# Advanced sample preparation by broad ion beam milling for EBSD analyses

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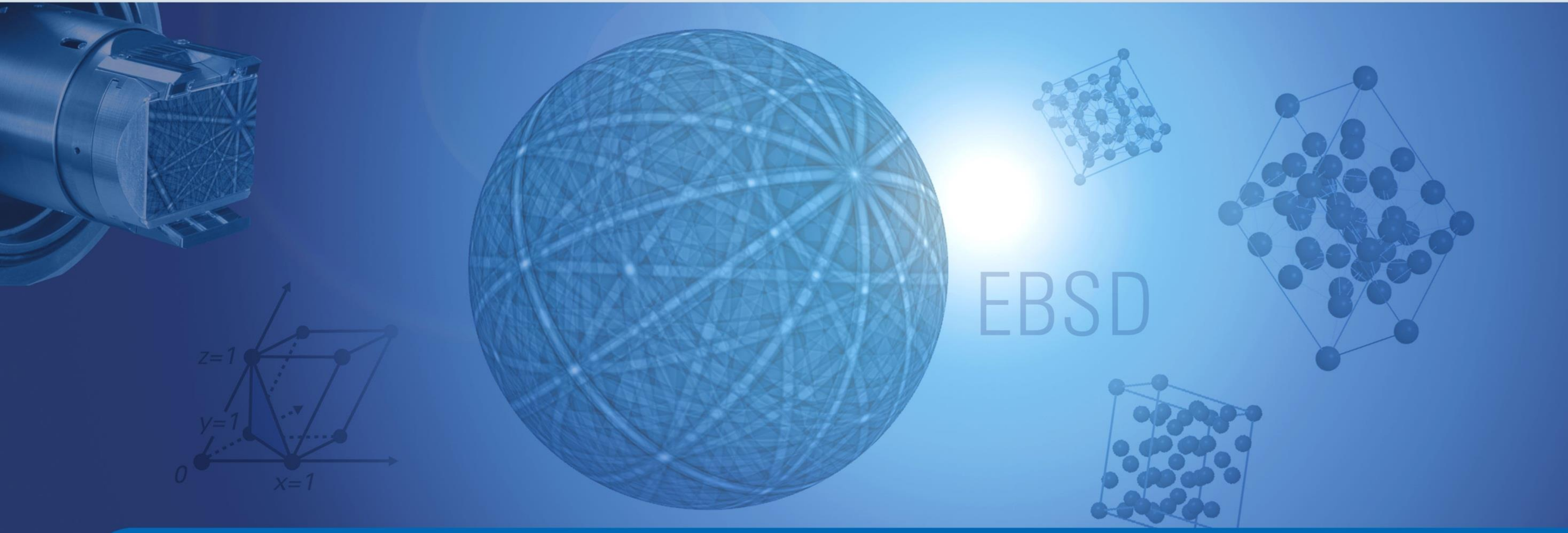
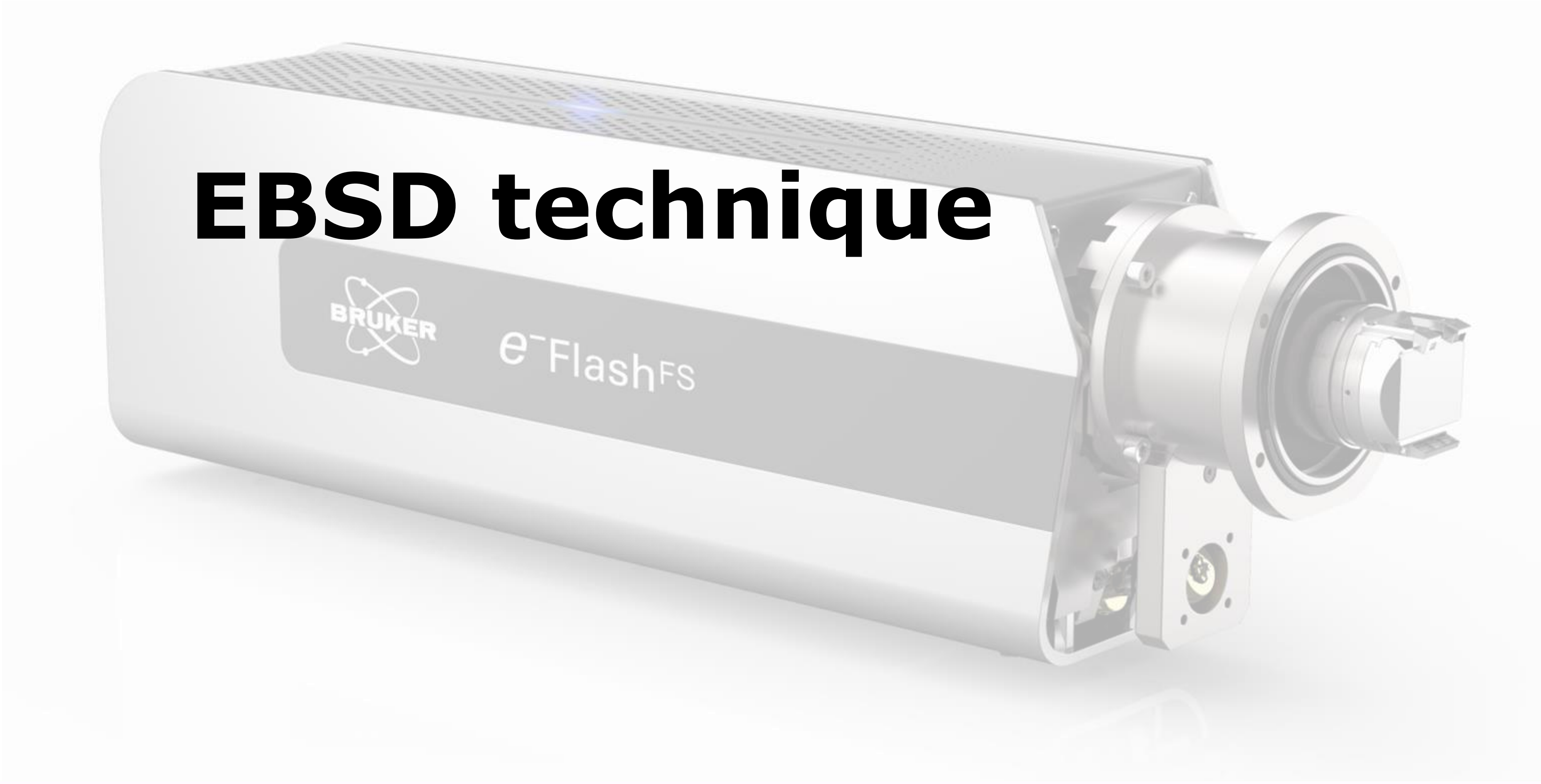



10.11.2020

# Outline

- EBSD technique overview
- ARGUS™ imaging system
- Broad ion beam milling: Model 1061 SEM Mill
- Application examples:
  - Austenitic steel
  - Zirconium alloy
  - Ti CP
  - Ti alloy
  - Solder bumps

# EBSD technique



# Introduction

## What is EBSD?

- **EBSD** - **E**lectron **B**ack**S**catter **D**iffraction
- SEM based technique to measure **crystal orientations**
- Applicable to any **crystalline material** (in theory)
- Provides the absolute crystal orientation with sub-micron spatial resolution:
  as fine as 300 nm grain size for bulk sample, 10 nm with Transmission Kikuchi Diffraction (TKD)
- Complementary technique between X-ray diffraction and TEM studies

➡ *The only technique that correlates local texture (orientation) with microstructure (grain metrics, grain boundary, ...)*

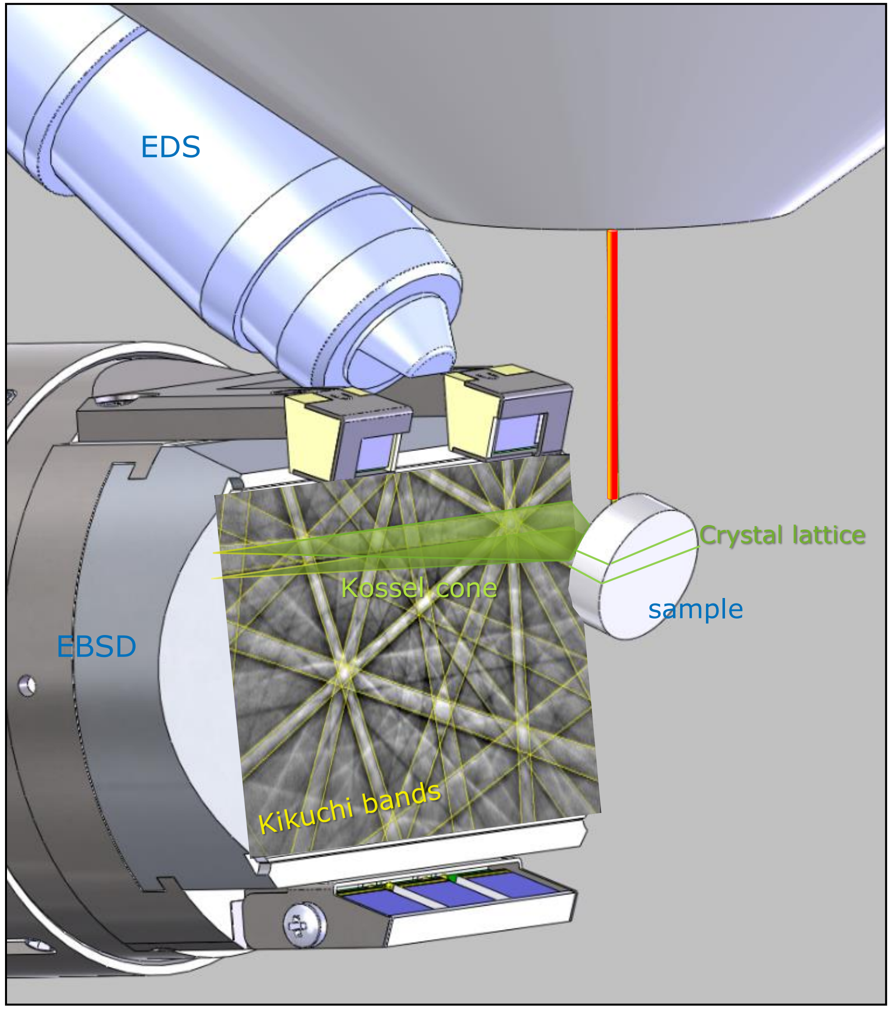
# Introduction

## EBSD: What is it used for?

**To understand the material properties by:**

- Analysing the grain structure, crystal orientation, phase ID, and distribution
- Residual stress, quality control, phase transformation, fracture analysis, strain analysis, tectonophysic studies...
- **Texture analysis**: Assessing the effects of the thermomechanical processes
- Complement the chemical information; **maximise your knowledge of the sample** and the processes involved

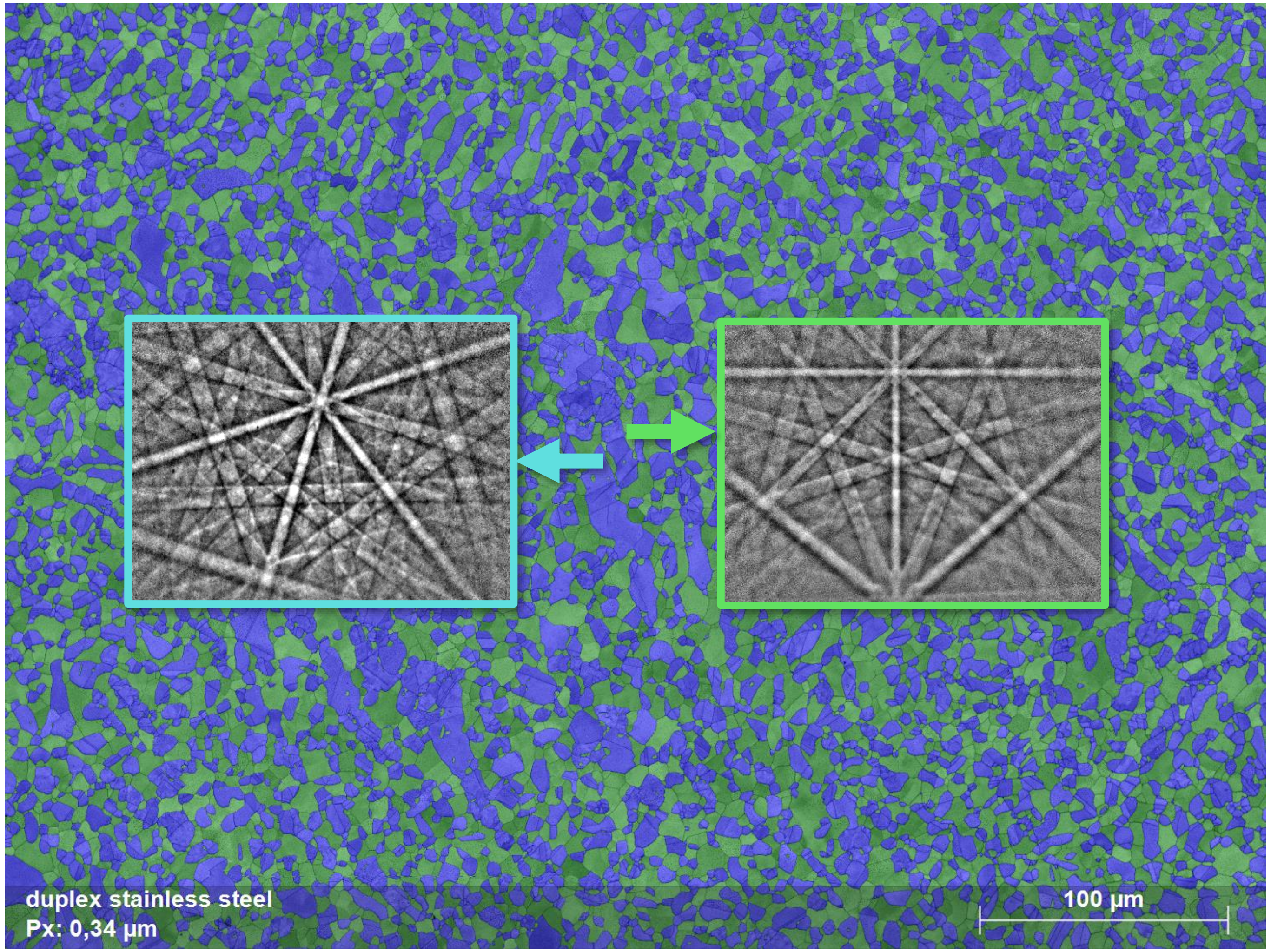
# EBSD technique
## EBSP formation




- *e*- beam strikes specimen
- Scattering produces *e*- travelling in all directions
- $e^-$ that satisfy the Bragg condition ($n\lambda$=2d.sin$\theta$) for a plane (hkl) are channeled $\Rightarrow$ 2 hyperbolas/diffraction cones (**Kikuchi bands**) corresponding to the various diffracting planes
- Backscatter volume: Top few nanometers below the surface
- $e^-$ strike the phosphor screen and produce light (gnomonic projection)
- Which is detected by a CCD or CMOS camera and digitised
- The resulting diffraction pattern "**EBSP**" is automatically analysed and indexed...

# EBSD technique

## EBSD measurement phase identification & distribution



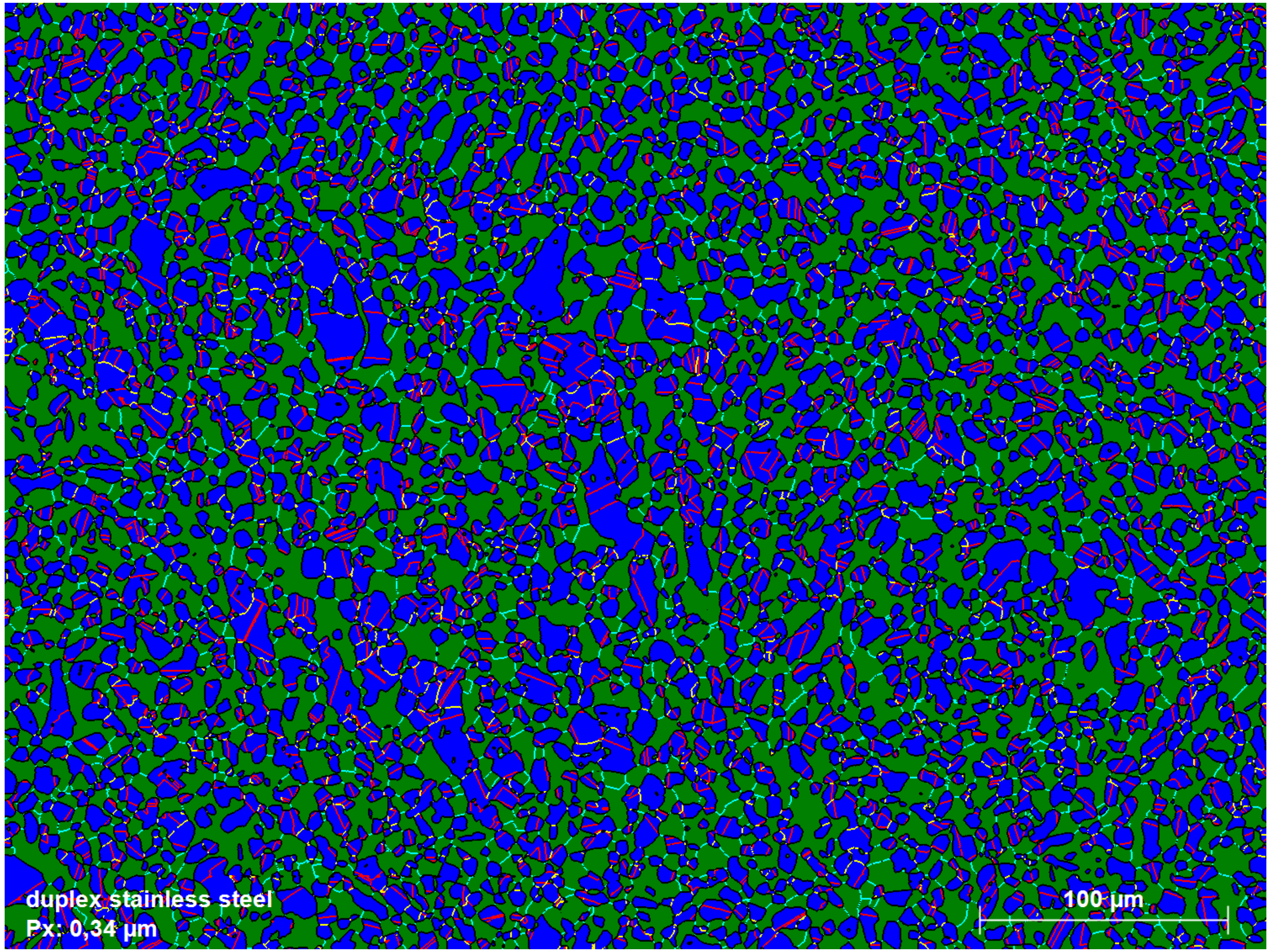

**EBSD phase map**

Hit rate 99,4%

| | | |
|---|---|---|
| (blue) | Fe fcc | 49,1% |
| (green) | Fe bcc | 50,2% |

# EBSD technique

## EBSD measurement phase identification & distribution



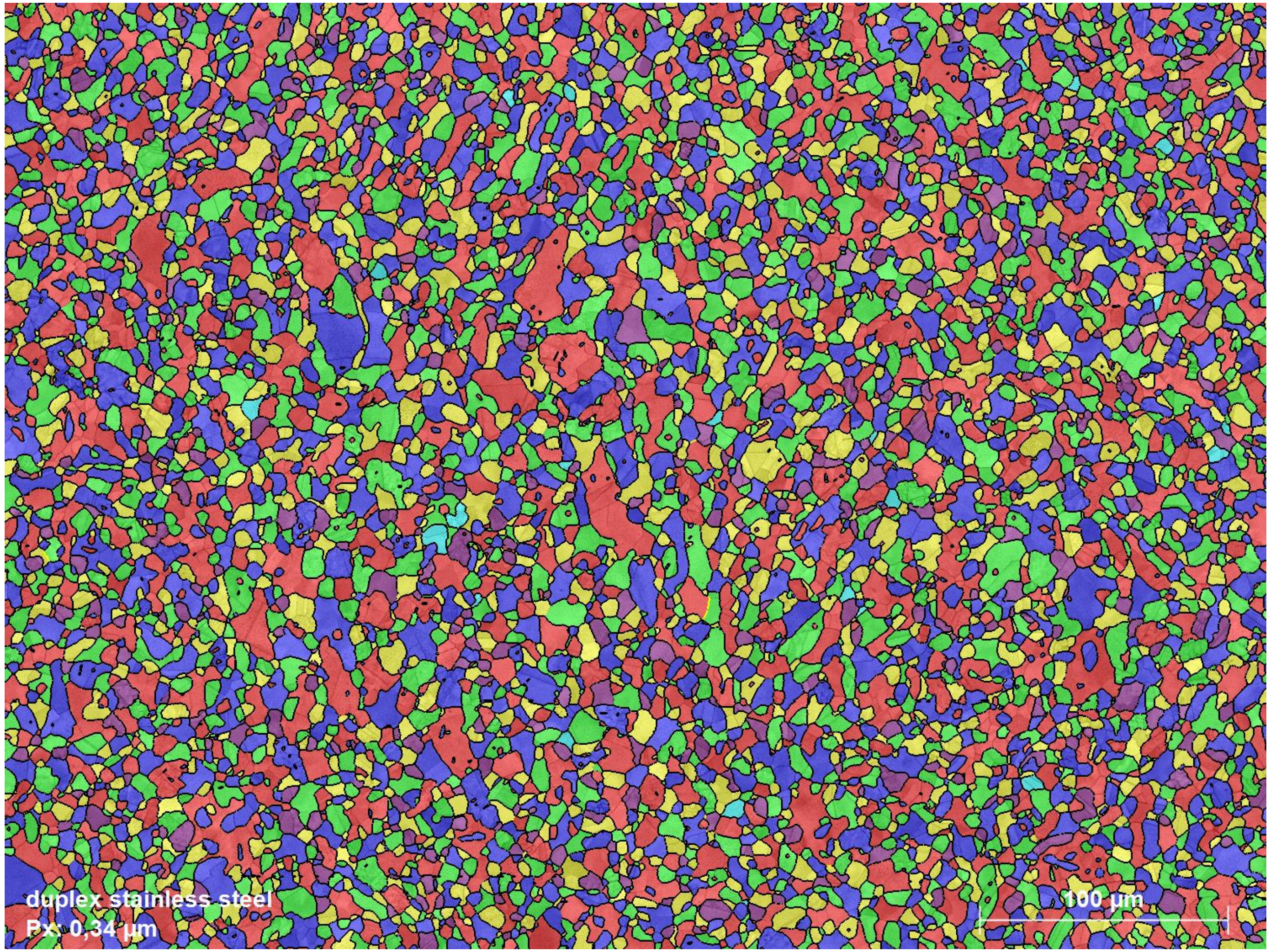

**EBSD phase map**

Hit rate 99,4%

- Fe fcc 49,1%
- Fe bcc 50,2%
- Grain boundaries (13,1 mm)
- Phase boundaries (63,4 mm)
- ∑3 (17 mm)

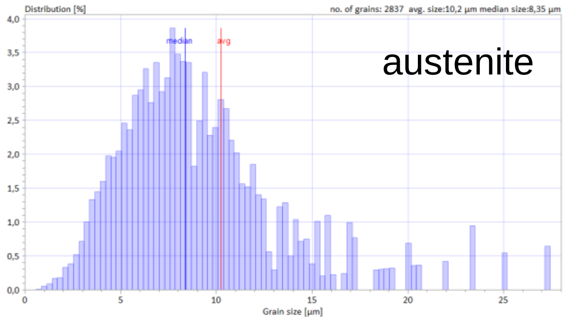
# EBSD technique

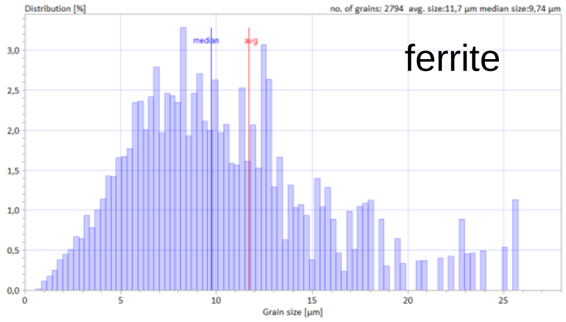
## EBSD measurement – grain metrics




### Grain distribution map

1,7M points in 30 min
5631 grains measured
Average grain size: 11 µm



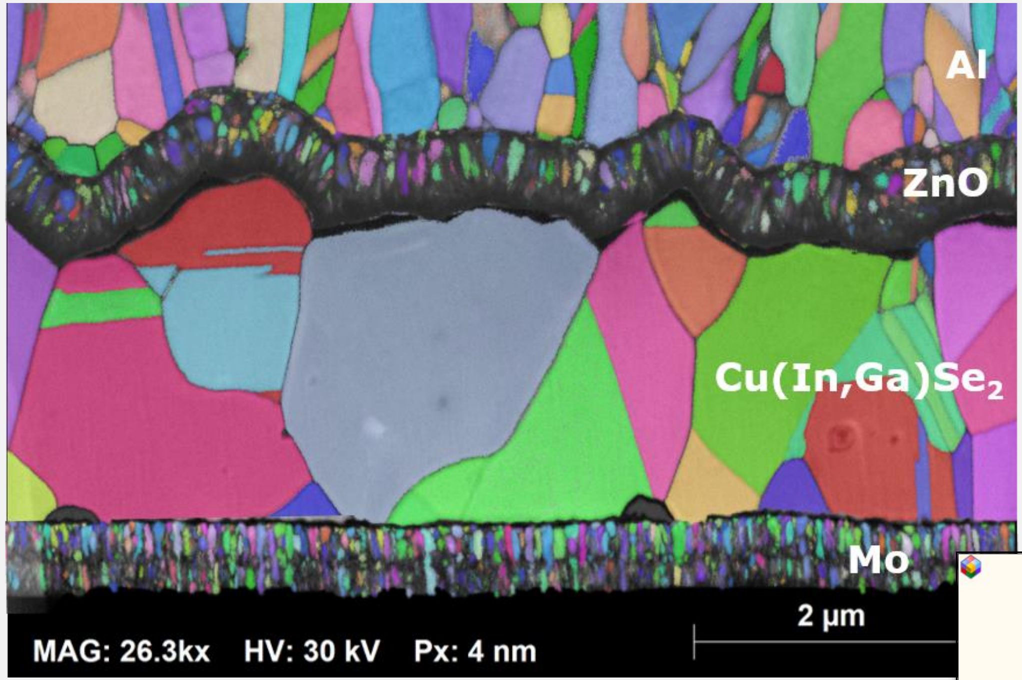




*Correlation between local texture, microstructure, grain boundary and grain size analysis, ...*

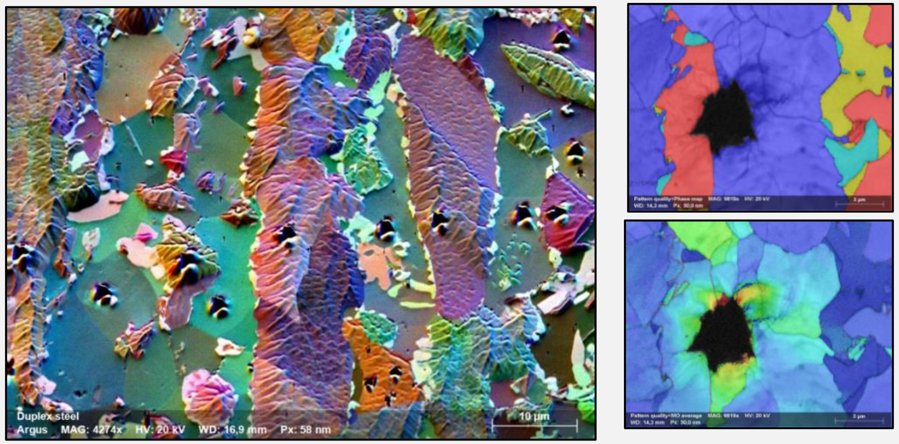
# EBSD technique

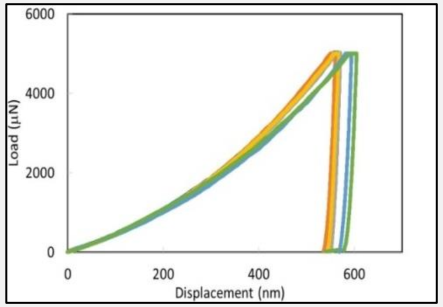
## EBSD measurement – other applications

### Transmission Kikuchi Diffraction

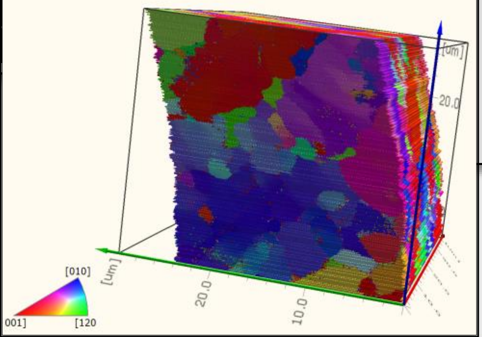



### In situ EBSD/EDS & mechanical testing



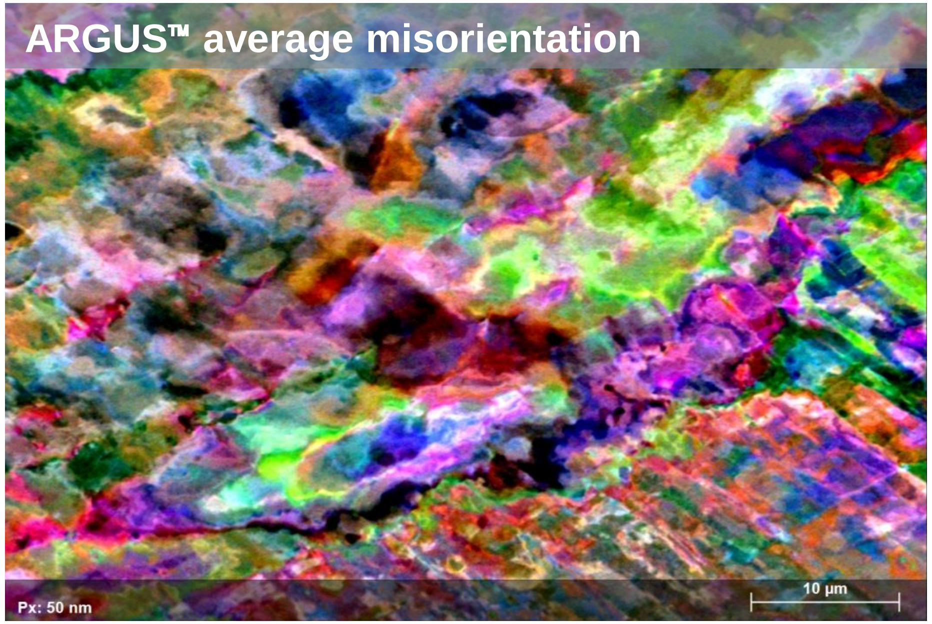

### 3D EBSD/EDS



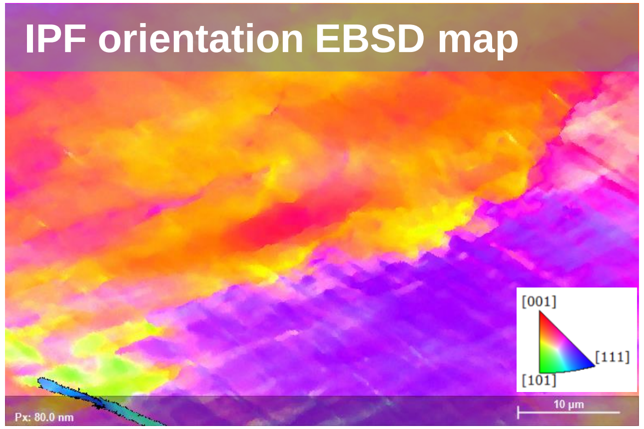
# High-quality EBSD measurement requires high-quality sample preparation

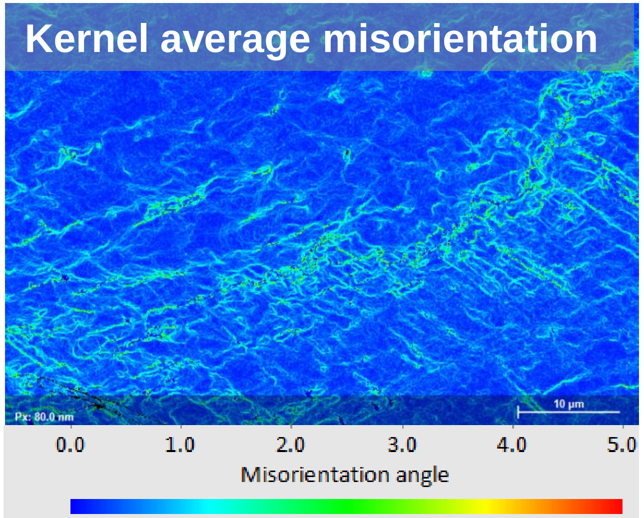


Highly deformed copper sample prepared by ion milling



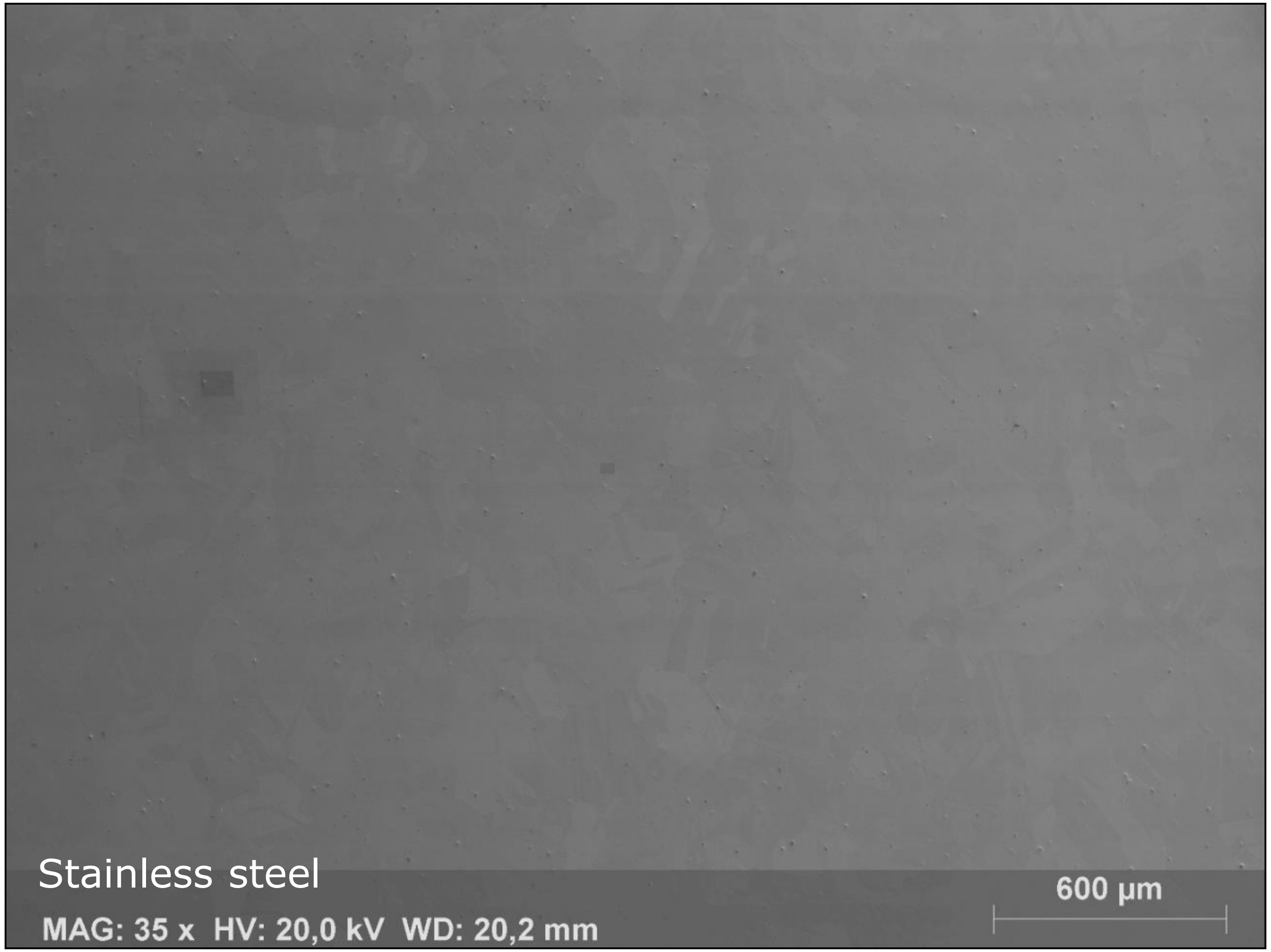

- Ion milling removes topography and mechanical preparation artifacts from the sample. An iterative workflow between the SEM Mill and the ARGUS™ imaging system results in optimal sample preparation.
- ARGUS™ orientation contrast imaging displays the quality of the sample preparation; orientation (IPF) and misorientation (KAM) maps reveal the microstructure of the material.



# ARGUS™ imaging system

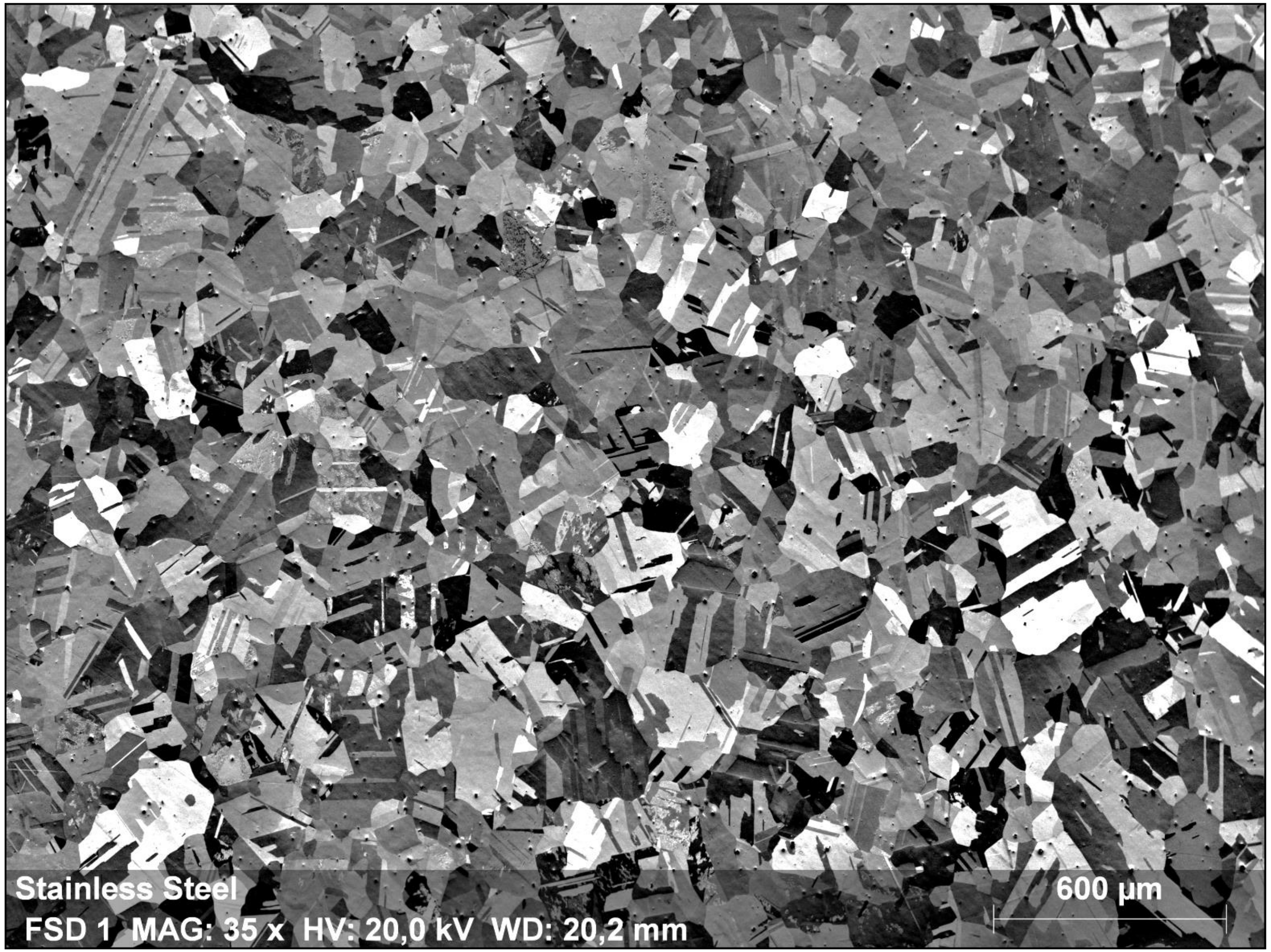
# FSE imaging system
## Color-coded orientation contrast




- SE images have very low contrast; not easy to find region of interest

# FSE imaging system

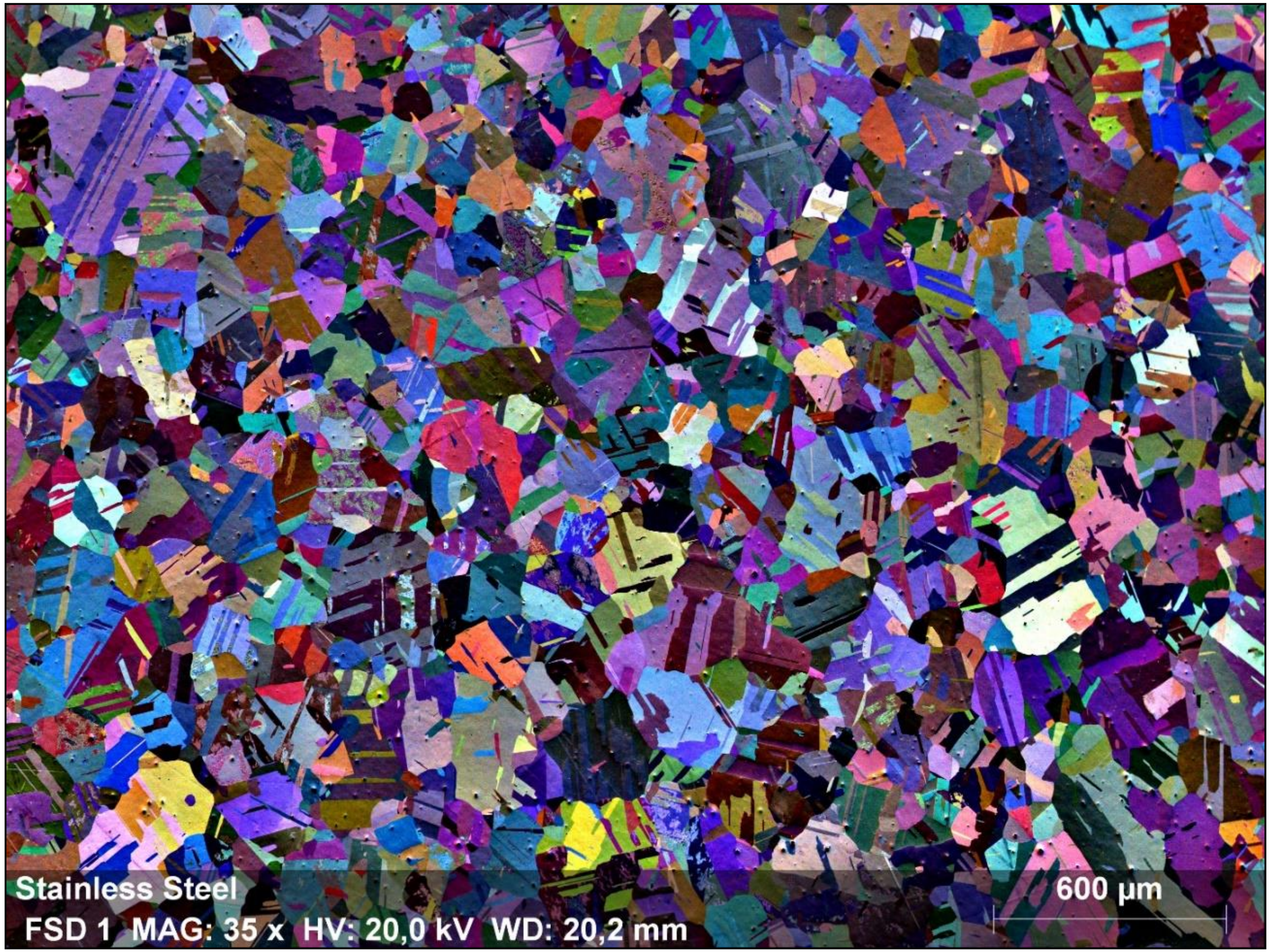
## Color-coded orientation contrast




- SE images have very low contrast; not easy to find region of interest
- Common Fore Scattered Electron (FSE) detectors acquire images with better contrast

# FSE imaging system

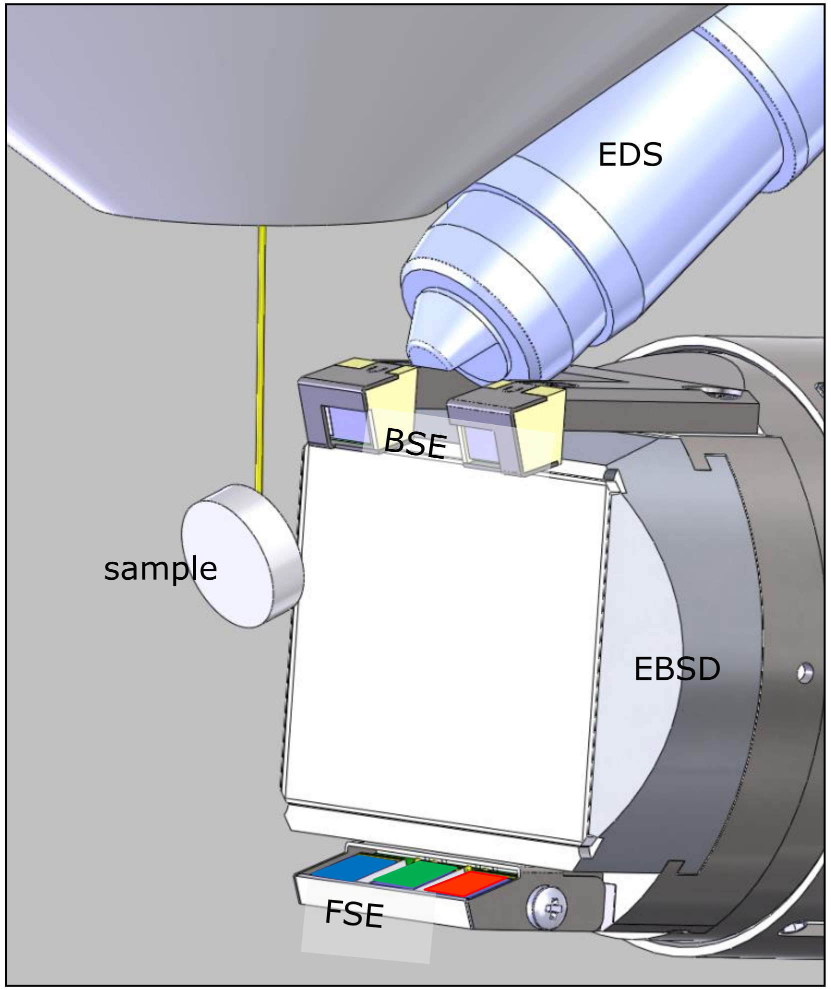
## Color-coded orientation contrast




- SE images have very low contrast; not easy to find region of interest
- Common Fore Scattered Electron (FSE) detectors acquire images with better contrast
- However, ARGUS™ color-coded FSE images can reveal much more detail with **acquisition speed of 125 000 pps**

# FSE imaging system with ARGUS™

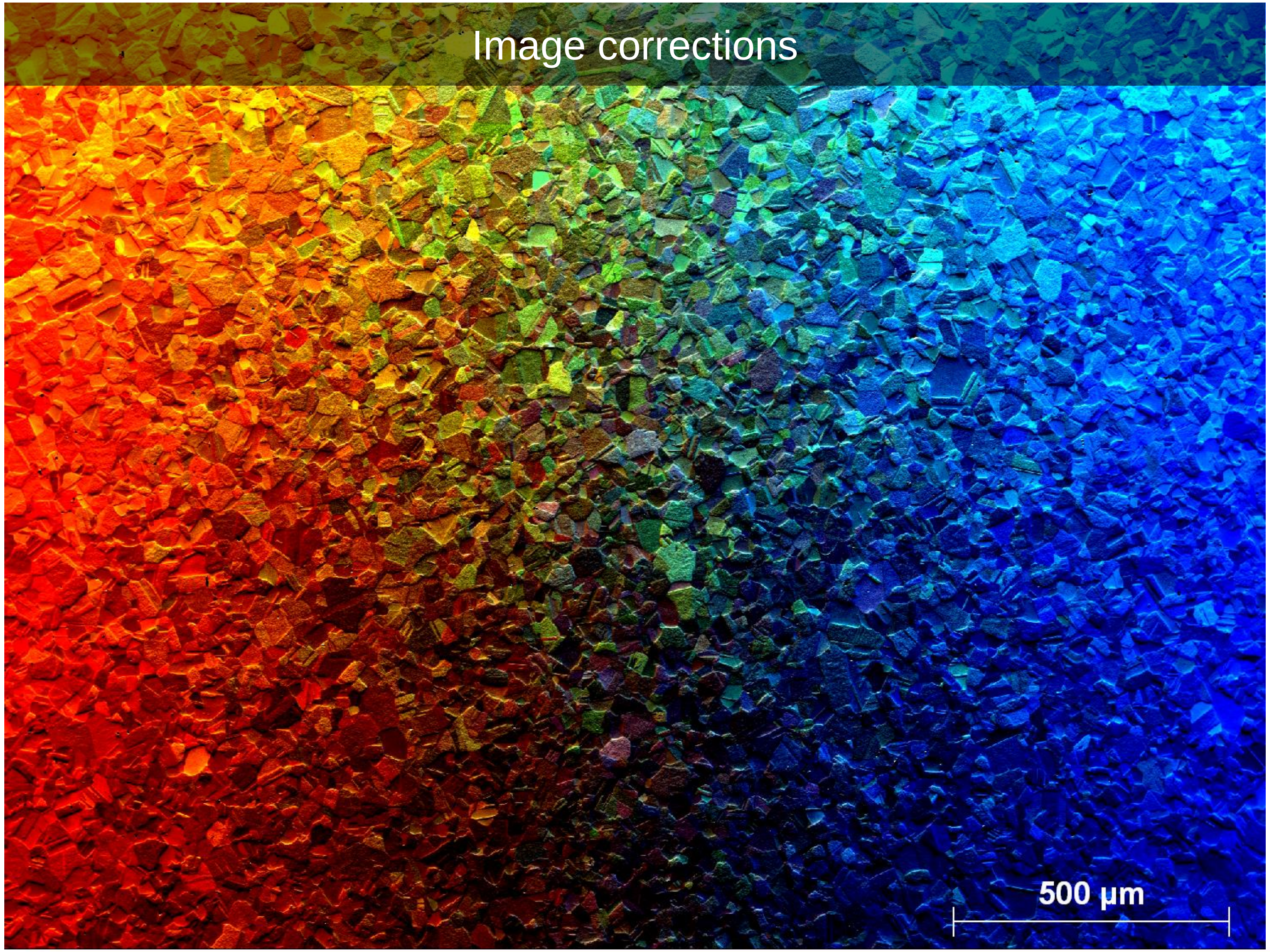
## How does it work?




- Each diode captures a similar amount of noisy backscattered electrons and a different small part of the diffracted backscattered electrons, i.e., EBSD signal
- Signal ($e^-$ counts) is transferred using a separate channel for each diode
- When scanning, for each pixel the system will obtain three numbers which will be transformed into three RGB levels
- When the three signals/RGB levels are mixed we obtain…

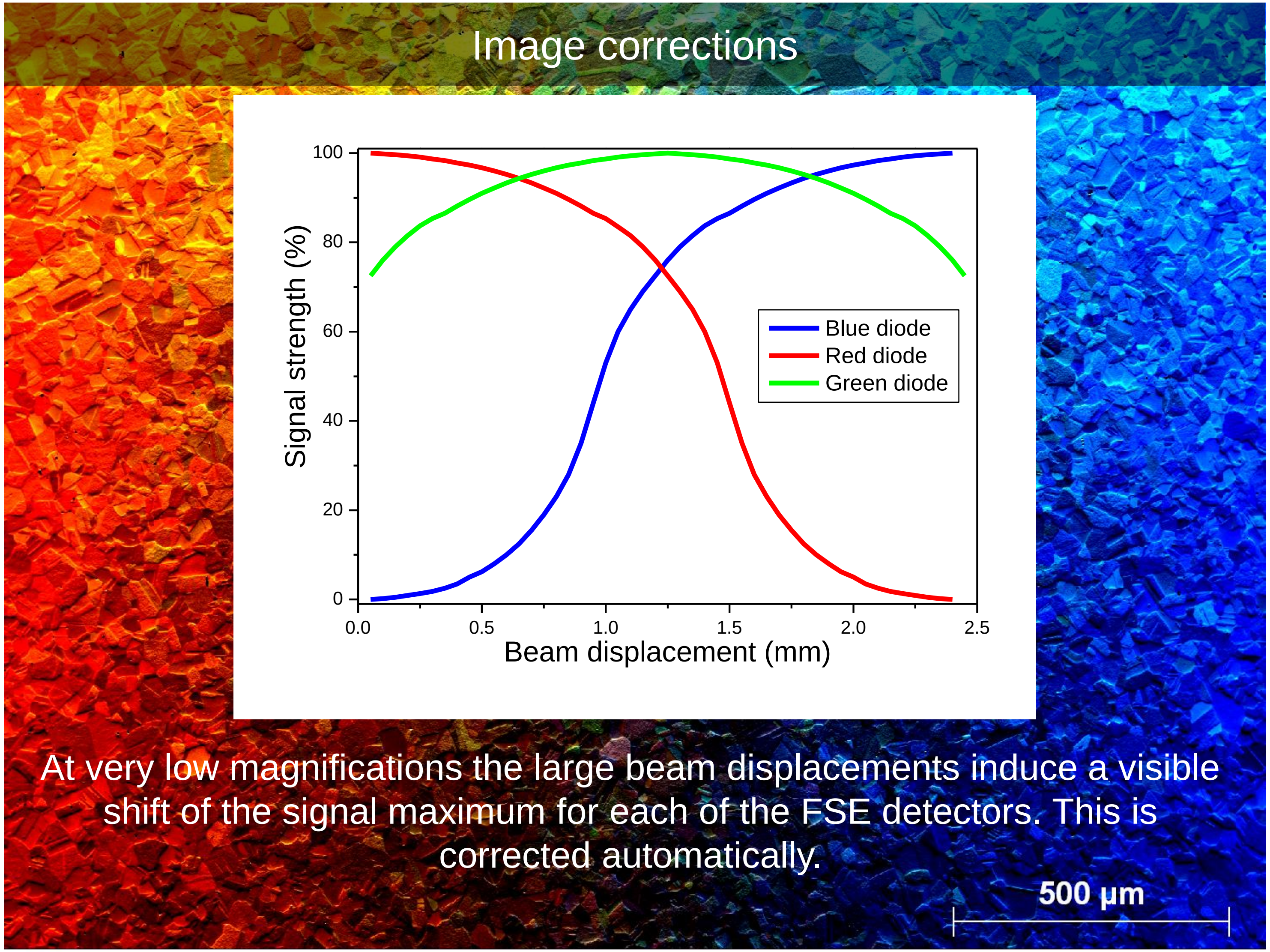
# FSE imaging system with ARGUS™

## How does it work?



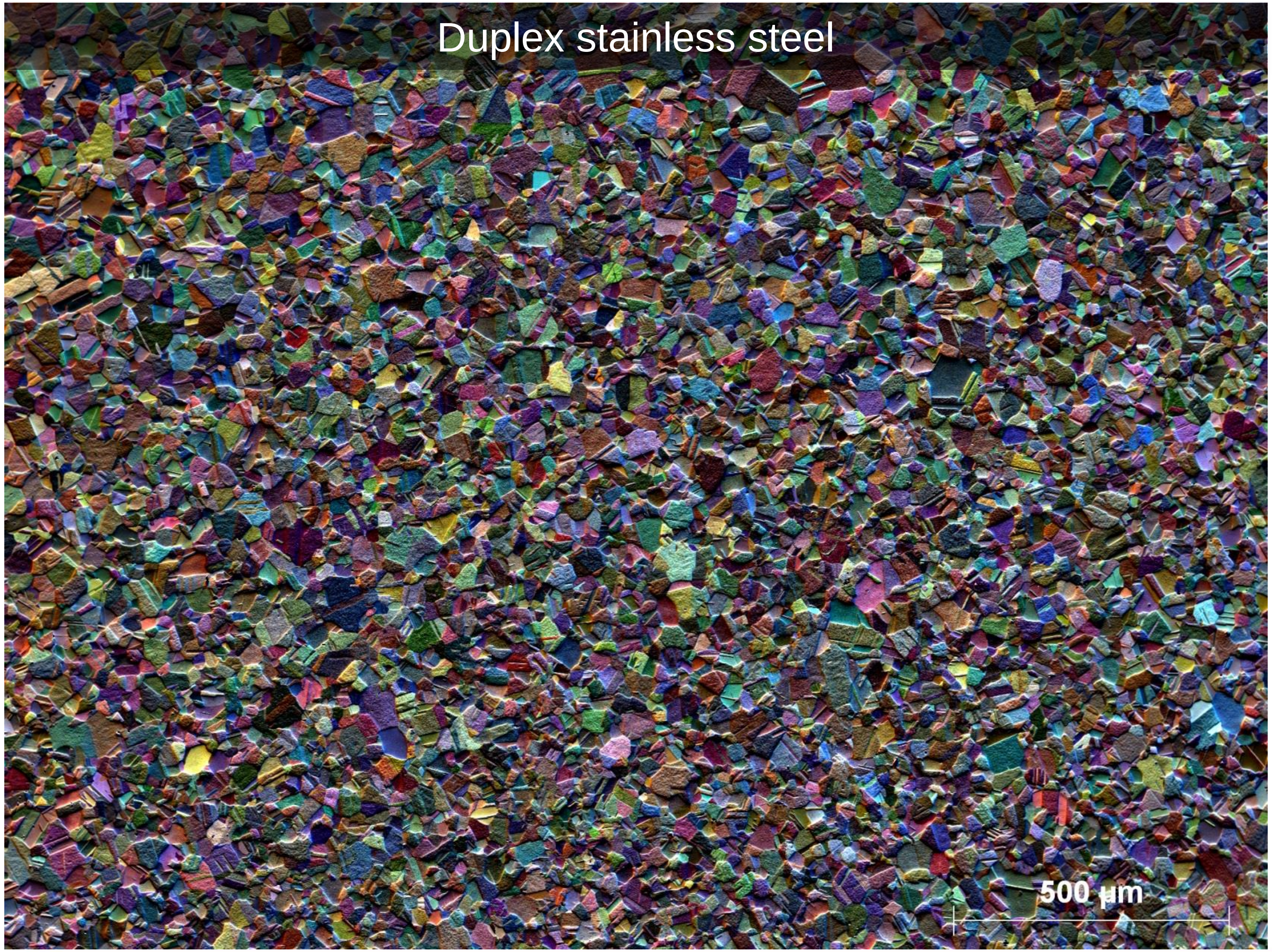
# FSE imaging system with ARGUS™

## How does it work?



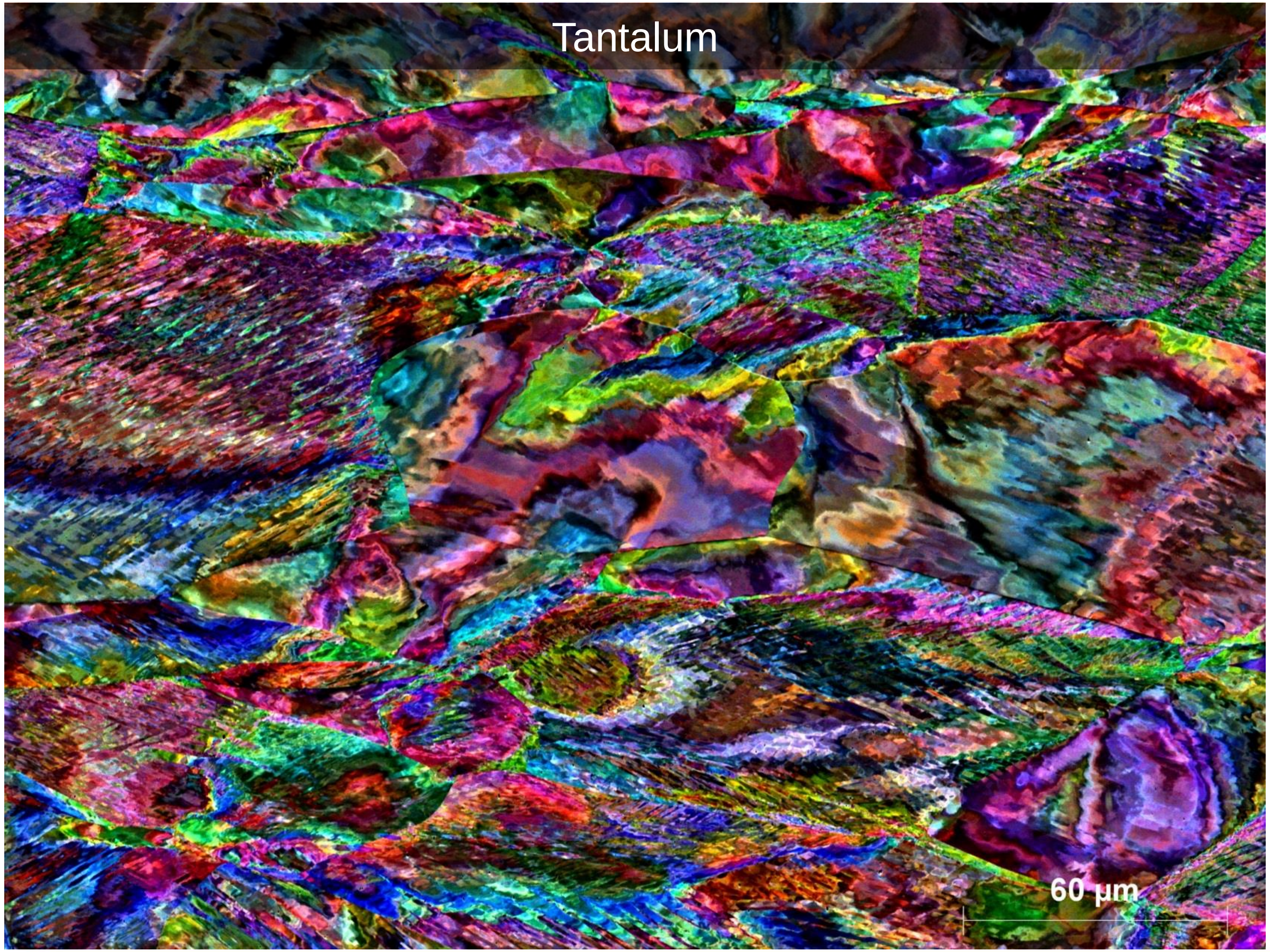
# FSE imaging system with ARGUS™

## How does it work?



# FSE imaging system with ARGUS™

## How does it work?



# Broad Ion Beam Milling
# Model 1061 SEM Mill

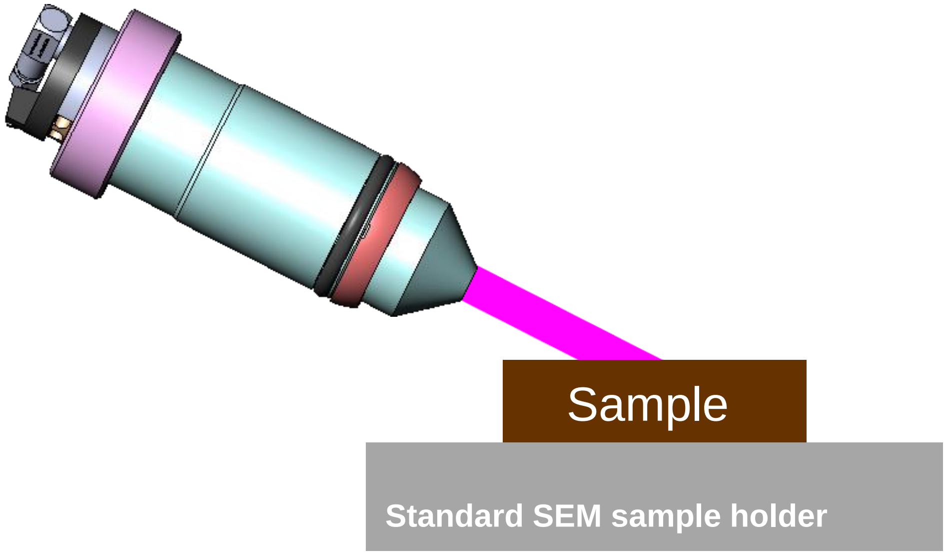
# Model 1061 SEM Mill

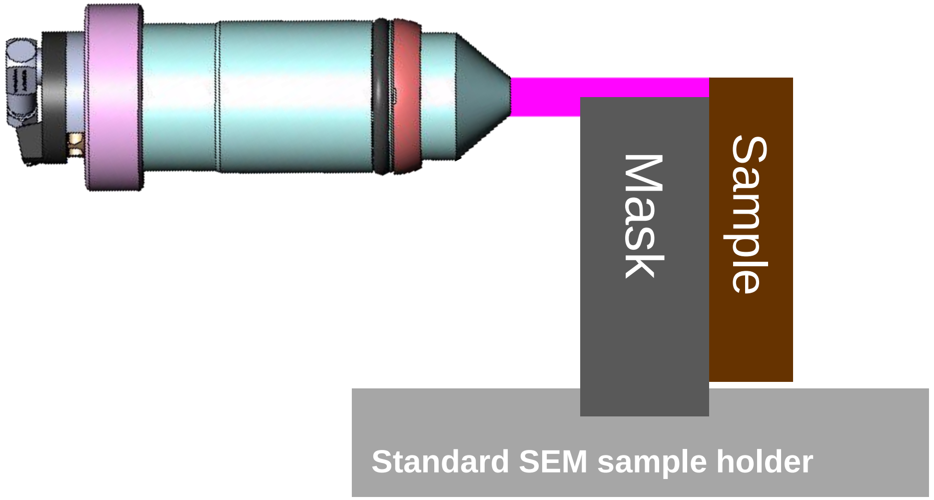
## A versatile argon broad ion beam mill for SEM applications


- Produces ideal results for both **planar and cross-section** samples
- **Wide-ranging ion energies** allow either rapid milling or gentle polishing on a broad variety of sample materials
- **Artifact-free samples** are readily produced
- **Easy-to-use interface**
- **Fully automated**, including precise sample height detection, for high-throughput applications
- Liquid nitrogen **stage cooling**
- **Vacuum / inert gas transfer** to SEM capability

# Model 1061 SEM Mill
## Milling geometries

| Planar milling | Cross-section milling |
| --- | --- |



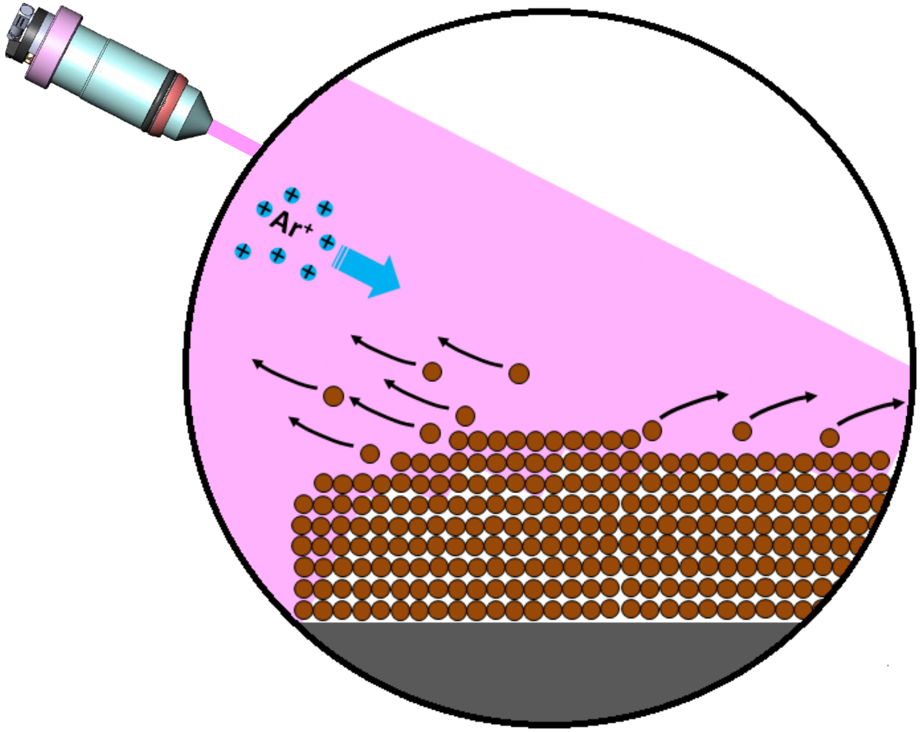




Ion energies range from 0.1 to 10 keV
Variable milling angle range: 0 to 10°
Stage movement: Continuous rotation or rocking

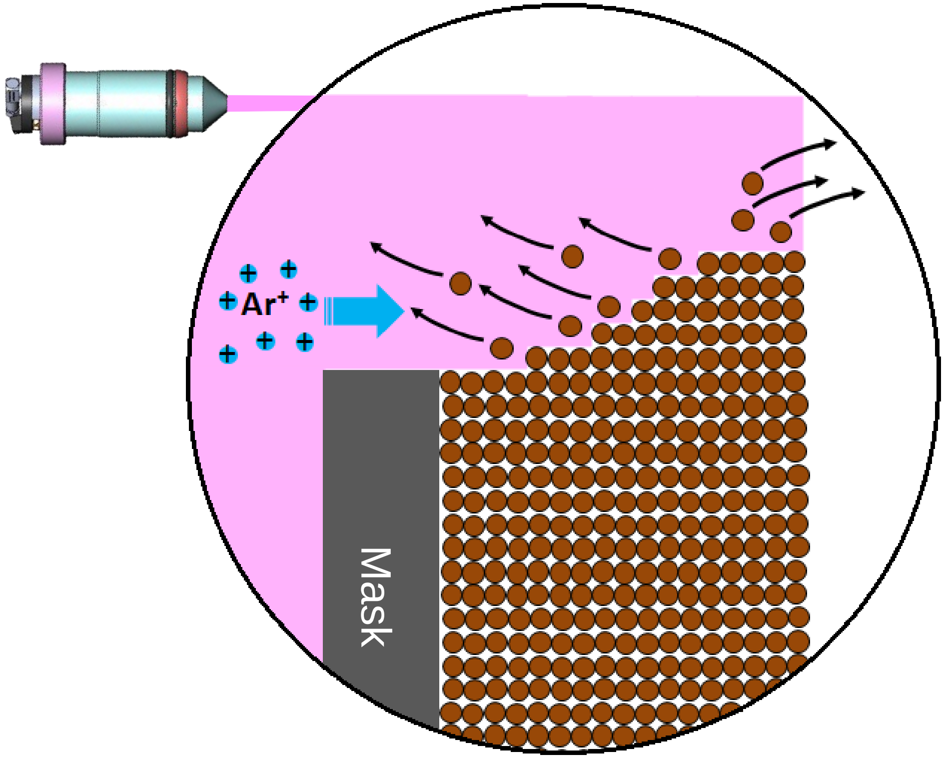
# Model 1061 SEM Mill

## Milling geometries

**Planar milling**

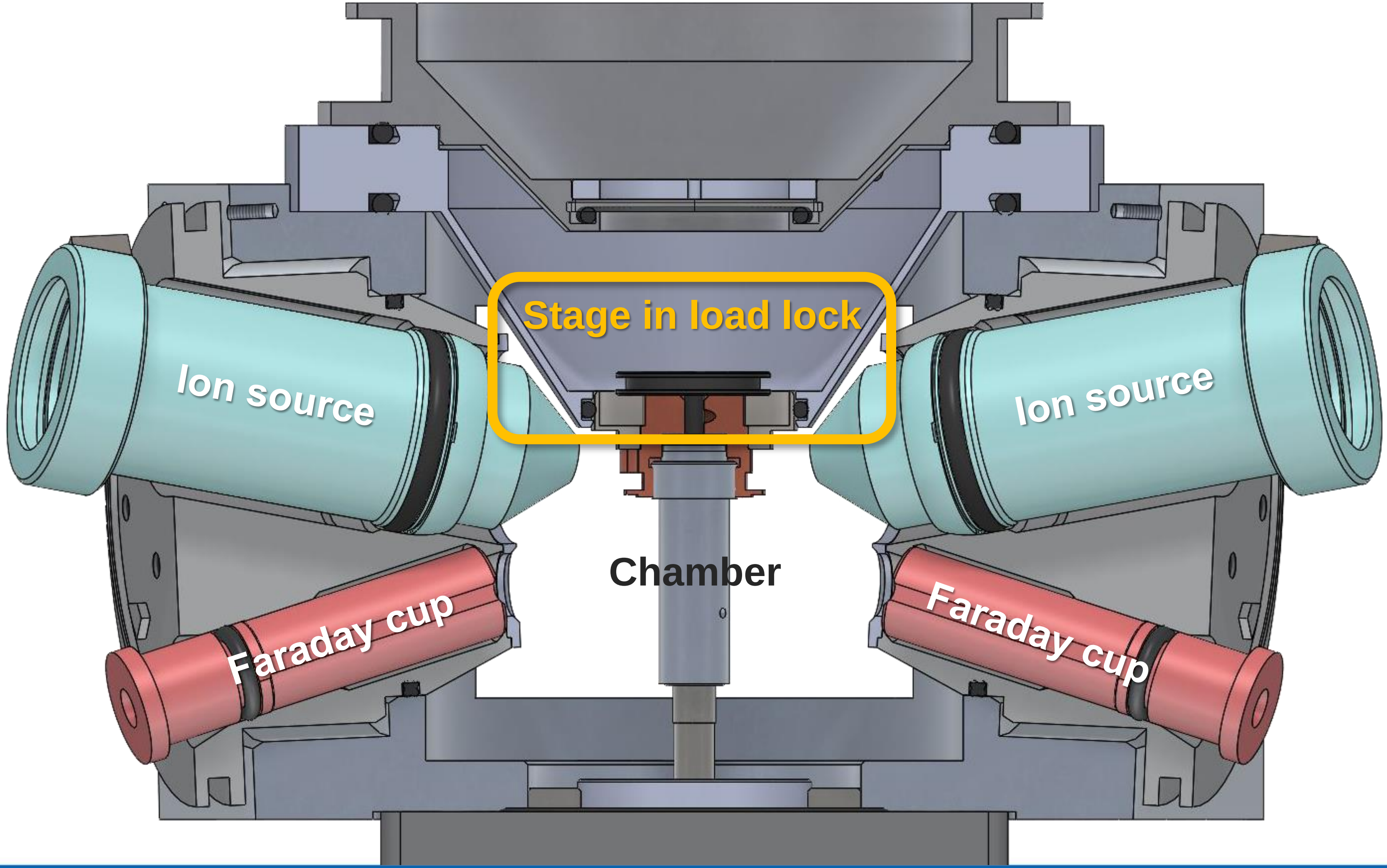



**Cross-section milling**



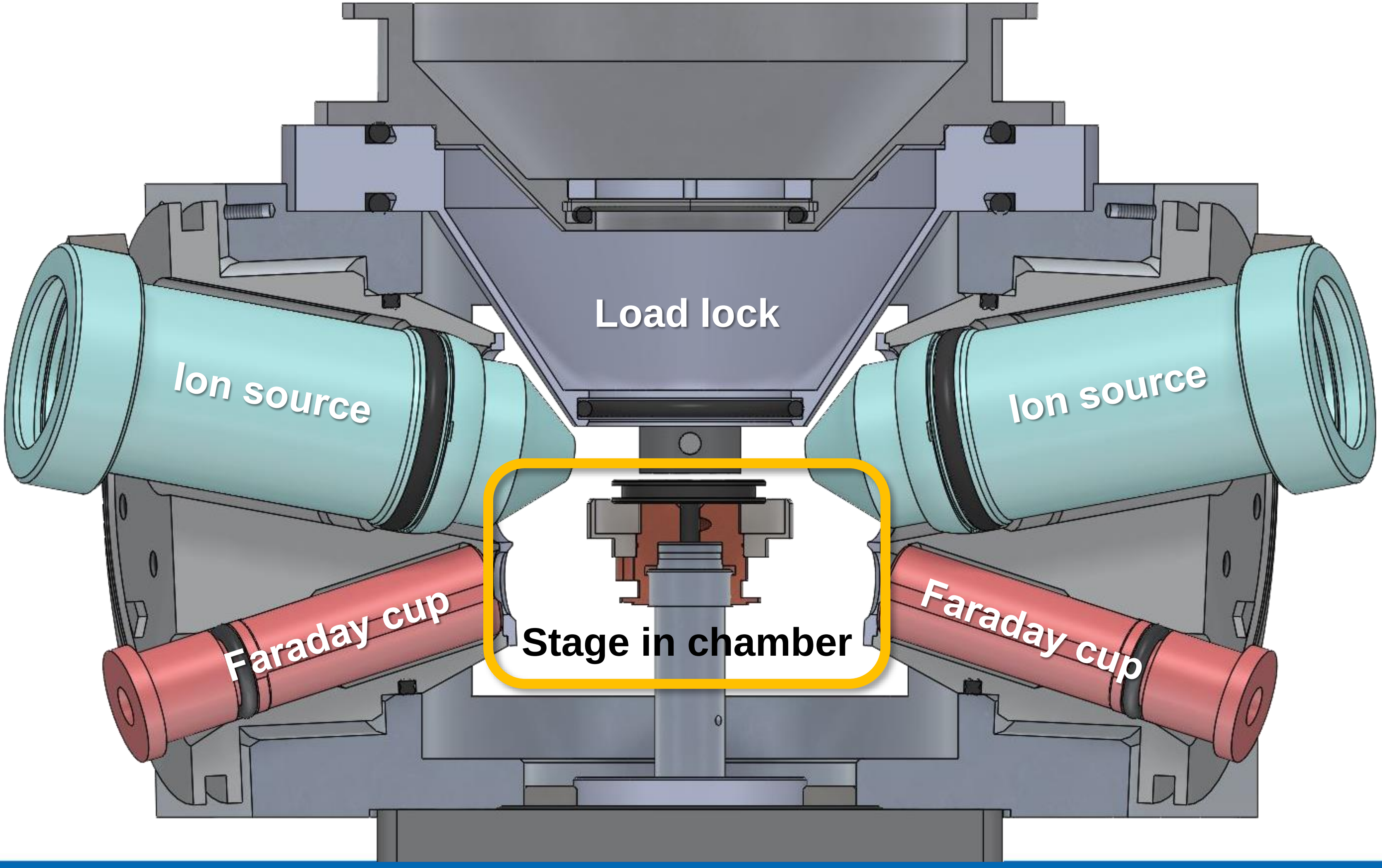
# Model 1061 SEM Mill

## Planar sample milling

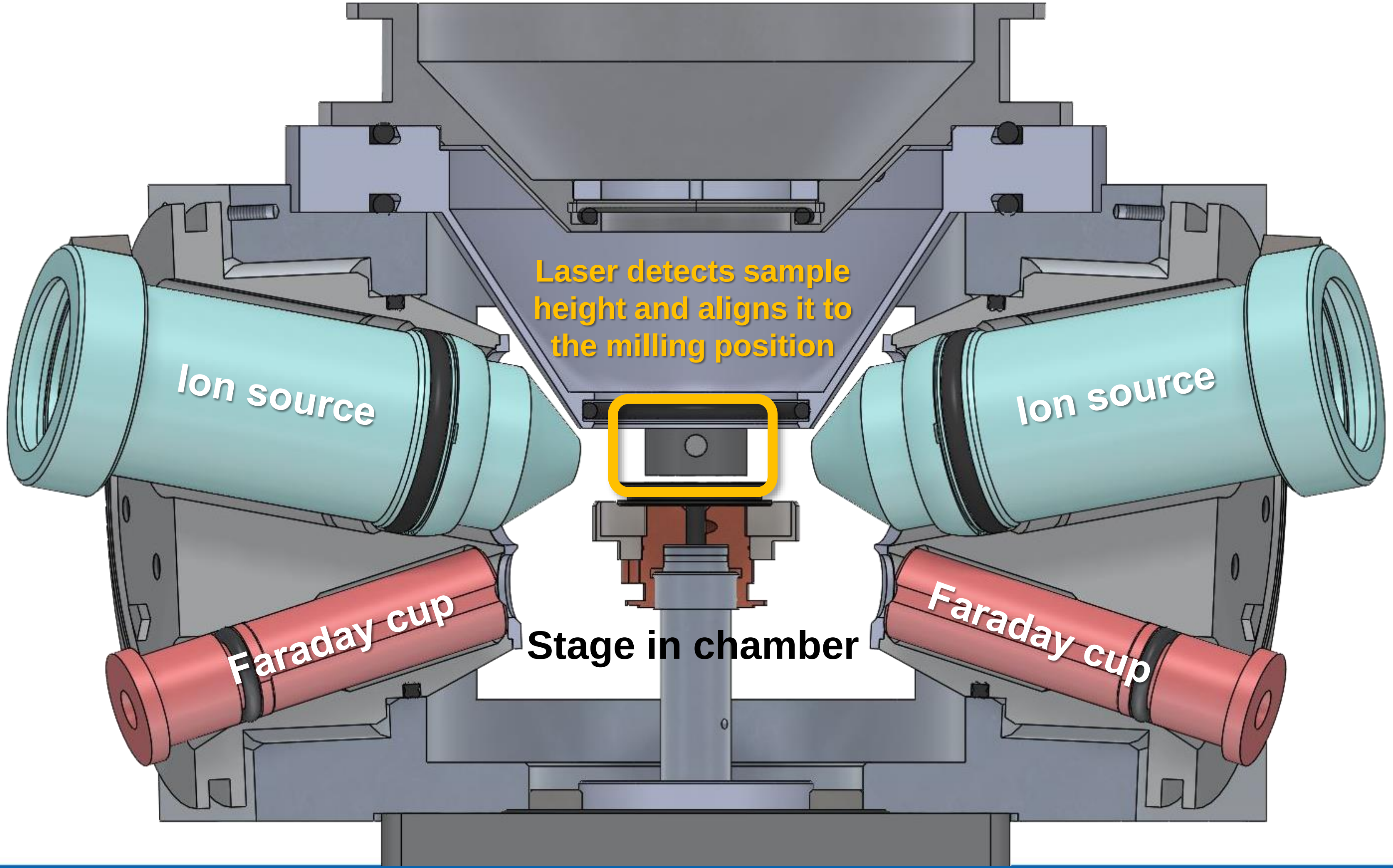


# Model 1061 SEM Mill

## Planar sample milling

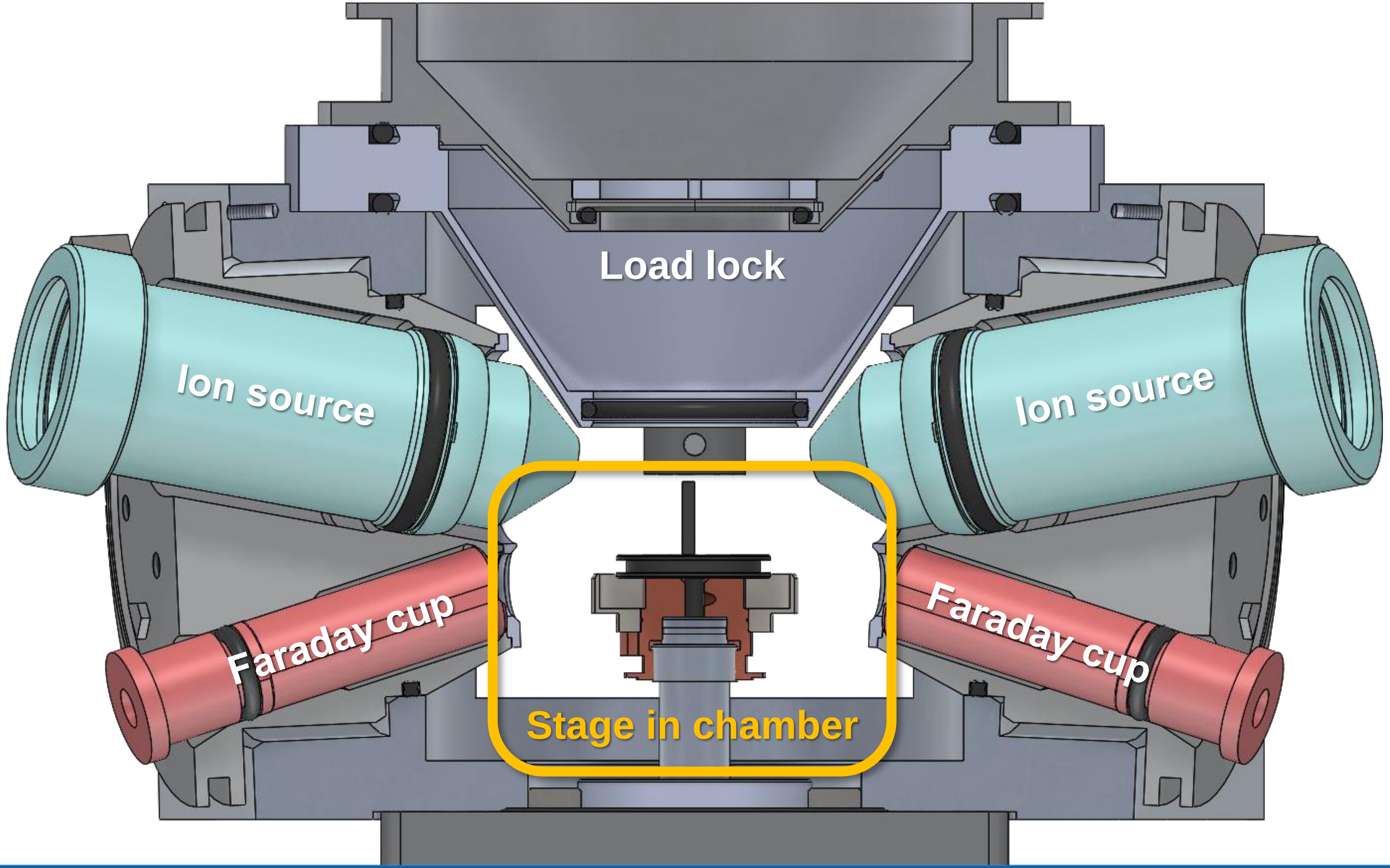


# Model 1061 SEM Mill
## Sample height detection



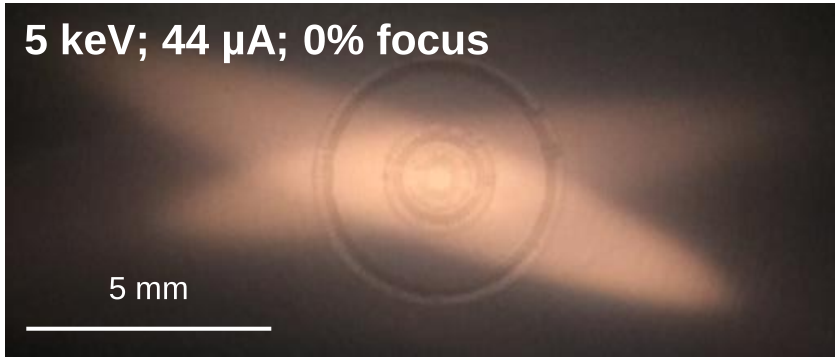
# Model 1061 SEM Mill

## Cross-section sample milling



# Model 1061 SEM Mill

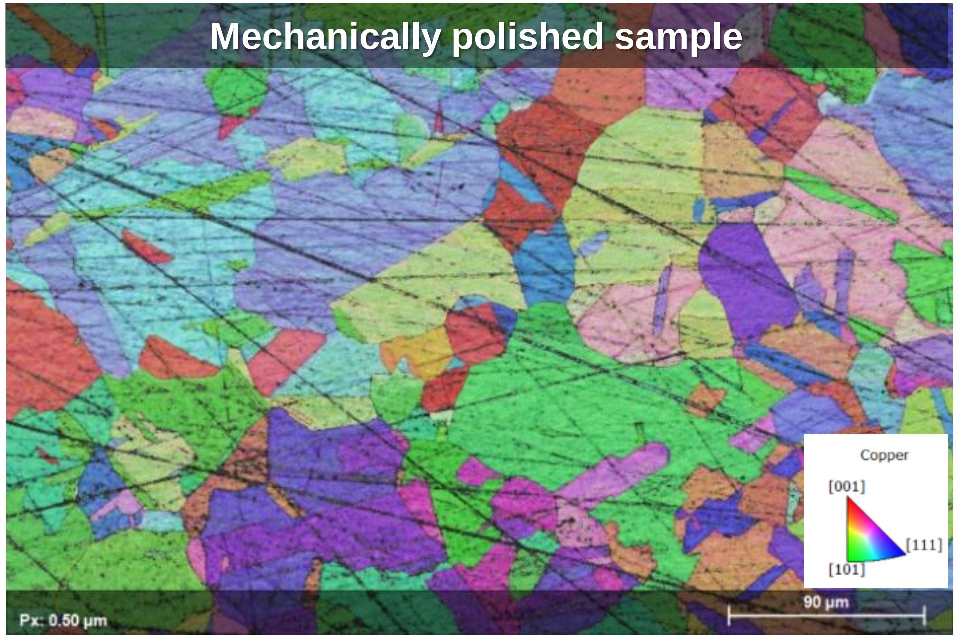
## TrueFocus ion sources



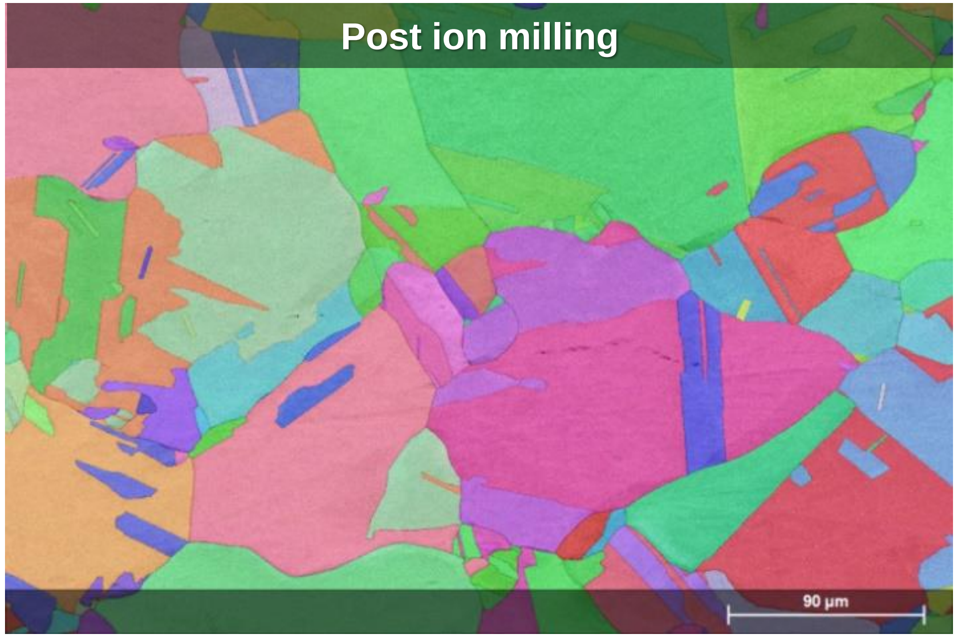



- Adjustable energy range from 100 eV to 10 keV
- Ion beam spot size ranges from 300 µm to 5 mm
- Consistent beam current
- Motorized ion sources enable touch screen control of beam angle adjustments

# Broad ion milling
## Sample preparation




The specimen surface must be deformation-free to conduct EBSD measurements.




Scratches and damages caused by mechanical polishing are removed by the ion milling process.

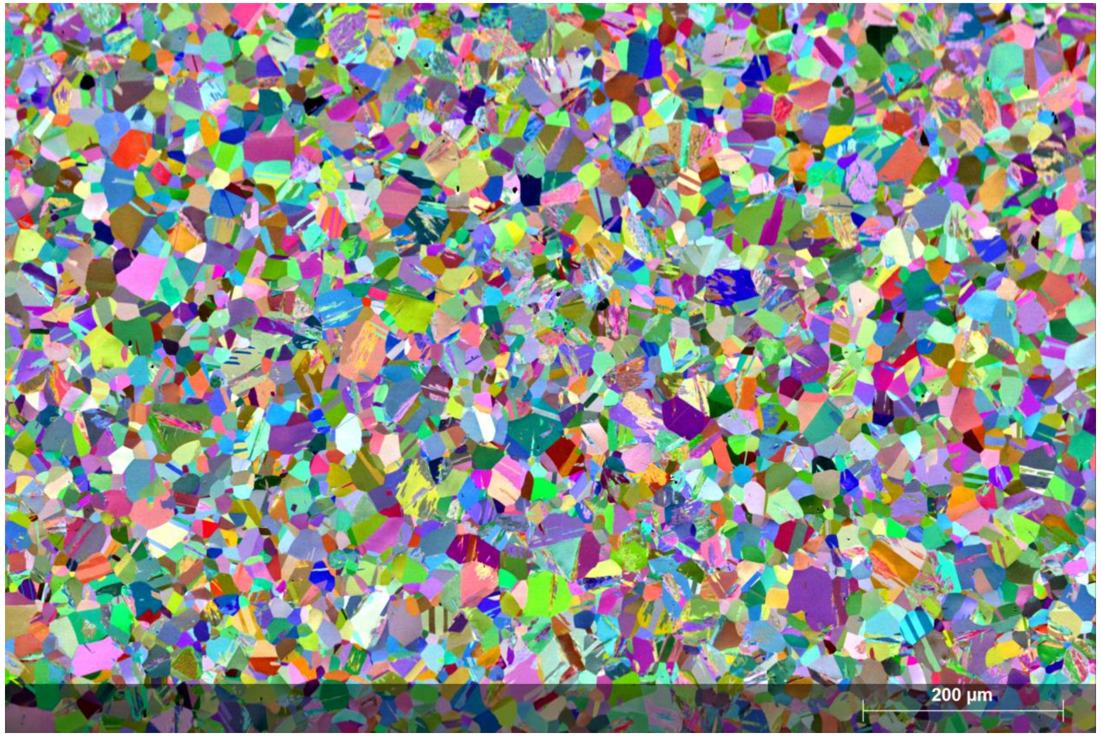
As a result, the material's microstructure is revealed, and statistical data can be acquired.

# Austenitic steels

# Austenitic 300 series

## Orientation contrast imaging with ARGUS™

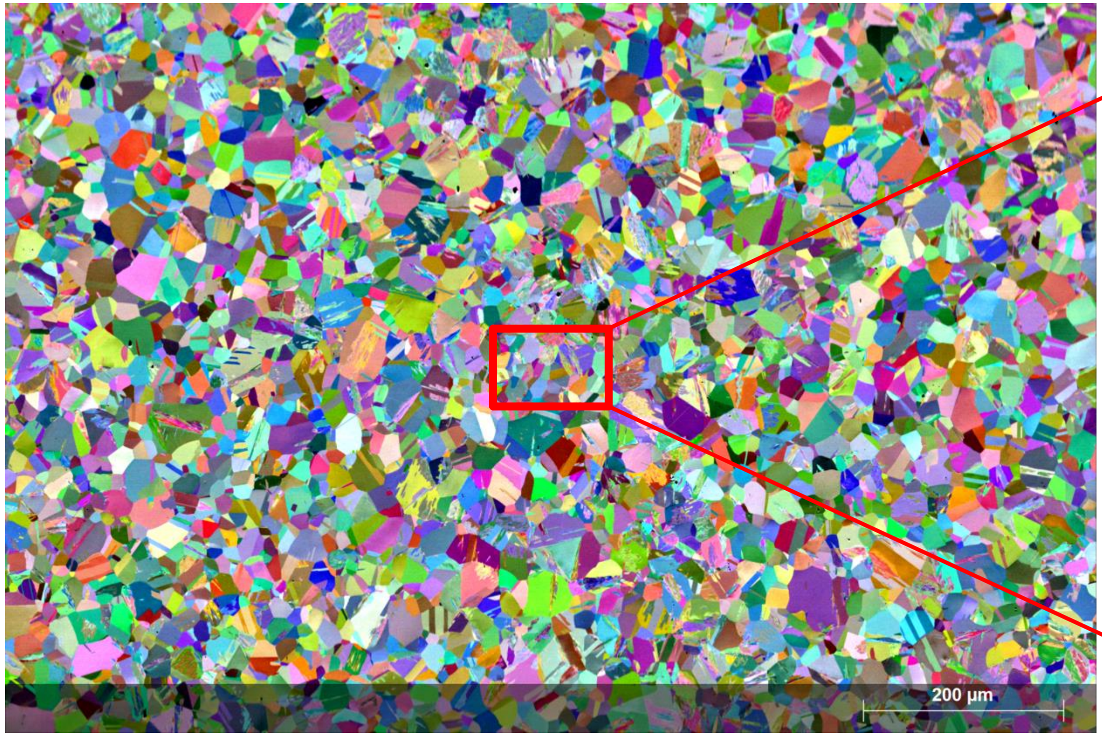
Mechanically polished using colloidal silica

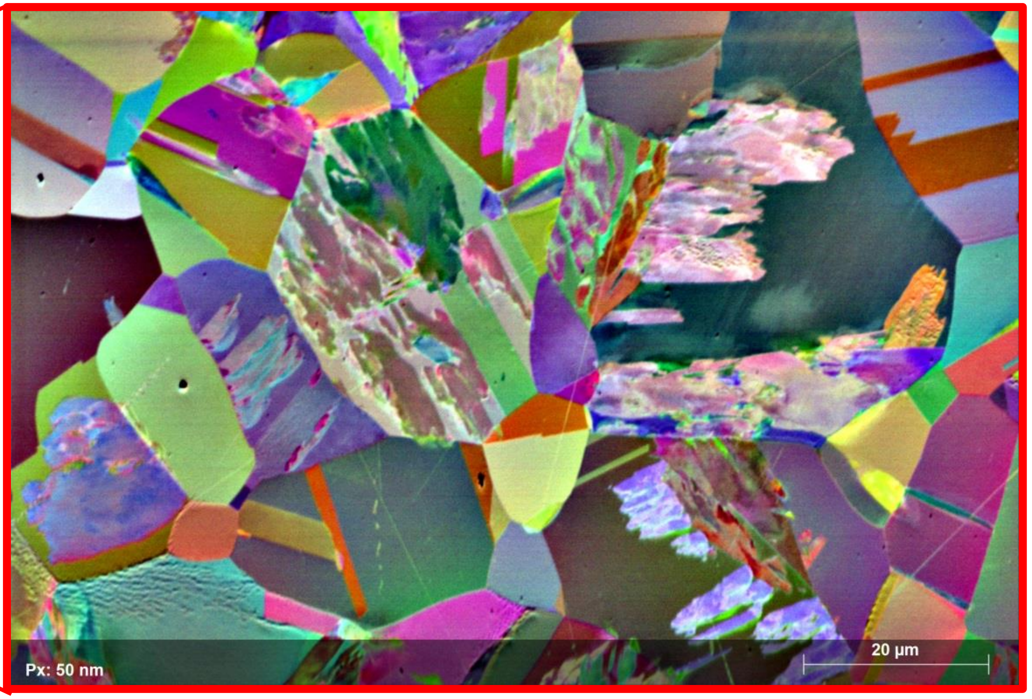


# Austenitic 300 series

## Orientation contrast imaging with ARGUS™

Mechanically polished using colloidal silica

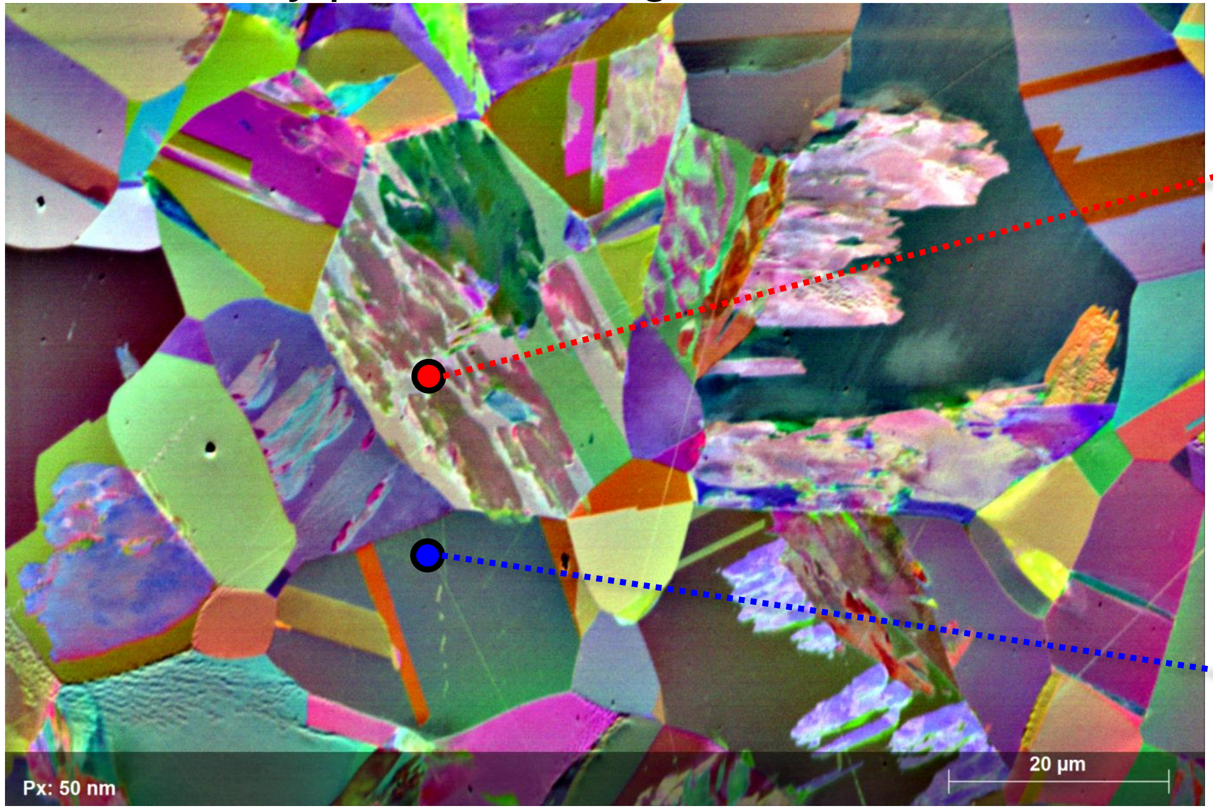


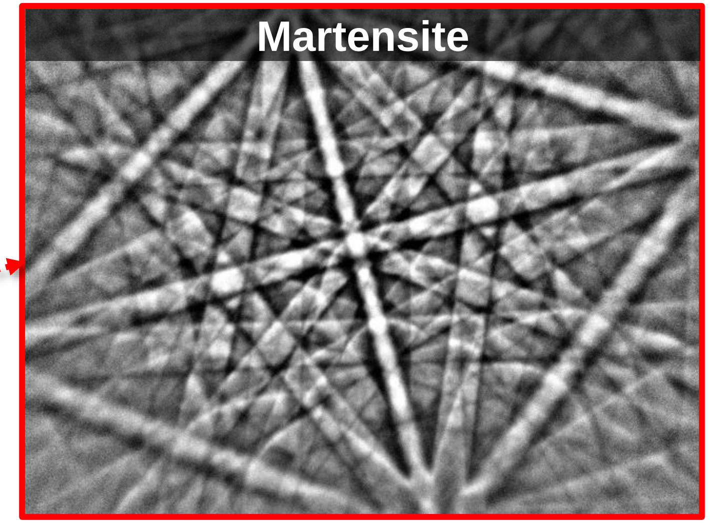
# Austenitic 300 series

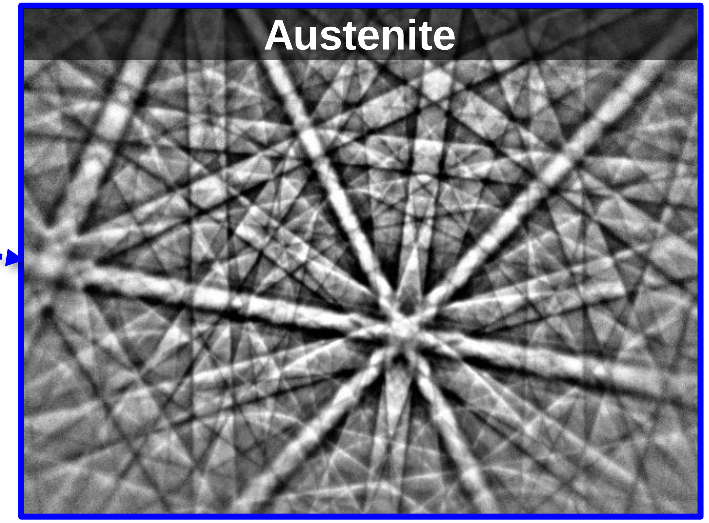
## Orientation contrast imaging with ARGUS™

Mechanically polished using colloidal silica



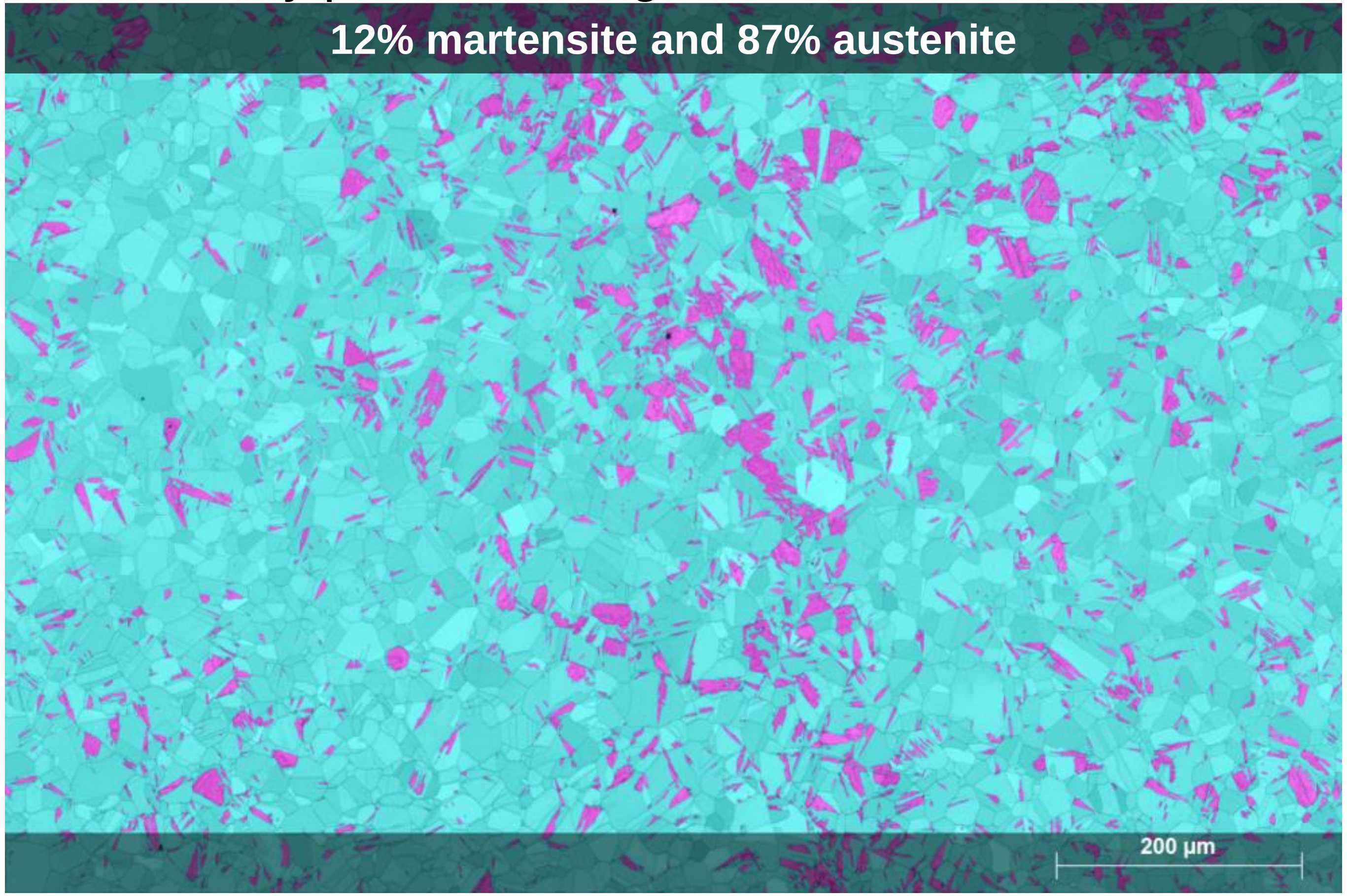

Dynamic strain-induced phase transformation of austenite to martensite during mechanical polishing



# Austenitic 300 series
## EBSD phase distribution map

Mechanically polished using colloidal silica




**Phase map**
Step size: 0.5 µm
Pattern resolution: 80x60 pixels
Acquisition speed: 950 Hz

Indexing rate: 99%

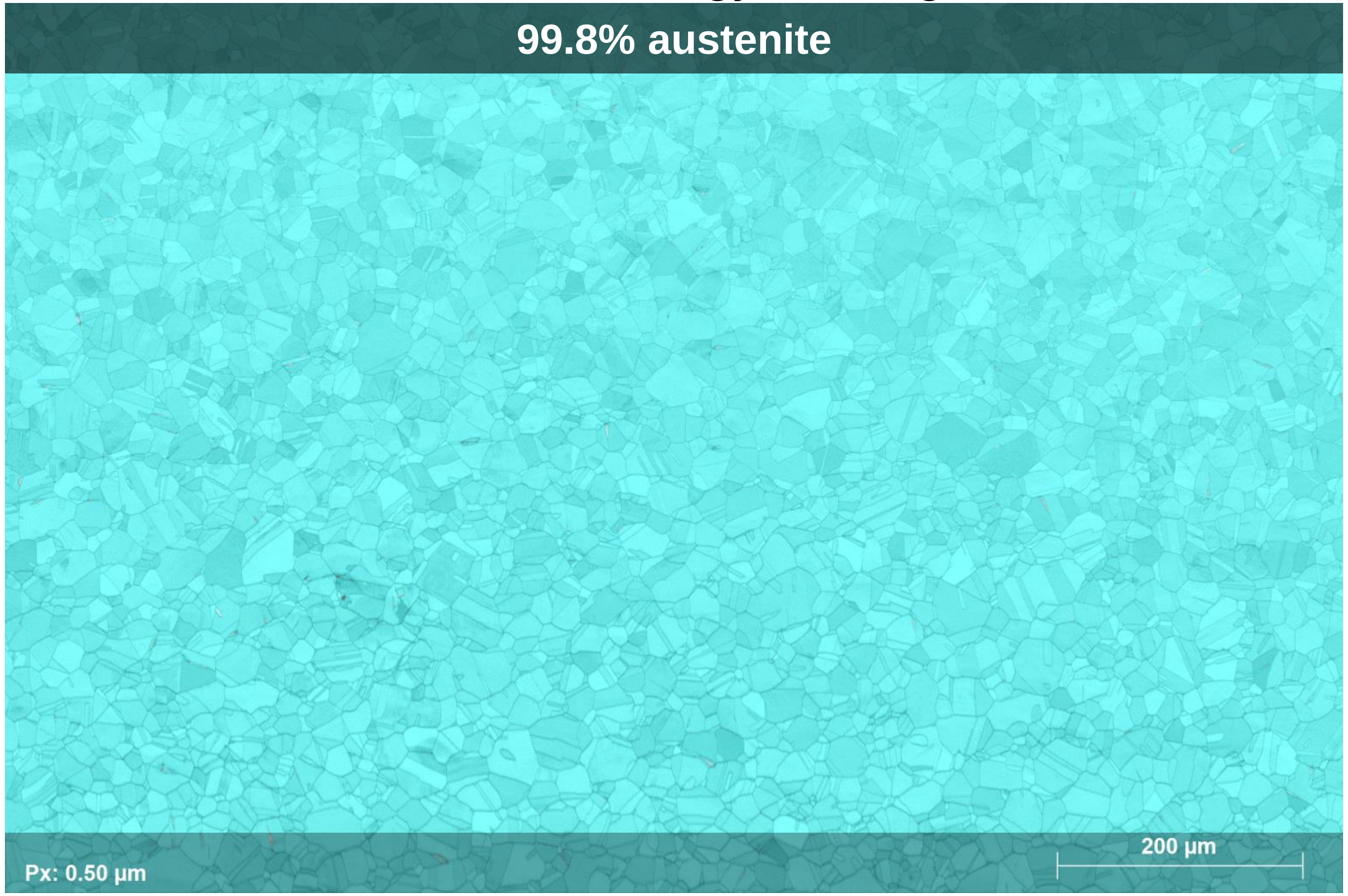
| | | |
|---|---|---|
| | Martensite | 12% |
| | Austenite | 87% |

Dynamic strain-induced phase transformation of austenite to martensite during mechanical polishing

# Austenitic 300 series

## EBSD phase distribution map

Ion milled at 5 and 2 keV energy; 2° angle




**Phase map**
Step size: 0.5 µm
Pattern resolution: 80x60 pixels
Acquisition speed: 950 Hz

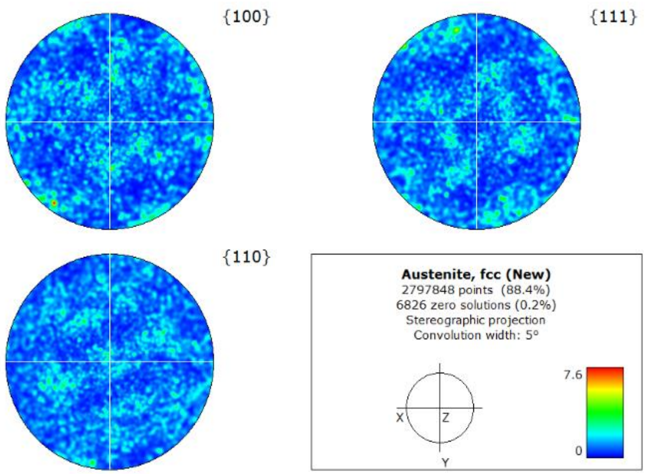
Indexing rate: 99.8%

| | | |
|---|---|---|
| | Martensite | 0% |
| | Austenite | 99.8% |

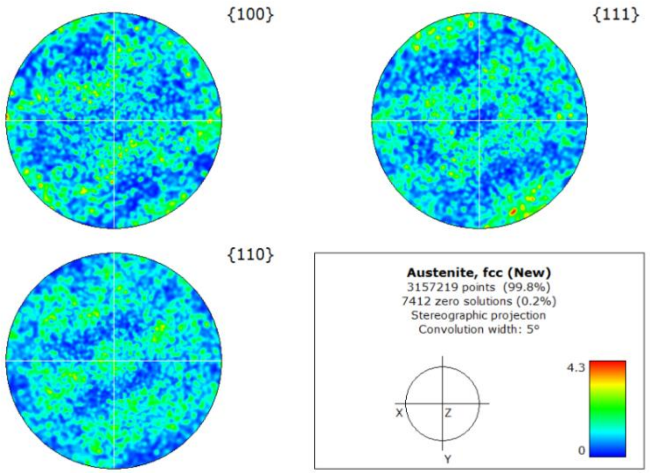
**No dynamic strain-induced phase transformation**

# Austenitic 300 series
## Texture

**Mechanically polished: colloidal silica**

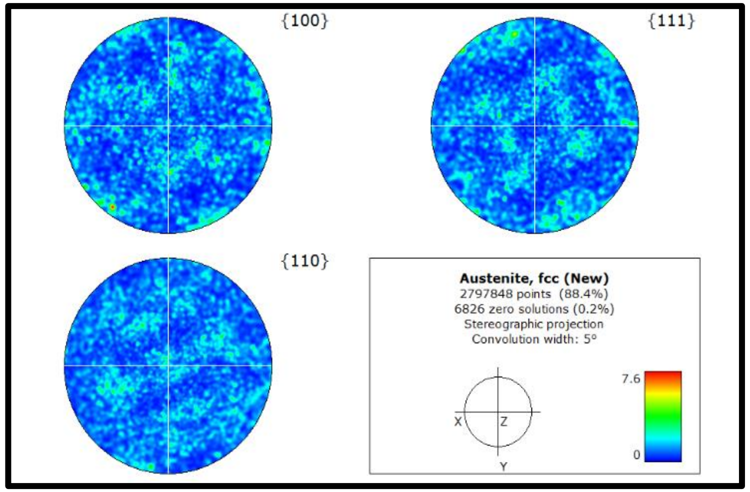


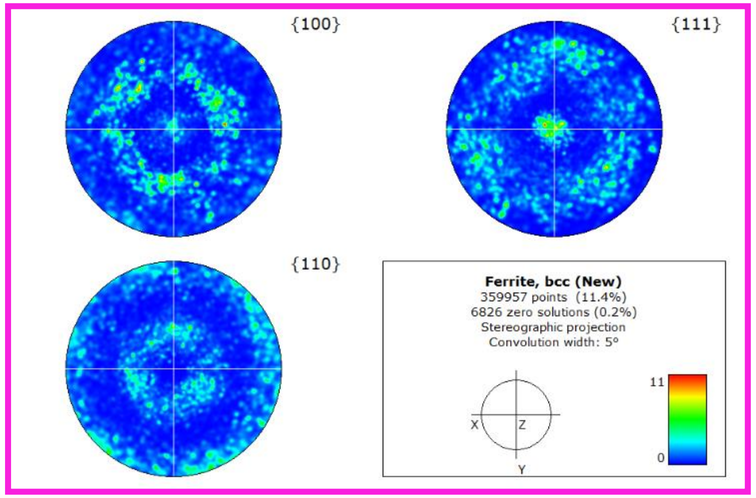

**Ion milled: 5 and 2 keV energy; 2° angle**



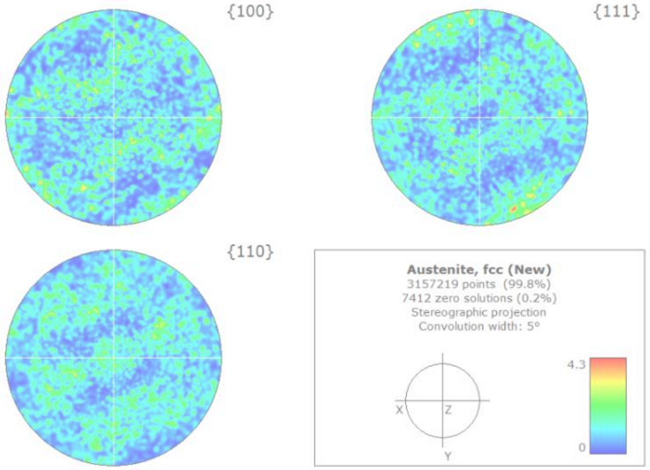
# Austenitic 300 series
## Texture

**Mechanically polished: colloidal silica**



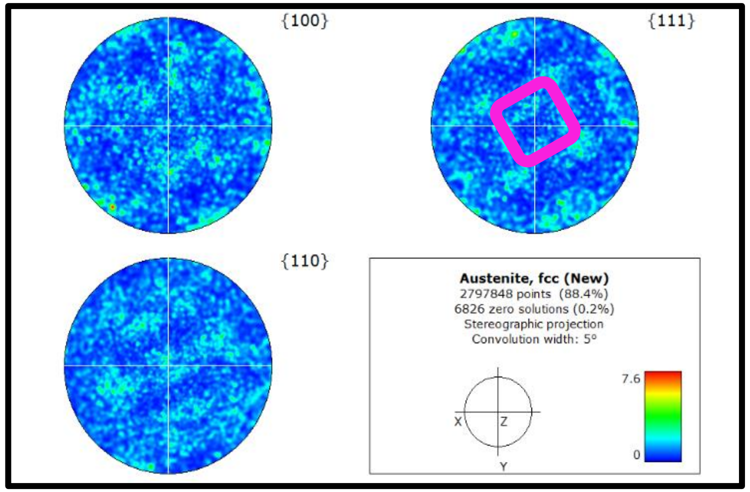

**Ion milled: 5 and 2 keV energy; 2° angle**

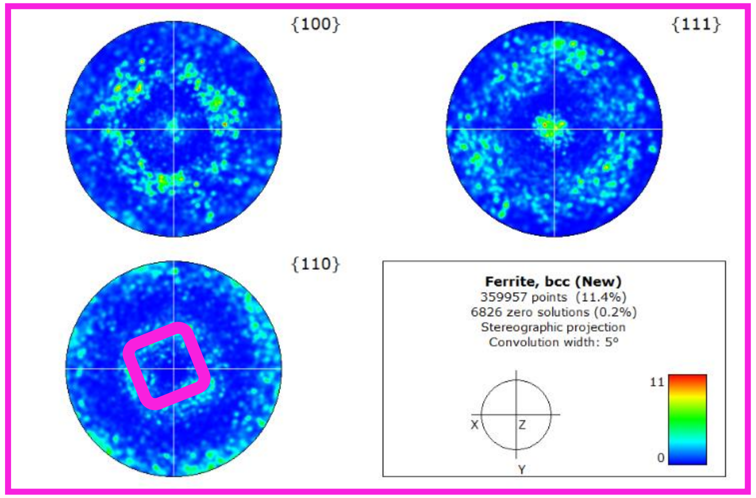


# Austenitic 300 series

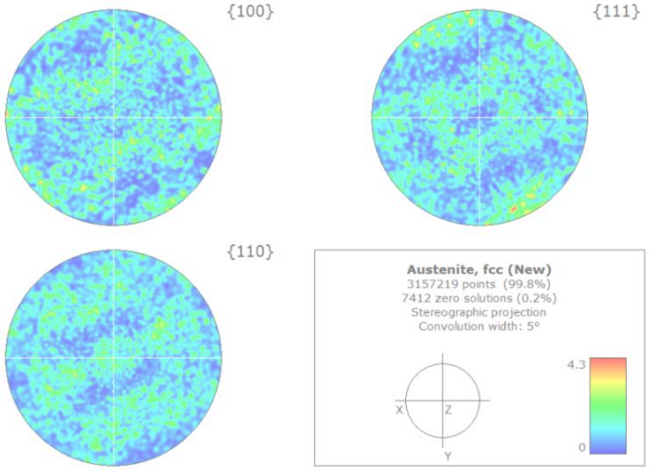
## Texture

**Mechanically polished: colloidal silica**




**Ion milled: 5 and 2 keV energy; 2° angle**

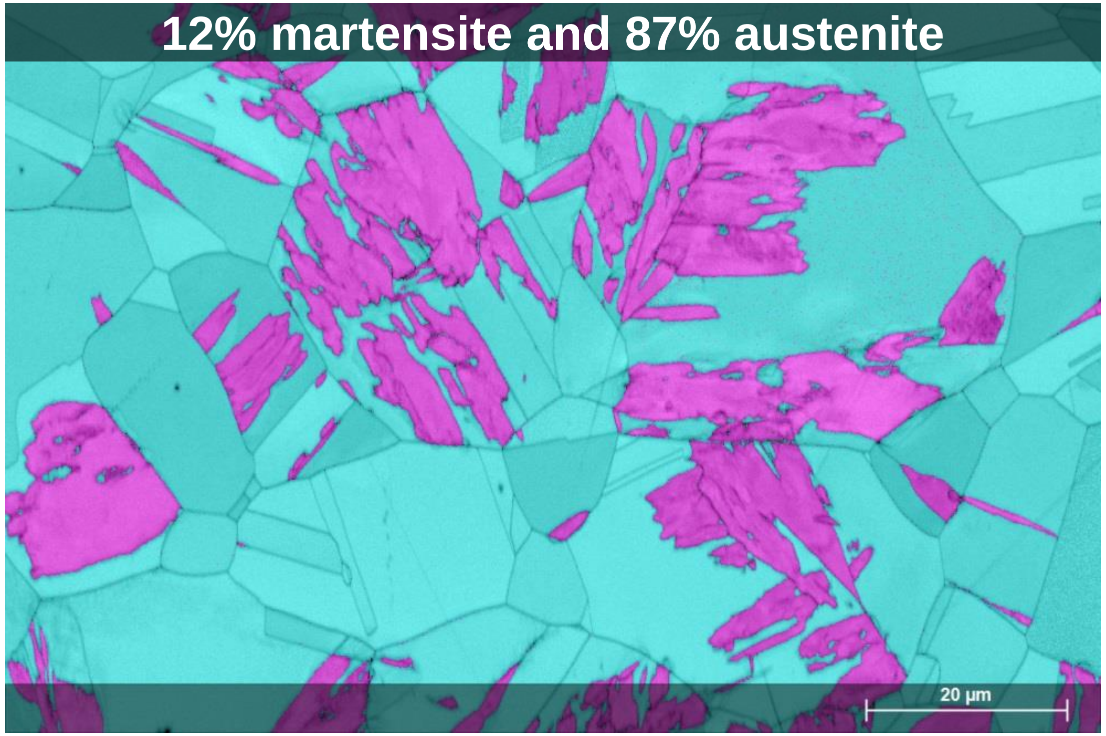



Kurdjumov–Sachs orientation relationship
$\{111\}_{\gamma} \,||\, \{110\}_{\alpha'}$ and $\mathbf{<1\text{-}10>}_{\gamma} \,||\, \mathbf{<11\text{-}1>}_{\alpha'}$

# Austenitic 300 series

## EBSD phase distribution map

Mechanically polished: colloidal silica




Ion milled: 5 and 2 keV energy; 2° angle

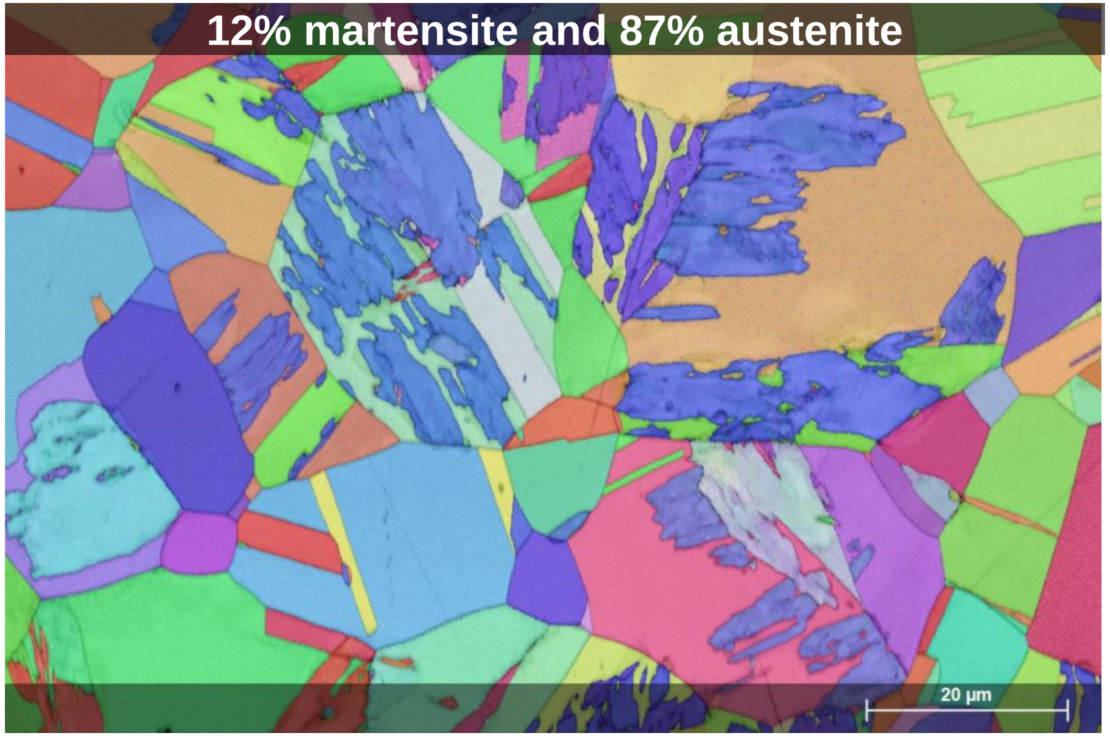

Step size: 100 nm
Pattern resolution: 160x120 pixels
Indexing rate: 99%

Martensite
Austenite

# Austenitic 300 series

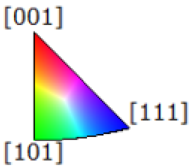
## EBSD IPF map

Mechanically polished: colloidal silica




Ion milled: 5 and 2 keV energy; 2° angle

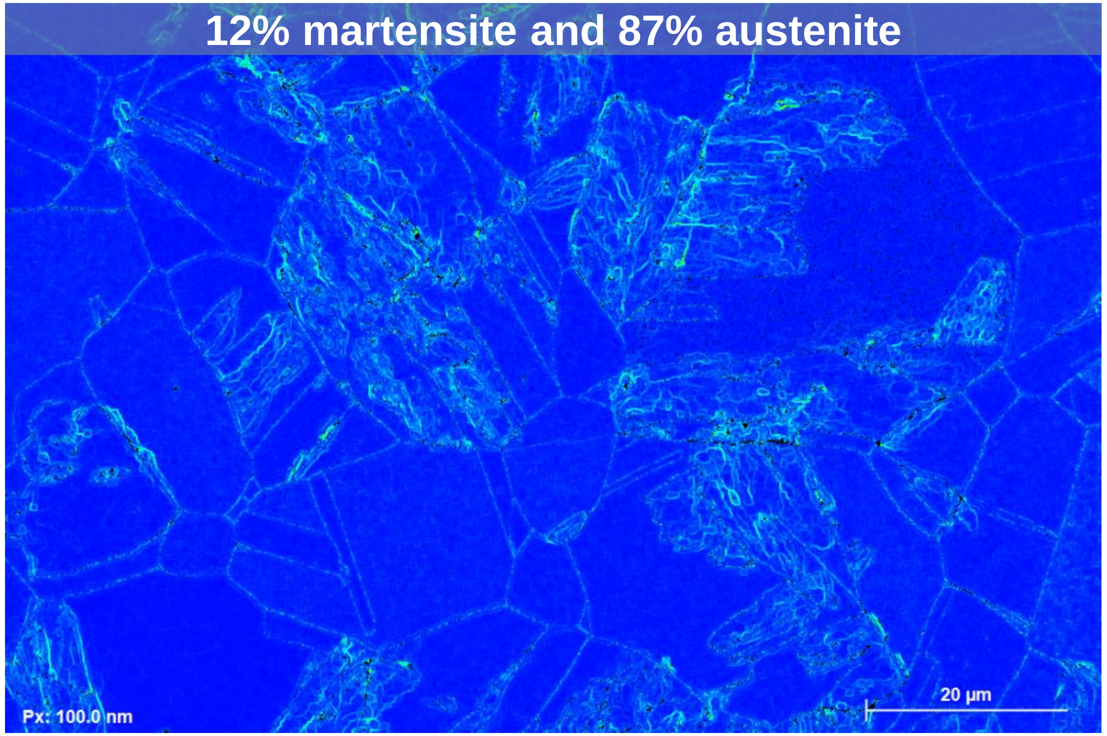

Step size: 100 nm
Pattern resolution: 160x120 pixels
Indexing rate: 99%



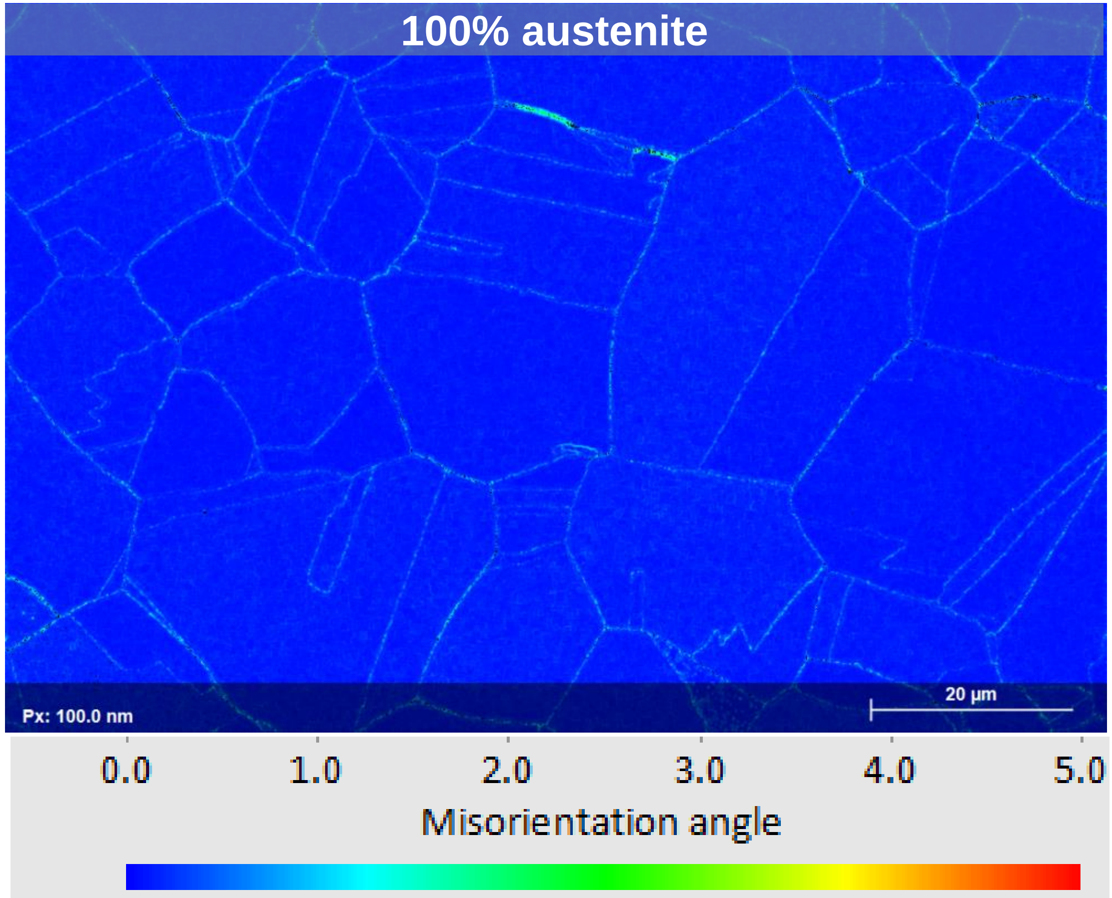
# Austenitic 300 series

## Kernel average misorientation map

Mechanically polished: colloidal silica




Ion milled: 5 and 2 keV energy; 2° angle



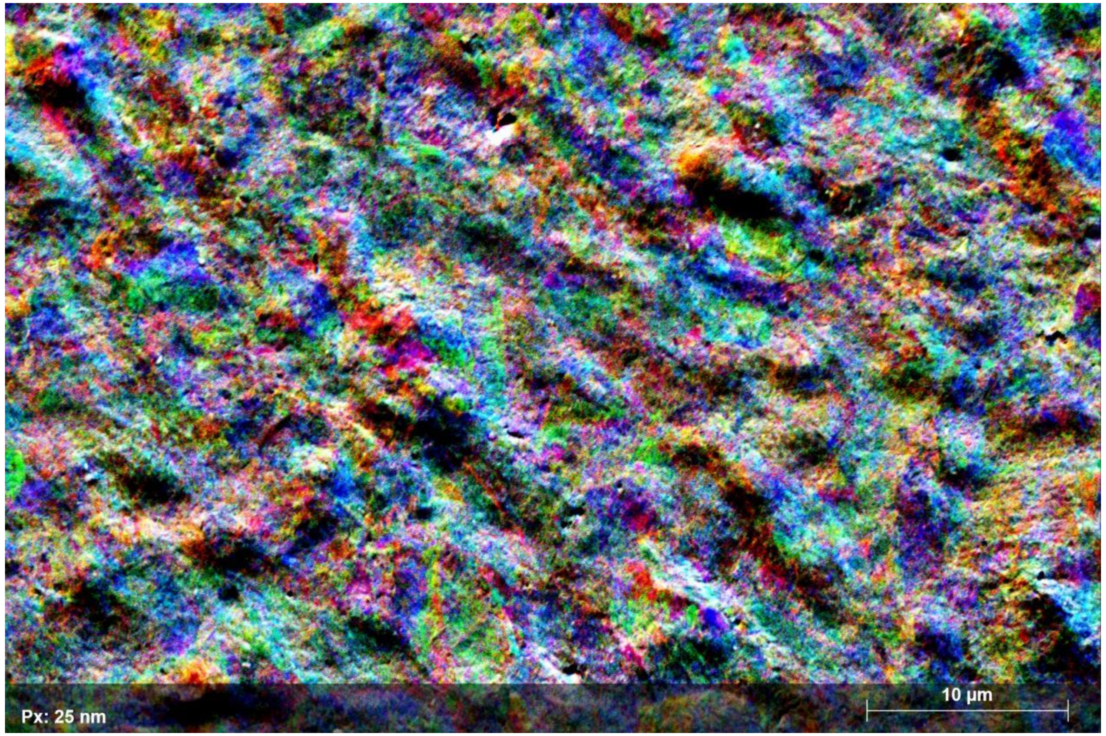

Step size: 100 nm
Pattern resolution: 160x120 pixels
Indexing rate: 99%

# Zirconium alloy

# Zirconium 702 alloy

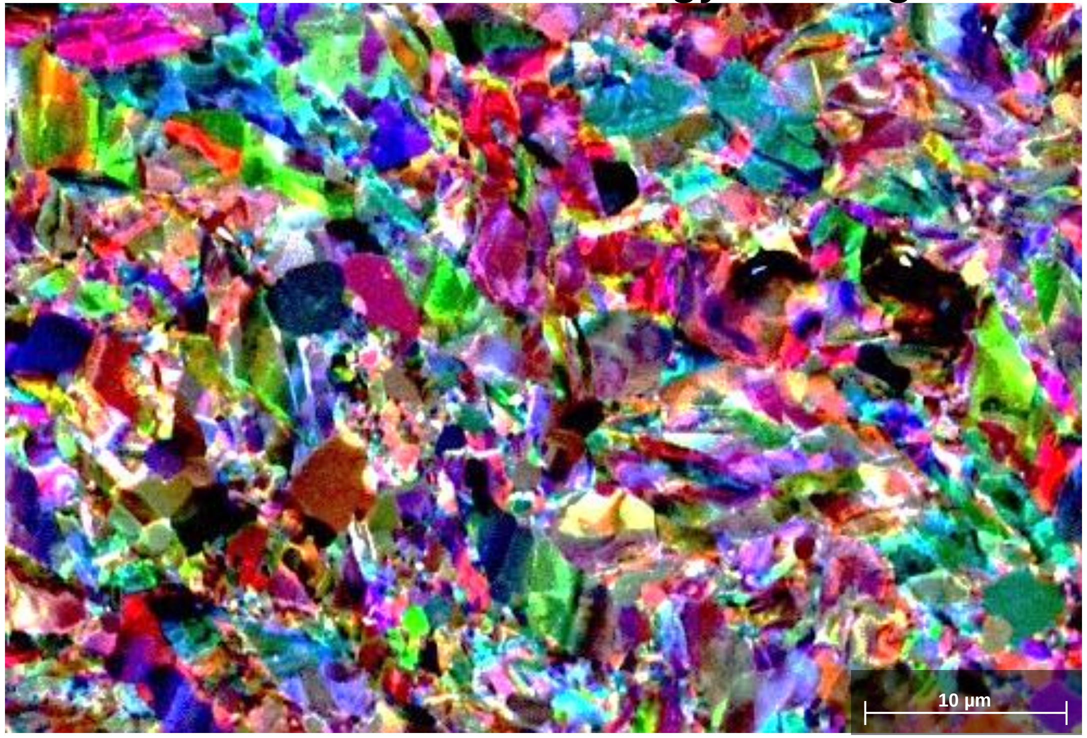
## Orientation contrast imaging with ARGUS™

Mechanically polished: colloidal silica




The microstructure is not visible

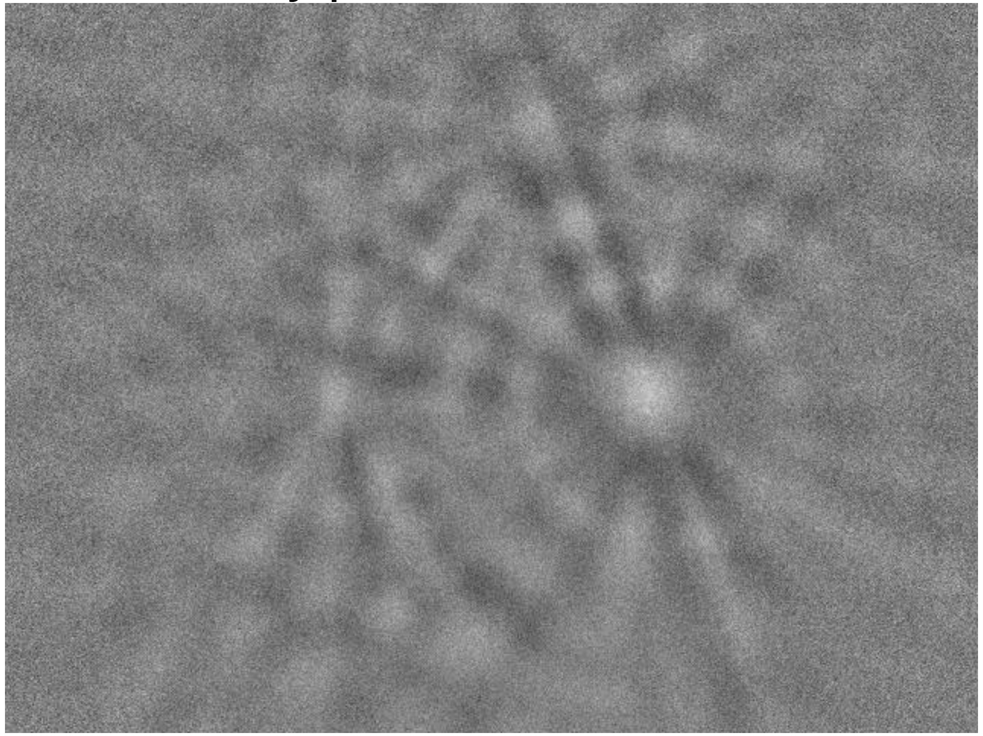
Ion milled: 8 and 2 keV energy; 3° angle




The fine microstructure is revealed (inter- and intragranular)

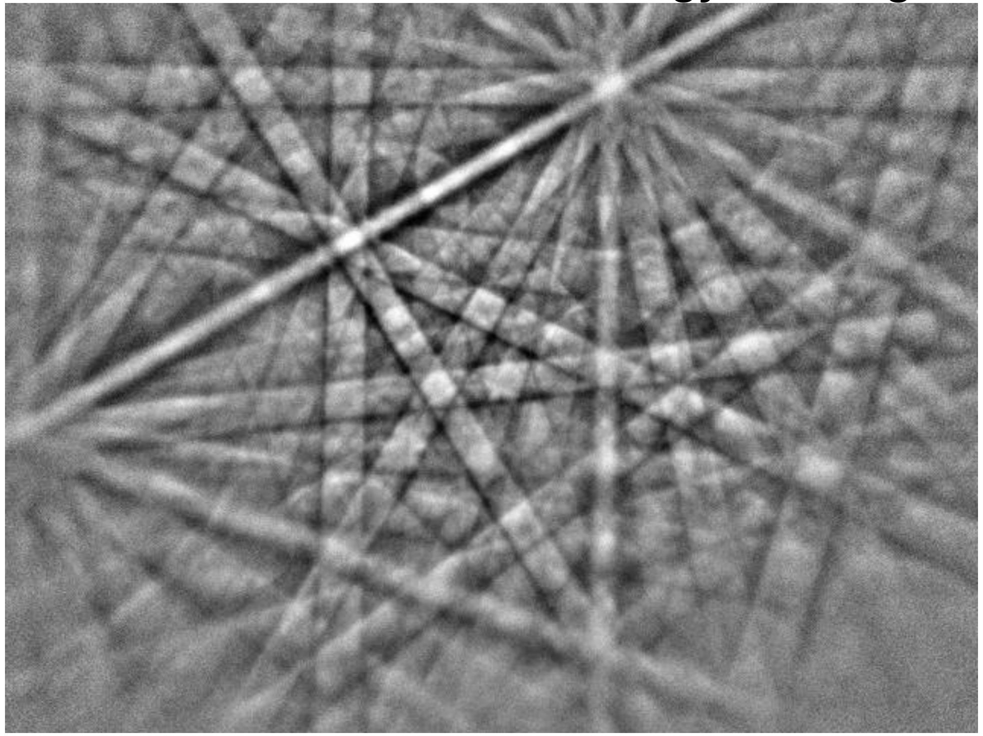
# Zirconium 702 alloy
## EBSD patterns

Mechanically polished: colloidal silica



Very few patterns

Ion milled: 8 and 2 keV energy; 3° angle

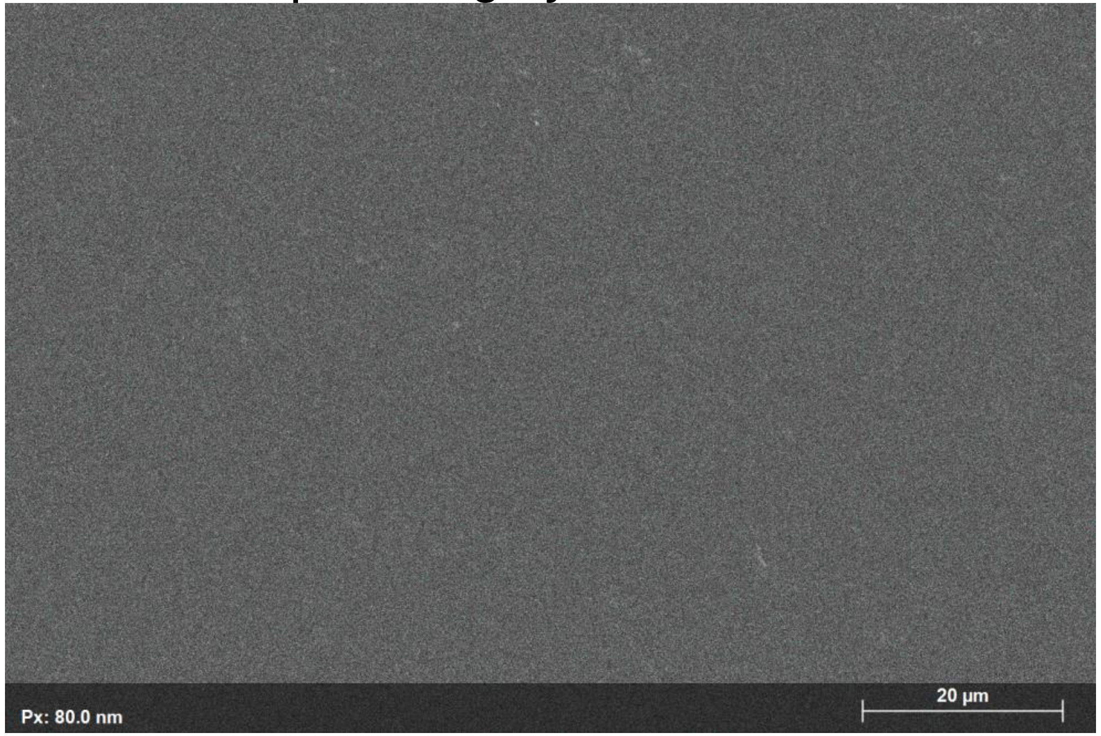


Many excellent quality patterns

# Zirconium 702 alloy

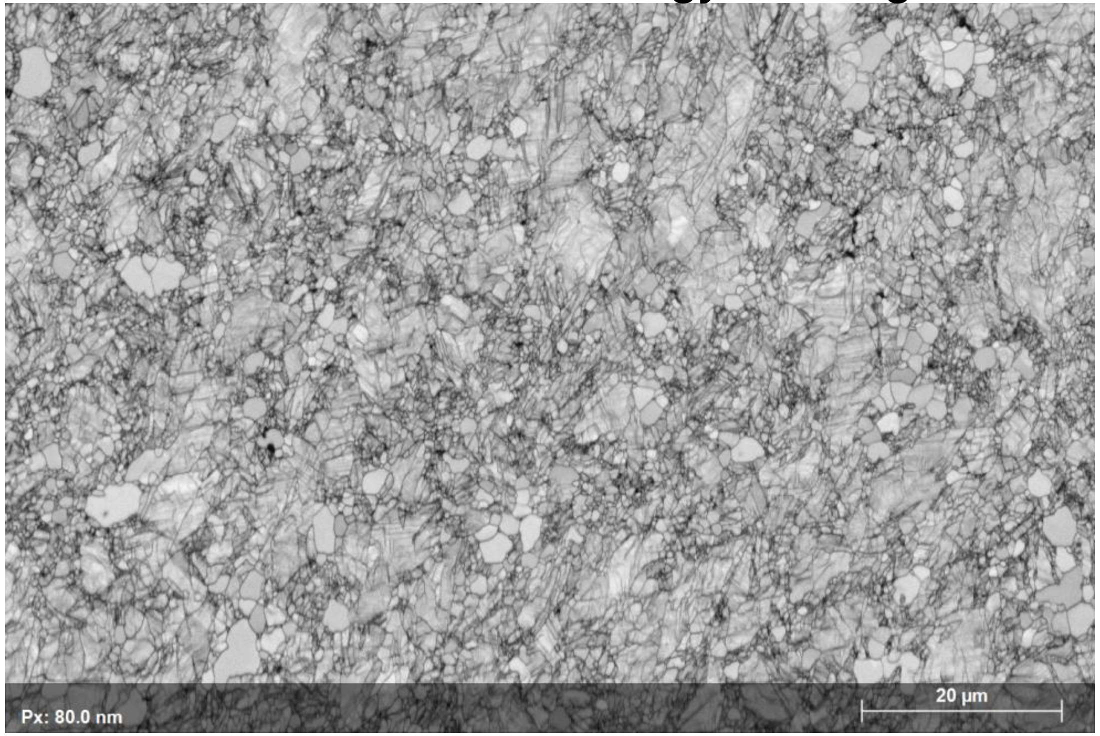
## Pattern quality EBSD maps

Mechanical polishing by colloidal silica




Indexing rate: < 1%

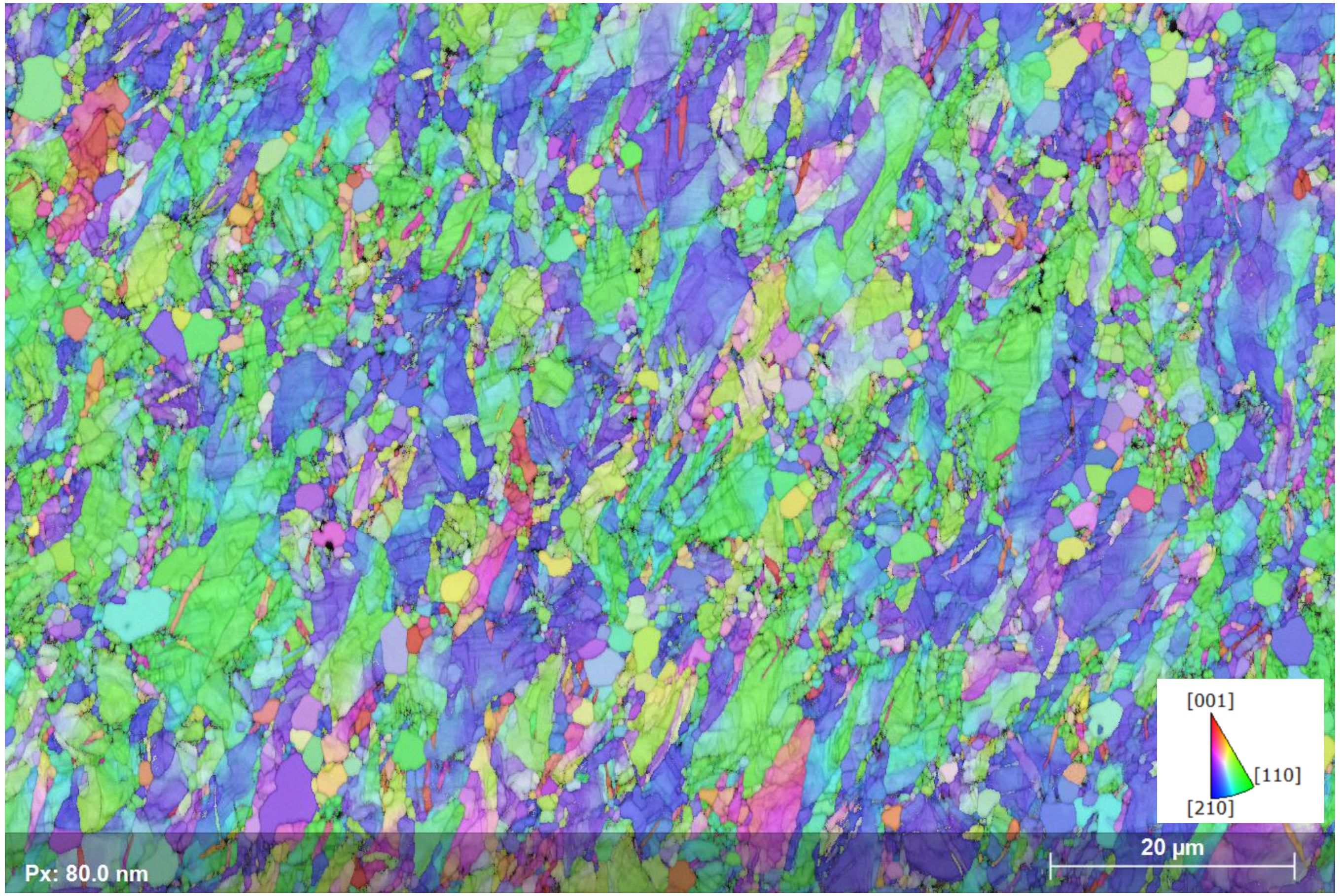
Ion milled: 8 and 2 keV energy; 3° angle



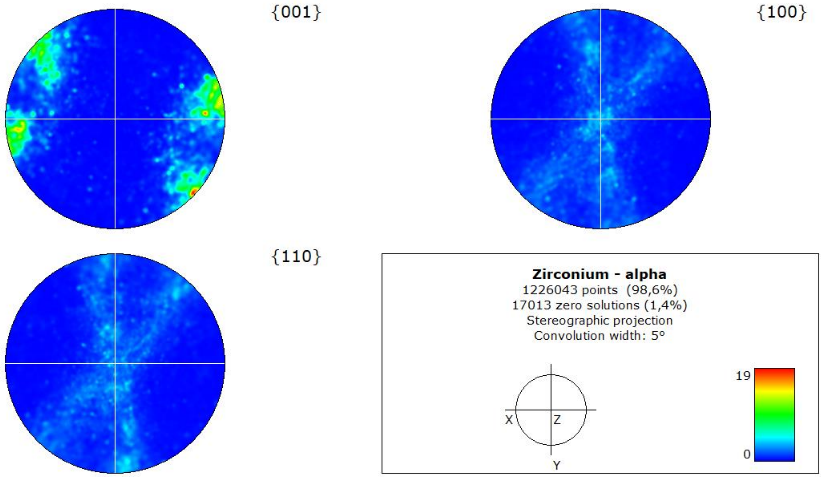

Indexing rate: 98.6%

# Zirconium 702 alloy
## EBSD IPF maps

Ion milled: 8 and 2 keV energy; 3° angle



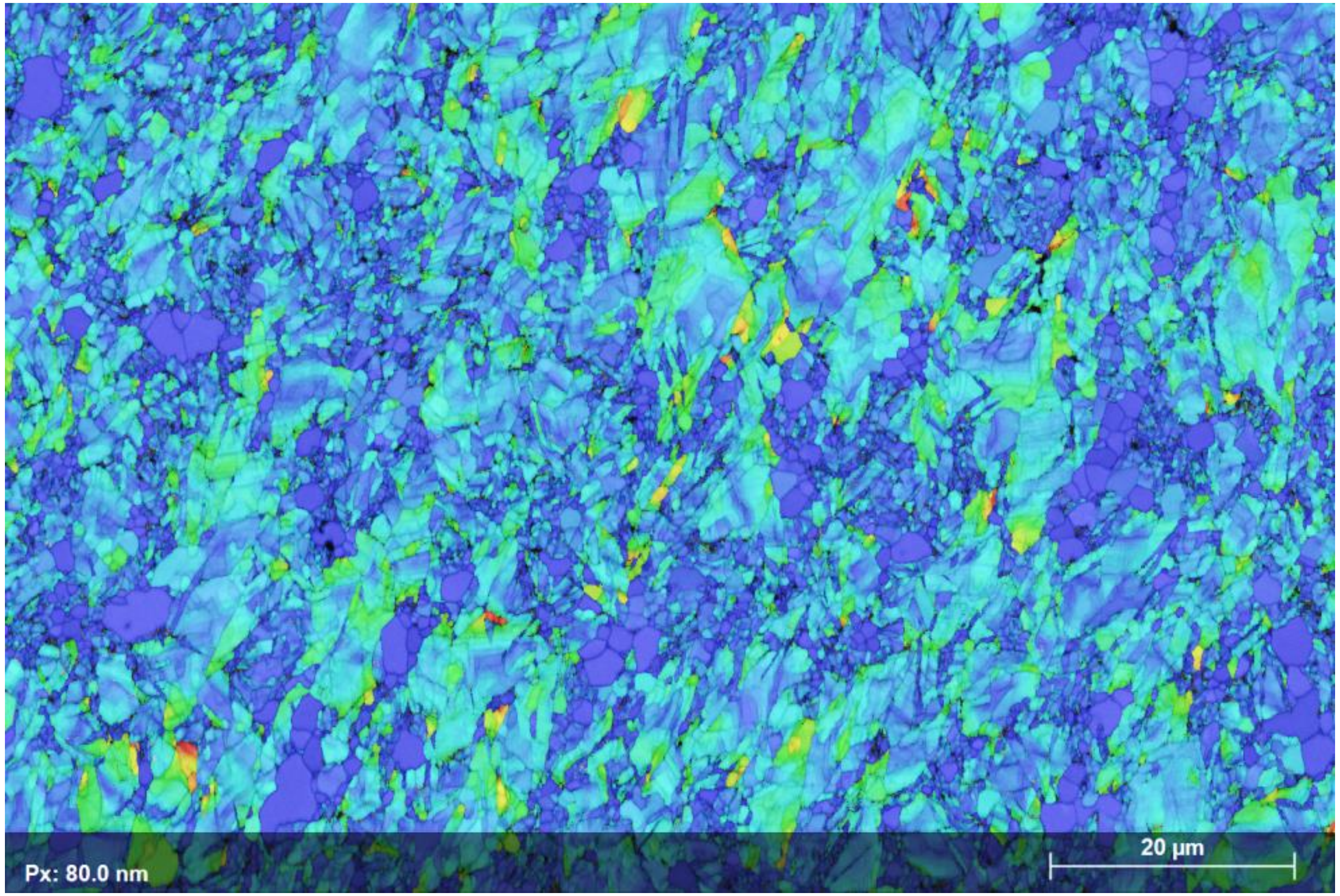

**IPF Z map**
Step size: 80 nm
Indexing rate: 98.6%



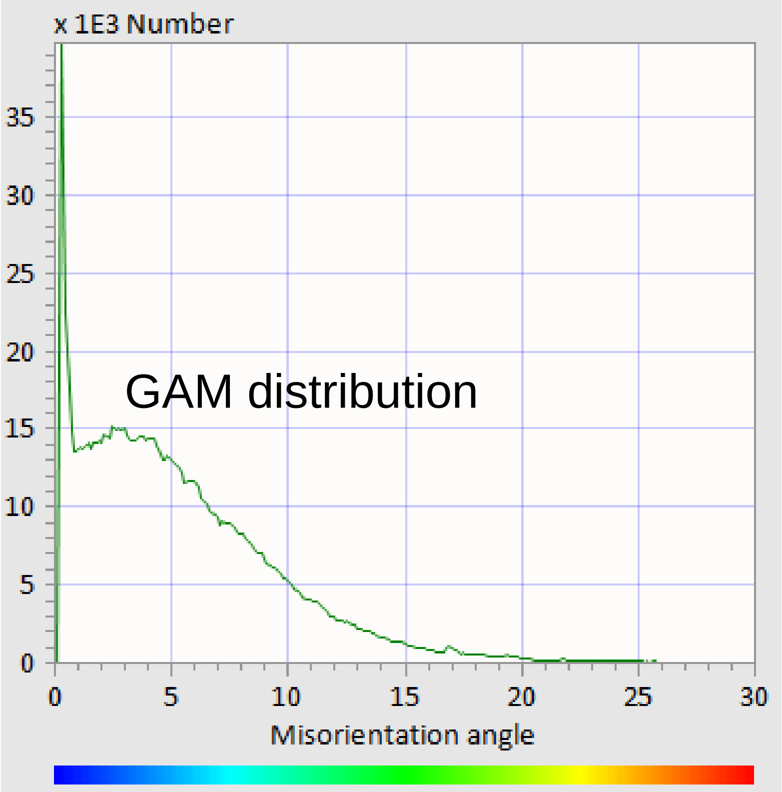
# Zirconium 702 alloy

## Grain average misorientation: Strain localization

Ion milled: 8 and 2 keV energy; 3° angle



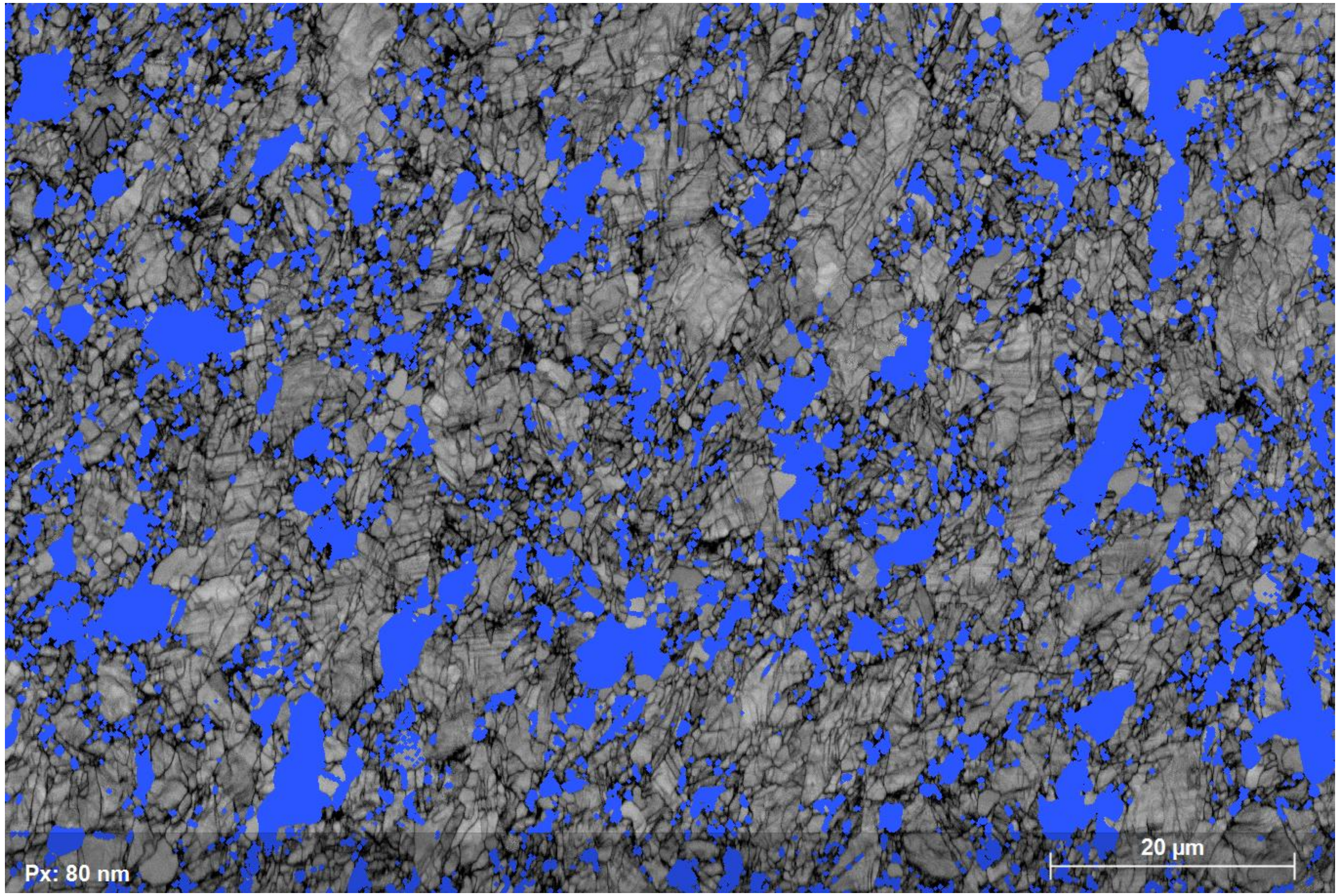

No artifact is observed after ion milling



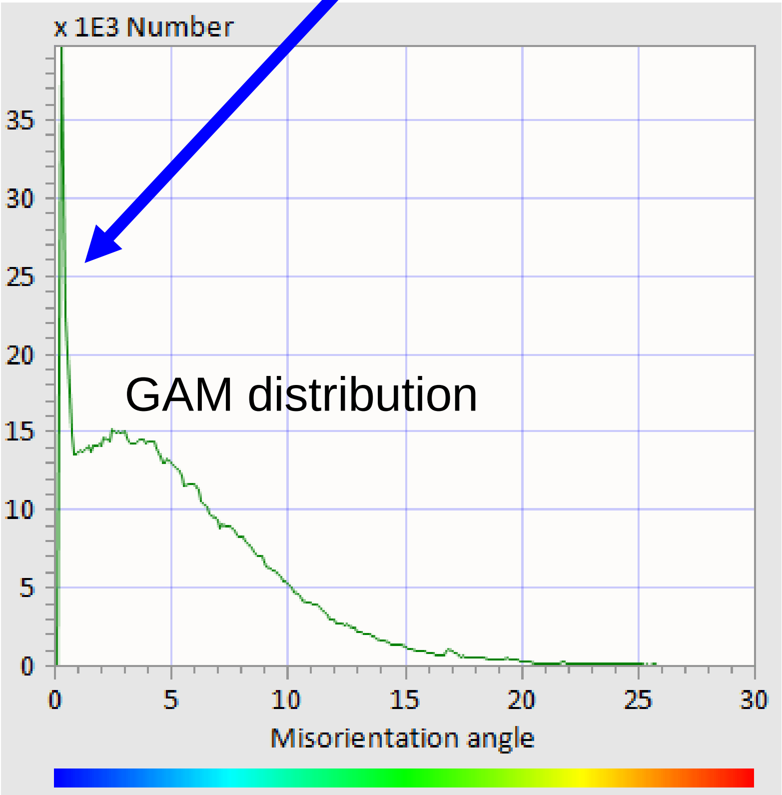
# Zirconium 702 alloy

## Grain average misorientation: Recrystallized grains

Ion milled: 8 and 2 keV energy; 3° angle

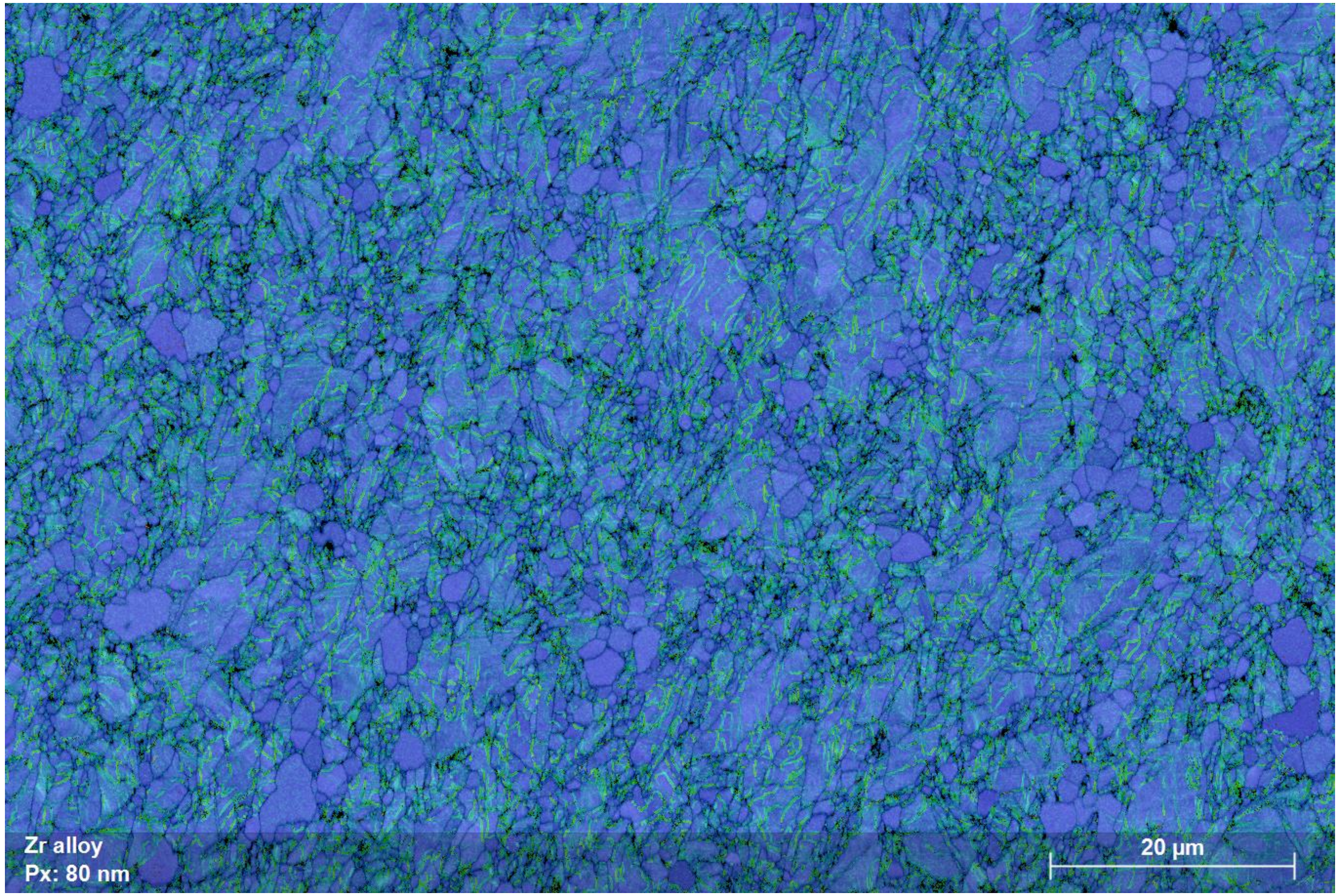



Recrystallized grains:
Misorientation angle from 0.01° to 0.6°

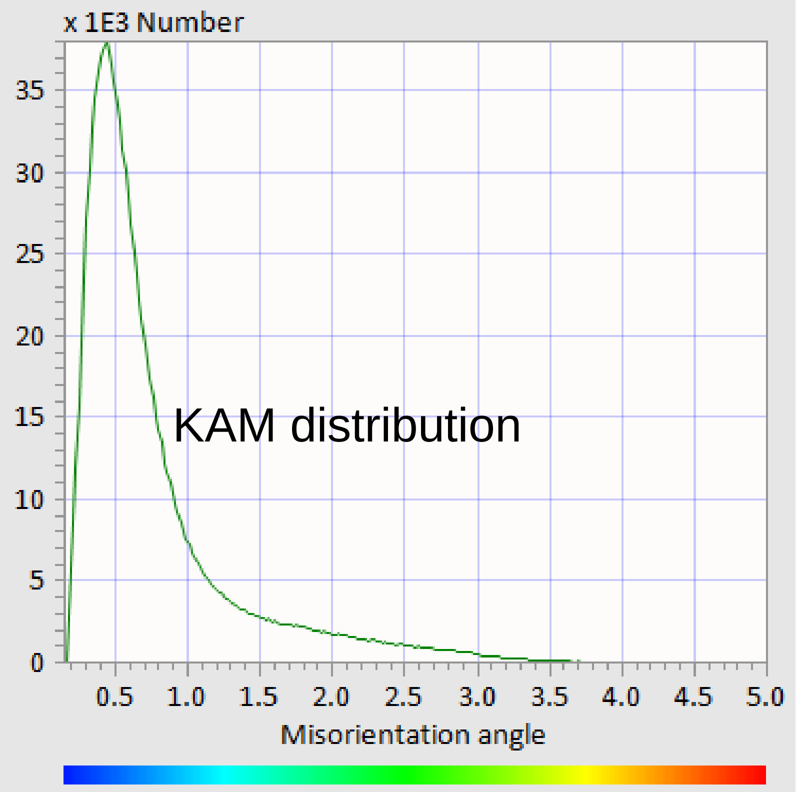


# Zirconium 702 alloy
## Kernel average misorientation

Ion milled: 8 and 2 keV energy; 3° angle




No artifact is observed after ion milling

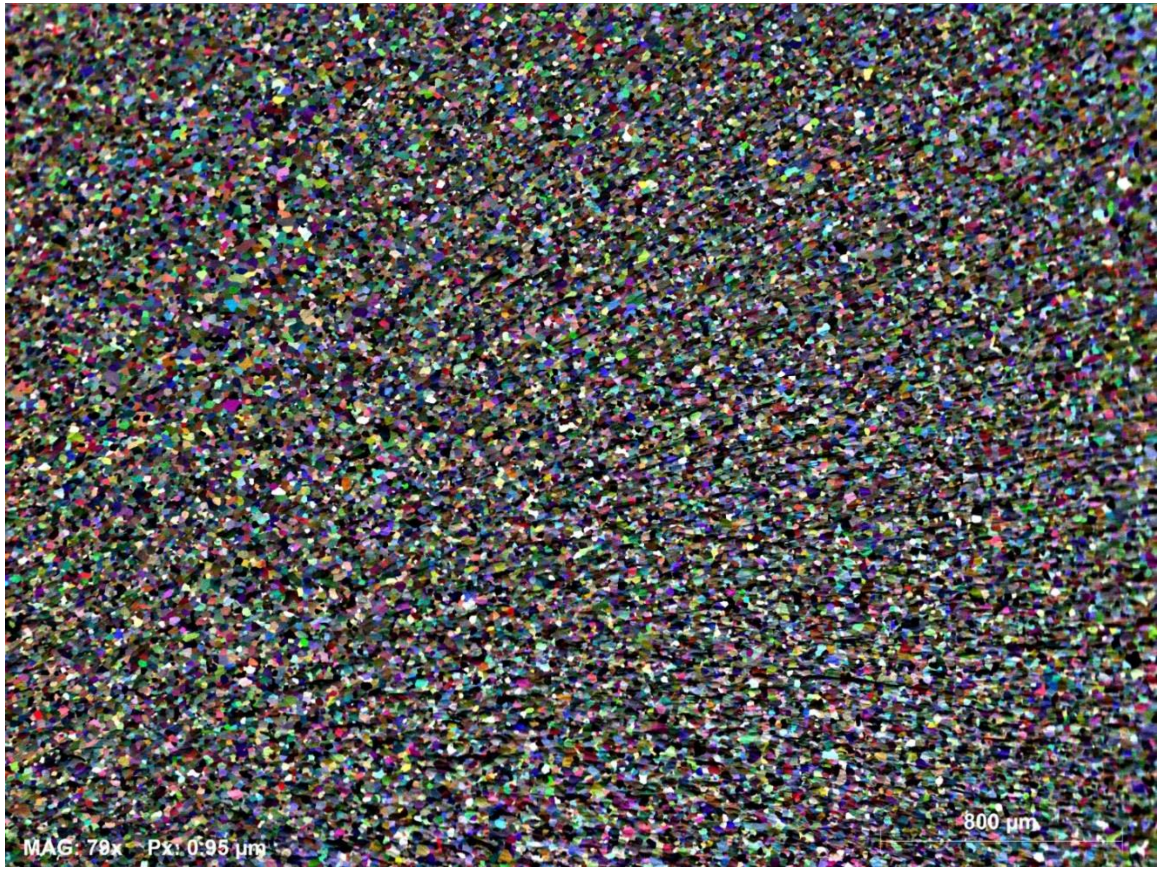


# Titanium grade 2

# Titanium grade 2

## Orientation contrast imaging with ARGUS™

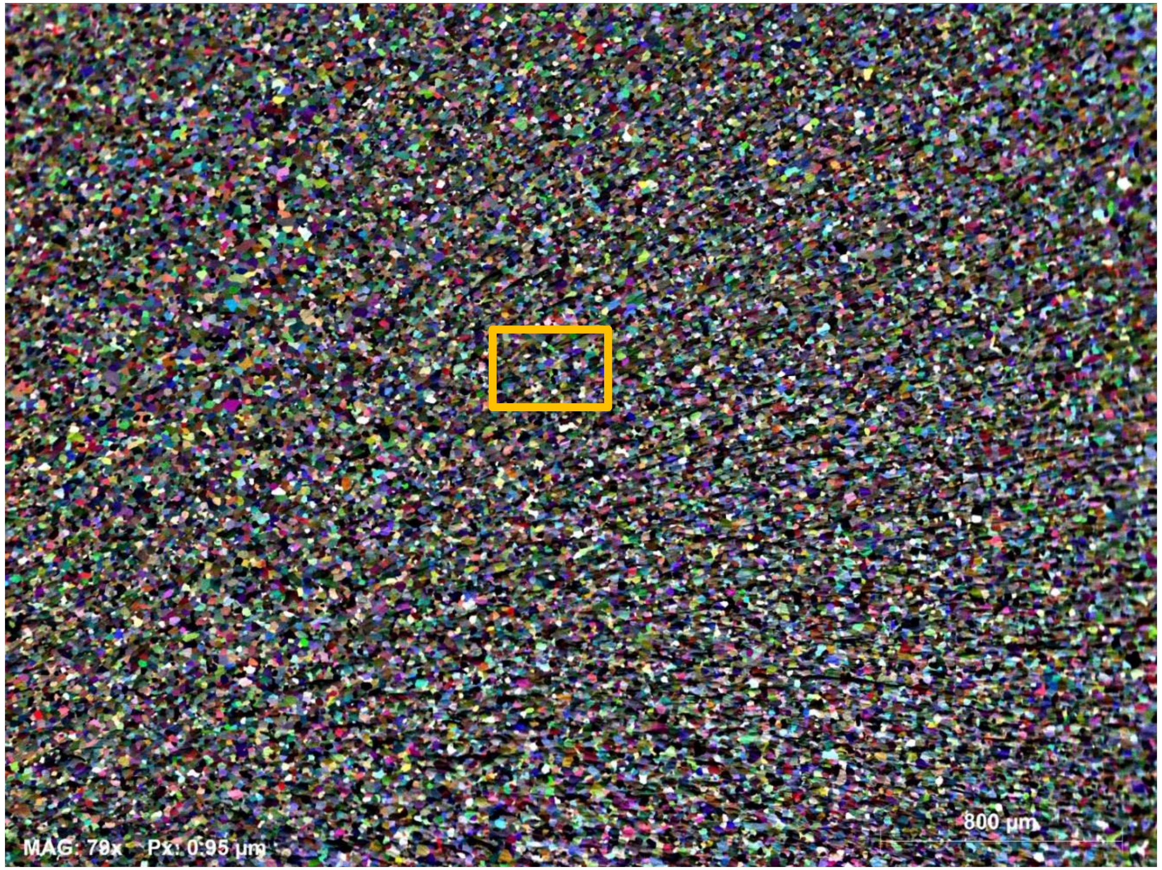
Ion milled: 8, 6, and 2 keV; 3° angle



# Titanium grade 2

## Orientation contrast imaging with ARGUS™

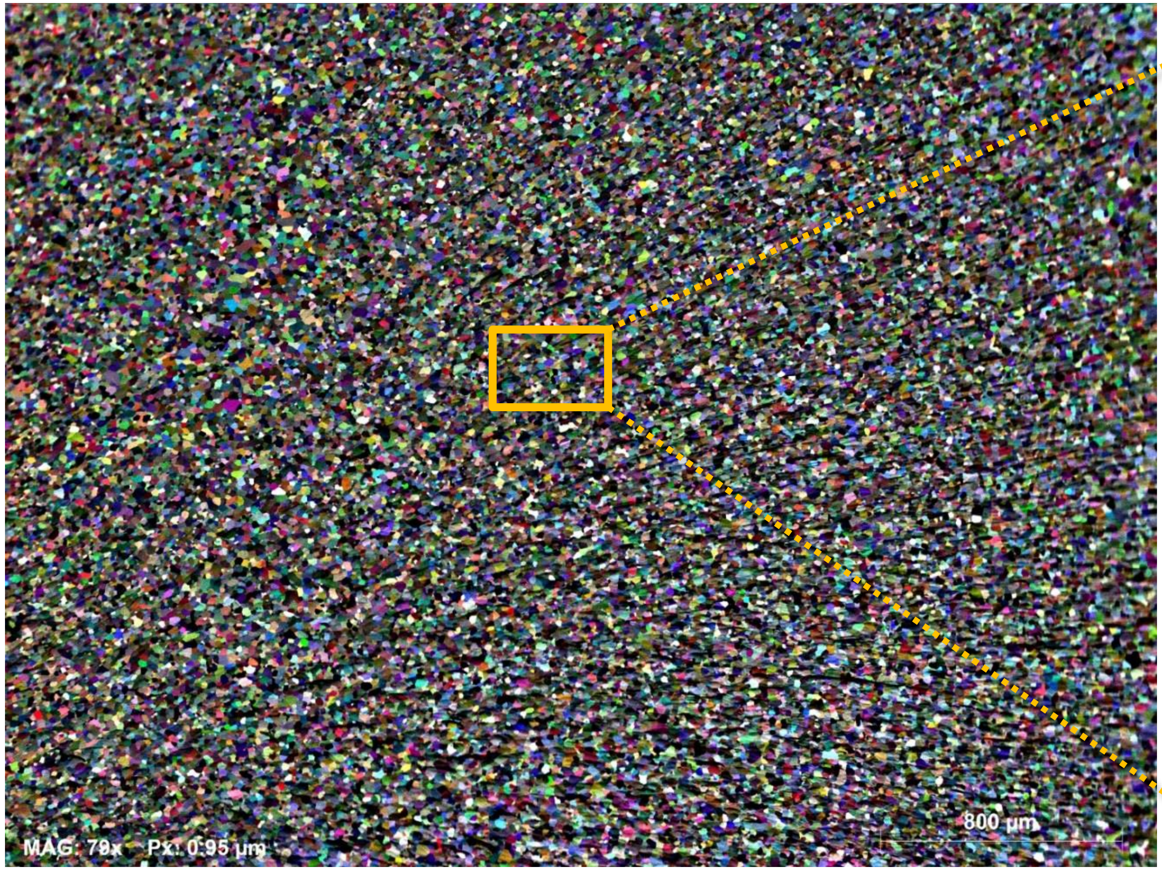
Ion milled: 8, 6, and 2 keV; 3° angle

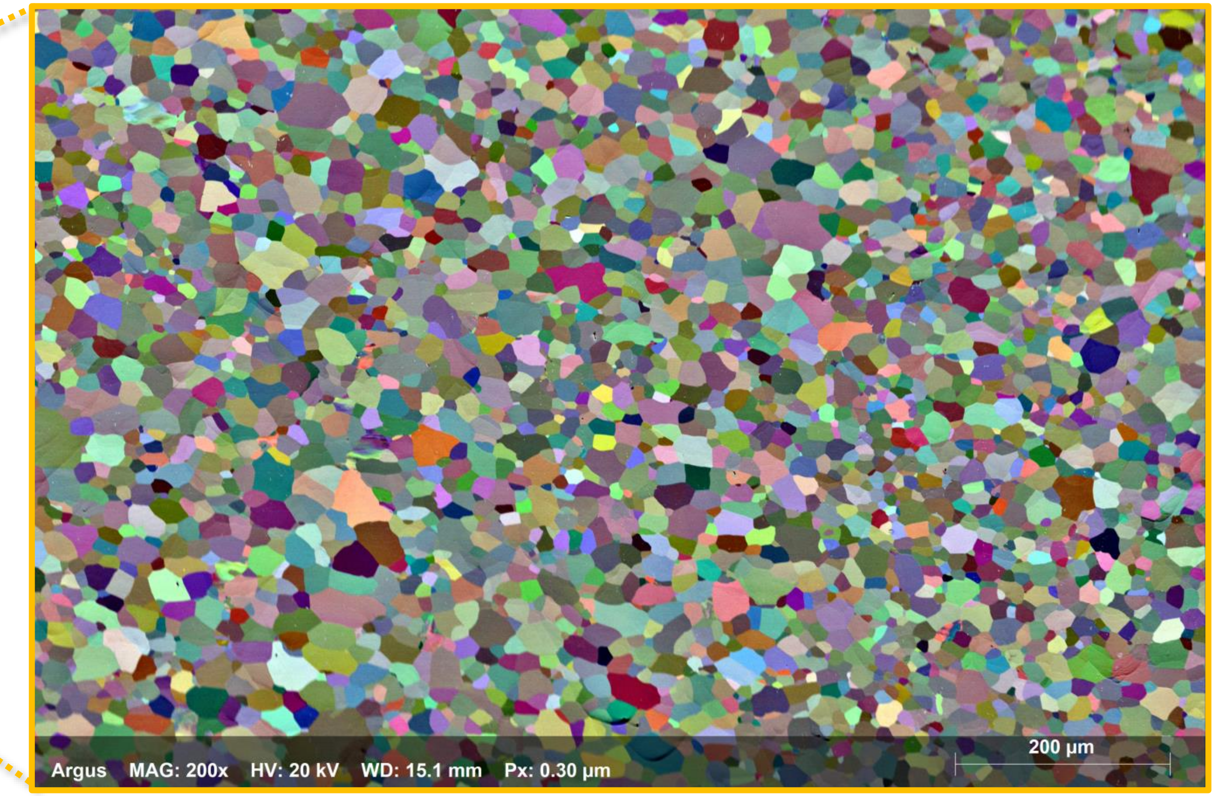


# Titanium grade 2

## Orientation contrast imaging with ARGUS™

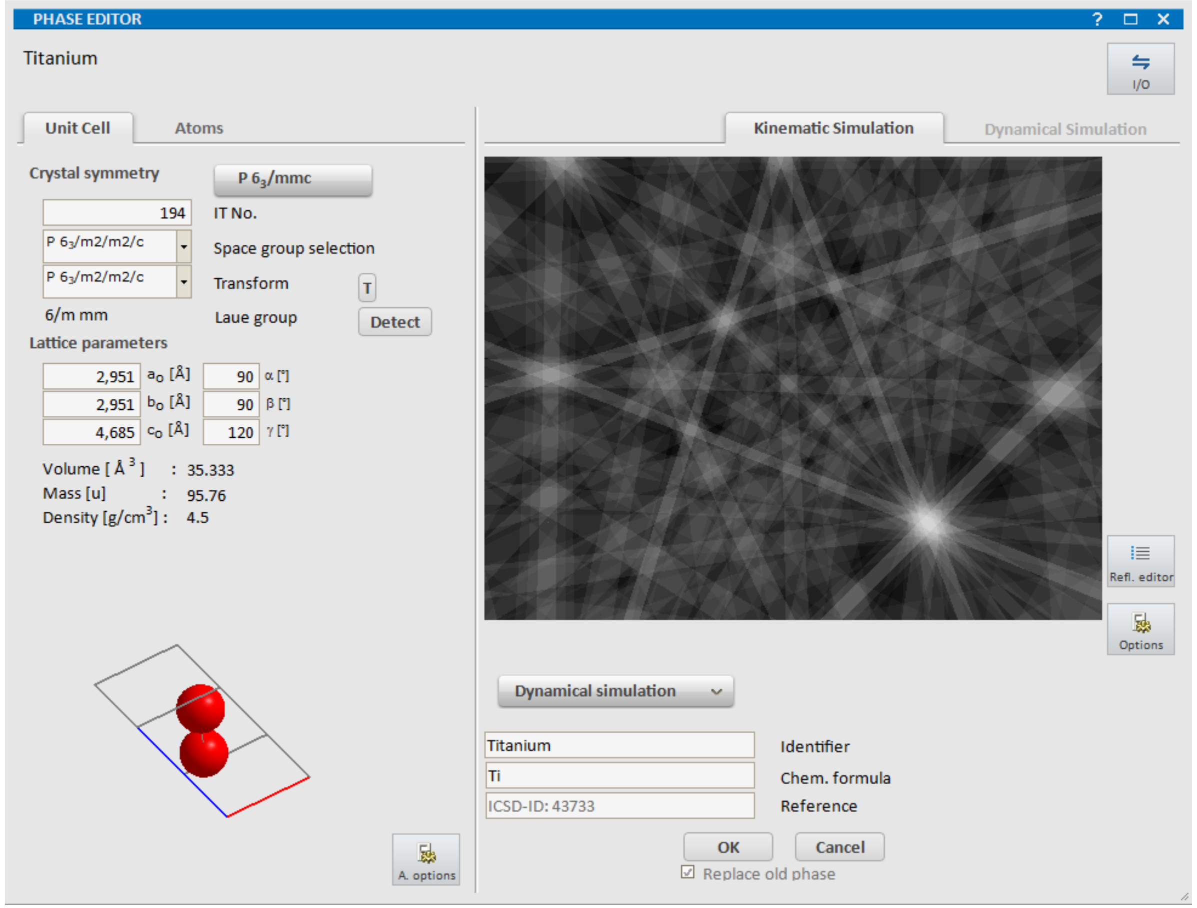
Ion milled: 8, 6, and 2 keV; 3° angle



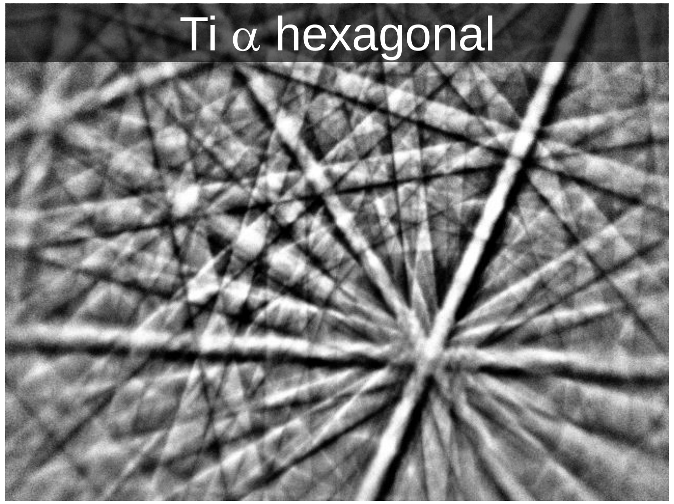



# Titanium grade 2
## EBSD pattern

### Phase file



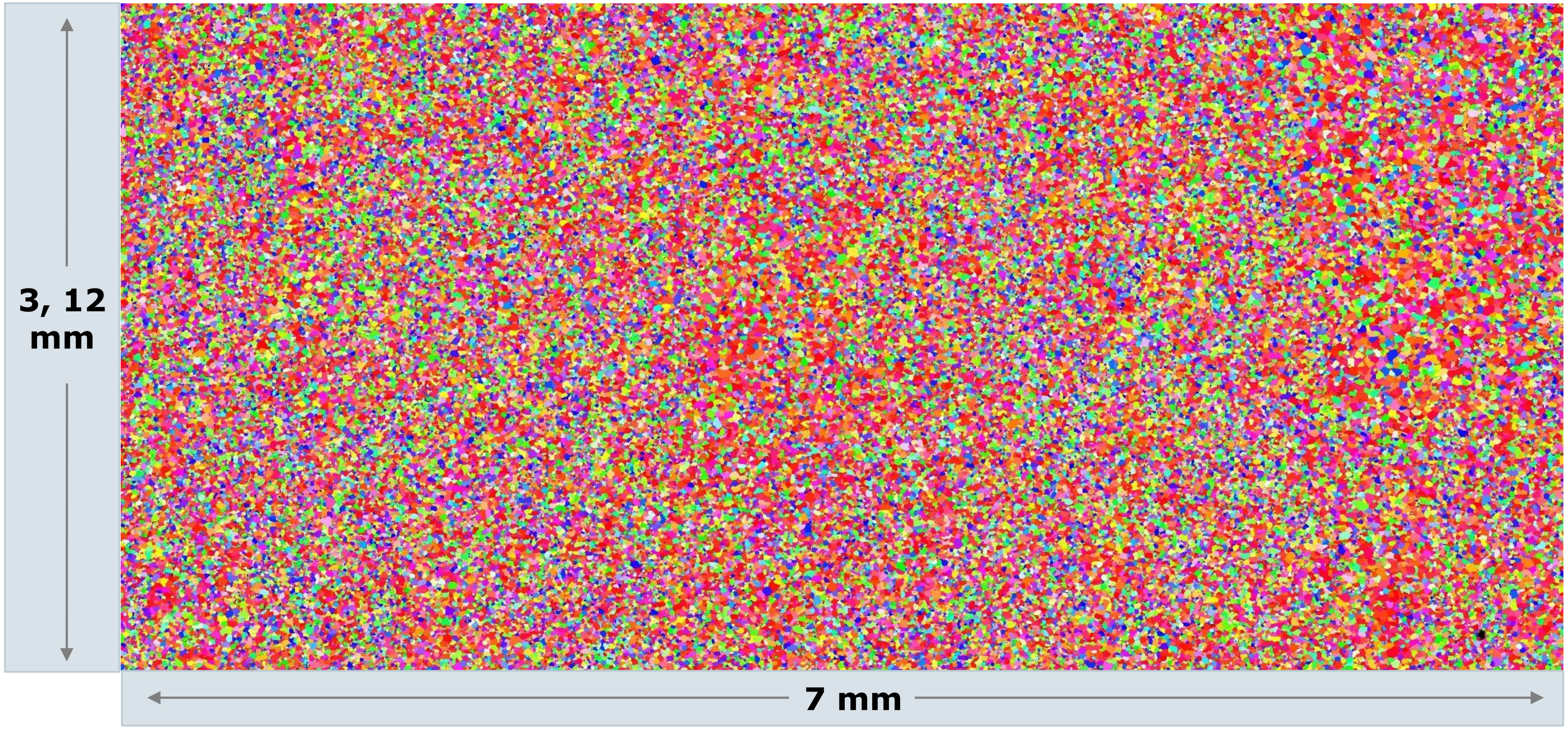

### Diffraction pattern



# Titanium grade 2

## Large-area milling and EBSD map

Ion milled: 8, 6, and 2 keV; 3° angle

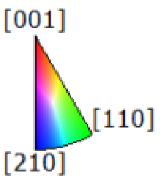



Step size: 1 µm

Indexing rate: 99%

EBSP resolution:
80x60 pixels

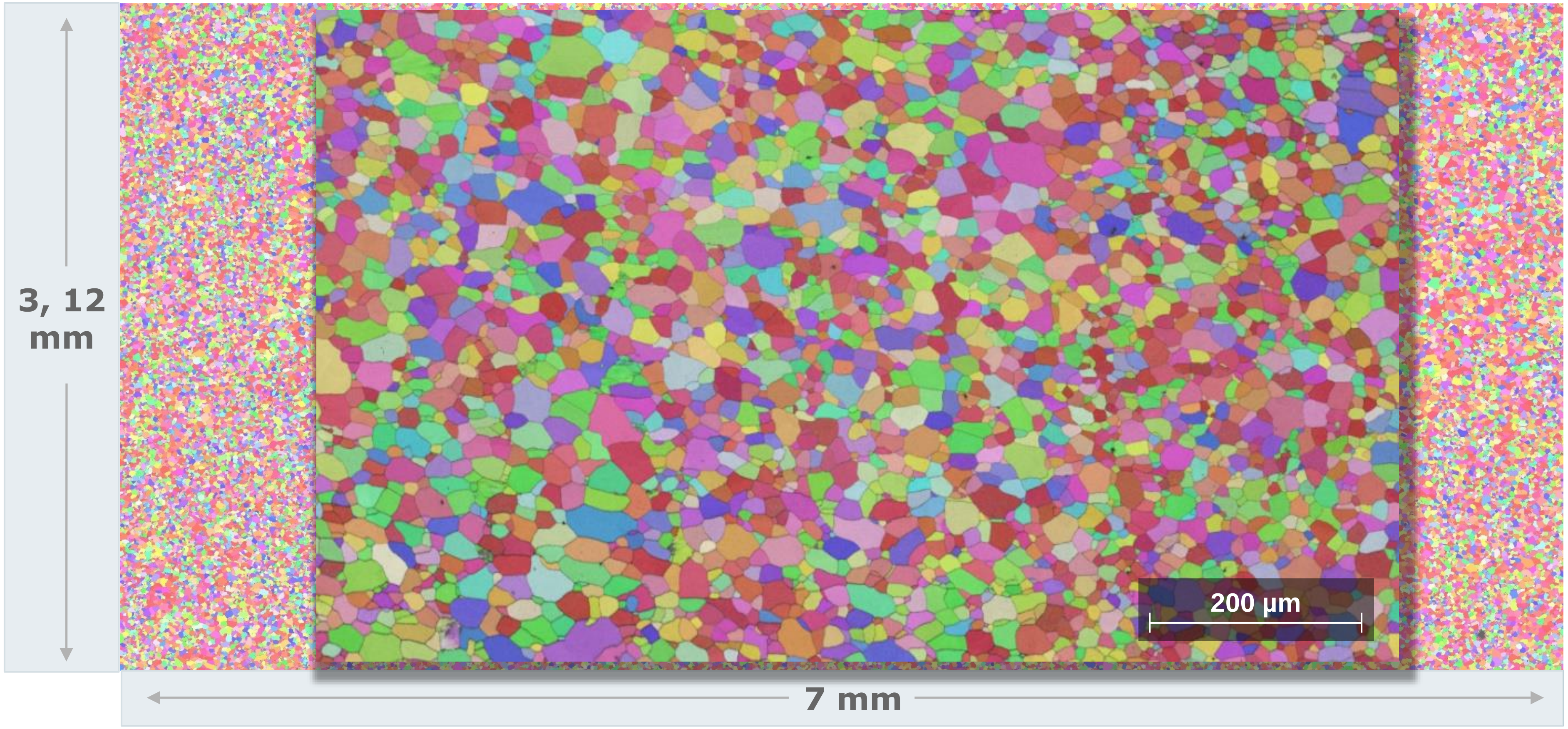
Measurement speed:
936 Hz



# Titanium grade 2

## Large-area milling and EBSD map

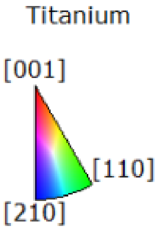
Ion milled: 8, 6, and 2 keV; 3° angle




Step size: 1 µm

Indexing rate: 99%

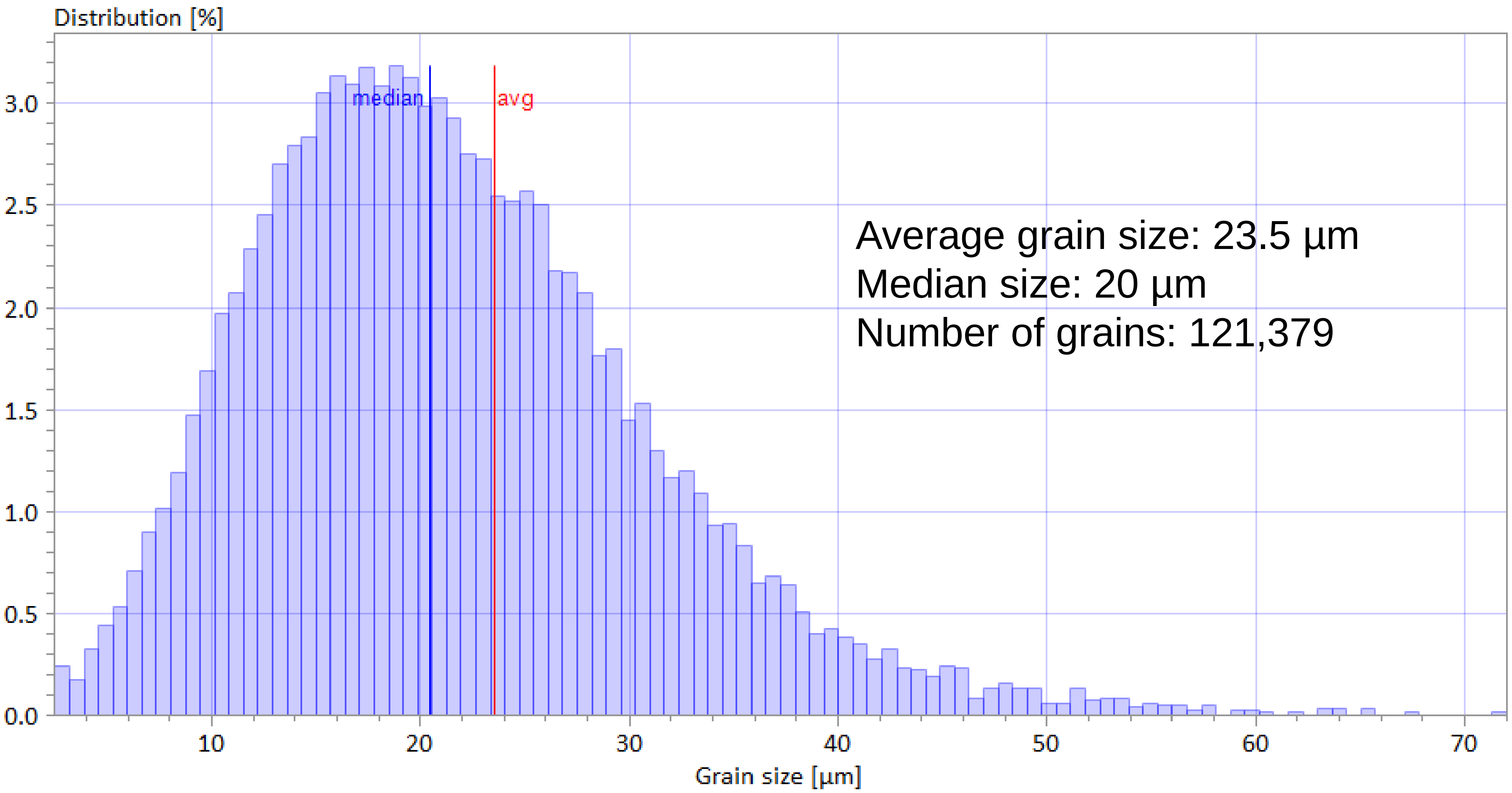
6 min per map



# Titanium grade 2

## Large-area milling and EBSD map

Grain size distribution

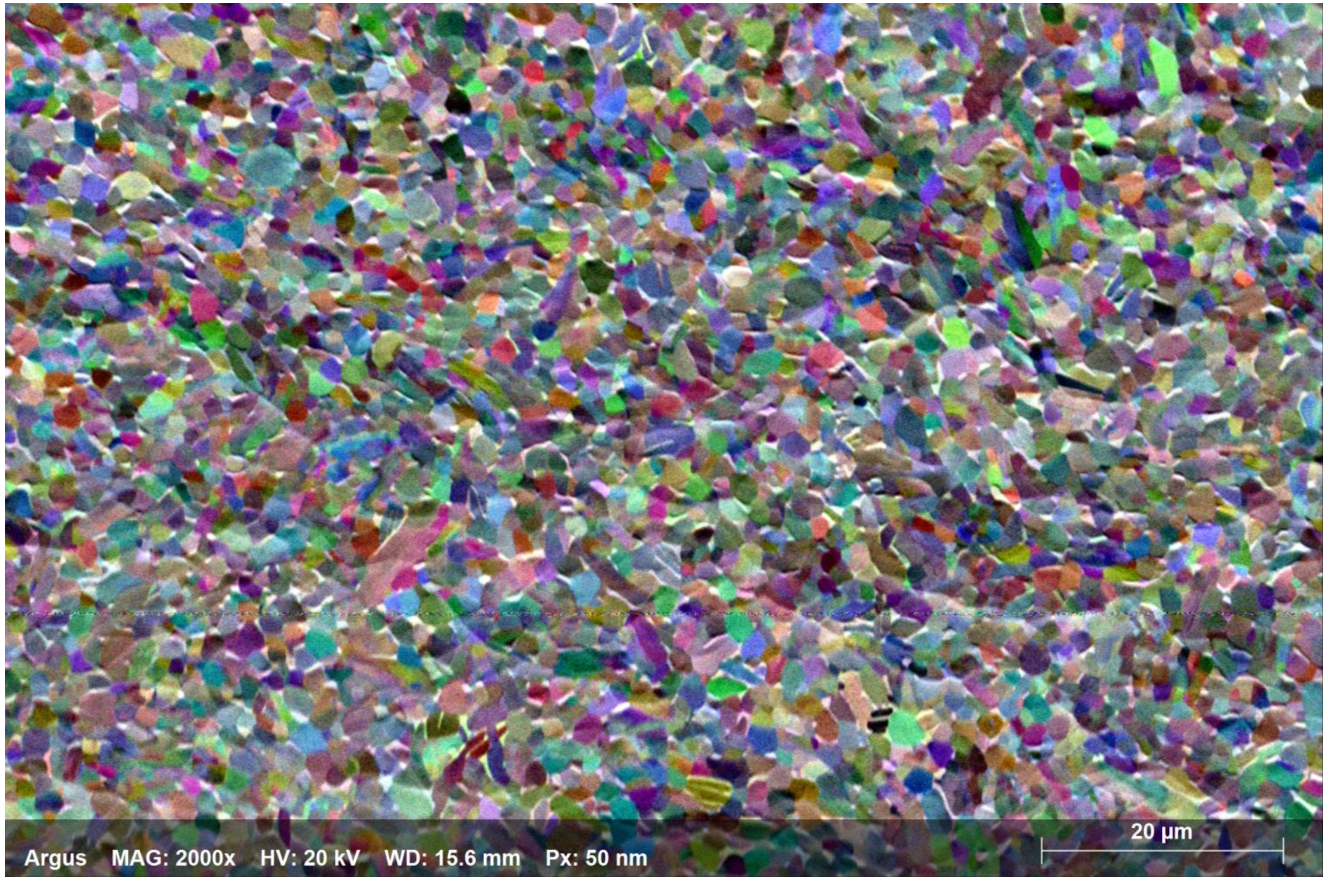


# Titanium grade 5 (Ti-6Al-4V)

# Titanium grade 5 (Ti-6Al-4V)

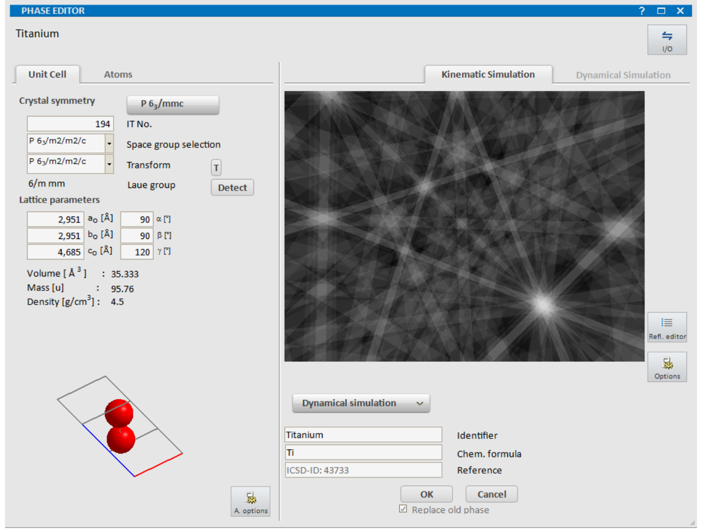
## Orientation contrast imaging with ARGUS™

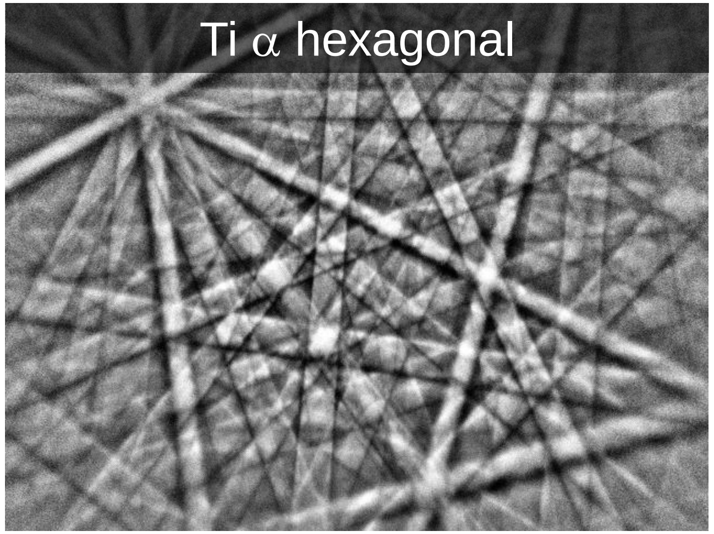
Ion milled: 6 and 2 keV; 3° angle

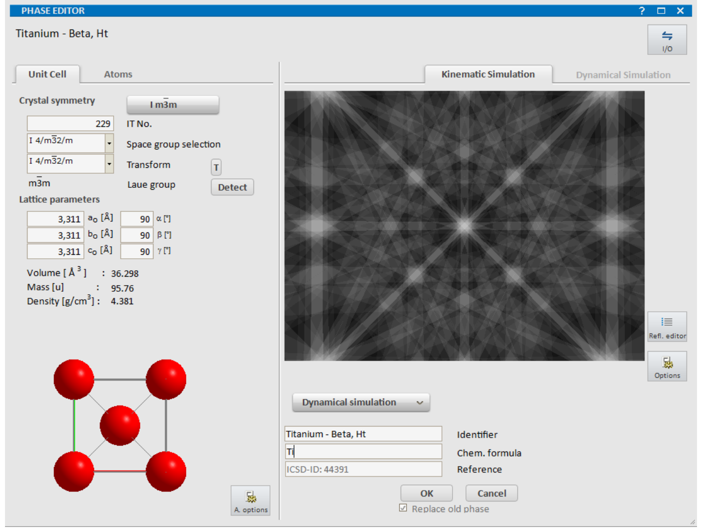


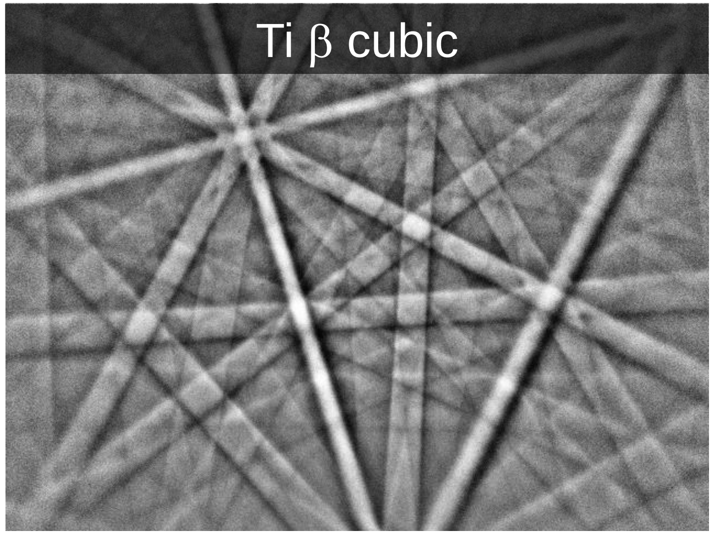
# Titanium grade 5 (Ti-6Al-4V)

## EBSD patterns



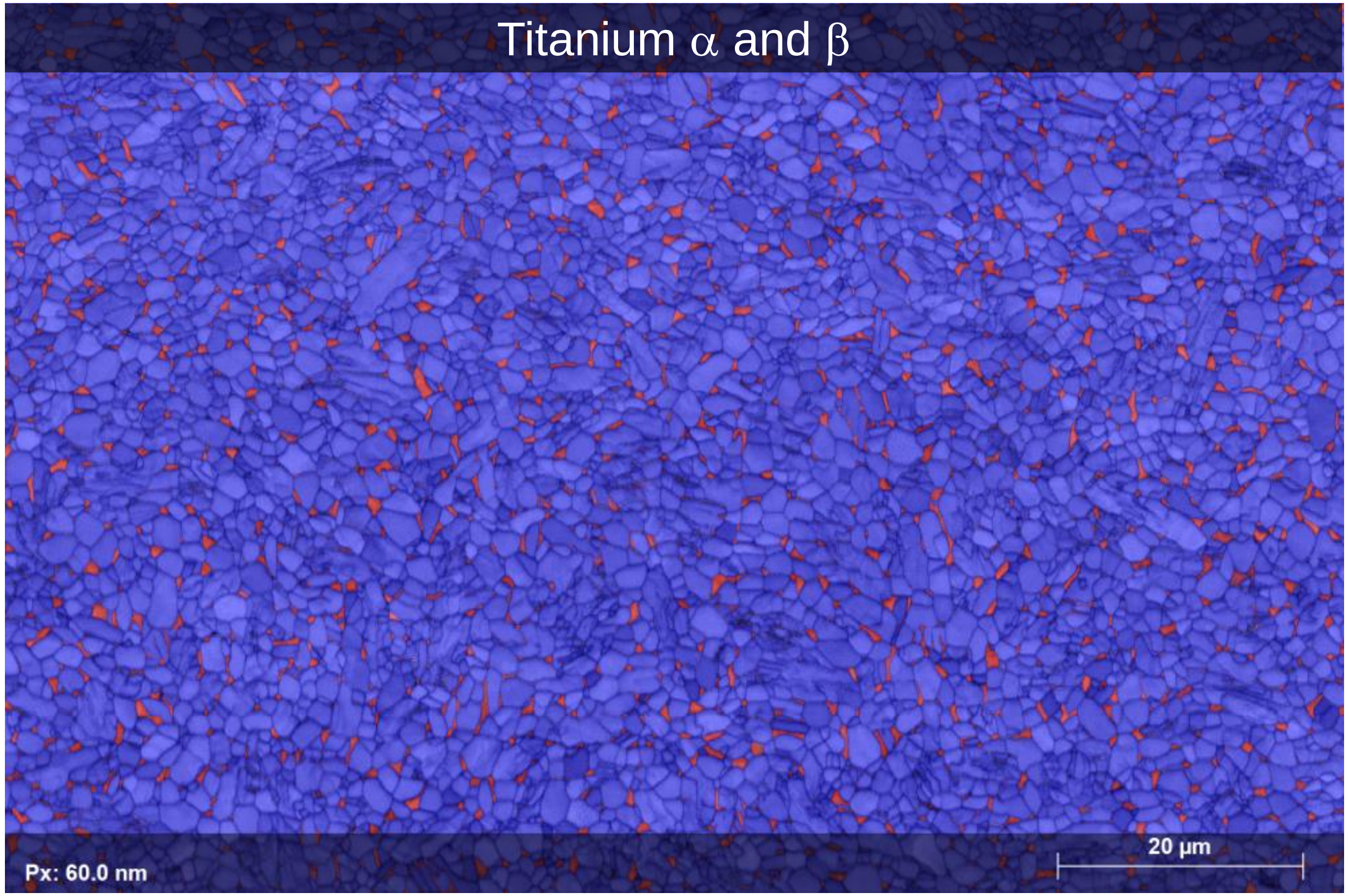



# Titanium grade 5 (Ti-6Al-4V)

## EBSD phase distribution map

Ion milled: 6 and 2 keV; 3° angle




**Phase map**
Step size: 80 nm

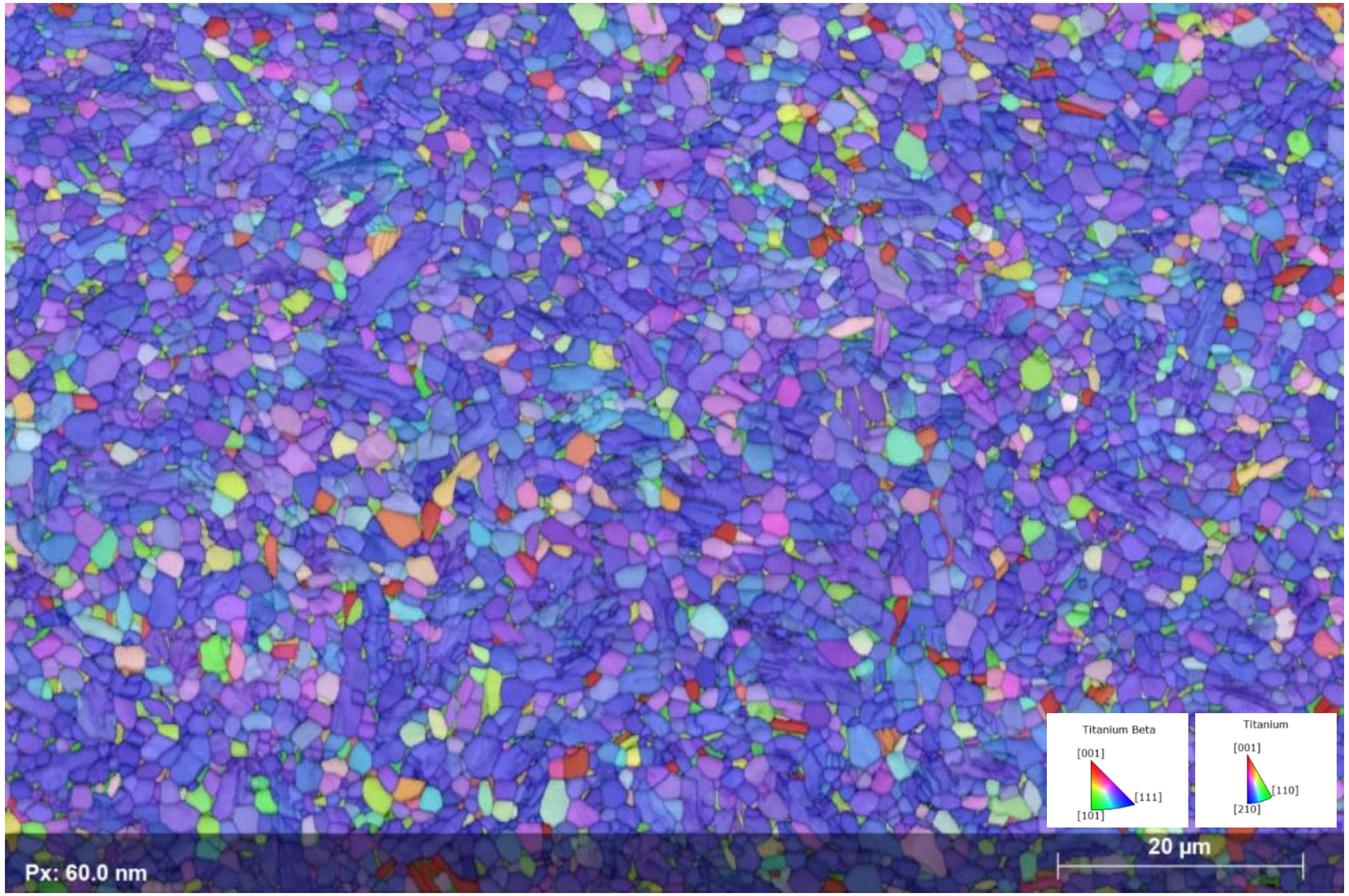
Indexing rate: 98.6%

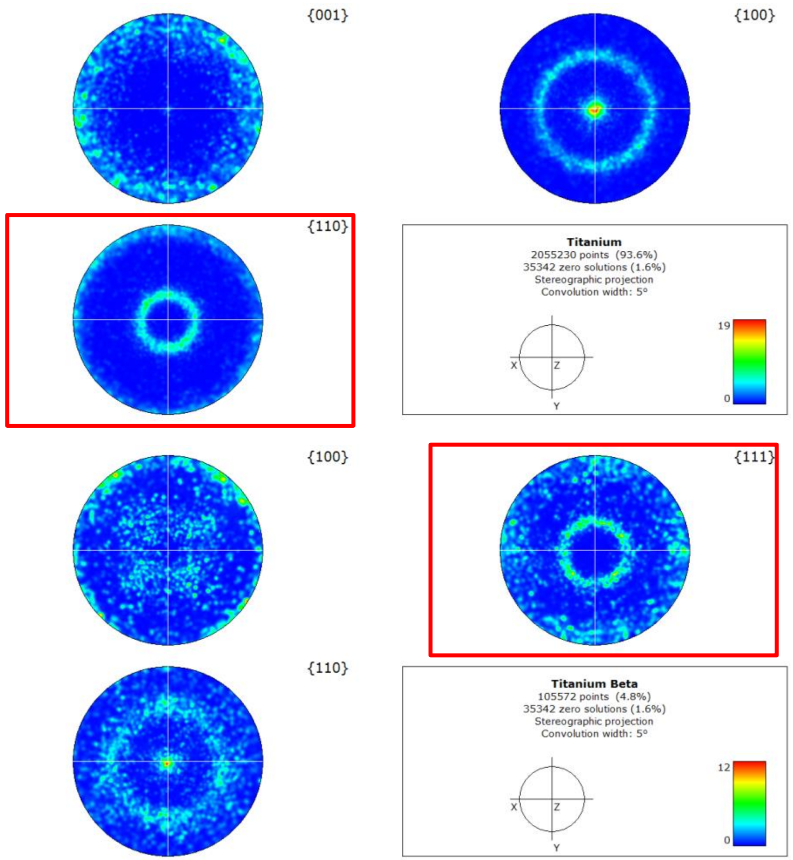
| | |
|---|---|
| Ti β | 5% |
| Ti α | 93% |

# Titanium grade 5 (Ti-6Al-4V)

## EBSD IPF map

Ion milled: 6 and 2 keV; 3° angle



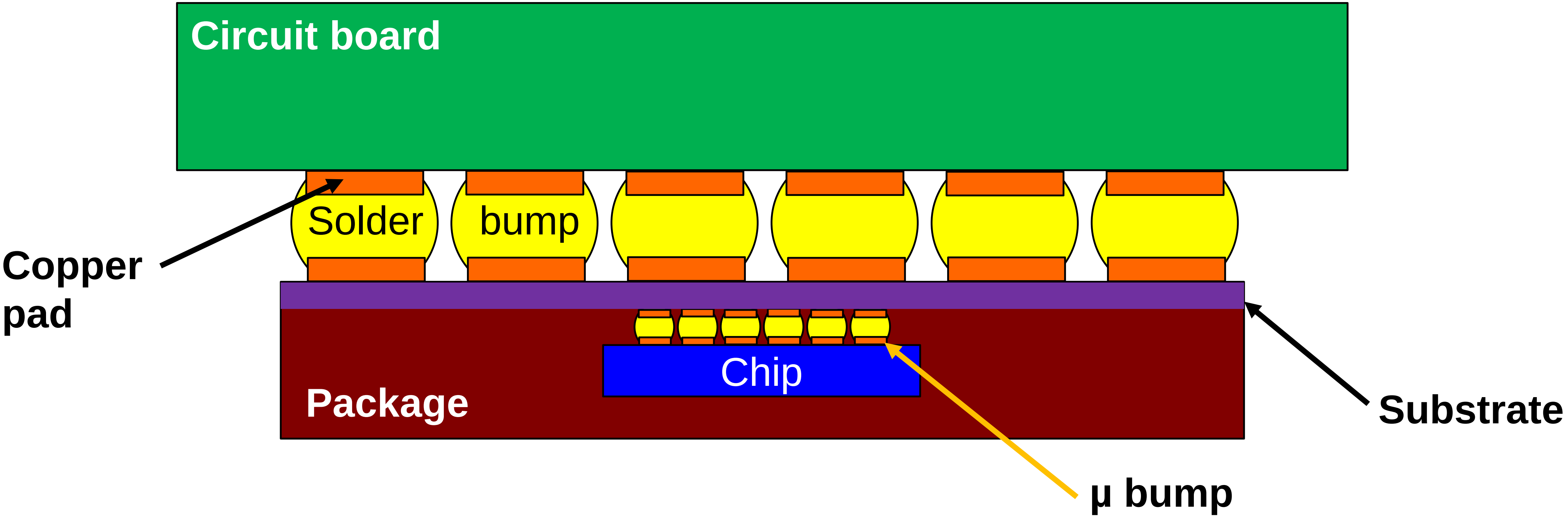




Burgers relationship **<111>**$_\beta$ || **<11-20>**$_\alpha$ and {110}$_\beta$ || {0001}$_\alpha$

# Solder bumps

# Solder bumps

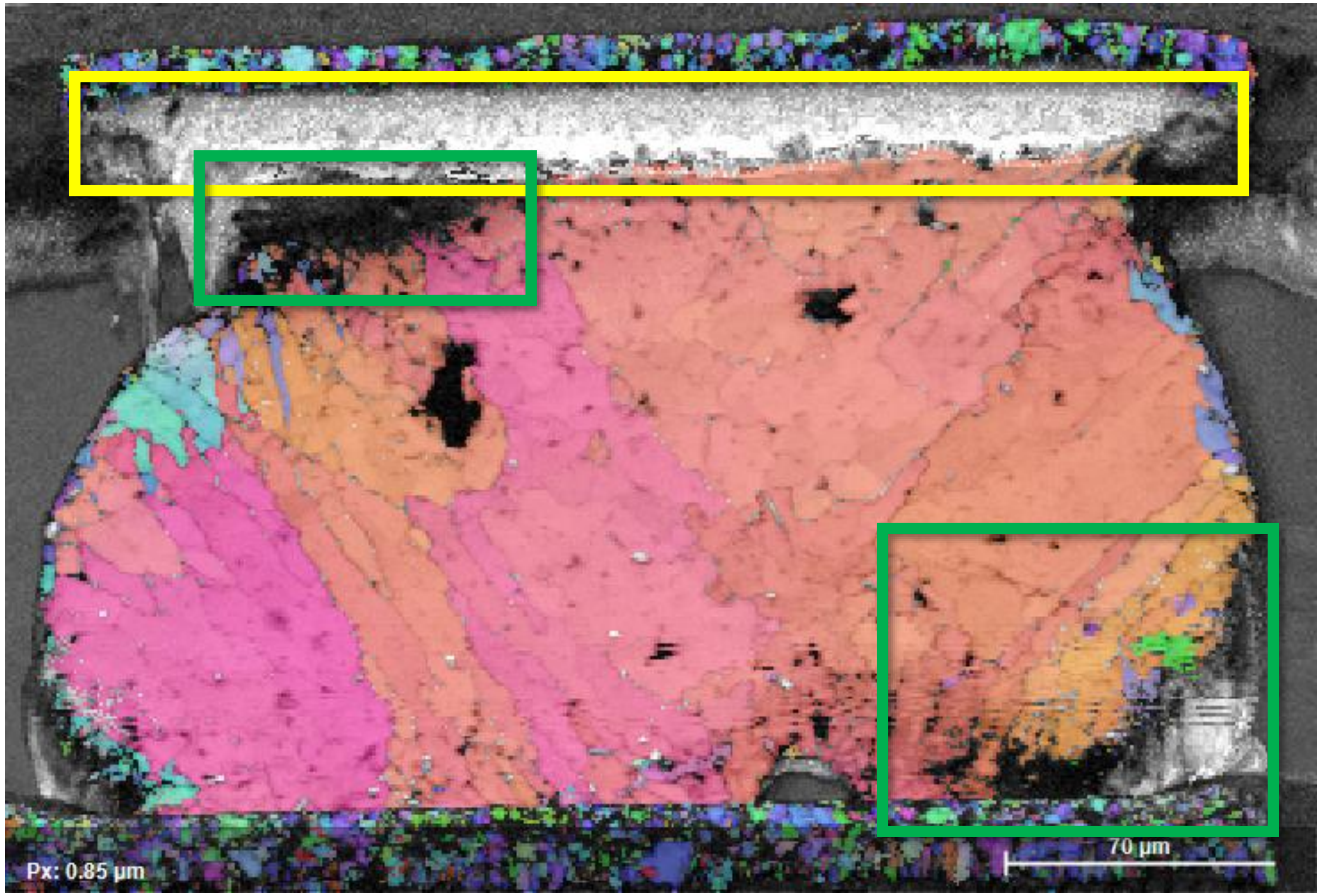
## Cross-section sample schematic




Structure is fragile

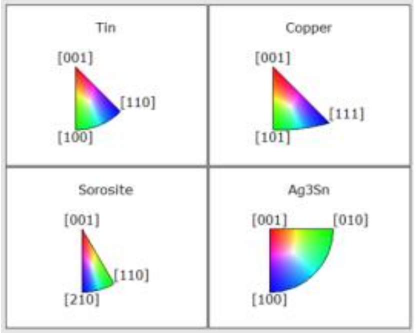
# Solder bumps
## EBSD IPF map

Mechanical polishing by colloidal silica



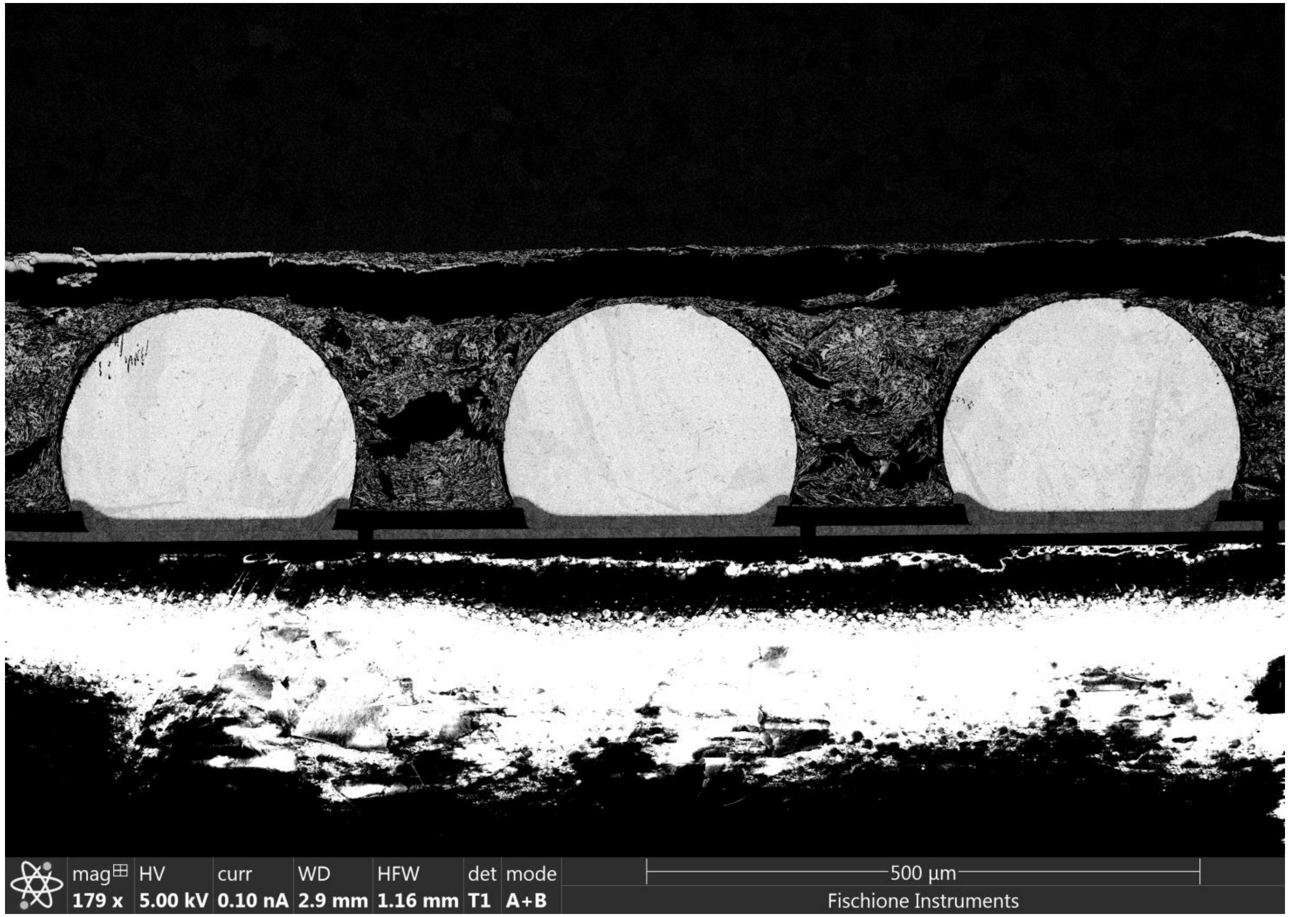

**IPF Z map**

- $SiO_2$ particles are difficult to remove
- Areas of the interface are not visible due to shadowing by the copper pad caused by differential material removal
- Corrosion of packaging components leaves surface contamination



# Solder bumps
## Cross-section sample

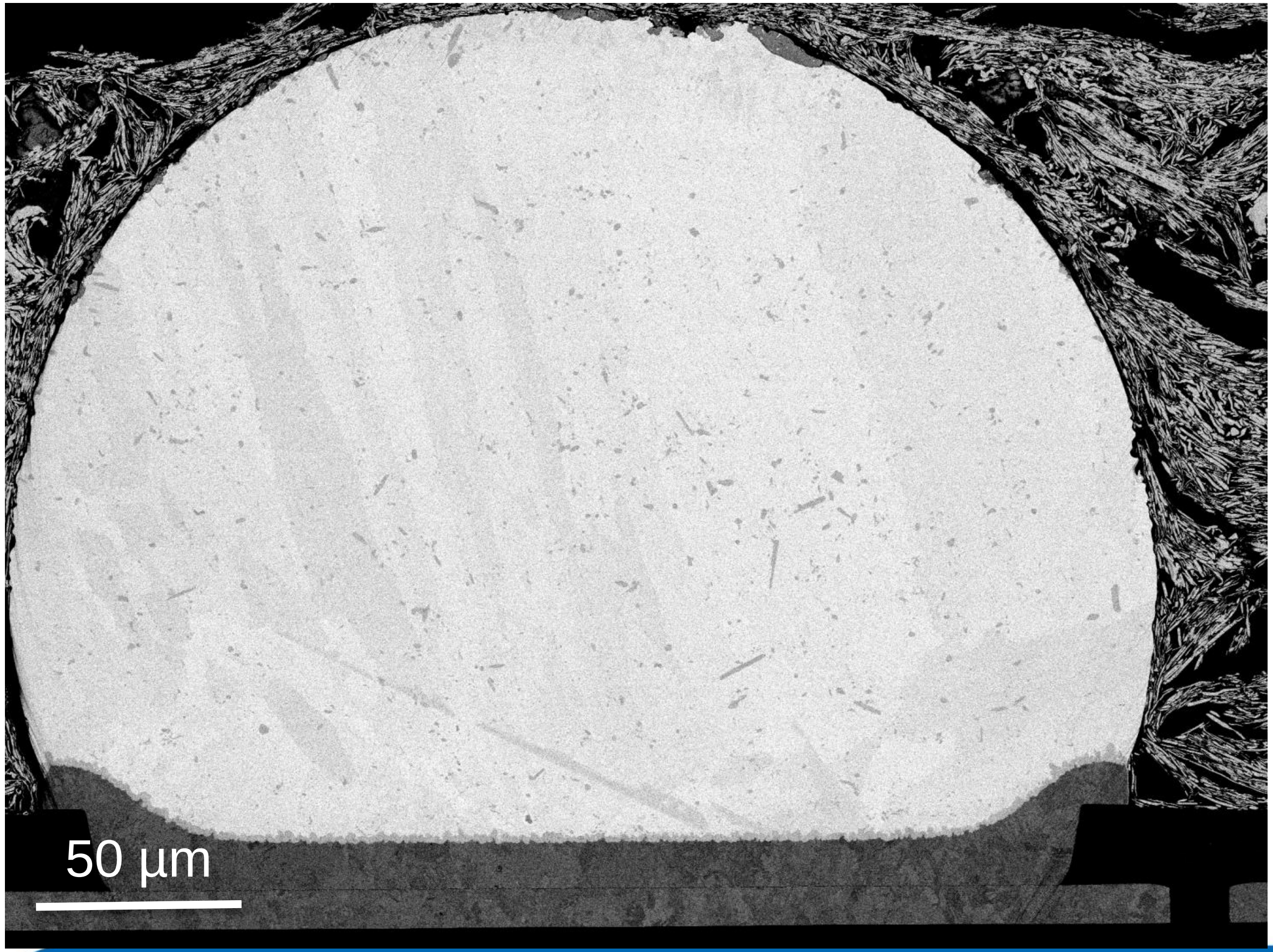
Ion milled at 6 and 2 keV; cross-section milling with mask under **cryo conditions**



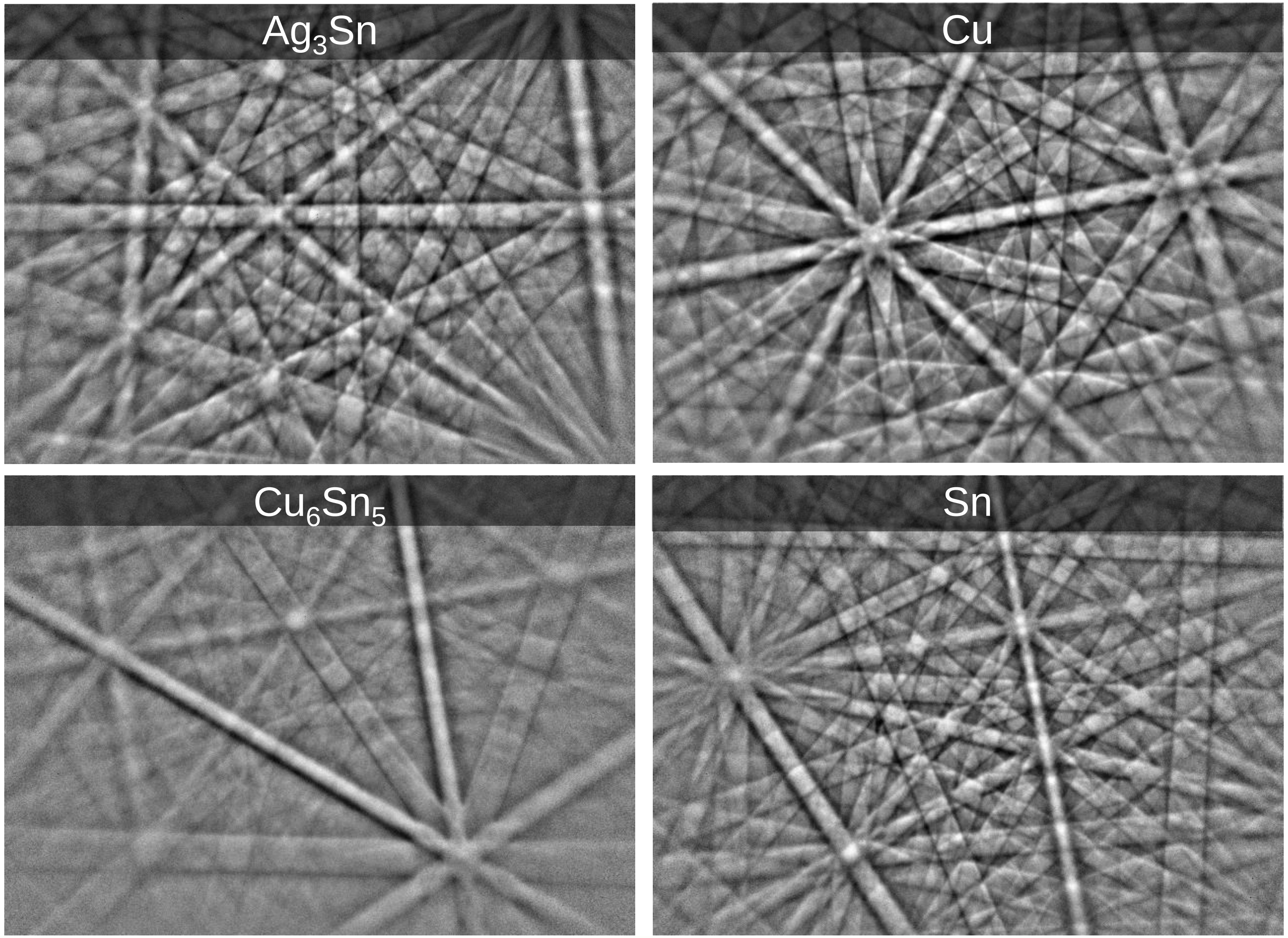
# Solder bumps

## Backscatter electron contrast imaging

Ion milled at 6 and 2 keV; cross-section milling with mask under **cryo conditions**



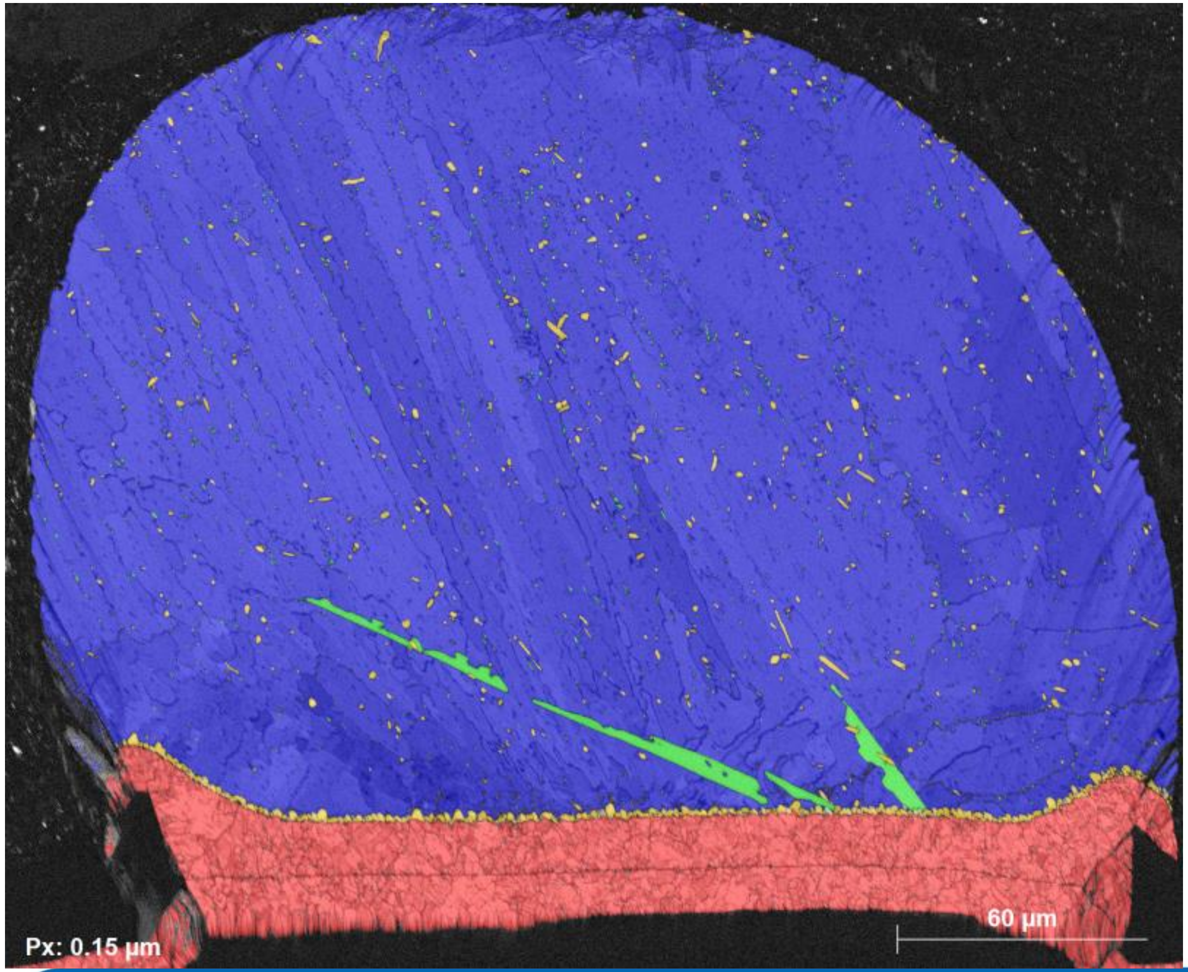
# Solder bumps
## EBSD patterns



# Solder bumps

## EBSD phase distribution map

Ion milled at 6 and 2 keV; cross-section milling with mask under **cryo conditions**

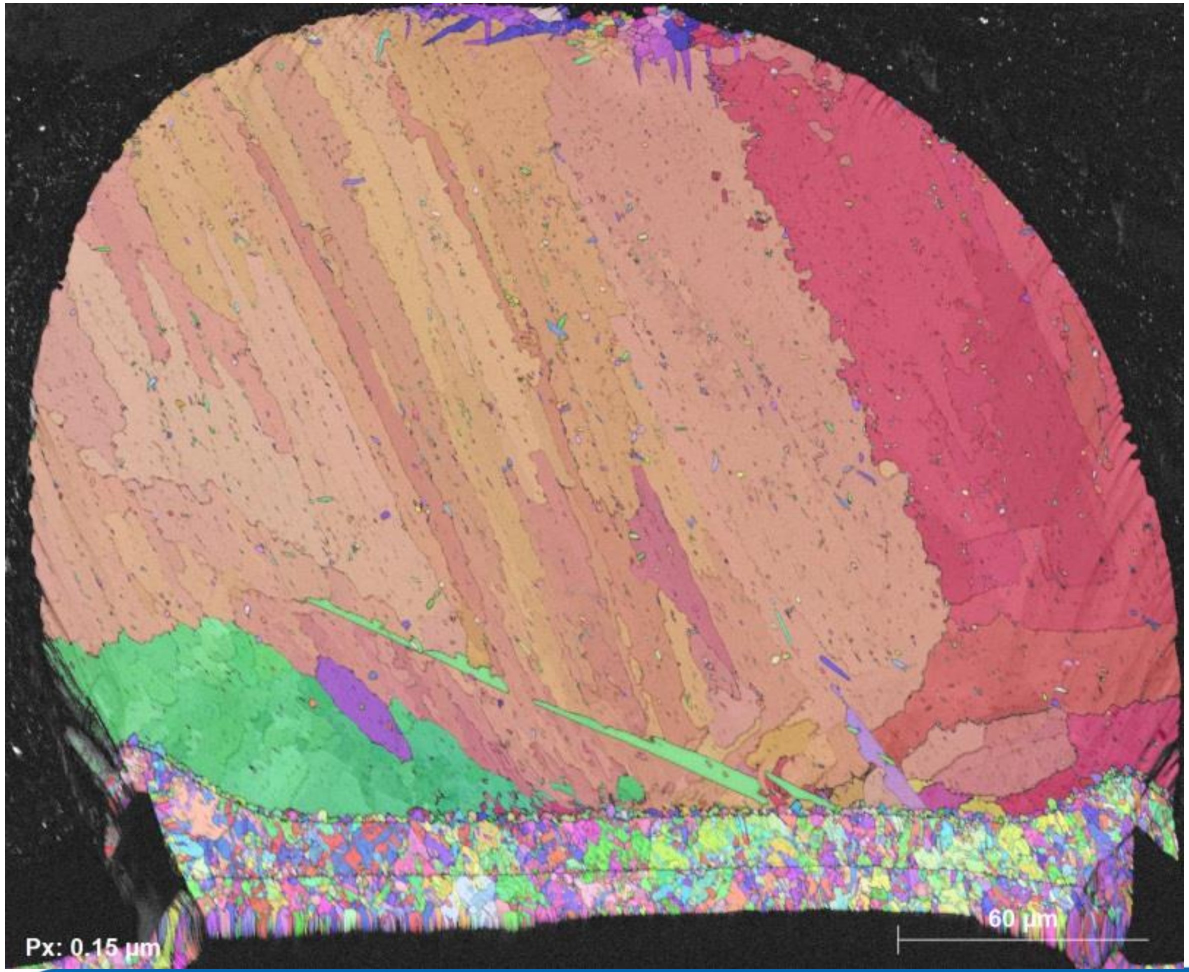



**Phase map**
Step size: 150 nm
Pattern resolution: 80x60 pixels
Indexing rate: 95%

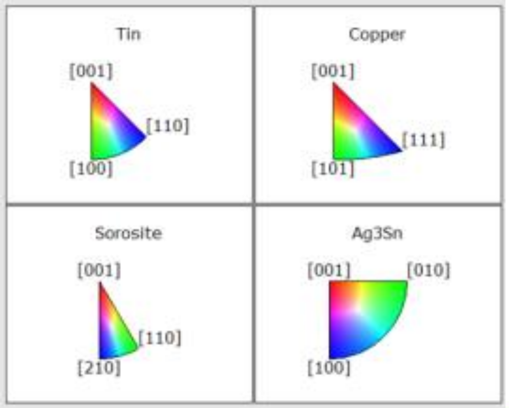
| Phase | Color | % normalized | Average grain size |
|---|---|---|---|
| Cu | | 12.5 | 10 µm |
| $Cu_6Sn_5$ | | 1.5 | 1,7µm |
| $Ag_3Sn$ | | 1 | 700 nm (fine grains only) |
| Sn | | 85 | NA (only 4 grains) |

# Solder bumps
## EBSD IPF map

Ion milled at 6 and 2 keV; cross-section milling with mask under **cryo conditions**



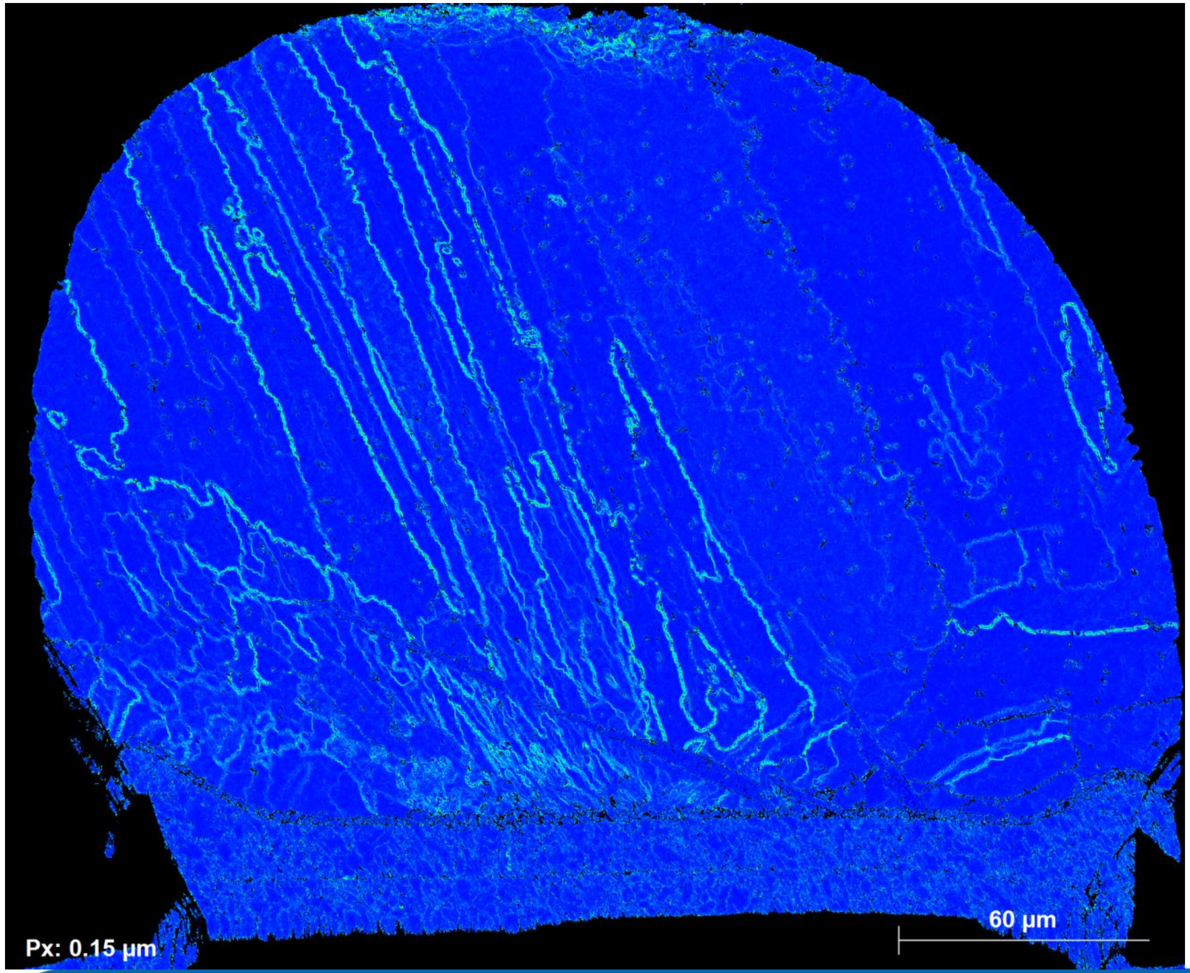

**IPF Z map**

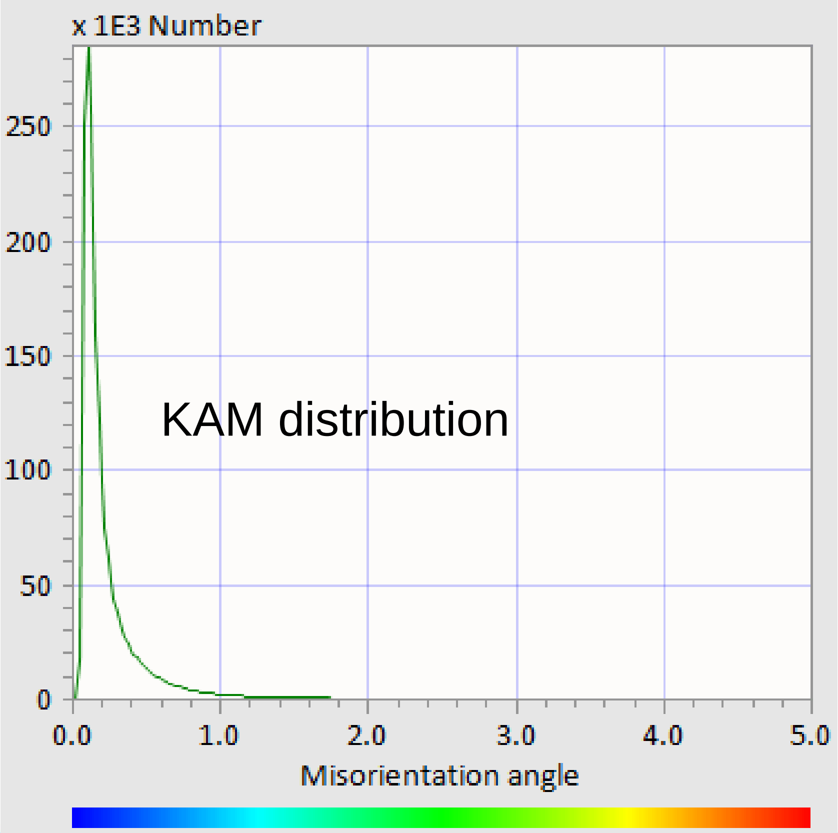


# Solder bumps

## Kernel average misorientation

Ion milled at 6 and 2 keV; cross-section milling with mask under **cryo conditions**

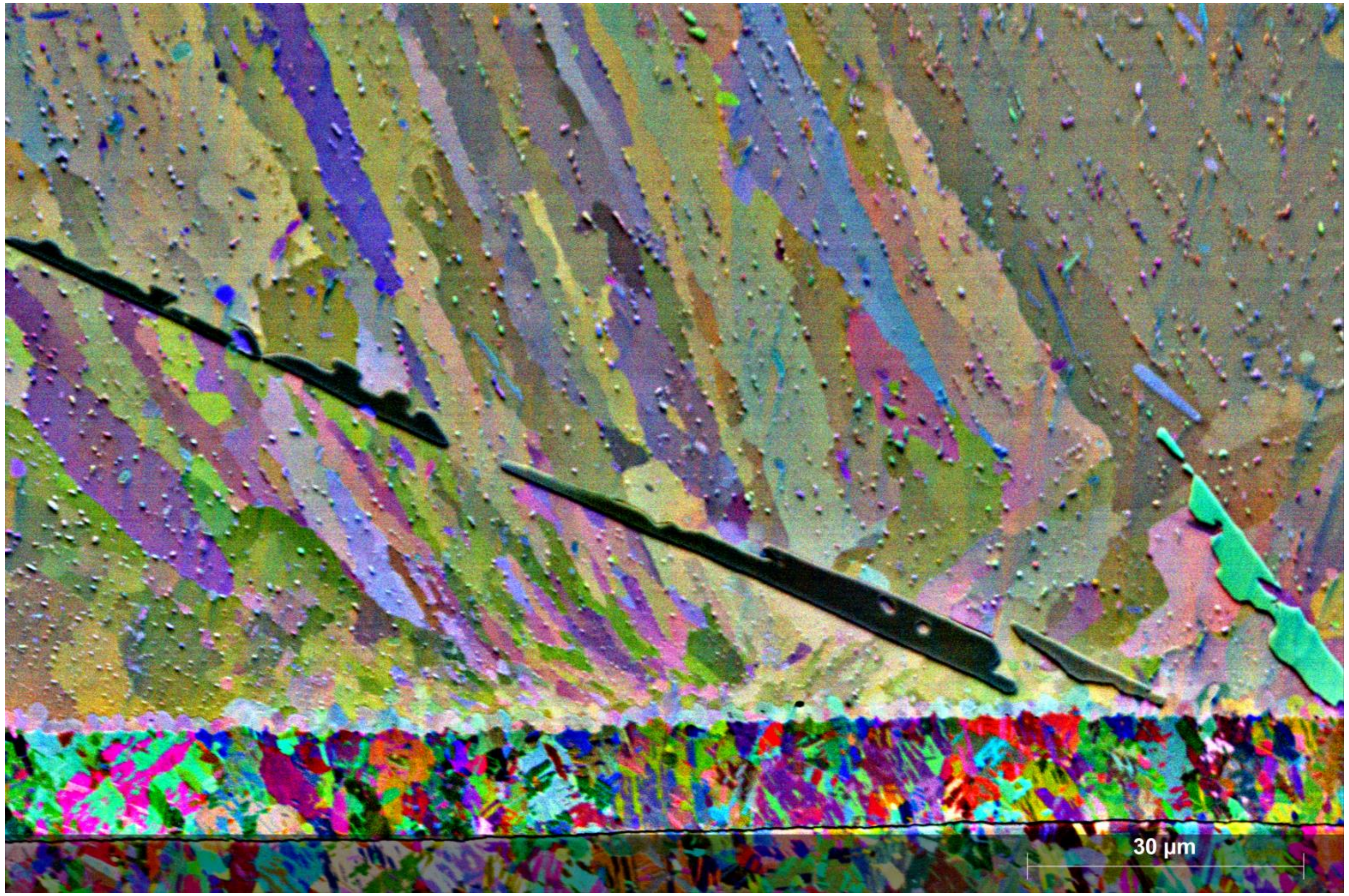


# Solder bumps

## Orientation contrast imaging with ARGUS™

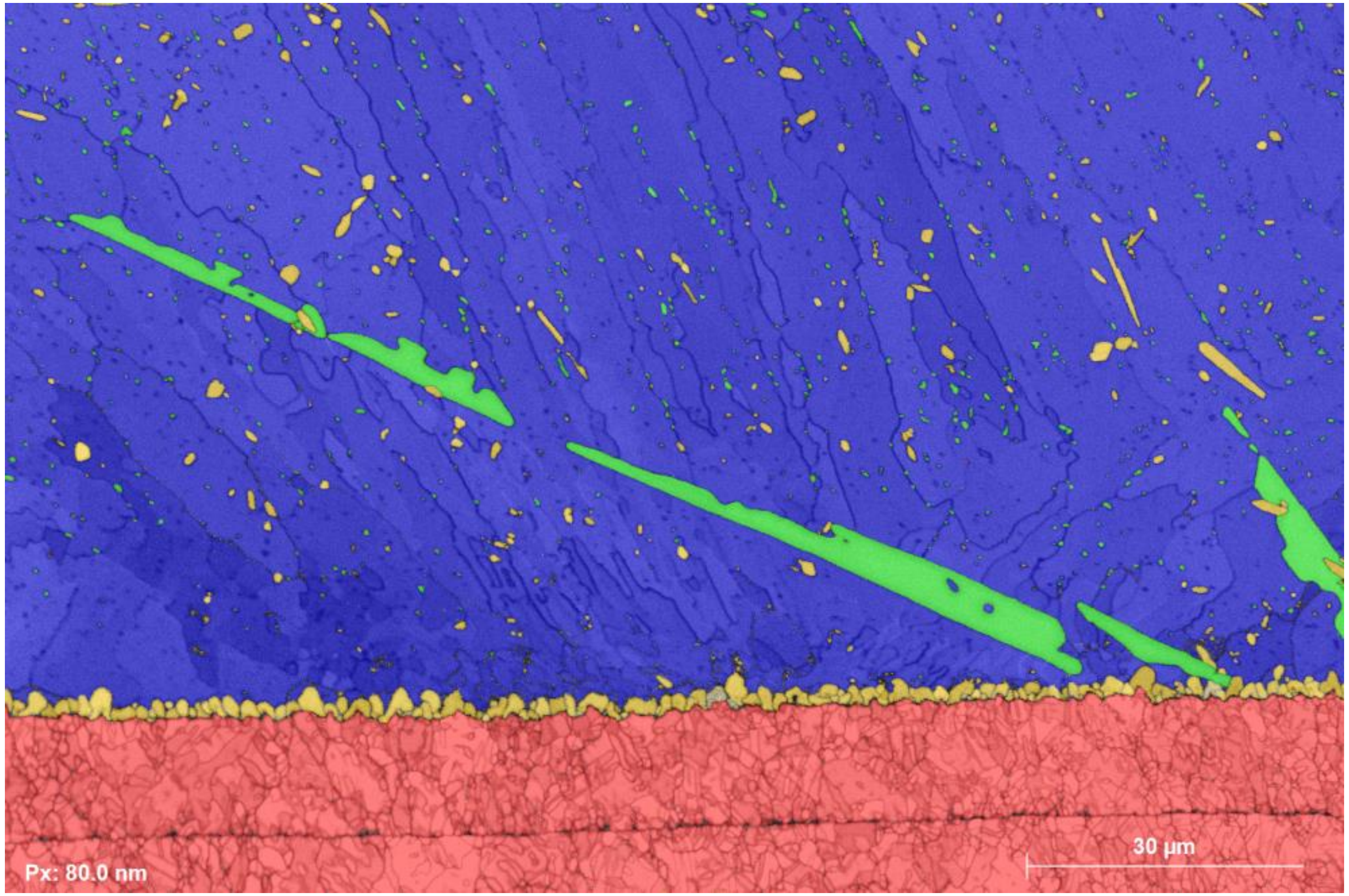
Ion milled at 6 and 2 keV; cross-section milling with mask under **cryo conditions**



# Solder bumps

## EBSD phase distribution map

Ion milled at 6 and 2 keV; cross-section milling with mask under **cryo conditions**




**Phase map**
Step size: 80 nm
Pattern resolution: 80x60 pixels
Indexing rate: 96%

- Cu
- $Cu_6Sn_5$
- $Ag_3Sn$
- Sn

# Achieve excellence in EBSD analyses
## Summary

Combining Fischione Instruments and Bruker NanoAnalytics technologies allows all user levels to quantitatively characterize a sample’s microstructure in a timely and accurate manner.

- **Fischione Instruments** provides fast and reliable sample preparation solutions for a wide range of applications and a wide range of challenging materials, such as thermally sensitive samples (solder bumps) and environmentally sensitive samples (lithium-ion batteries)
- **Bruker QUANTAX EBSD** delivers a complete solution for EBSD and TKD, with:
  - ARGUS™ imaging system with unmatched sensitivity and speed for sample preparation quality control and qualitative mapping of plastic and elastic strain in the specimen
  - high resolution EBSD measurement to characterize quantitatively the micro/nano-structure, phase distribution, texture,…, making it the ideal analytical tool for deformation studies

## Are there any questions?

Please type your questions

in the Q&A box and press *Send*

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