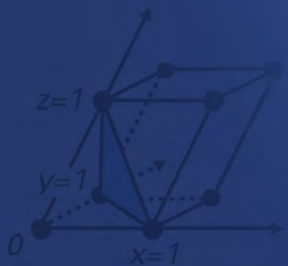
Advances in materials characterization using the new *e*-Flash EBSD Detectors

Bruker Nano Analytics, Berlin, Germany

Webinar, January 24th, 2017

Speakers:
Daniel Goran, PhD and Laurie Palasse, PhD



EBSD

e⁻Flash Generation II
Introduction

Old e⁻Flash EBSD detectors – not perfect! ☺

New e⁻Flash EBSD detectors – What changed?

- ***e*⁻Flash**$^{\mathbf{HD}}$ - High Definition detector for “HR-EBSD” applications
  - Upgraded cooling system (passive) - higher signal/noise ratio
  - Longer detector tube – optimum detector reach on all SEM chambers
- ***e*⁻Flash**$^{\mathbf{FS}}$ - Fast and Sensitive for all EBSD applications
  - Custom-made high quality optics system – maximized light sensitivity
  - Improved cooling system – higher signal/noise ratio
  - Longer detector tube – optimum detector reach on all SEM chambers

e^{-}FlashHD

Unmatched pattern resolution and quality

e^{-}FlashHD – improved pattern quality:

- Better cooling - dark current lowered by at least a factor of x8
- Significantly lower noise levels in the patterns

e-Flash^{HD}
High Resolution EBSD

e^{-}Flash^{HD}

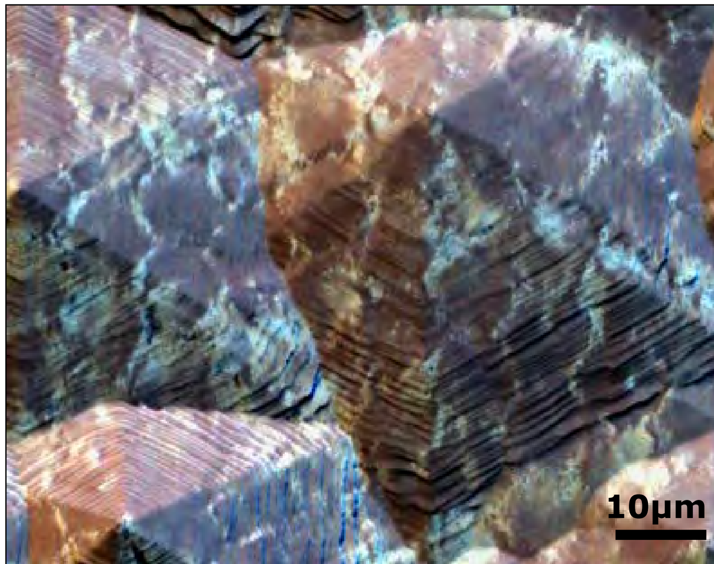
Unmatched pattern resolution and quality

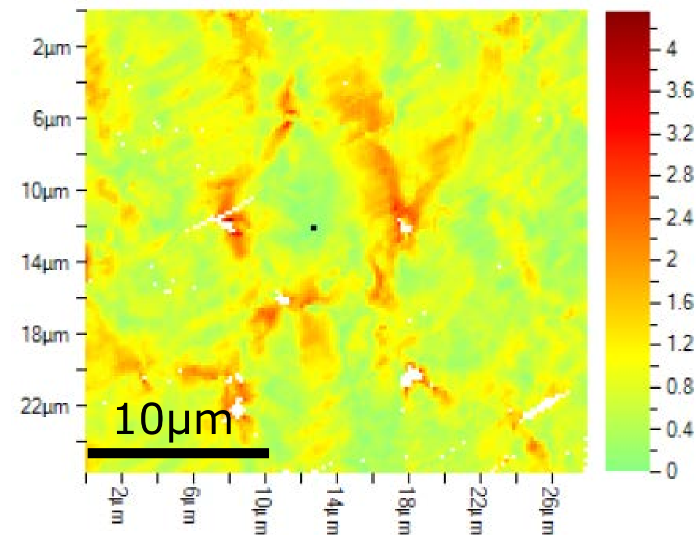
High **D**efinition EBSD detector

- 1600x1200 pixels CCD native resolution
- State of the art optics system
- Screen positioning precision better than 10µm
- Best fitted for pattern correlation based analysis:
    - residual strain analysis or HR-EBSD (CrossCourt4)
    - high accuracy phase ID (ESPRIT DynamicS)
    - advanced crystallographic analysis, etc.

QUANTAX EBSD
ARGUS™ FSE/BSE - sensitivity

- AlGaN single crystalline layer deposited on patterned Sapphire substrate for isotropic strain relief
- ARGUS™ FSE images show presence of residual strain confirmed by analysis with CrossCourt 4 software





e⁻Flash^{FS}
High speed EBSD

e⁻Flash^{FS} Generation II – greatly improved pattern quality:

- At least 3x more sensitive
- At least 8x lower dark current on the CCD chip

e⁻FlashFS

Fast and Sensitive – no compromise!

High speed EBSD:

- No longer needs high probe currents
- Can be applied to most materials

e-Flash^{FS}
outline

- High speed EBSD
  - low density materials
  - deformed materials
- Low kV EBSD
- Combined EBSD/EDS analysis
- 3D EBSD
- High speed TKD

e-Flash^FS
High speed EBSD

IPFZMap MAG: 525x HV: 20 kV WD: 16,4 mm Px: 0,49 µm
80 µm

Poly Si (solar cell)

Binning: 8x8

Pattern resolution:

80x60

Measurement speed:

650fps

Map size:

800x533pixels

Measurement time:

10:56min

Hit rate (indexed points):

98.9%

e-Flash^{FS}

High speed EBSD on deformed material

Pattern quality+IPFYMap MAG: 800x HV: 20 kV WD: 22,3 mm
Px: 0,32 µm
50 µm

3D printed SS

Binning:

8x8

Pattern resolution:

80x60

Speed:

938fps

Map size:

800x533pixels

Measurement time:

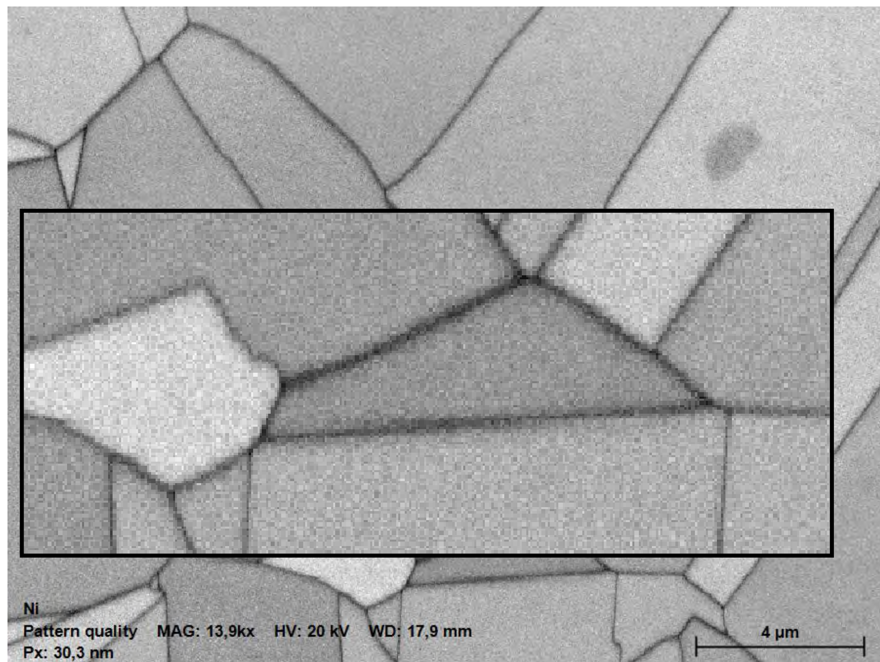
7:34min

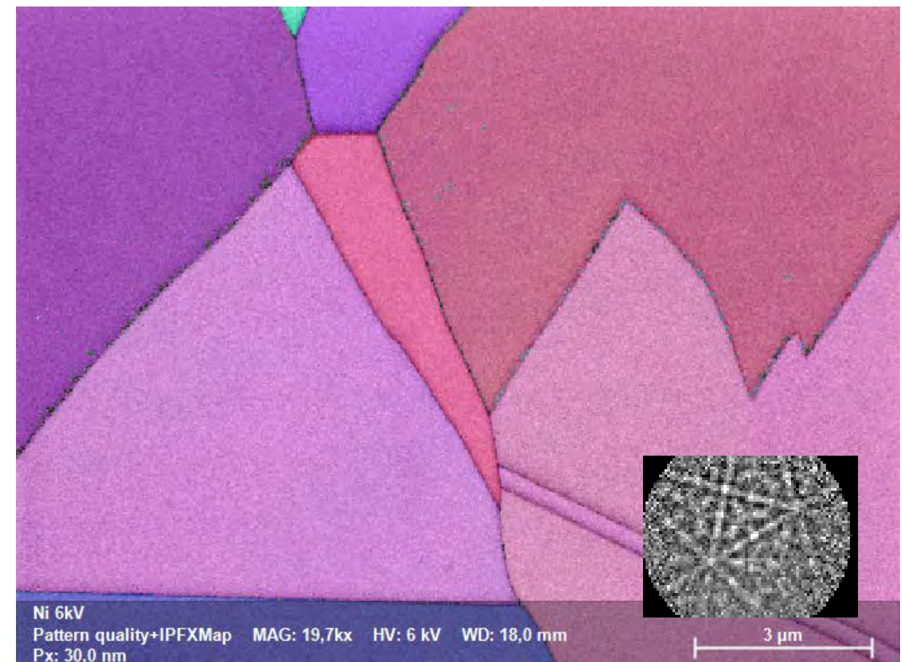
Hit rate:

98.6%

e-Flash^{FS}
low kV EBSD on Nickel alloy

kV	20kV
EBSP resolution (pixels)	80*60
Step size	30nm
Measurement speed	930 fps (1ms)
Raw hit rate	99,9%





e-Flash^{FS}

High speed simultaneous EBSD/EDS

Fe-Si alloy
Pattern quality+Phase map MAG: 404x HV: 20 kV WD: 16,5 mm
Px: 0,50 µm
100 µm

Fe-Si alloy

Fast measurement setup

Not all phases known

XFlash 6|60

Pattern resolution:

160x120

Measurement speed:

628 fps

Map size:

1 586 314 points

Measurement time:

42:05min

e-Flash^{FS}

High speed simultaneous EBSD/EDS

Fe-Si alloy
Pattern quality+Phase map MAG: 404x HV: 20
Px: 0,50 µm

Fe-Si alloy

Offline Phase ID

XFlash 6|60

Pattern resolution:

160x120

Measurement speed:

628 fps

Reanalysis speed:

3100 fps

Hit rate:

96,3%

9 Phases:

Si 227	ZrS_{i2} 63
Ba_8Si_{48} 223	$TiFeSi_2$ 55
$FeSi_2$ 123	$CaSi_2$ 166
$CaSi_2$ 141	$CrTiSi_2$ 180
	ZrFeSi 194

e-Flash^{FS}

combined EDS/EBSD analysis

Fe-Si alloy
Pattern quality+Phase map MAG: 687x HV: 20 kV WD: 16,5 mm
Px: 0,50 µm
90 µm

Fe-Si alloy

ultra high speed EBSD/EDS

Xflash 6|60

Pattern resolution:

80x60

Measurement speed:

930fps

Map size:

856 x 642 pixels

Measurement time:

9:49min

Hit rate:

94,2%

e-Flash^{FS}

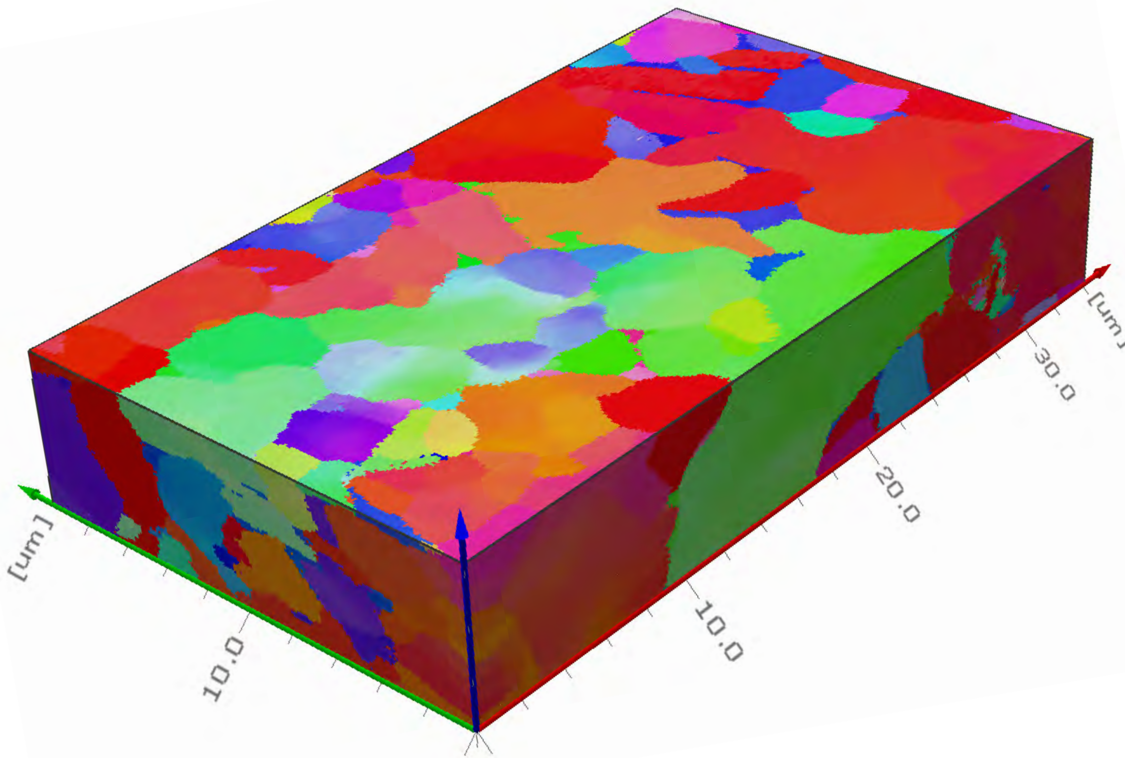
Full resolution EBSPs (Duplex steel and $(Mg,Fe)_2SiO_4$)

e-Flash^{FS}

high speed 3D EBSD

BRUKER

3D EBSD on deformed Titanium alloy
With FEI HELIOS and e-Flash ^{FS}



Ti beta & Ti alpha

62 slices

EBSD measurement speed :

164 fps (7 min per slice)

FIBing time :

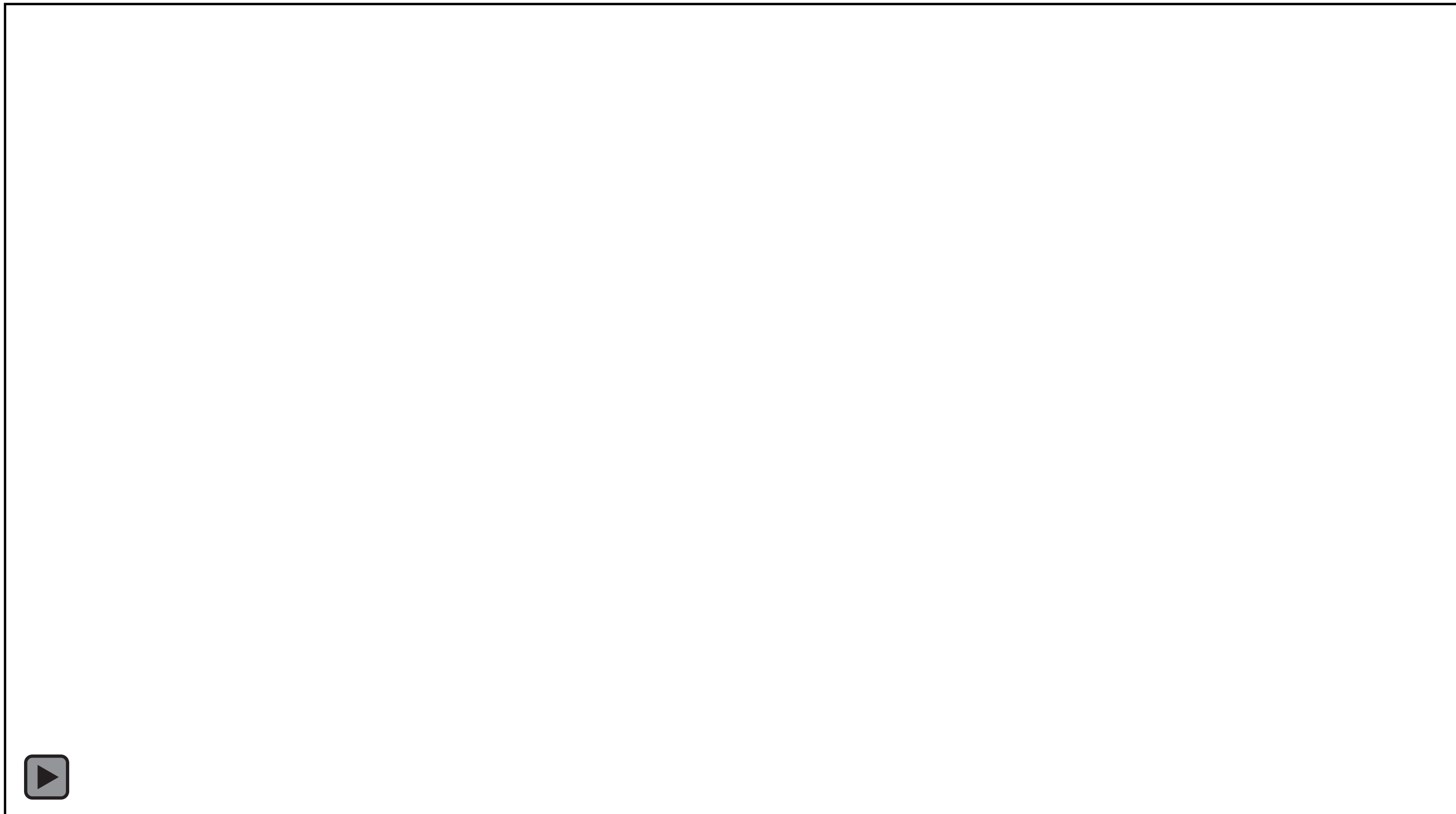
1min per slice

e-Flash^{FS}

3D EBSD with ESPRIT QUBE

BRUKER

Local Average Disorientation (0 – 5°)



e^{-}Flash^{FS}
High speed TKD mapping

e^{-}Flash^{FS} Generation II – greatly improved pattern quality:

- At least 3x more sensitive
- At least 8x lower dark current on the CCD chip

TKD in SEM using on-axis detector
Spatial resolution & high speed TKD mapping

Pt on Si
Pattern quality MAG: 72,2kx HV: 30 kV WD: 4,8 mm Px: 4,0 nm
1 µm

Pt thin film on Si:

Indexed points: 93.1%

Binning: 4x4

Pattern resolution:
160x120

Speed: **328fps**

Map size:
1017x763pixels

Measurement time:
39:27min

Step size: 4nm

TKD in SEM using on-axis detector

Spatial resolution & high speed TKD mapping

Pt on Si
Pattern quality+IPFZMap MAG: 72,2kx HV: 30 kV WD: 4,8 mm
Px: 4,0 nm
1 µm

Pt thin film on Si:

Indexed points: 93.1%

Binning: 4x4

Pattern resolution: 160x120

Speed: **328fps**

Map size: 1017x763pixels

Measurement time: 39:27min

Step size: 4nm

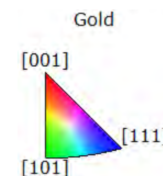
TKD in SEM using on-axis detector

Spatial resolution & high speed TKD mapping

20nm Au film on 5nm Si_3N_4 membrane

20nm Au on 5nm Si3N4 membrane
Pattern quality+IPFZMap MAG: 184kx HV: 30 kV WD: 5,1 mm
Px: 2,8 nm
300 nm

- Step size: 2.8nm
- Acq. speed: 200 fps
- Total acquisition time: 23:05min
- Map size: 270,000 pixels
- Pattern resolution: 160x120 pixels
- Zero sol.: 15.9%



Thanks to Dr. Alice Bastos da Silva Fanta from DTU in Denmark for generously providing the sample

TKD in SEM using on-axis detector

Spatial resolution & high speed TKD mapping

20nm Au film on 5nm Si_3N_4 membrane

20nm Au on 5nm Si3N4 membrane
Pattern quality+IPFZMap MAG: 184kx HV: 30 kV WD: 5,1 mm
Px: 2,8 nm

angle [°]
60
40
20
0
0,00 0,02 0,04 0,06 0,08 0,10
Distance [µm]

TKD in SEM using on-axis detector

Large area & high speed TKD mapping

Partially recrystallized martensitic stainless steel

4 µm
Pattern quality MAG: 14,8kx HV: 30 kV WD: 4,7 mm Px: 10,0 nm

Thanks to Prof. Zeng Yi from Shanghai Institute of Ceramics in China for generously providing the sample

- "Large" area TKD mapping ($20x15 \mu m^2$)
- Map size: 2000x1500 points
- Step size: 10nm
- Acq. speed: 623 fps
- Total acquisition time: 1:19:30h
- Pattern resolution: 160x120 pixels
- Zero sol.: 14.4%

TKD in SEM using on-axis detector

Large area & high speed TKD mapping

Partially recrystallized martensitic stainless steel

Pattern quality+IPFXMap MAG: 14,8kx HV: 30 kV WD: 4,7 mm
Px: 10,0 nm
4 µm

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- Map size: 2000x1500 points
- Step size: 10nm
- Acq. speed: 623 fps
- Total acquisition time: 1:19:30h
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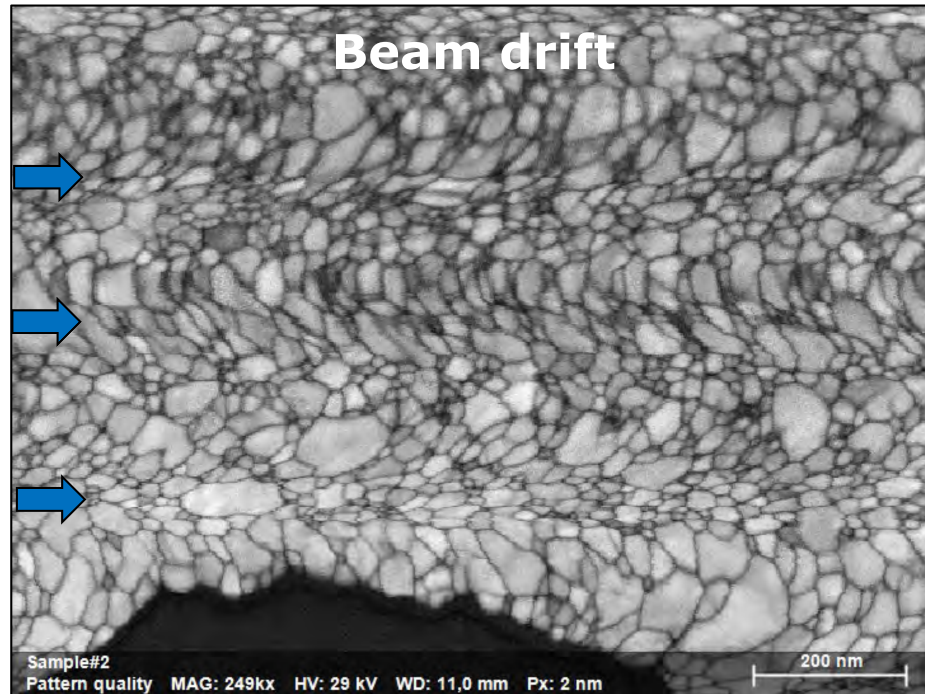
TKD in SEM using on-axis detector

High speed TKD mapping - benefits

40nm Au film on Si (dimpled)



20nm Au film on 5nm Si_3N_4 membrane

- 2.6nA and 10ms/point vs. 1.75nA and 3ms/point
- 43:05 min vs. 14:04min

Lower probe currents and faster data acquisition – less prone to beam instability

TKD in SEM using on-axis detector

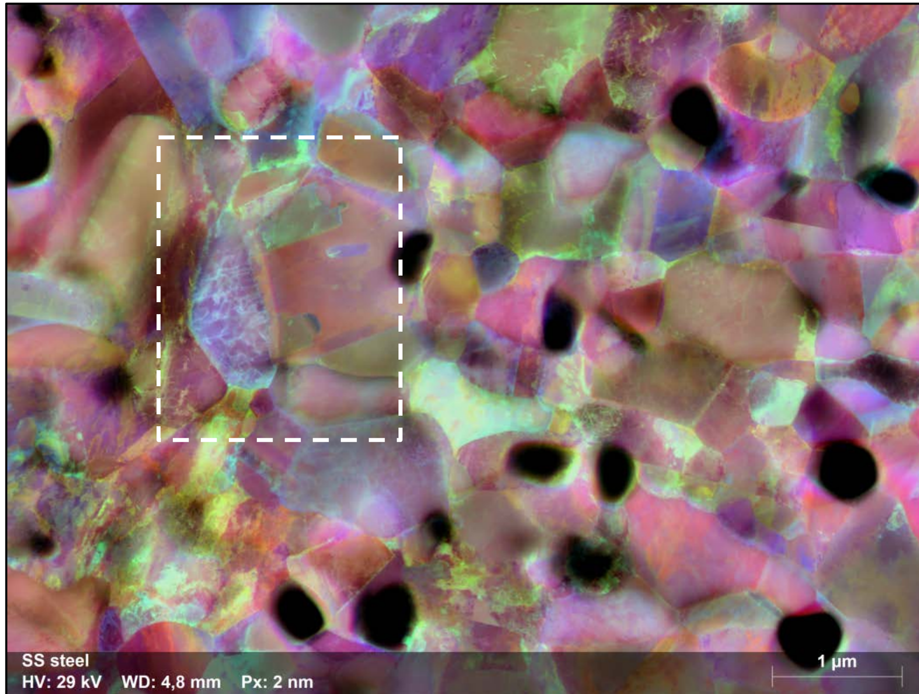
Integrated ARGUS™ imaging system

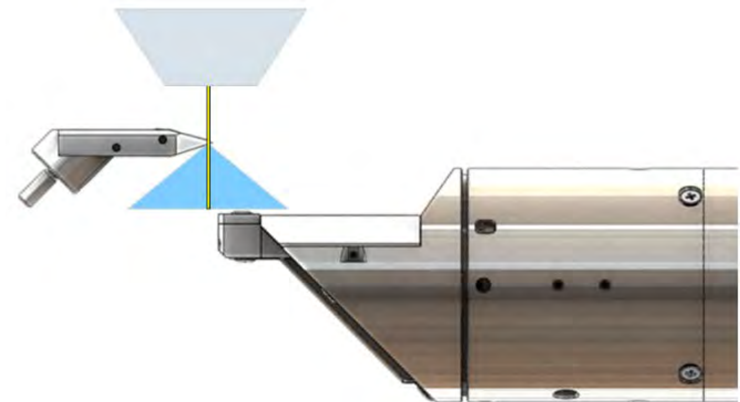
Feature:

- Built-in ARGUS™ imaging system (3x Si diodes)

Benefits:

- Color coded Dark Field imaging
- Direct detection – fast & sensitive (up to 125,000 p/s)





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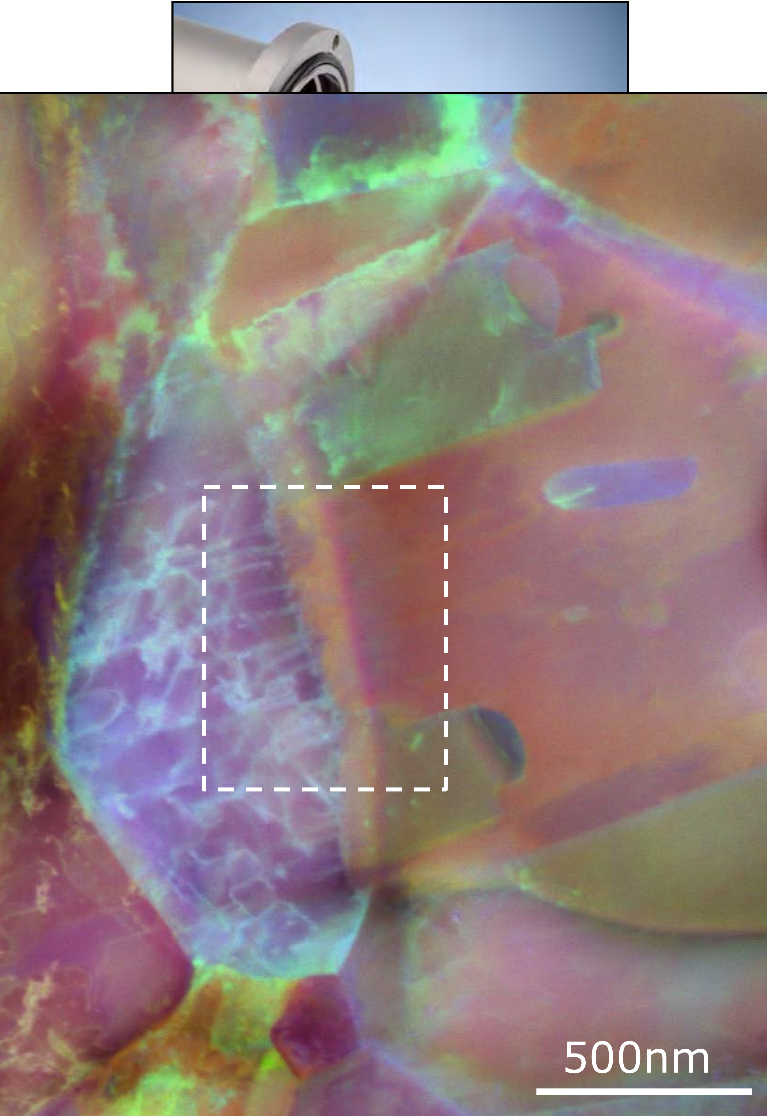
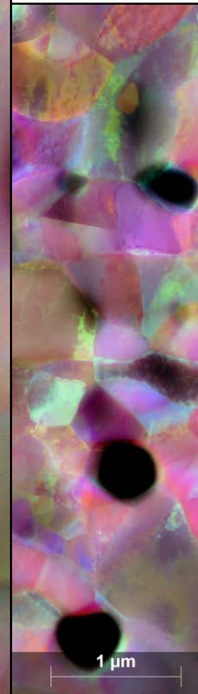
TKD in SEM using on-axis detector

Integrated ARGUS™ imaging system





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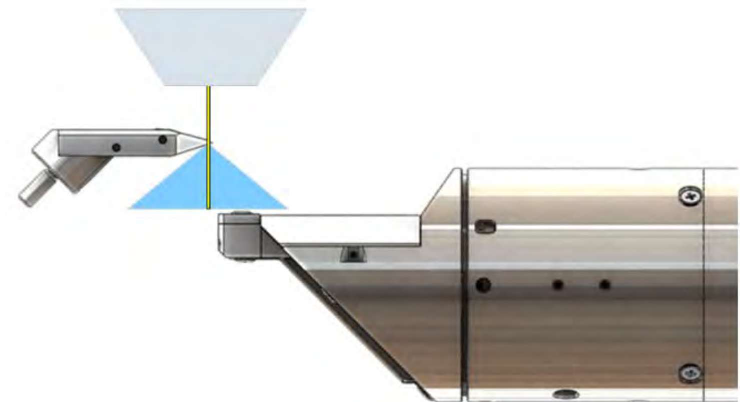

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TKD in SEM using on-axis detector

Color coded DF imaging

2 µm

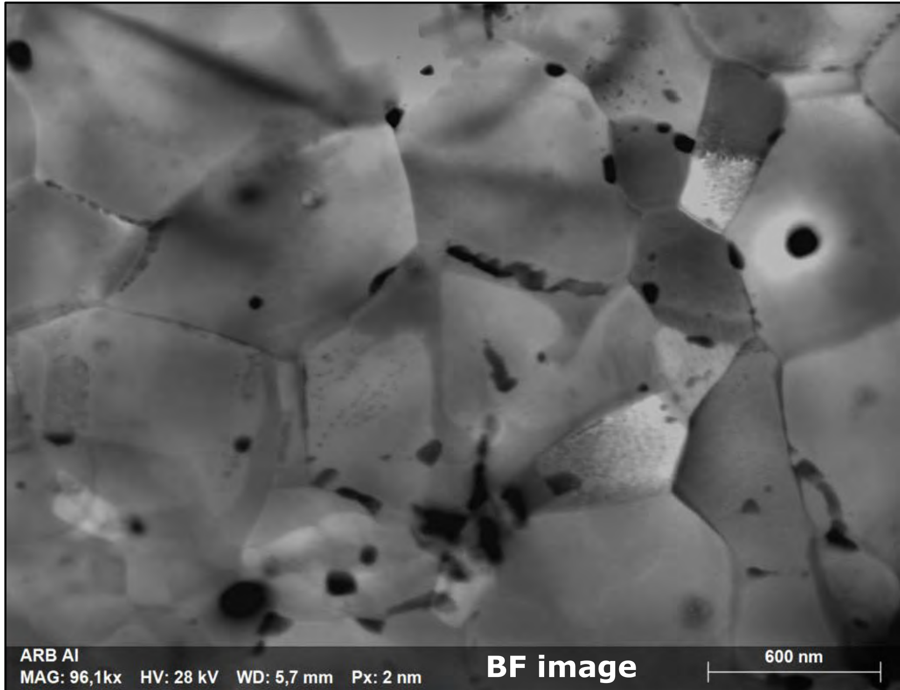
ARGUS MAG: 11322 x HV: 20,0 kV WD: 6,3 mm Px: 6 nm

TKD in SEM using on-axis detector

Color coded DF imaging

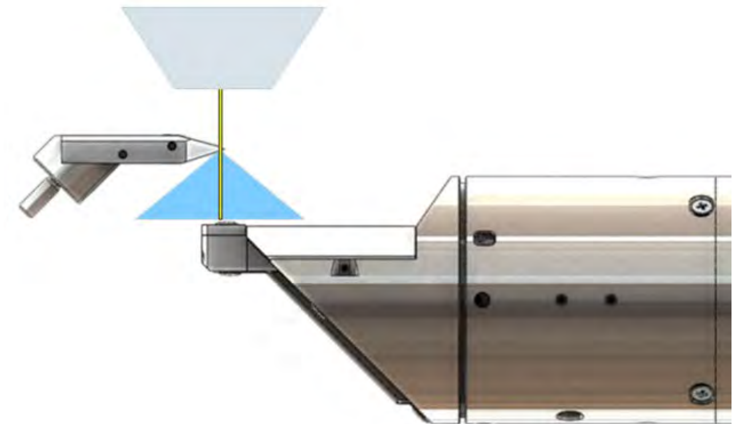
TKD in SEM using on-axis detector

Integrated ARGUS™ imaging system



Benefits:

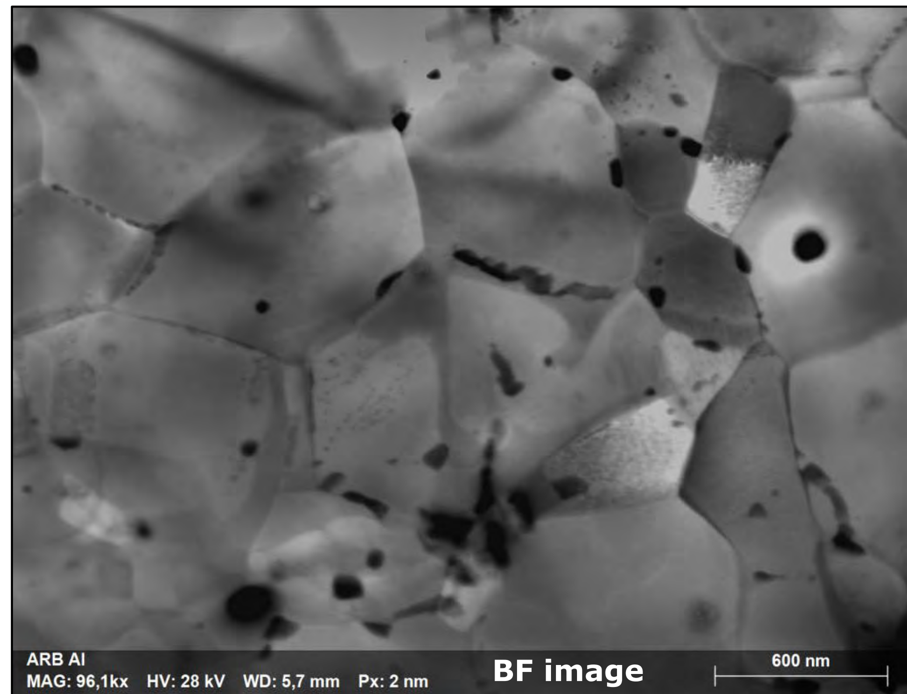
- Built-in BF/DF imaging system (3x Si diodes)
- Bright field imaging (middle diode)
- Dark field imaging (side diodes)



Thanks to Dr. Patrick Woo from Hitachi High Technologies Canada for generously providing the sample.

TKD in SEM using on-axis detector

Integrated ARGUS™ imaging system



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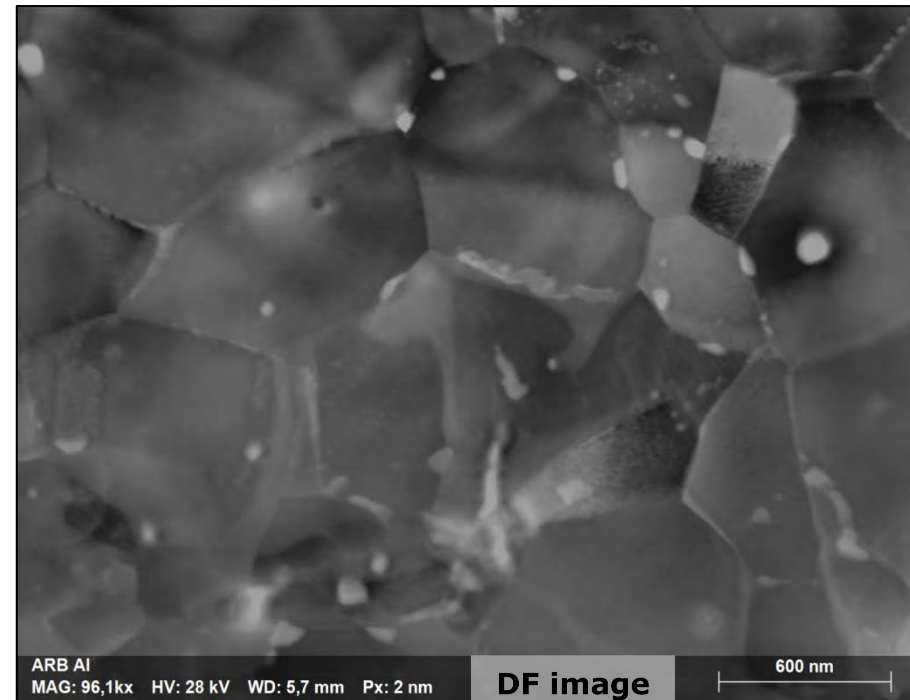

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e⁻Flash Generation II

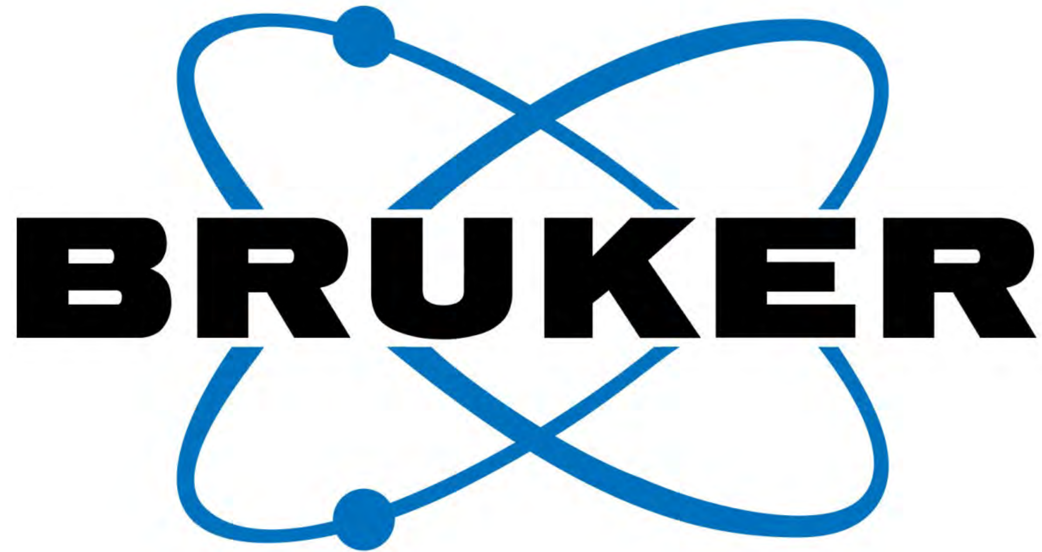
Summary

e⁻**Flash**[FS]

- High speed and high sensitivity detector
- Best solution for all Hough based EBSD applications

e⁻**Flash**[HD]

- High pixel resolution and high quality patterns
- Best solution for applications requiring high resolution and high quality patterns, e.g. residual strain analysis a.k.a. HR-EBSD



Innovation with Integrity

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