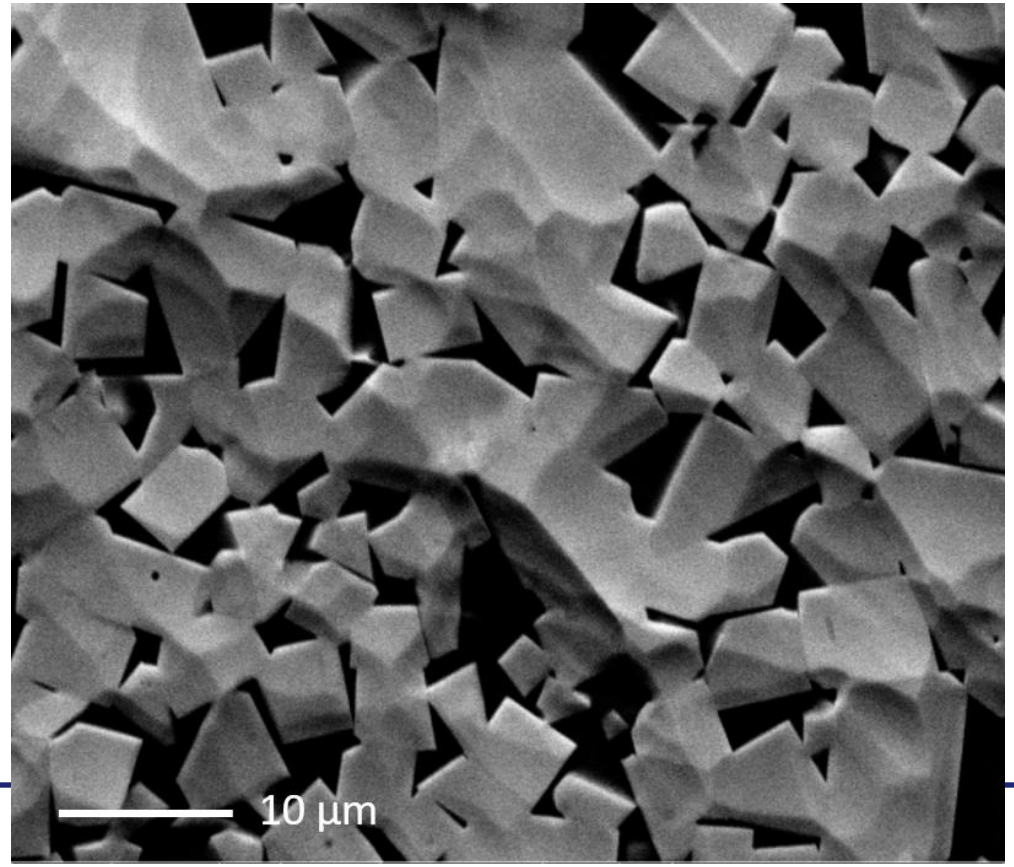


AFM topography



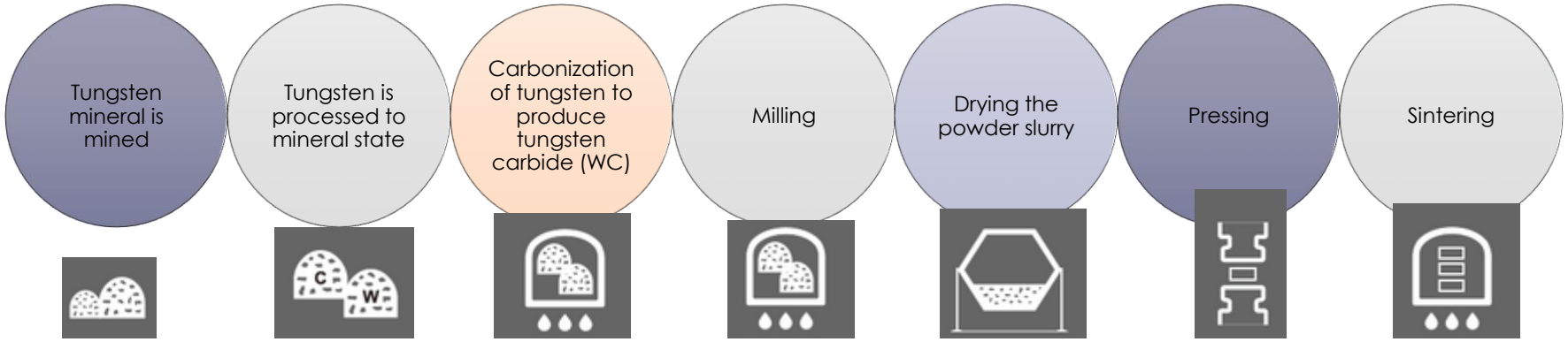
Applications:What is tungsten carbide

- Tungsten carbide is a hard metal
- Hard metals are common
- Hardmetals are known for their stiffness and hardness

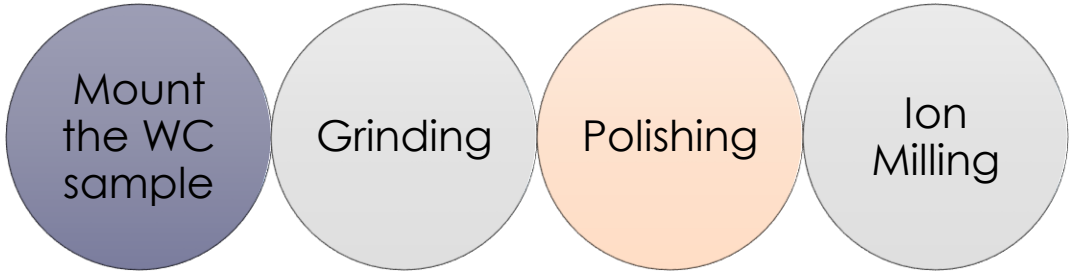


Tungsten carbide samples

Tungsten carbide fabrication



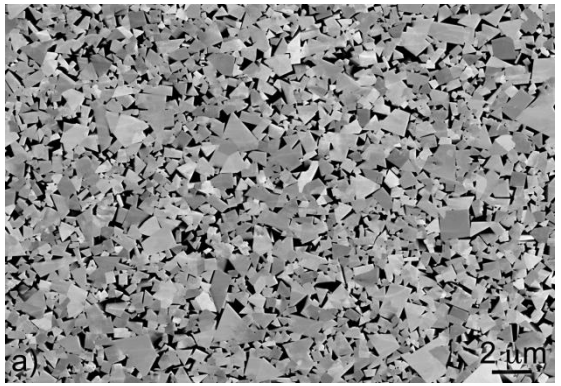
Sample Preparation



Backscattered electrons

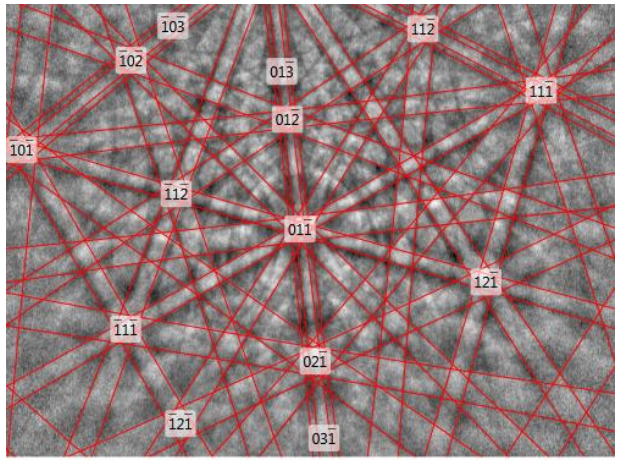
Primary electrons (back/forescattered)

- atomic contrast and topography
- structural information from crystalline material in the form of EBSPs
- reveal crystallographic defects (electron channelling contrast imaging or ECCI)

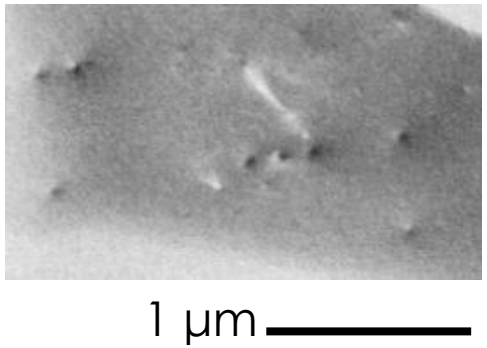


Z contrast of WC-Co

Yousfi, 2016

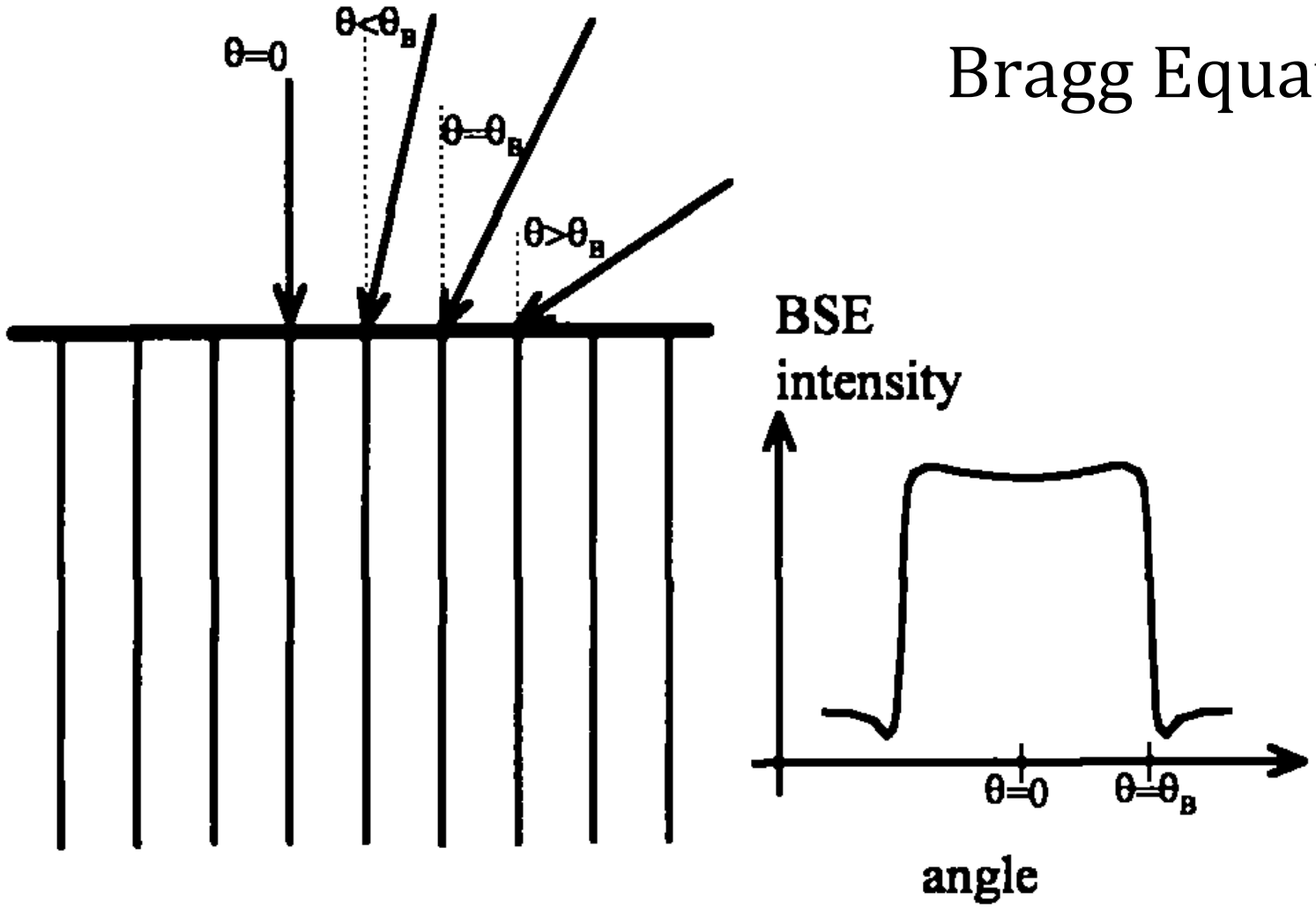


EBSD Pattern of WC-Co



ECCI of WC-Co

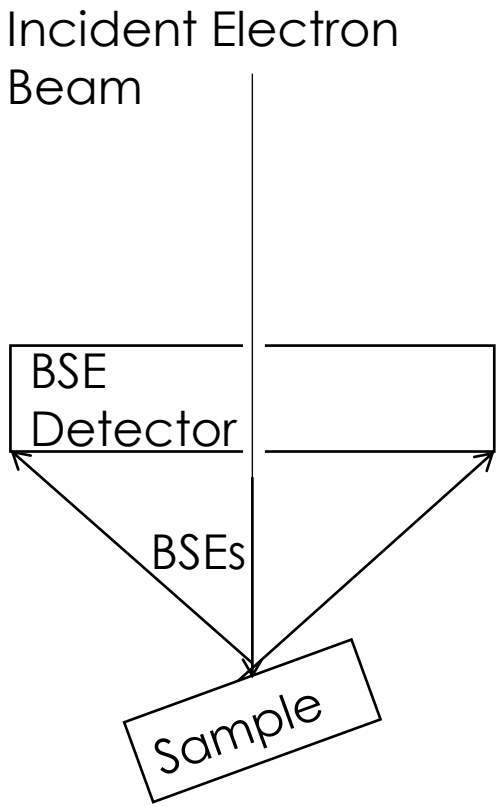
ECCI

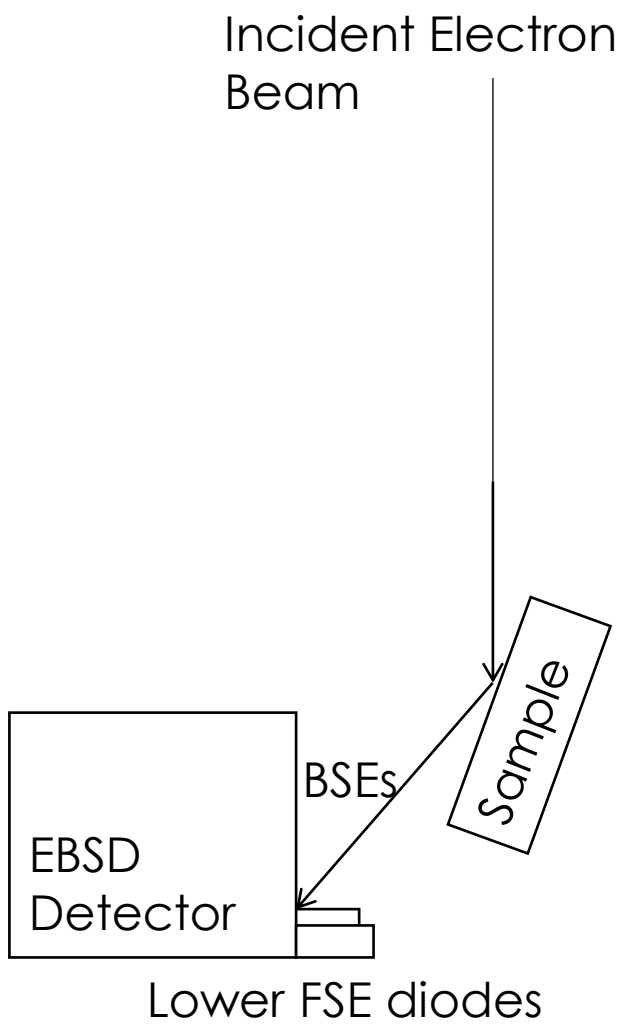


Bragg Equation: $n\lambda = 2dsin(\theta)$

(From Wilkinson and Hirsch, 1997)

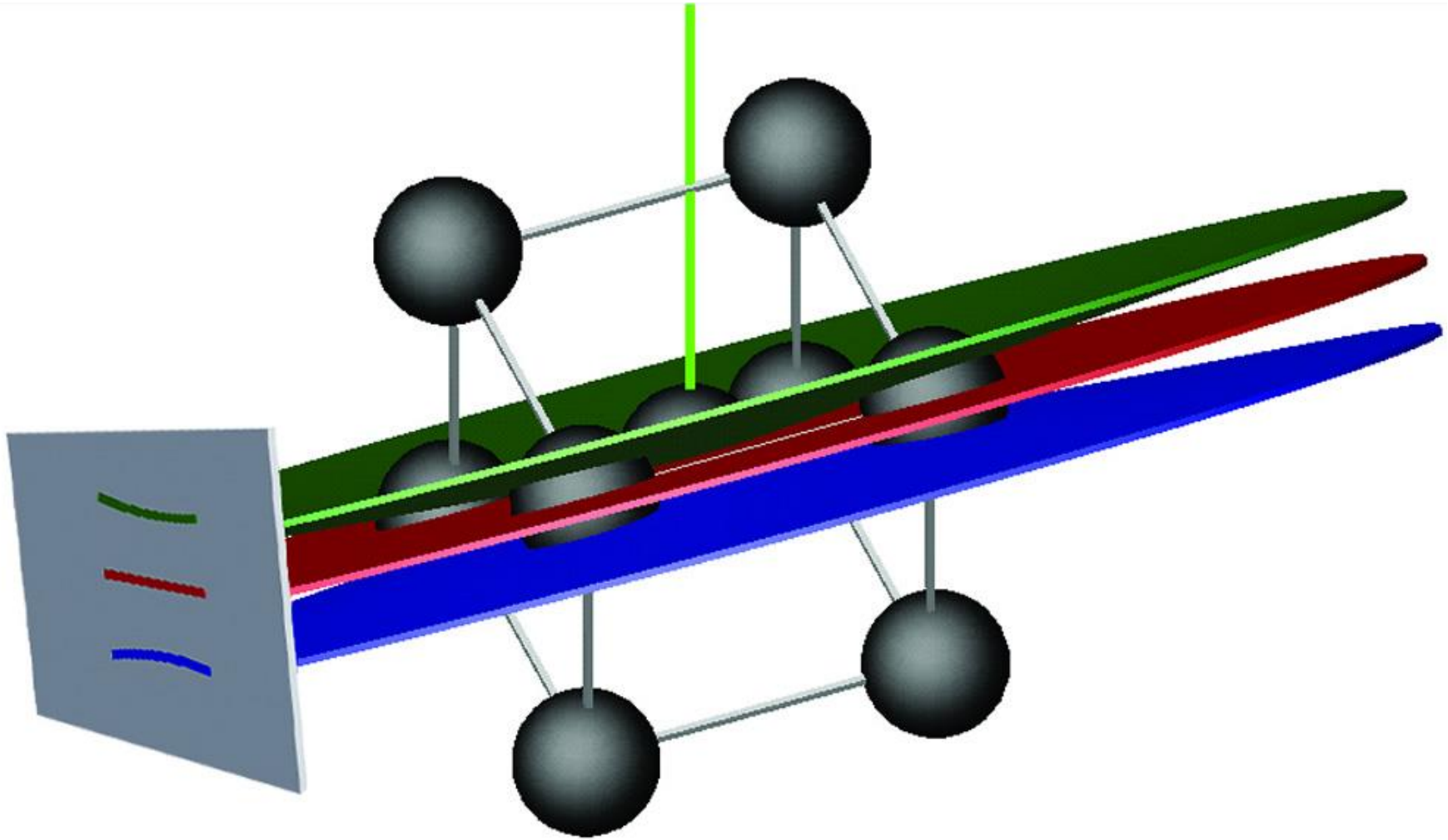
Potential ECCI configurations in an SEM



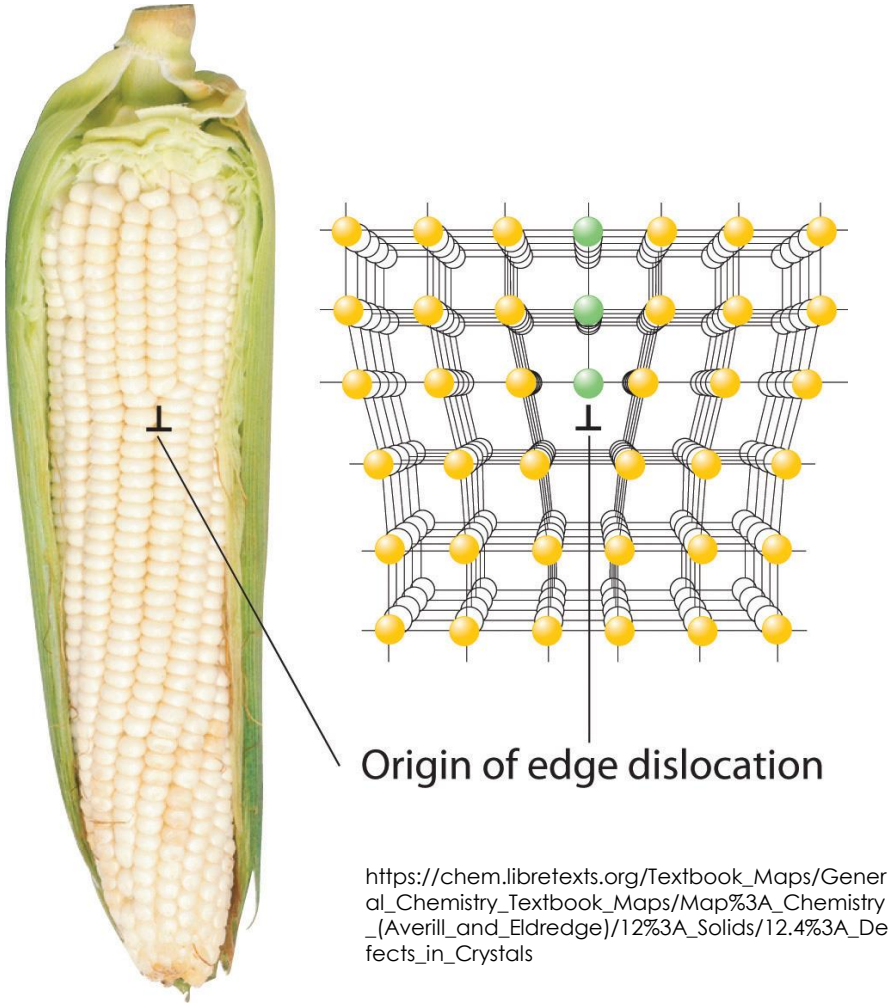


(Adapted from Kaboli et al, 2013)

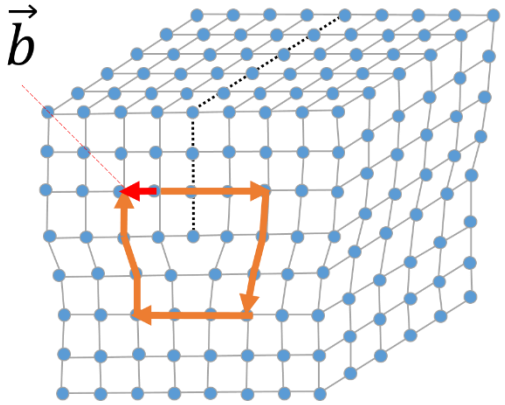
EBSD configuration $n\lambda = 2dsin(\theta)$



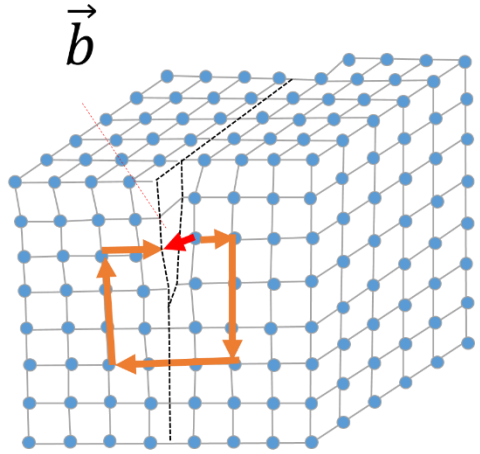
Dislocations



https://chem.libretexts.org/Textbook_Maps/General_Chemistry_Textbook_Maps/Map%3A_Chemistry_(Averill_and_Eldredge)/12%3A_Solids/12.4%3A_Defects_in_Crystals

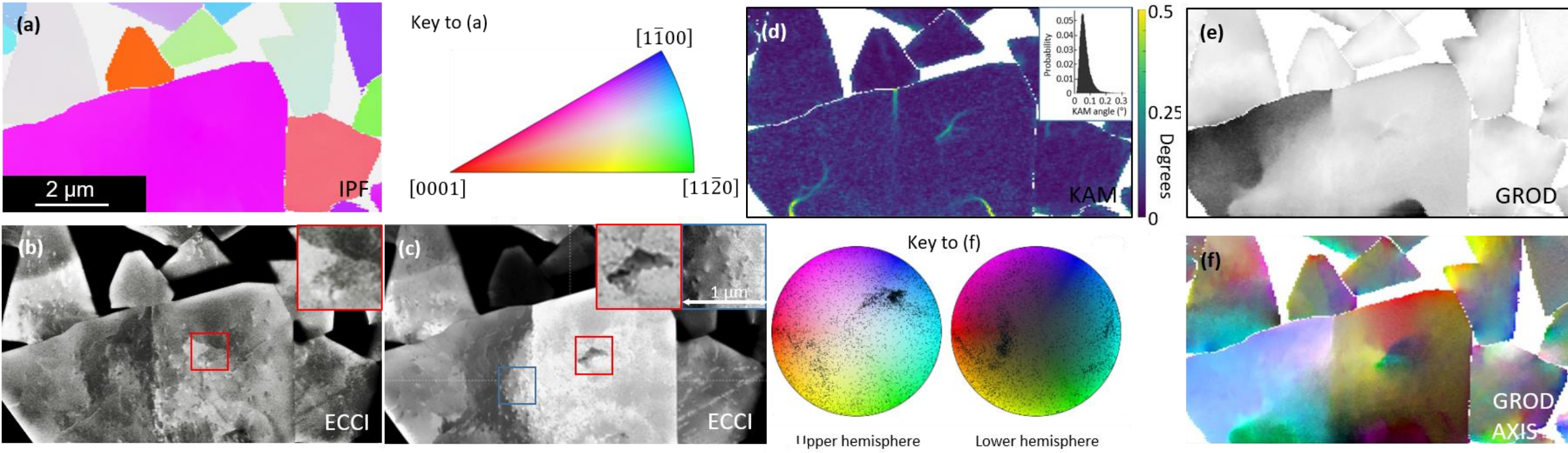


Edge dislocation



Screw dislocation

Subgrains, dislocations and HR-EBSD results

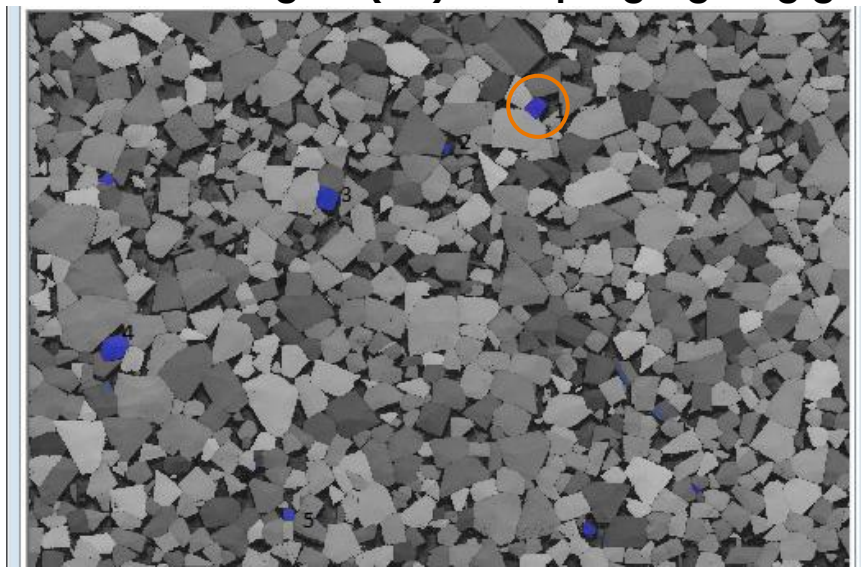


Accelerating Voltage: 20 kV, sample tilt: 20°

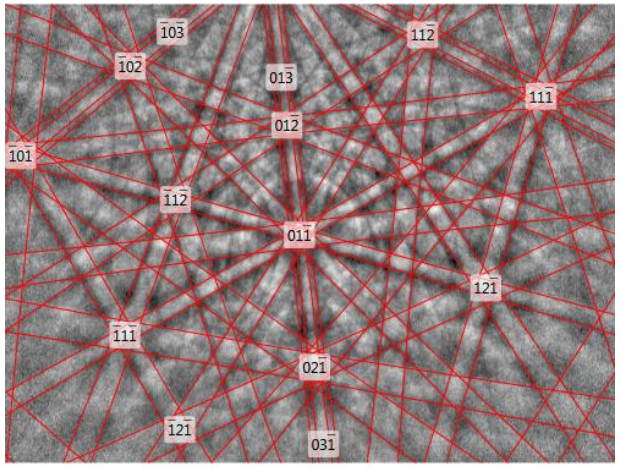
Image from Jablon et al;, 2020

Choosing a [10-10] oriented grain

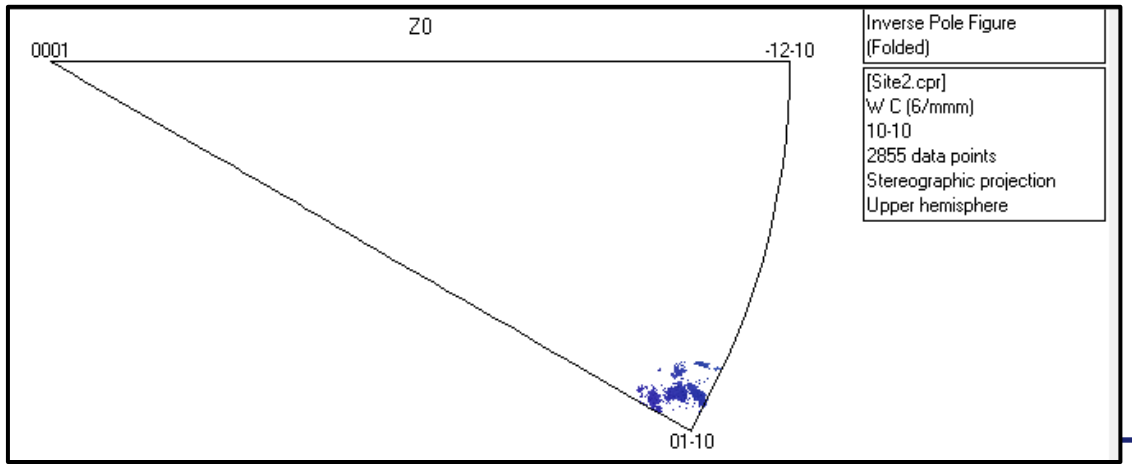
Inverse Pole Figure (IPF) Z map highlighting grains in $01\bar{1}0$ region



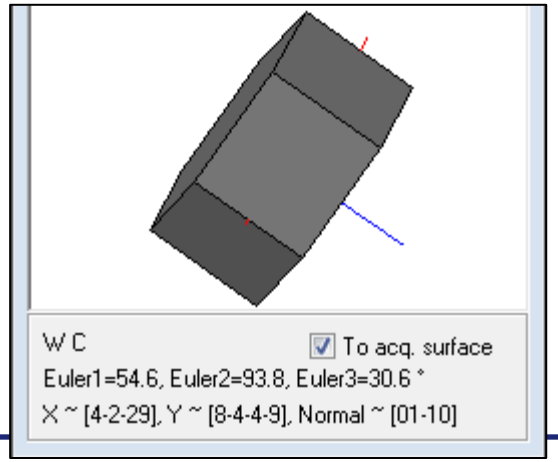
Electron backscattered pattern of $01\bar{1}0$ oriented grain



IPF Z map legend selecting grains in $01\bar{1}0$ region



Simulated grain direction

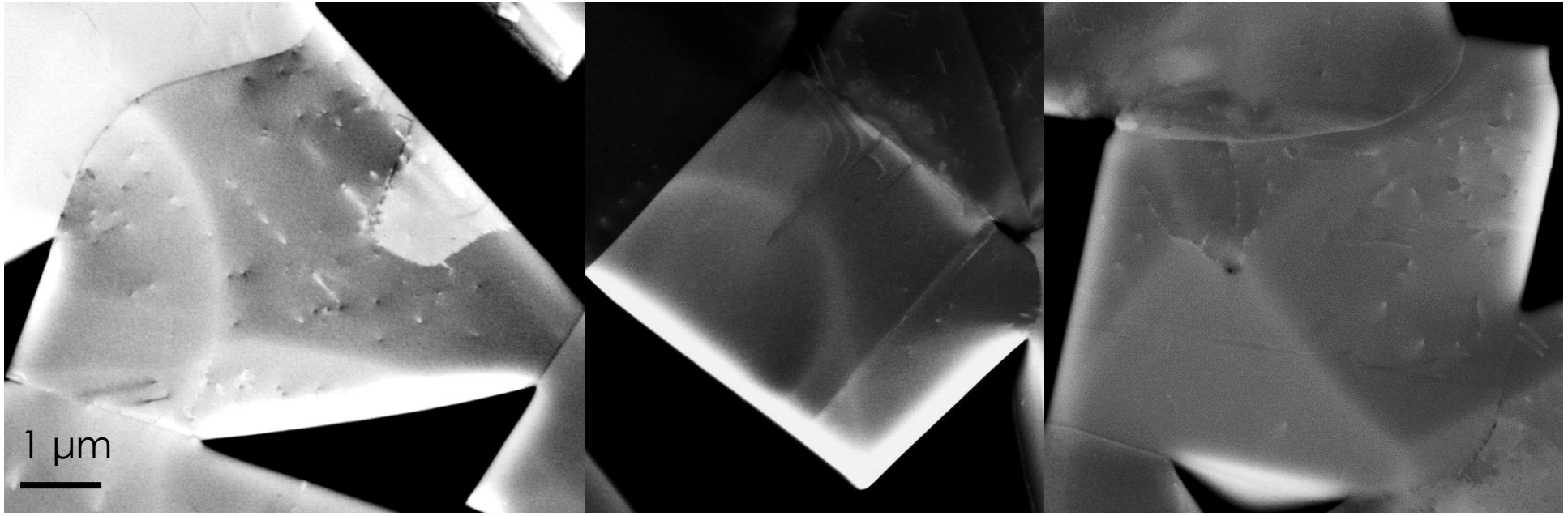


Defect distributions and crystal orientation

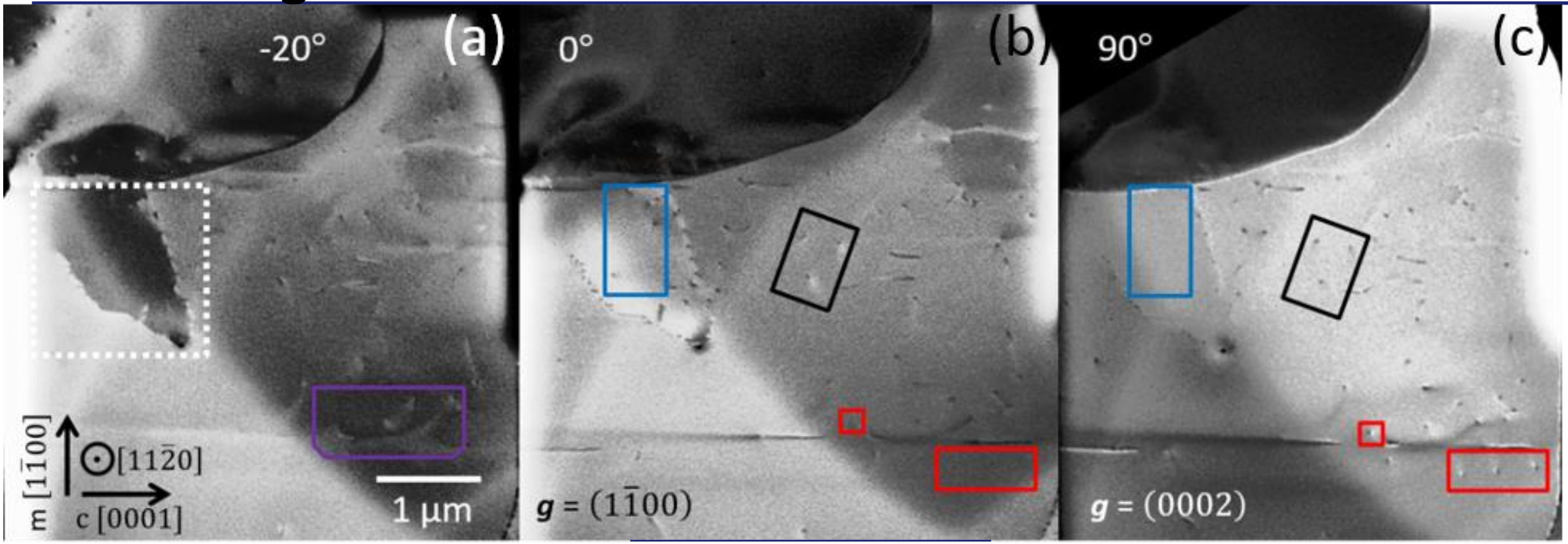
[0001]
≈ 4 x 10^8 cm^{-2}

$[10\bar{1}0]$
≈ 2 x 10^8 cm^{-2}

$[11\bar{2}0]$
≈ 2 x 10^8 cm^{-2}



Dislocations as revealed by ECCI in a [11-20] oriented grain



Euler angle (°)	Subgrain	Parent grain
Φ_1	0.8	0.7
Φ	92.8	93
Φ_2	59.1	59

Conclusions

- Users can image the internal structure of WC grains from EBSD data
- Qualitative ECCI data presented above is thereby quantified by the orientation data from pattern matching
- Compare ECCI and EBSD data using HR patterns

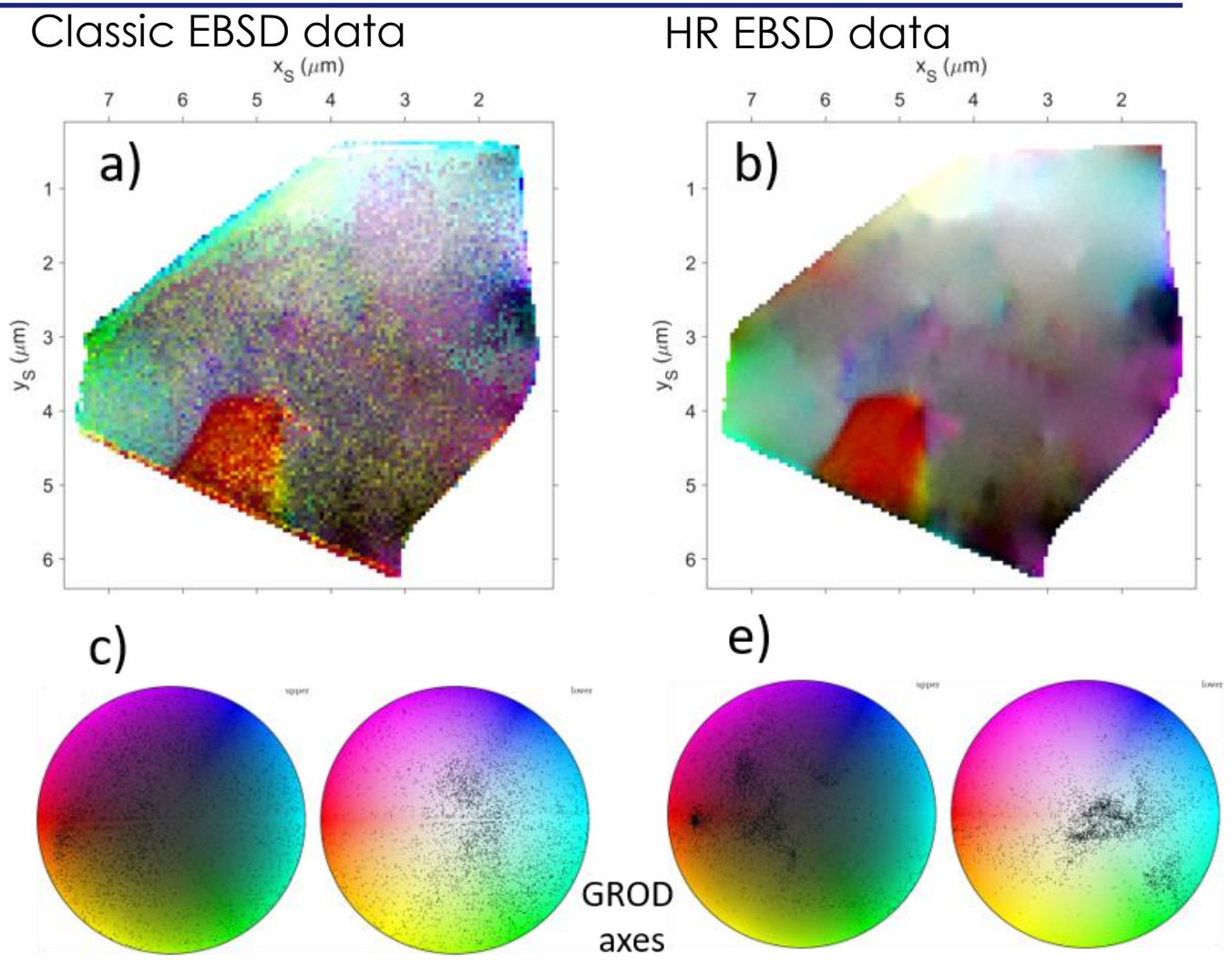


Image from Winkelmann et al;, 2020

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