# QUANTAX ED-XS: Cost-Effective Solution for Material Characterization

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# Overview

01

# Why EBSD?

# Introduction

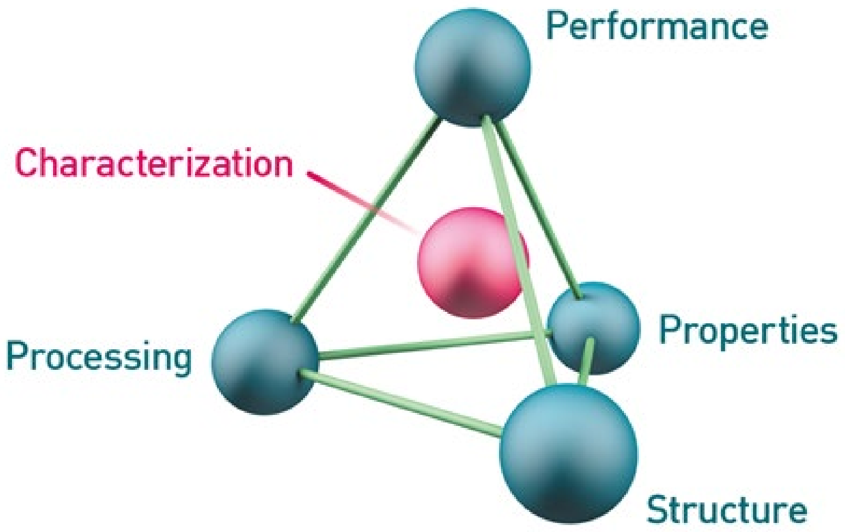
## Why EBSD?

- Understanding the material strength and deformation properties by:
  - Measuring crystal orientation
  - Analyzing the grain structure
  - Identify and Distinguish phases

- Complement the chemical information, maximize your knowledge of the sample and the processes involved
  - Texture analysis: assessing the effects of the thermomechanical processes
  - Control or investigate material properties
  - Process control: Manufacturing, R&D, Pure research
  - Complete other techniques (X-ray diffraction, TEM studies...)

>> Correlates local texture (orientation) with microstructure (grain metrics, GB, ...)

# Introduction
## Why EBSD?

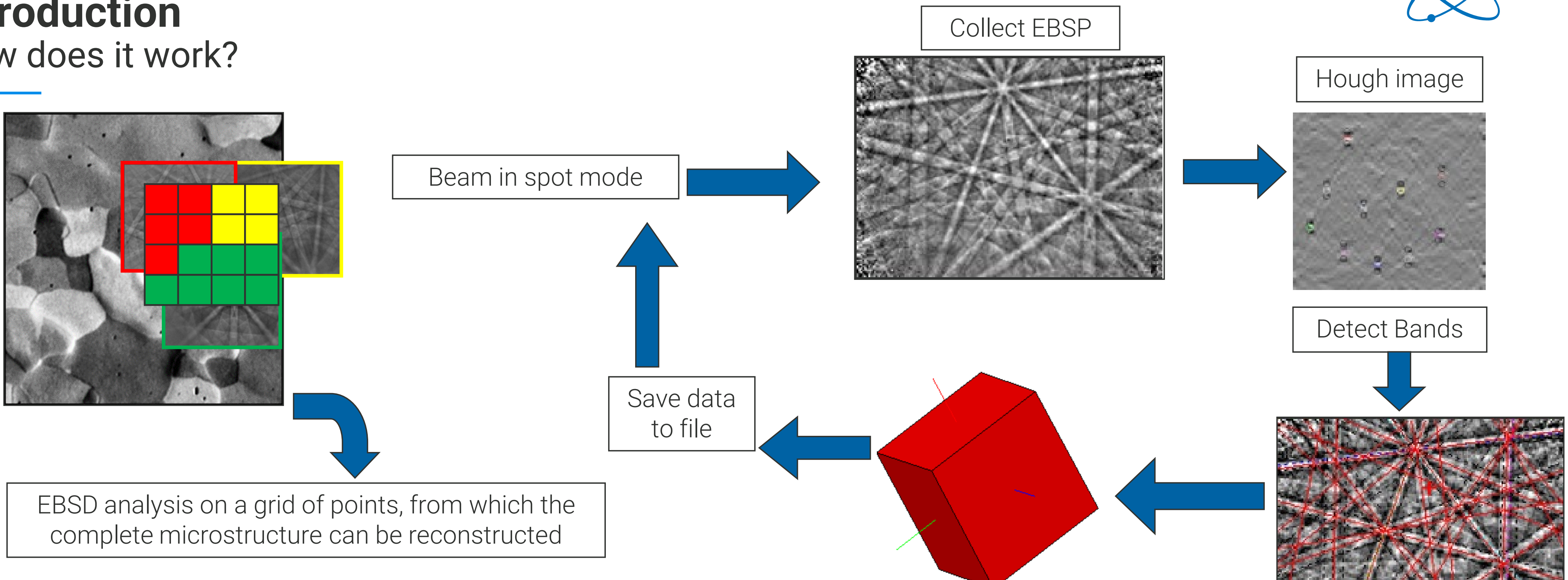



- Processing of metals & alloys
    - Control mechanical properties
    - Reach maximum performance

- Processing optimization:
    - Based on results from mechanical testing (Properties)
        - Iteratively refine processing parameters until desired properties are obtained
        - Little to no understanding of the structural changes happening during processing
    - Based on Structure-Properties relationship, i.e. **full understanding of mechanisms involved**
        - Correlate structural changes with resulting properties after applying a given thermomechanical process
        - EBSD is one of the few techniques that can help making this correlation
        - EBSD is the only technique enabling correlation between local crystal orientation distribution and microstructure (nano to meso to macro scale):

# Introduction

## How does it work?




### Advantages of EBSD analysis vs. XRD:

- corelation between local texture and microstructure
- grain boundary analysis
- grain size & shape analysis

https://www.youtube.com/watch?v=TOGRtACDw7M

02

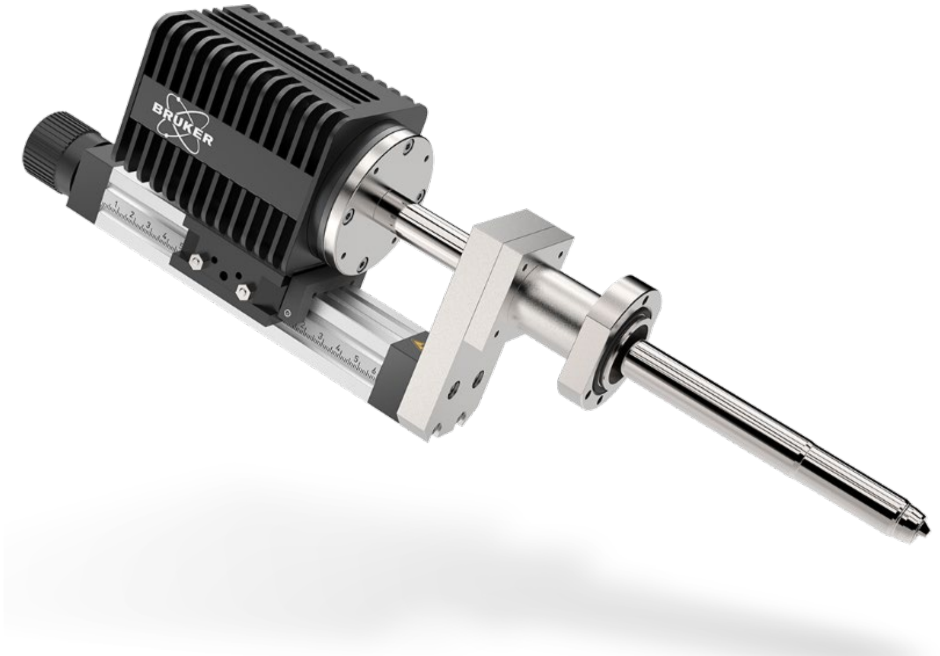
# QUANTAX ED-XS

# QUANTAX ED-XS

## The simplest and most affordable solution

**Complete integrated EDS and EBSD system:**

- eFlashXS
- Xflash® EDS detector 30mm²
- ESPRIT Software platform

# QUANTAX ED-XS
## The simplest and most affordable solution

Main specs:

- Binning capable CMOS camera with 720x540pixels native resolution
- Binning modes: 2x2, 3x3, 4x4, 5x5 and 6x6
- Max speed: 520 frames/second
- Dimensions (outer): L=84mm (3.3in), Φ=48mm (1.9in)
- Weight: ~0.85Kg
- Power and data transfer: 1x USB3.0 cable
- User replaceable phosphor screen
- User removable detector head with slide-in & -out mechanism
- Custom optics system with field lens for maximum light efficiency

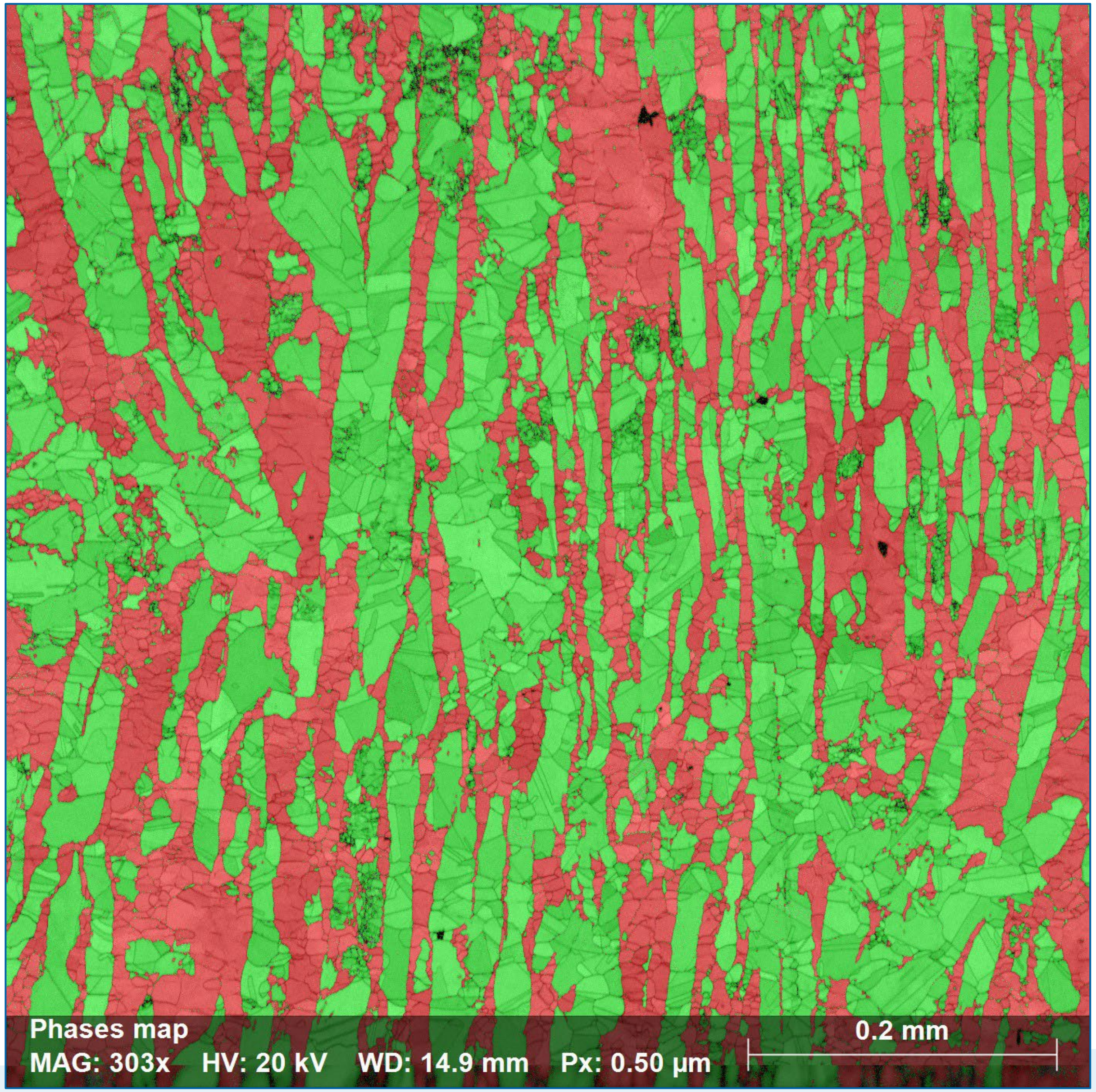
# QUANTAX ED-XS
## The simplest and most affordable solution

- EBSD detector is static – easy to operate
- Automatic gain and no -or little- exposure time adjustment – easy to operate
- No pattern center calibration required – easy to operate
- Automated crystal phase setup – easy to operate
- User replaceable phosphor screen and Field Replacement Unit – low downtime
- Automated data saving – Prevents data loss
- Automatic “EHT OFF” at the end of map acquisition – Saves filament life

# Introduction

## What is EBSD used for?

- Quantification of microstructural features:
  - Phase (crystallographic) ratio and spatial distribution
    - 43% Ferrite
    - 57% Austenite

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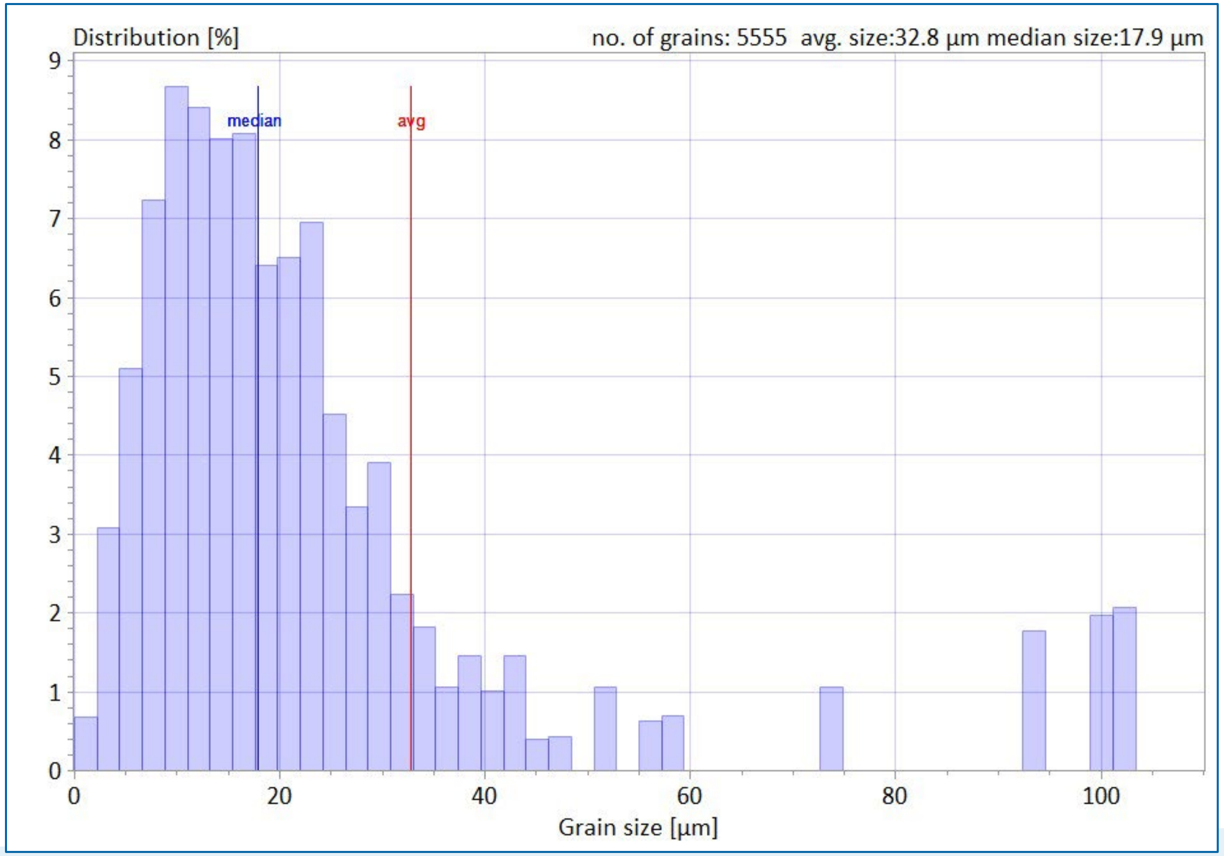
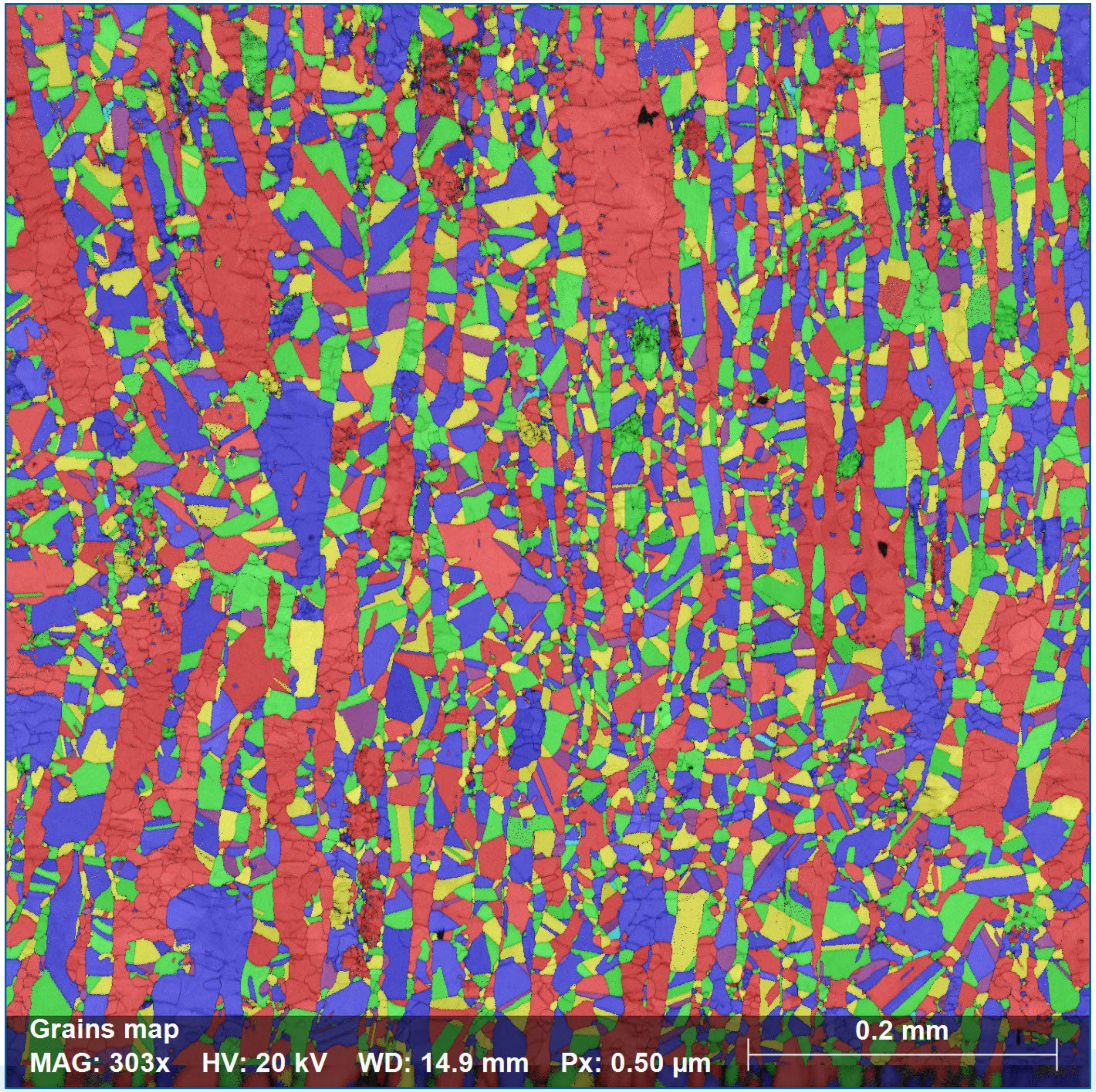
# Introduction

## What is EBSD used for?

- Quantification of microstructural features:
  - Phase (crystallographic) ratio and spatial distribution
  - Grain size and distribution

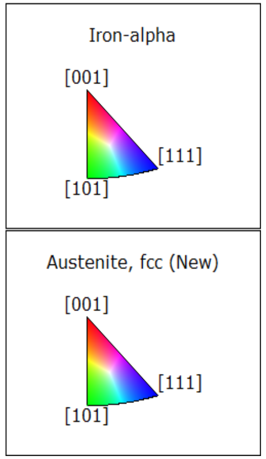


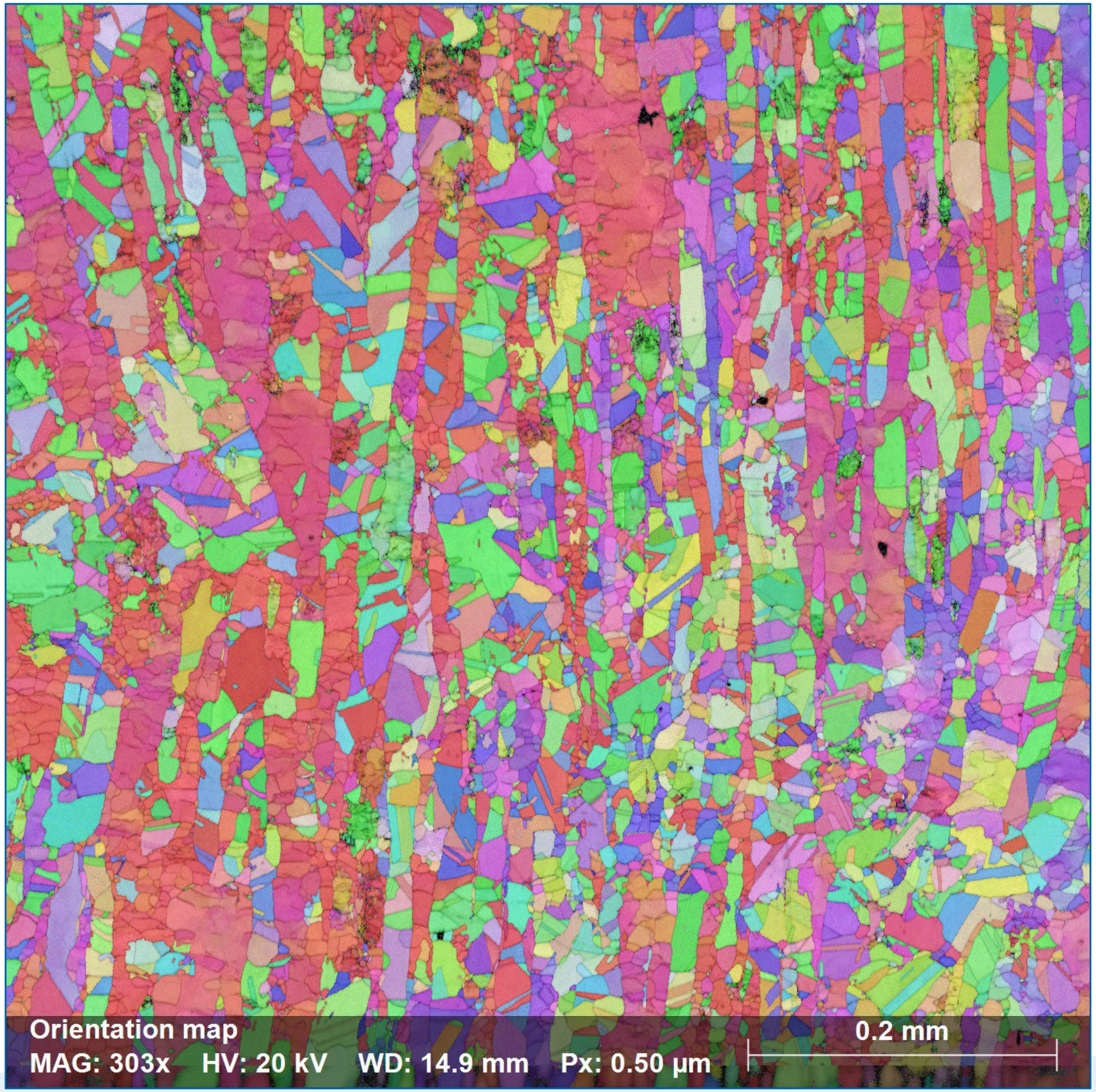



# Introduction

## What is EBSD used for?

- Quantification of microstructural features:
    - Phase (crystallographic) ratio and spatial distribution
    - Grain size and distribution
    - Orientation distribution map

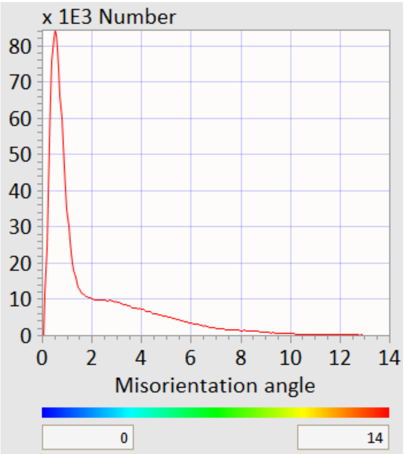


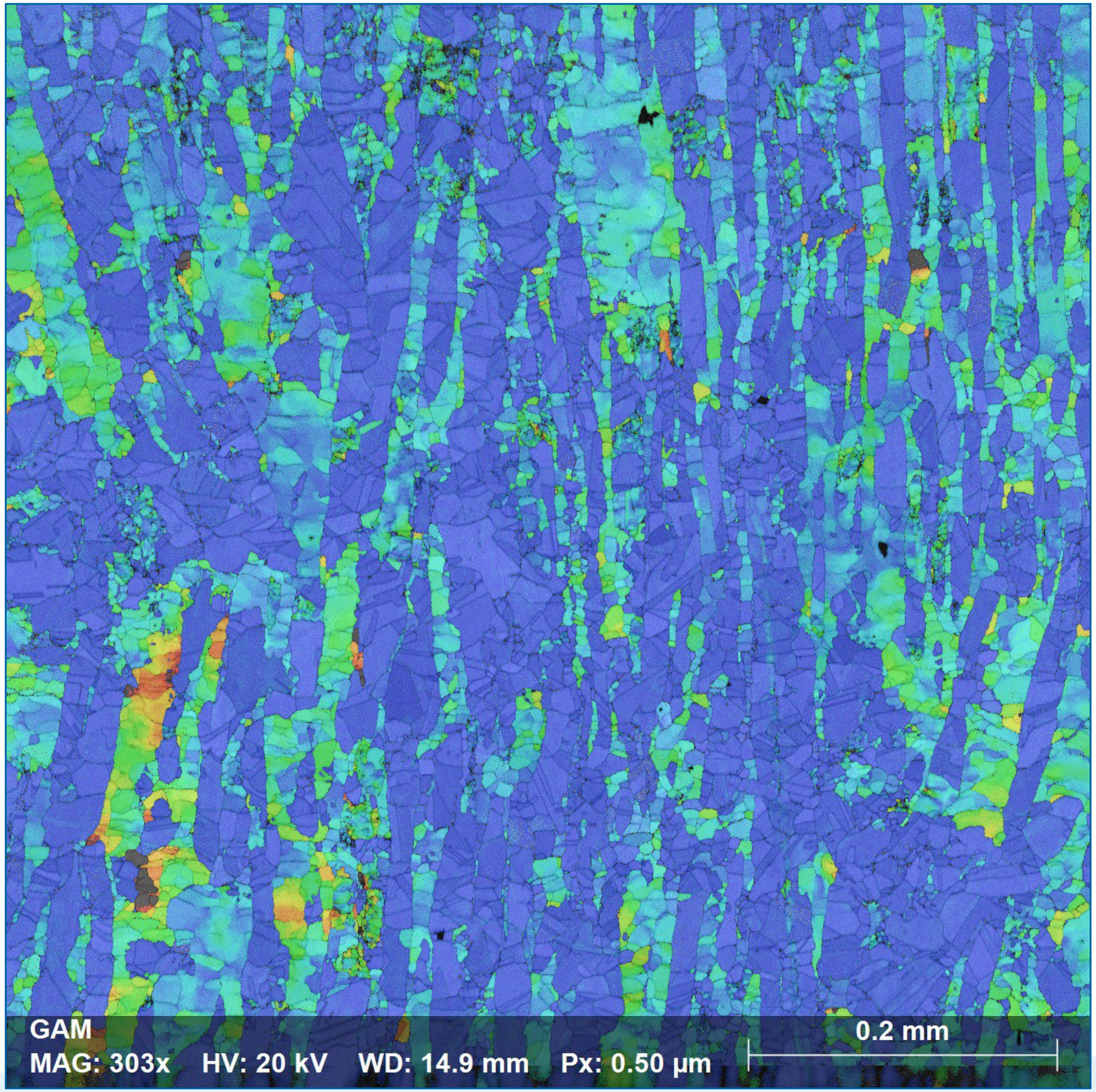



# Introduction

## What is EBSD used for?

- Quantification of microstructural features:
  - Phase (crystallographic) ratio and spatial distribution
  - Grain size and distribution
  - Orientation distribution map
  - Grain (and Kernal) Average Misorientation for:
    - Deformation state
    - Deformed vs. Recrystallized ratio



03

# Application Examples

# Simplicity Delivers Affordable Science

## Application example

Characterization of microstructural features in stainless steels

Duplex Steel with slightly deformed Ferrite grains

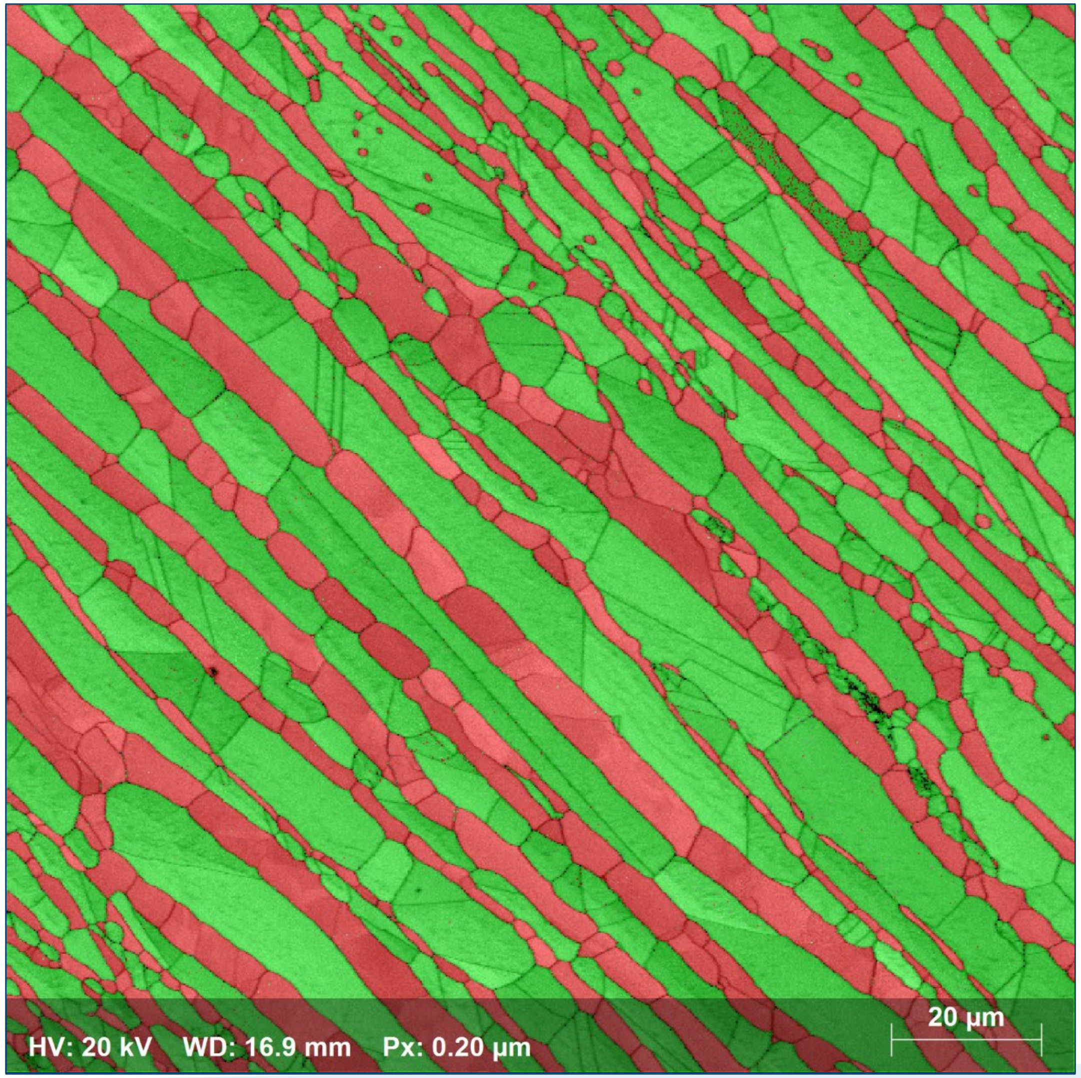
# Simplicity Delivers Affordable Science

## Qualitative & quantitative characterization

Important parameters:

- EHT: 20kV
- Probe current: ~5nA
- Step size: 200nm
- Acquisition speed: 502 frames/second
- Total acquisition time: 16:44 minutes
- Map size: 504k pixels
- Zero solutions: 0.8% NO DATA CLEANING!!
- Phase ratio (normalized):
  - 38% Ferrite
  - 62% Austenite

Duplex steel – phase ratio & distribution



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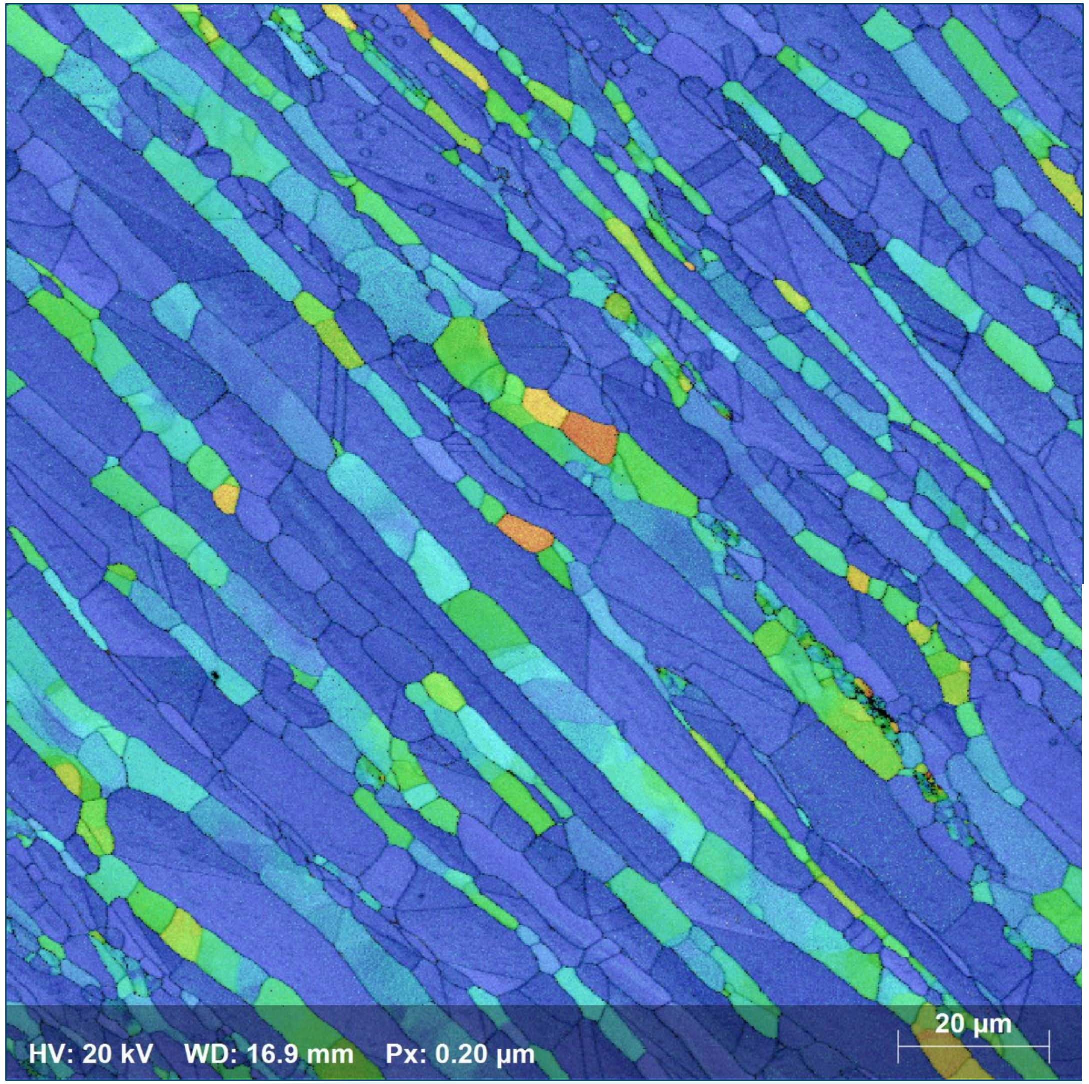
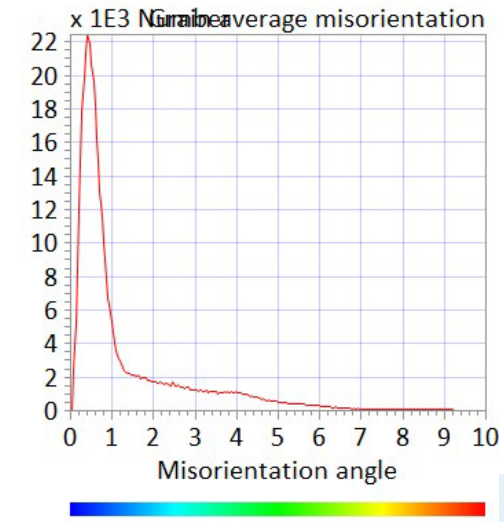
## Qualitative & quantitative characterization




Characterization of grain deformation state




Features:

- Orientation distribution map
- Grain Average Misorientation

Analysis options:

- Deformation state
- Deformed vs. Recrystallized ratio

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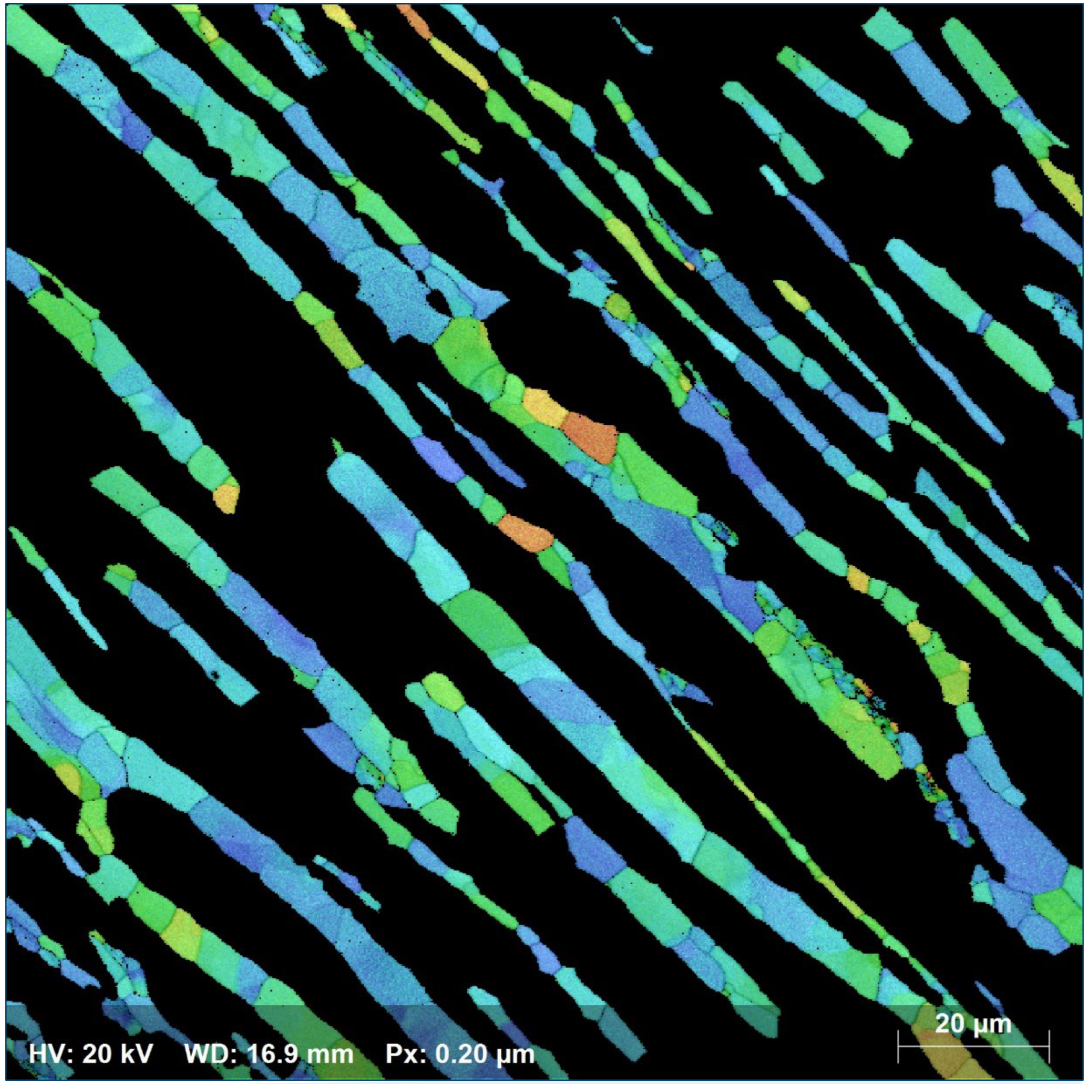
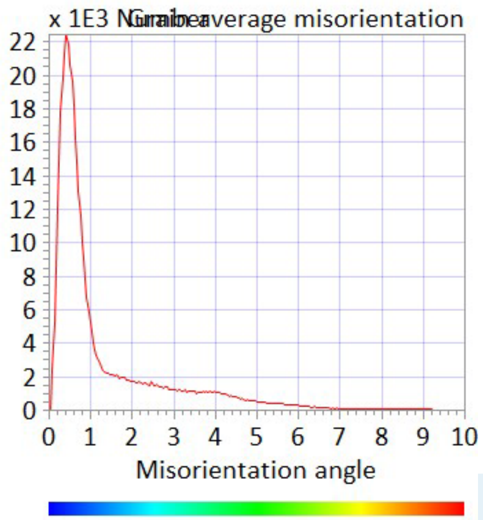
## Qualitative & quantitative characterization




Characterization of grain deformation state




Features and analysis options:

- GAM map for Austenite grains
- GAM map for Ferrite grains
- Subset of all deformed grains covering 30.2% of field of view

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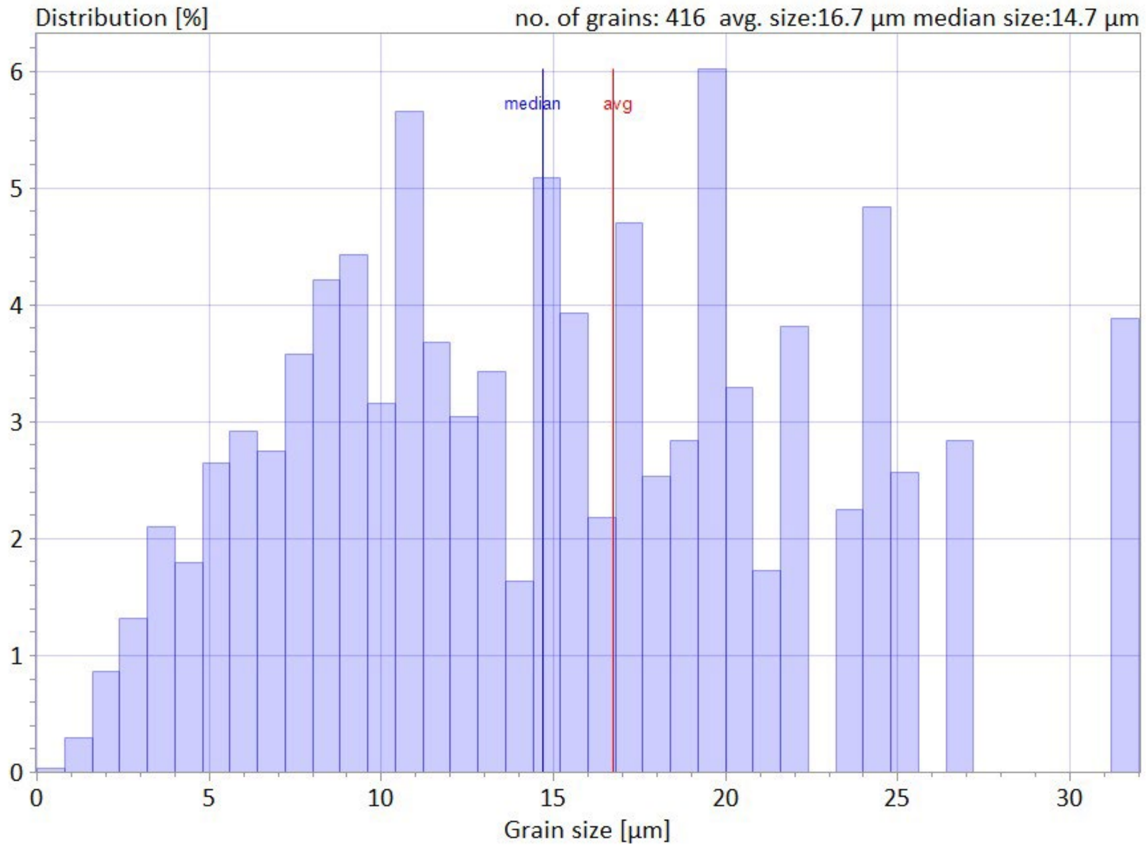
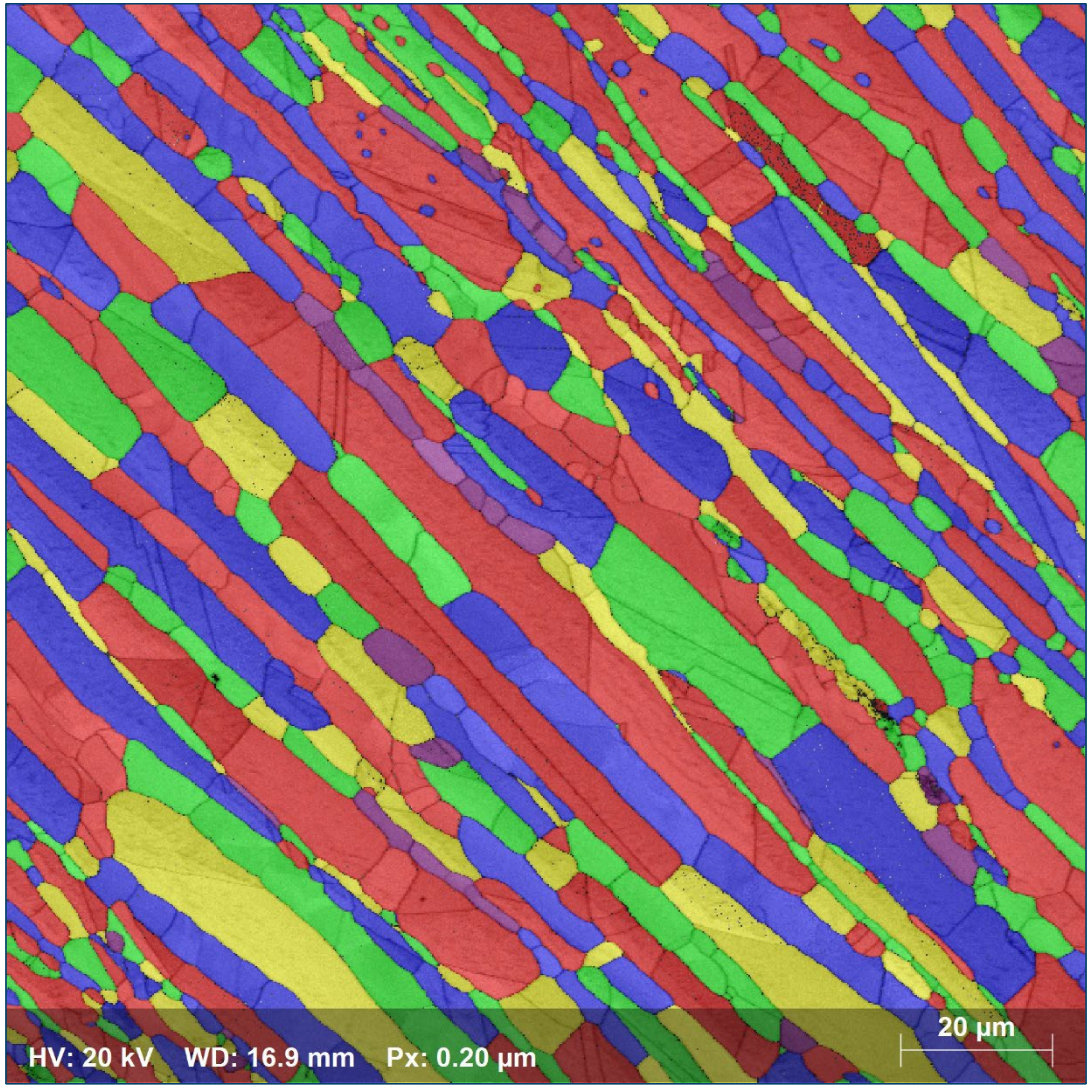
## Qualitative & quantitative characterization

Grain size distribution – both phases




Grains map – grains shown in random colors



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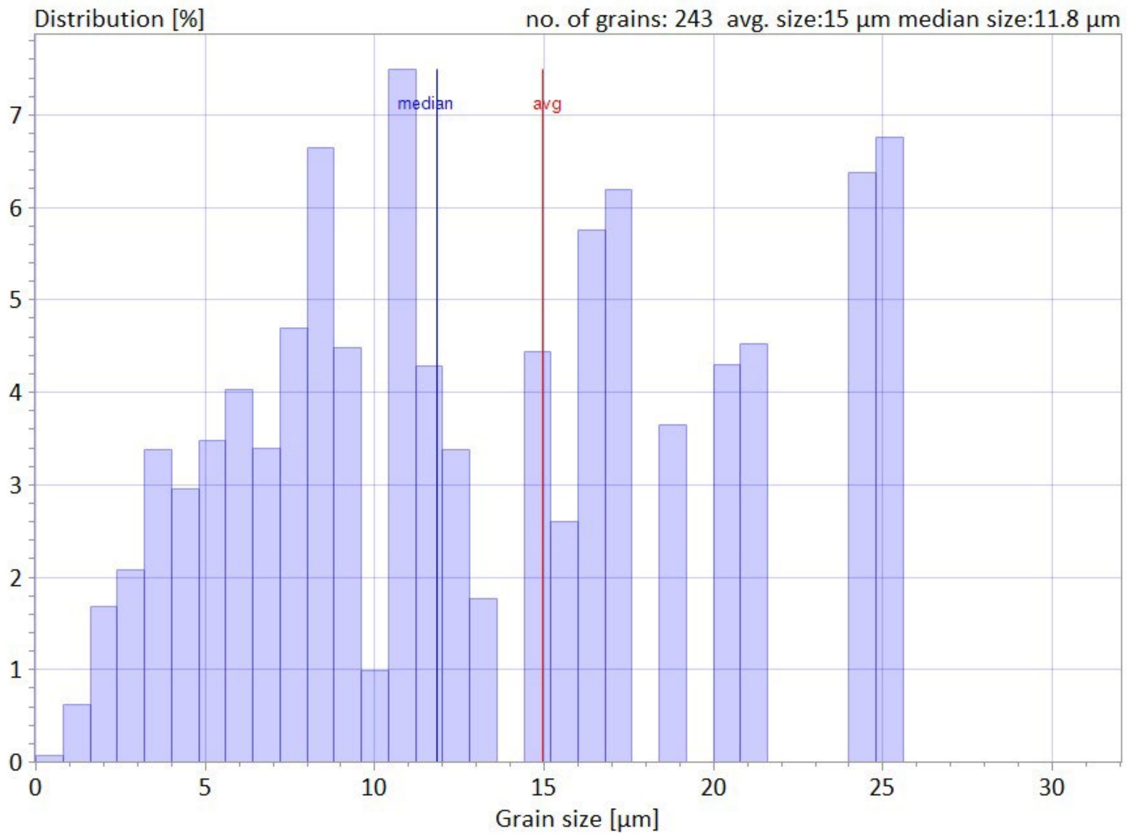
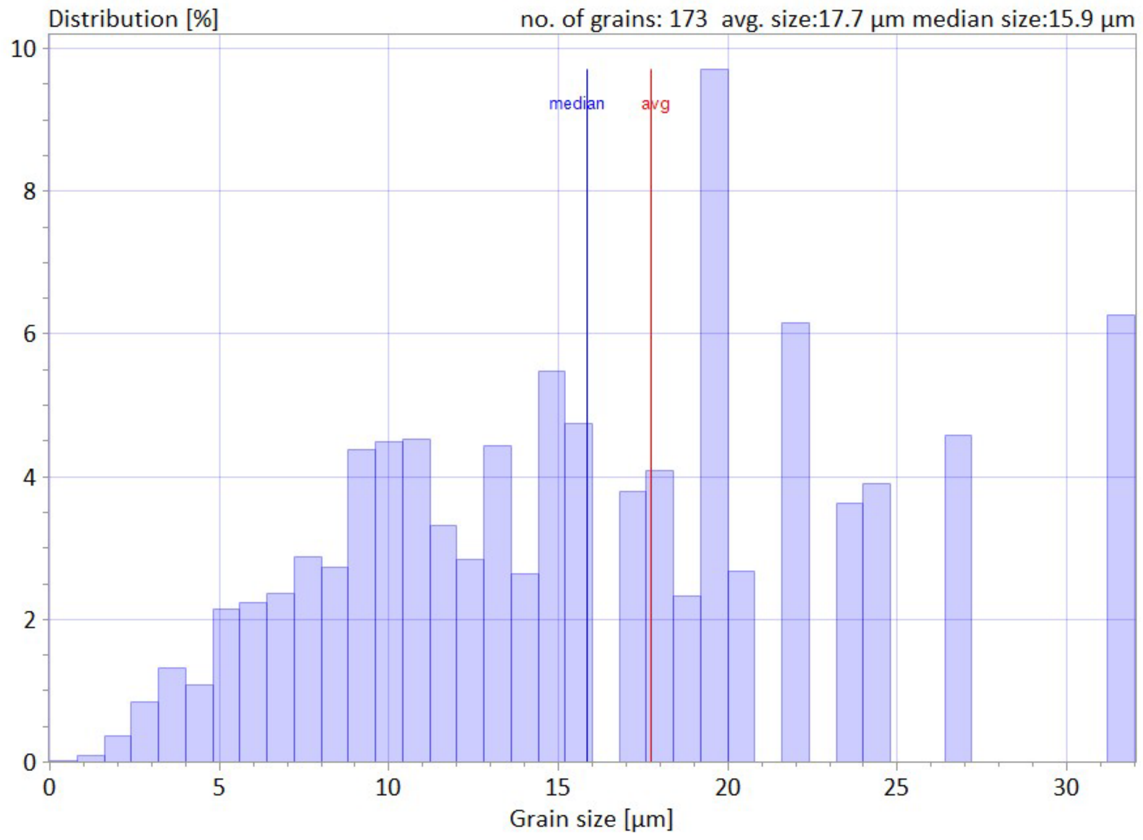
## Qualitative & quantitative characterization

### Grain size distribution Ferrite




### Grain size distribution Austenite



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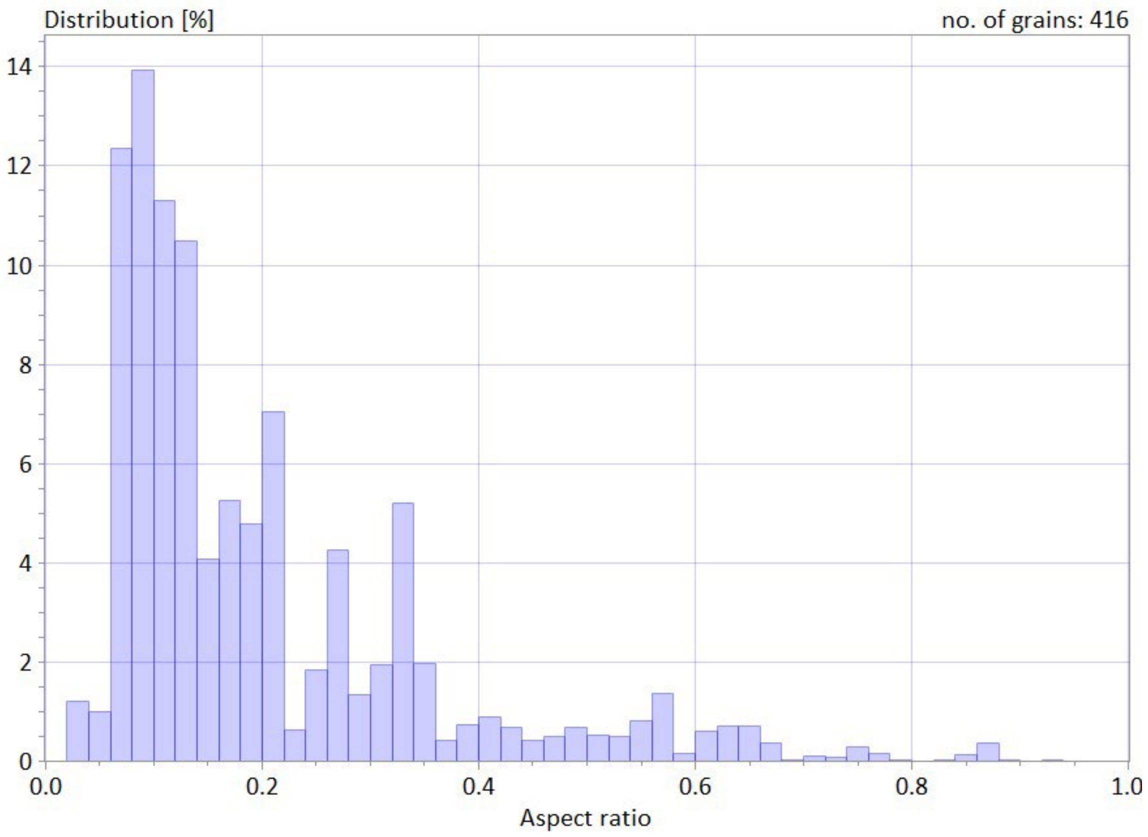
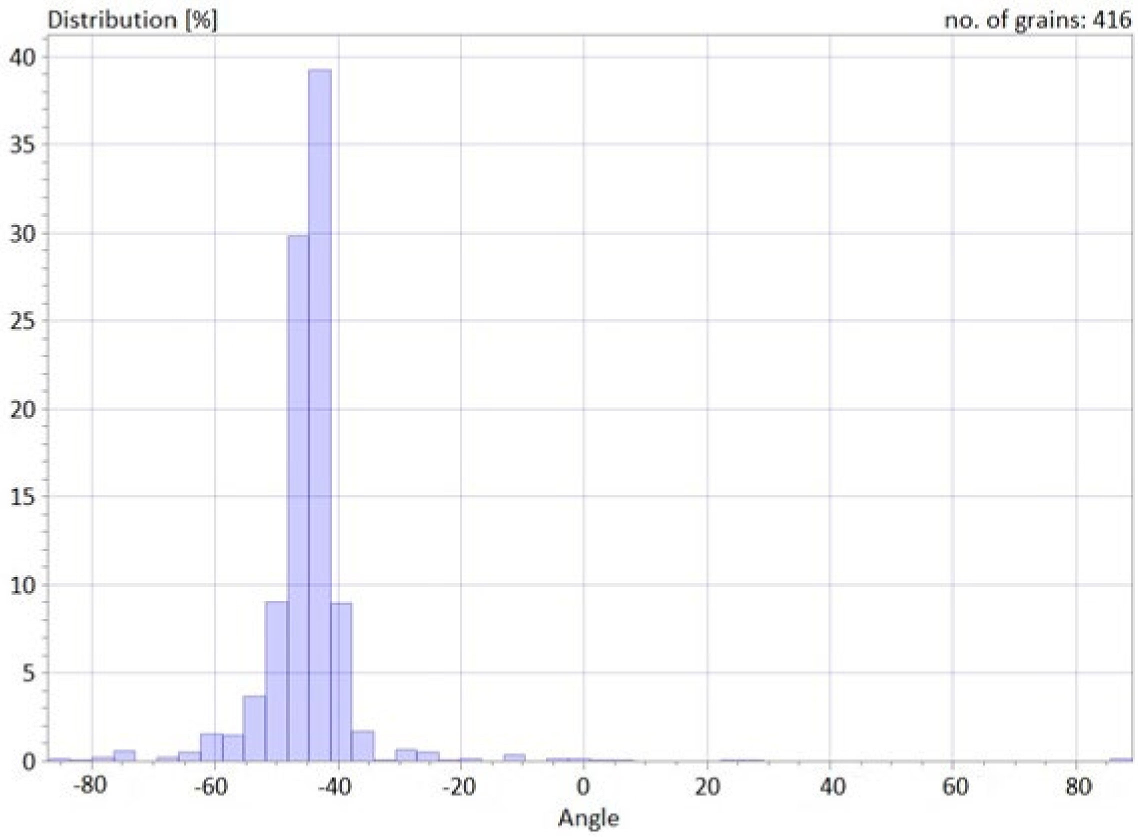
## Qualitative & quantitative characterization

Grain shape distribution




Main axis inclination or grain alignment distribution



# Simplicity Delivers Affordable Science

## Application example

High resolution measurements on dual-phase Ti-alloy

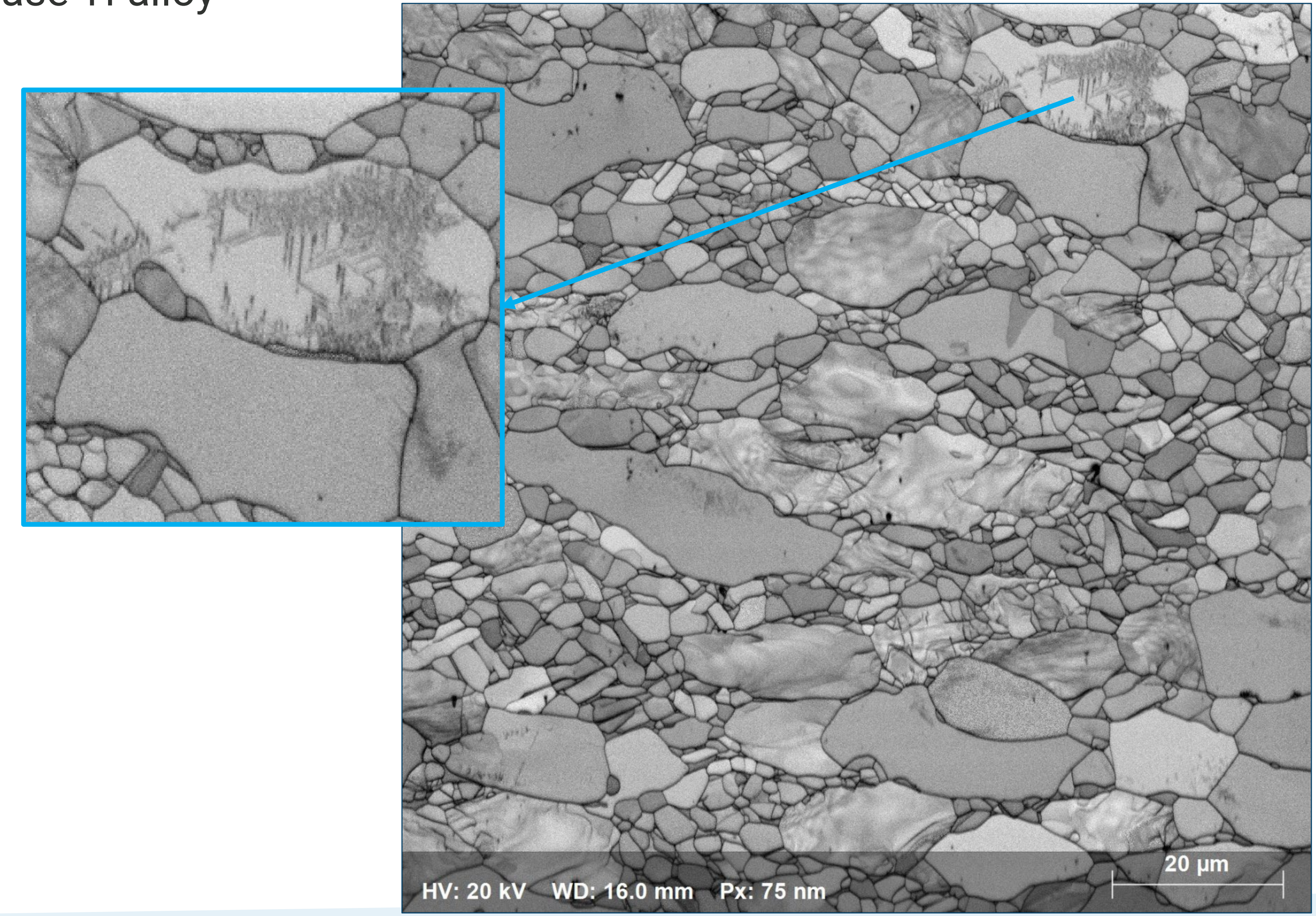
# Simplicity Delivers Affordable Science

## High spatial resolution_ dual-phase Ti-alloy

Important parameters:

- EHT: 20kV
- Probe current: ~3 nA
- **Step size: 75 nm**
- Acquisition speed: >100 fps
- Map size: **+2M pixels**
- > 1000 grains
- Pattern resolution: 180x135 pixels
- Zero sol.: 1.48 %

Pattern quality map



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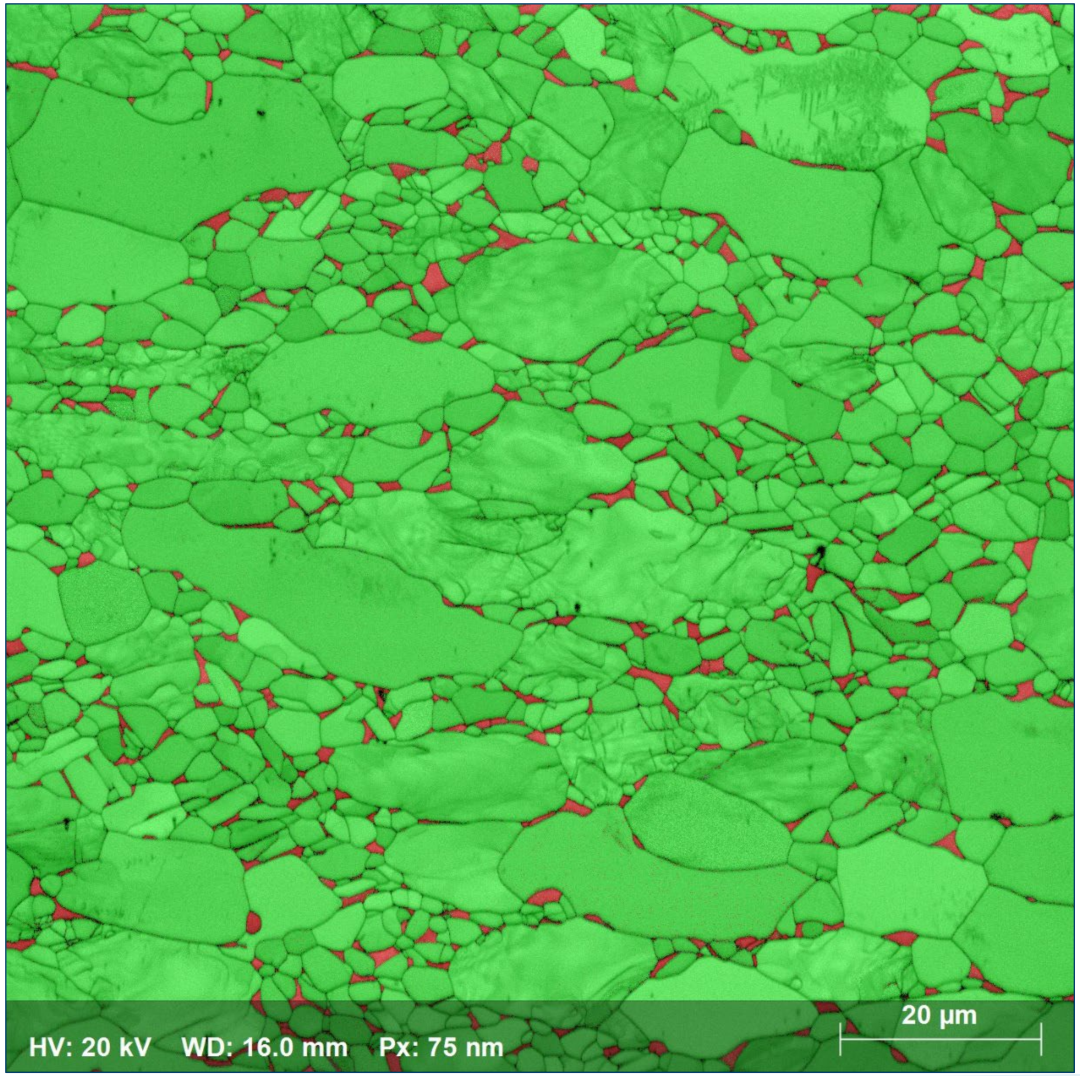
## Quantitative microstructure characterization_ dual-phase Ti-alloy




Phase fractions: 94,3% **alpha-Ti** and 4,22% **beta-Ti**

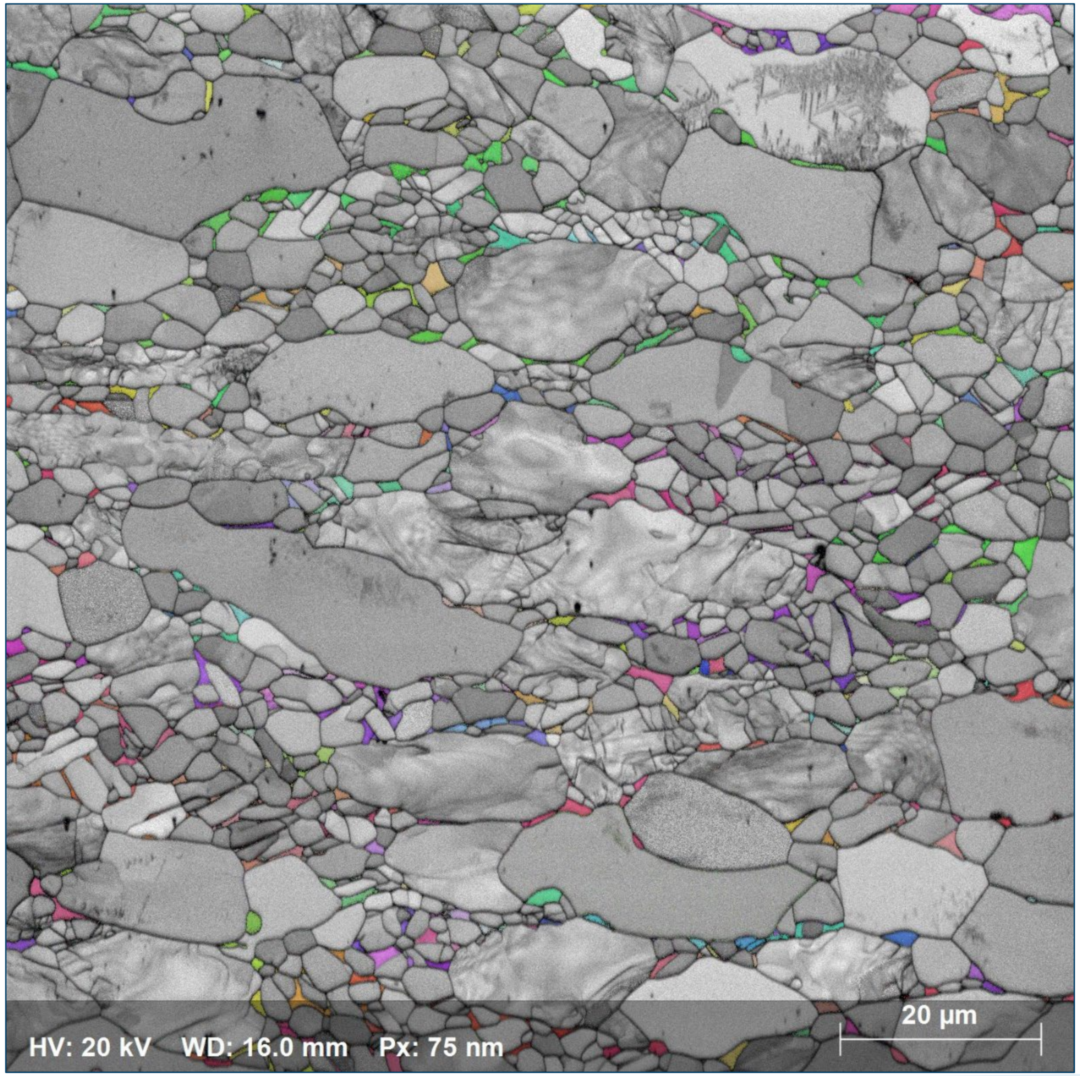
Possible subsetting based on:

- Phase
- Pattern quality value
- Crystal orientation (pole figure, texture component)
- GAM and Kernel Average Misorientation (KAM)
- Grain size, shape, main axis inclination

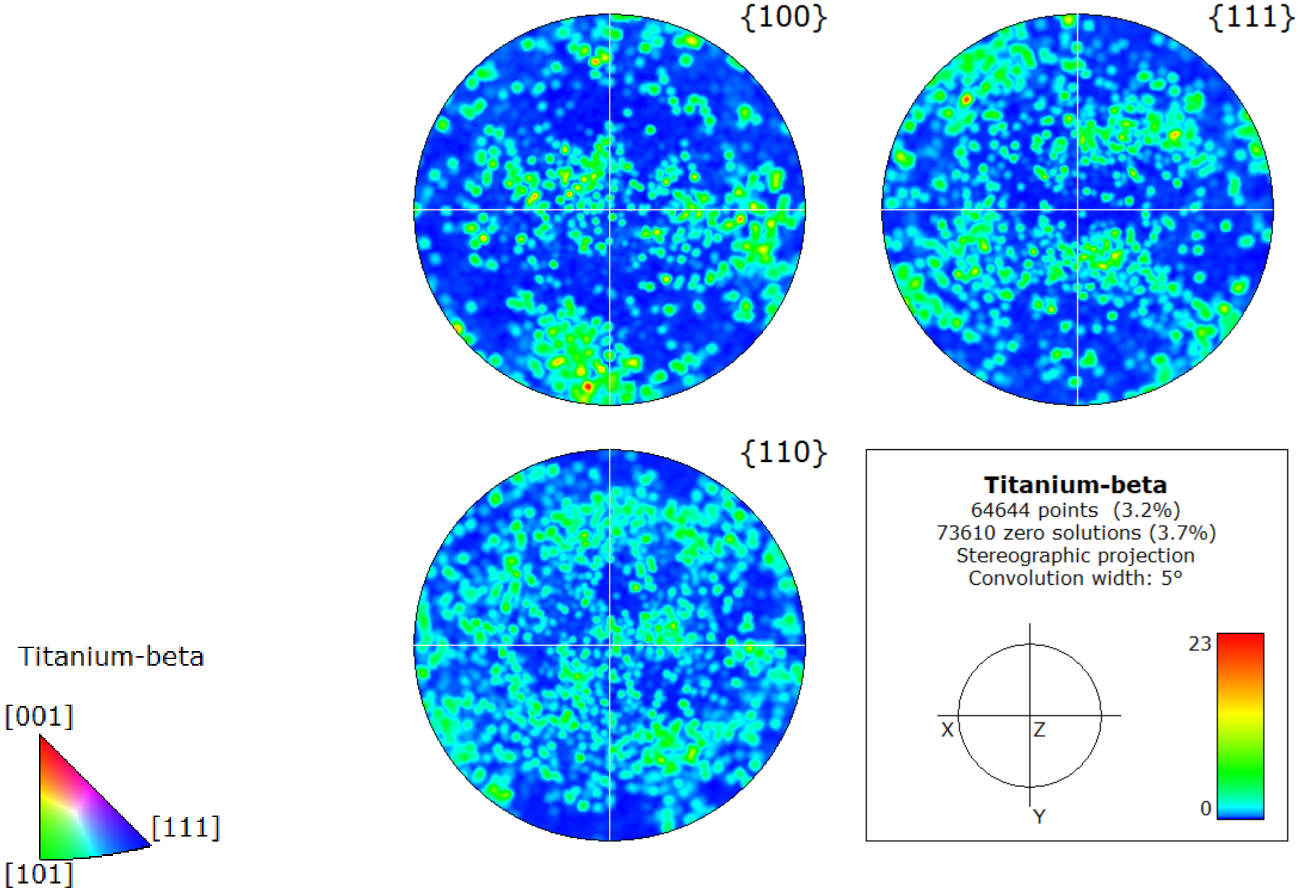
NO DATA CLEANING!!

# Simplicity Delivers Affordable Science

## Quantitative microstructure characterization_ dual-phase Ti-alloy

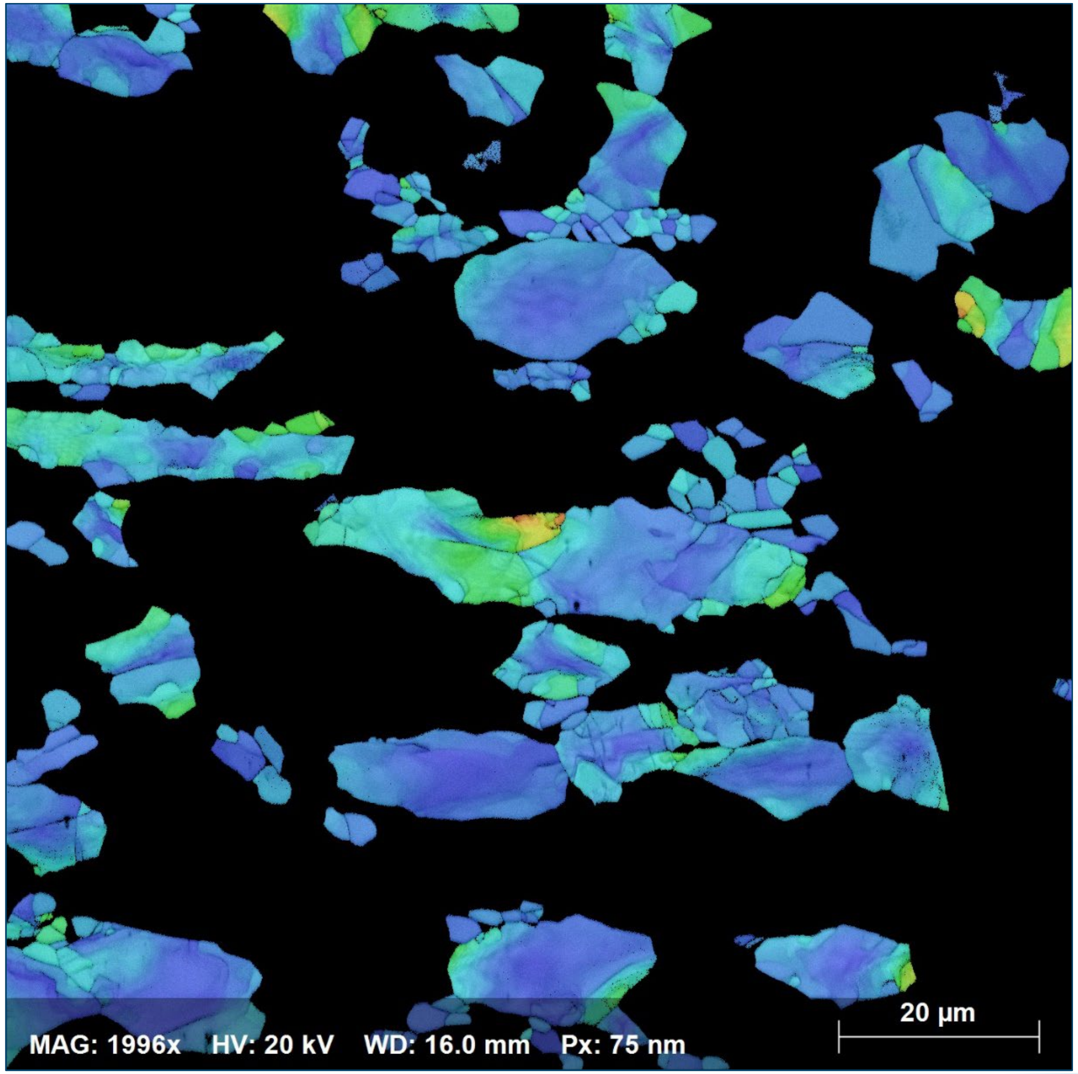


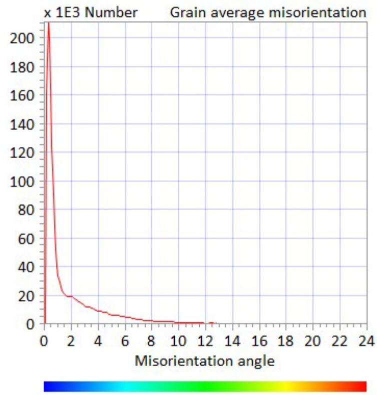

- Beta-Ti phase subset
- Beta-Ti pole figures



# Simplicity Delivers Affordable Science

## Quantitative microstructure characterization_ dual-phase Ti-alloy



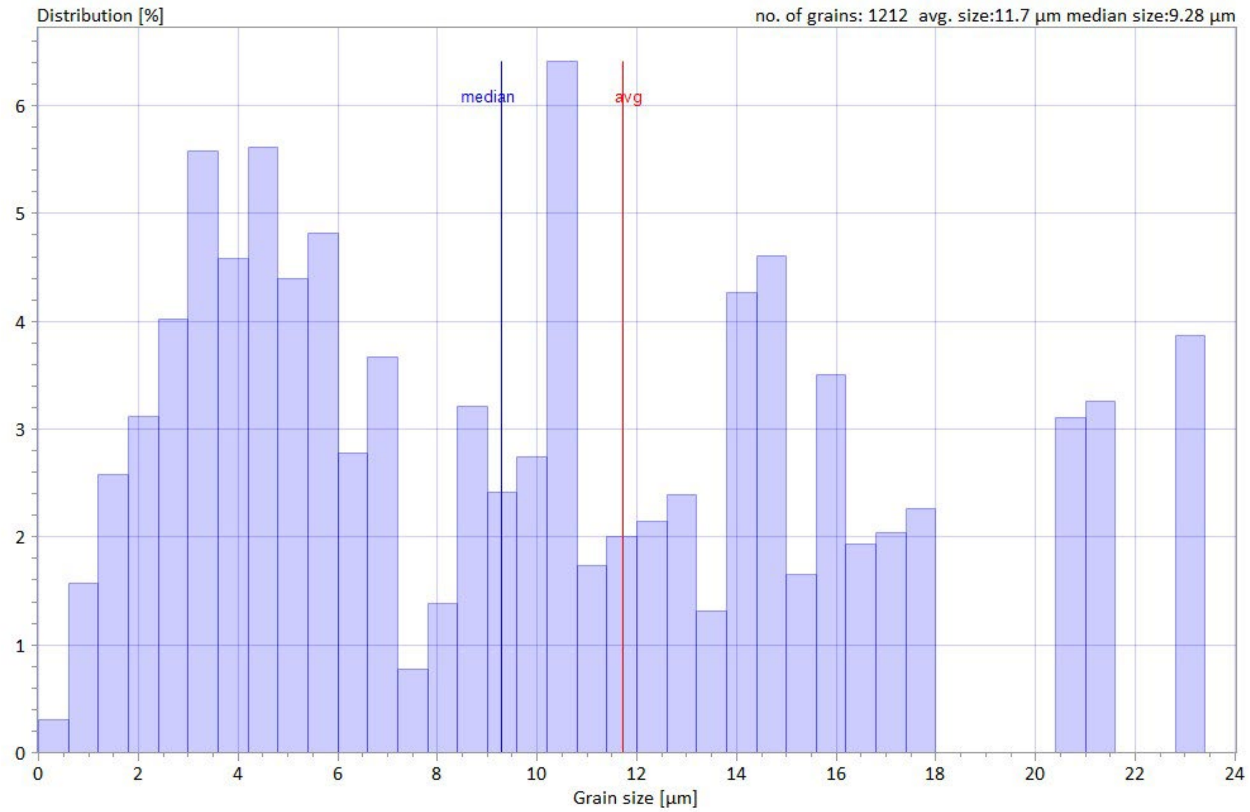

Important details:

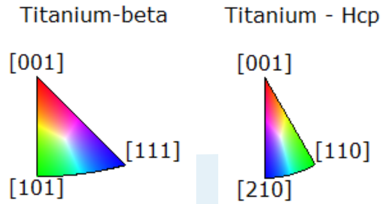
- No data cleaning applied
- Step size: 75 nm
- GAM map showing intragranular plastic deformation
- GAM used for creating subsets
- Deformed grains represent 31.2 % of mapped area

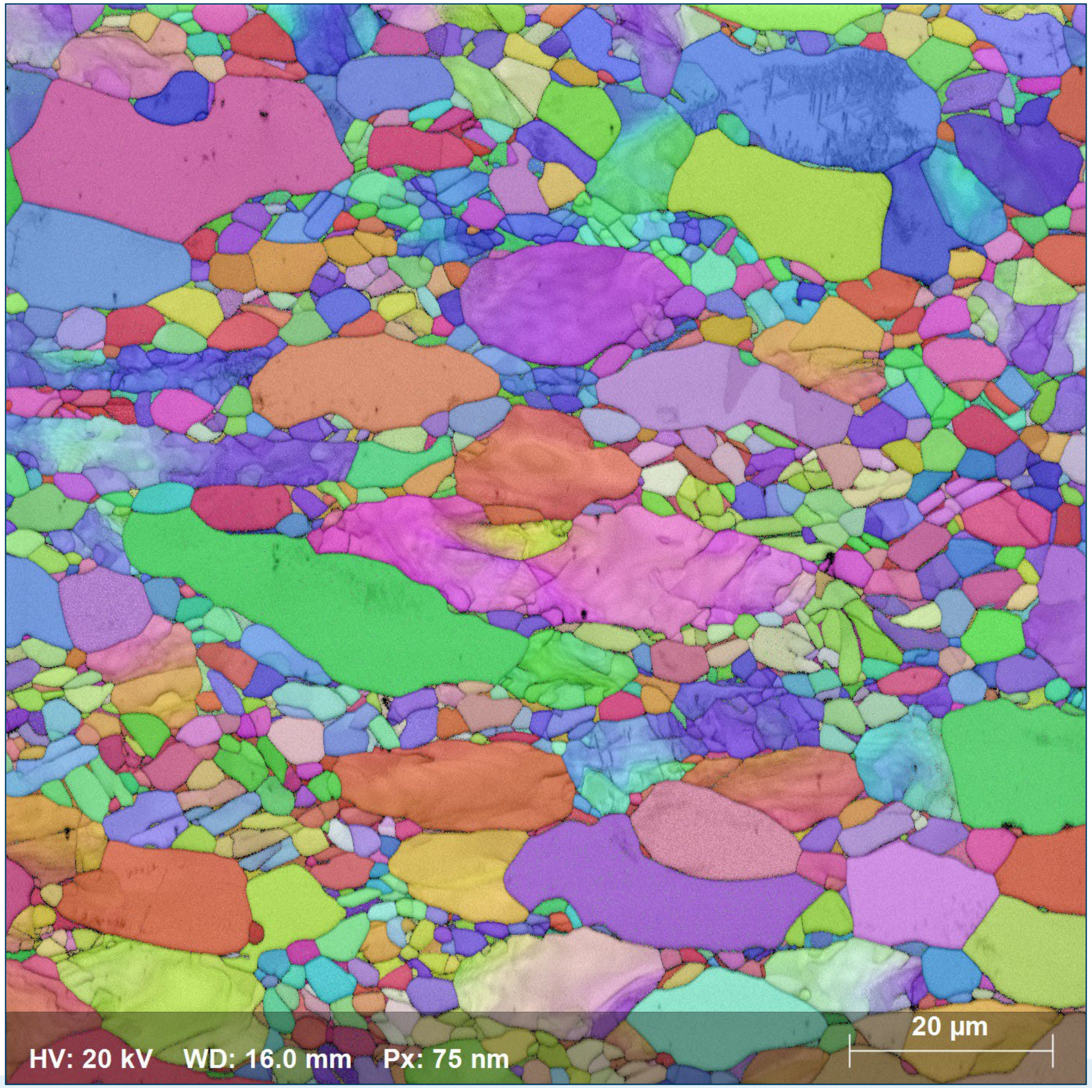
# Simplicity Delivers Affordable Science

## Quantitative microstructure characterization_ dual-phase Ti-alloy

Area weighted grain size distribution and statistics (all phases)



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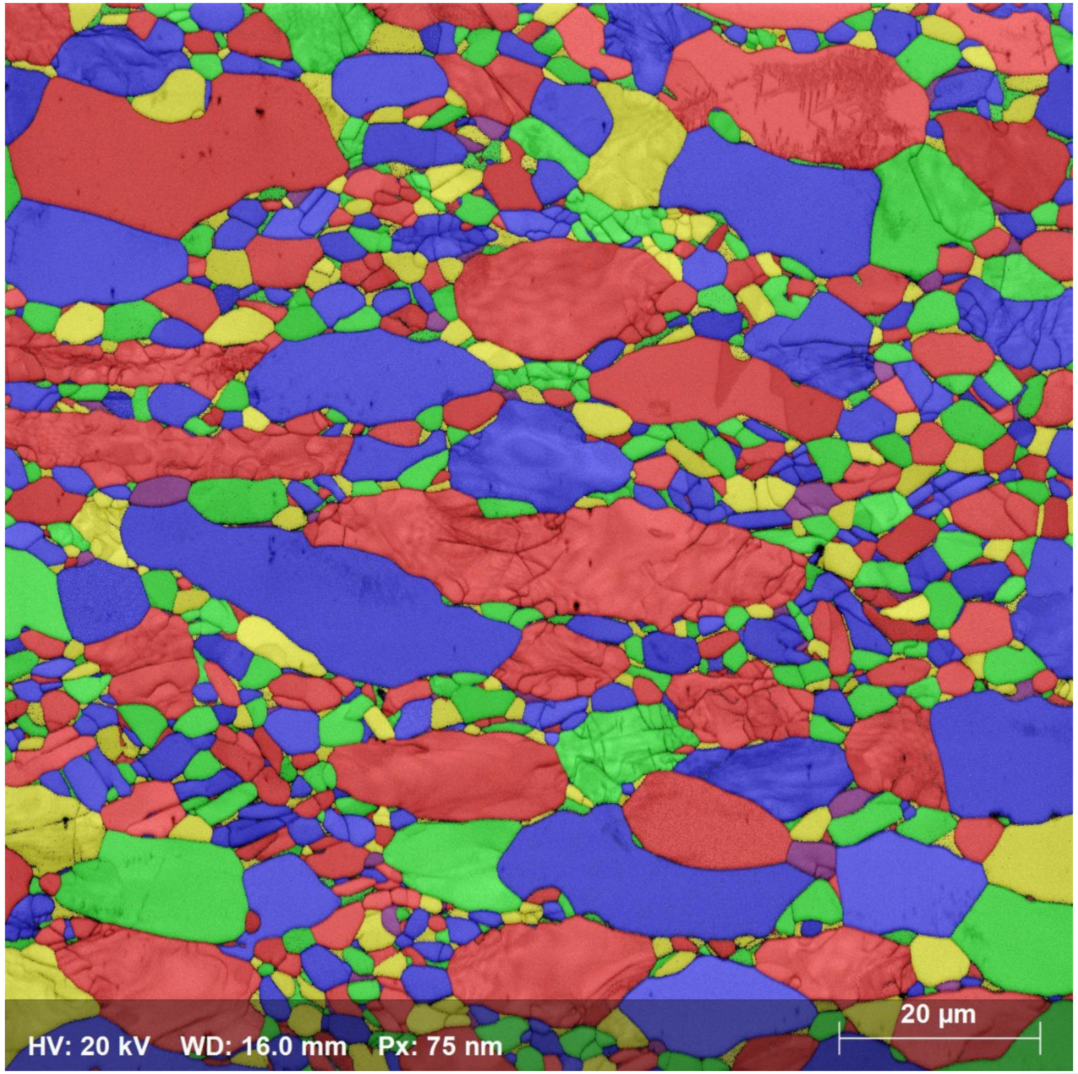
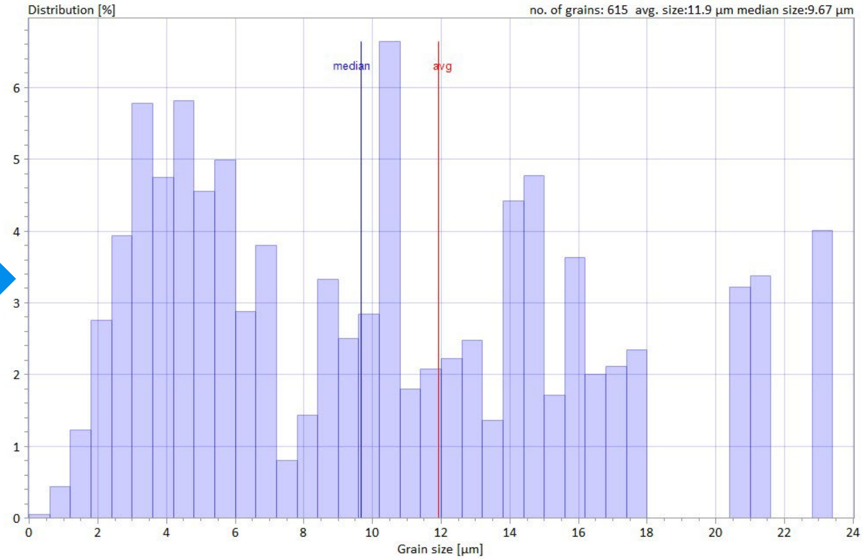
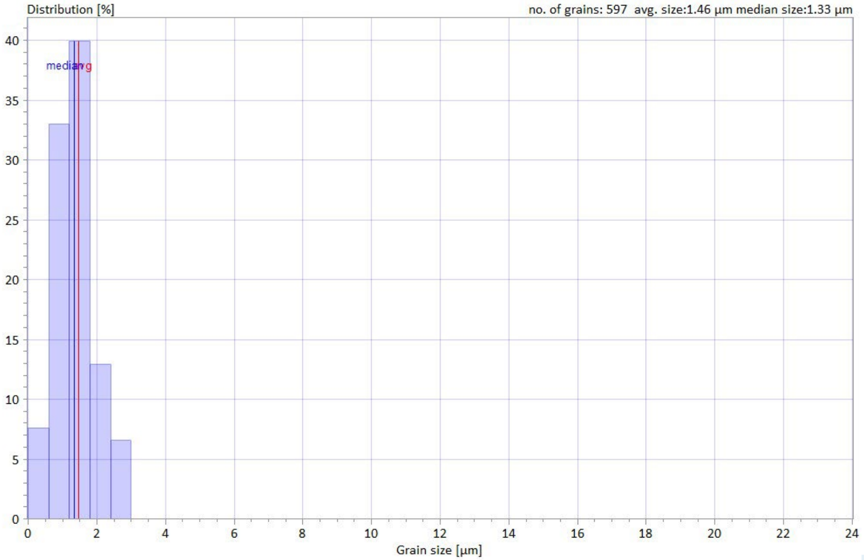
## Quantitative microstructure characterization_ dual-phase Ti-alloy




- Grains map
- Alpha-Ti grain stats
- Beta-Ti grain stats



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## Application example

High speed measurements on materials with low electron scattering yield

Amorphous Si film crystallized using a scanning laser beam

# Simplicity Delivers Affordable Science

## Qualitative & quantitative characterization_ Si film

Important parameters:

- EHT: 20kV
- Probe current: ~5nA
- Step size: 0.25 µm
- Acquisition speed: 505 frames/second
- Total acquisition time: 1:06 h
- Map size: ~2M pixels
- Pattern resolution: 144x108 pixels
- Zero sol.: 0.8% (excluding amorphous region)

NO DATA CLEANING!!

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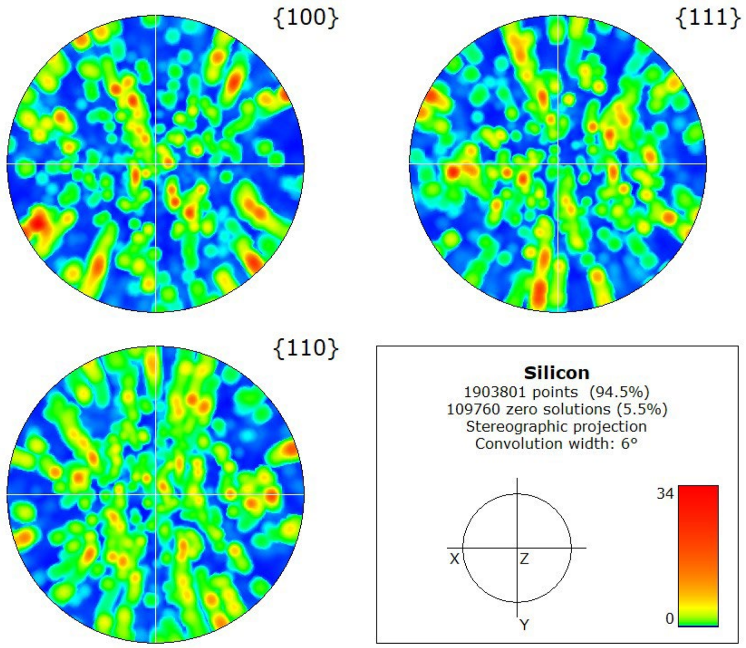
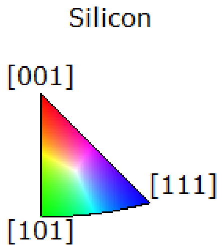
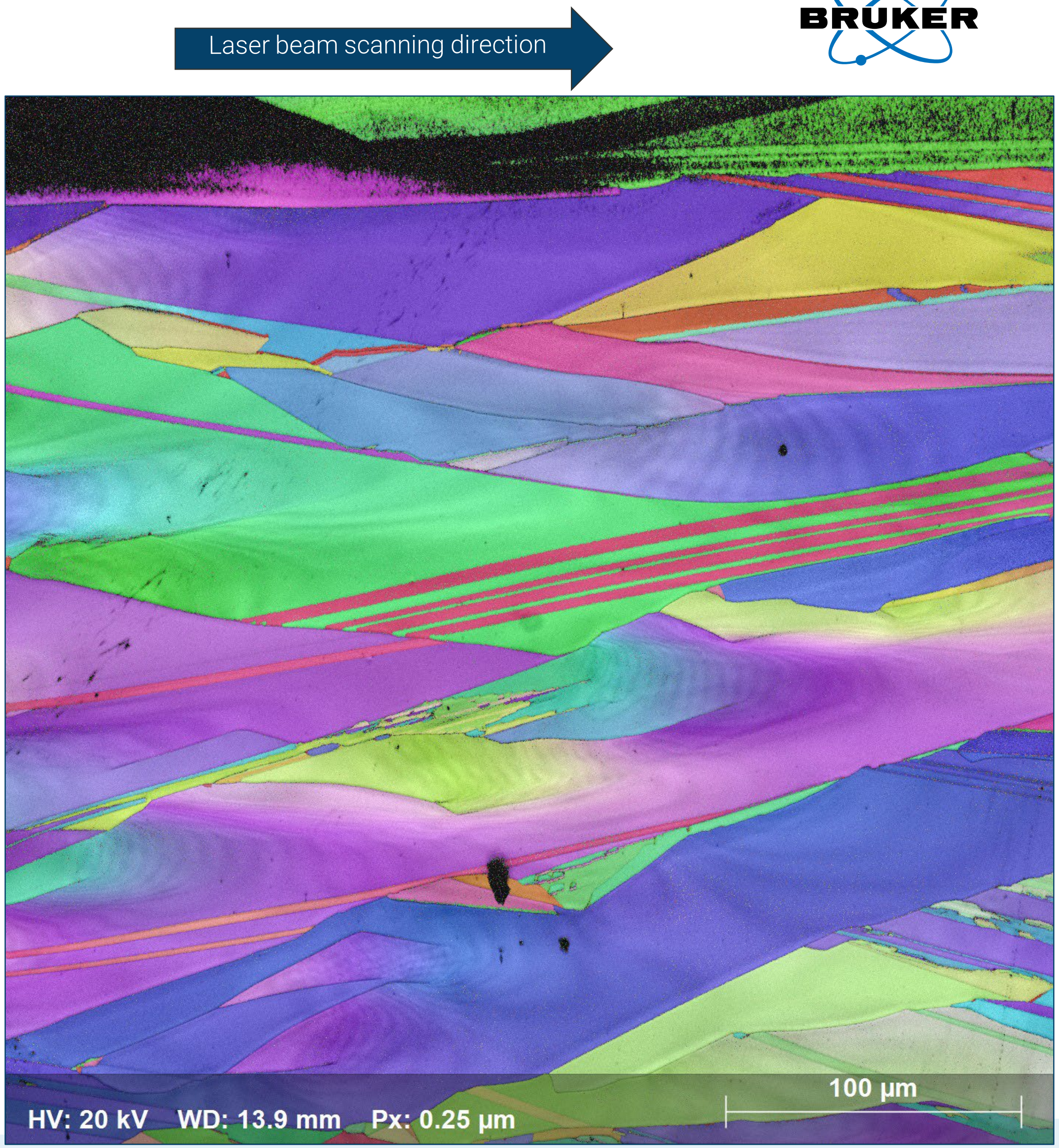
## Qualitative & quantitative characterization_ Si film

- Crystal orientation map
  - Fine twin domains
  - Significant lattice rotations across large elongated grains
- Crystallographic texture analysis
  - {110} partial fiber along the laser beam scan direction



# Simplicity Delivers Affordable Science

## Application example

Phase identification and distribution analysis of little-known multi-phase containing materials

(applicable to both: materials and earth sciences)

Highly alloyed Fe-Si composite

# Simplicity Delivers Affordable Science

## Analysis of little-known multi-phase materials

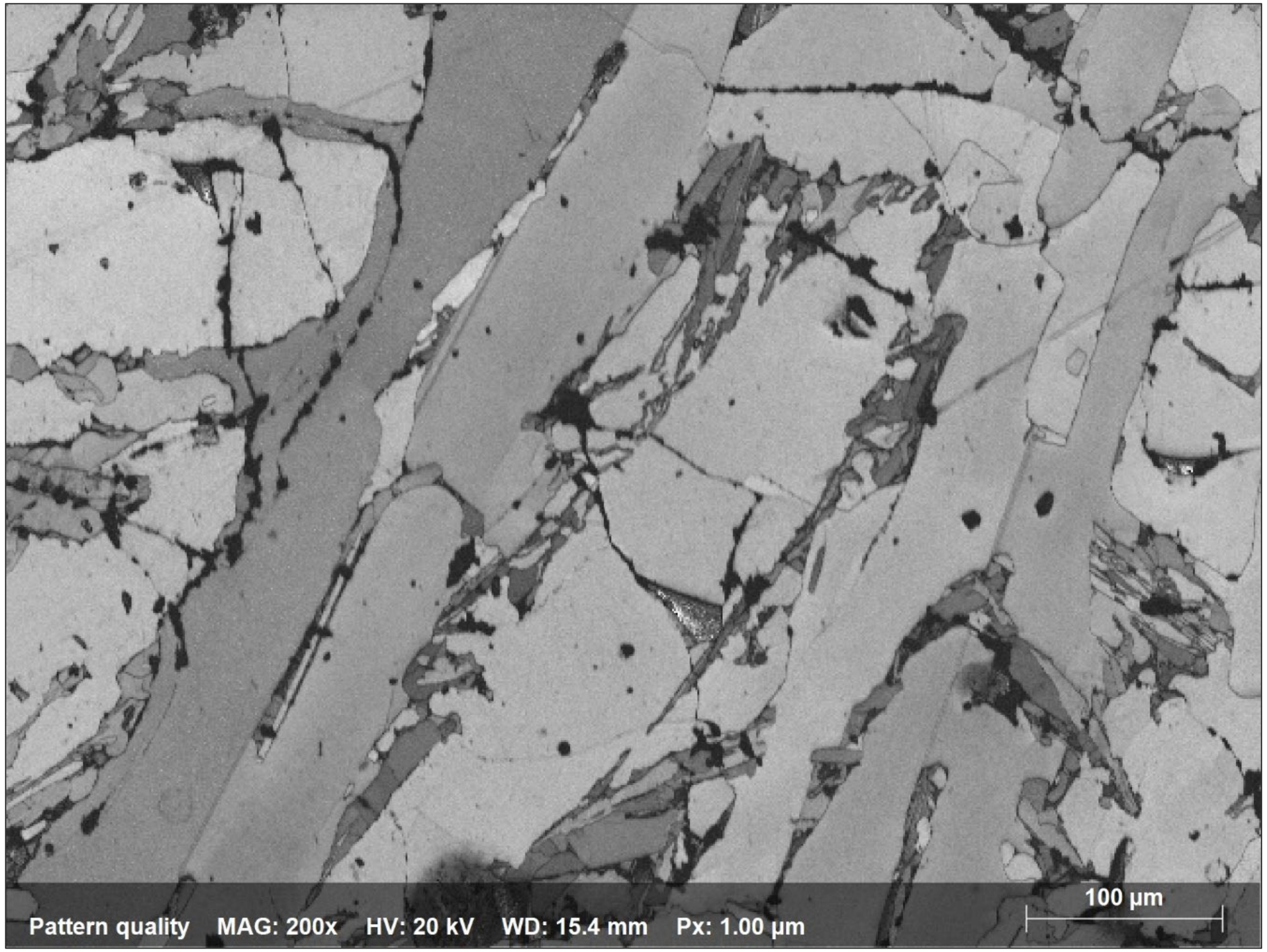
Important parameters:

- EHT: 20kV & Probe current: ~15nA
- Step size: 1µm
- Acquisition speed: 50 points/second
- Total acquisition time: 1:44 hours
- Map size: 309k pixels

Analysis type:

- Phase identification
- Phase distribution
- Phase ratio

Fe-Si composite material



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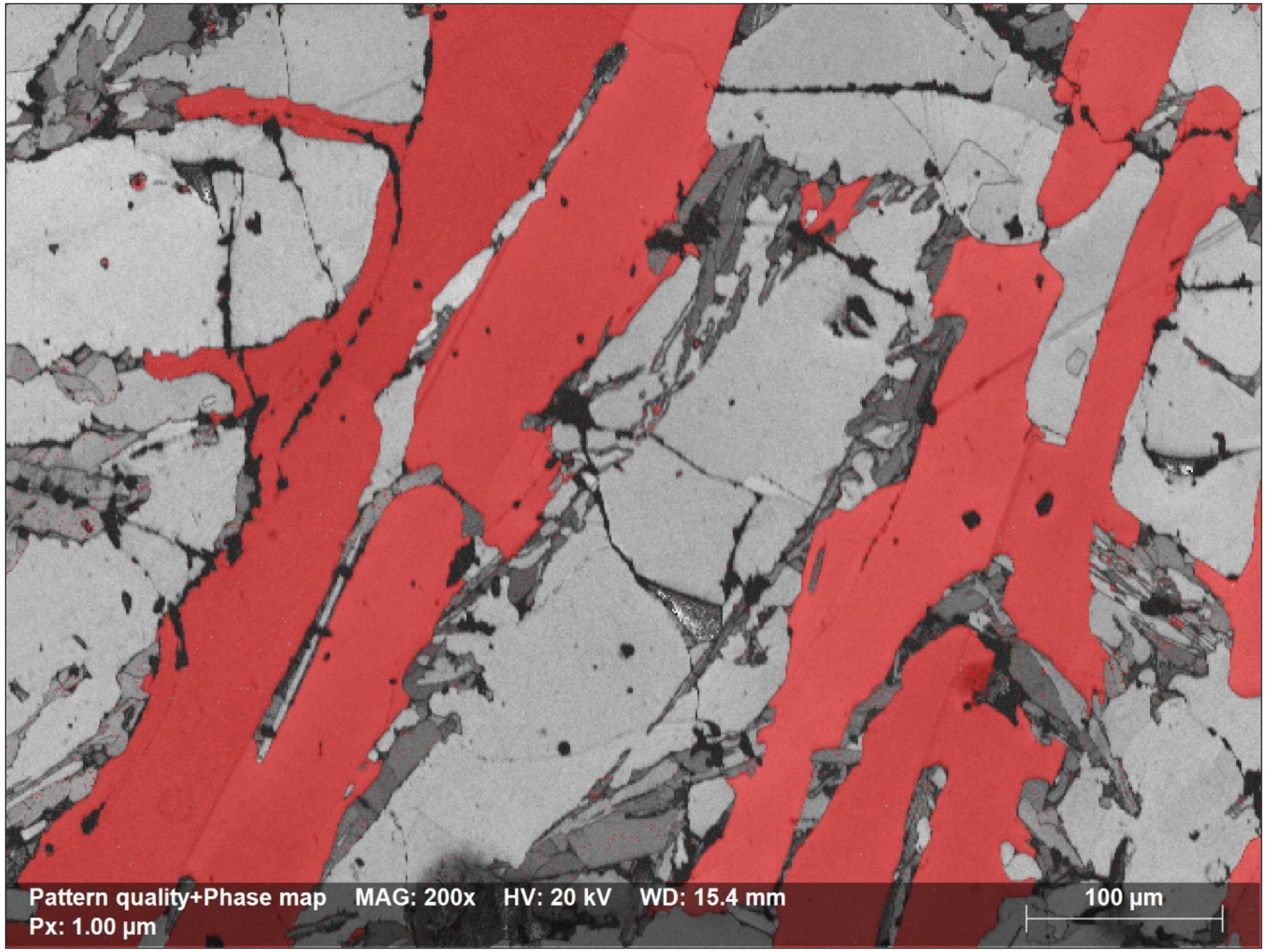
## Analysis of little-known multi-phase materials

Raw phase map: Fe-Si composite material




Important details:

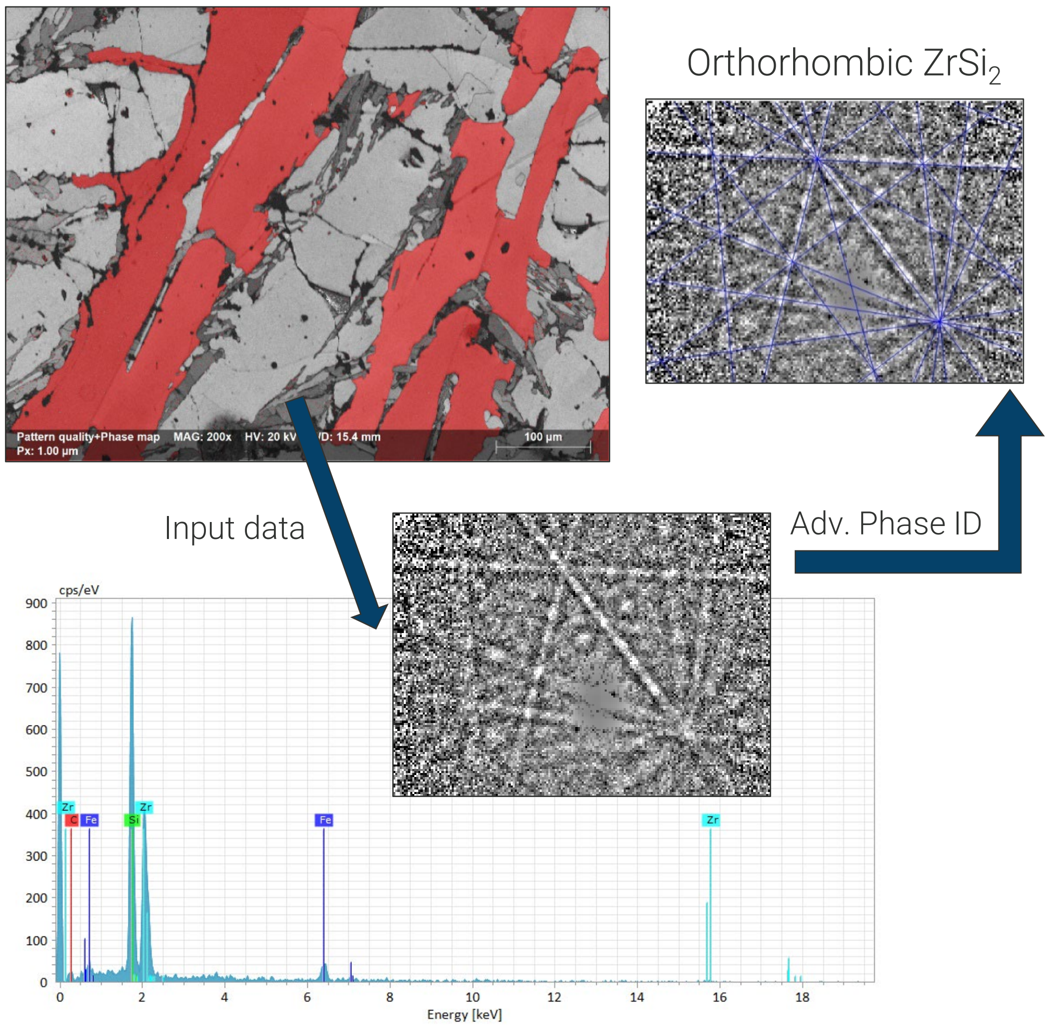
- Simultaneous acquisition of EBSPs and EDS spectra
- Only one phase was used during data acquisition resulting in reduced time to setup measurement
- All other crystallographic phases have been identified offline using Advanced Phase ID feature

# Simplicity Delivers Affordable Science

## Analysis of little-known multi-phase materials

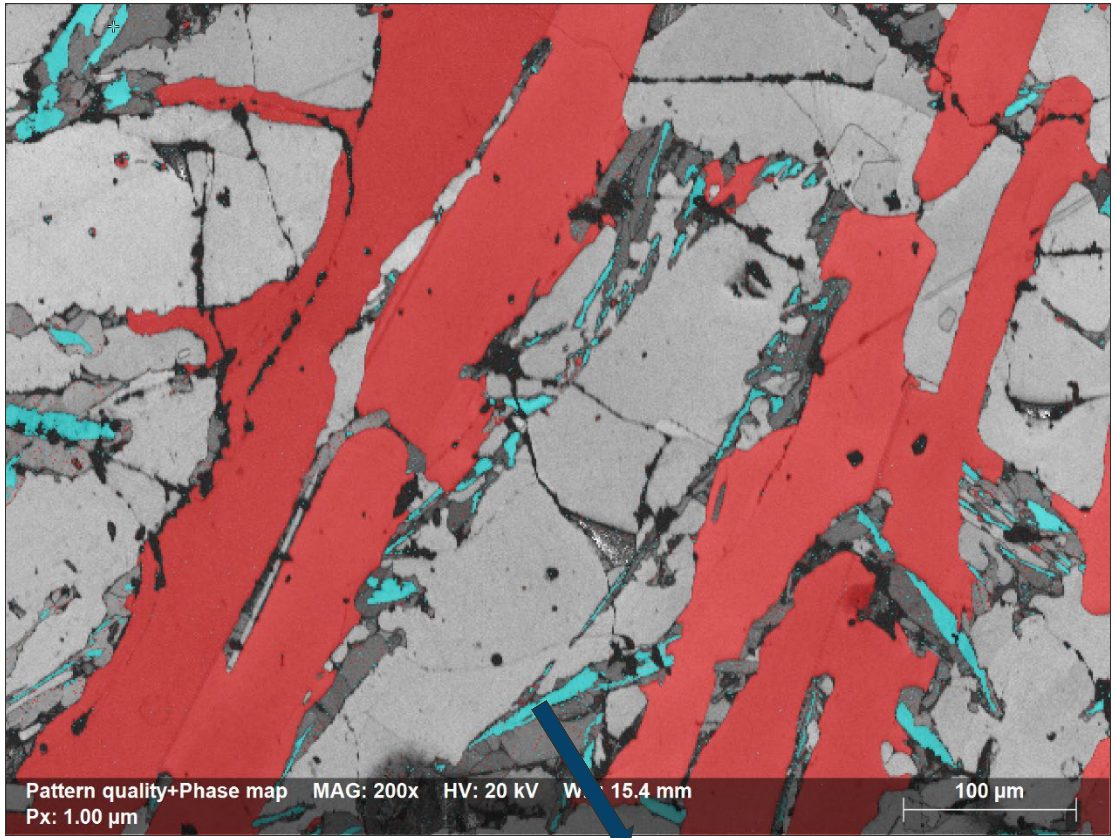
Advanced Phase ID:

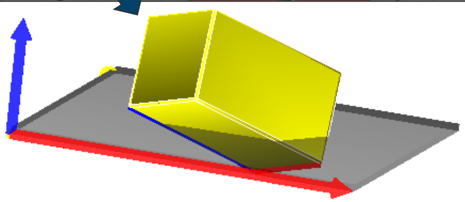
- Flexible semi-automatic search of phase candidates based on EDS spectrum (search through multiple databases with up to 900k phases)
- Ultra-fast indexing of all candidate phases (within seconds for a few thousand phase entries)
- Automatic classification based on best fit to experimental EBSP
- Multiple advanced features for improved identification, e.g. automatic search of pixels with similar chemistry, etc.



# Simplicity Delivers Affordable Science

## Analysis of little-known multi-phase materials



Ultra-fast map reanalysis:

- ESPRIT 2 is capable of reindexing speeds of up to 60,000 points/second (pps)
- Current map analyzed at 24,000pps with two phases
- Reindexing time: 13seconds
- Non-indexed areas of light gray levels are unknown phases
- Advanced Phase ID + Ultra-fast Reanalysis are applied iteratively until all present phases have been identified
- If necessary, the user can resave the map at any time and continue map completion at a latter time

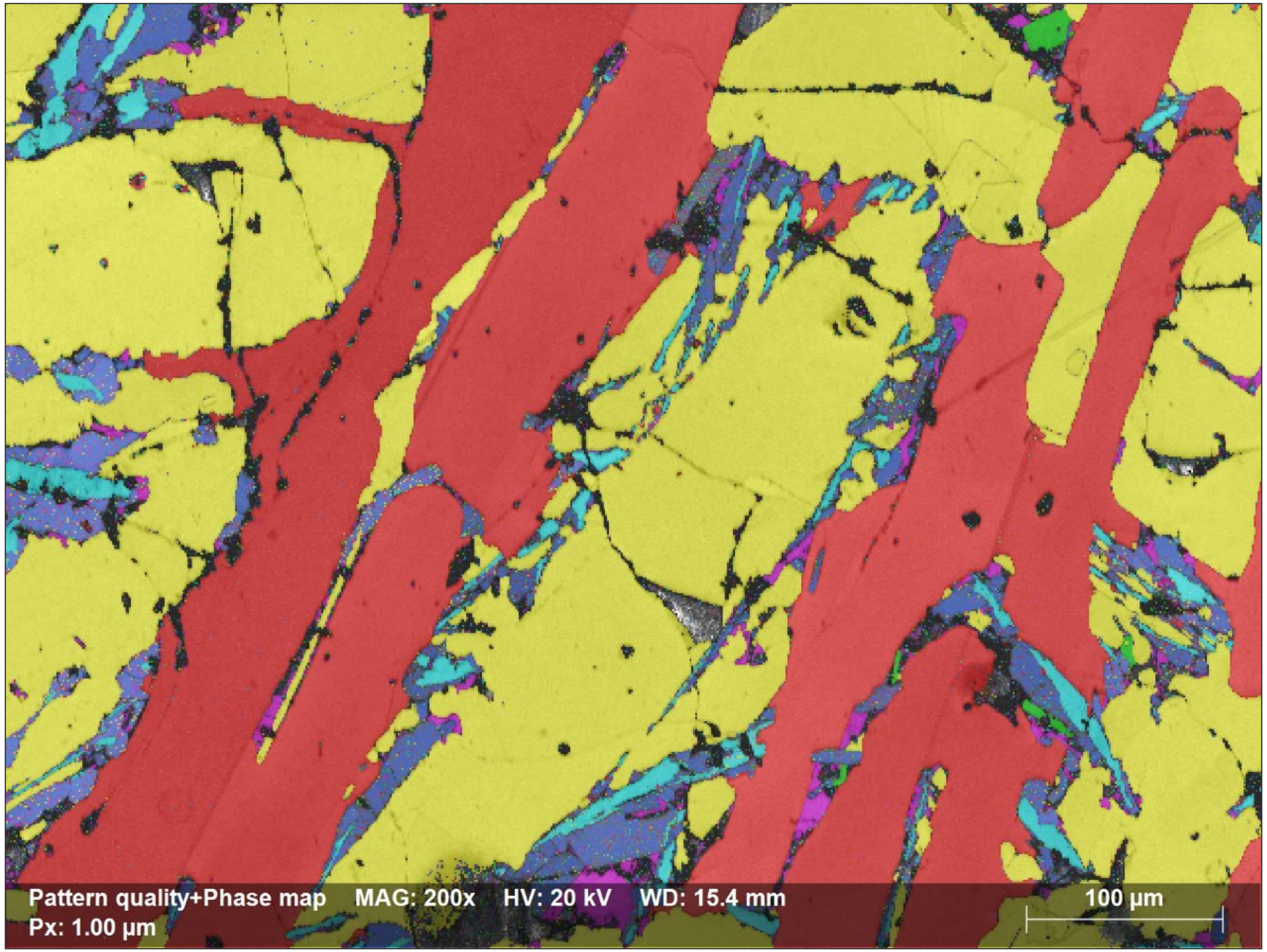
# Simplicity Delivers Affordable Science

## Analysis of little-known multi-phase materials

Ultrafast map reanalysis:

- Phase map after all phases have been identified:
  1. Silicon – cubic (IT227)
  2. Ferdisilicide – tetragonal (IT123)
  3. Titanium(II) iron silicide – orthorhombic (IT55)
  4. Zirconium silicide – orthorhombic (IT63)
  5. Calcium silicide – trigonal (IT166)
  6. Zirconium iron silicide – hexagonal (IT194)
- Map analyzed at ~6,500pps with six phases described above
- Reindexing time on a regular laptop: 48 seconds

NO DATA CLEANING!!



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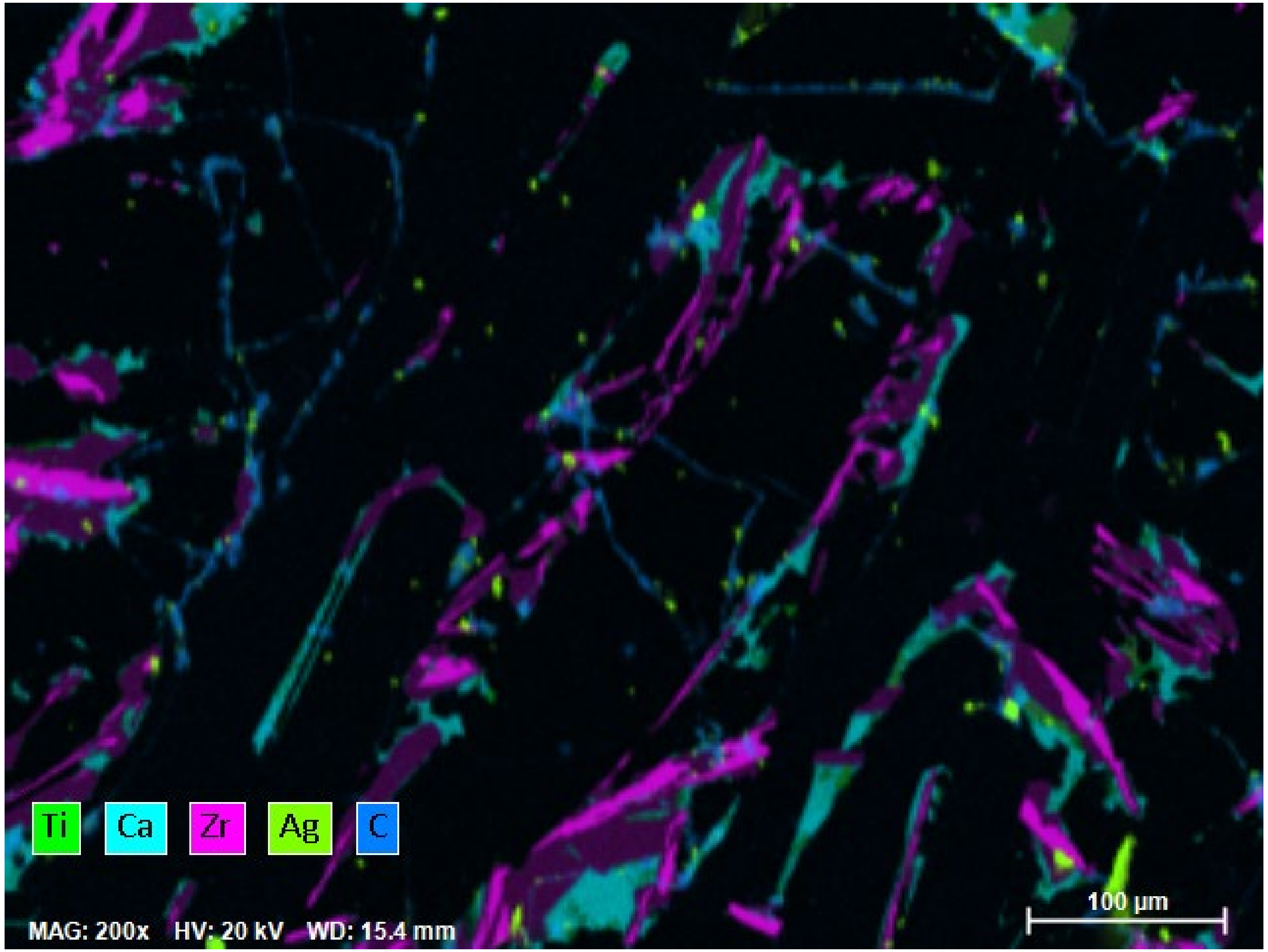
## Analysis of little-known multi-phase materials

Simultaneously acquired EDS HyperMap:

- Each pixel contains a spectrum acquired in 20ms
- Total of 309k spectra
- Multitude of EDS based analysis types enabled by the full analytical power of ESPRIT 2



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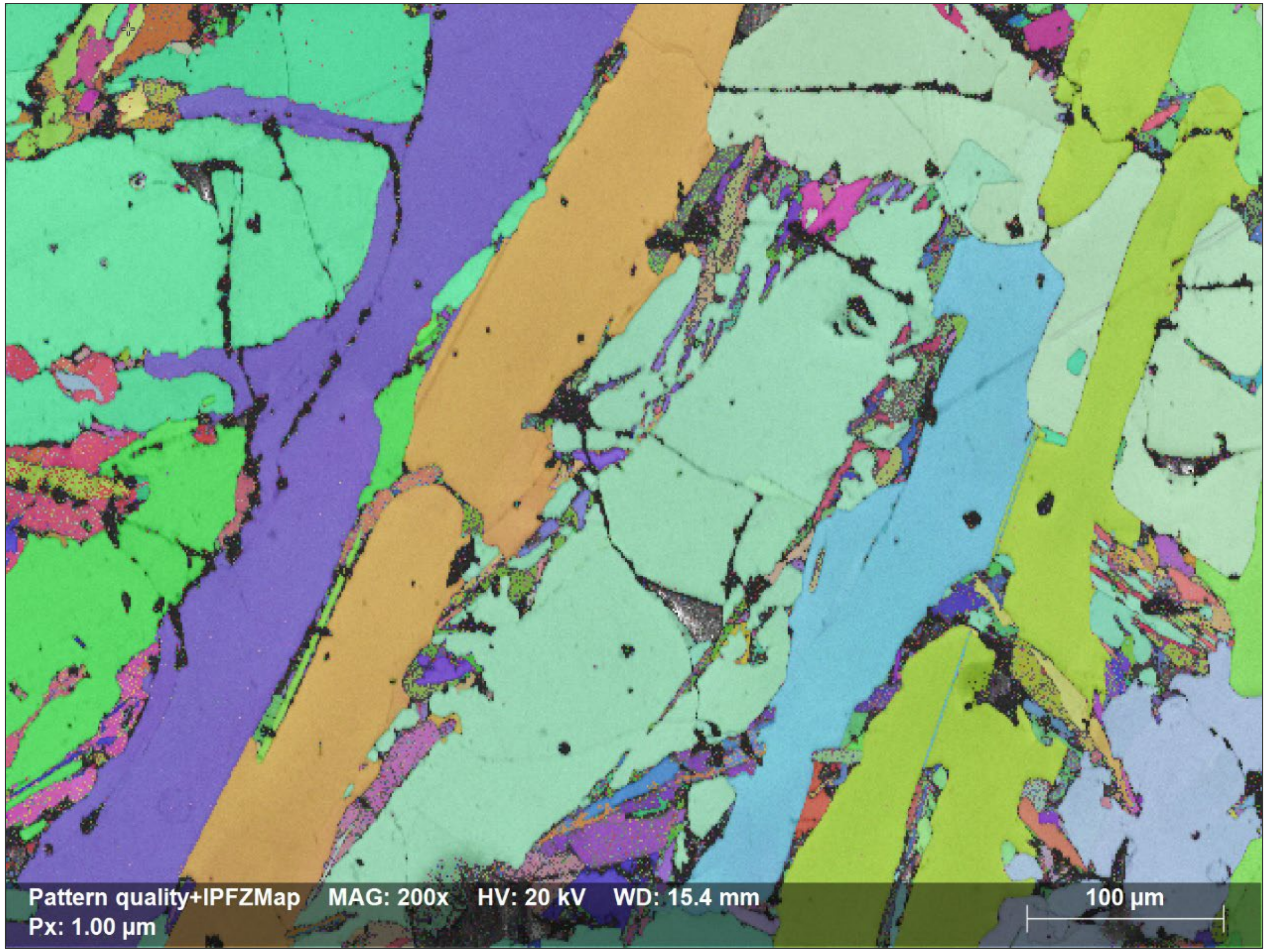
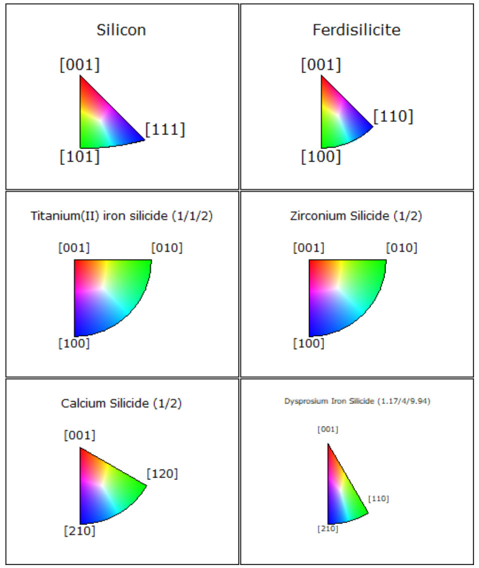
## Analysis of little-known multi-phase materials




Benefits:

- ESPRIT 2 offers the most powerful combination of EDS and EBSD techniques
- Minimized SEM use / maximized SEM throughput
- Map completion and data interpretation done offline without time constraints and on a separate computer



# Simplicity Delivers Affordable Science

## Application example

Phase Identification & Distribution Analysis of Oxide Ceramics: ZnO

# Simplicity Delivers Affordable Science

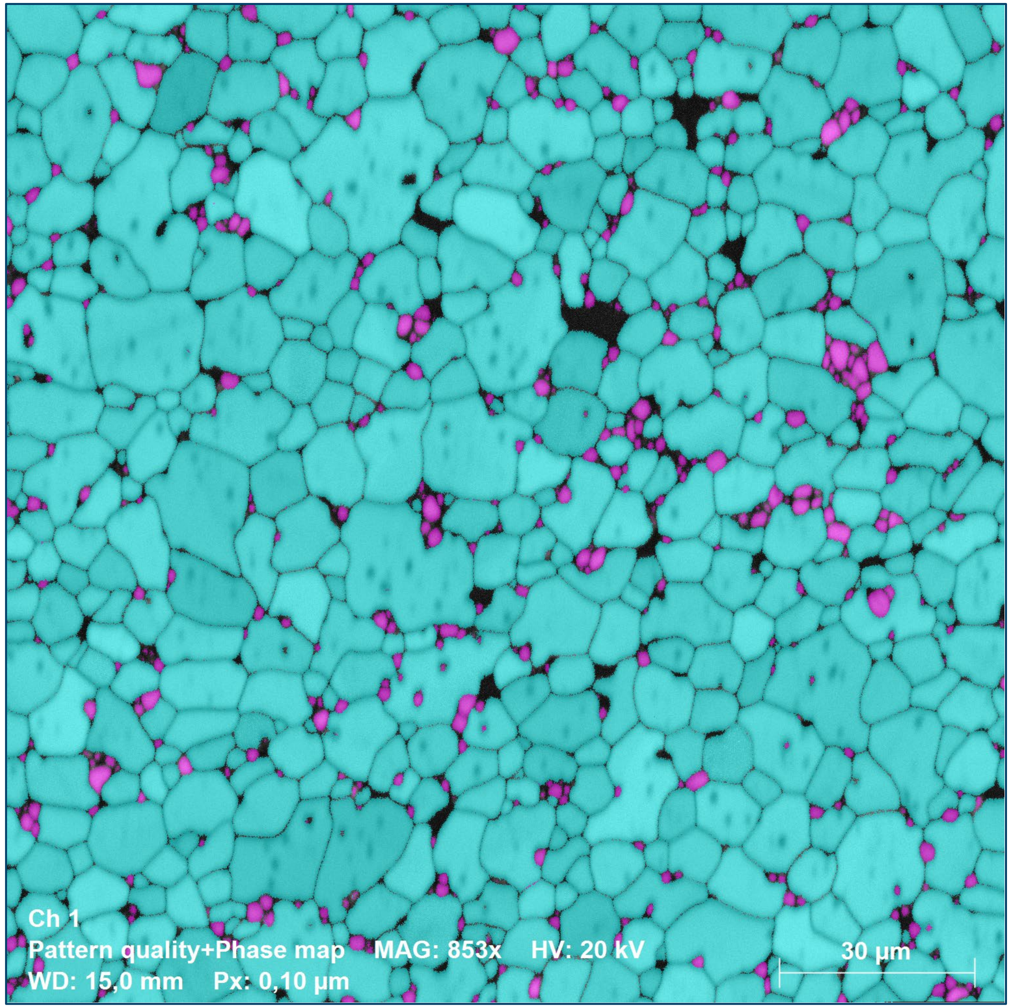
## Phase Identification & Distribution of Zn Oxides

Important parameters:

- EHT: 20kV
- Probe current: ~10 nA
- Step size: 100 nm
- Acquisition speed: 495 frames/second
- Total acquisition time: 80 min
- Map size: >2M points
- Pattern resolution: 180x135 pixels
- Phase fraction ZnO in blue (91%), Sb-ZnO in pink (4%).
- Porosity: 4,9 %

| | | | |
|---|---|---|---|
| 1 | ✓ | (cyan) | Zincite |
| 2 | ✓ | (magenta) | O Sb Zn |

Pattern quality + Phase map

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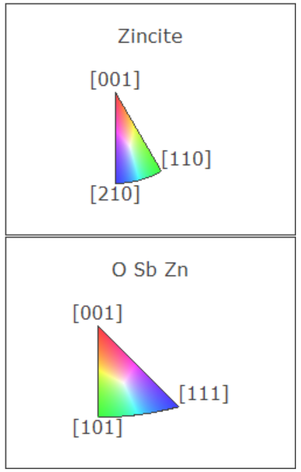
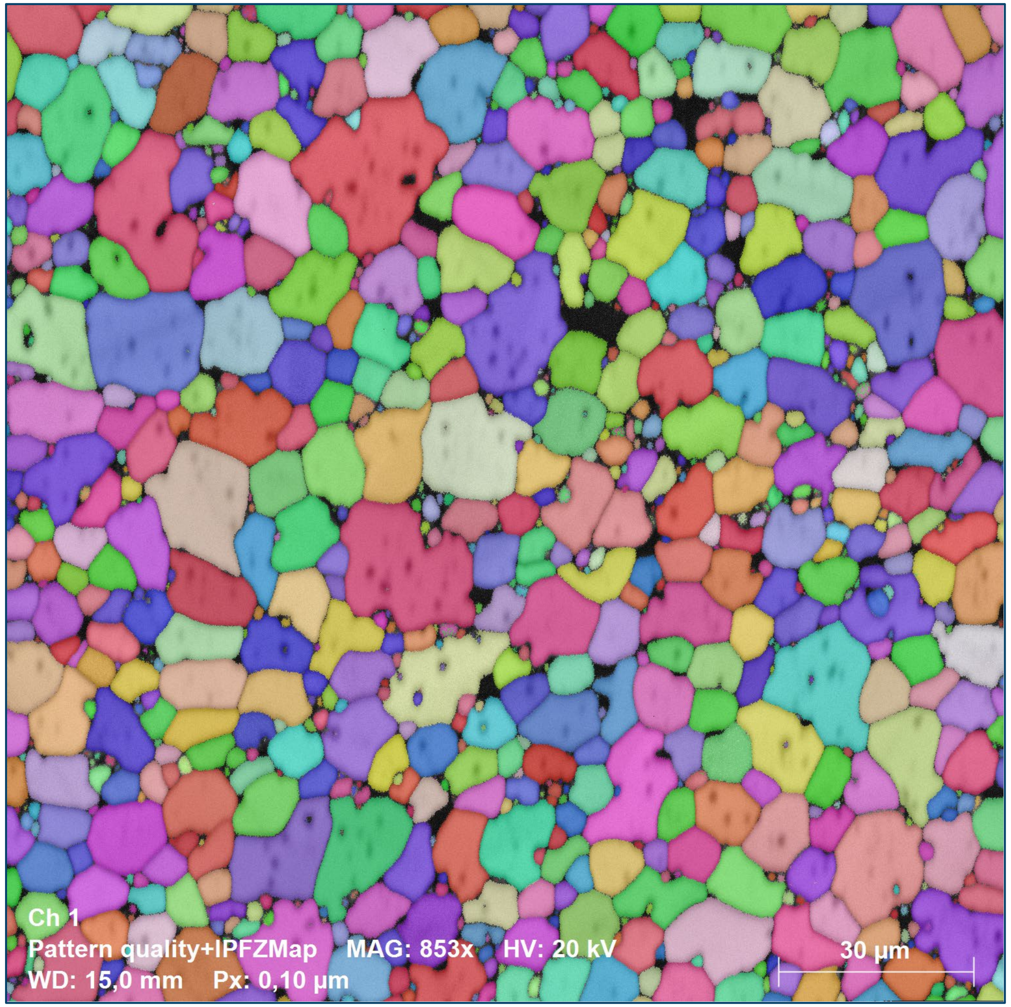
- Phase fraction ZnO in blue (91%), Sb-ZnO in pink (4%).
- Porosity: 4,9 %.




IPFx map

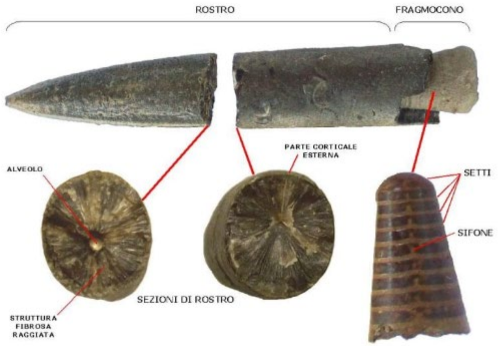



No data cleaning

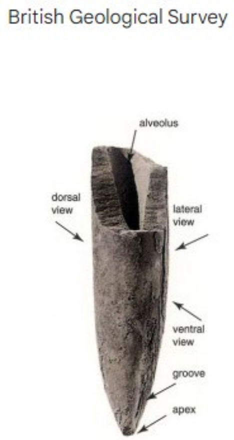
# Simplicity Delivers Affordable Science

## Application example

Microstructure and chemistry correlation for mineral characterization : Calcite (belemnite fossil)




Parts of the rostrum. This shows the phragmocone (FRAGMOCONO) and cross sections of the rostrum (SEZIONI DI ROSTRO). Image by Antonov via Wikimedia Commons, public domain



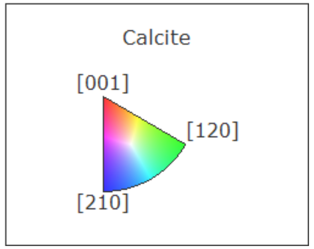
# Simplicity Delivers Affordable Science

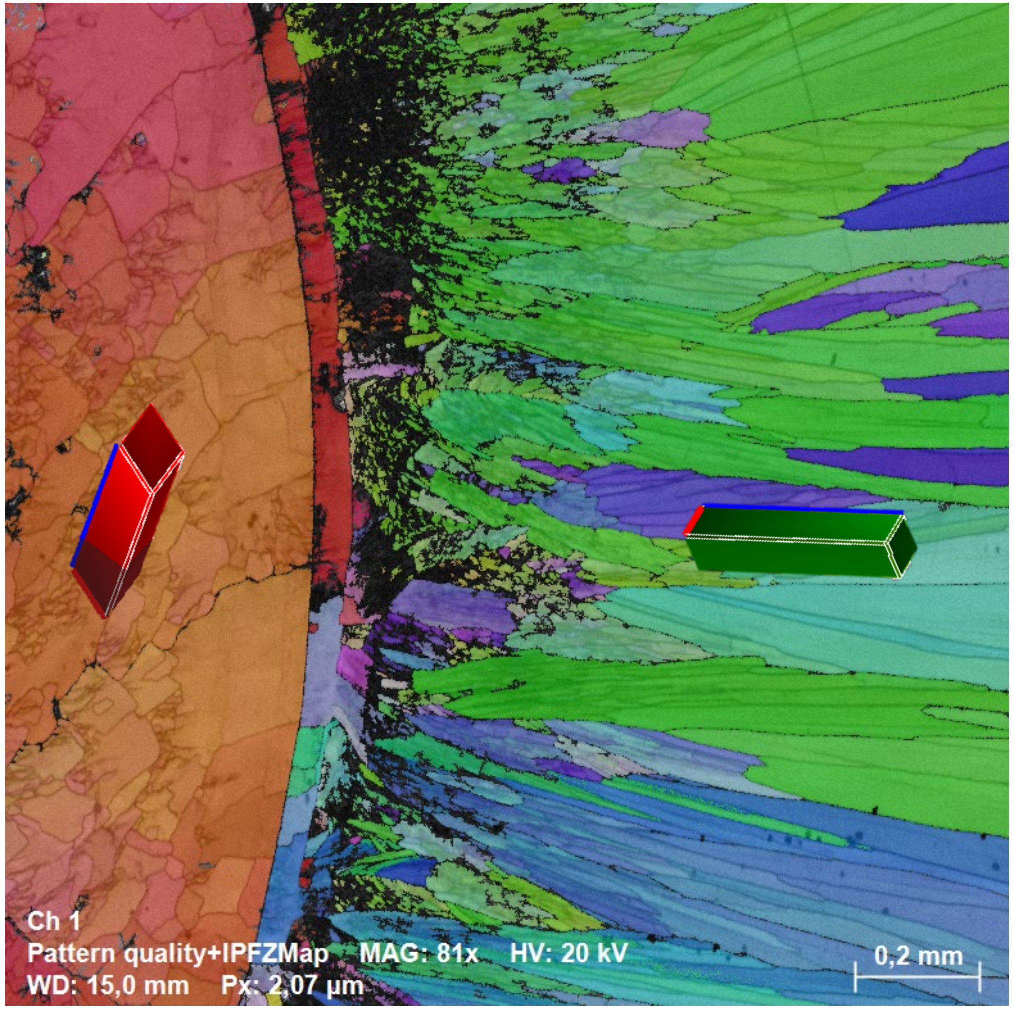
## Microstructure and chemistry correlation for mineral characterization : Calcite

Parameters:

- EHT: 20kV
- Probe current: 15 nA
- Step size: 2 µm
- Acquisition speed: 122 frames/second
- Total acquisition time: 80 min
- Map size: 613k pixels
- Pattern resolution: 180x135 pixels
- Zero sol.: 13 %
- NO DATA CLEANING!

- C-coating too thick (~5nm is ideal)



# Simplicity Delivers Affordable Science

## Microstructure and chemistry correlation for mineral characterization : Calcite

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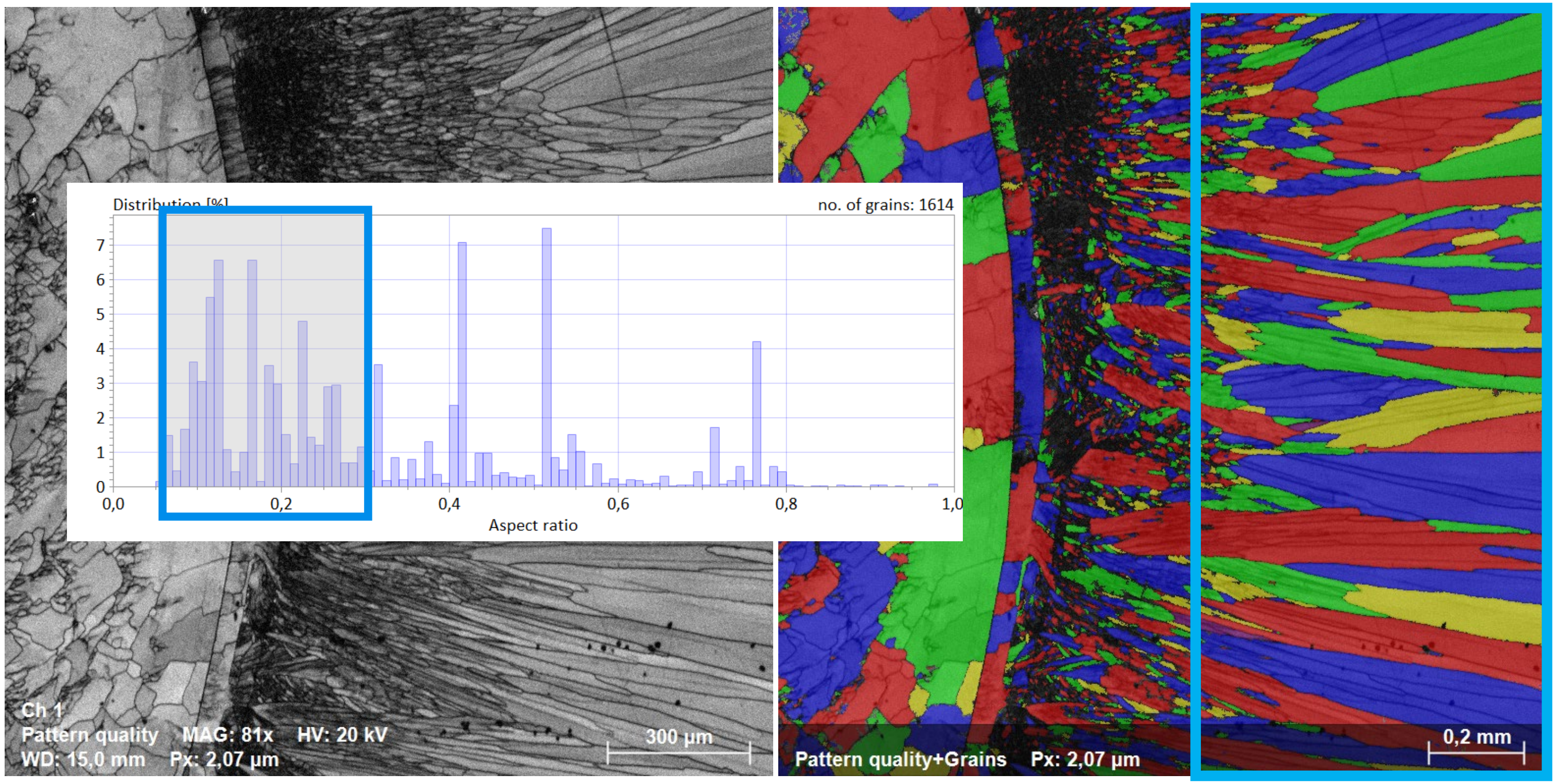
- Map size: 613k pixels
- Pattern resolution: 180x135 pixels
- Zero sol.: 13 %
- C-coating too thick

Pattern quality map

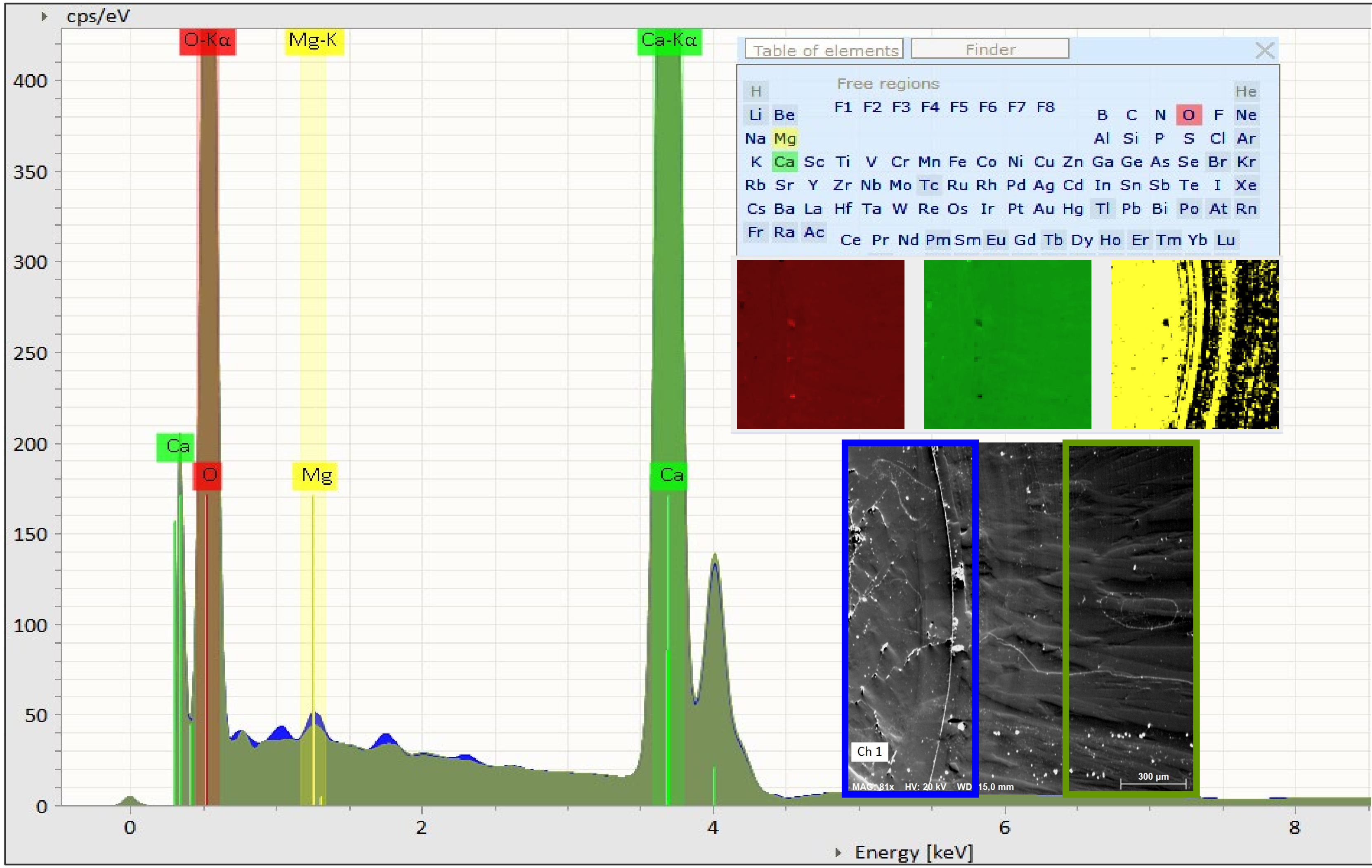
Pattern quality + Grain map



# Simplicity Delivers Affordable Science

## Microstructure and chemistry correlation for mineral characterization : Calcite

- Mg concentration can be correlated to the growth morphology of Calcite
- EDS spectrum acquired simultaneously with EBSD showing different Mg concentration between the inner and the outer shell/rostrum (with less than 5% at. in the richest region)



04

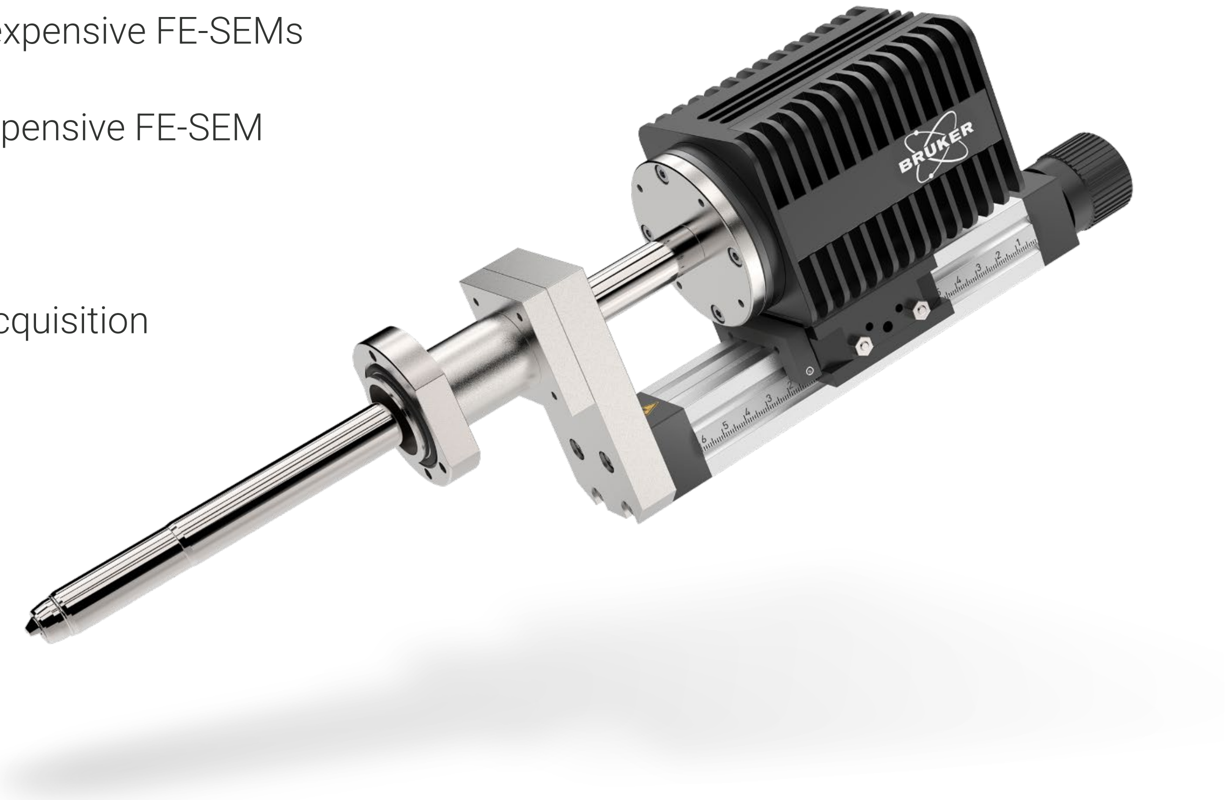
# Summary

# QUANTAX ED-XS

## Simplicity delivers affordable science

**Efficient use of lab' resources by:**

- Running routine analyses with QUANTAX ED-XS to relieve backlog on expensive FE-SEMs
- Checking sample preparation quality, before an EBSD session on an expensive FE-SEM
- Training of new users with much lower time/cost constraints
- Giving more time to entry level users for practicing EBSD & EDS data acquisition

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