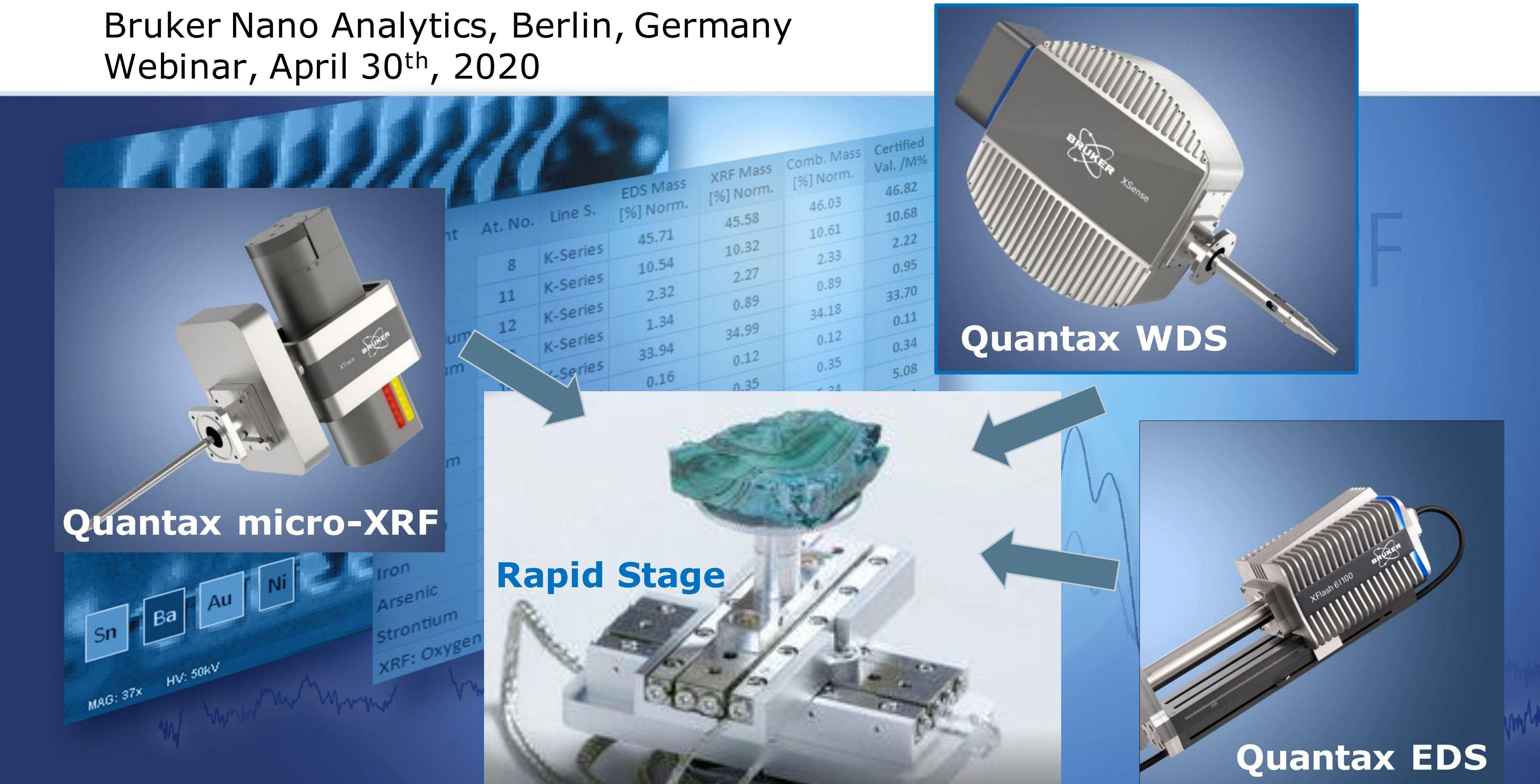
Large area SEM mapping using the Rapid Stage and its benefits for EDS, WDS and micro-XRF analysis

Bruker Nano Analytics, Berlin, Germany
Webinar, April 30th, 2020



Presenters

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Bruker Nano Analytics, Berlin, Germany

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Bruker Nano Analytics, Berlin, Germany

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Bruker Nano Analytics, Berlin, Germany

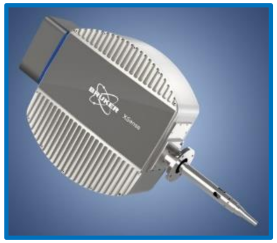
Overview

- Terminology
- Introduction to Rapid Stage
- Types of Large Area Mapping on a SEM and Potential Issues
- Differences between SEM-EDS / SEM-WDS / SEM-XRF - Rapid Stage Technical Description
- Example Applications and Benefits
- Summary and Conclusion

Introduction
Terminology

SEM-EDS: Analysis based on the sample interaction with an electron beam source from the SEM and the resultant X-rays that are detected using an EDS (simultaneous element detection)

SEM-WDS: Analysis based on the sample interaction with an electron beam source from the SEM and the resultant X-rays that are detected using a WDS (sequential element detection)



SEM-XRF-EDS: MicroXRF on SEM (XTrace): Analysis based on the sample interaction with an X-ray beam source from the Micro XRF attached to the SEM and the resultant X-rays that are detected using an EDS (simultaneous element detection)

Rapid Stage: New high speed stage with precision movement

SEM Stage: Standard stage that comes with the SEM

Analytical Parameters and Conditions
SEM-EDS vs SEM-WDS vs SEM-XRF

Parameter	EDS: E-beam (SEM-EDS)	WDS: E-beam (SEM-WDS)	EDS: Micro-XRF (SEM-XRF-EDS)
Analyzed Volume	Ø: few µm Information depth: µm; (depending primarily on electron energy)	Ø: few µm Information depth: µm; (depending primarily on electron energy)	Ø: 15-30 µm Information depth: µm to mm; (depending on analysed element and matrix)
Detectable Elements	Atomic number Z ≥ 4 (beryllium)	Atomic number Z ≥ 4 (beryllium)	Atomic number Z ≥ 6 (carbon)
Energy range	K- L –M – Lines (up to 20 keV)	70 eV – 3.6 keV (L- M- Lines)	K- L –M – Lines (up to 40 keV)
Concentration Range	Down to 1000 ppm	Down to 100 ppm	Down to 10 ppm
Quantification	Standard less and Standard based	Standard based	Standard less and standard based
Data collection	Simultaneously	Sequentially	Simultaneously
Sample Preparation	Sample needs to be electrically conductive (commonly carbon-coated), polishing required	Sample needs to be electrically conductive (commonly carbon-coated), polishing required	Electrical Conductivity not required, samples doesn´t need to be polished
Sample Stress	Heating due to absorbed electrons	Heating due to absorbed electrons	Minimal
Typical SEM beam current	Variable	Variable > 10 nA	N/A

Introduction
Historic and Current Webinars

www.bruker.com/events/webinars.html

Filter: EDS, WDS, EBSD, Micro-XRF on SEM

Application of Rapid Stage
Motivation

Why using Rapid Stage for SEM-EDS and SEM-WDS?

- Enhancing speed for mapping applications
- Enhancing EDS / WDS usability
- Enabling WDS mapping with vertical beam at the speed of a beammap
- Avoiding Bragg`s law violation
- Avoiding lateral intensity loss
- Enabling large scale mapping

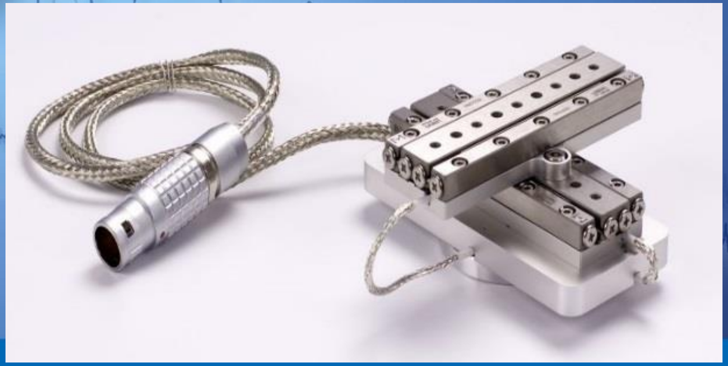
Why using Rapid Stage for SEM-XRF?

- Enhancing speed for mapping applications as only stage mapping is possible as the X-ray beam is fixed in space and can not be rastered

Rapid Stage for SEM´s
Introduction

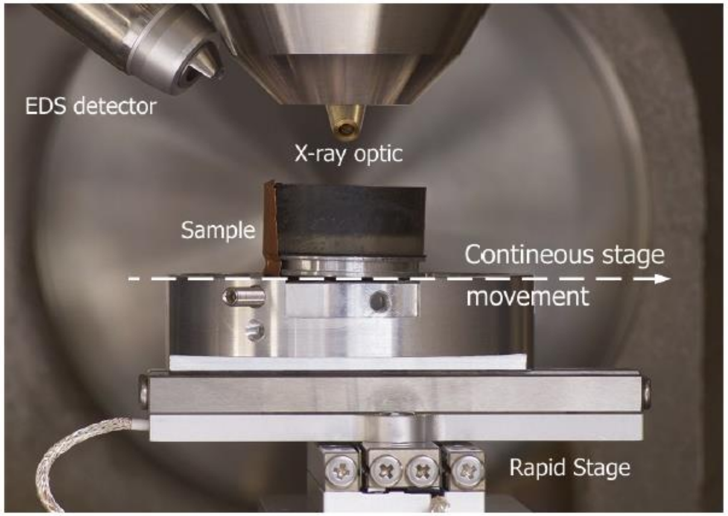
Element	At. No.	Line S.	EDS Mass [%] Norm.	XRF Mass [%] Norm.	Comb. Mass [%] Norm.	Certified Val. /M%
Oxygen	8	K-Series	45.71	45.58	46.03	46.82
Sodium	11	K-Series	10.54	10.32	10.61	10.68
Magnesium	12	K-Series	2.32	2.27	2.33	2.22
Aluminium	13	K-Series	1.34	0.89	0.89	0.95
Silicon	14	K-Series	33.94	34.99	34.18	33.70
Sulfur	16	K-Series	0.16	0.12	0.12	0.11
Potassium	19	K-Series	0.37	0.35	0.35	0.34
Calcium	20	K-Series	5.30	5.34	5.34	5.08
Titanium	22	K-Series	0.00	0.01	0.01	0.01
Iron	26	K-Series	0.28	0.03	0.03	0.03
Arsenic	33	K-Series	0.04	0.06	0.06	0.04
Strontium	38	L-Series	0.00	0.04	0.04	–

XRF: Oxygen quantification by stoichometry



Introduction
Rapid Stage

- The **Rapid Stage** has been developed to enable high-speed mapping over large areas via Stage movement
- It is mounted on top of an existing SEM stage, including stage adaption and sample holder.
- The Rapid Stage is controlled independently from the SEM stage and can operate up to a maximum travel speed of 4 mm/s.

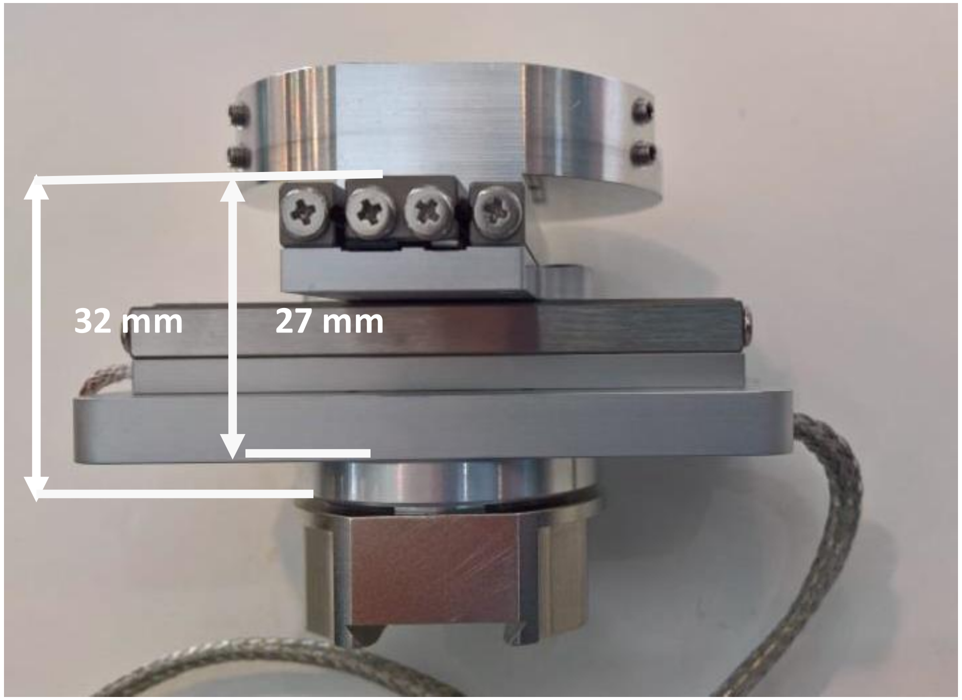


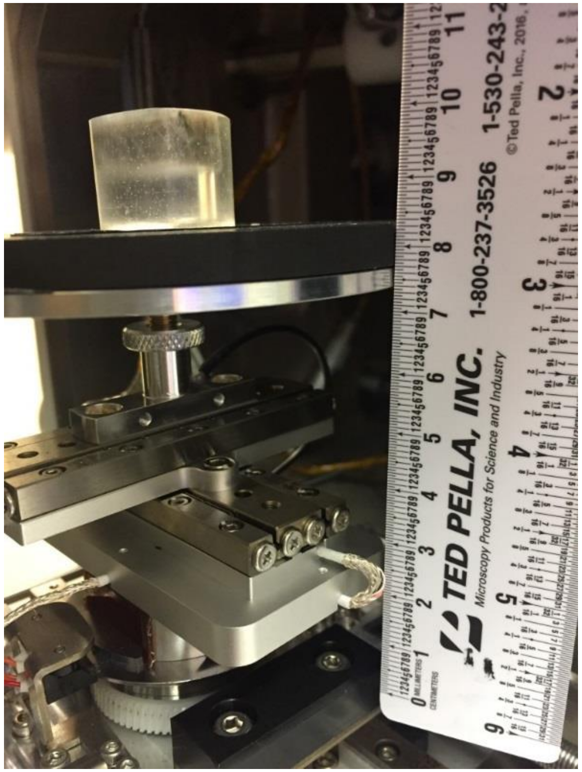
Rapid Stage Dimensions

Stage itself (including x- and y linear positioners and basis plate): **27 mm**

Including footplate: **32 mm**

Without dovetail and sample holder → SEM depending

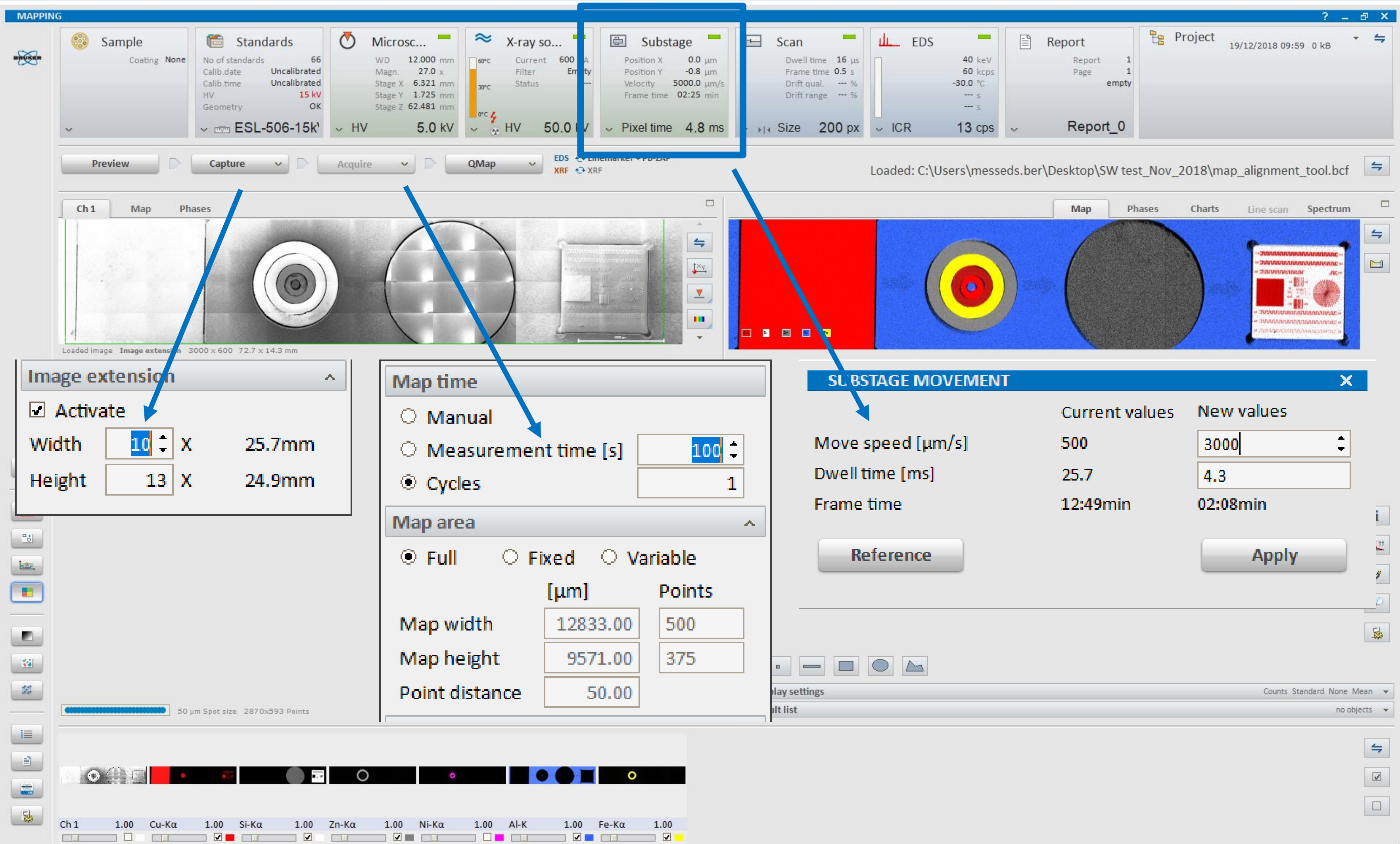




for Jeol IT 500 setup: **60 mm**

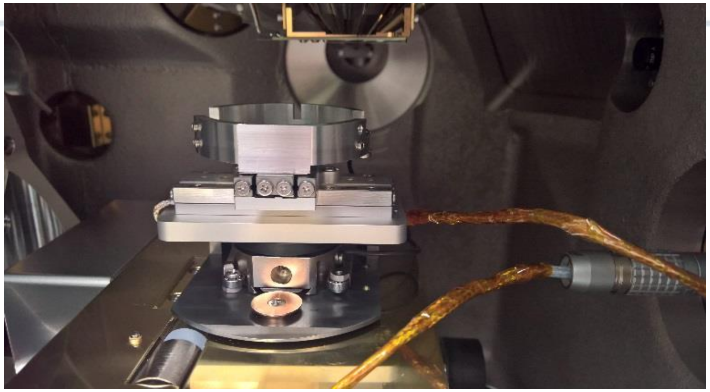
for Hitachi S3700N setup: **73 mm**

Rapid Stage Integration in ESPRIT

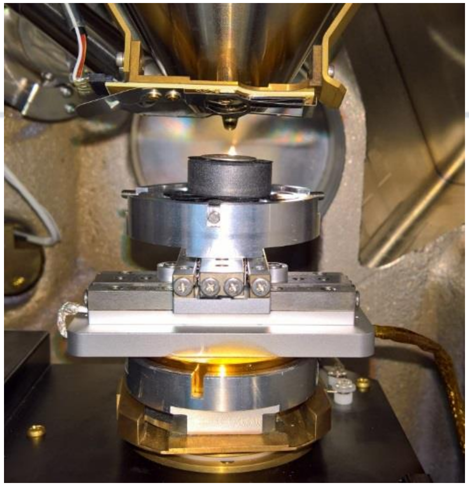


Introduction Video
Rapid Stage

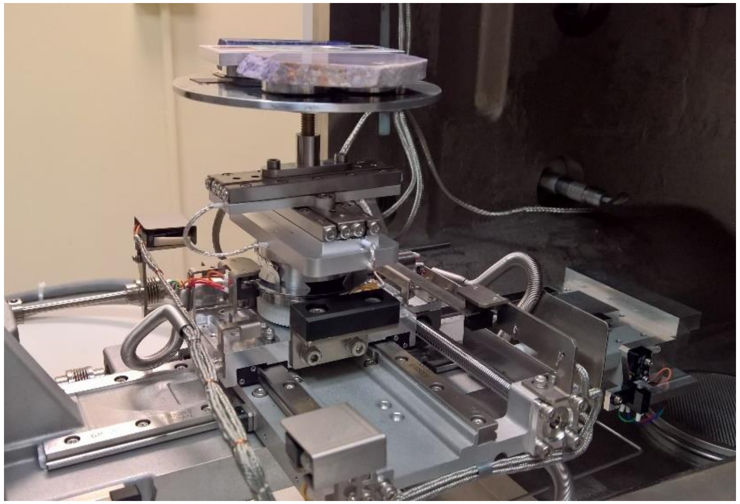
Rapid Stage Installations:
Adaptable to various SEMs



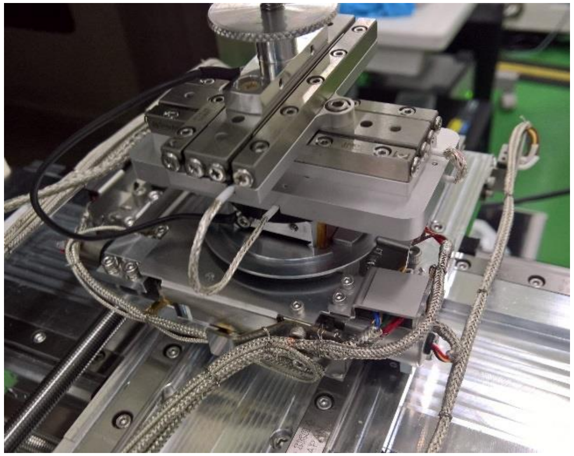
Jeol IT-500



Jeol JSM 6490



Hitachi S 3700N

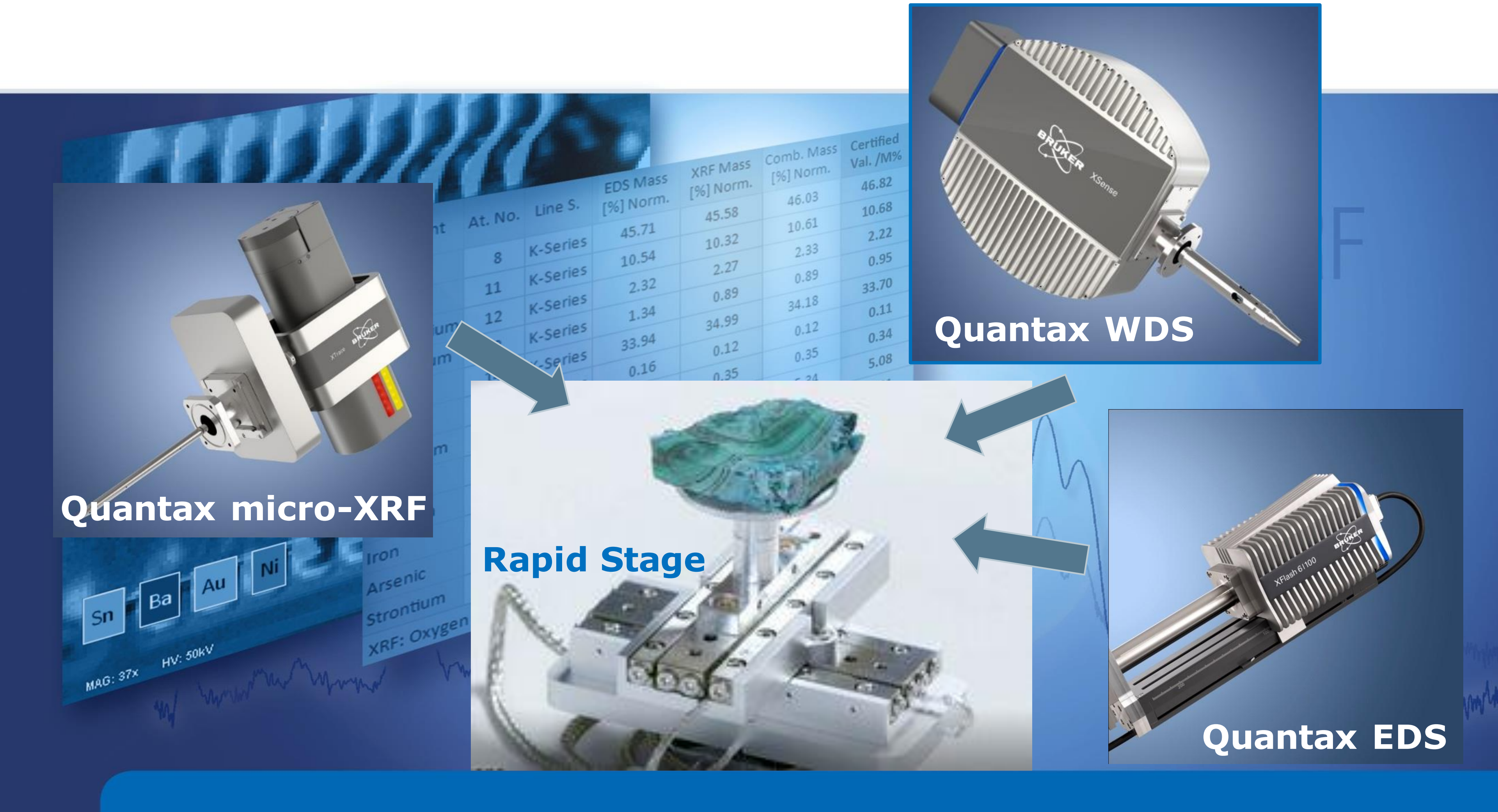


Hitachi SU 3900

Rapid Stage
Specification

Parameter	Description
Height	27 mm (without sample holder and SEM stage adaption)
Weight	300 g
Sample load	3 kg
Stage travel speed	4 mm /sec
Travel distance	50 mm
Vacuum resistance	10^{-6} mbar (higher vacuum resistance on request)
Resolution	< 1 nm

Examples



Rapid Stage
Examples - Settings

- **Large Area Maps Overview: Mapping Types**

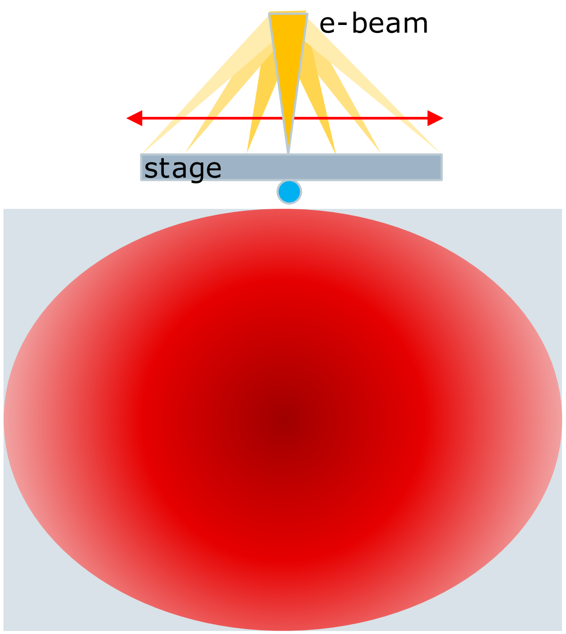
- **Source: Electron Beam (e-beam) and X-ray beam**
- **Detector: EDS and WDS**
- **Stage: SEM-Stage and Rapid Stage**

- **Note: Combination (Simultaneous) data-sets**
  - **Rapid Stage with e-beam + X-ray beam + EDS, or**
  - **Rapid Stage with e-beam + EDS and WDS**

Large Area Mapping

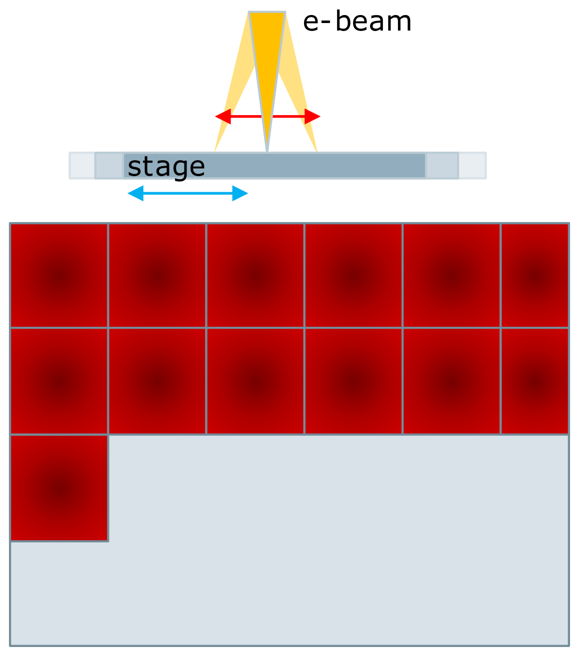
3 Different Mapping Modes

Type I



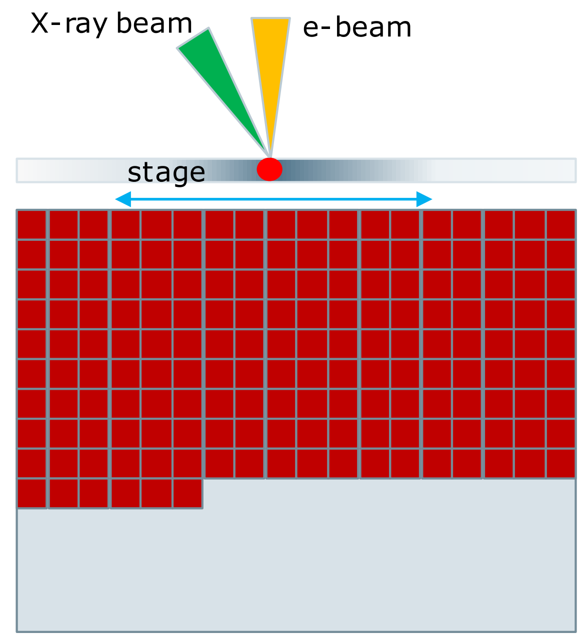
Scan Map Without Limits
(Raster over Full Area)
e.g. Low Mag. 30x

Type II



Scan Map With Limits
(Raster over Restricted Area
with Stage Movement)

Type III

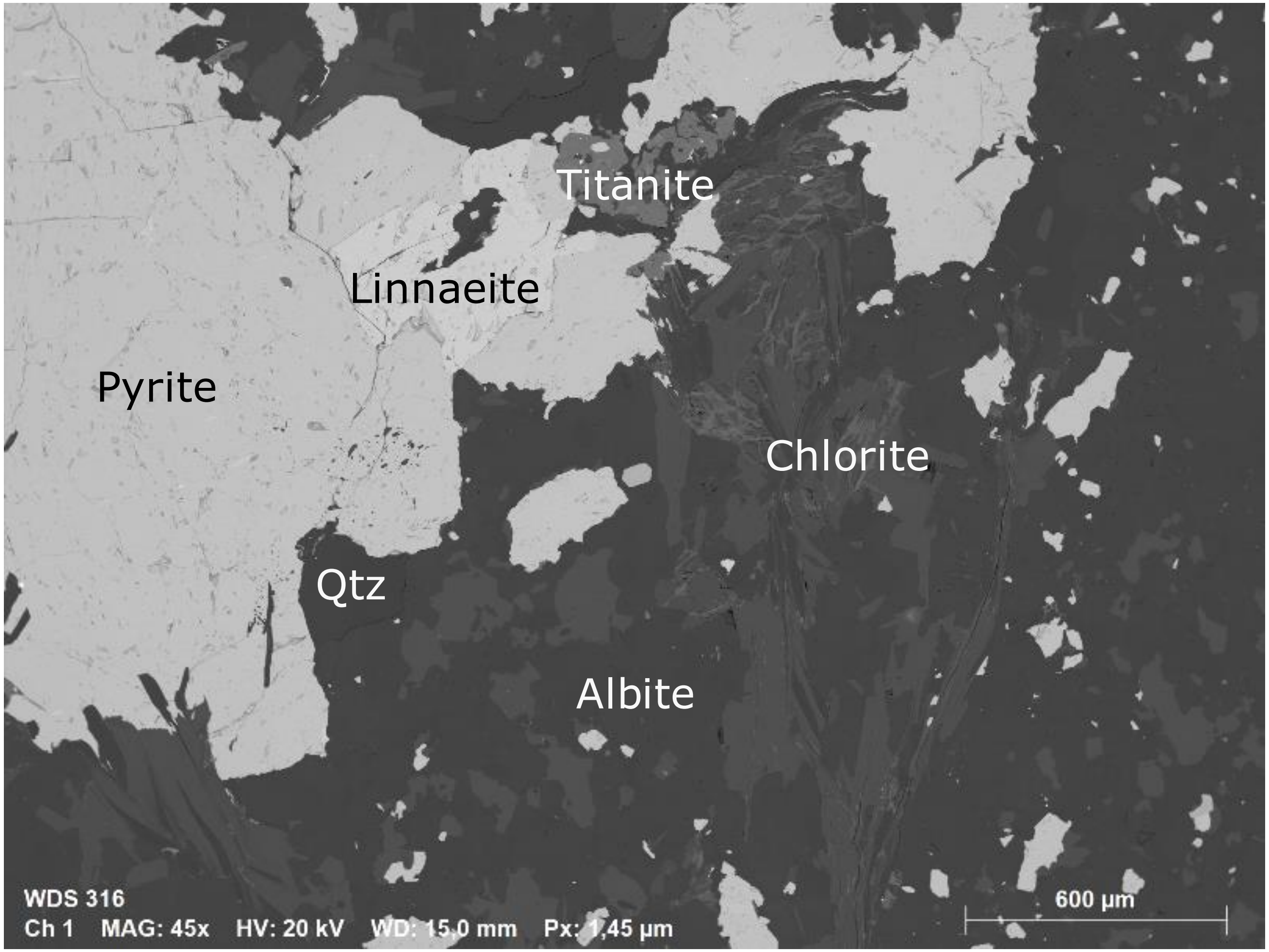


Stage Map
Independent of Magnification

Increasing accuracy - WDS

Increasing speed – SEM Stage

Sample 1
Sulphide bearing rock



Low magnification

45x

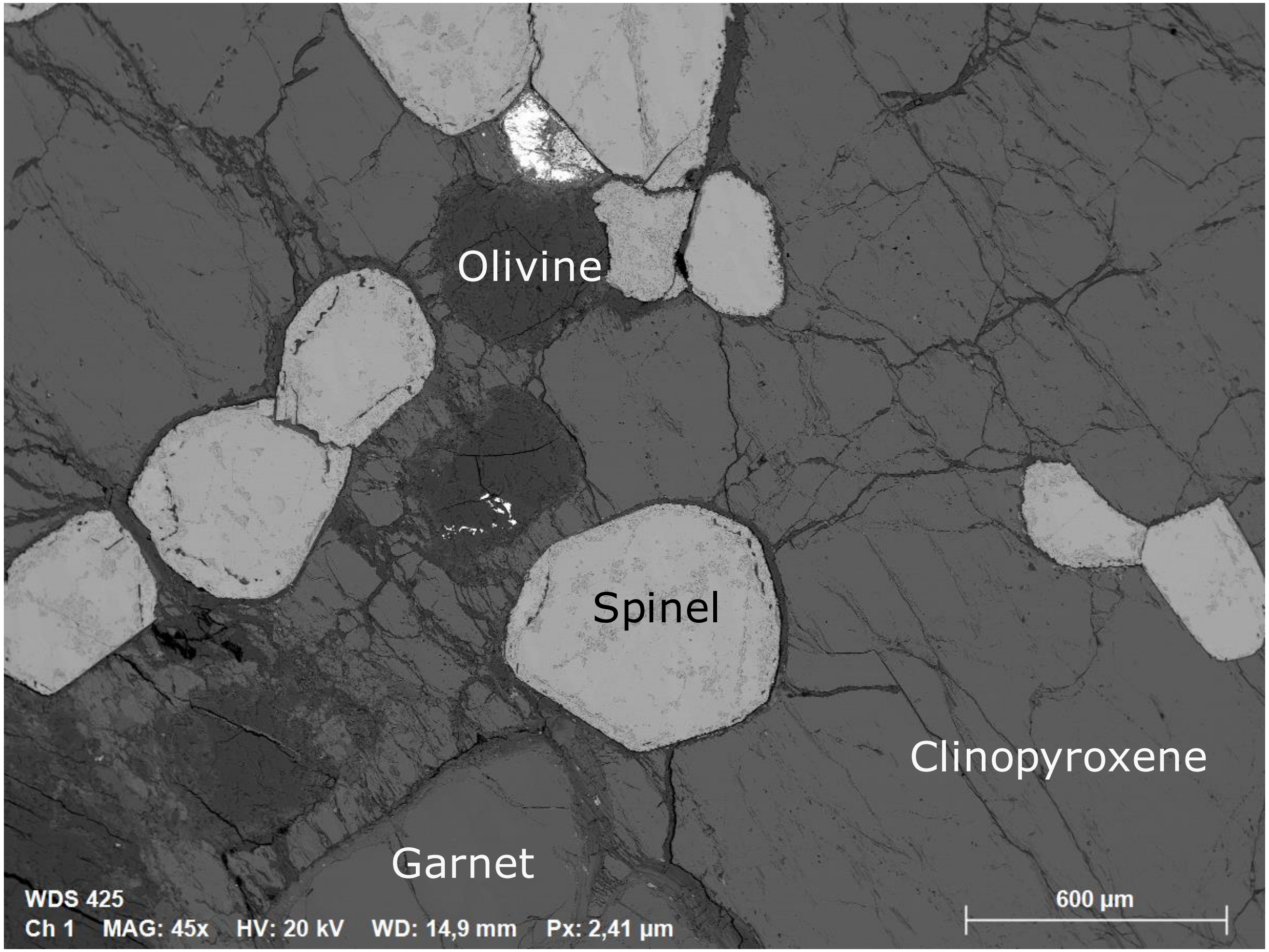
Image dimensions:

2.9 x 2.2 mm

Resolution:

2000 x 1500 px (1.45 µm / px)

Sample 2
Garnet-Spinel Peridotite



Low magnification

45x

Image dimensions:

2.9 x 2.2 mm

Resolution:

1200 x 900 px

(2.4 µm / px)

Sample 3
Electronics Microchip

Low magnification

30x

Image dimensions:

4.3 x 3.2 mm

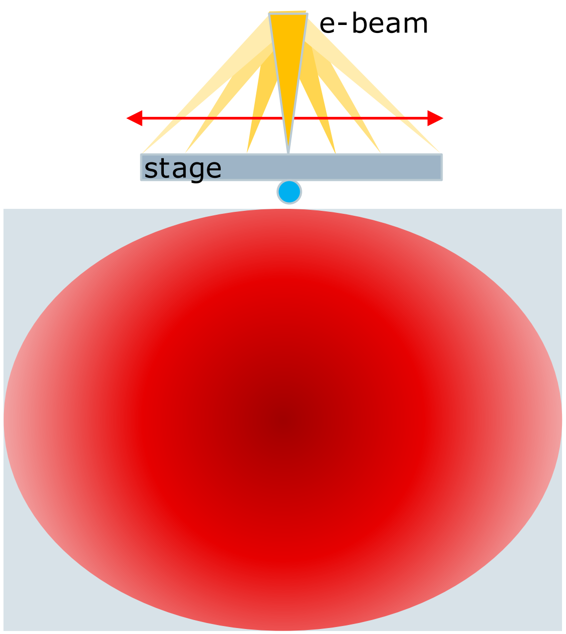
Resolution:

1200 x 900 px

(3.6 µm / px)

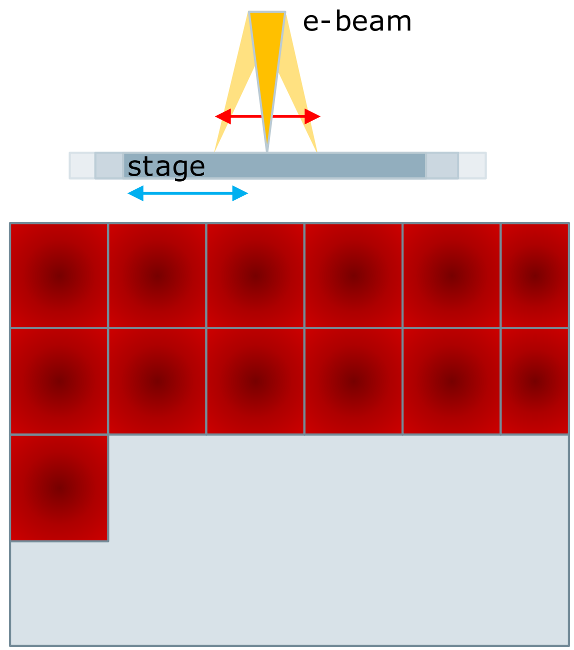
Large Area Mapping
Type I

Type I



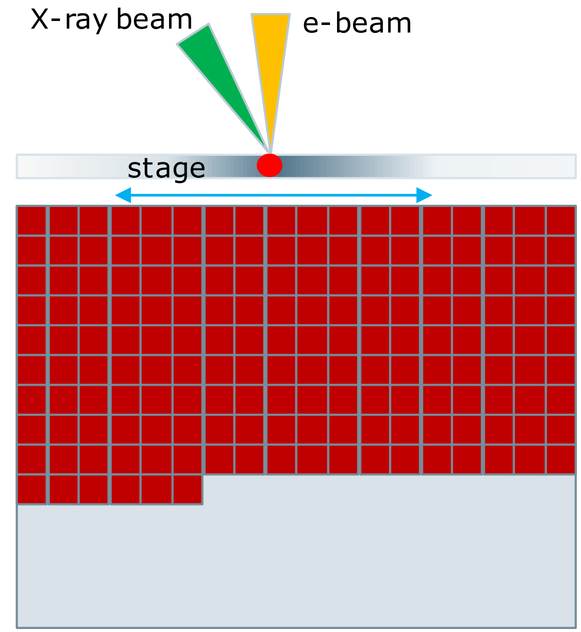
Scan Map Without Limits
(Raster over Full Area)
e.g. Low Mag. 30x

Type II



Scan Map With Limits
(Raster over Restricted Area
with Stage Movement)

Type III



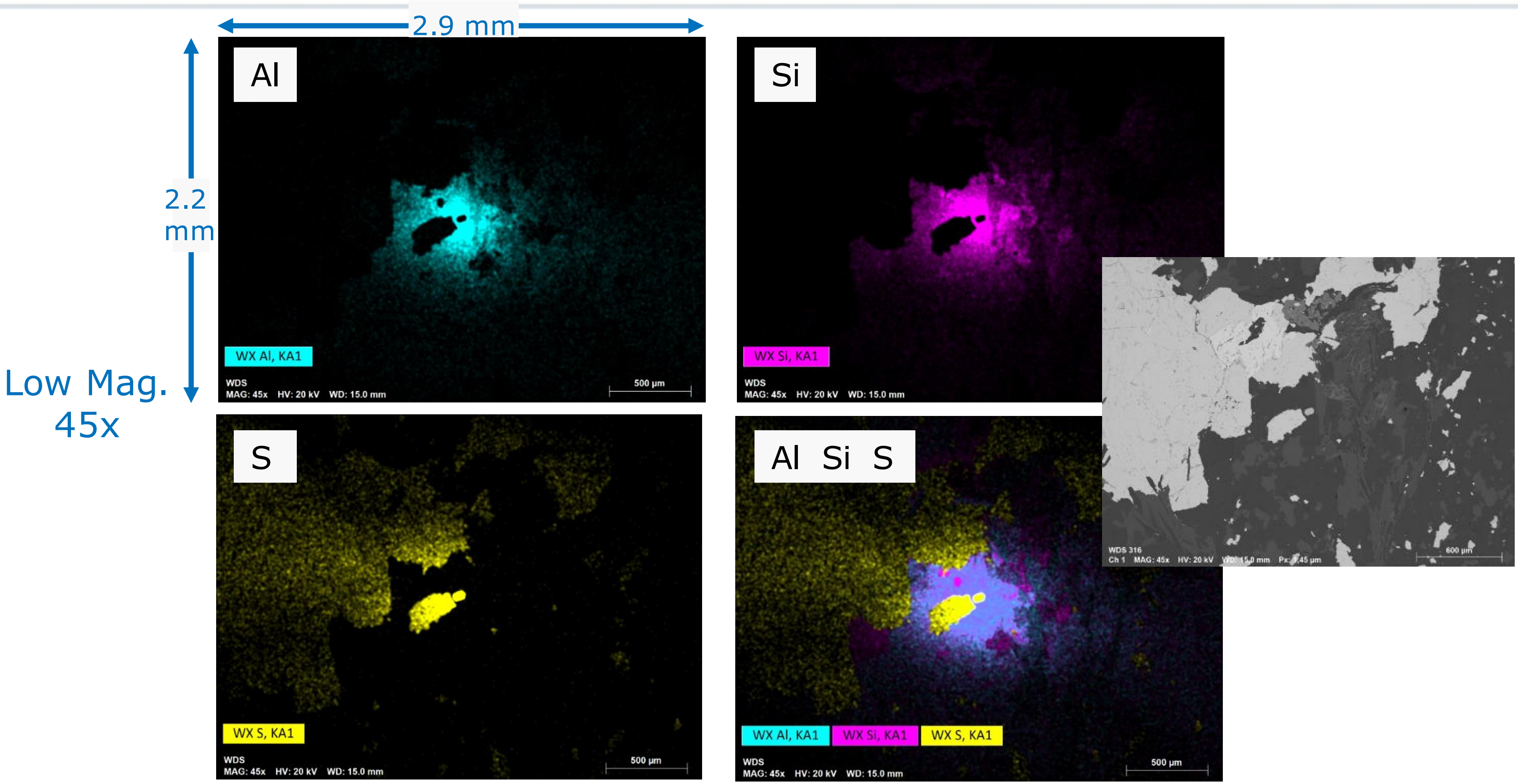
Stage Map
Independent of Magnification

Increasing accuracy - WDS

Increasing speed – SEM Stage

Scan Map Without Range Limits

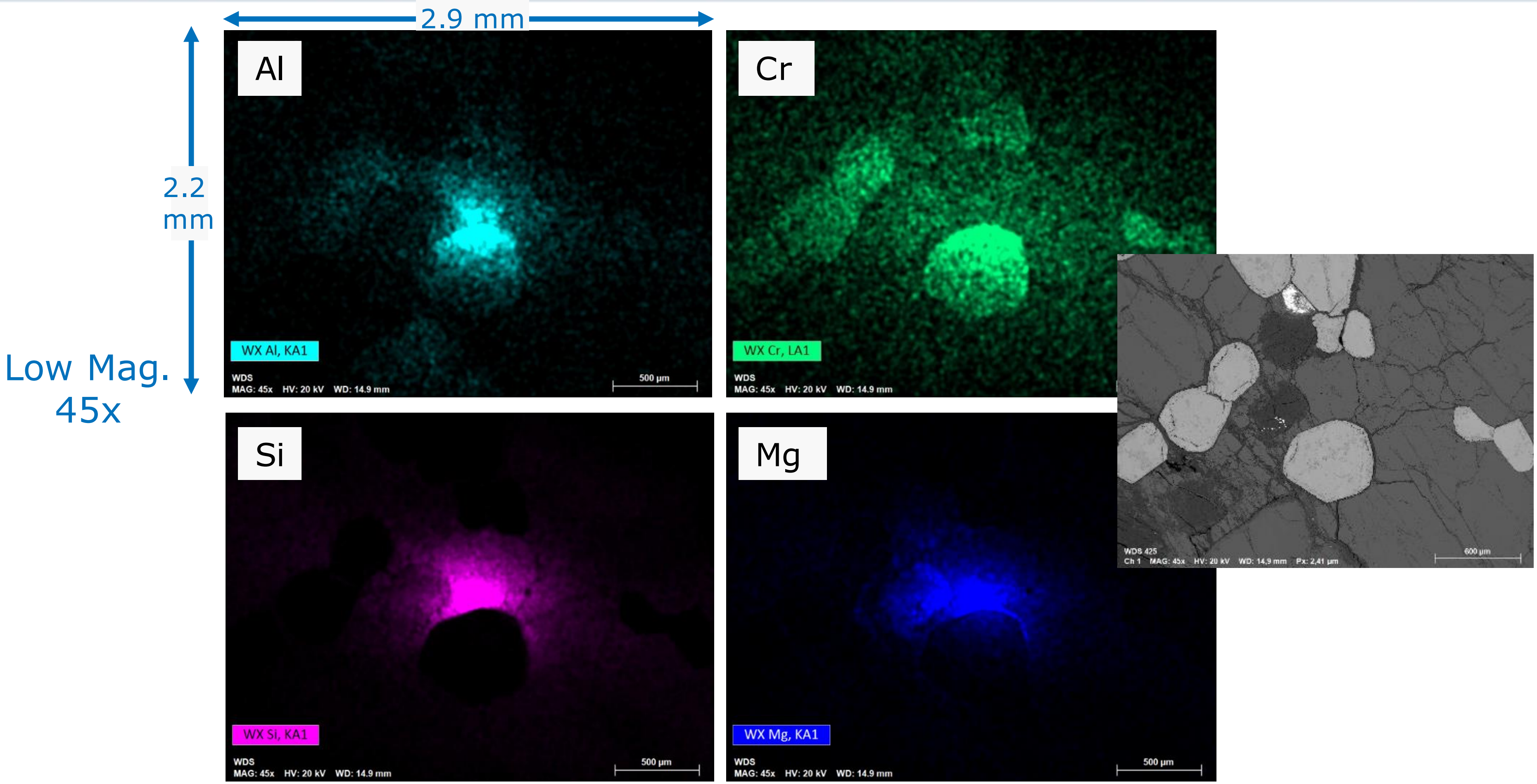
Sample 1 at low magnification Single Field



Type I Mapping: Low Magnification – Single Field
Problems for WDS Mapping due to violation of Bragg's Law

Scan Map Without Range Limits

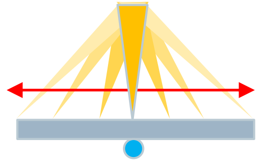
Sample 2 at low magnification
Single Field



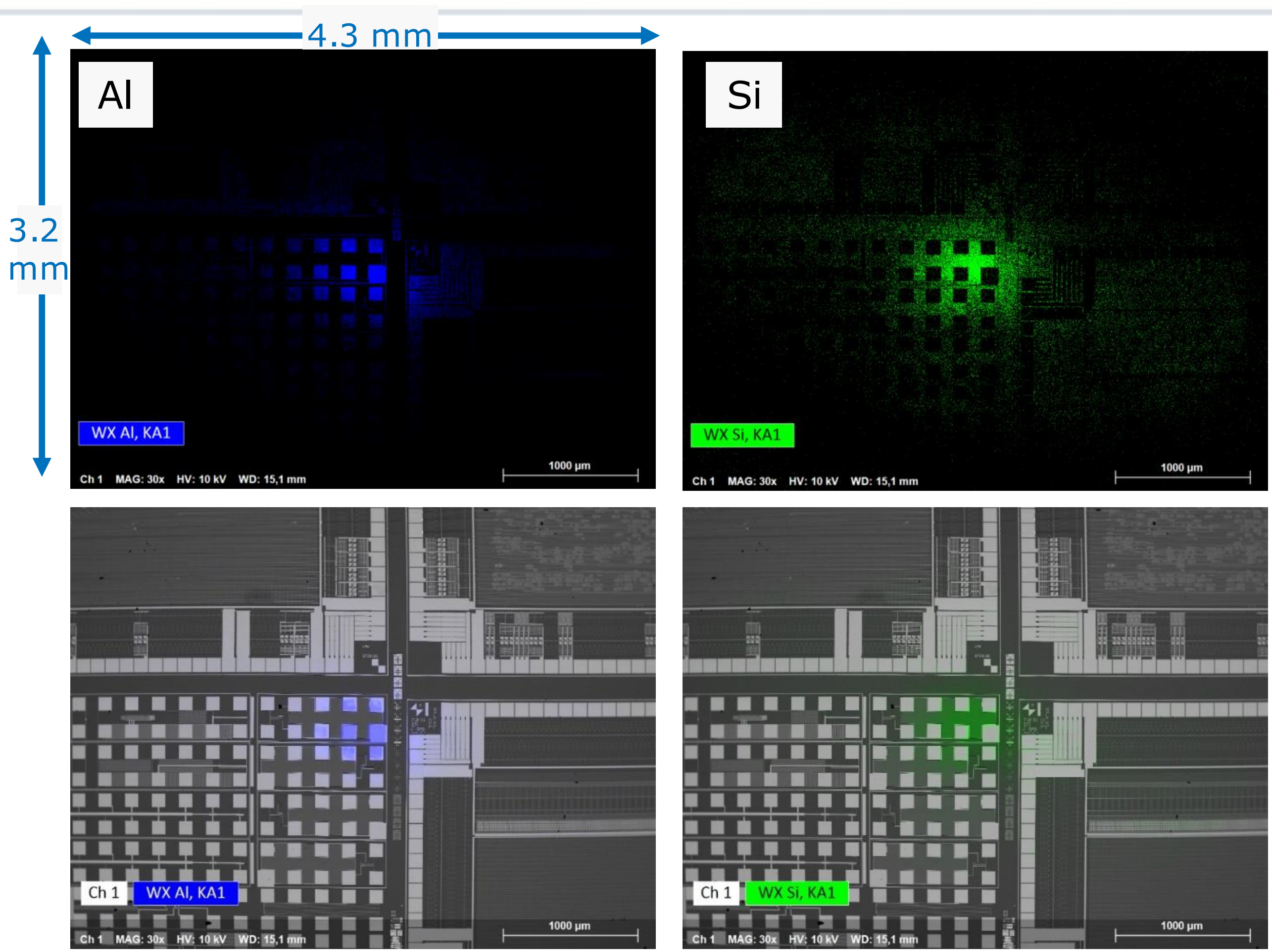
Type I Mapping: Low Magnification – Single Field
Problems for WDS Mapping due to violation of Bragg's Law

Scan Map Without Range Limits

Sample 3 at low magnification
Single Field



Low Mag.
30x

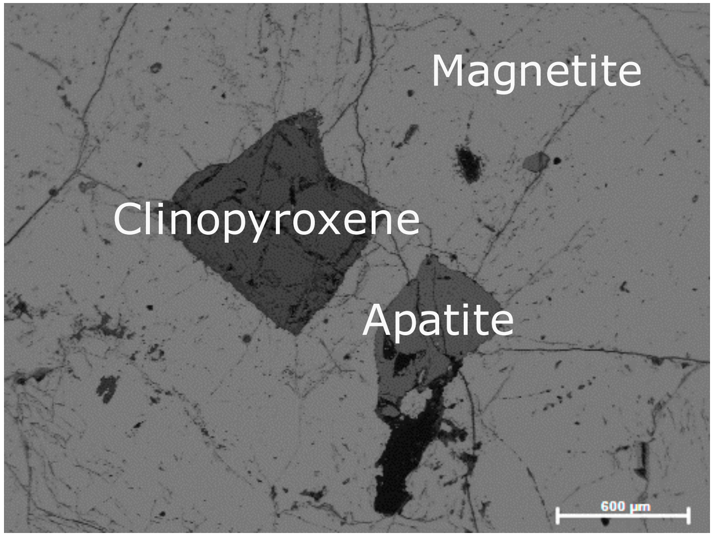


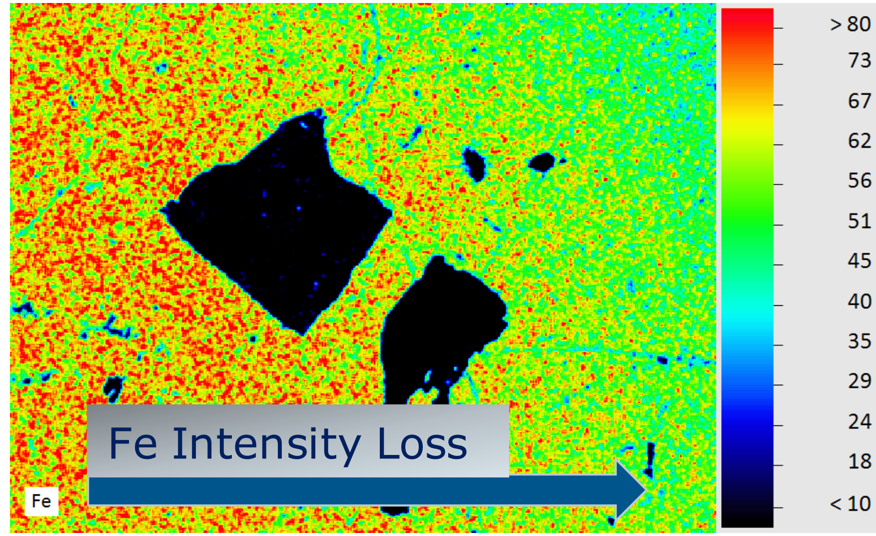
Type I Mapping: Low Magnification – Single Field
Problems for WDS Mapping due to violation of Bragg's Law

Scan Map Without Range Limits

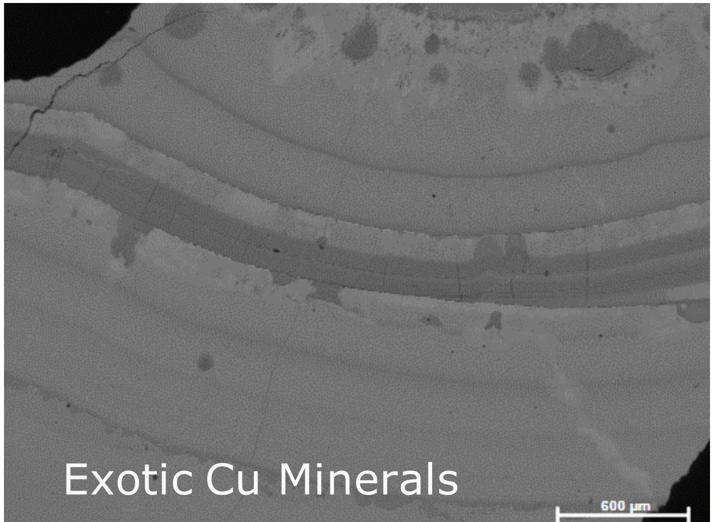
Sample 3 at low magnification
Single Field

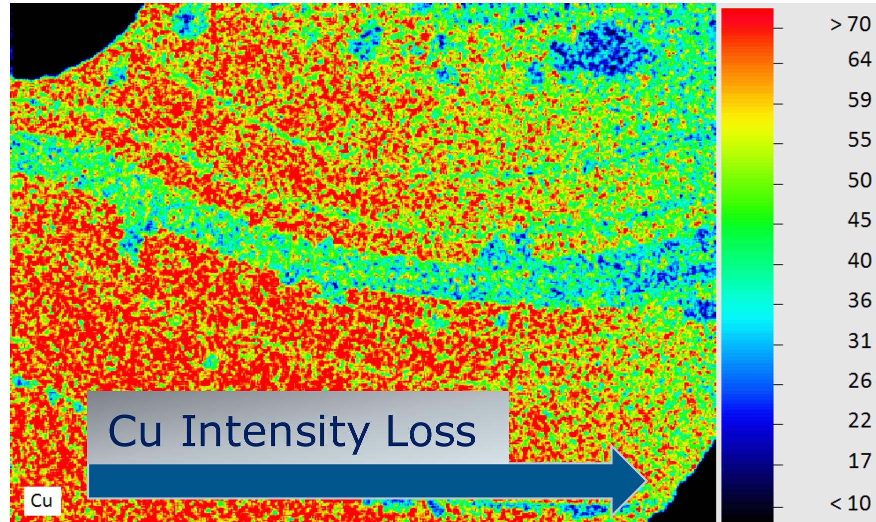
El Laco Deposit



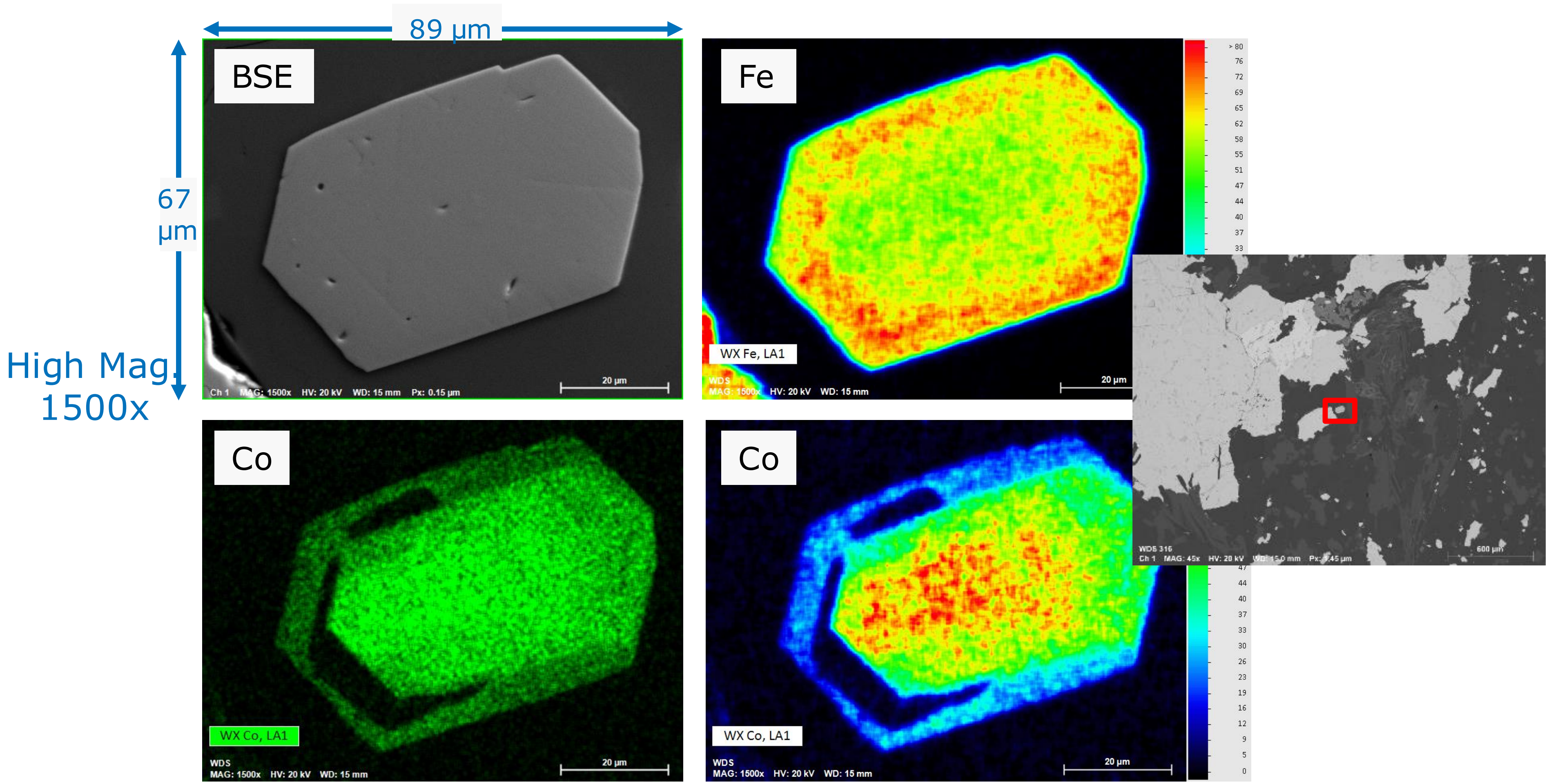


El Tesoro Deposit





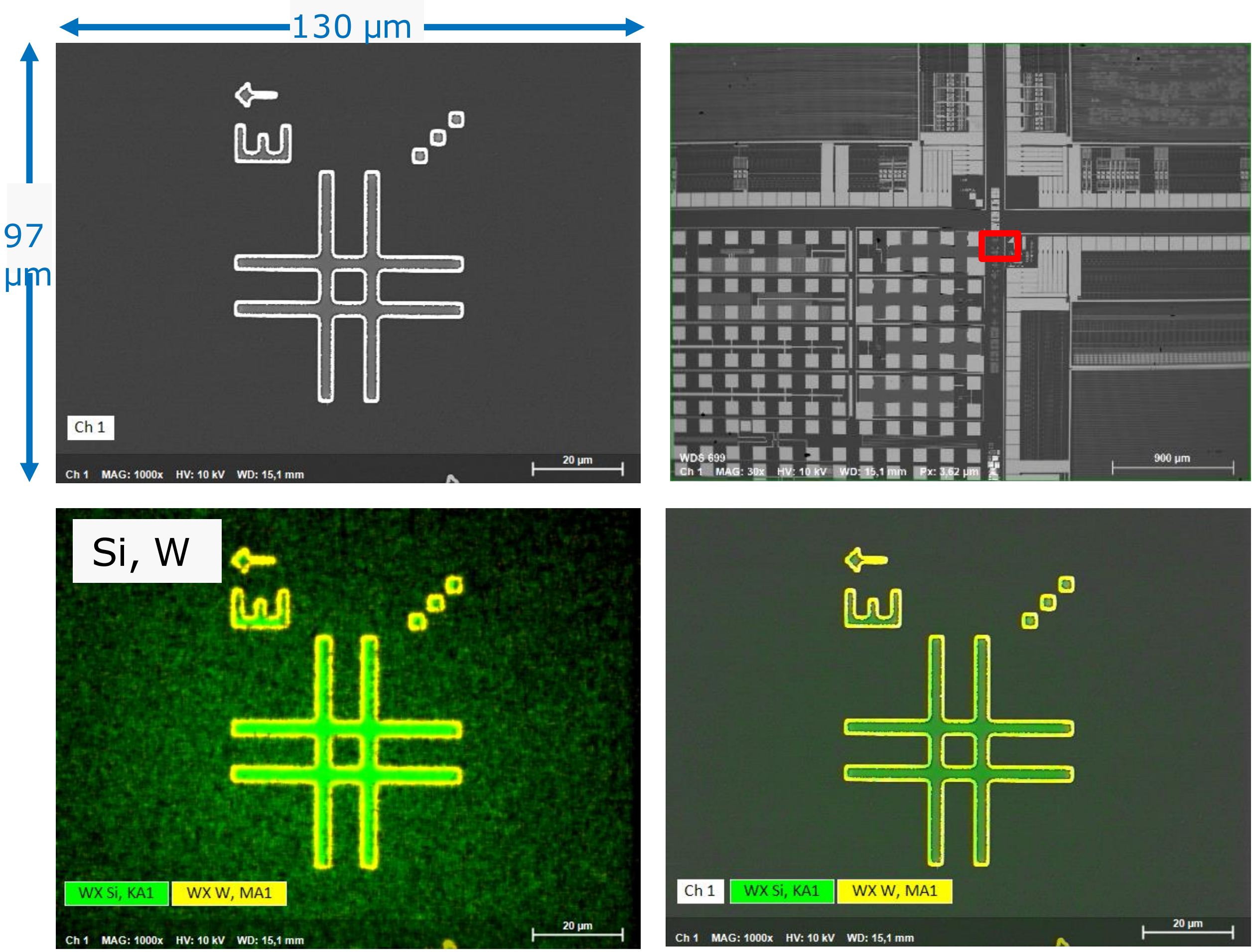
Scan Map Without Range Limits
Sample 1 at high magnification
Single Field



Type I Mapping: High Magnification – Single Field
Suitable method for Small Areas where Bragg Angle Effect is negligible

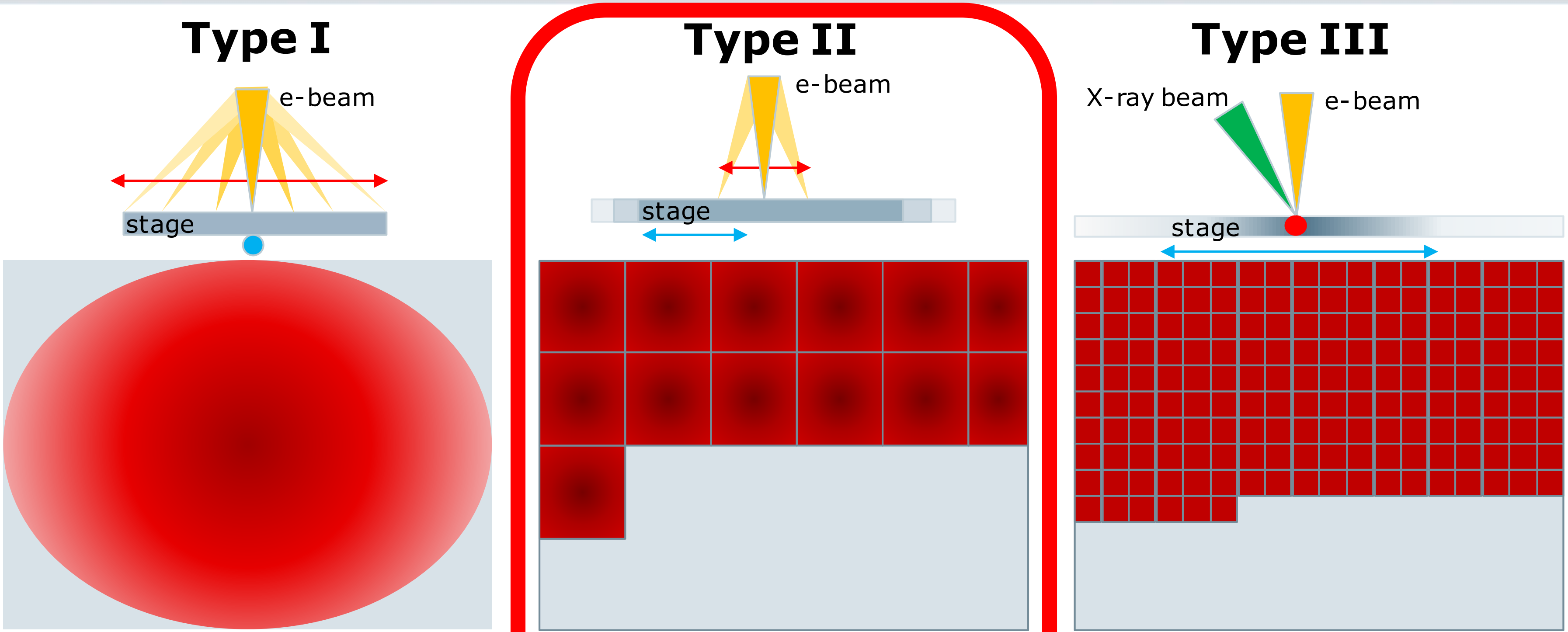
Scan Map Without Range Limits
Sample 3 at high magnification
Single Field

High Mag.
1000x



Type I Mapping: High Magnification – Single Field
Suitable method for Small Areas where Bragg Angle Effect is negligible

Large Area Mapping
Type II



Scan Map Without Limits
(Raster over Full Area)
e.g. Low Mag. 30x

Scan Map With Limits
(Raster over Restricted Area
with Stage Movement)

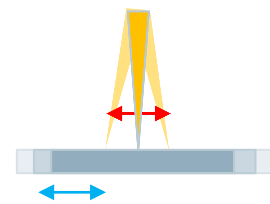
Stage Map
Independent of Magnification

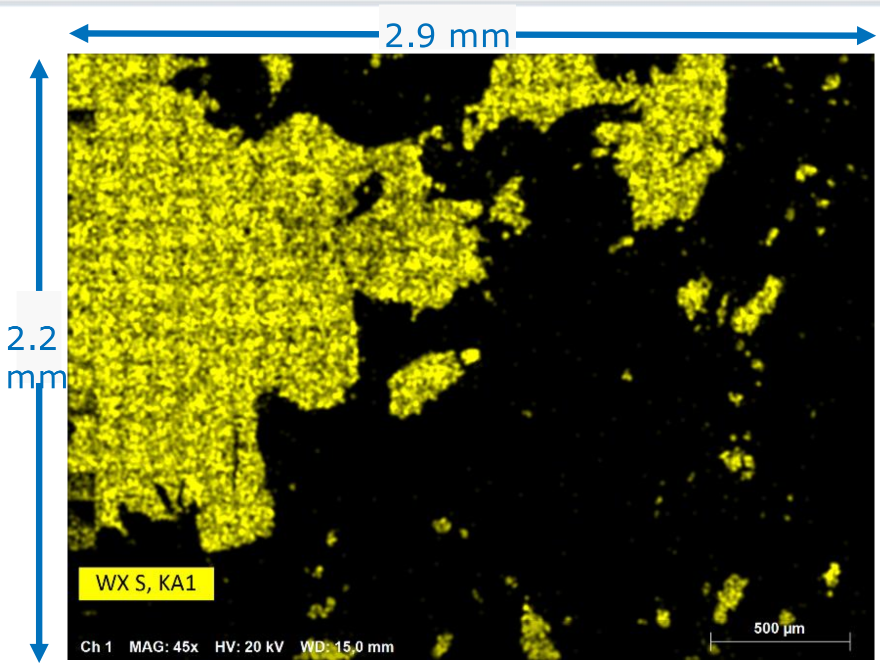
Increasing accuracy - WDS

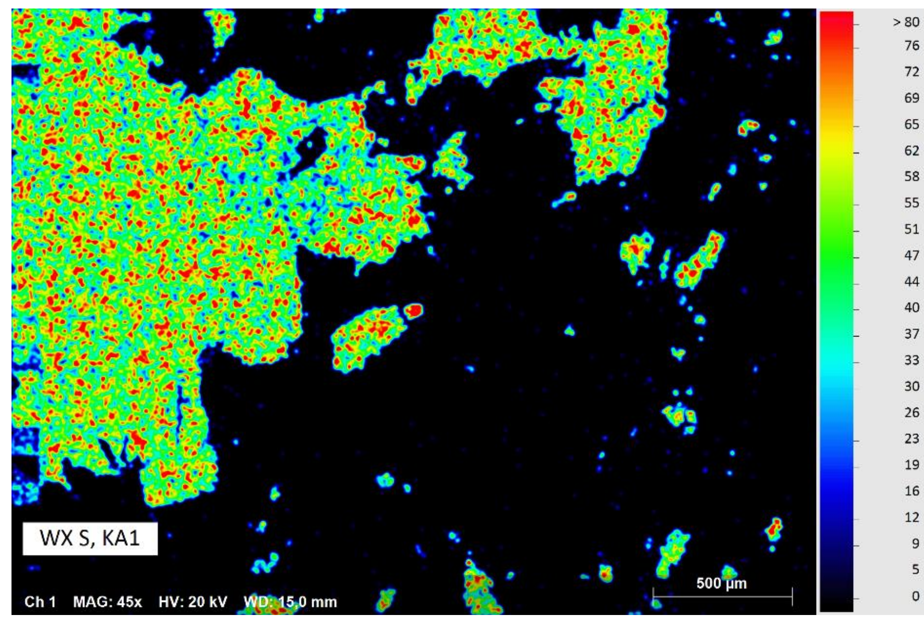
Increasing speed – SEM Stage

Scan Mapping with Range Limits

Sample 1 at low magnification
Segmented Map: 29 x 22 Tiles







- ✓ Faster than stage map
- ✓ Intensity fading reduced

- Slower than scan map without limits
- Tiling may be visible

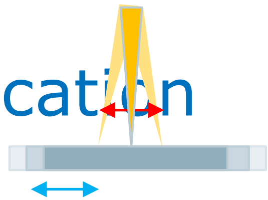
Type II Mapping: Low Magnification – Multiple Tiles 29 x 22

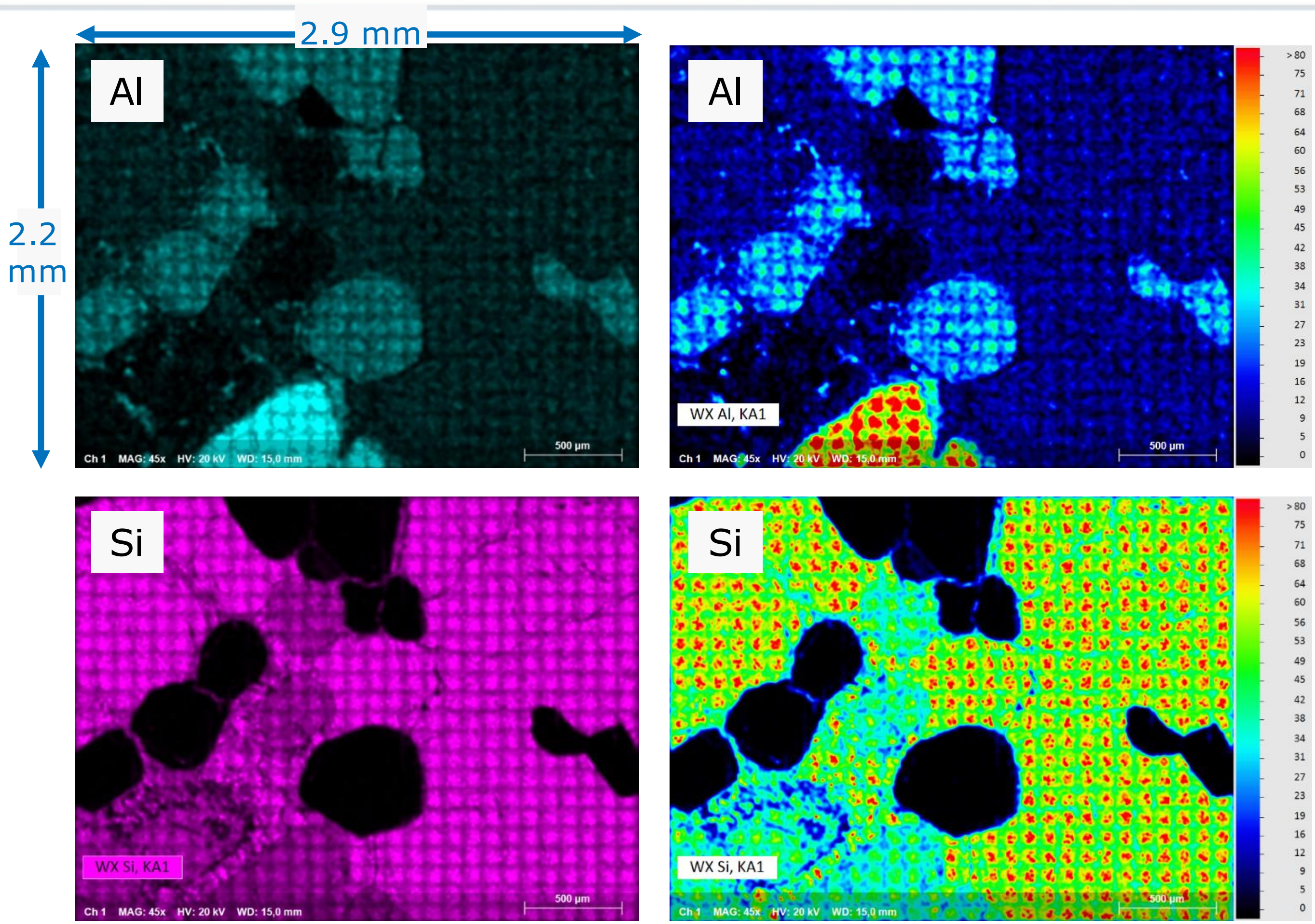
Bragg Angle Effect is lowered but may still be visible in mosaics

Scan Mapping with Range Limits

Sample 2 with Tiling at low magnification

Extended Map: 29 x 22 Tiles



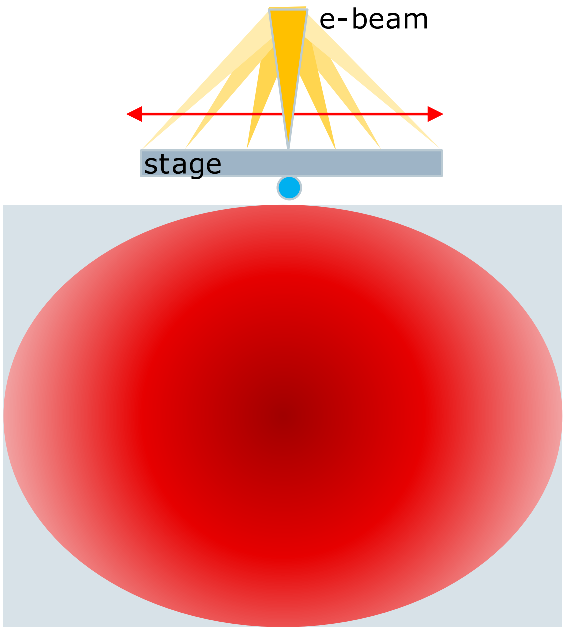


Type II Mapping: Low Magnification – Multiple Tiles 29 x 22

Bragg Angle Effect is lowered but may still be visible in mosaics

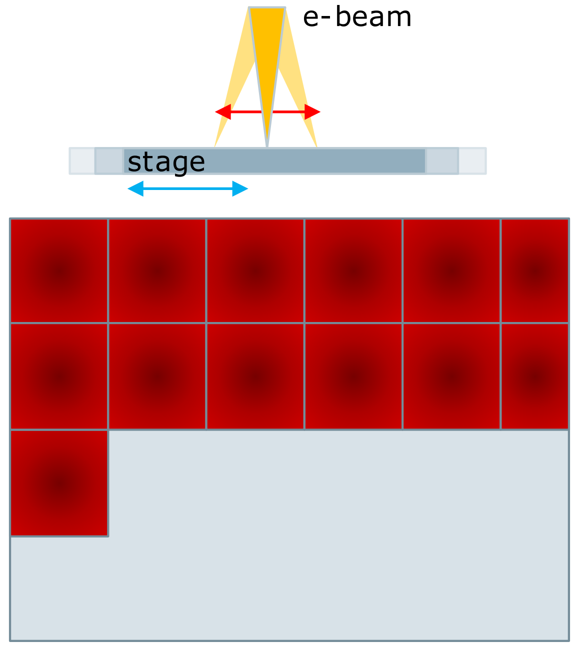
Large Area Mapping
Type IIIa: SEM Stage

Type I



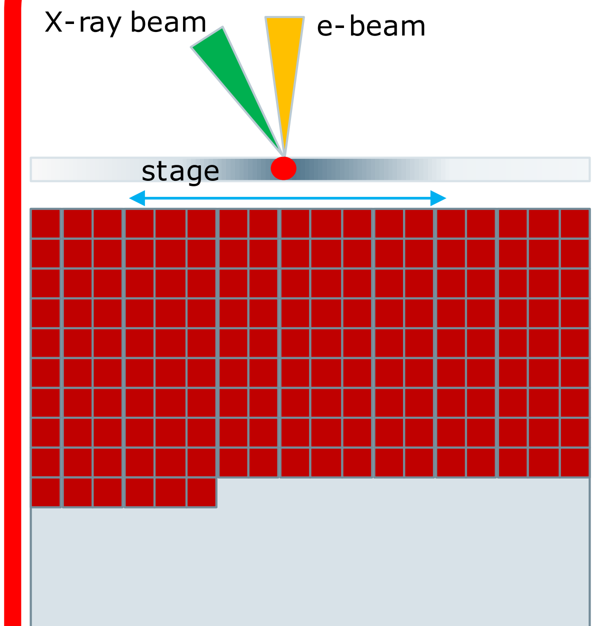
Scan Map Without Limits
(Raster over Full Area)
e.g. Low Mag. 30x

Type II



Scan Map With Limits
(Raster over Restricted Area
with Stage Movement)

Type III

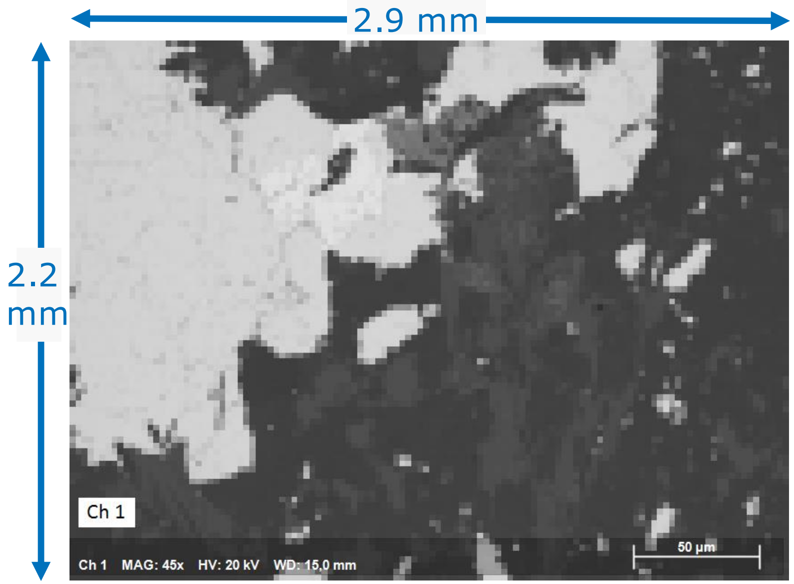


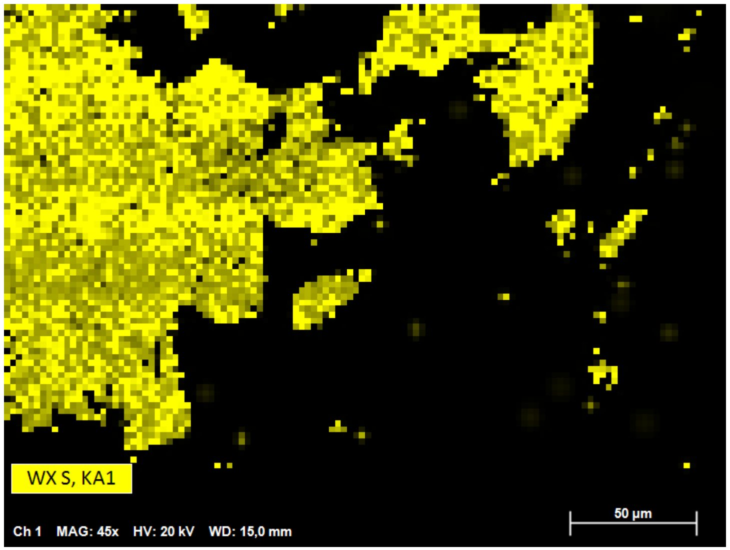
Stage Map
Independent of Magnification

Increasing accuracy - WDS

Increasing speed – SEM Stage

Stage Mapping using SEM Stage
Sample 1 at low magnification SEM-EDS/WDS





120 x 90 = 10,800 px → 10 h

1200 x 900 = 1,080,000 px → 42.5 d

Type IIIa Mapping: Low Magnification – Single Field Equivalent
High Analytical Time due to slow SEM Stage Movement

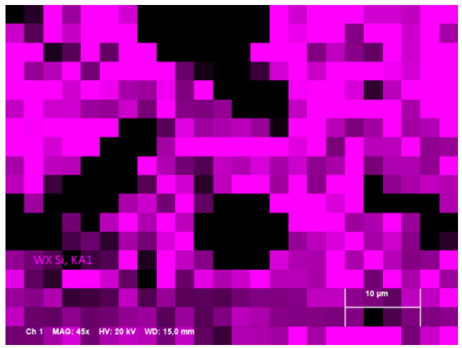
For SEM-EDS/WDS:
Not Practical or Realistic use of Analytical Equipment.

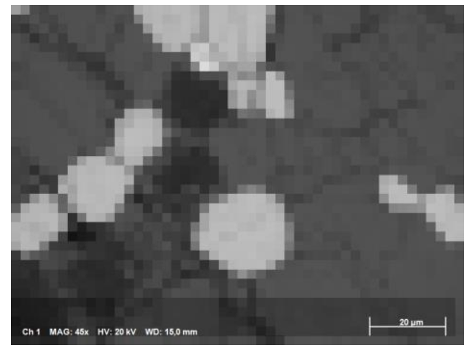
For SEM-XRF (Micro-XRF)
This is standard method, but slow. Fixed X-ray beam.

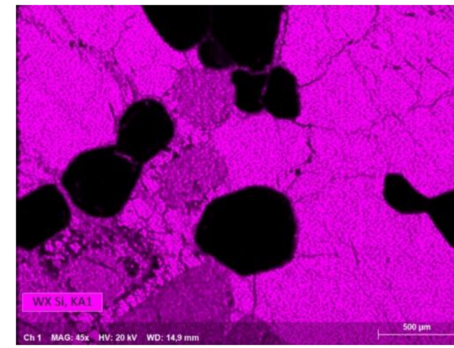
Stage Mapping using SEM Stage

Sample 2 at low magnification
SEM-EDS/WDS

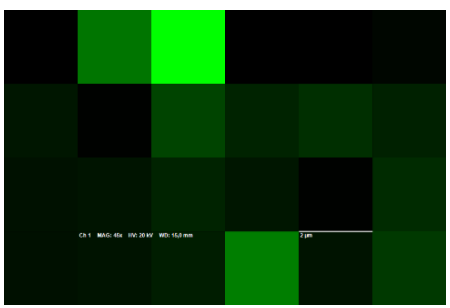
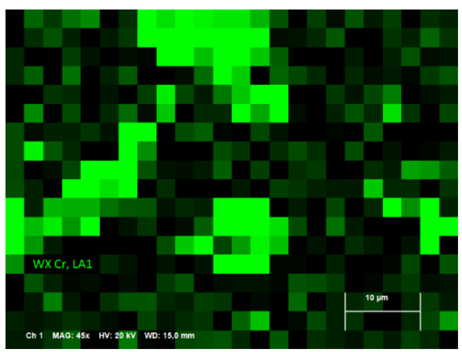
Effective time: 3.4 s / per pixel

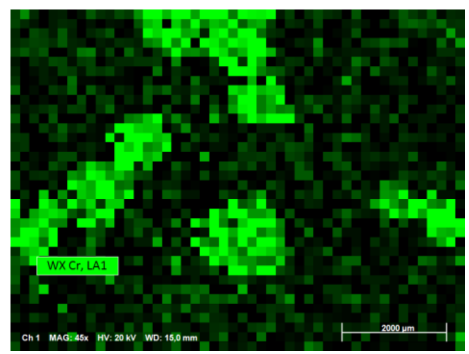



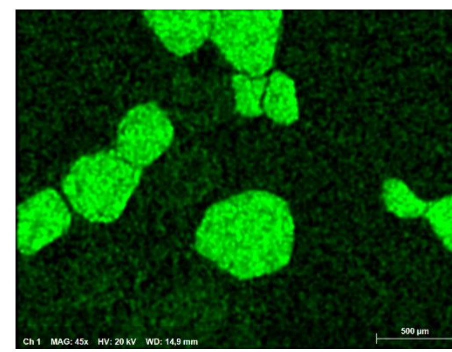




6x4 px = 82 s | 24x18 px = 25 min | 48x36 px = 97 min | **1200x900 px = 42 d**

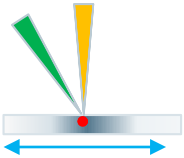




Type IIIa Mapping: Low Magnification – Single Field Equivalent
High Analytical Time due to slow SEM Stage Movement

Stage Mapping using SEM Stage

Sample 2 at low magnification
SEM-XRF



Large Area Map

Sample Size: Polished Section: 45 x 30 mm

Sample from El Tesoro, Chile.

Clearly Defined Elemental and Mineralogical Phases

Can identify the presence of trace elements, in this case, Cobalt (Co), Manganese (Mn), Strontium (Sr)

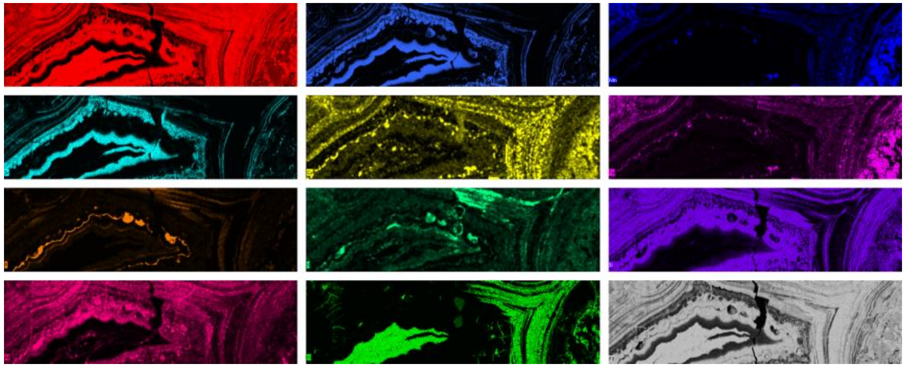
Analytical Parameters:

Tube Voltage: Rh at 50 kV

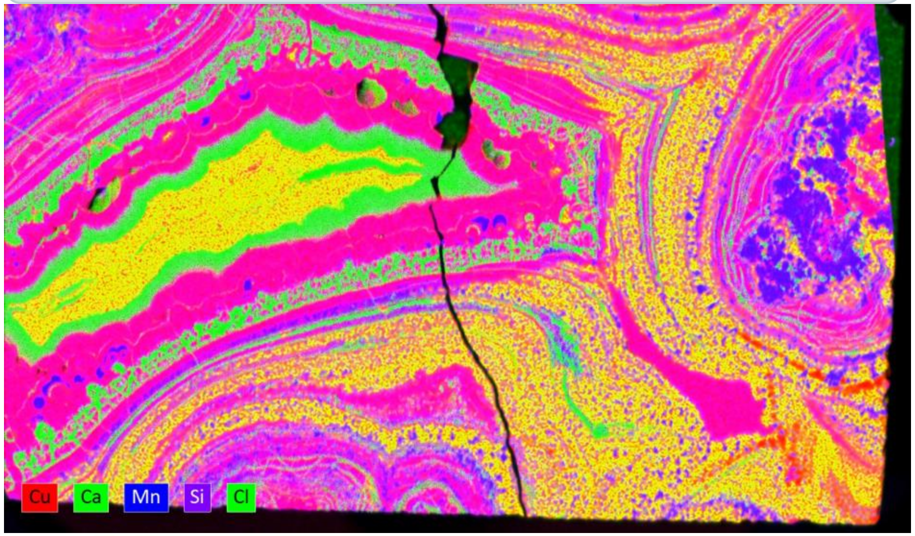
Anode Current: 600 µA

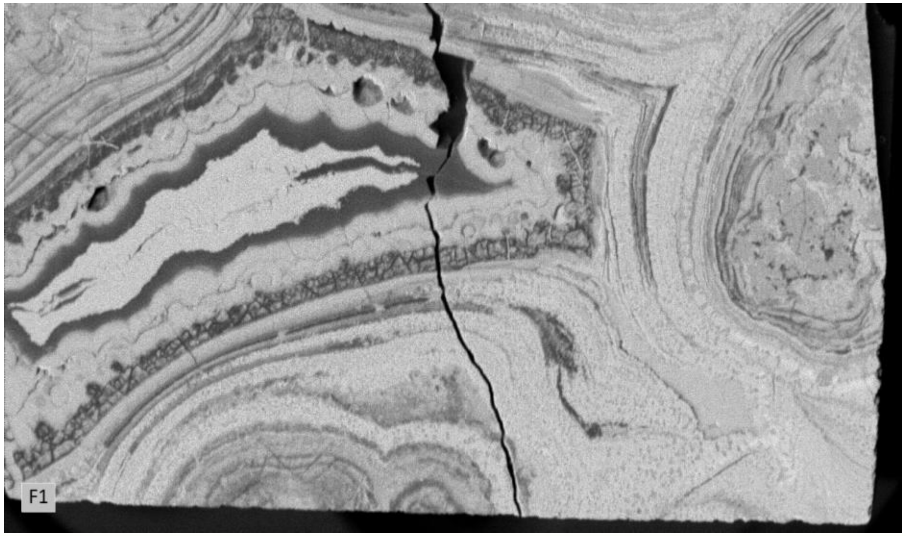
Pixel Spacing: 25 µm

Analytical Time:	**Rapid Stage 101 mins**
	SEM Stage >800 mins



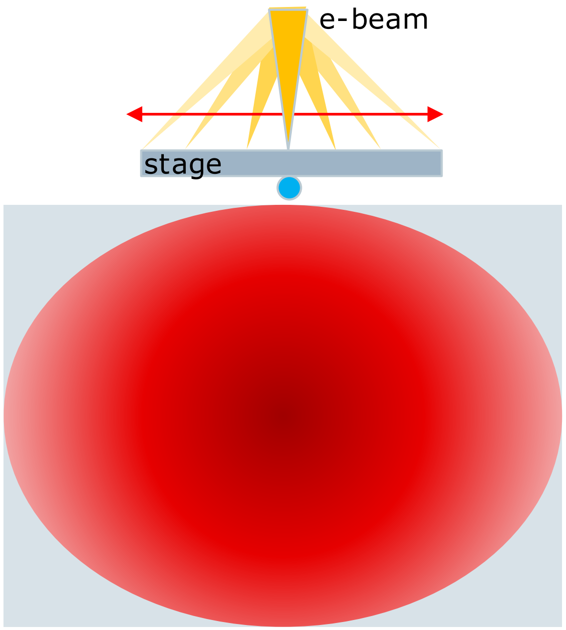
Top: Elemental Maps; Bottom Left: Mixed Elemental Map; Bottom, Right: X-Ray Intensity Map.





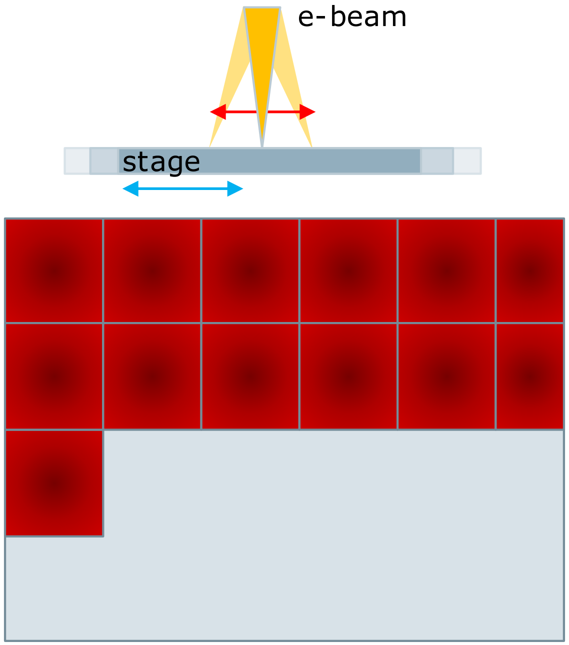
Large Area Mapping
Type IIIb: Rapid Stage

Type I



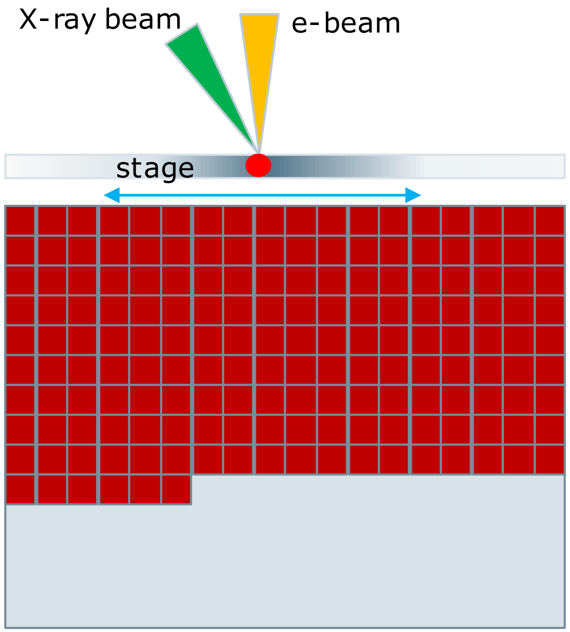
Scan Map Without Limits
(Raster over Full Area)
e.g. Low Mag. 30x

Type II



Scan Map With Limits
(Raster over Restricted Area
with Stage Movement)

Type III



Stage Map
Independent of Magnification

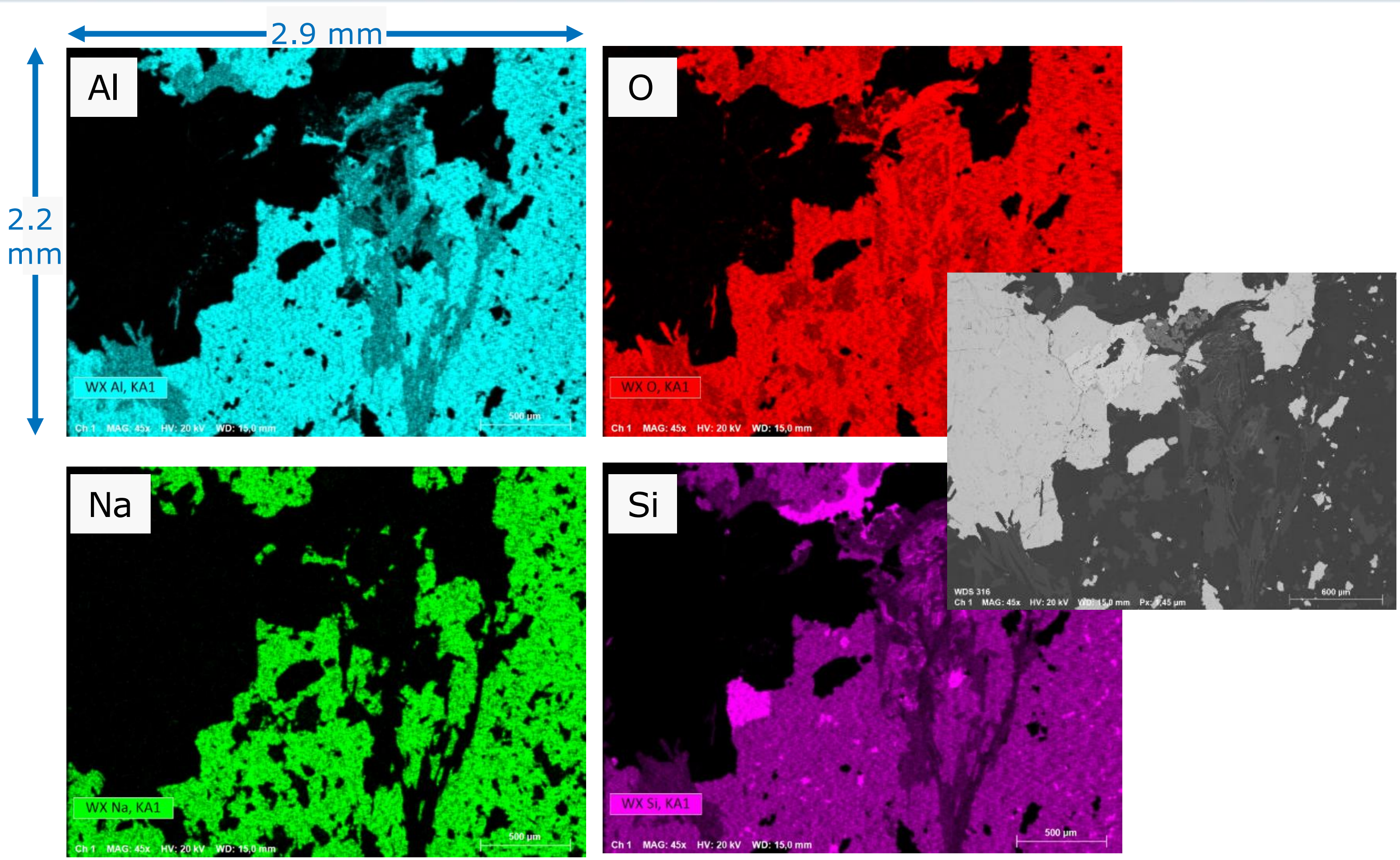
Increasing accuracy - WDS →

← Increasing speed – SEM Stage

Stage Mapping using Rapid Stage
Sample 1 at low magnification

Dwell time:
4 ms / px

Analytical time: 1 h / frame

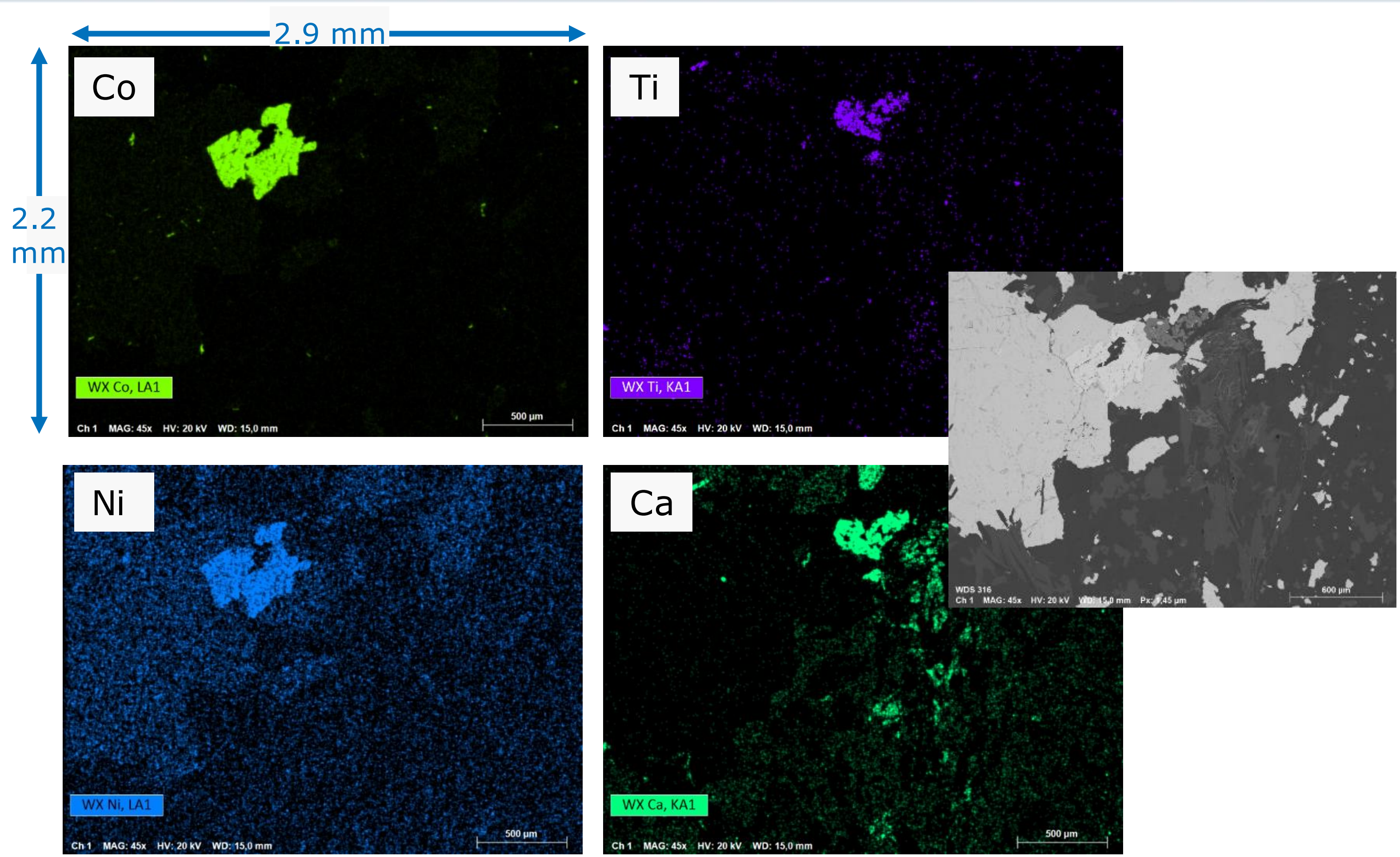


Type IIIb Mapping: Low Magnification – Single Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 1 at low magnification

Dwell time: 4 ms / px

Analytical time: 1 h / frame

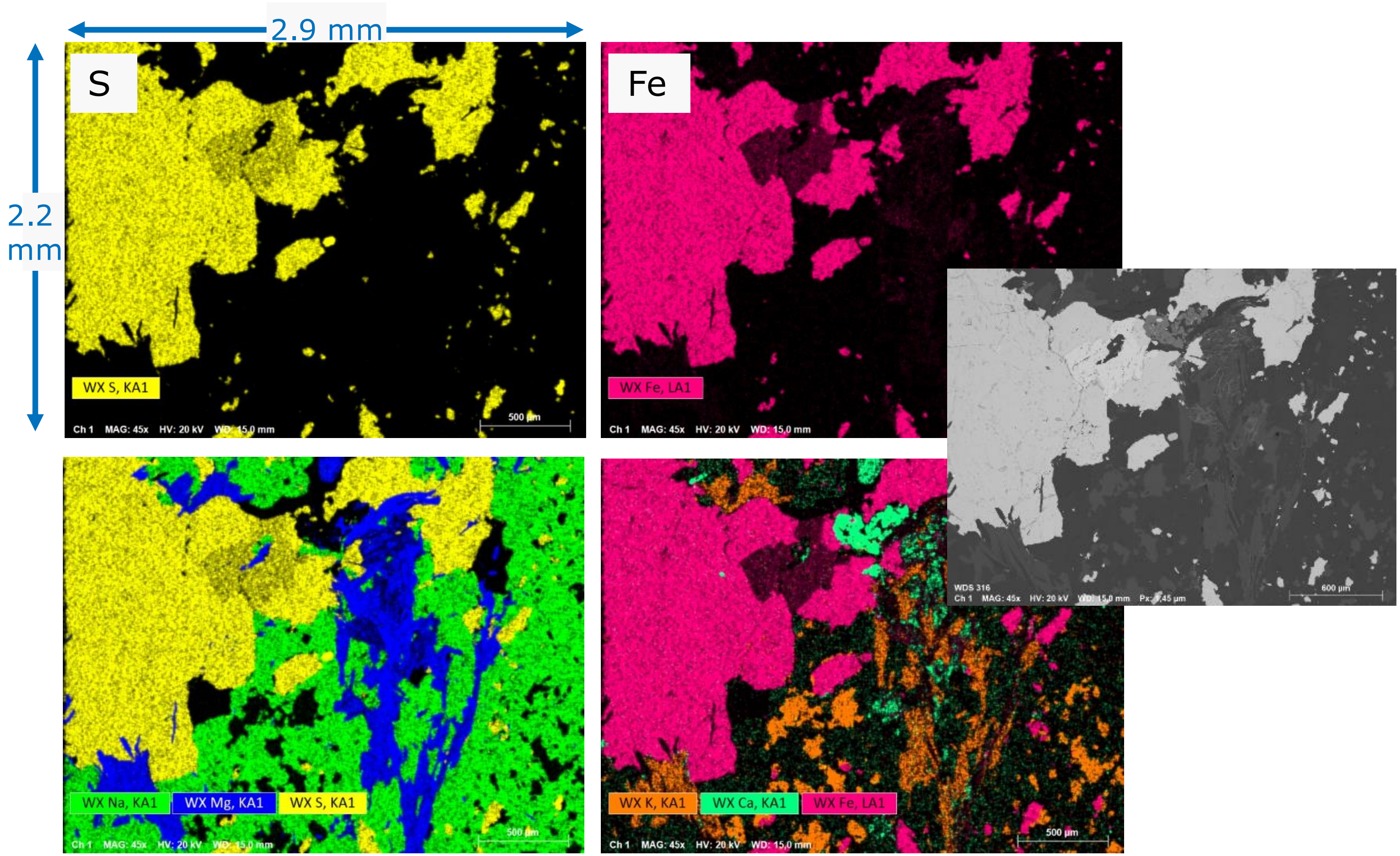


Type IIIb Mapping: Low Magnification – Single Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 1 at low magnification

Dwell time:
4 ms / px

Analytical time: 1 h / frame

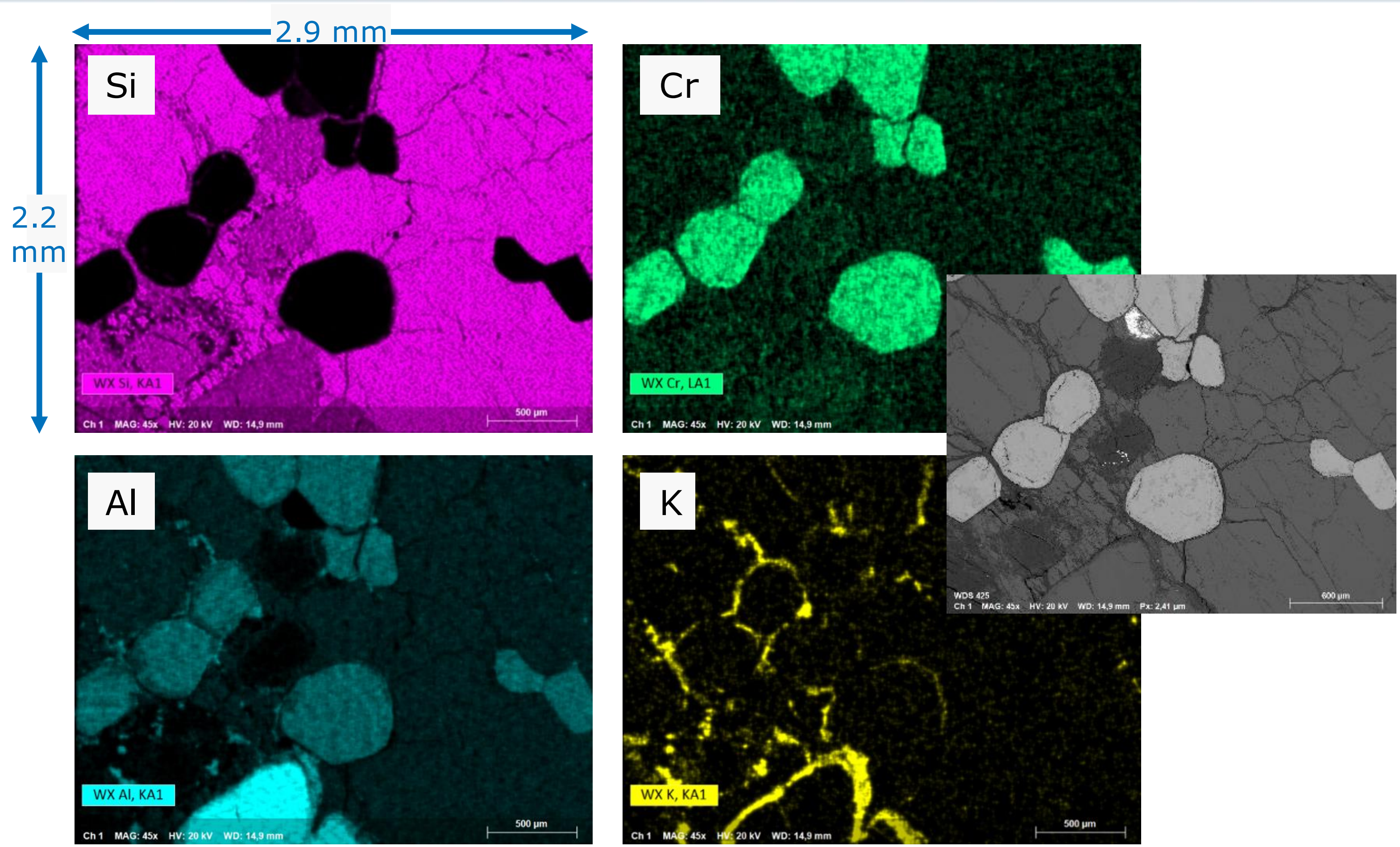


Type IIIb Mapping: Low Magnification - Single Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 2 at low magnification

Dwell time: 4 ms / px

Analytical time: 72 min / frame

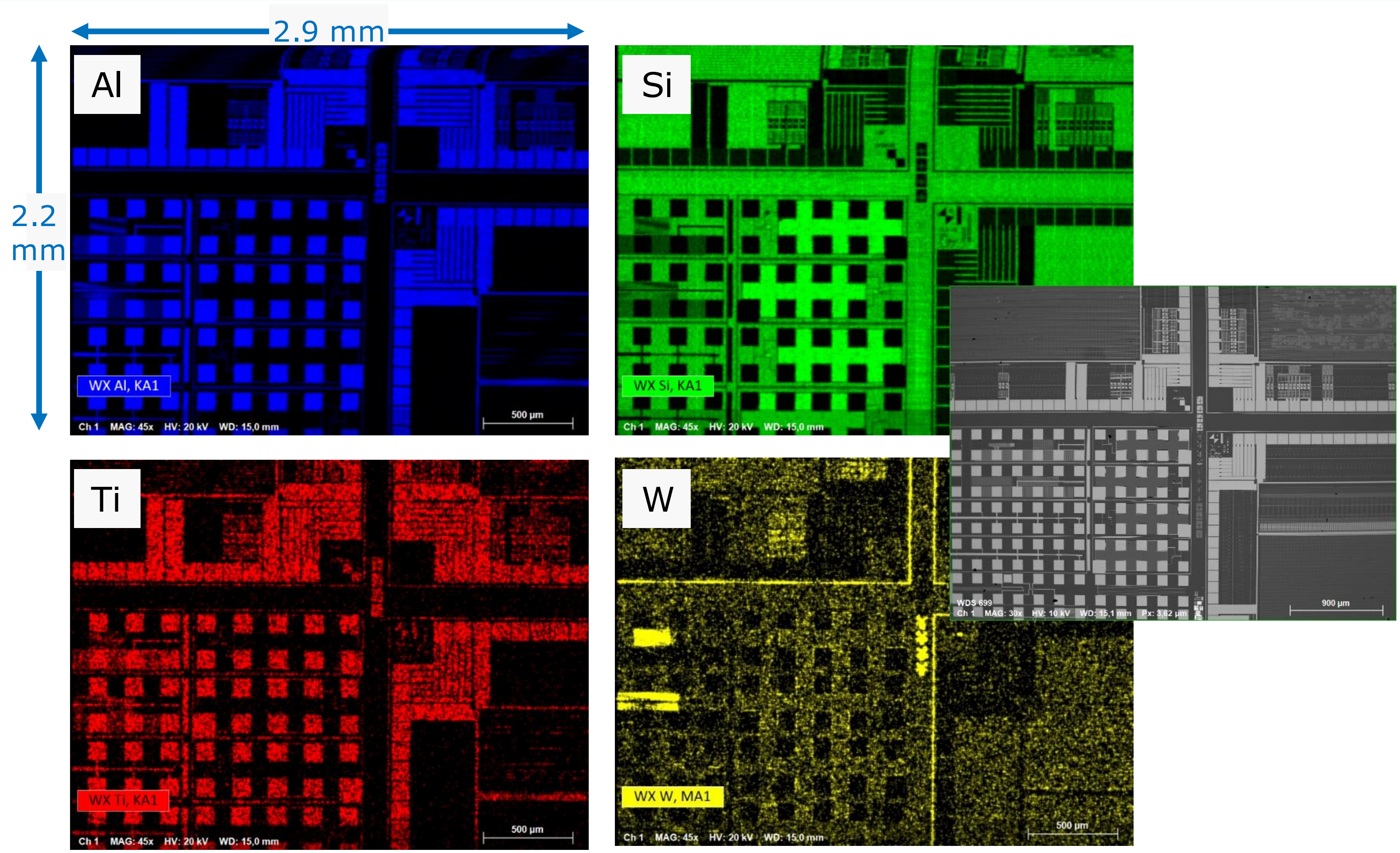


Type IIIb Mapping: Low Magnification – Single Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 3 at low magnification

Dwell time: 6.9 ms / px

Analytical time: 2 h / frame

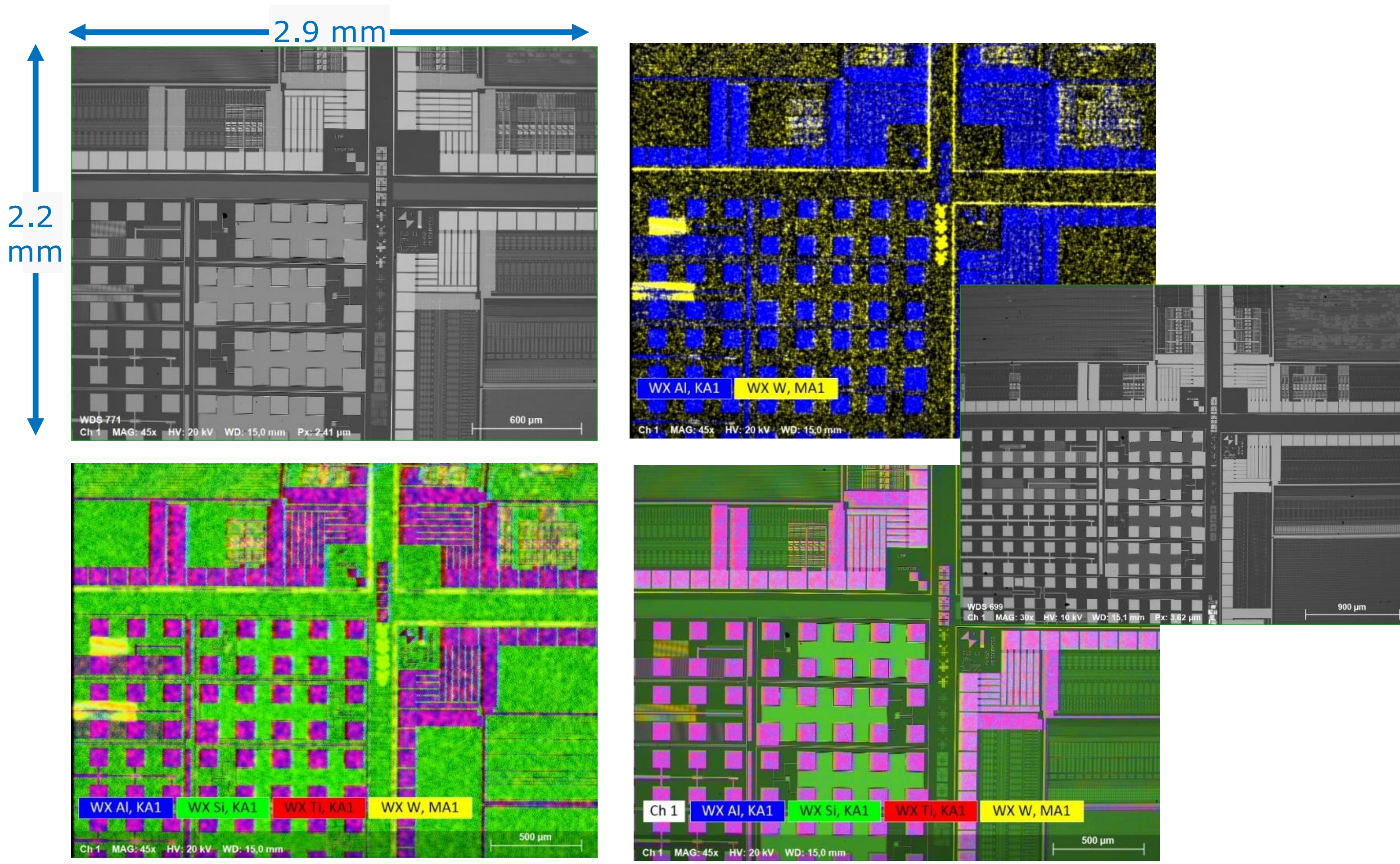


Type IIIb Mapping: Low Magnification – Single Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 3 at low magnification

Dwell time:
6.9 ms / px

Analytical time: 2 h / frame

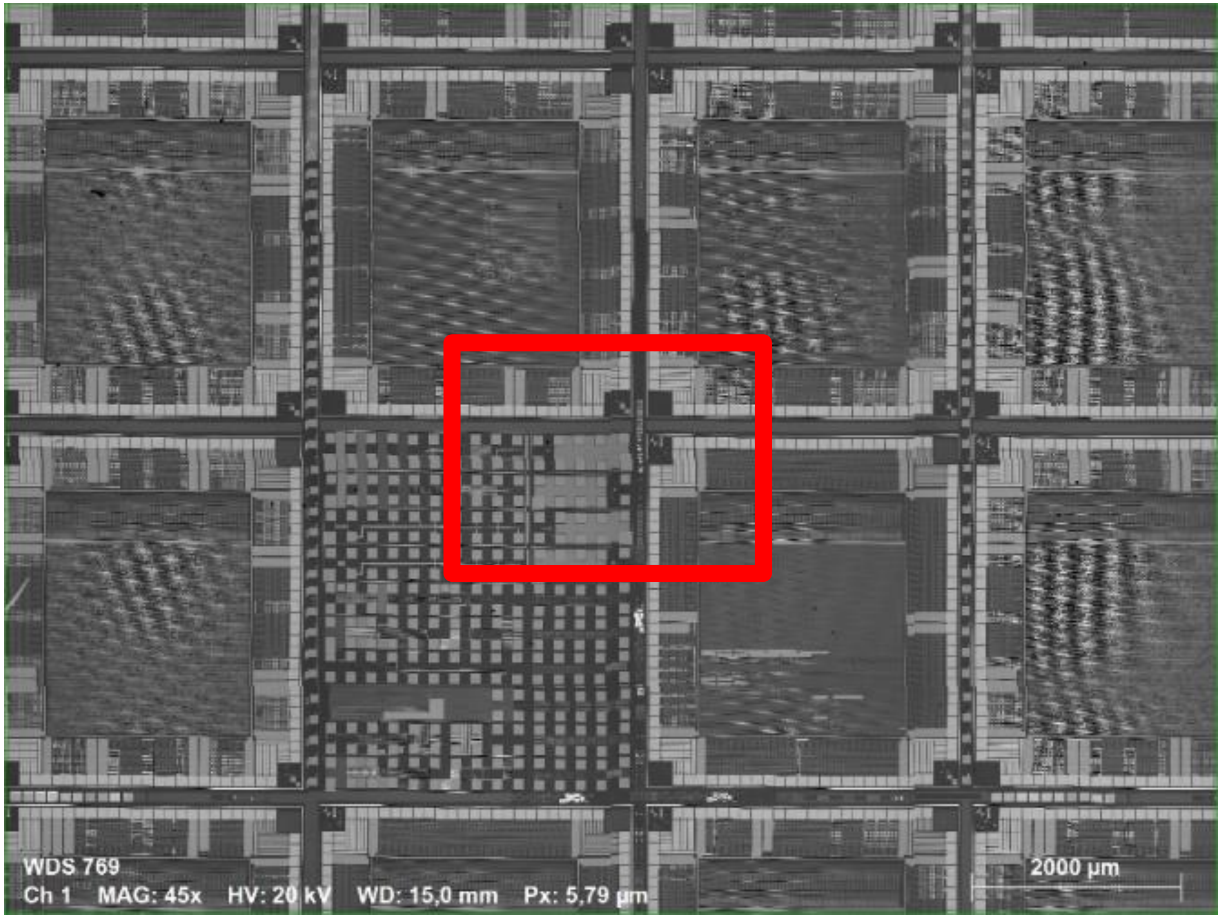


Type IIIb Mapping: Low Magnification – Single Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 3 extended area: 4x4

11.6 mm

8.6 mm



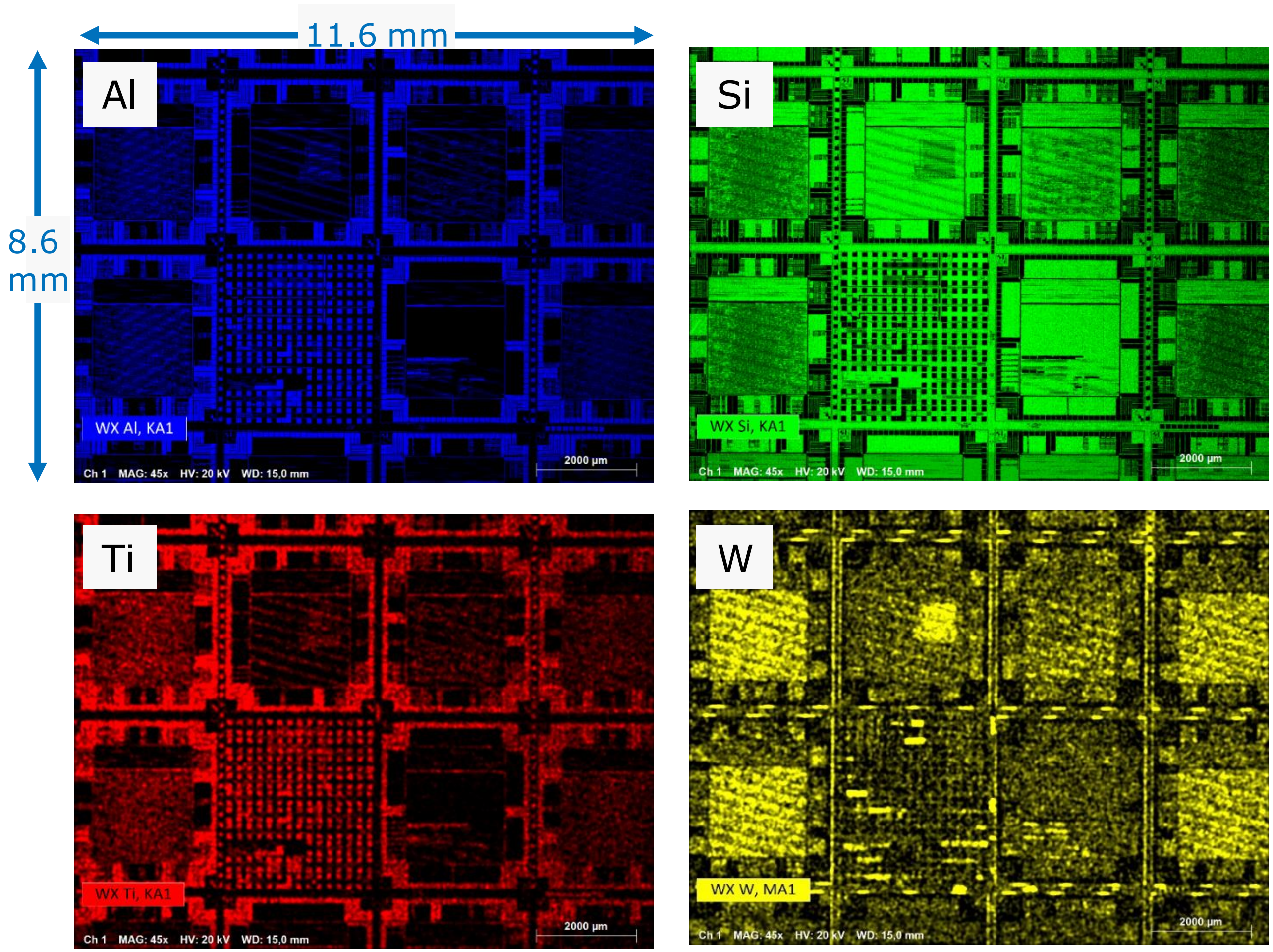
Type IIIb Mapping: Low Magnification – Multiple Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage

Sample 3 extended area

Dwell time: 4.8 ms / px

Analytical time: 4 h / frame

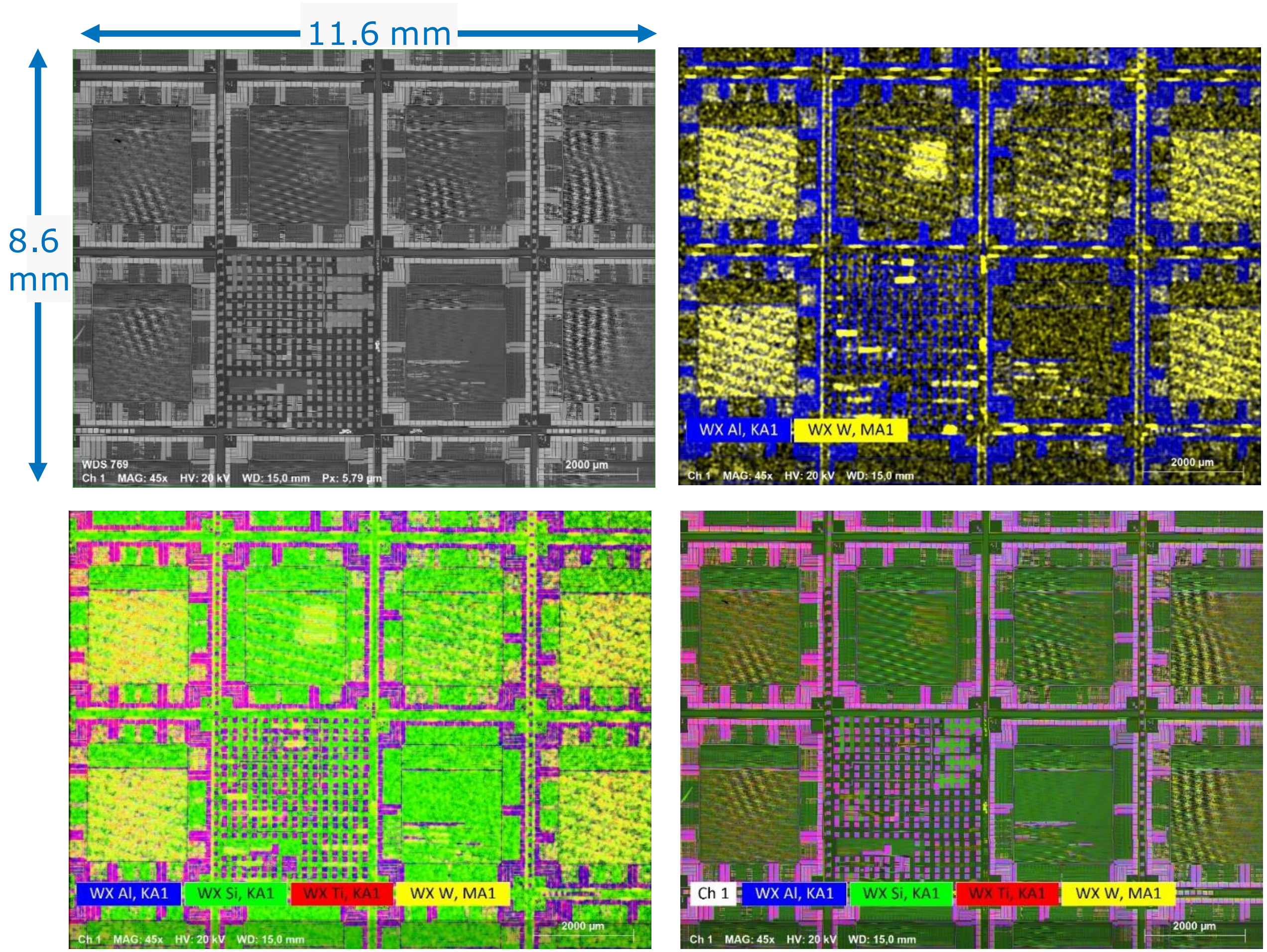


Type IIIb Mapping: Low Magnification – Multiple Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 3 extended area

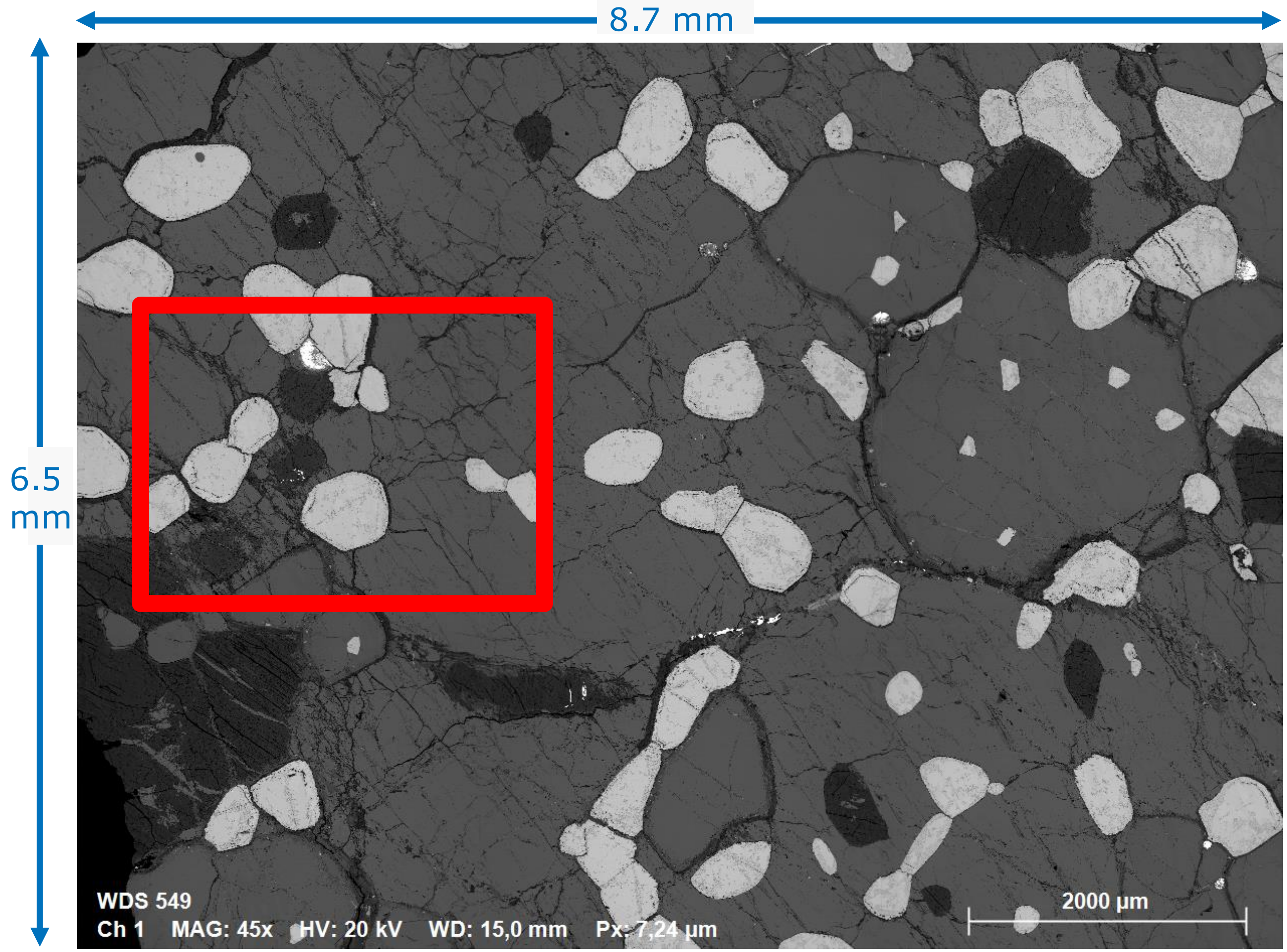
Dwell time: 4.8 ms / px

Analytical time: 4 h / frame



Type IIIb Mapping: Low Magnification – Multiple Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 2 extended area: 3x3

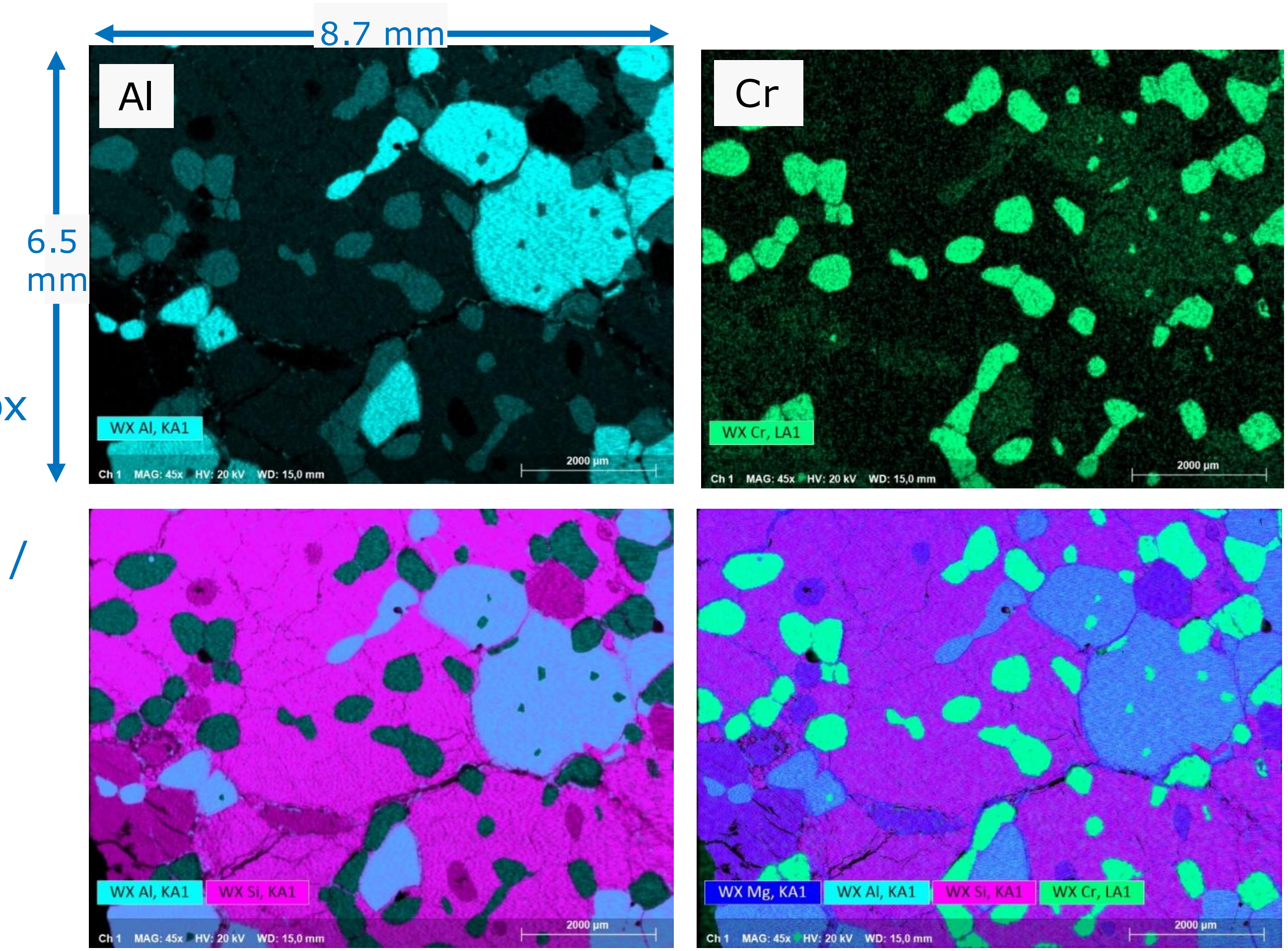


Type IIIb Mapping: Low Magnification – Multiple Field Equivalent
High speed mapping with no artifacts

Stage map using Rapid Stage
Sample 2 extended area

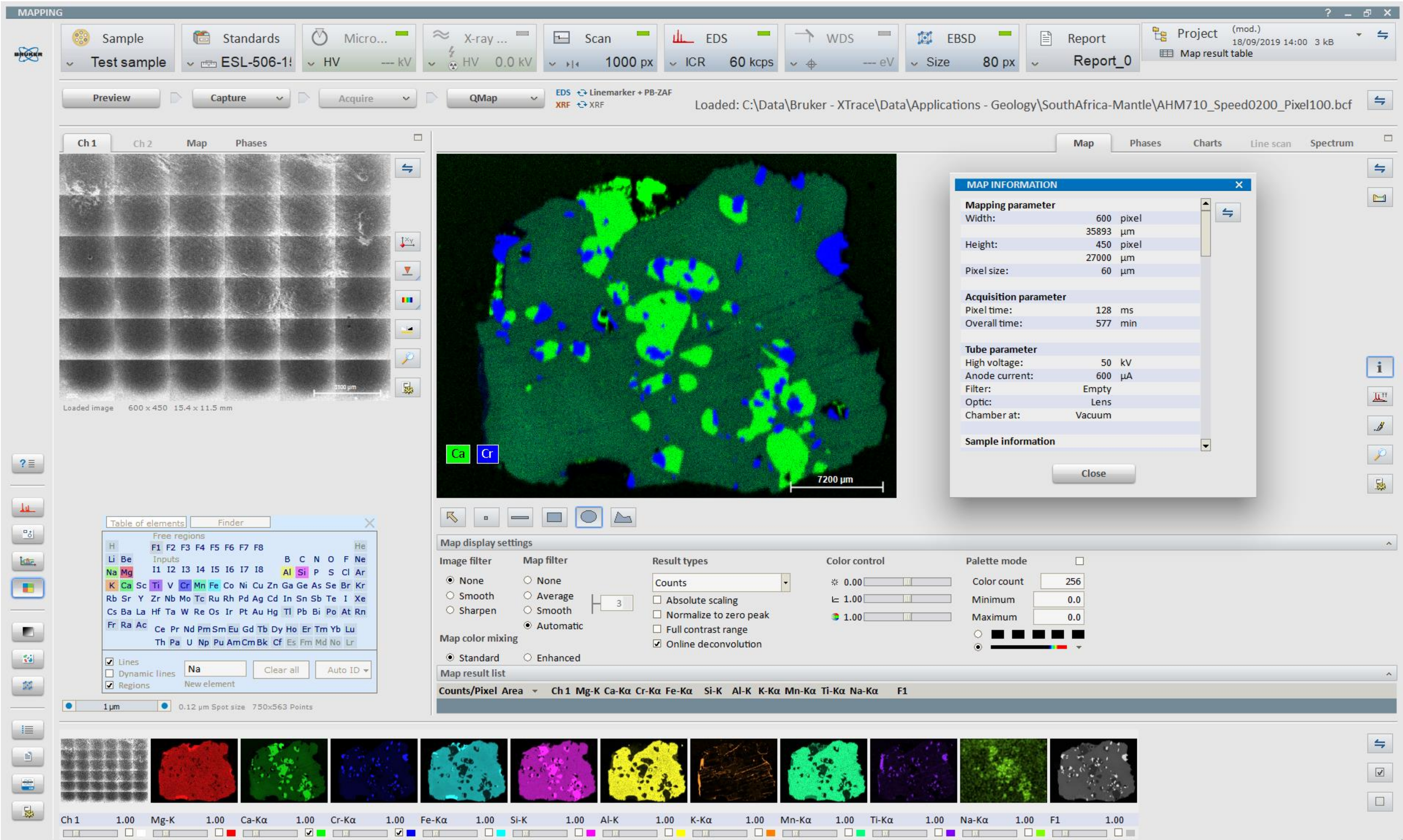
Dwell time: 15.4 ms / px

Analytical time: 4.5 h / frame

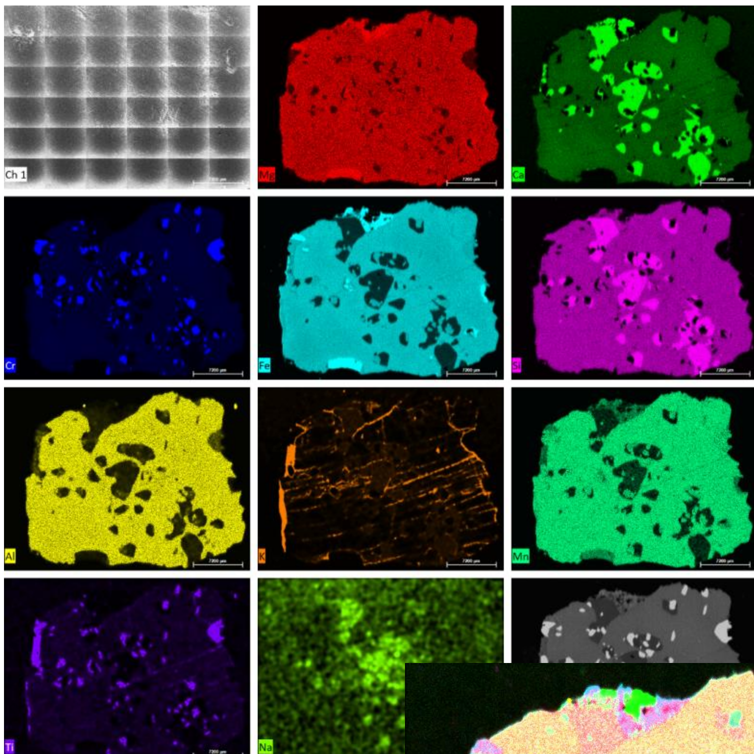


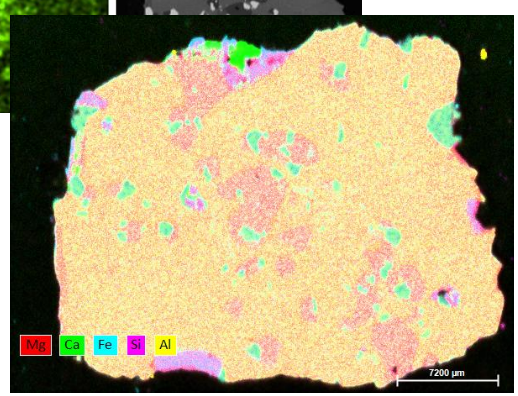
Type IIIb Mapping: Low Magnification – Multiple Field Equivalent
High speed mapping with no artifacts

Stage Mapping using Rapid Stage
Sample 2 extended area

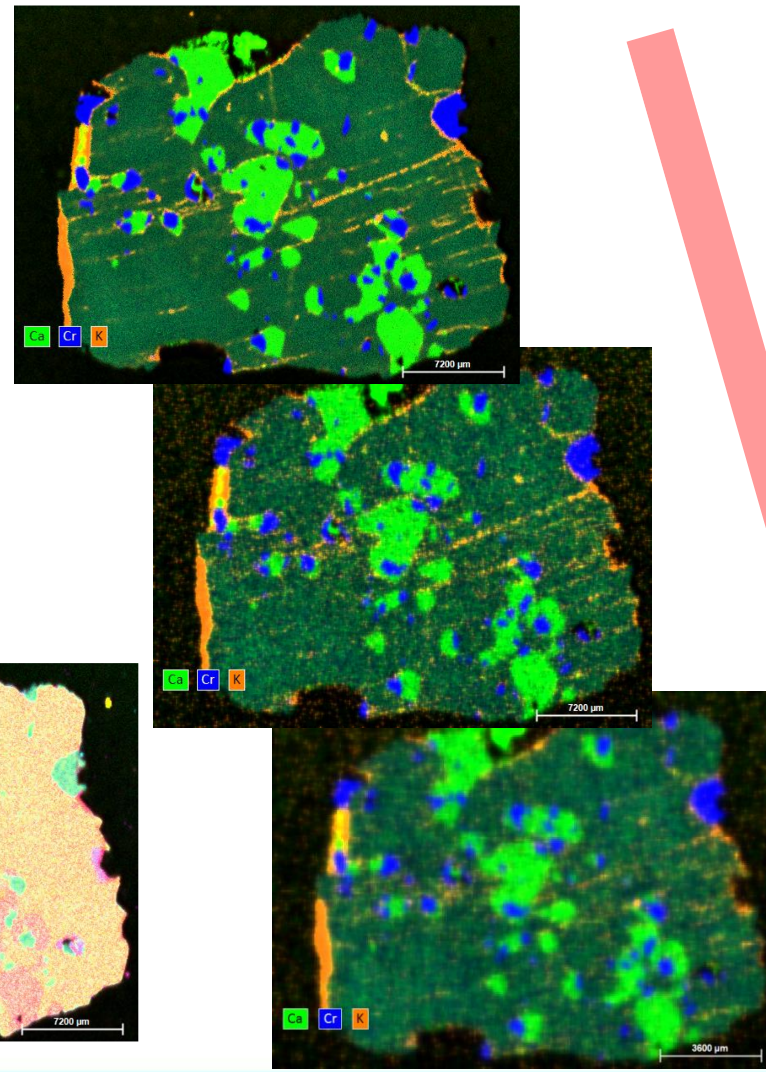


Stage Mapping using Rapid Stage
Sample 2 extended area





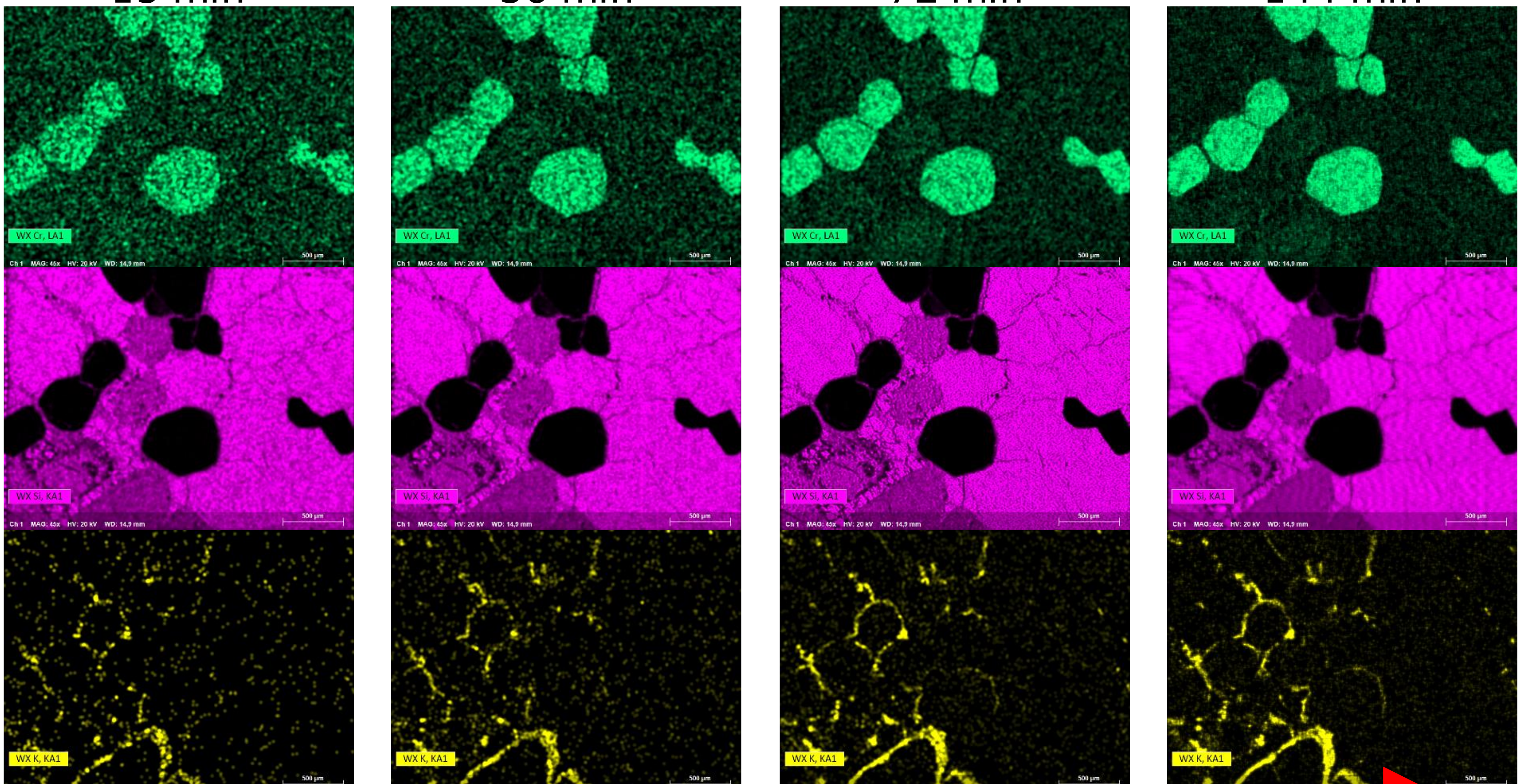
Mantle Peridotite:
Garnet
Chromite
Clinopyroxene
Olivine



Decreased Analytical Time :
(minutes)
577
23
11
Decreased Information per pixel

Stage Mapping using Rapid Stage
Effect of acquisition time

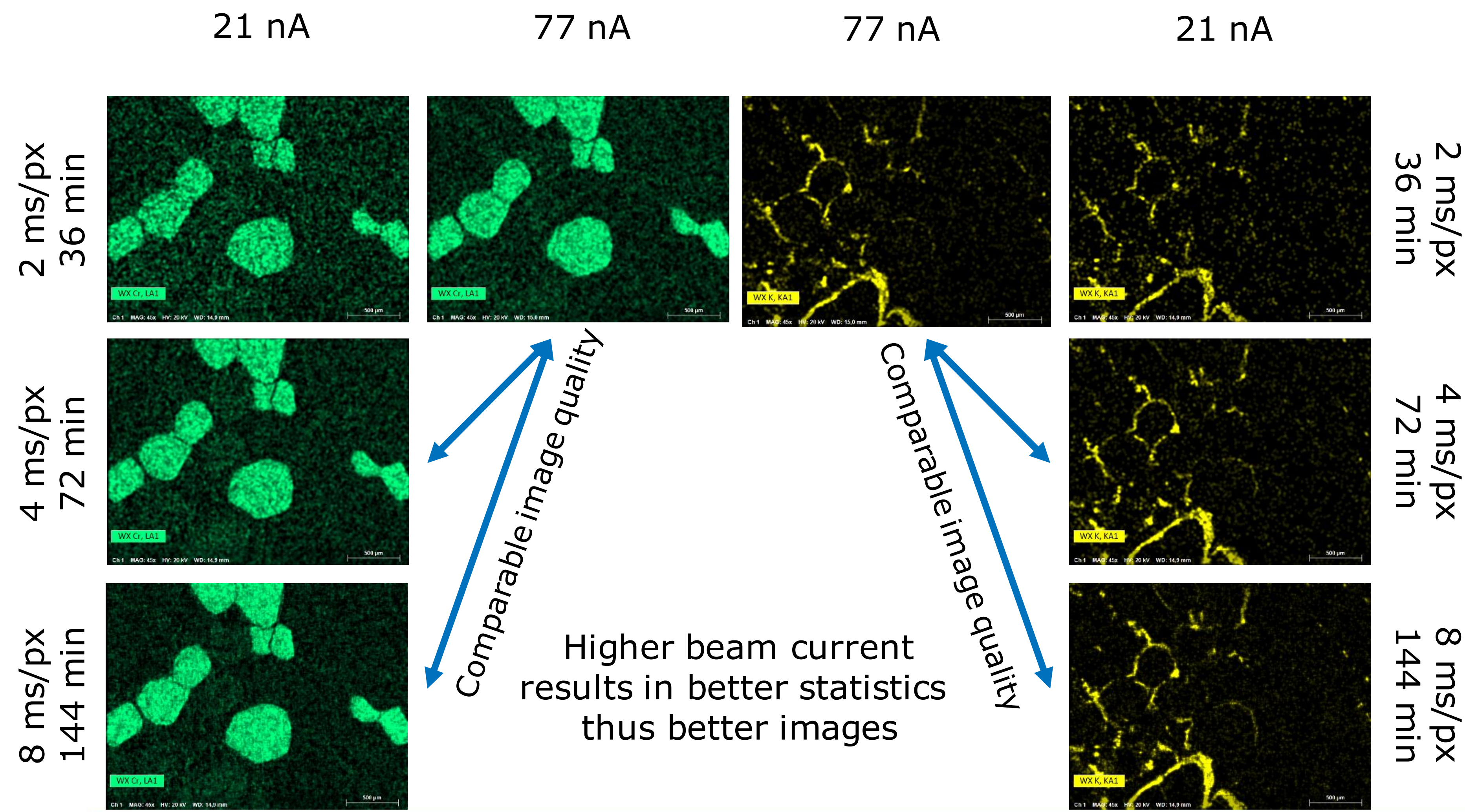
0.9 ms/px 15 min	2 ms/px 36 min	4 ms/px 72 min	8 ms/px 144 min



Increasing Information

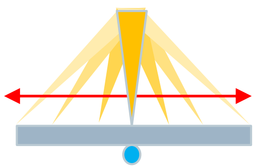
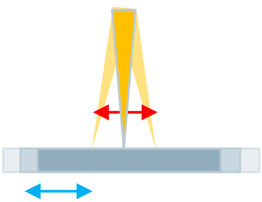
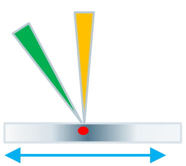
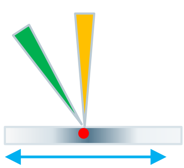
Increasing Analytical Speed

Stage Mapping using Rapid Stage
Effect of beam current



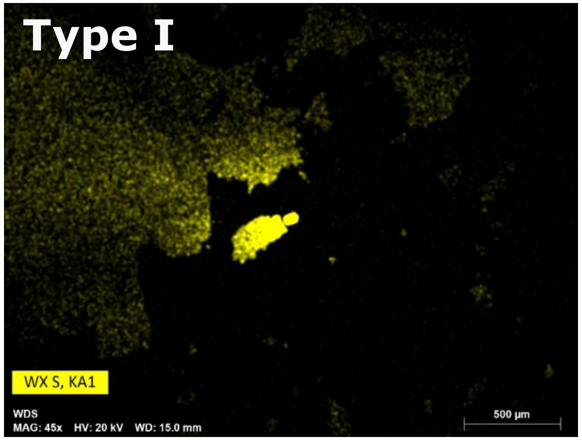
Higher beam current results in better statistics thus better images

Large Area Mapping Summary
Mapping Types I, II, IIIa and IIIb

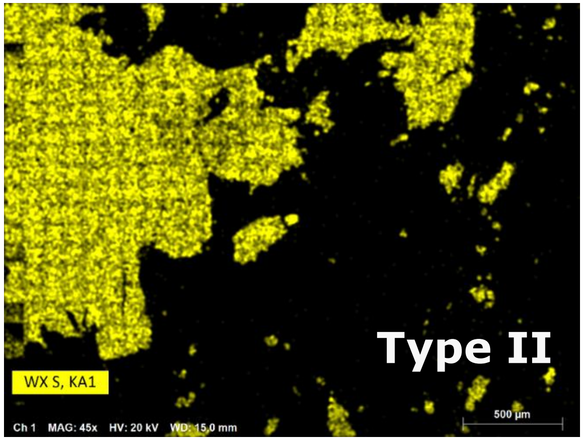





	FOV (mm)	FOV (px)	WDS Dwell time (ms/px)	Time /image (min)	Remarks
Type I: Raster Map	2.9 x 2.2	1200 x 900	8	60	**Lateral intensity loss**
Type II: Raster Map with Tiles	2.9 x 2.2	1200 x 900	8	360	**Tiling may be visible**
Type IIIa: SEM Stage Map	2.9 x 2.2	1200 x 900	8	60,480	**Extremely slow**
Type IIIb: Rapid Stage Map	2.9 x 2.2	1200 x 900	8	144	**Fast and correct**

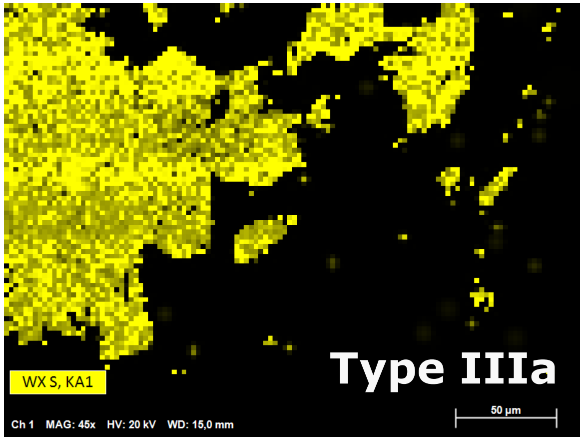
Comparing mapping types
Sample 1 at low magnification



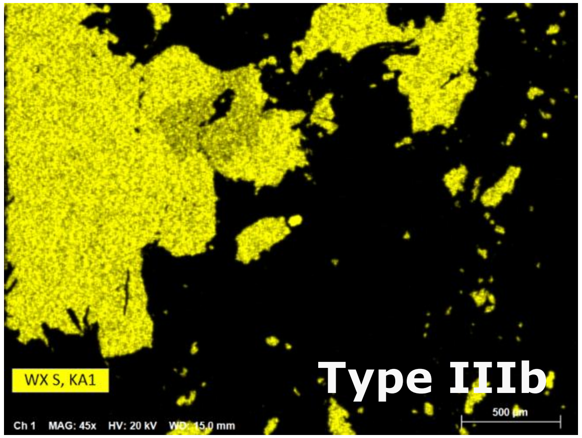
- ✓ Extremely fast
- • Inaccurate at low magnificat ion



- ✓ Relatively fast
- • Tiling may be visible



- ✓ Accurate
- ✓ Extremely slow



- ✓ Very fast
- ✓ Accurate

Summary and Conclusions:
Rapid Stage Benefits

Fast mapping

- Increase in speed by significant factor:
  - Especially for WDS and SEM-XRF use
- Enhancing WDS and SEM-XRF usability
- SEM Stage communication not designed for on-the-fly measurment

Accurate results

- Mapping with a vertical beam
- Avoiding violation of Bragg conditions
- Avoiding lateral intensity loss
- Supports simultaneous analysis with e-beam and X-ray beam or EDS and WDS.

Summary and Conclusions:
Rapid Stage and Micro-XRF

- Can work in combination with SEM e-beam
  - Commonly Low-KV due to charging and sample interaction

- The analysis of samples in mircometers (µm) scale on a standard SEM.
- Able to perform large area maps on a variety of samples.

- Sample Preparation Minimal for Micro-XRF
  - No carbon-coating
  - No polishing
  - Directly into the SEM

- Able to detect and resolve minor and trace elements
- Identification of high energy X-Ray lines

Questions and Answers

Are There Any Questions?

Please type in the questions you might have
in the Q&A box and press *Send*.

More Information

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