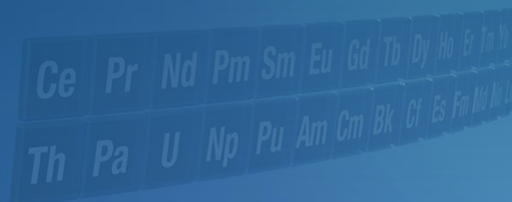
BRUKER NANO ANALYTICS

XFlash® 7 EDS Series for Electron Microscopy 高空間分解能EDSでの最速な定量および定性分析



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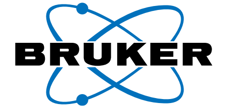

Presenters

Hiroki Baba
EMA Sales Account Manager, Nano Analytics Division

Rie Nakajima
EMA Application Engineer, Nano Analytics Division

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Outline

01 XFlash® 7 検出器のご紹介

02 ESPRIT LiveMap

03 バルク試料の高空間分解能SEM-EDS分析例

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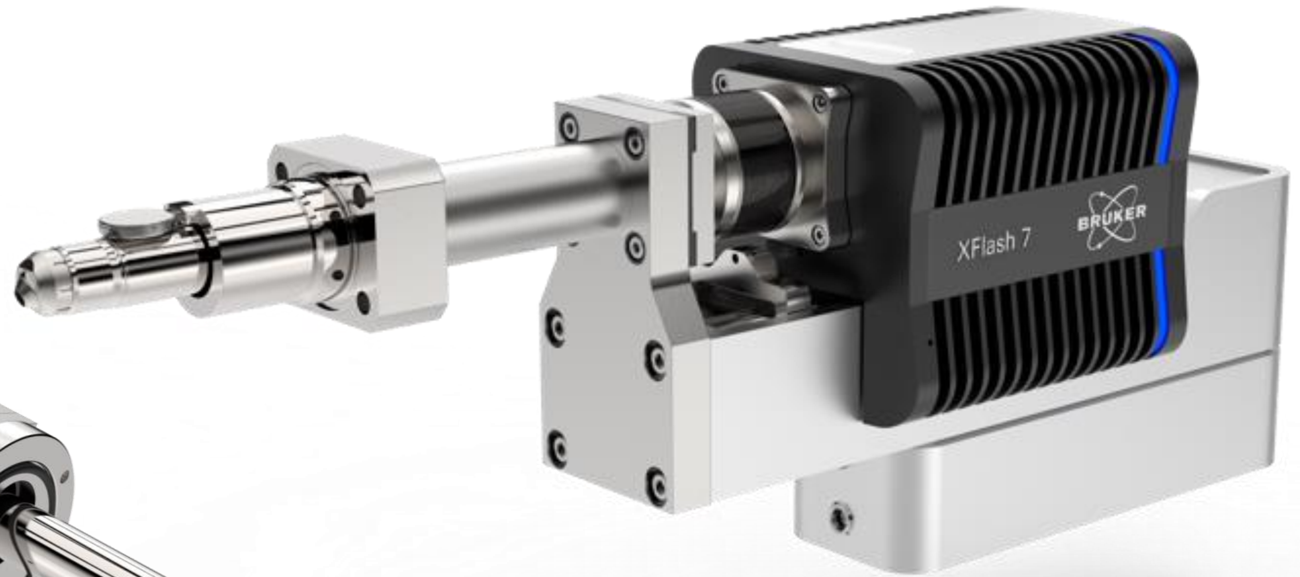
01

XFlash® 7 検出器のご紹介

XFlash® 7

高速に、正確に、信頼性の高いデータを提供します。



XFlash® 7 - the detector for SEM and FIB-SEM

XFlash® 7T - the detector for TEM and STEM

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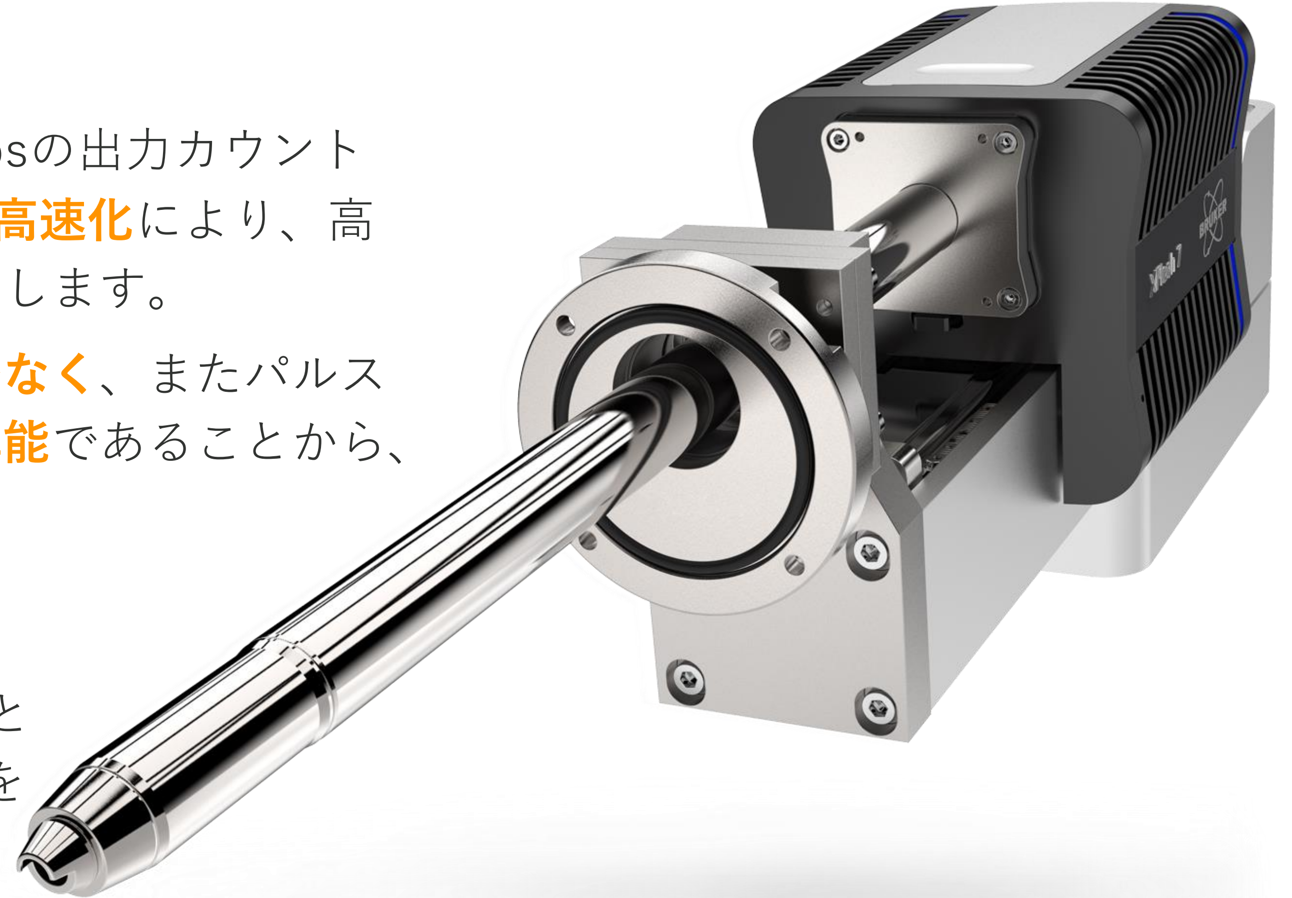
XFlash® 7 – Key facts

最大1,000,000 cpsの分析スループット

- 高入力カウントレート（ICR）で、最大1,000Mcpsの出力カウントレート（OCR）を実現しました。**X線処理速度の高速化**により、高速マップ・粒子解析が可能となり**分析速度が向上**します。
- デッドタイムを低く抑えられるため**信号損失が少なく**、またパルススループットレートに関わらず**高エネルギー分解能**であることから、品質を損なわずに効率良い測定ができます。

2,200以上の元素ピーク

- K, L, M, N線を網羅するエネルギーデータベースとESPRITソフト機能ピークデコンボリューションを使用することで、複雑な元素を定性・定量できます。



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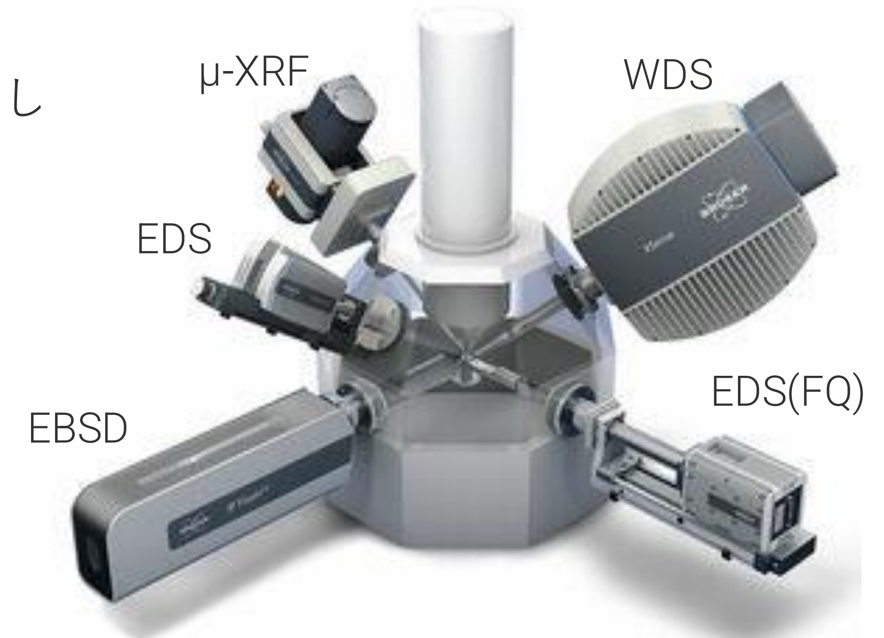
XFlash® 7 – Benefits

これまで以上に効率的なEDS分析を提供します。

- ユーザーフレンドリーなEspritソフトウエアを介すことにより、EDS, WDS, EBSD, micro-XRFを同一のソフトウエアGUIでコントロールし測定・解析・データ比較を行う事が可能です。
- **検出器修理はオンサイト、日本法人オフィスで可能**となり、修理時の時間短縮につながります。
- **オンデマンドで装置状況を確認する事が可能**であり、現状のシステム状態を確認する事により予知保全を実施可能これにより装置寿命の延長にもつながります。
- 様々な分野の皆様にご使用いただけるEDSシステムです。e.g., 材料化学、ライフサイエンス、半導体、電気電子、アカデミア....



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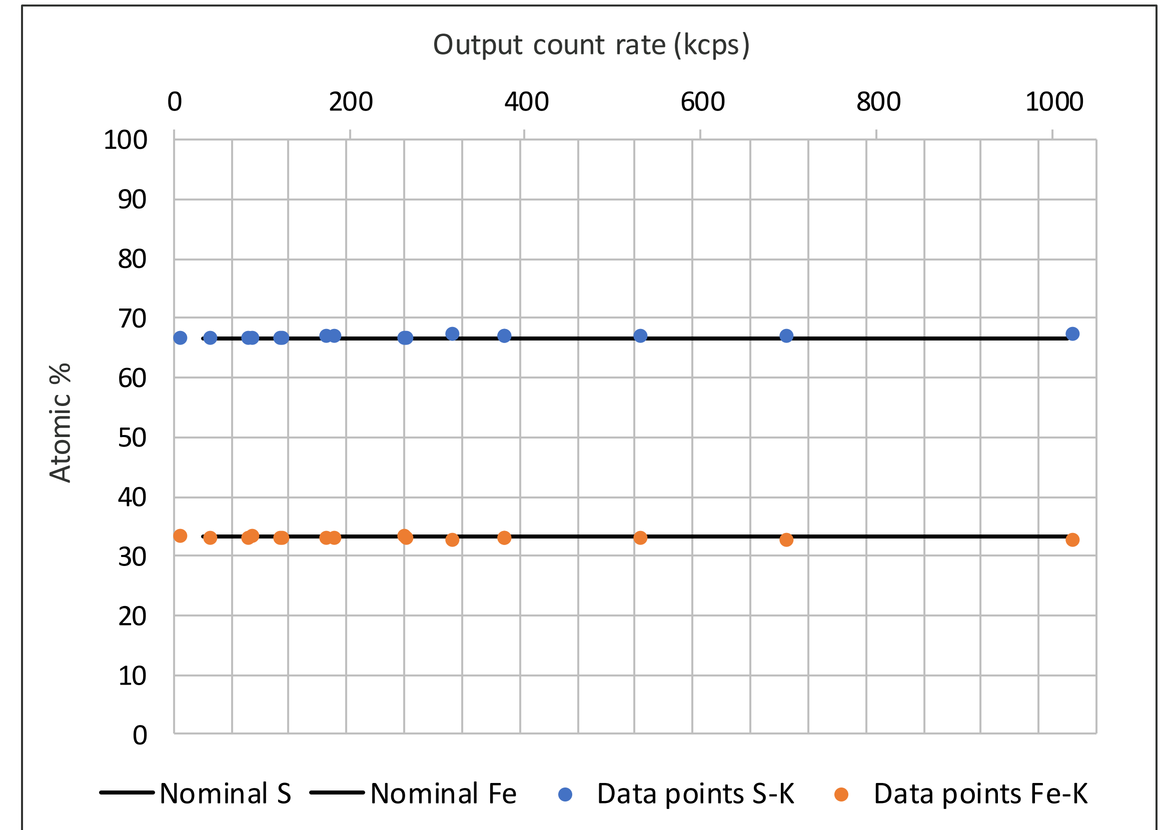
XFlash® 7 – Ensure reliability!

検出器の安定性は、データ取得と信頼性の高い定量結果を得る基礎です。

- 検出器を頻繁に調整する必要がないため、時間が節約できます。
- 最短測定時間で最高品質の定量結果を取得します。
- パルススループットとエネルギー分解能に関わらず、オーバーラップするピークのデコンボリューション（ピーク分離）が可能です。
- 極端な測定条件下でも、ESPRITソフトの独自な定量アルゴリズムにより定量結果の精度を維持できます。



Fe and S quantification results of stoichiometric FeS_2 at different output count rates (OCR)

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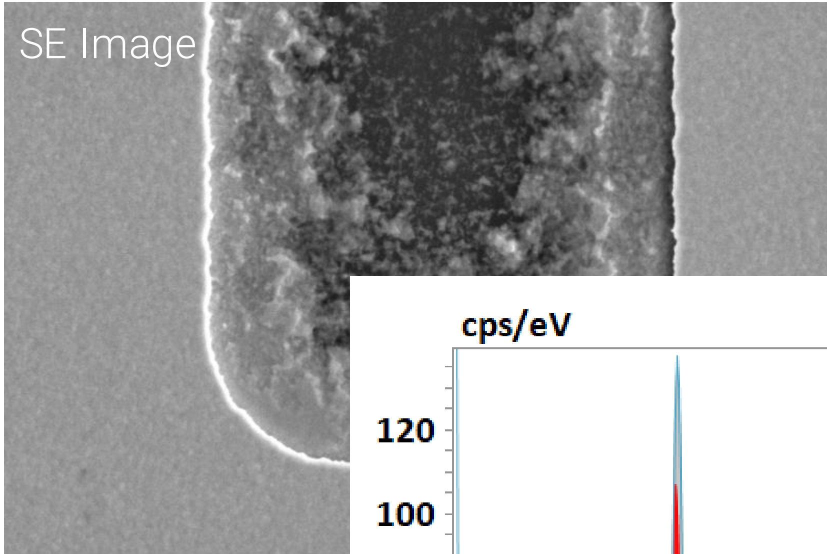
Is spectrum quality still sufficient at high throughput?

Task：ウェハーエッチング後のAu上の残留物Si, Wの特定。

Challenge：SiとWのオーバーラップ。ピークデコンボリューションを機能させるために、スペクトル品質を維持する必要がある。



cps/eV
120
100
80
60
40
20
0
O
Ti
W
Au
C
W
Au
Si
Ti
0
2
4
6
Energy [keV]

cps/eV
140
120
100
80
60
40
20
0
Si - Kα線：
1,740 keV
Si
W
W - Mα線：
1,775 keV
Au
1,40
1,60
1,80
2,00
2,20
2,40
Energy [keV]

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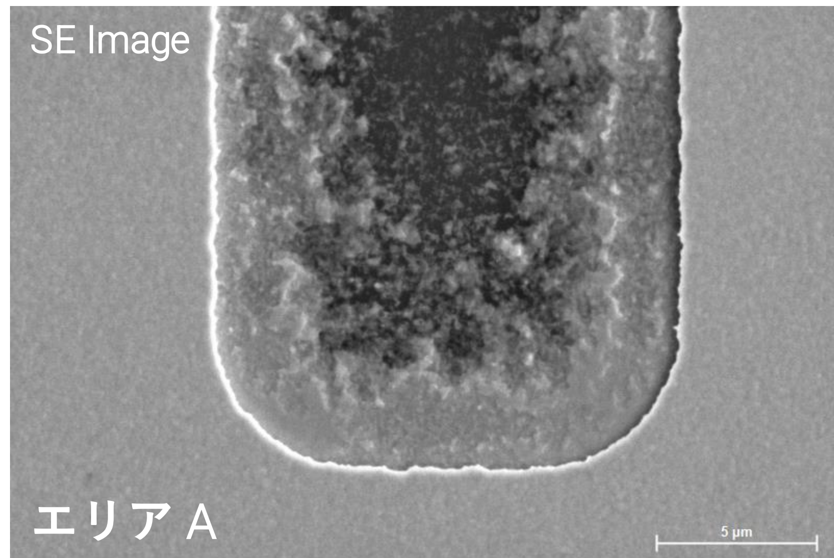
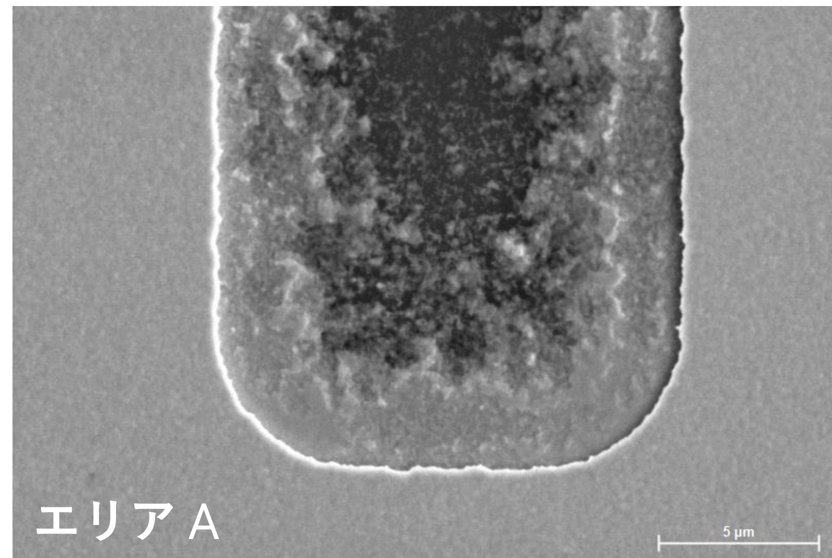
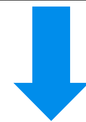
Is spectrum quality still sufficient at high throughput?

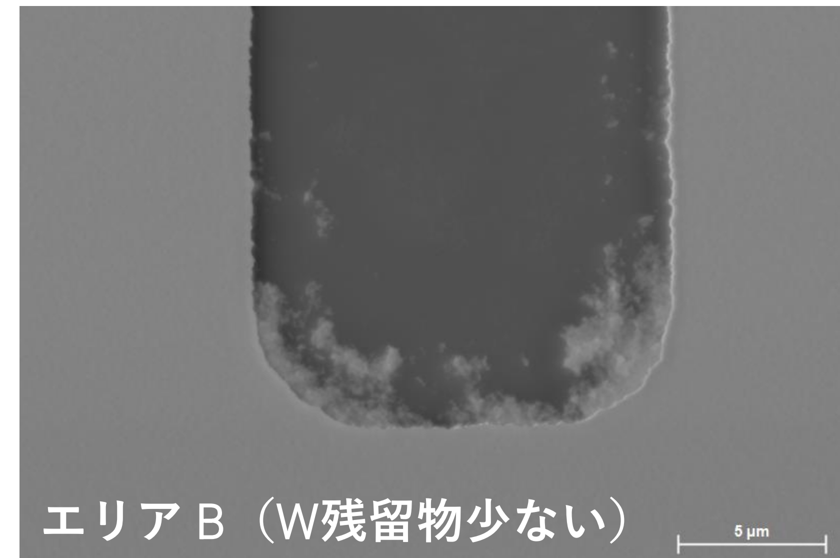


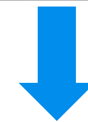
ビーム電流：0.4 nA
でマッピング⇒①





ビーム電流：5 nA
でマッピング⇒②





ビーム電流：20 nA
でマッピング⇒③

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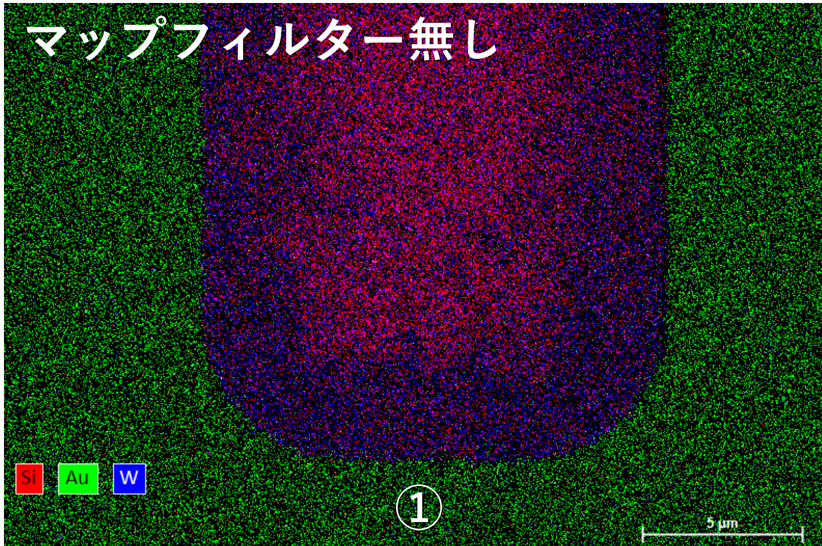
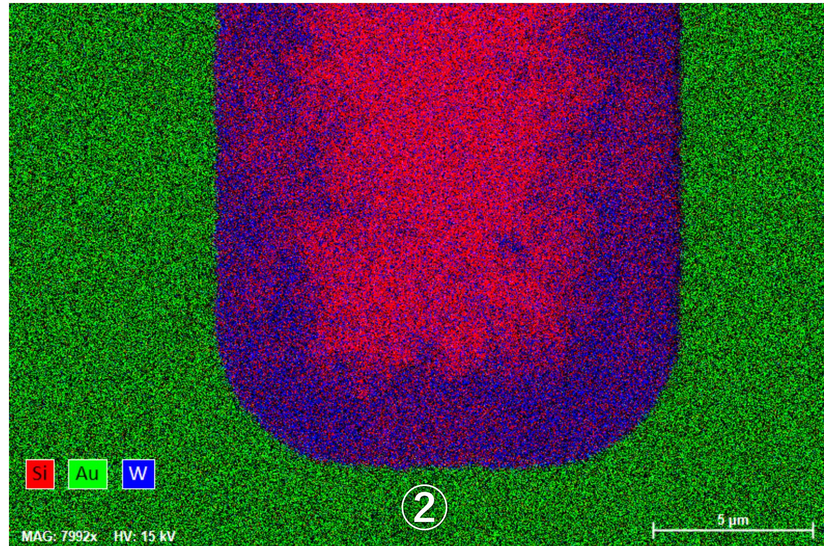
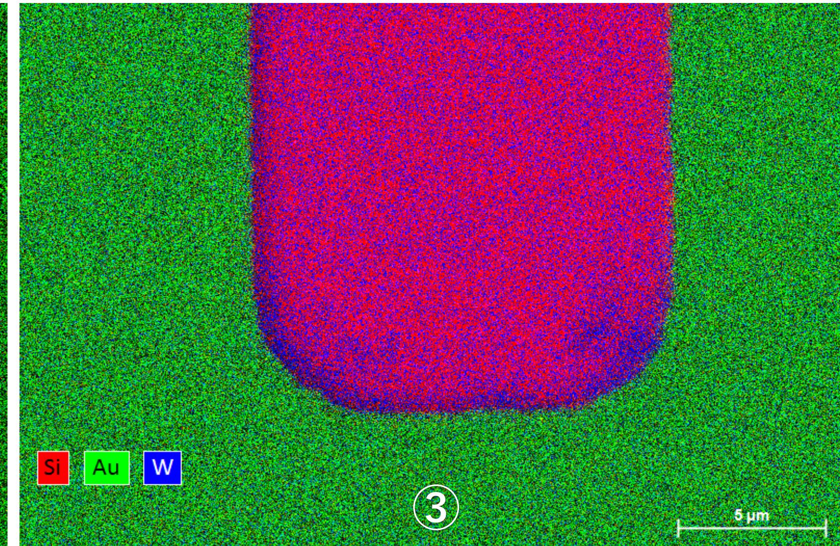
Is spectrum quality still sufficient at high throughput?







Measurement time	7 s (1 frame)	7 s (1 frame)	7 s (1 frame)
Beam current	0.4 nA	5 nA	20 nA
Input count rate	63 kcps	700 kcps	2600 kcps
Output count rate	51 kcps	410 kcps	1024 kcps
Dead time	18 %	38 %	61 %
Total counts	$3.5*10^5$	$2.7*10^6$	$6.6*10^6$
Relative counts	1x	7x	19x

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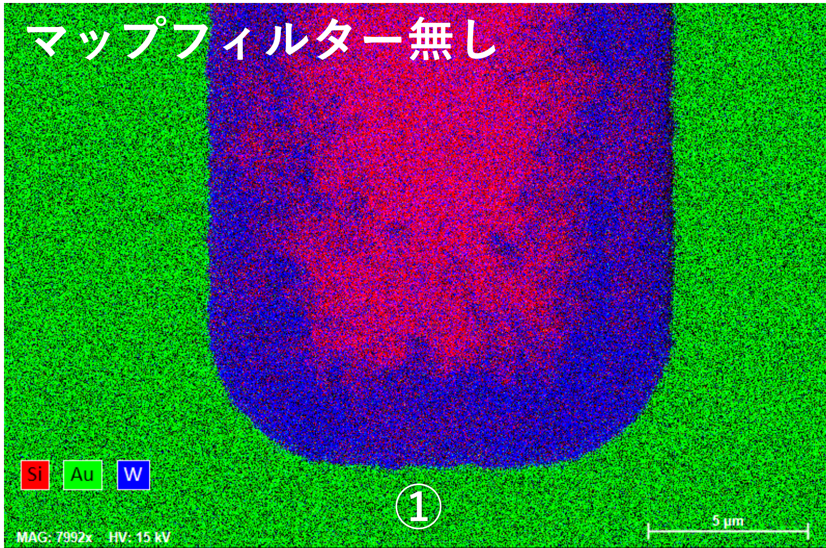
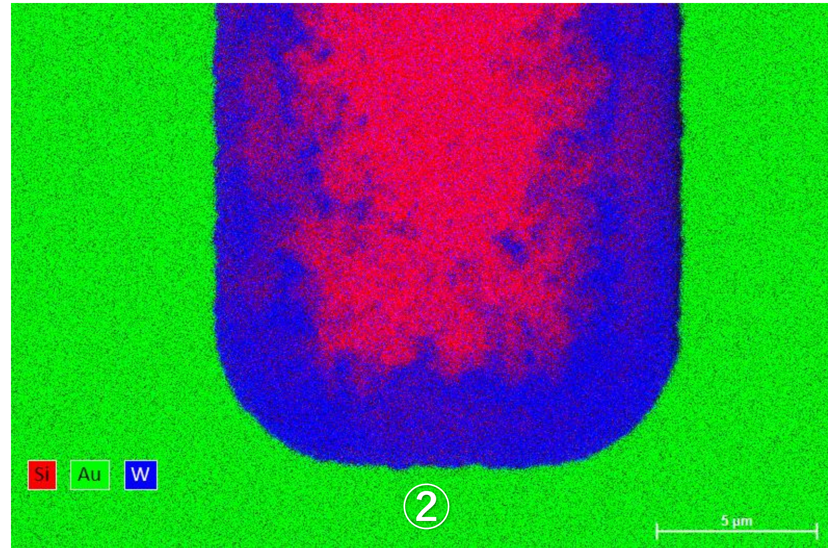
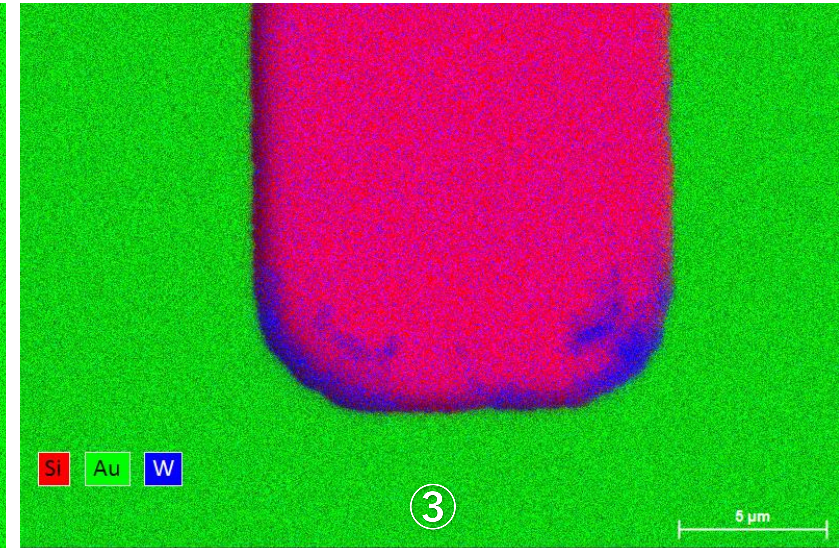
Is spectrum quality still sufficient at high throughput?







Measurement time	70 s (10 frame)	70 s (10 frame)	70 s (10 frame)
Beam current	0.4 nA	5 nA	20 nA
Input count rate	63 kcps	700 kcps	2600 kcps
Output count rate	51 kcps	410 kcps	1024 kcps
Dead time	18 %	38 %	61 %
Total counts	$3.5*10^6$	$2.7*10^7$	$6.6*10^7$

x7.7 more signal!

x19 more signal!

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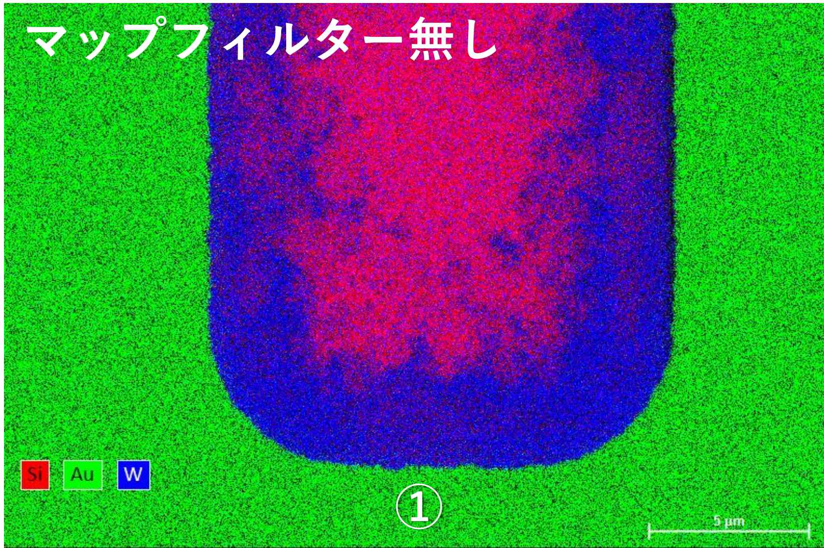
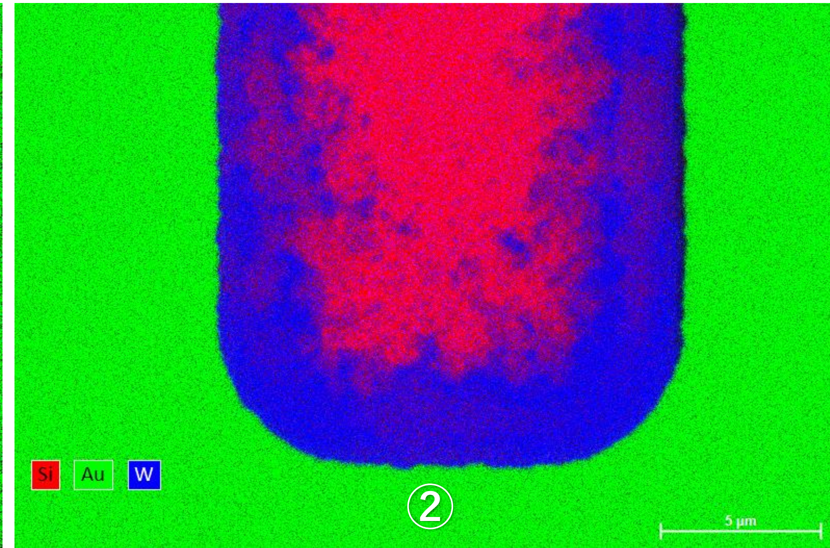
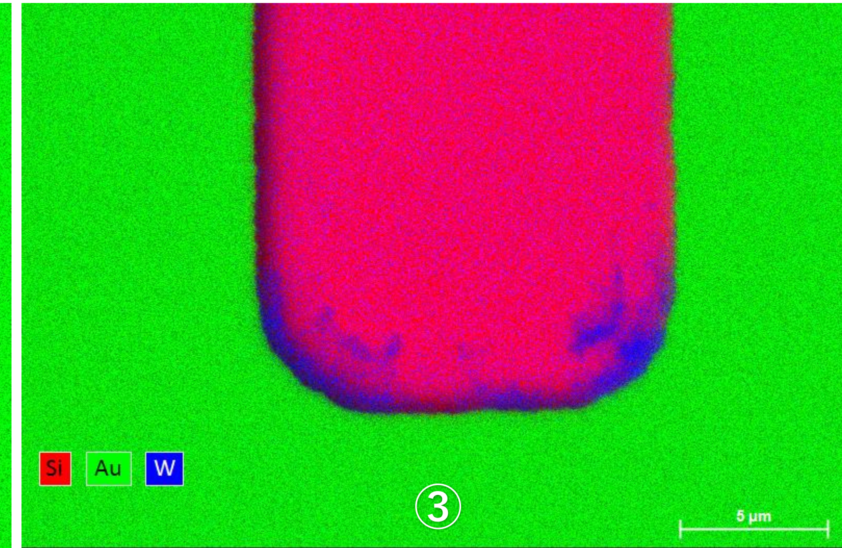
Is spectrum quality still sufficient at high throughput?







Measurement time	140 s (20 frame)	140 s (20 frame)	140 s (20 frame)
Beam current	0.4 nA	5 nA	20 nA
Input count rate	63 kcps	700 kcps	2600 kcps
Output count rate	51 kcps	410 kcps	1024 kcps
Dead time	18 %	38 %	61 %
Total counts	$7*10^6$	$5.5*10^7$	$1.3*10^8$

x7.7 more signal!

x19 more signal!

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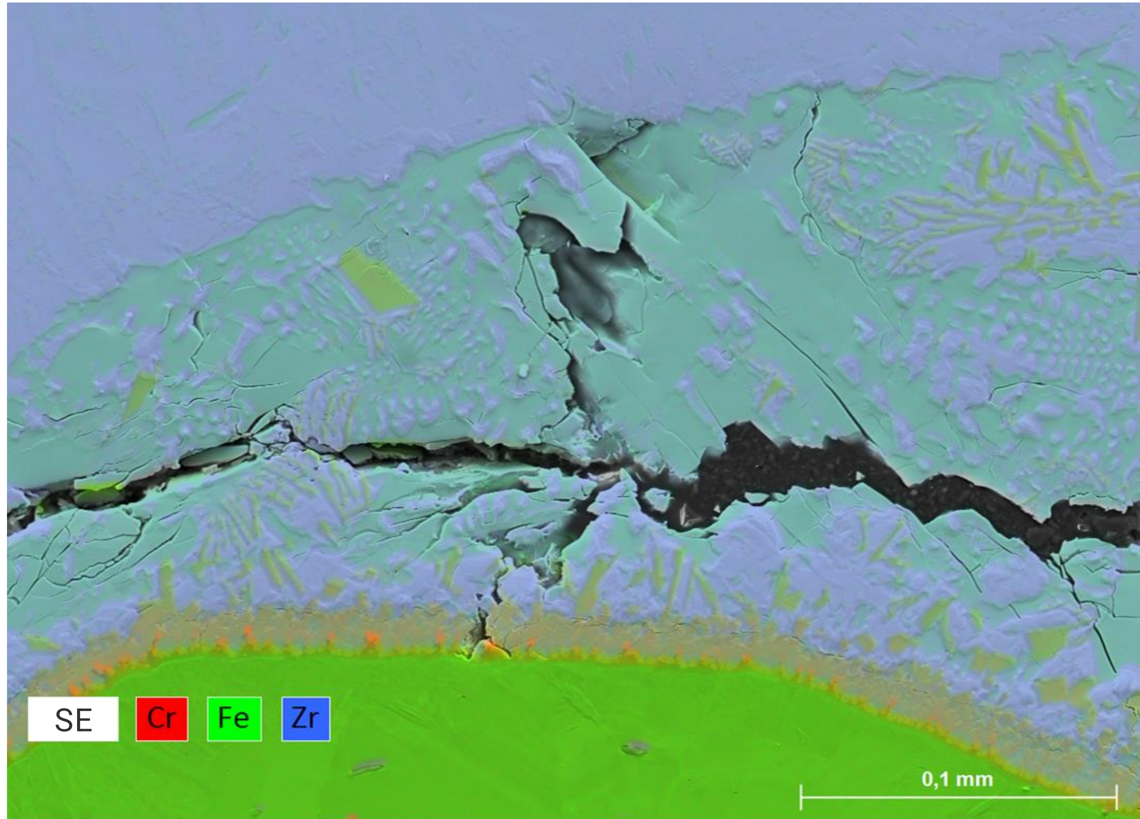
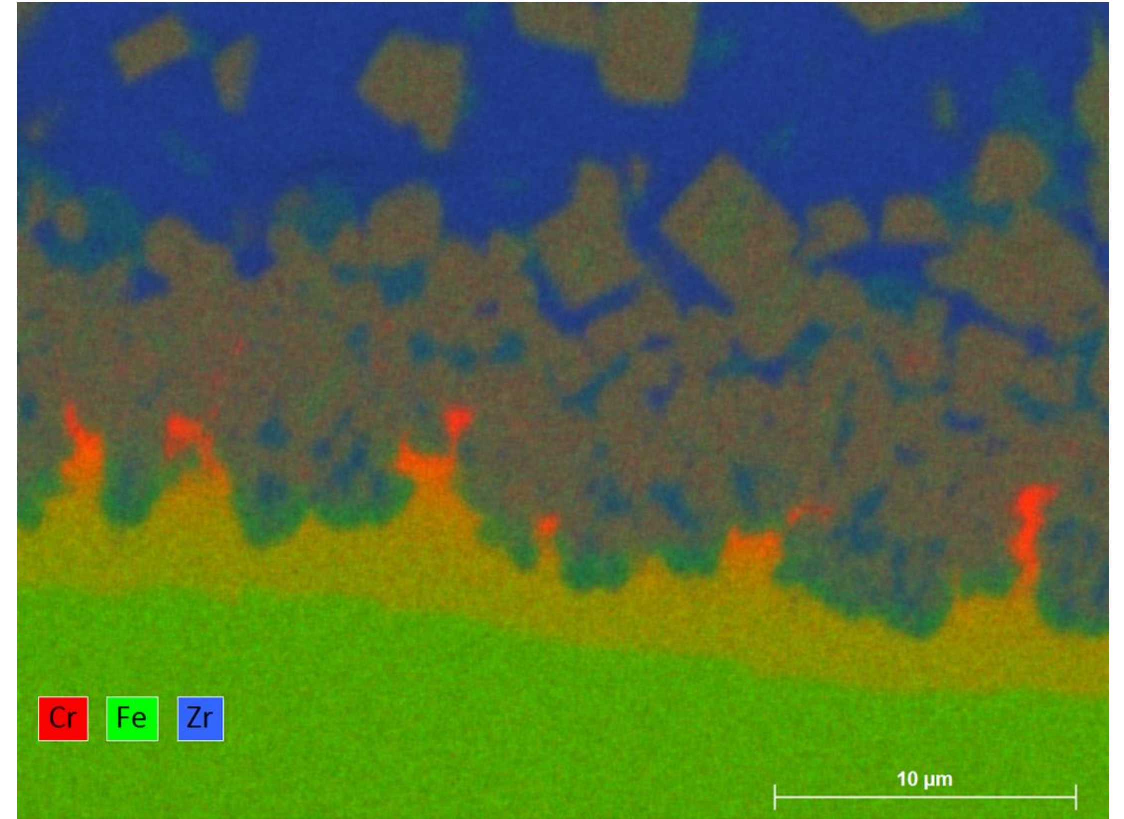
High throughput element mapping of Zr-steel welding seam



ICR: 2.25 Mcps, OCR: 900 kcps, Mapping time: 11 mins



ICR: 1.375 Mcps, OCR: 550 kcps, Mapping time: 5 mins

加速電圧：15 kV、ビーム電流：40 nA

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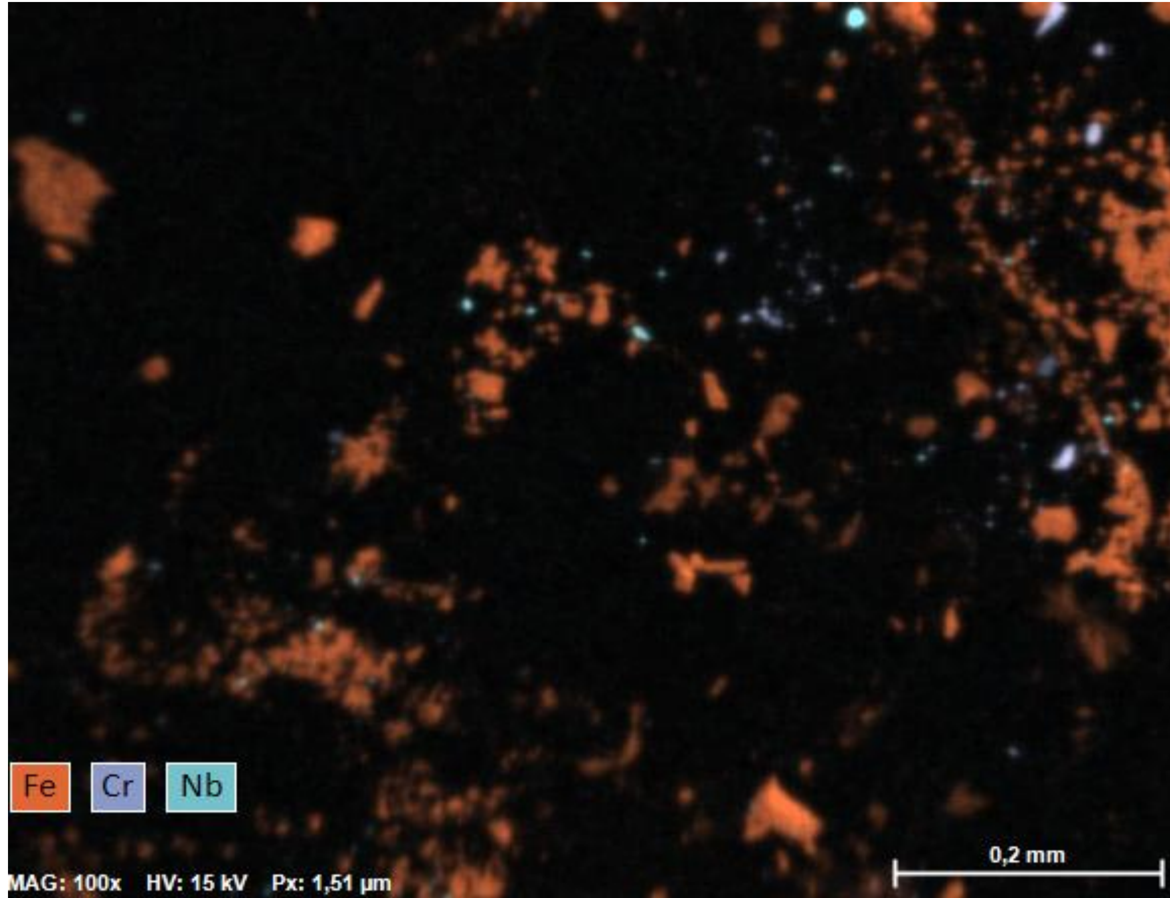
High throughput element mapping of Cr, Fe, Nb particles – single frame



加速電圧：15 kV, ビーム電流：10 nA

Task：粘着性カーボンパッド（コーティングなど前処理無し）上の金属粒子（Fe, Cr, Nbなど）を可能な限り短時間で位置を含めて特定。

Challenge：サンプルは、高ビーム電流に耐えられるか？スペクトル品質はこれらの元素マッピングをするのに十分であるか？

Measurement time	5 minutes
Input count rate	231 kcps
Output count rate	113 kcps
Dead time	50 %
Total counts	$3.5*10^7$

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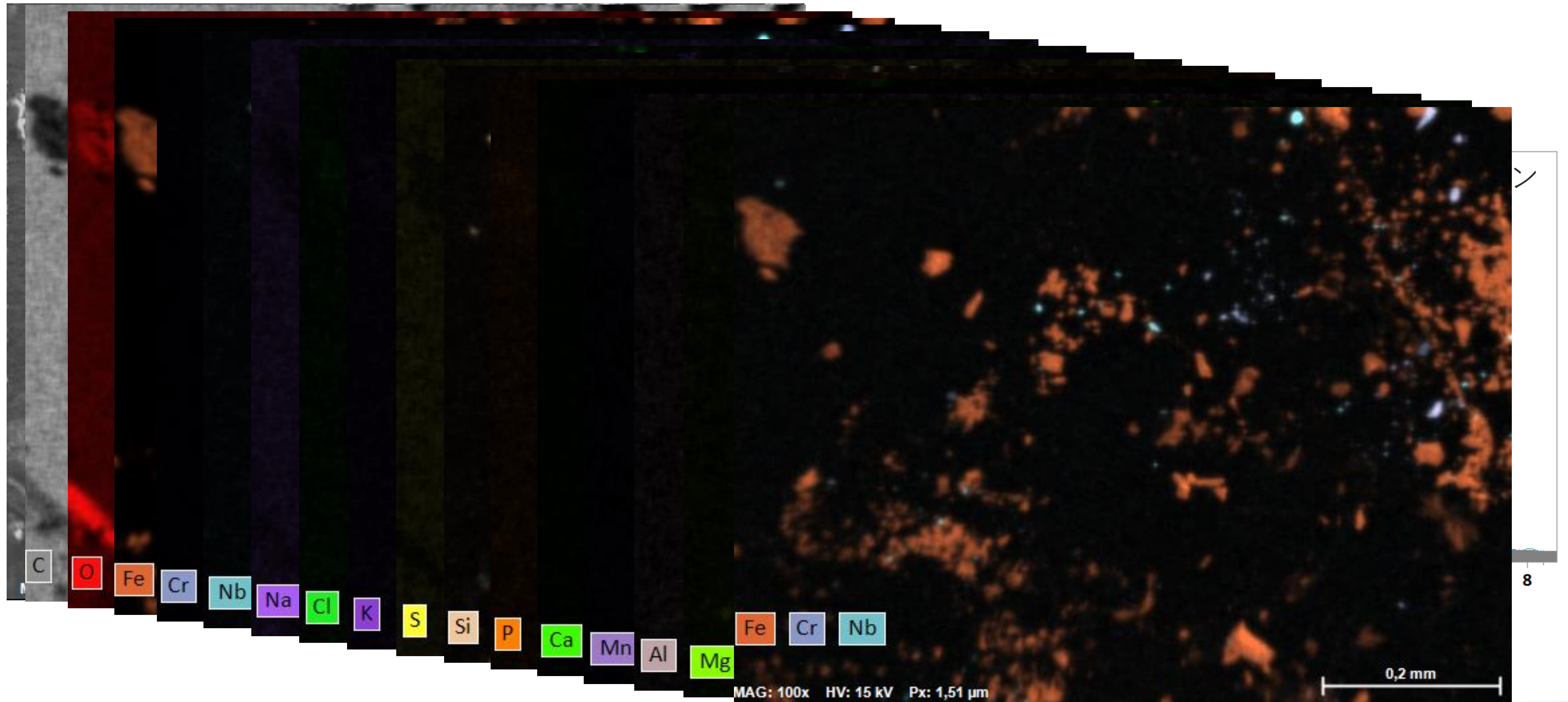
High throughput element mapping of Cr, Fe, Nb particles – single frame



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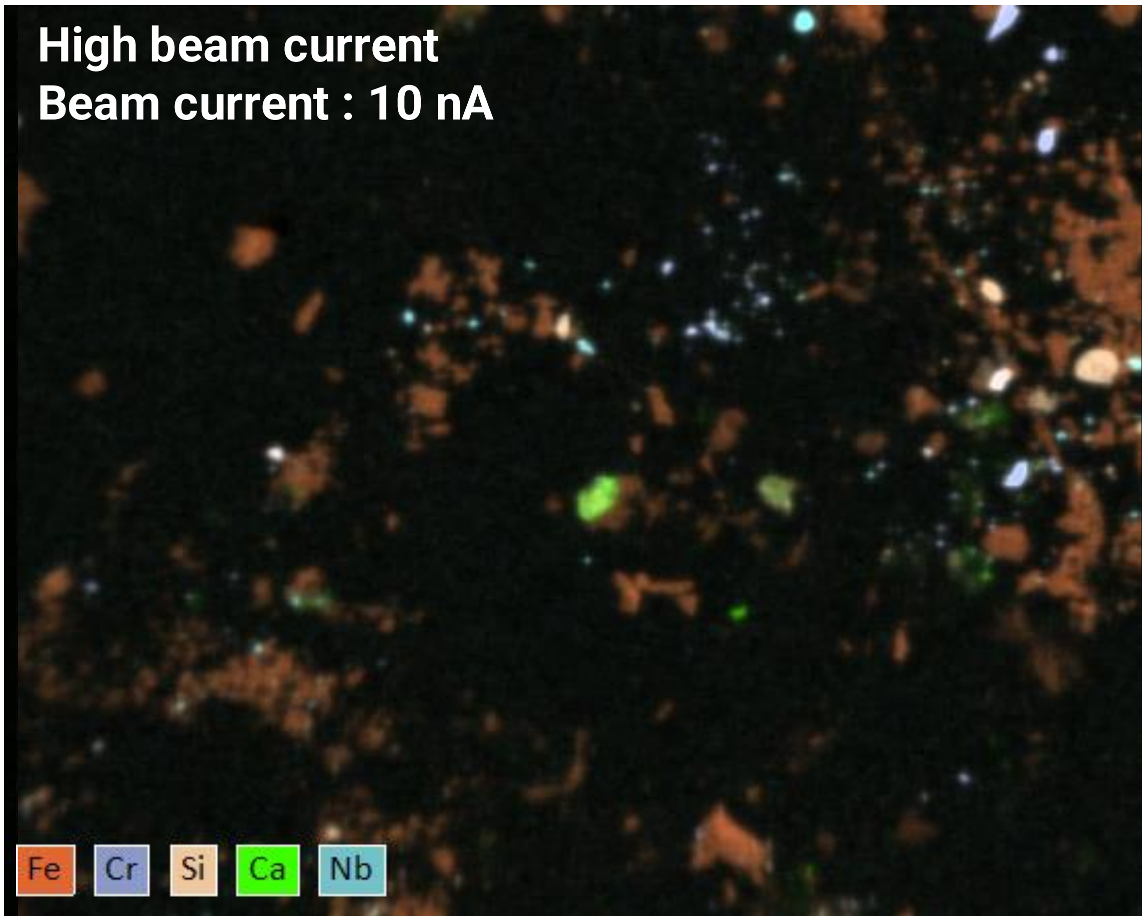
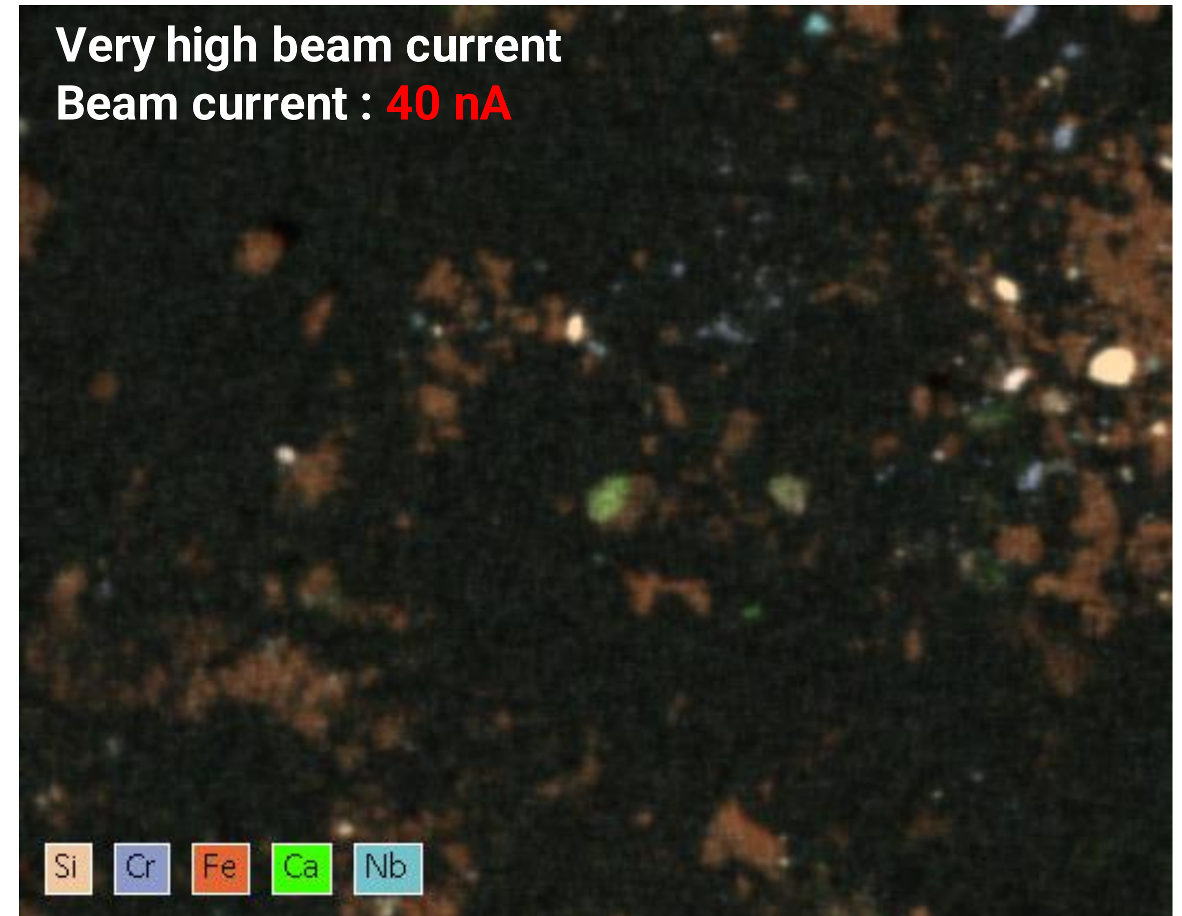
High throughput element mapping of Cr, Fe, Nb particles – single frame



map time: 5 minutes
Average ICR: 231 kcps, OCR: 113 kcps



map time: 15 seconds (1 frame)
Average ICR: 630 kcps, OCR: 440 kcps
Maximal count rates reached ICR : 2000 kcps, OCR: 900kcps

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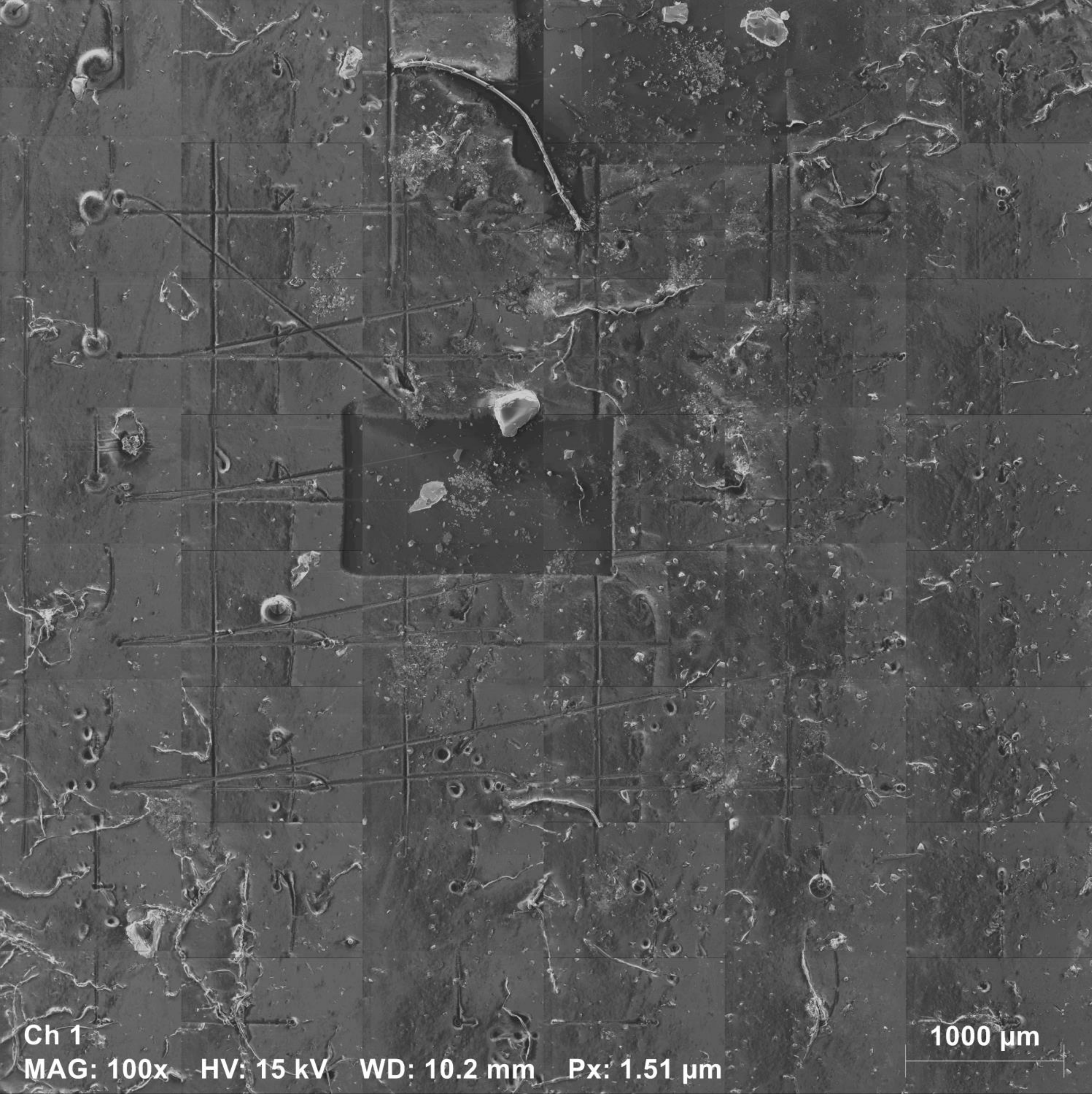

High throughput element mapping of Cr, Fe, Nb particles

- Large are Mapping with Esprit JOBS

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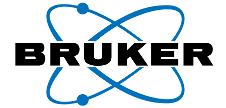
1 single frame map time	: 15 seconds
Total Image resolution	: 4583x4568 pixel
Mapping area	: 7mm x 7mm= 49 mm^2
6x8=48 frames map time	: 15x48 seconds=12 mins + stage positioning, data saving, generating of mosaics
Average ICR	**: 630 kcps**
OCR	**: 440 kcps**
加速電圧	: 15 kV
ビーム電流	: 40 nA

Si
MAG: 100x HV: 15 kV WD: 10.2 mm Px: 1.51 µm
1000 µm

High throughput element mapping of Cr, Fe, Nb particles



- Large are Mapping with Esprit JOBS

1 single frame map time	: 15 seconds
Total Image resolution	: 4583x4568 pixel
Mapping area	: 7mm x 7mm= 49 mm^2
6x8=48 frames map time	: 15x48 seconds=12 mins + stage positioning, data saving, generating of mosaics
Average ICR	**: 630 kcps**
OCR	**: 440 kcps**
加速電圧	: 15 kV
ビーム電流	: 40 nA

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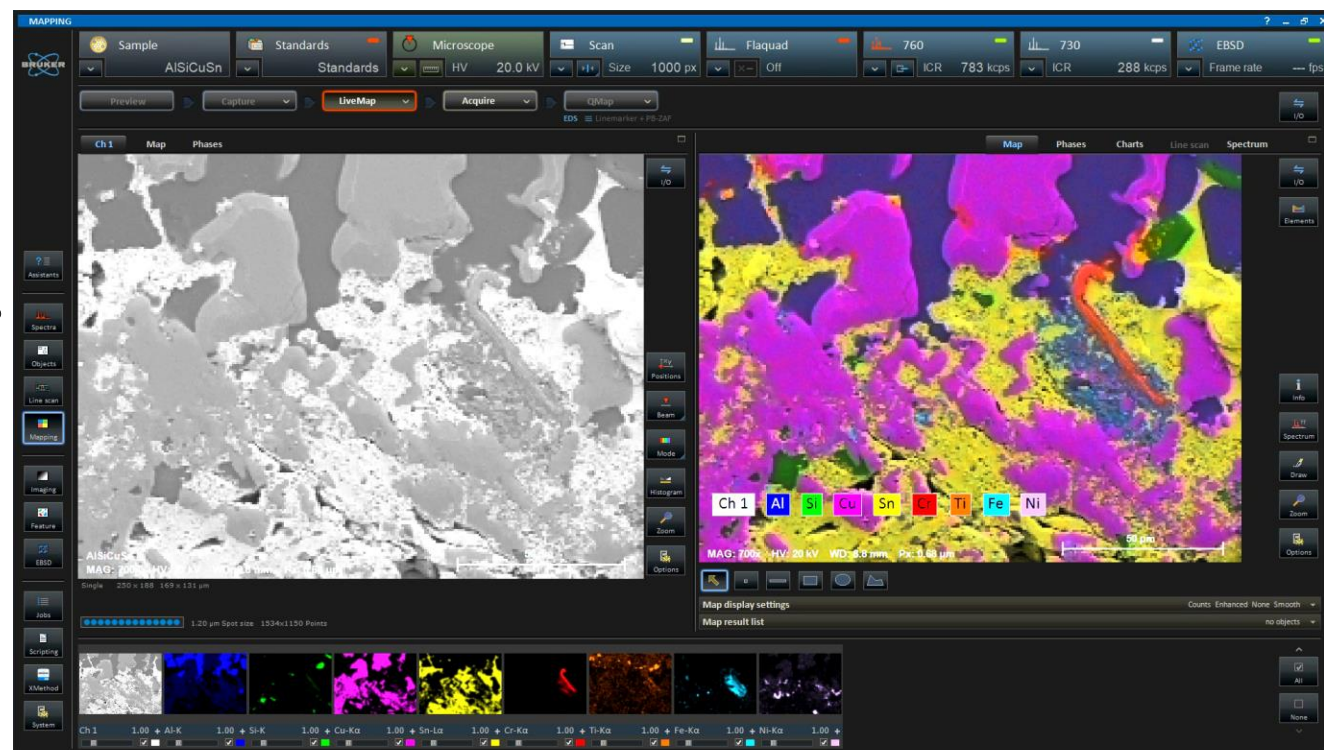
02

ESPRIT LiveMap

ESPRIT LiveMap

最大1,000,000 cpsの高OCRによるリアルタイムEDSマッピングが可能です！

- 検出元素のライブアップデート（自動/手動）ができます。
- LiveMapの解像度はユーザーで決定できます。
- 指定部分のスペクトルを抽出・表示できます。
- 微小領域に局在する微量元素をライブ検出できます。
- 従来マッピングへ簡単に切り替えられます。



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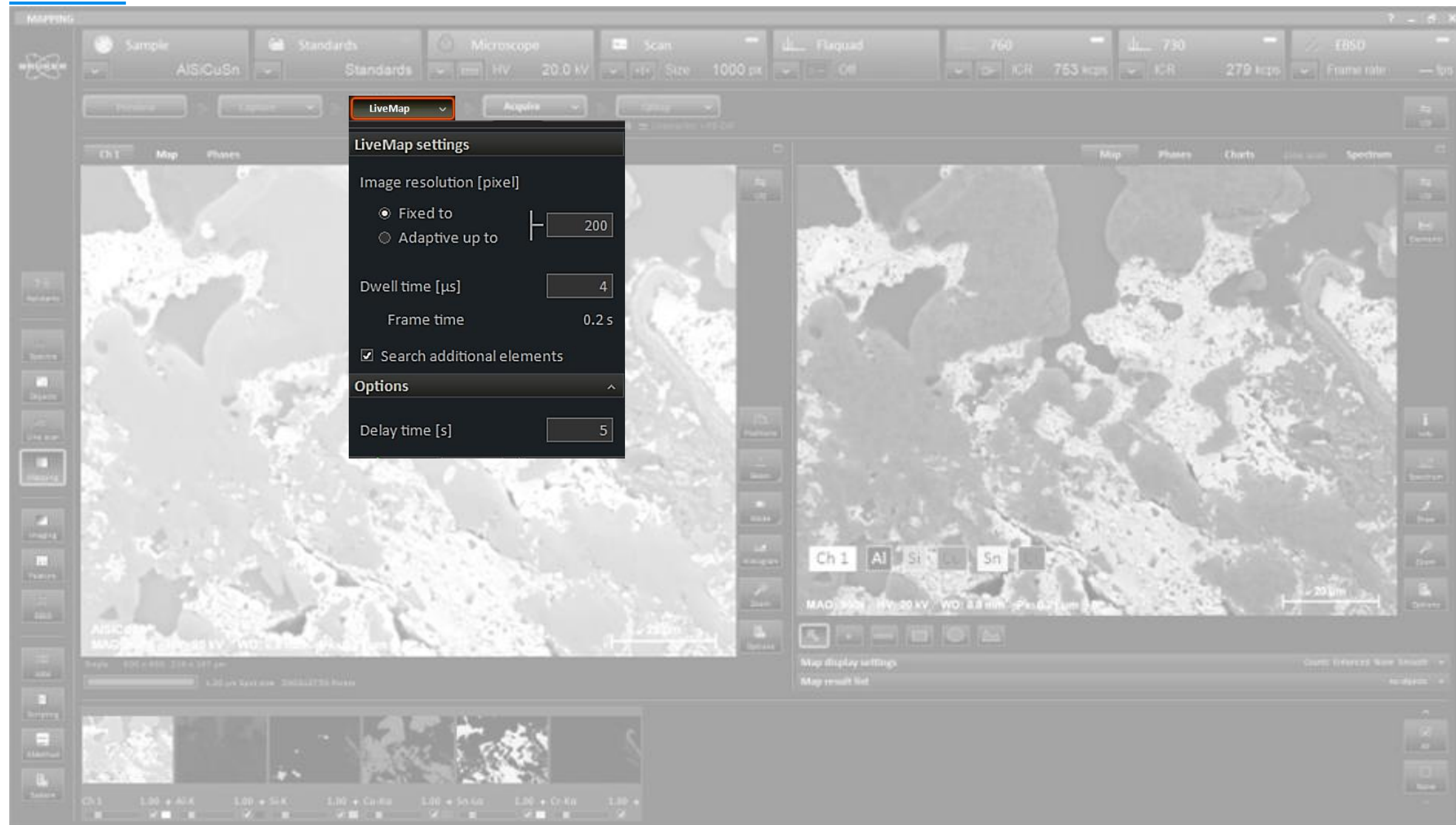
ESPRIT LiveMaps – GUI



ESPRIT LiveMap

LiveMap interface and Auto ID

Flexible interface, extraction of object spectra

- マッピングでは、左側にSEM像、右側にEDS map像を表示させる他、左側にEDS map像、右側にスペクトルを表示させることができます。
- 画面全体の色合いは、default（グレー）またはdark（ブラック）のどちらかが選択できます。

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ESPRIT LiveMap

Map objects for spectrum extraction in LiveMap mode

ESPRIT LiveMap

Map objects to identify chemistry of
a specific region in LiveMap mode

ESPRIT LiveMap

Maximum pixel spectrum for low concentration/trace element identification in LiveMap mode

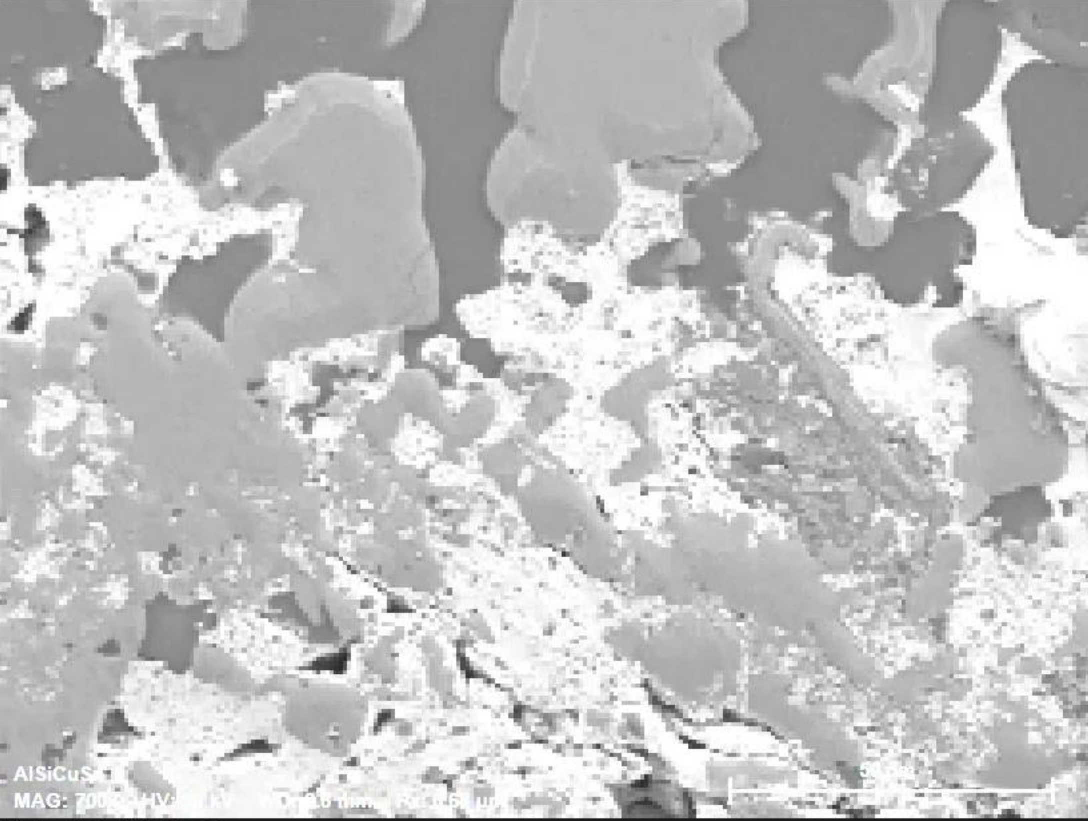
MAPPING

Sample AlSiCuSn | Standards Standards | Microscope HV 20.0 kV | Scan Size 1000 px | Flaquad Off | 760 ICR 764 kcps | 730 ICR 280 kcps | EBSD Frame rate --- fps

Preview | Capture | LiveMap | Acquire | QMap

EDS Linemarker + PB-ZAF

Ch 1 | Map | Phases



Single 250 x 188 169 x 131 µm

1.20 µm Spot size 1534x1150 Points

Map | Phases | Charts | Line scan | Spectrum

Assistants, Spectra, Objects, Line scan, Mapping, Imaging, Feature, EBSD, Jobs, Scripting, XMethod, System

I/O, Positions, Beam, Mode, Histogram, Zoom, Options

	All	cps	Real	Live	Dead	Pulses	Input	Output	Max. pulse throughput
EDS	Map	3015.42	114 s	65 s	43 %	40907611	659.1 kcps	358.1 kcps	600 kcps
EDS	Maximum pixel spectrum	32588.06	0.0 s	0.0 s	0 %	28644	2439.0 kcps	2203.4 kcps	600 kcps

ESPRIT LiveMap

Switching from LiveMaps to conventional mapping

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03

バルク試料の高空間分解能SEM-EDS分析例

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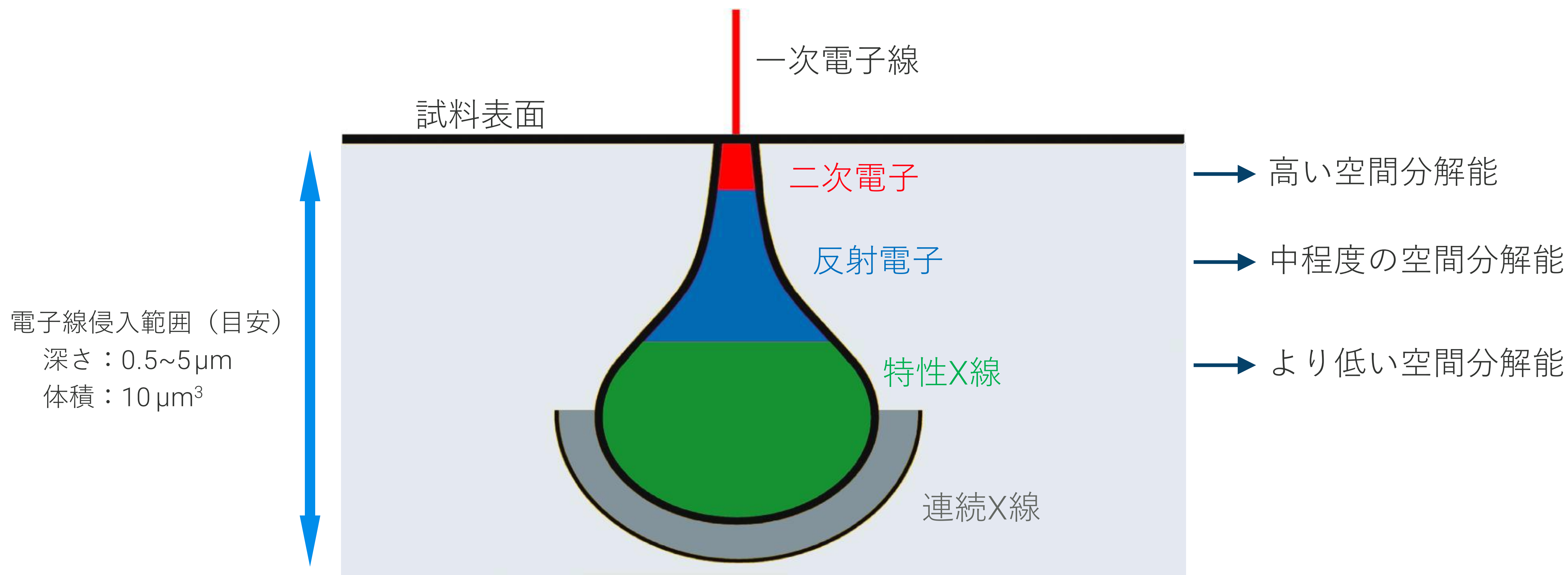
Interaction volume - signal generation



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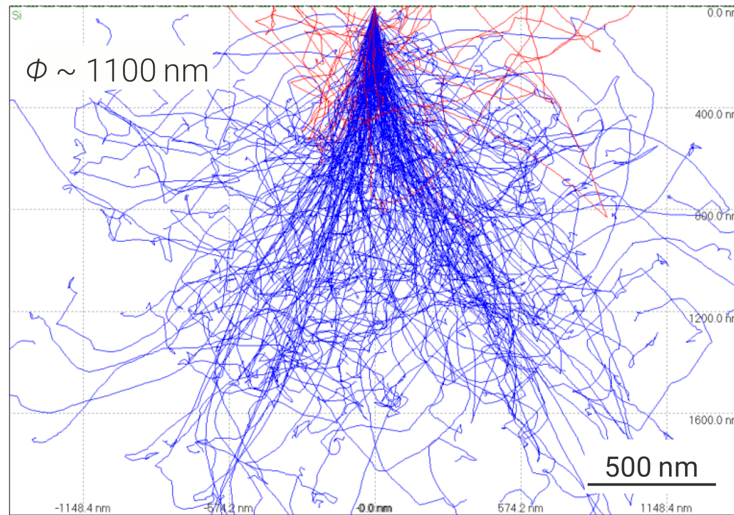
Interaction volume - signal generation

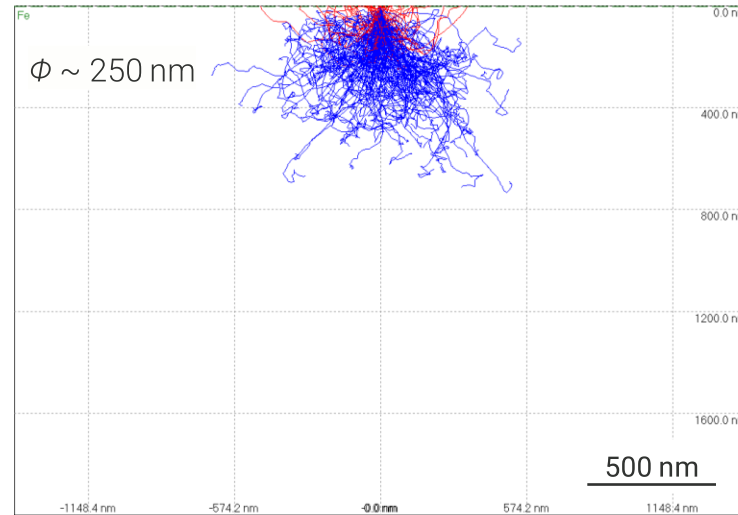
- Signal generation depth (interaction volume) at 15 kV for different materials

Si - 15 kV



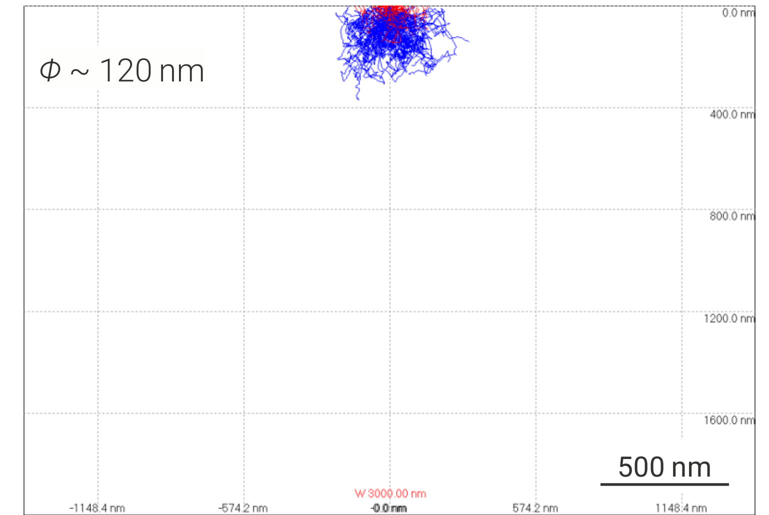
$Z = 14$ $\rho = 2.33\ g/cm^3$ Kα = 1.74 keV

Fe - 15 kV



$Z = 26$ $\rho = 7.86\ g/cm^3$ Kα = 6.40 keV

W - 15 kV

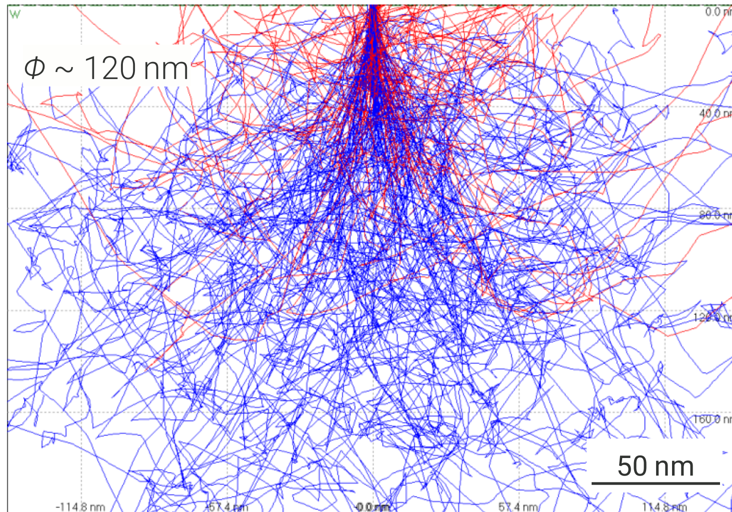


$Z = 79$ $\rho = 19.3\ g/cm^3$ Lα = 9.70 keV

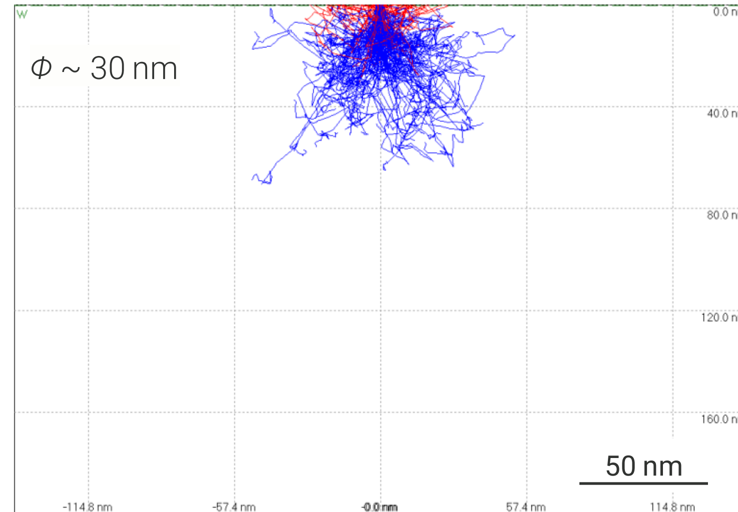
Interaction volume - signal generation

- Signal generation depth (interaction volume) for W at different primary energies

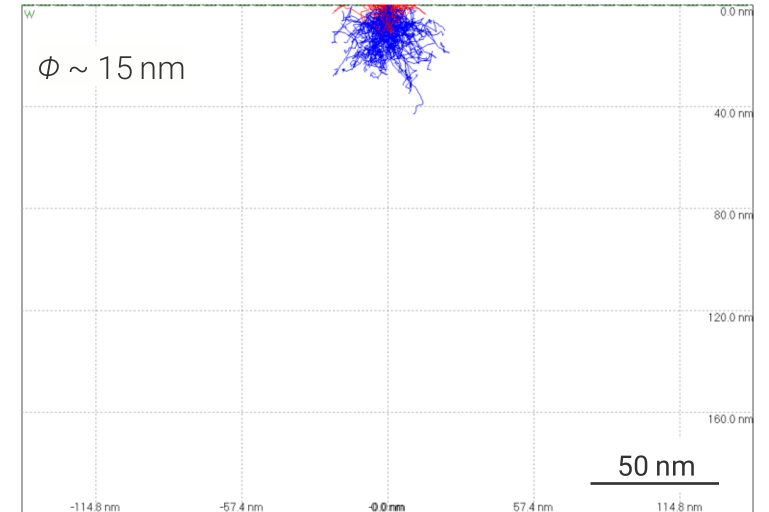
W - 15 kV



W - 5 kV



W - 3 kV



Z = 74 ρ = 19.3 g/cm^3 Lα = 8.39 keV

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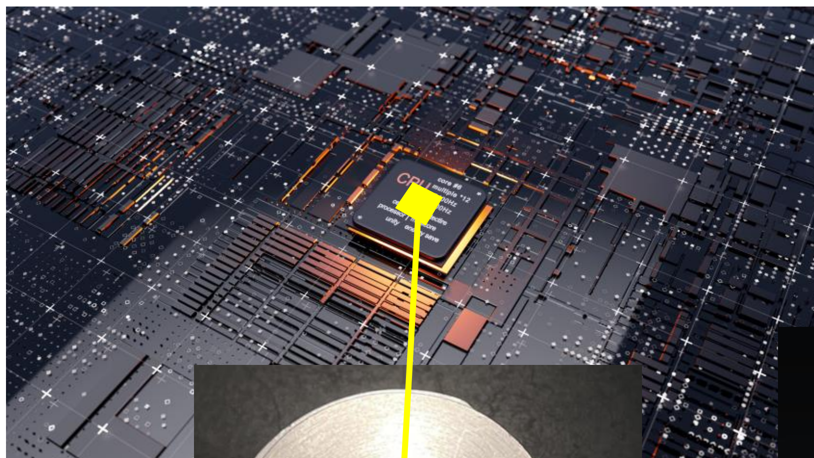
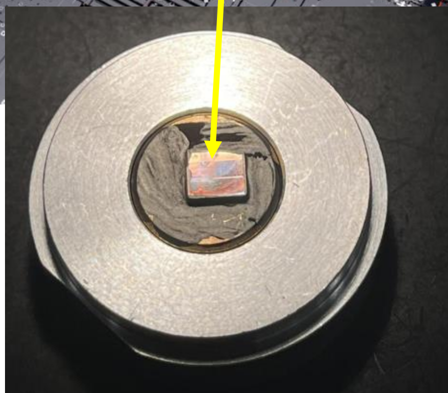
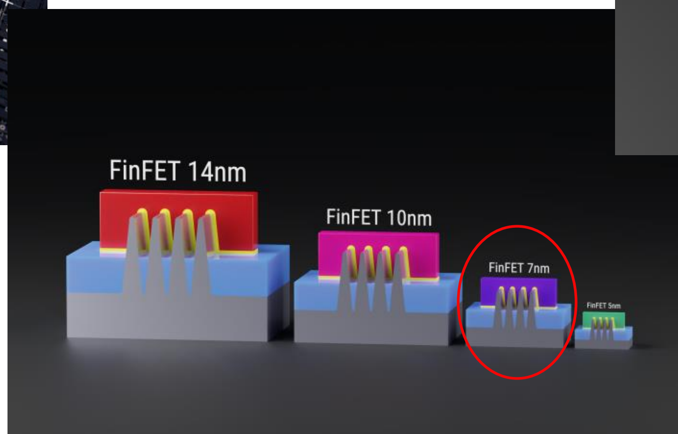
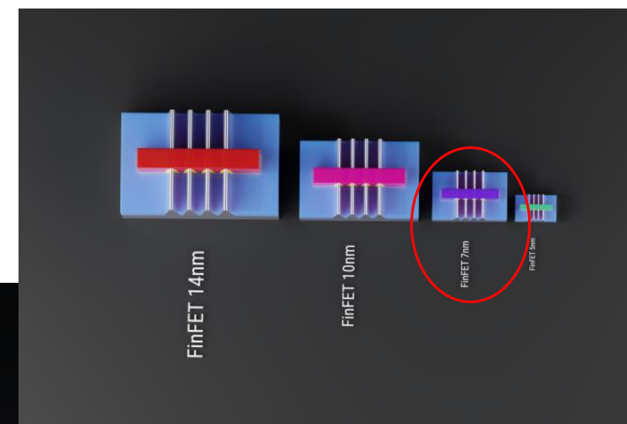
測定例1: FinFET 7 nm



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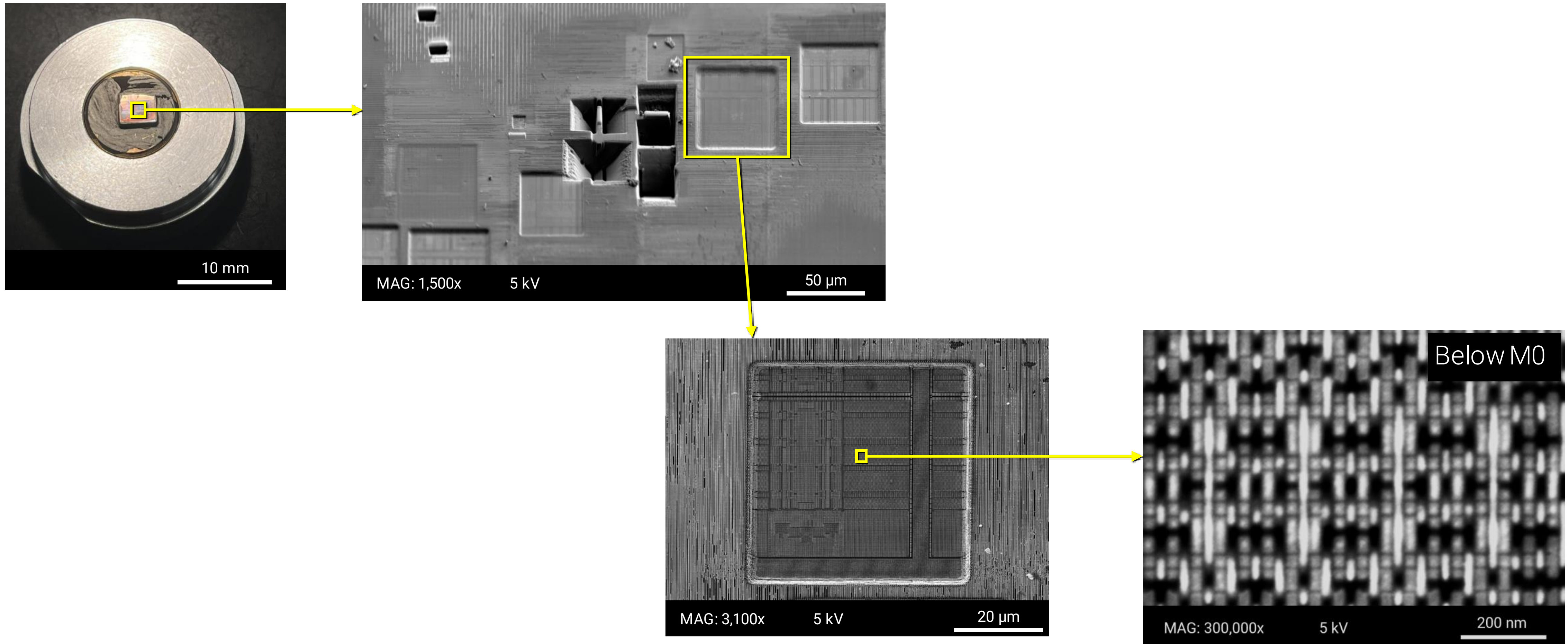
Deprocessed CPU (FinFET) – PFIB delayering



Sample courtesy: Dr. Andrey Denisyuk, TESCAN ORSAY HOLDING, a.s, Brno, CZ

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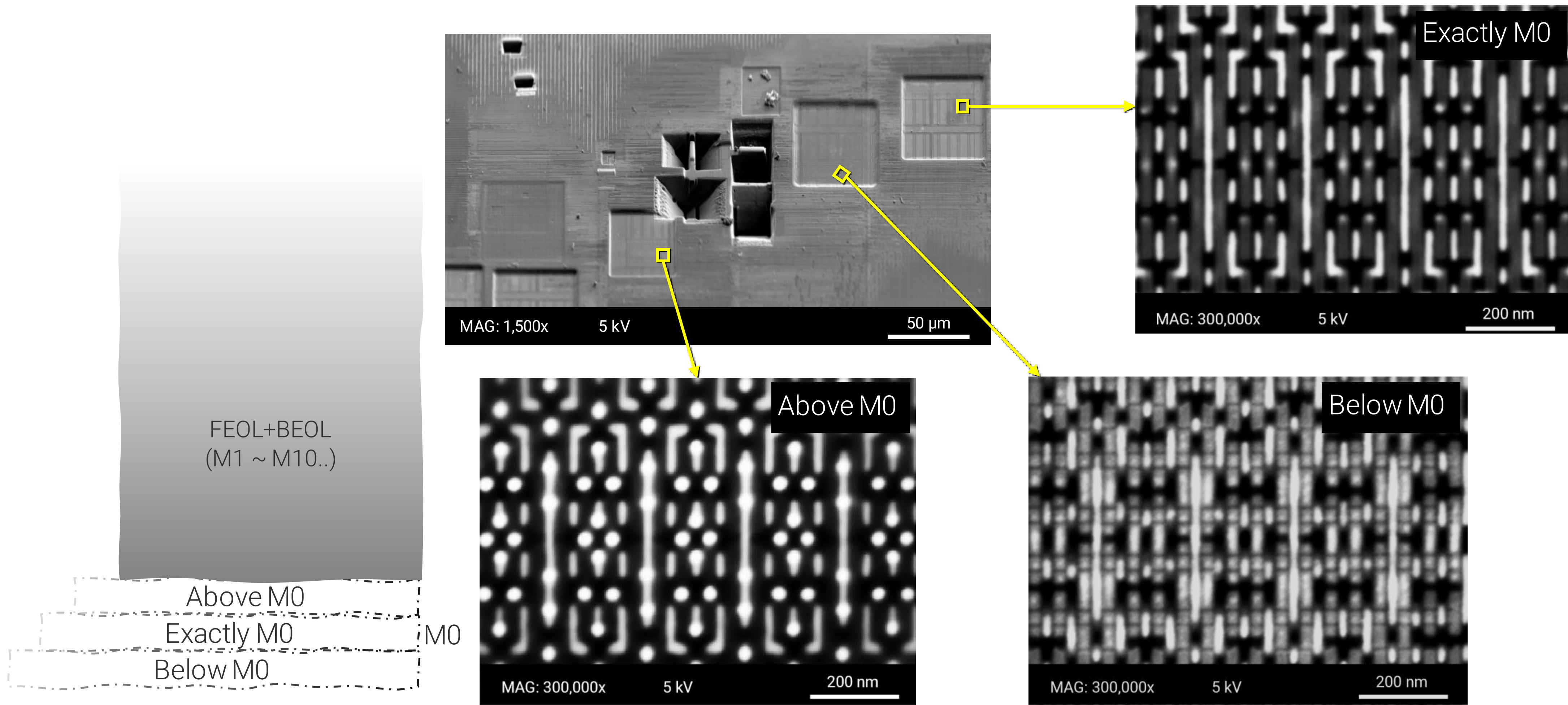
Deprocessed CPU (FinFET) – PFIB delayering



Sample courtesy: Dr. Andrey Denisyuk, TESCAN ORSAY HOLDING, a.s, Brno, CZ

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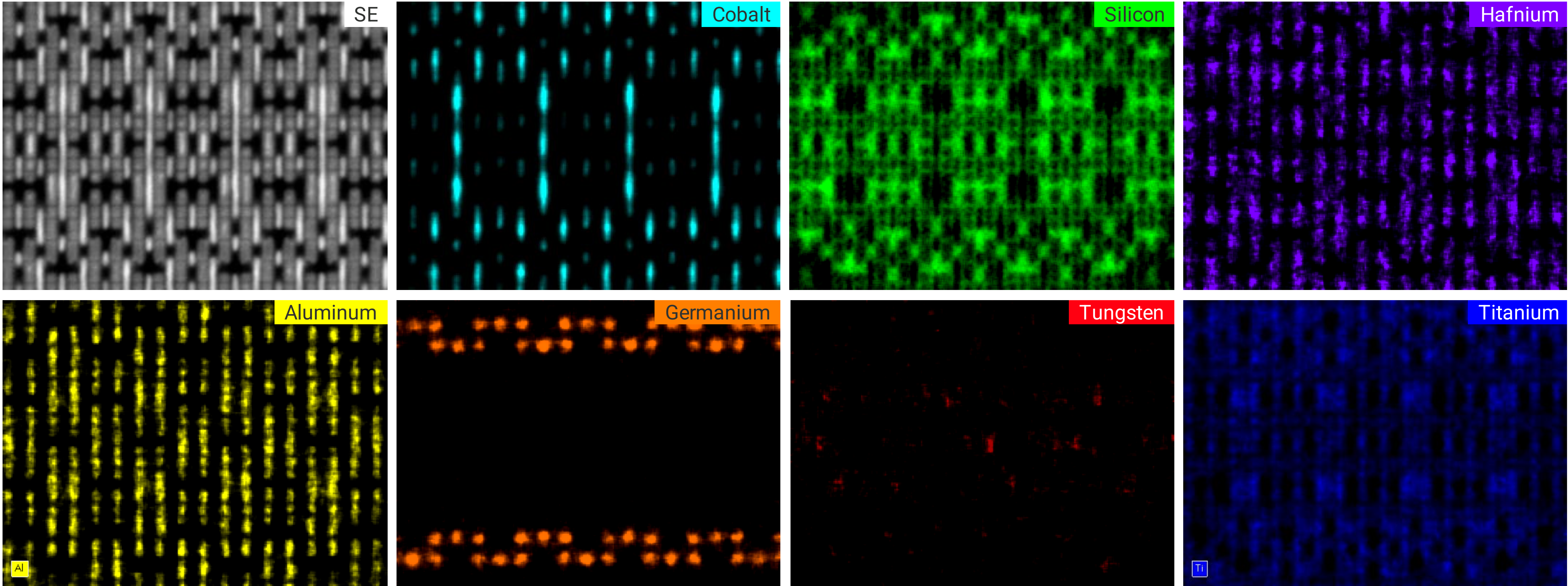
5 kV, 300kx MAG, Below M0



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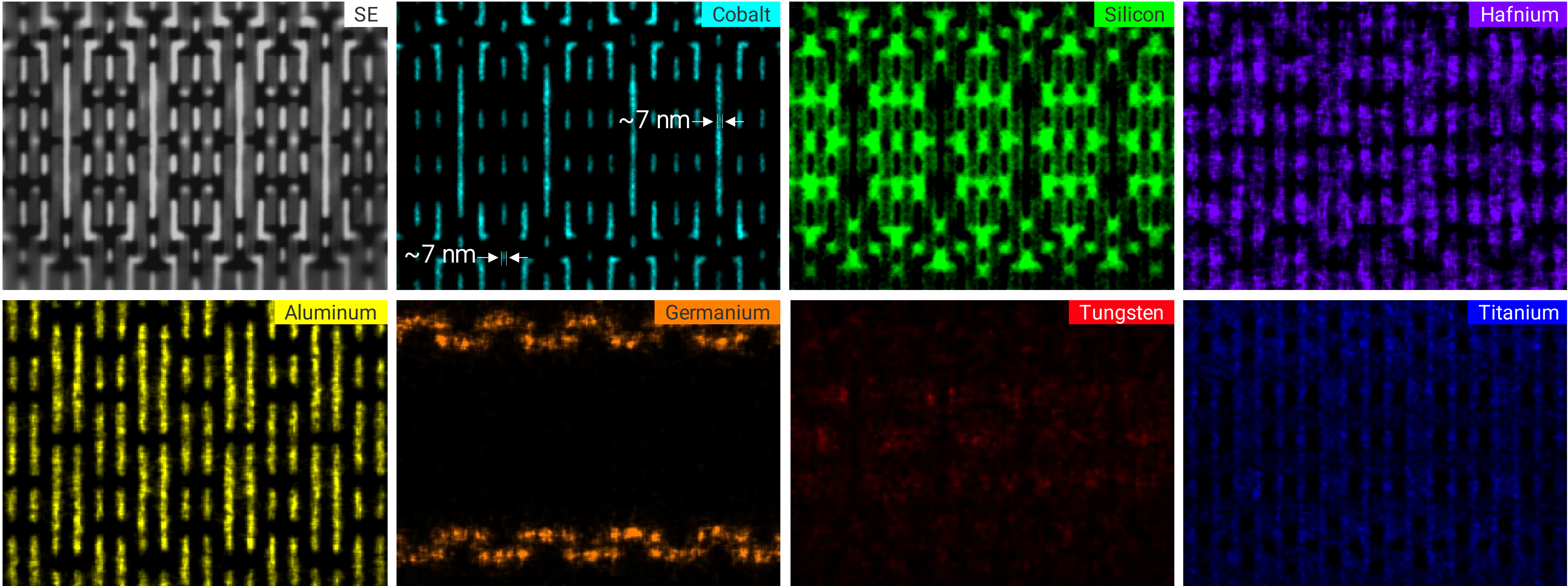
5 kV, 300kx MAG, Exactly M0



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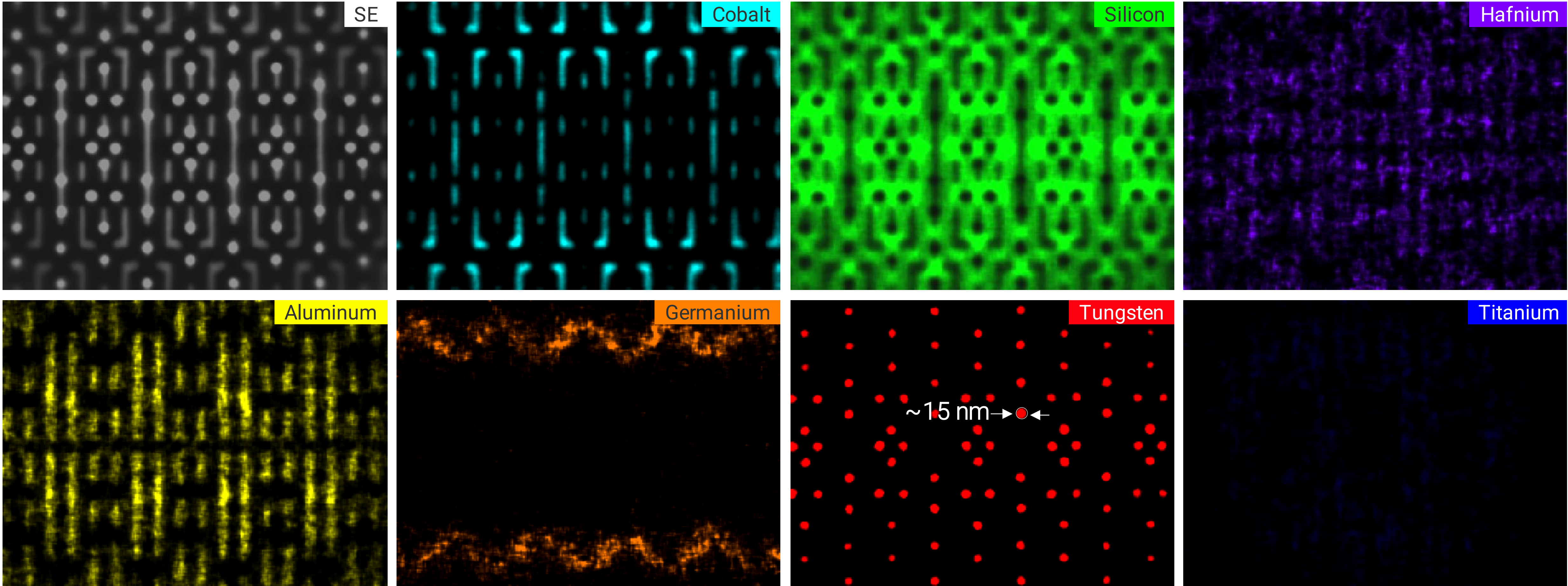
5 kV, 300kx MAG, Above M0



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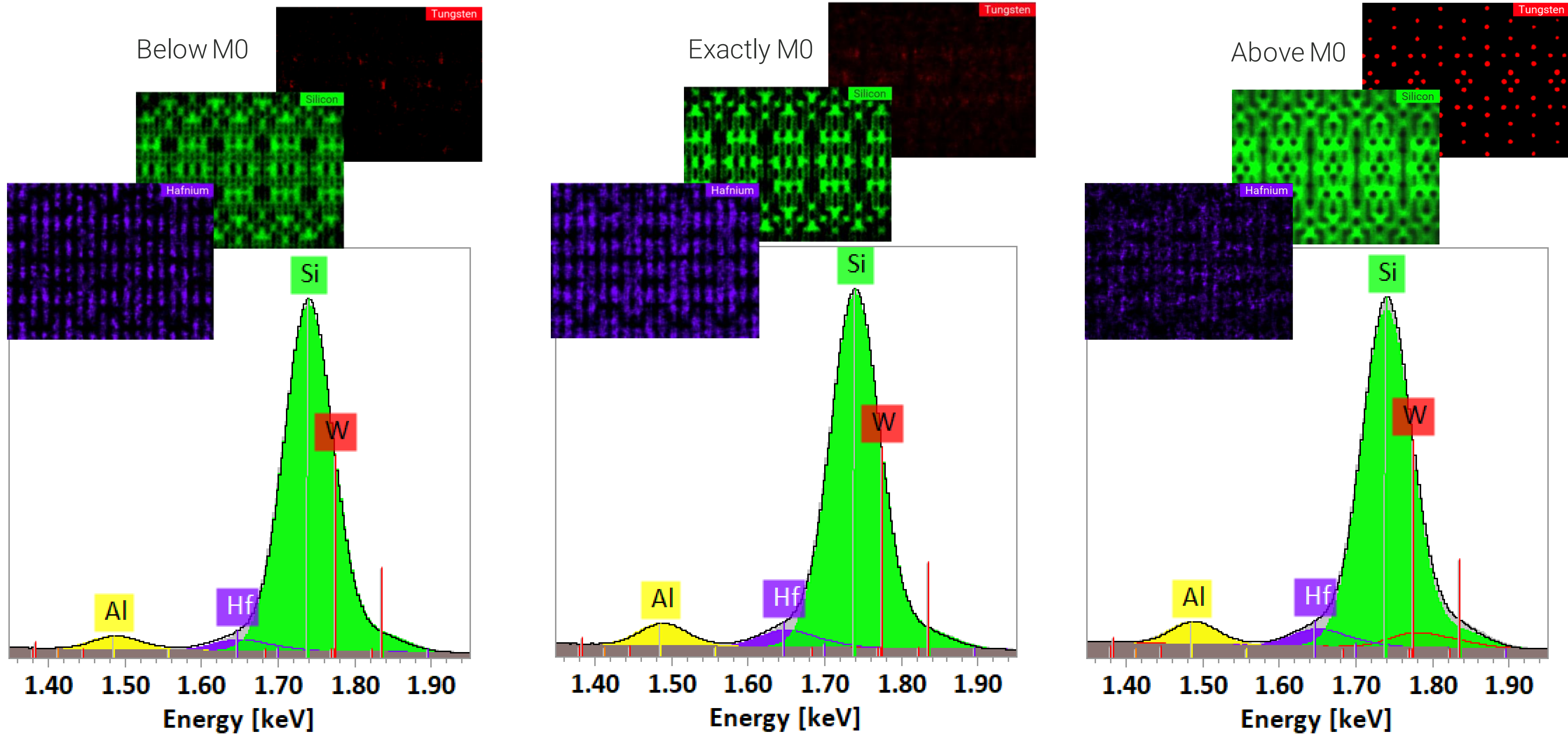
Automatic peak deconvolution



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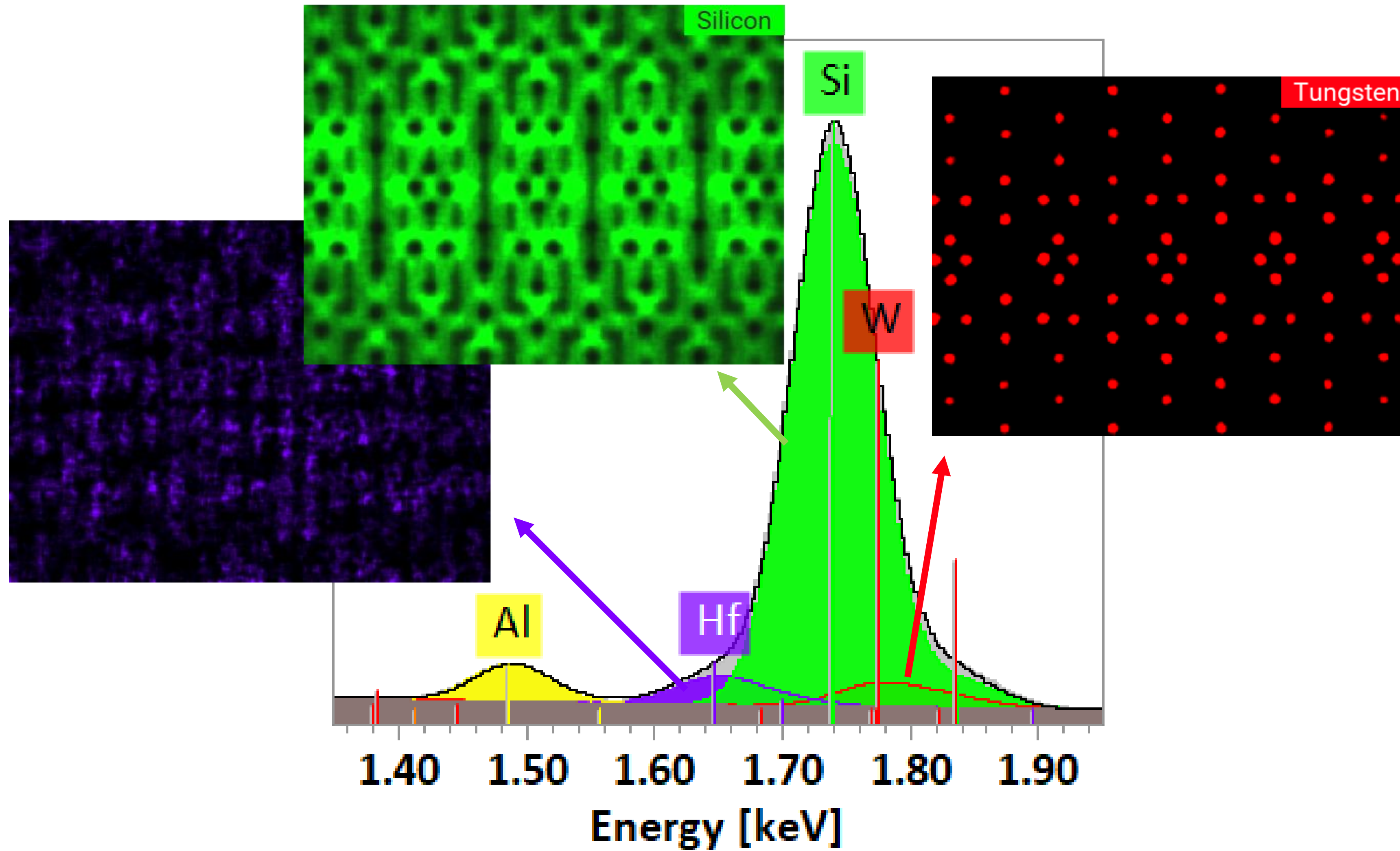
Automatic peak deconvolution – Above M0



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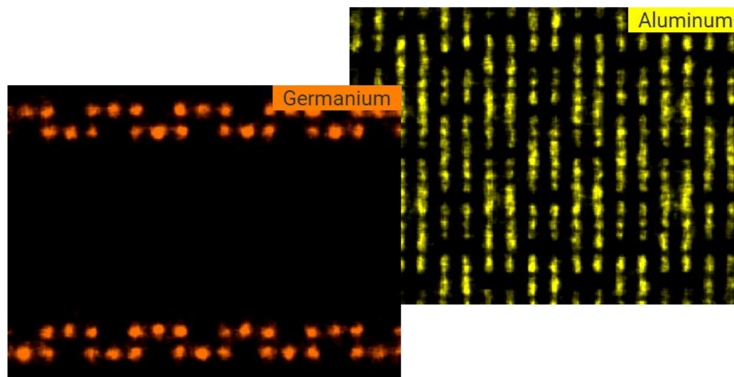
Measurement parameters

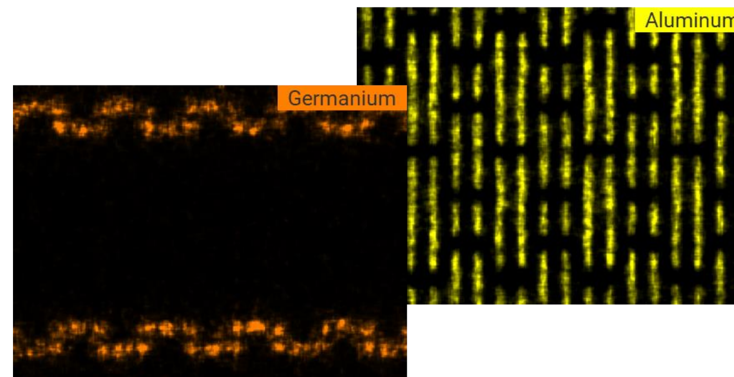
Below M0

EDS MEASUREMENT PARAMETERS	
Measurement time	60 min
Input Count rate	15500 cps
Probe current	380 pA
Dead time	18%
Total counts	4.6E+7 (46M)
H-FOV	1000 nm
Map size	300 x 225 px



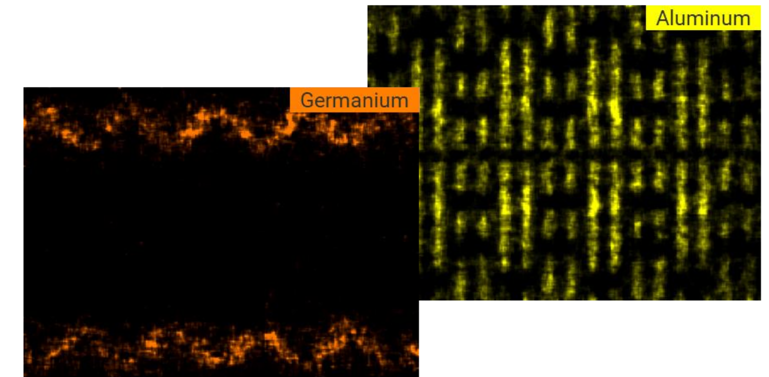
Exactly M0

EDS MEASUREMENT PARAMETERS	
Measurement time	55 min
Input Count rate	15200 cps
Probe current	350 pA
Dead time	17%
Total counts	4.2E+7 (42M)
H-FOV	1000 nm
Map size	300 x 225 px



Above M0

EDS MEASUREMENT PARAMETERS	
Measurement time	60 min
Input Count rate	15600 cps
Probe current	390 pA
Dead time	18%
Total counts	4.6E+7 (46M)
H-FOV	1000 nm
Map size	300 x 225 px



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20 kV, 300kx MAG, Above M0

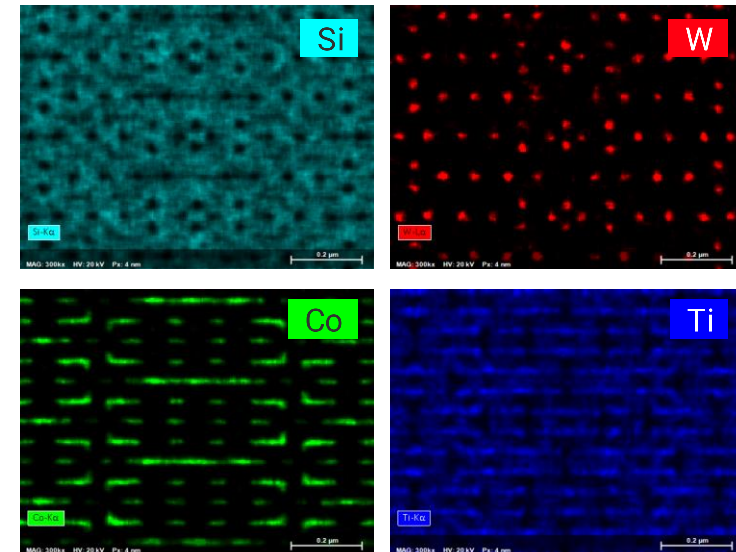
Ch 1 | Co-Kα | Ti-Kα | W-Lα

MAG: 300kx HV: 20 kV Px: 4 nm 0.2 µm

200 nm

EDS MEASUREMENT PARAMETERS

Measurement time	30 min
Input Count rate	50700 cps
Probe current	290 pA
Dead time	27%
Total counts	6.6E+7 (66M)
H-FOV	1000 nm
Map size	250 x 187 px



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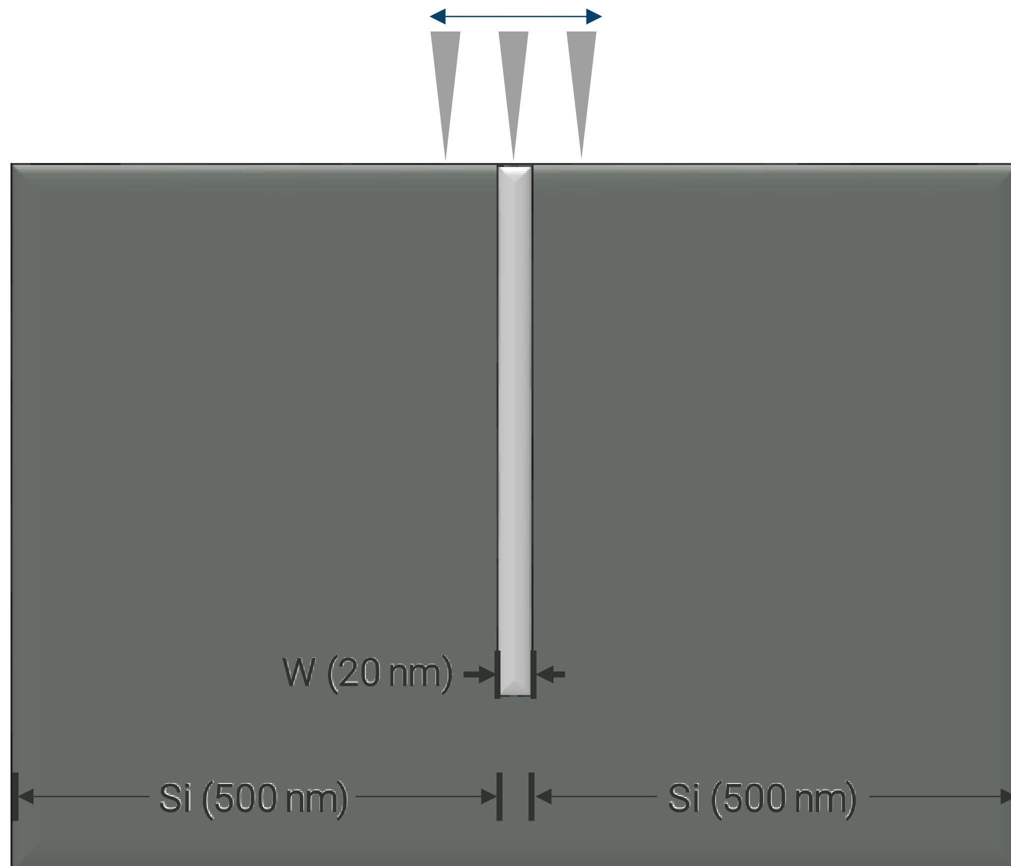
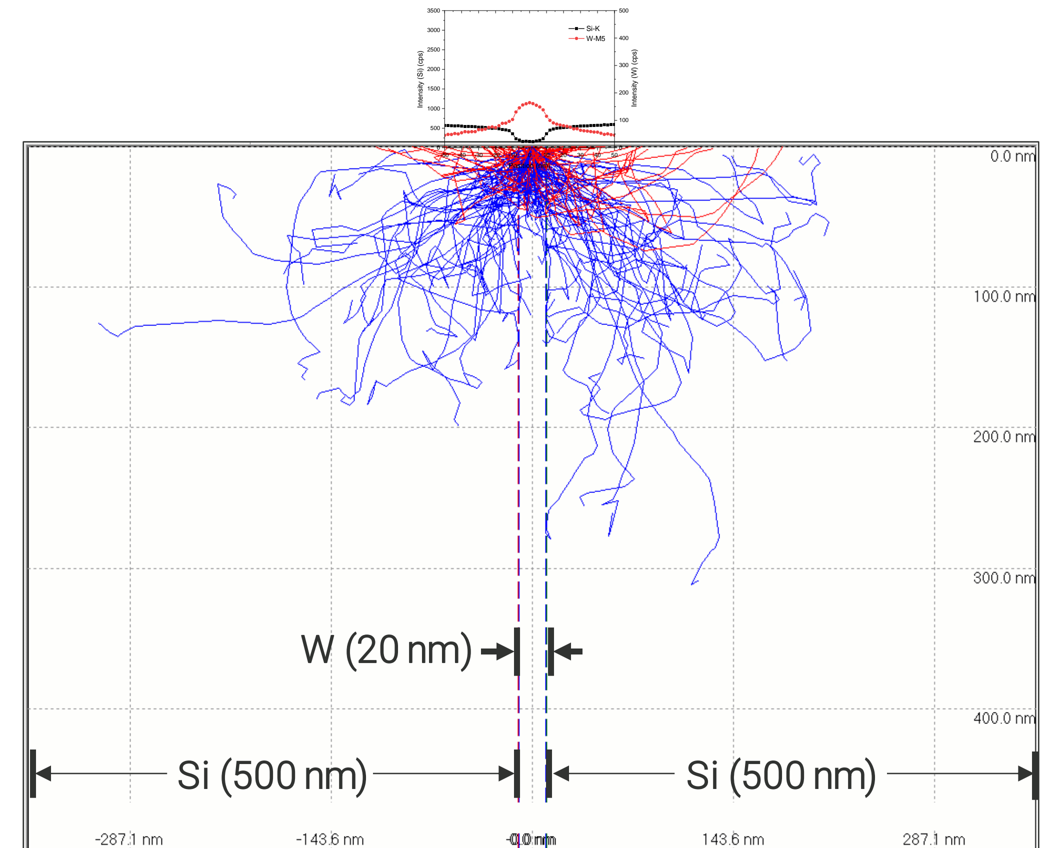
Interaction volume generation Si – W – Si

- 5 kV beam scanning across a 20 nm tungsten (W) layer in Silicon (Si)





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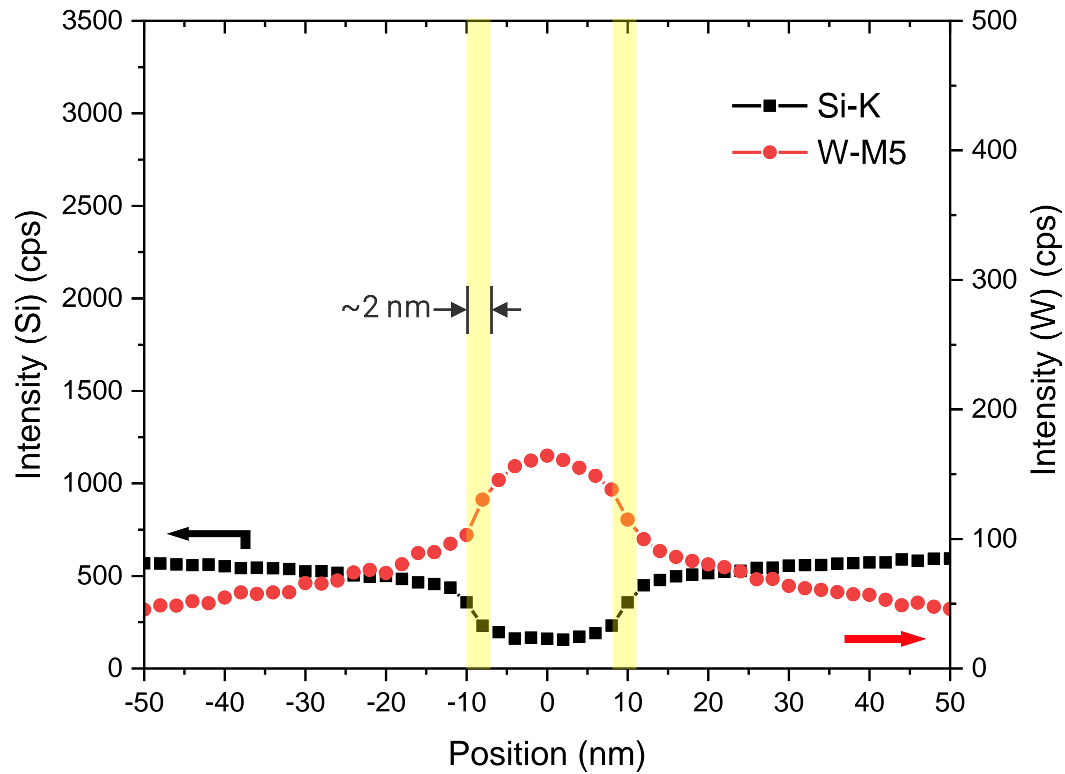
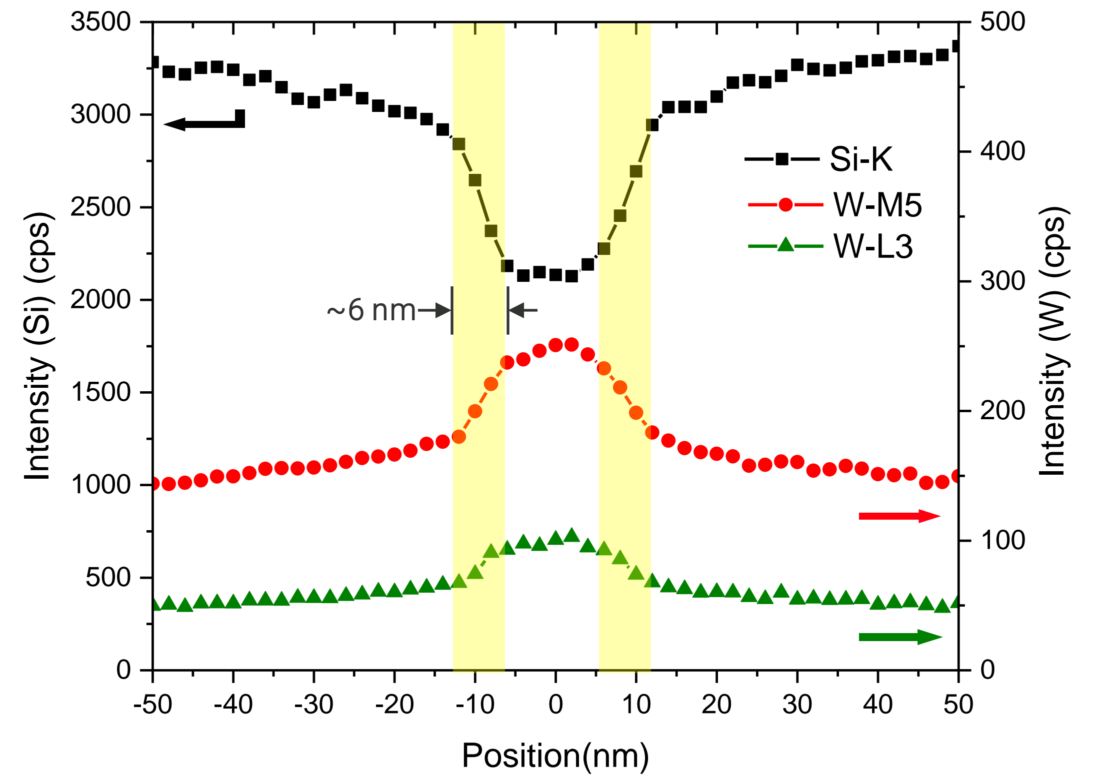
Intensity of X rays generated across Si – W – Si

5 kV



20 kV



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20 kV, 750kx MAG, Above M0

Ch 1 Co-Kα Ti-Kα W-Lα

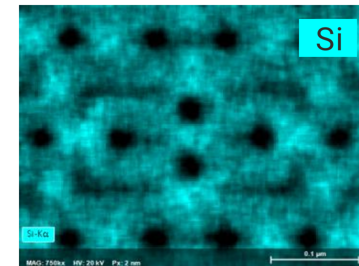
MAG: 750kx HV: 20 kV Px: 2 nm

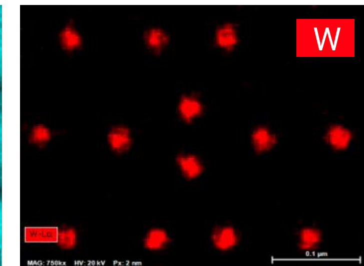
0.1 µm

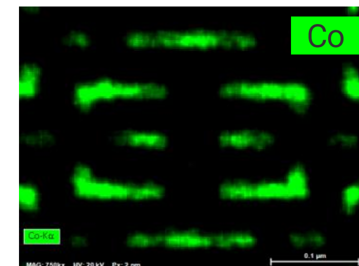
100 nm

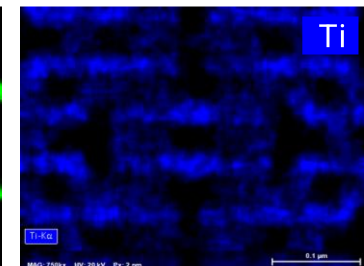
EDS MEASUREMENT PARAMETERS

Parameter	Value
Measurement time	21 min
Input Count rate	50500 cps
Probe current	220 pA
Dead time	27%
Total counts	3.7E+7 (37M)
H-FOV	500 nm
Map size	250 x 187 px









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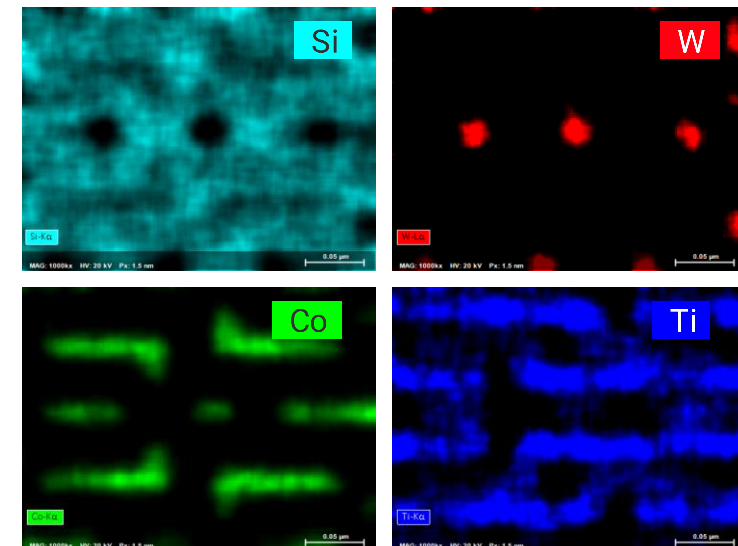
20 kV, 1Mx MAG, Above M0

Ch 1 Co-Kα Ti-Kα W-Lα
MAG: 1000kx HV: 20 kV Px: 1.5 nm
0.05 µm
50 nm

EDS MEASUREMENT PARAMETERS

Measurement time	577 s
Input Count rate	50900 cps
Probe current	210 pA
Dead time	27%
Total counts	2.1E+7 (27M)
H-FOV	300 nm
Map size	200 x 150 px



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5 kV, 1Mx MAG, Below M0

Ch 1 Co-L Al-K Si-Kα Ge-L

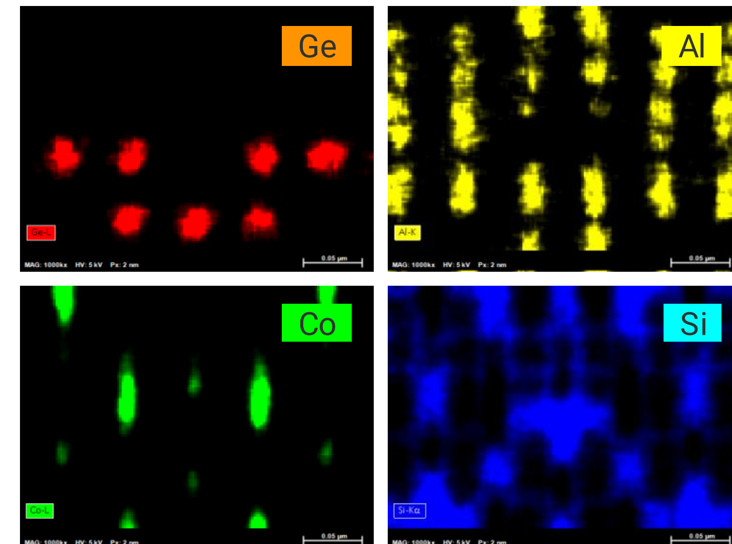
MAG: 1000kx HV: 5 kV Px: 2 nm

0.05 µm

50 nm

EDS MEASUREMENT PARAMETERS

Measurement time	545 s
Input Count rate	15600 cps
Probe current	390 pA
Dead time	18%
Total counts	7E+6 (7M)
H-FOV	300 nm
Map size	200 x 150 px



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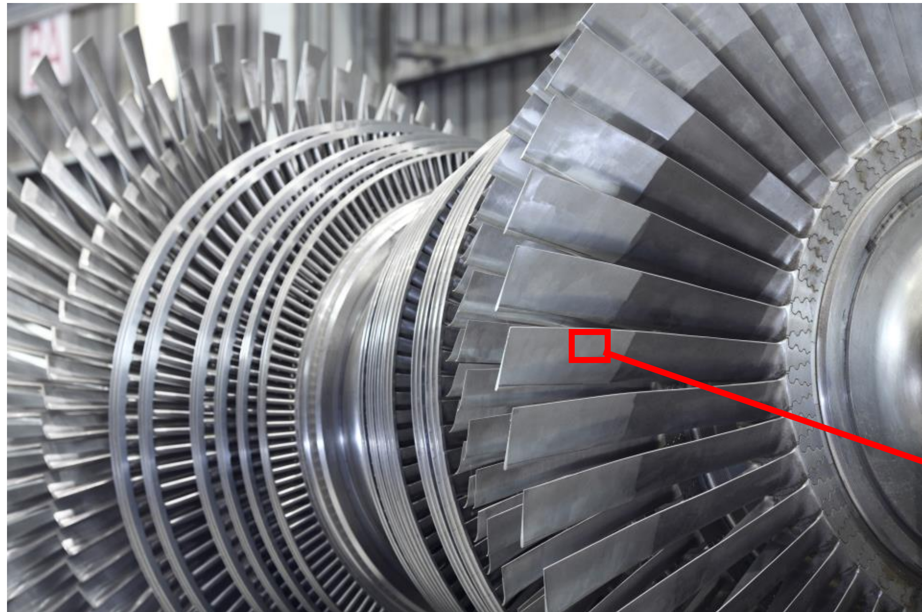
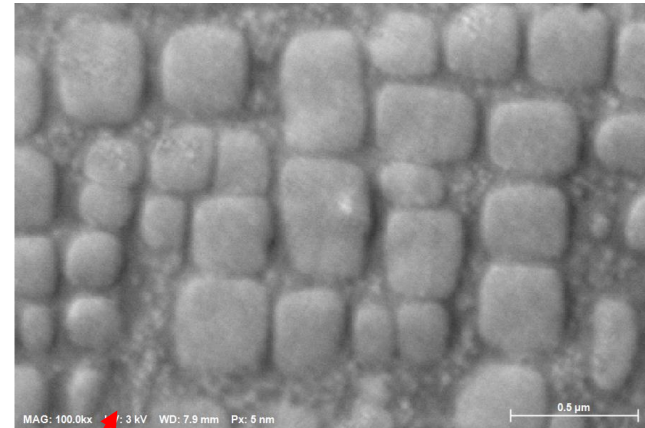
測定例2: Ni-based single crystal superalloy





100,000x

500 nm

10 mm

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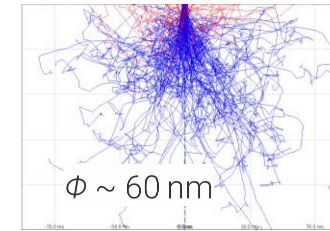
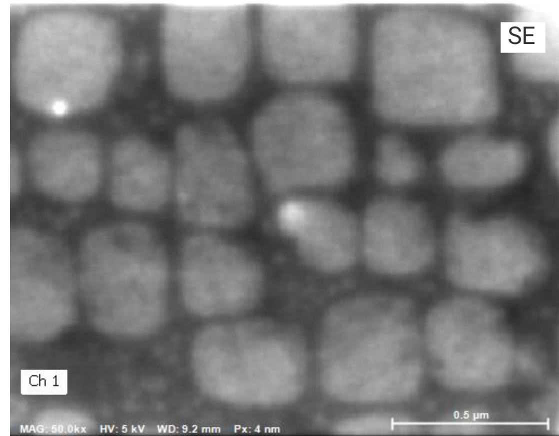
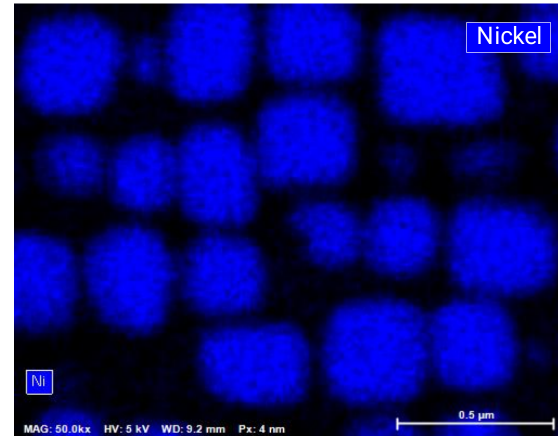
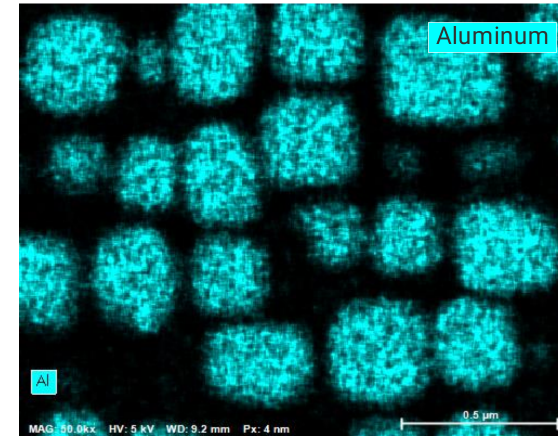
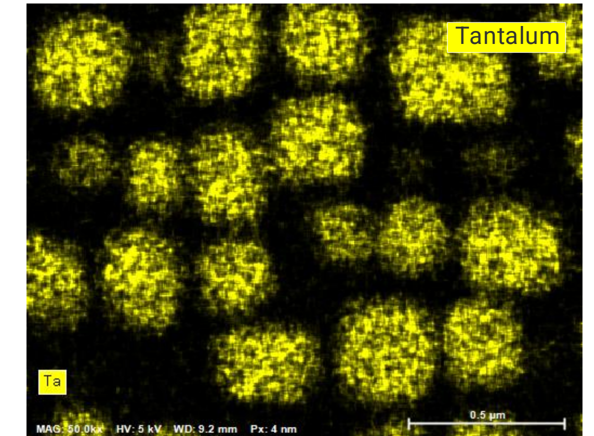
5 kV measurement at 50,000x magnification

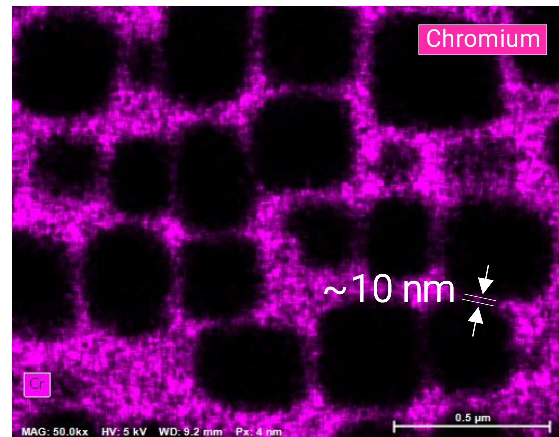


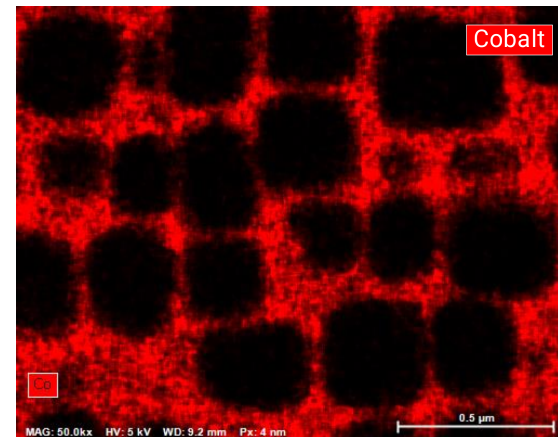


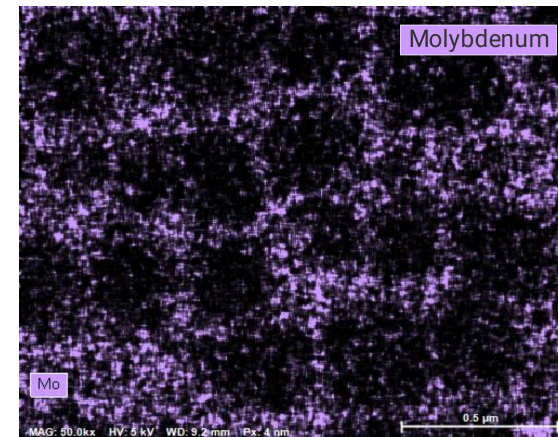


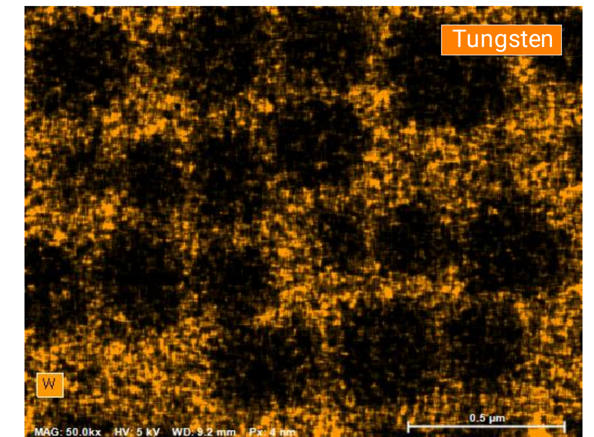












MAG: 50,000x HV: 5 kV

500 nm

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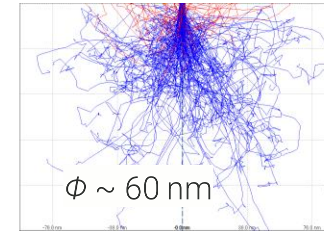
5 kV measurement at 50,000x magnification



cps/eV
Energy [keV]
Tantalum
Tungsten

EDS MEASUREMENT PARAMETERS

Measurement time	15 min
Input Count rate	39,000 cps
Dead time	23%
Total counts	3.6E+7 (36M)
Magnification	50,000 x (FOV 2.4 µm)
Map size	600 x 450 px

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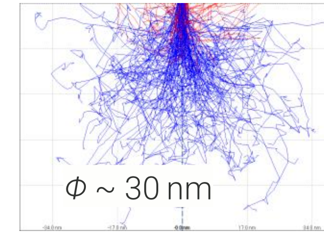
3 kV measurement at 50,000x magnification



cps/eV

Ni, Co, O, W, Ti, C, Cr, Ta, Mo, Al, Ta, W, Mo

Energy [keV]

Tantalum

Tungsten

cps/eV

Al, Ta, W

Energy [keV]

EDS MEASUREMENT PARAMETERS

Measurement time	35 min
Input Count rate	20,000 cps
Dead time	12%
Total counts	3.4E+7 (34M)
Magnification	50,000 x (FOV 2.4 µm)
Map size	500 x 375 px

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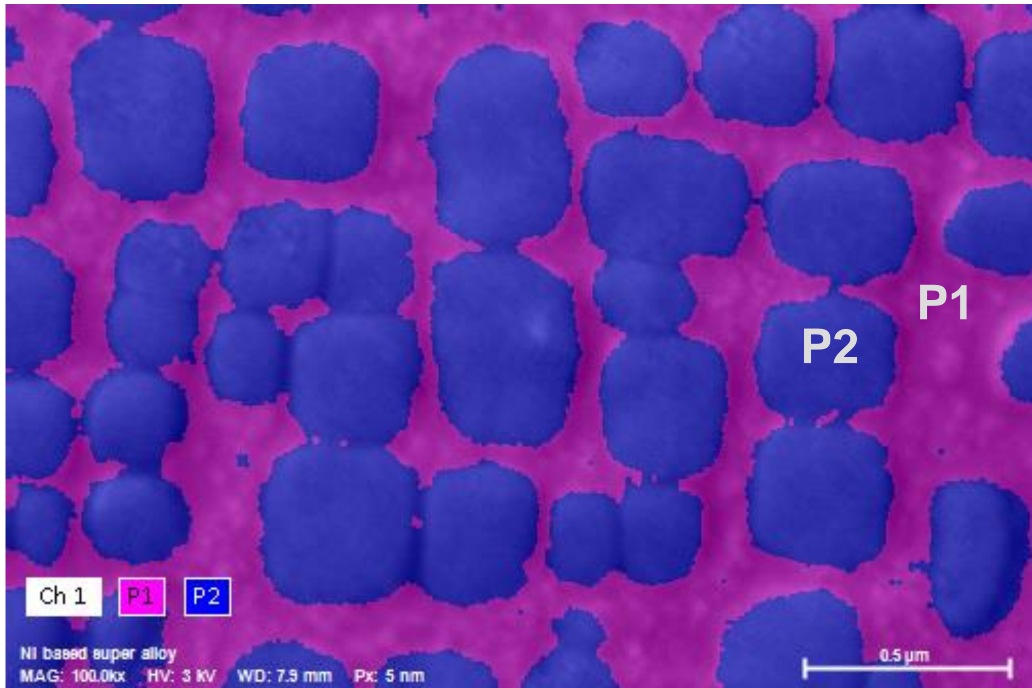
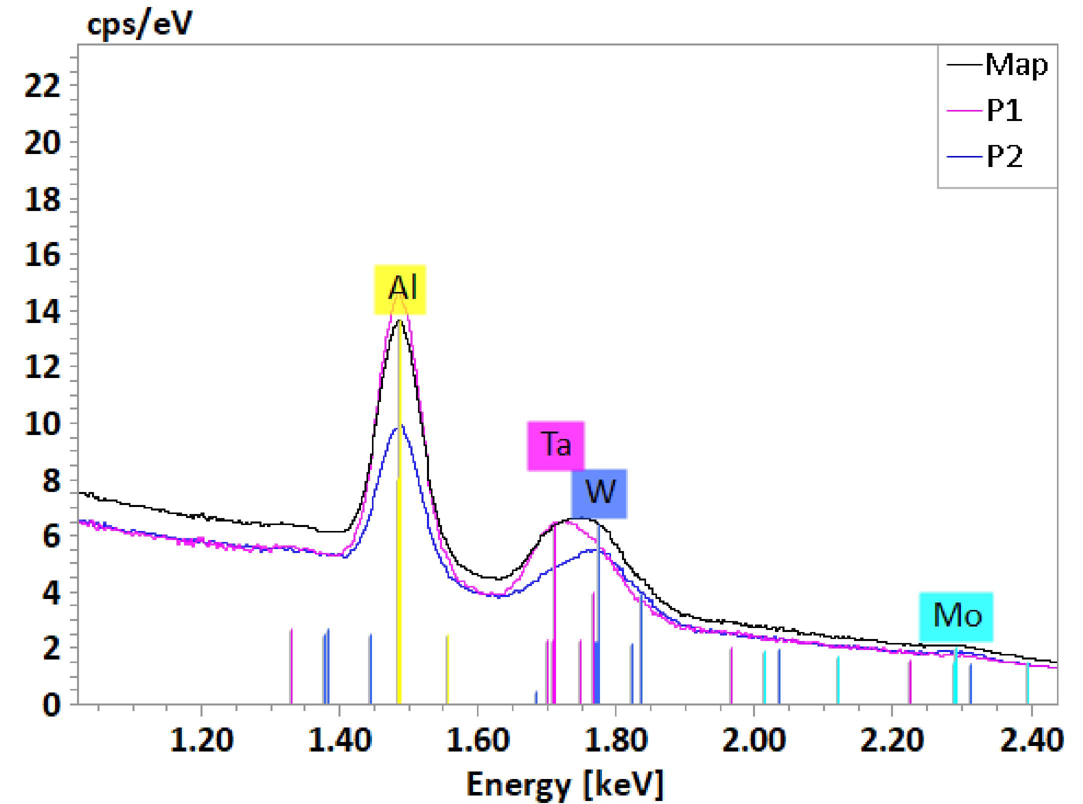
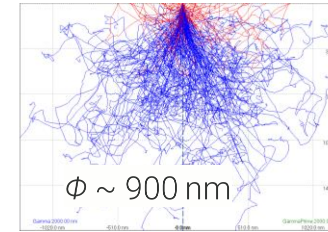
3 kV measurement at 100,000x magnification

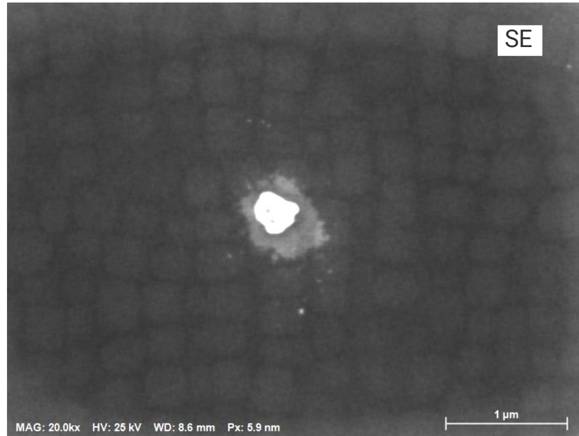


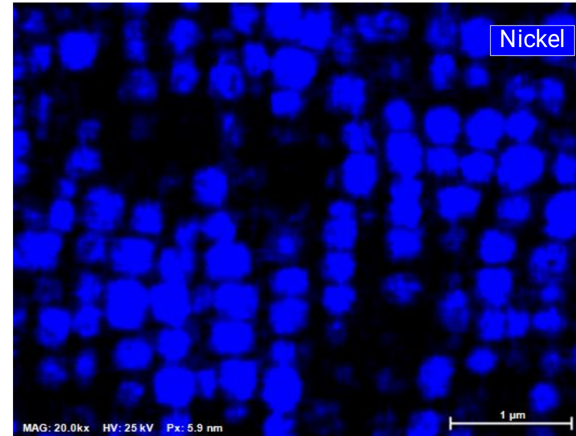


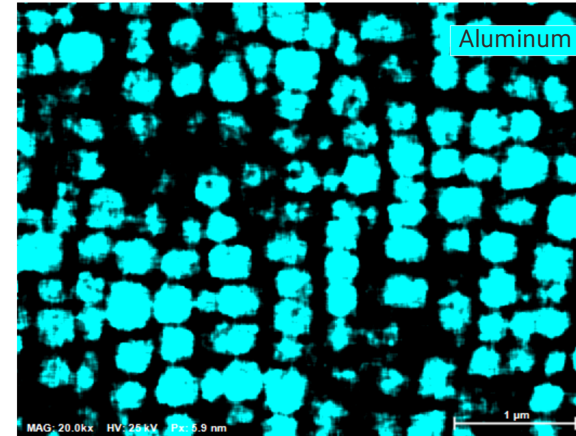
Gamma - P1
Gamma prime - P2

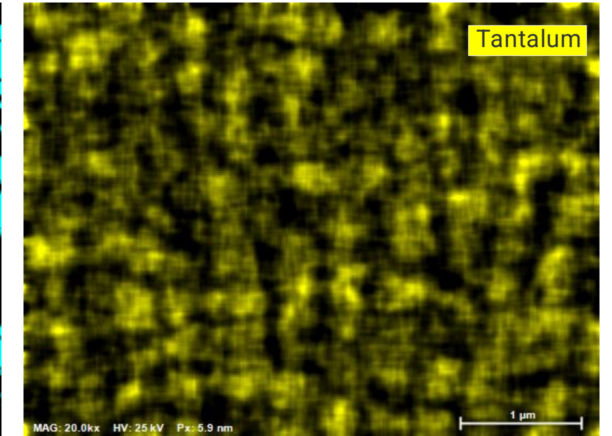
25 kV measurement at 20,000x magnification

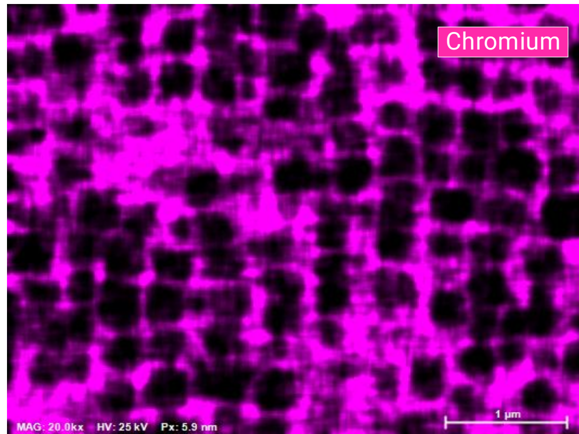


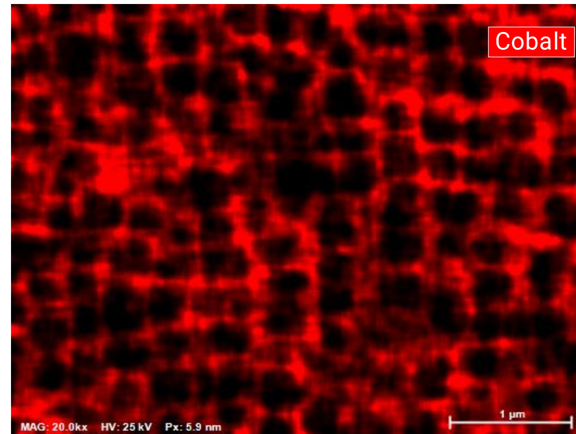


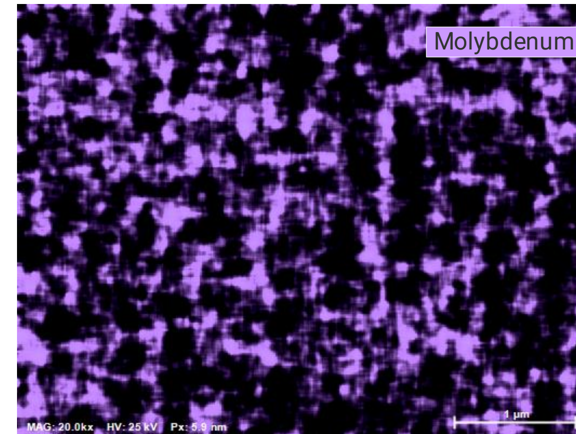


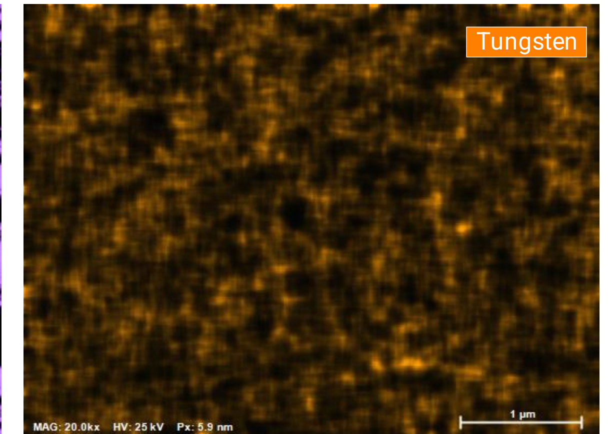




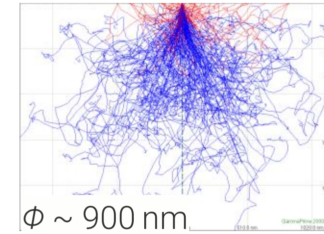








MAG: 20,000x HV: 25 kV 1 µm



25 kV measurement at 20,000x magnification

EDS MEASUREMENT PARAMETERS

Measurement time	30 min
Input Count rate	119,000 cps
Dead time	32%
Total counts	1.46E+8 (146M)
Magnification	20,000 x
Map size	1000 x 750 px

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Detector used?

EDS/SEM SYSTEM CONFIGURATION

Detector type	Bruker 7th Gen XFlash
Resolution	126 eV @Mn Kα
Window	SLEW AP3.3

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Thank you!

Name
Email or phone number

Q&A

ご質問がある場合は

Q&A boxにご質問を入力して、送信ボタンをクリックしてください。

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