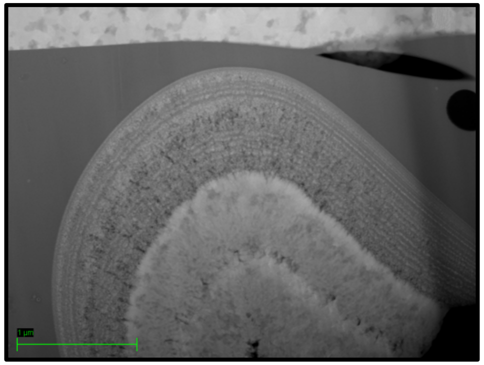
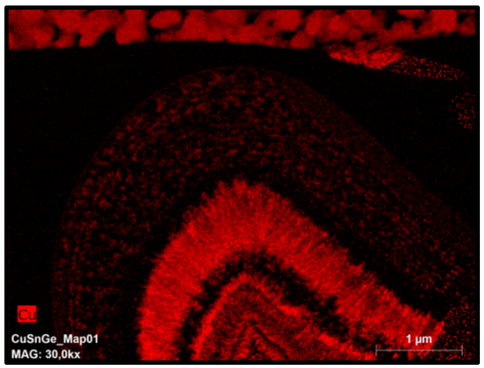
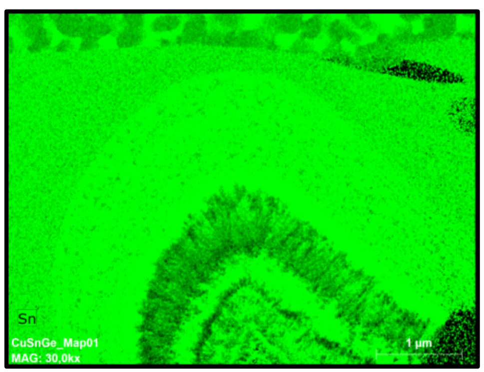
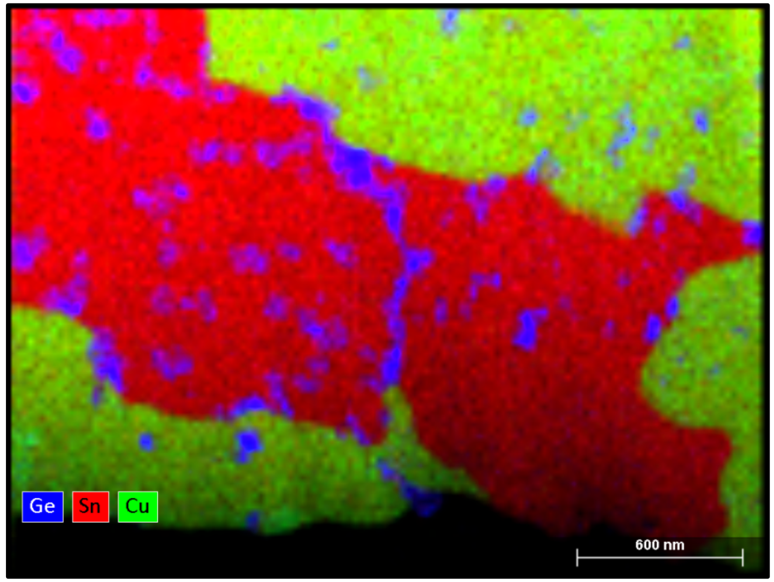
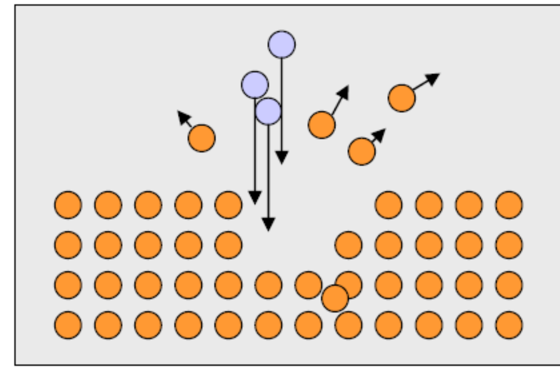










Advanced Electron Transparency Sample Preparation

Focused Ion Beam Techniques

Riza Iskandar

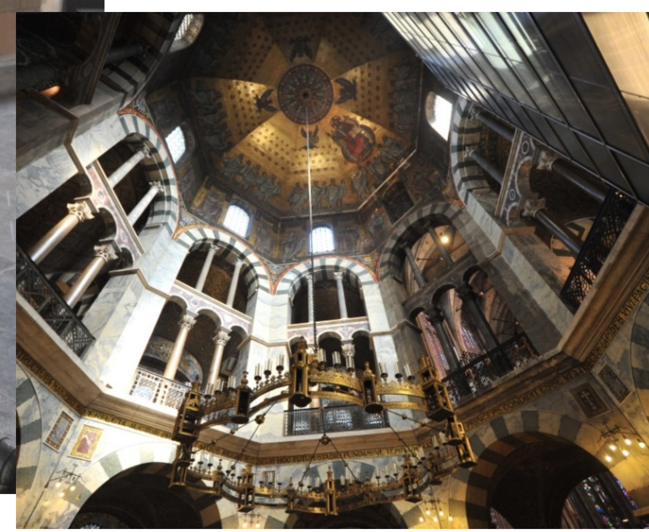
Workshop Laboratorium TEM UI 2017

Depok, November, 16^{th} 2017

Aachen

Krefeld
Düsseldorf
Mönchengladbach
Köln
Netherland
Germany
Aachen
Bonn
Lüttich
Belgium
Verviers
© 2012 Cnes/Spot Image
Image © 2012 GeoContent
© 2009 GeoBasis-DE/BKG
© 2012 Google
Google earth
Bildaufnahmedatum: 1/1/2005
50°57'15.43" N 6°13'58.72" O Höhe 91 m
Sichthöhe 112.43 km
US Dept of State Geographer
Data SIO, NOAA, U.S. Navy, NGA, GEBCO
© 2012 Google
Google earth
Sichthöhe 15213.99 km

Aachen



GFE

Central Facility for Electron Micrsocope (GFE)

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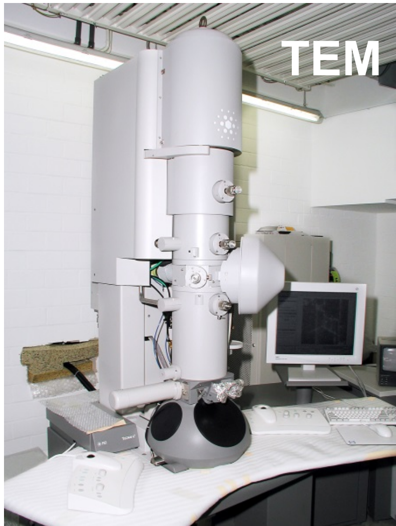
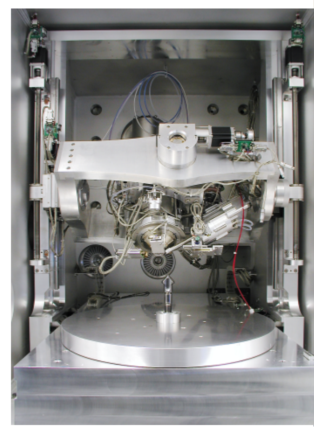


EPMA

AFM



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in der Helmholtz-Gemeinschaft

RWTH RHEINISCH-WESTFÄLISCHE TECHNISCHE HOCHSCHULE AACHEN

Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons

ER-C

GFE

ER-C

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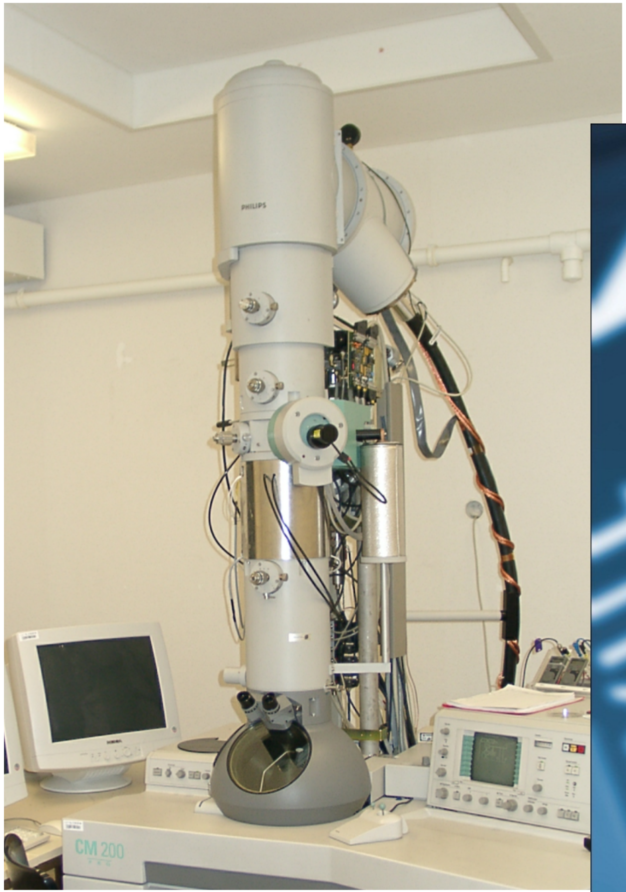
Cs-corrected protoype



Rose, Haider, Urban (1998)

Cs-corrected

FEI TITAN 80 – 300 (2006)

Cs/Cc-corr.

PICO (2011)

Three generations of aberration corrected HRTEMs

Outline

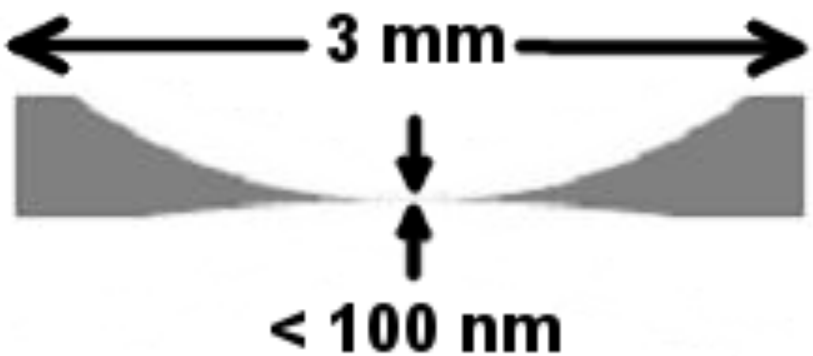
- Electron Transparency sample preparations
- Focused Ion Beam Techniques
  - Principle
  - Techniques
  - Advantages and Disadvantages
- Applications
  - Ceramics
  - Steel
  - Life Science

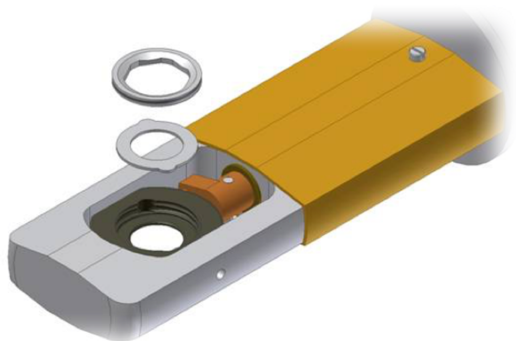
ELECTRON TRANSPARENCY SAMPLE PREPARATIONS

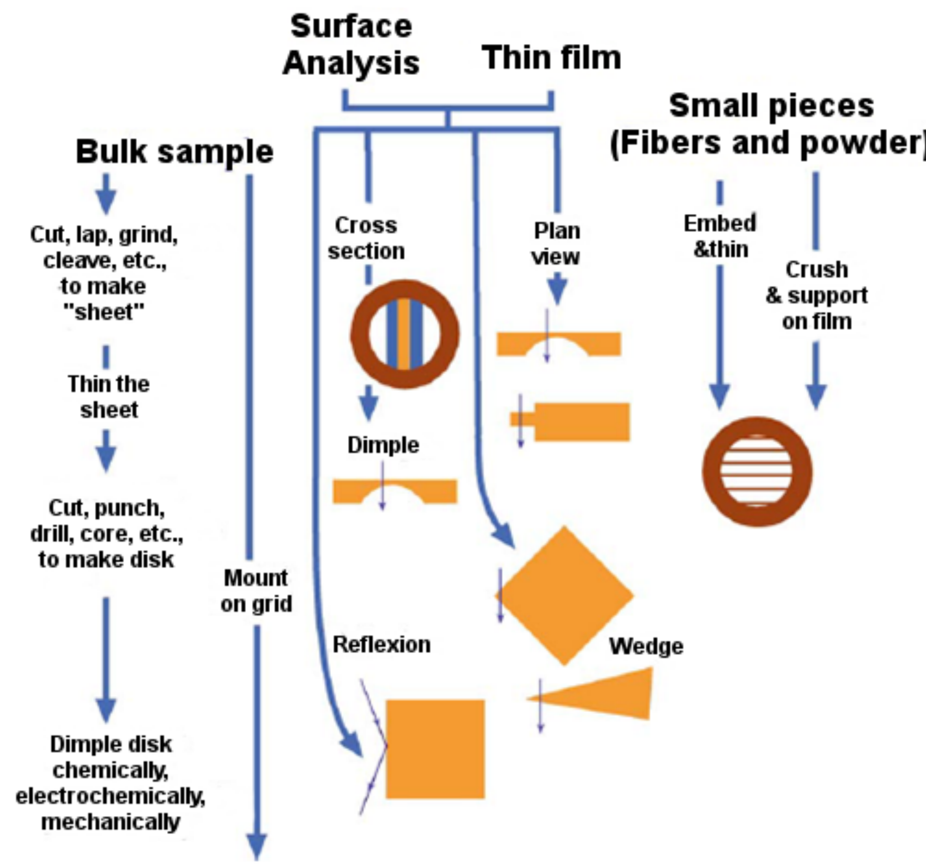
Electron Transparency Sample Preparations [1]

Specimen's requirements:

- Fit into TEM holder cup (3 mm disc)
- Electron transparent (< 100 nm)

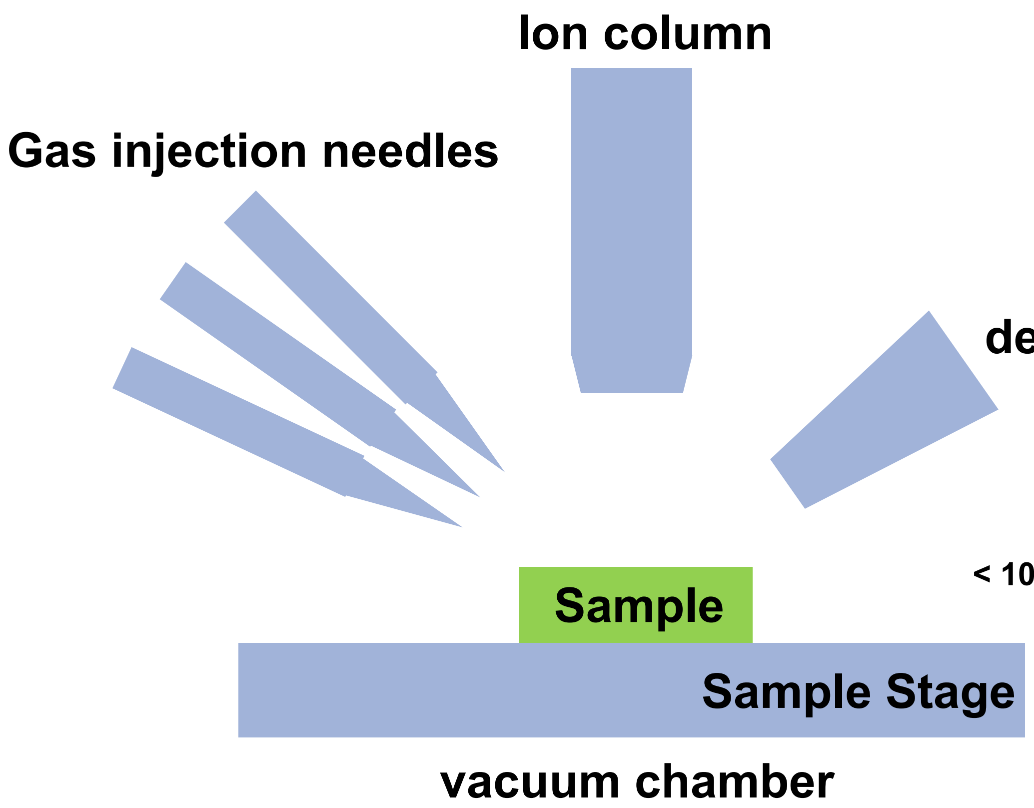


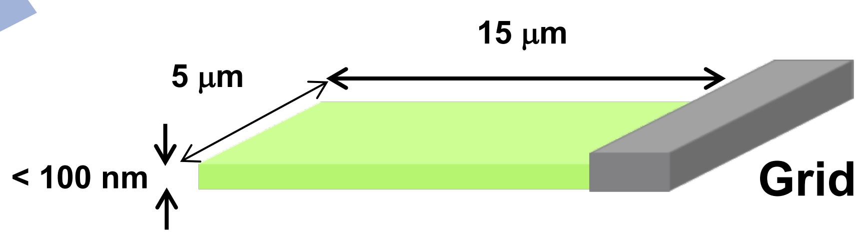




FOCUSED ION BEAM TECHNIQUES

Focused Ion Beam Chamber and Lamella

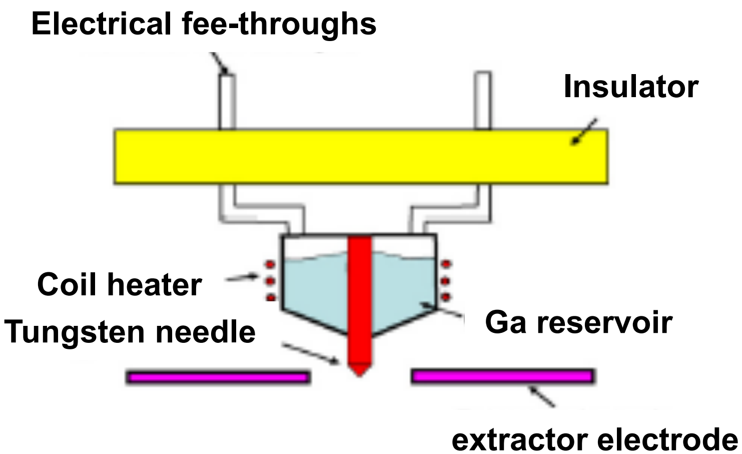




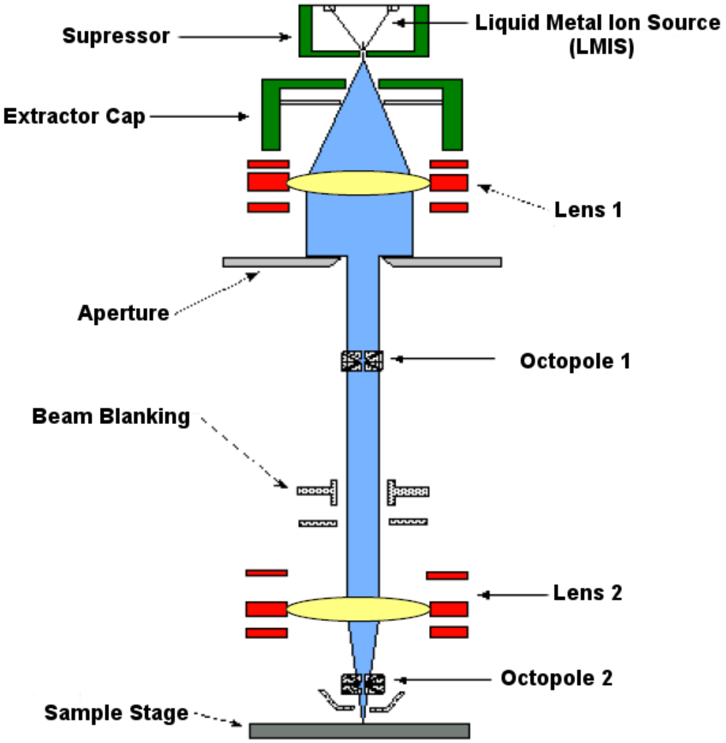
Focused Ion Beam Source

Basic Principle

- Use liquid metal ion sources (LIMS), eg. Ga
- Liquid flow from reservoir
- Ion formation
- External beam interaction



Schematic diagram of Ga LMIS [2]



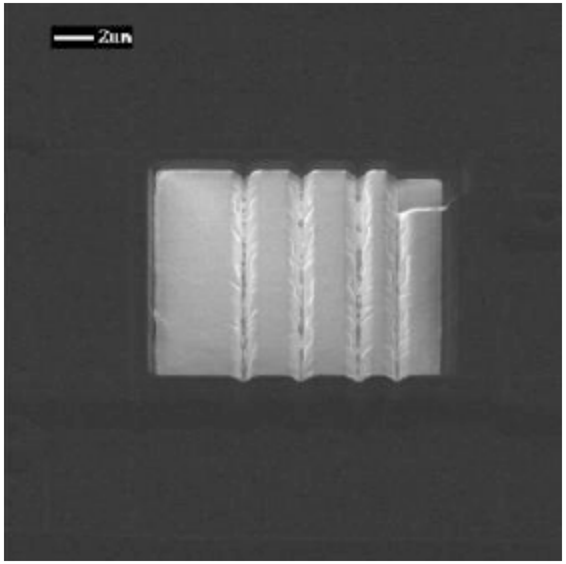
Quantitative comparison of FIB ions and SEM electrons [3]

Table 1.1 *Quantitative comparison of FIB ions and SEM electrons*

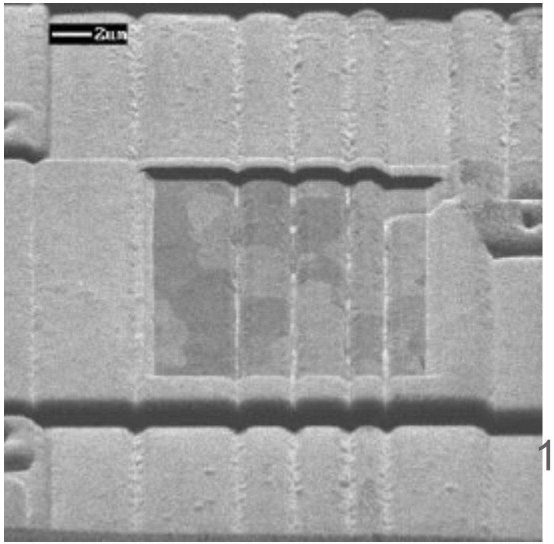
Particle	FIB	SEM	Ratio
Type	Ga^{+} ion	Electron	
Elementary charge	+1	–1	
Particle size	0.2 nm	0.00001 nm	20 000
Mass	1.2×10^{-25} kg	9.1×10^{-31} kg	130 000
Velocity at 30 kV	2.8×10^{5} m/s	1.0×10^{8} m/s	0.0028
Velocity at 2 kV	7.3×10^{4} m/s	2.6×10^{7} m/s	0.0028
Velocity at 1 kV	5.2×10^{4} m/s	1.8×10^{7} m/s	0.0028
Momentum at 30 kV	3.4×10^{-20} kg m/s	9.1×10^{-23} kg m/s	370
Momentum at 2 kV	8.8×10^{-21} kg m/s	2.4×10^{-23} kg m/s	370
Momentum at 1 kV	6.2×10^{-21} kg m/s	1.6×10^{-23} kg m/s	370
Beam			
Size	nm range	nm range	
Energy	up to 30 kV	up to 30 kV	~
Current	pA to nA range	pA to µA range	~
Penetration depth			
In polymer at 30 kV	60 nm	12000 nm	0.005
In polymer at 2 kV	12 nm	100 nm	0.12
In iron at 30 kV	20 nm	1800 nm	0.11
In iron at 2 kV	4 nm	25 nm	0.16
Average signal per 100 particles at 20 kV			
Secondary electrons	100–200	50–75	1.33–4.0
Backscattered electron	0	30–50	0
Substrate atom	500	0	infinite
Secondary ion	30	0	infinite
X-ray	0	0.7	0

Secondary Electron (SEM) vs Secondary Ion Mode (FIB)

- Secondary electron
  - Detector biased positive
  - Images generated from e-
  - Emitted from top 50 – 100 Å
  - Only charging up a few volts to go dark
  - Grounded metals very bright, oxides dark
- Secondary ion mode
  - Detector biased negative
  - Images generated with I+
  - Emitted from top 5-10 Å
  - Very surface sensitive
  - No voltage-contrast
  - Oxides brighter
  - Less yield, so images noisier



SE Image

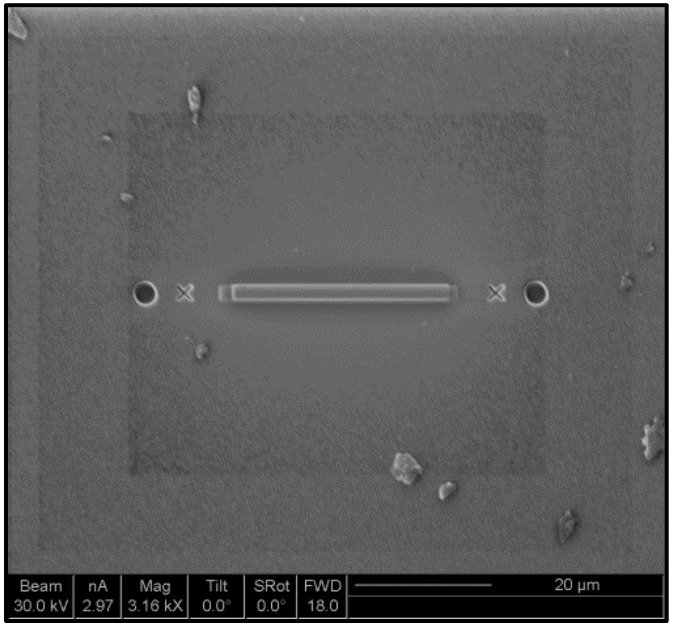


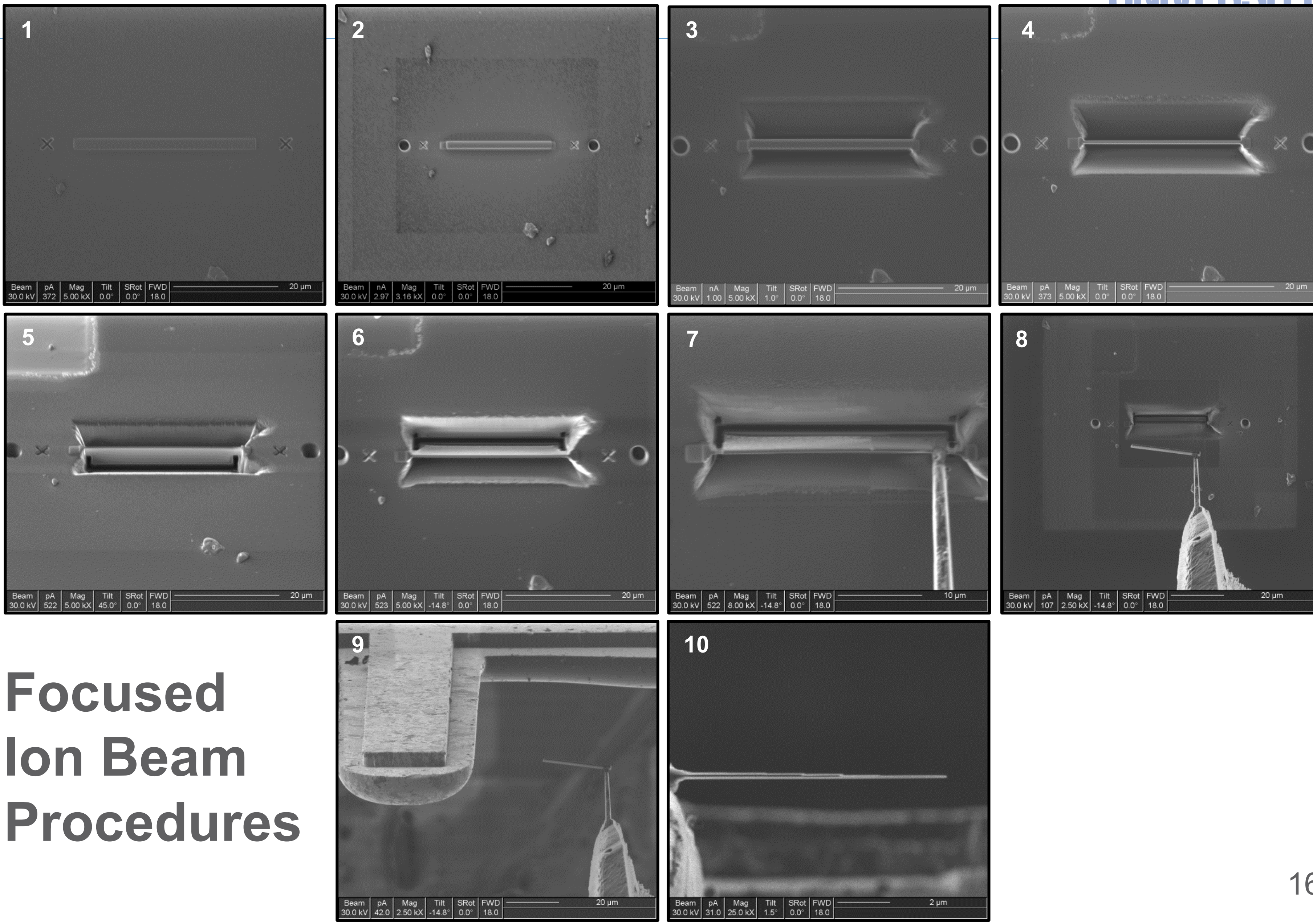
SI Image

Depositing protection layer

- Platinum
  - (methylcyclopentadienyl) trimethyl platinum - $C_5H_4CH_3Pt(CH_3)_3$
  - Solid at room temperature
  - Operating temperature 38-42°C
  - About 10 minute warm-up period
  - User refillable (use fume hood)
  - Very hard: tougher for probing and thermal cycling
  - Chemically resistant

- Tungsten
  - Tungsten hexacarbonyl - $W(CO)_6$
  - Lower resistivity than Pt (better for circuit edit)
  - Slower deposition than Pt
  - Solid at room temperature
  - User refillable
  - Operates at 50° C

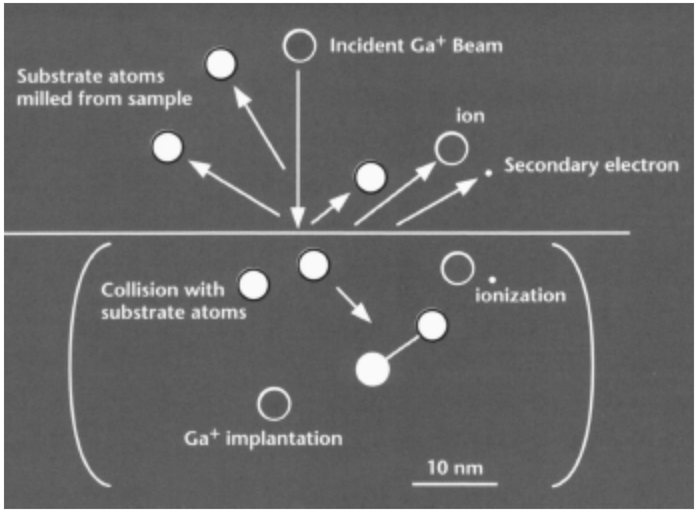




Focused Ion Beam Procedures

What a FIB does (but a SEM does not) [4]

- Removes material
- Adds material
- Secondary ion imaging shows material contrast
- Channelling contrasts
- Prepares sample in situ
- Combines high magnification imaging and sample modification



Interactions of the ion beam with sample surface. The unique control offered by beam currents and spot sizes allow use of the FIB for both nano engineering as well as for high resolution imaging using secondary electrons as well as ions [5].

Advantages vs Disadvantages

- Advantages
  - Reduces preparation time
  - Prepare almost any kind of materials
  - Large homogenous sample thickness
  - Specific area can be selected
  - Can be combined with other analysis techniques

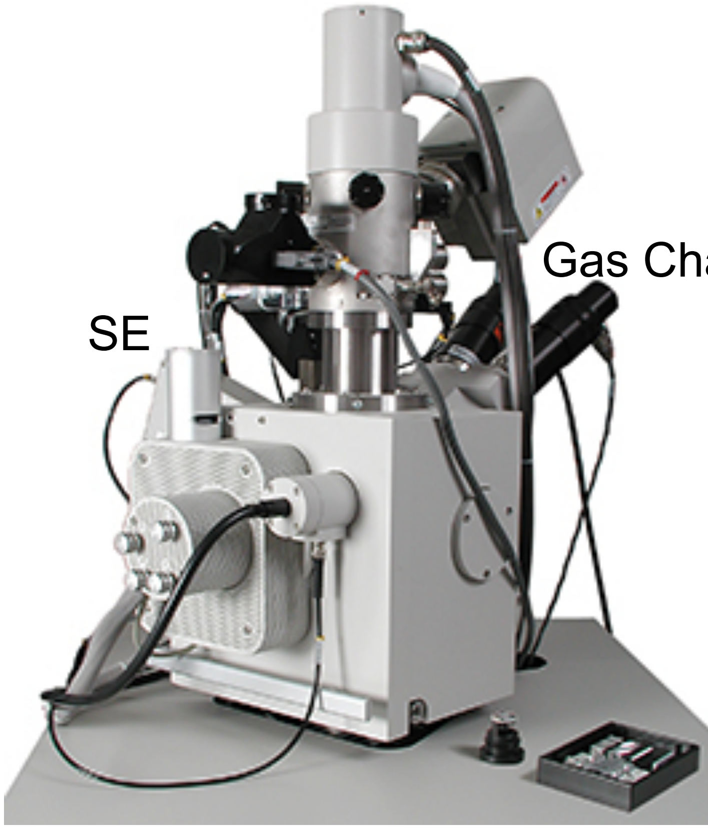
- Disadvantages
  - Sample thickness less than 50 nm forms amorphous layer
  - Localized, needs prior analysis techniques

Single vs Dual Beam Focused Ion Beam

FIB

SE

Gas Chamber



SEM

EDS
+ WDS

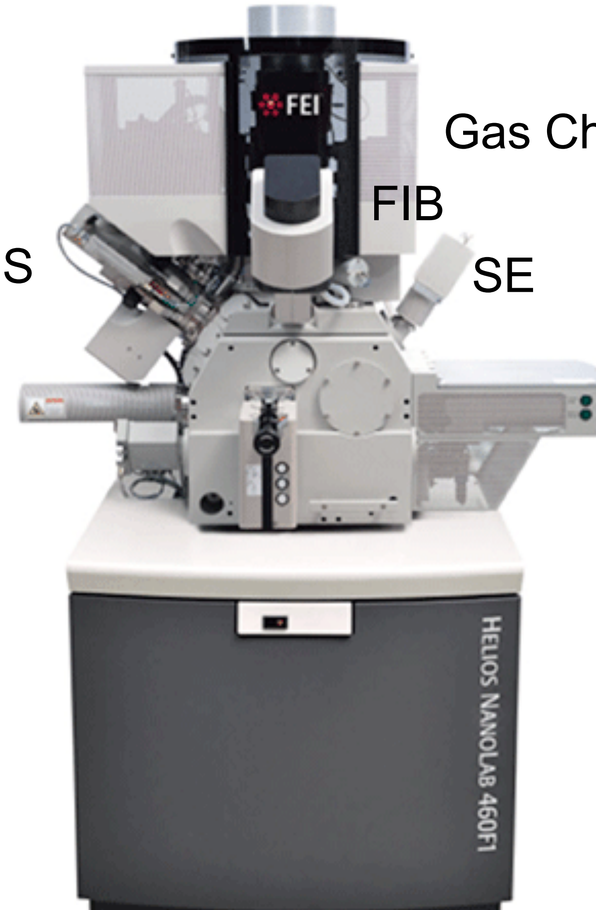
BSE

Gas Chamber

FIB

SE

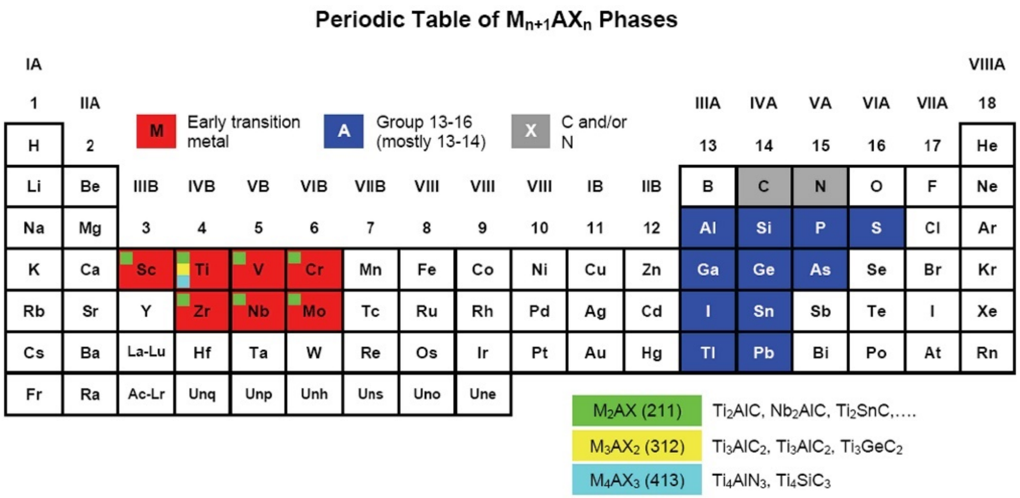
EBSD

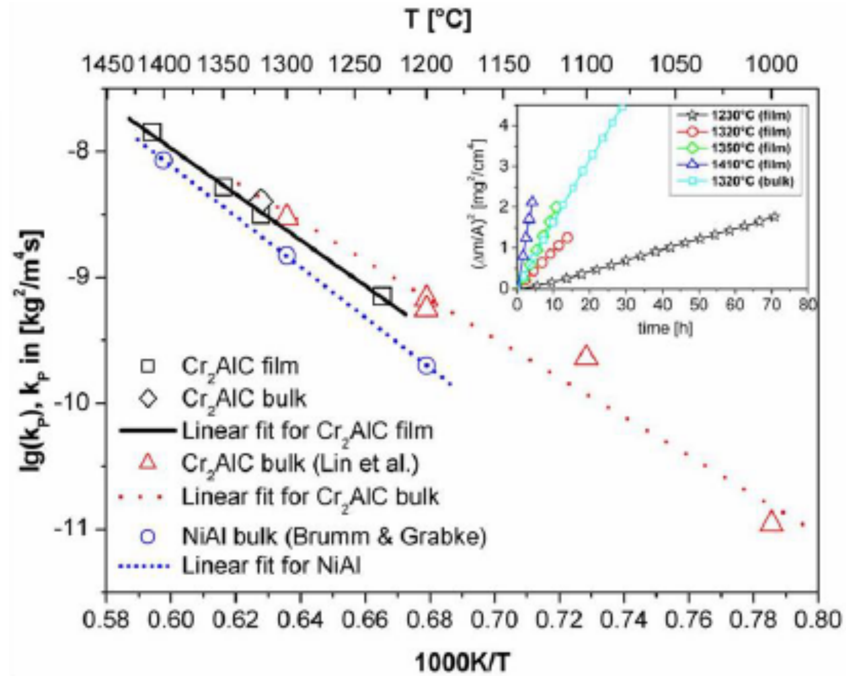


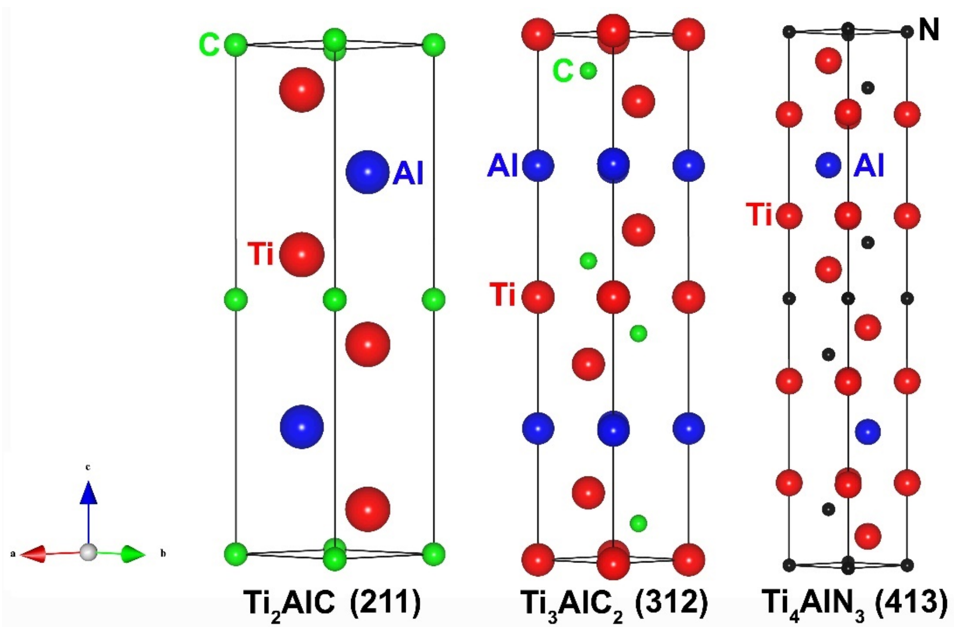
APPLICATIONS

OXIDATION RESISTANCE OF MAX PHASE

MAX Phase [6,7]

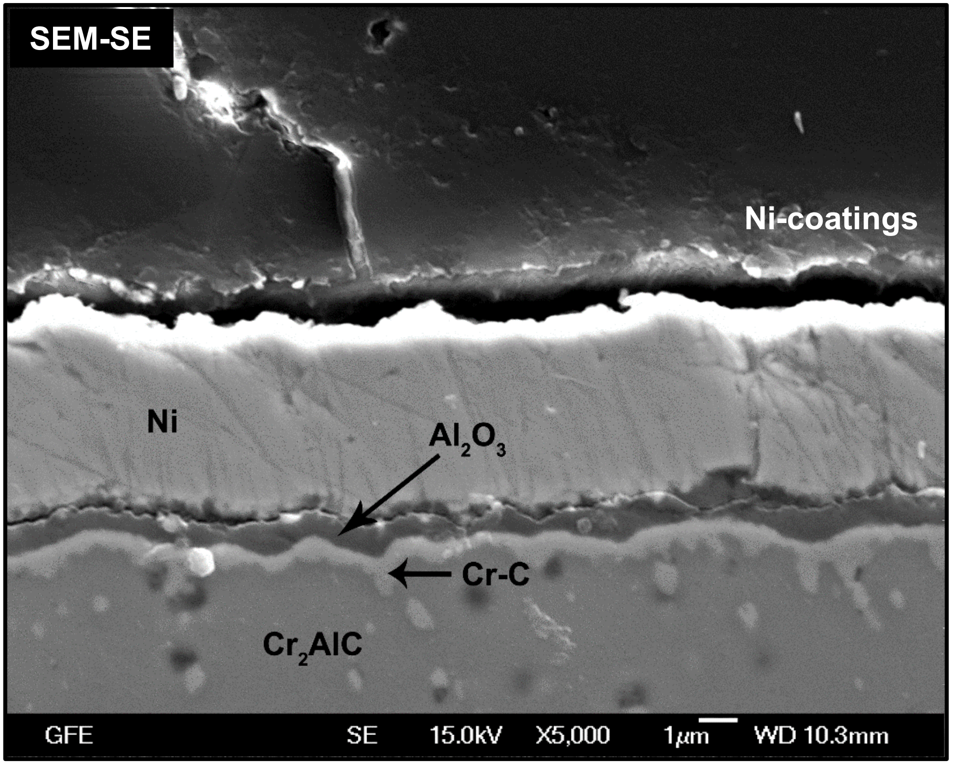


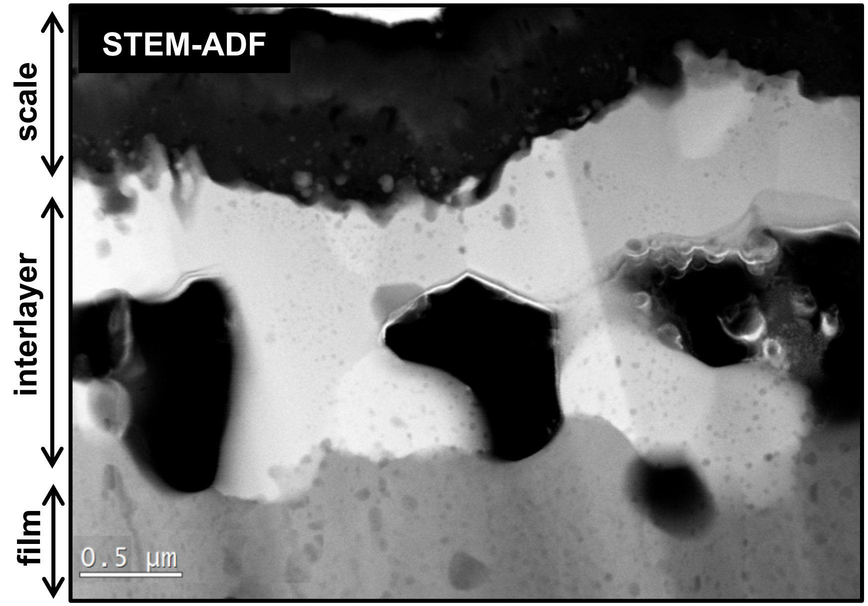


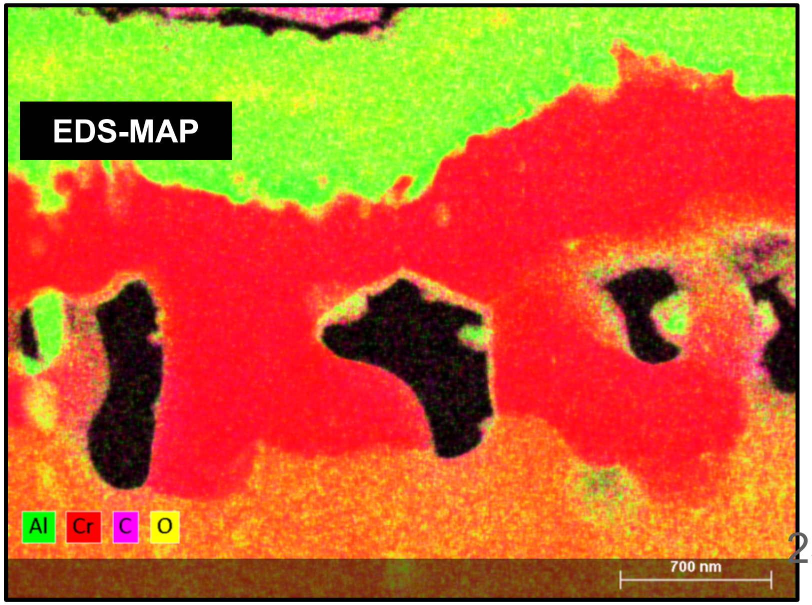


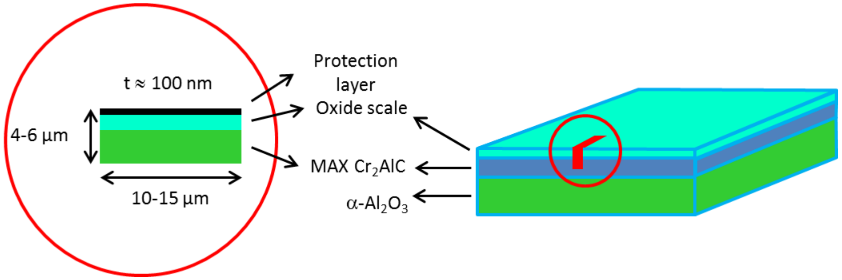
- Exhibit both metals and ceramic properties
- Exhibit ductility at room temperature
- Chemical stability at high temperatures.
- Excellent thermal shock resistance
- Possess a high thermal conductivity
- Machinable

SEM, STEM and EDS Map [8]

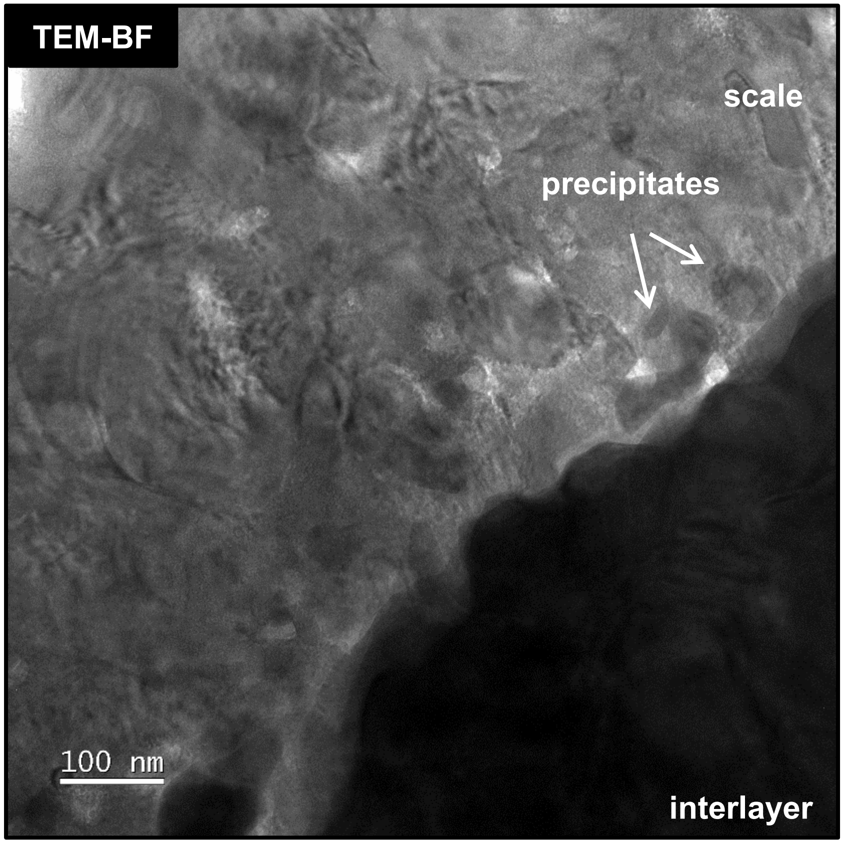


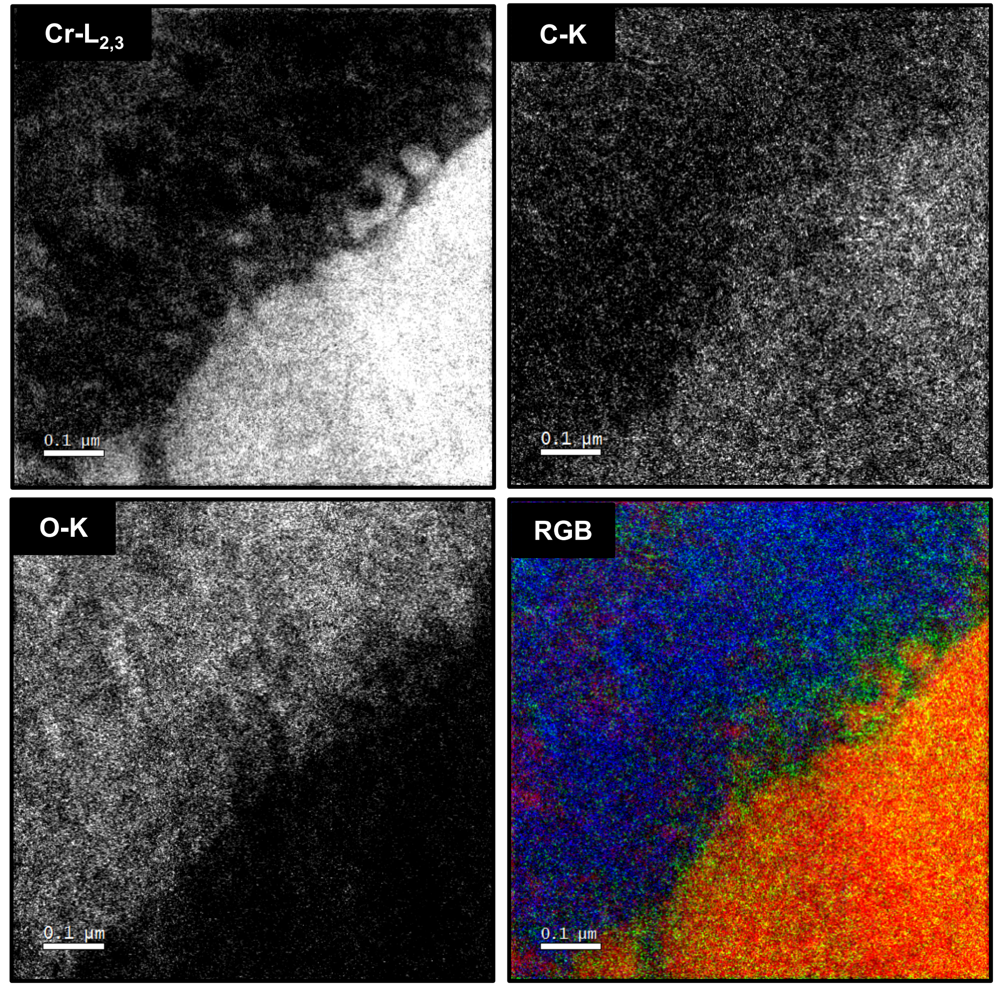






EFTEM elemental maps





Red: Cr; Green: C; Blue: O

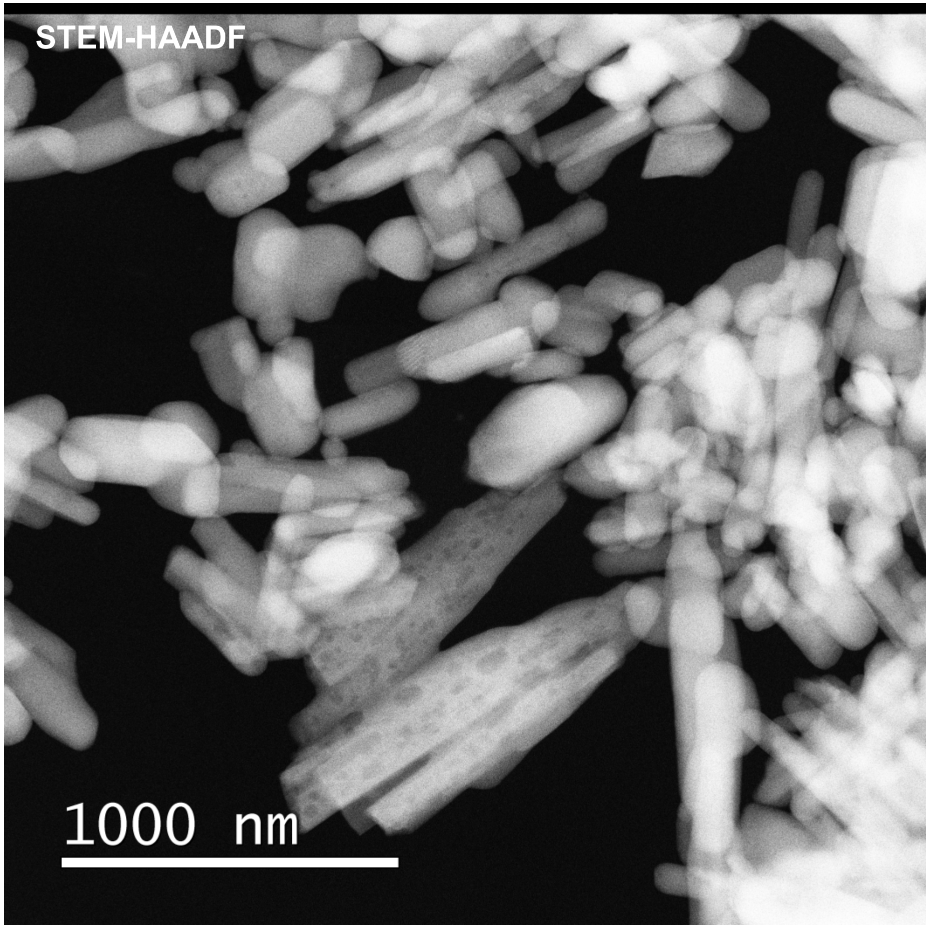
- Interlayer contains no oxygen
- Slightly C enrichment at precipitates area
- Cr-enrichment at outer scale

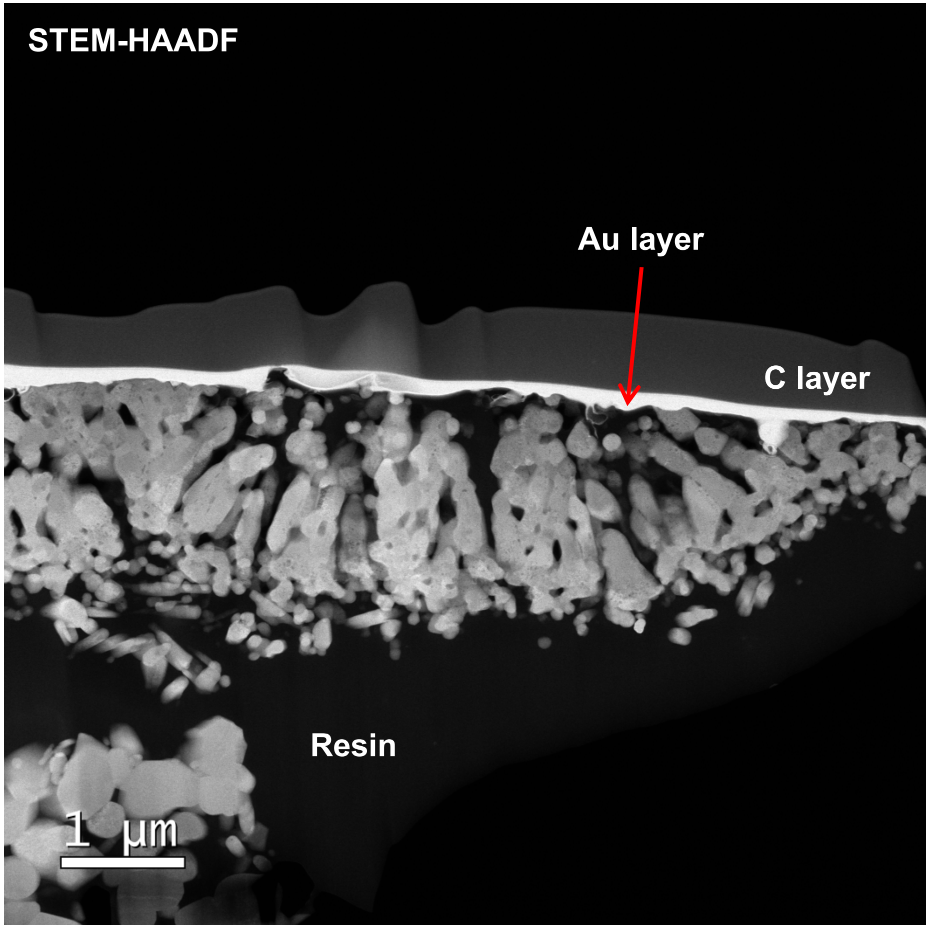
LITHIUM DETECTION ON ZNO NANO-PARTICLES WITH SMALL LITHIUM- ADDITION

Lithium detection on ZnO nano-particles with small Li-Addition

- To investigated effect of Li addition on ZnO nano particles structural properties

ZnO + 6at.% lithium

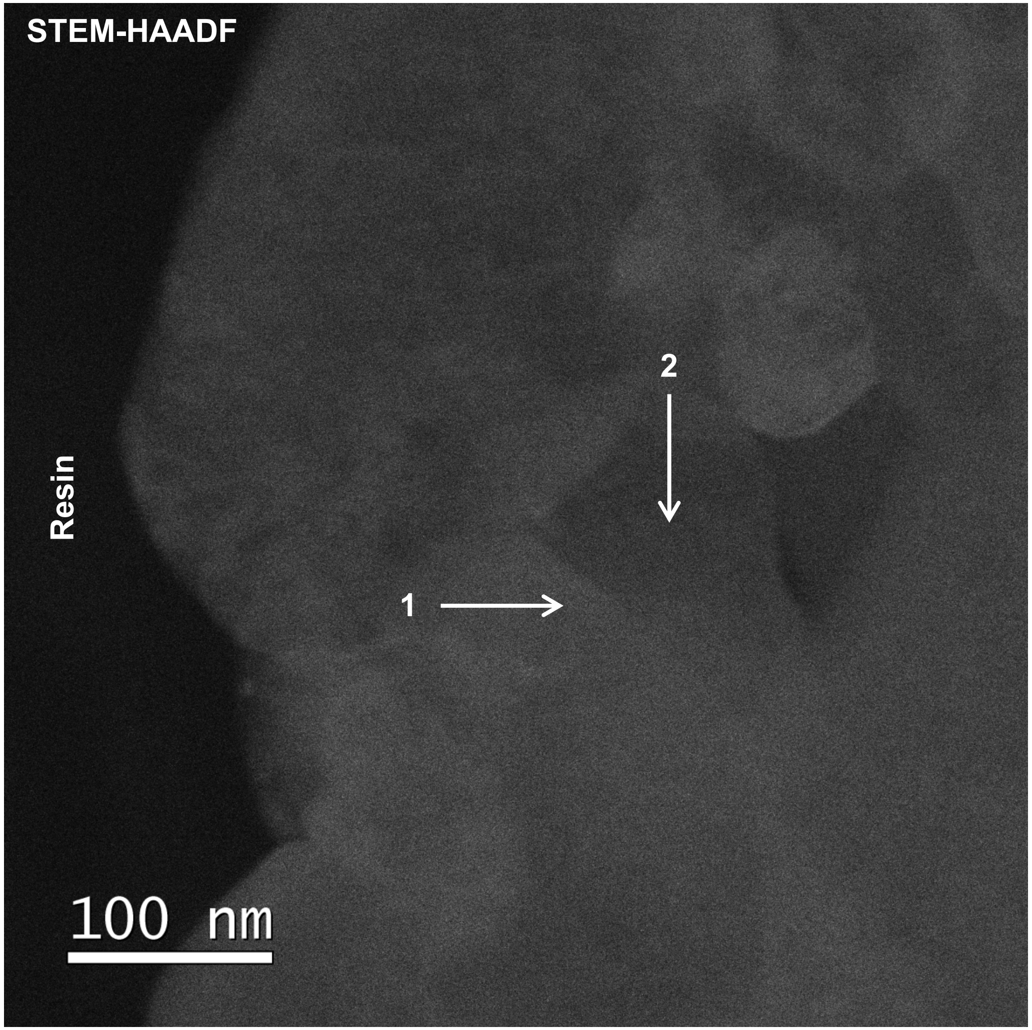


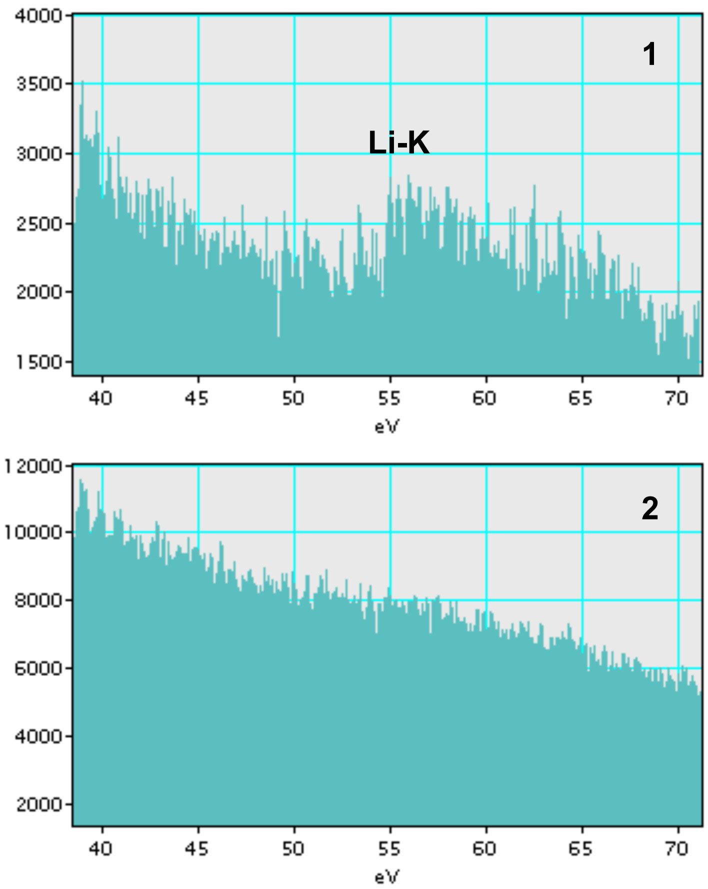


- Conventional preparation
- Lateral size app. 200 nm
- Impossible to detect Li

- Imbedded into resin
- Prepared with FIB techniques

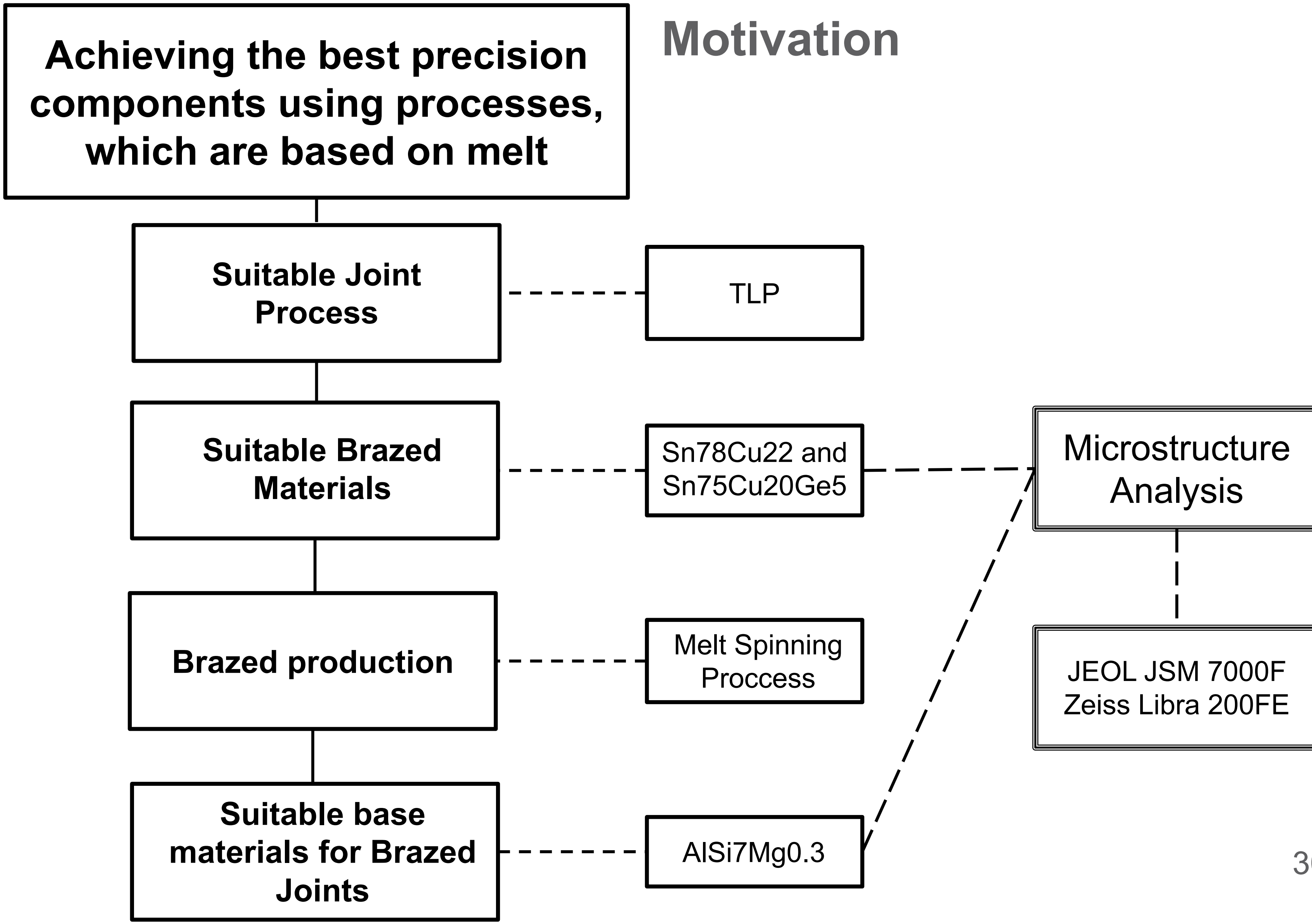
Lithium detection with Electron Energy Loss Spectroscopy (EELS)





MICROSTRUCTURAL ANALYSIS OF GERMANIUM MODIFIED TIN-COPPER BRAZING FILLER METALS FOR TRANSIENT LIQUID PHASE (TLP) BONDING OF ALUMINUM [9]

Motivation

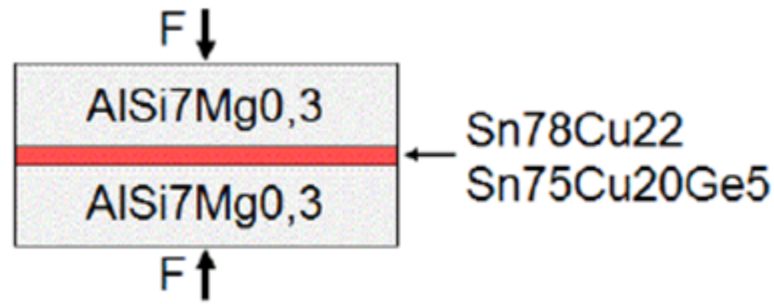

Achieving the best precision components using processes, which are based on melt
Suitable Joint Process
TLP
Suitable Brazed Materials
Sn78Cu22 and Sn75Cu20Ge5
Microstructure Analysis
Brazed production
Melt Spinning Proccess
JEOL JSM 7000F Zeiss Libra 200FE
Suitable base materials for Brazed Joints
AlSi7Mg0.3

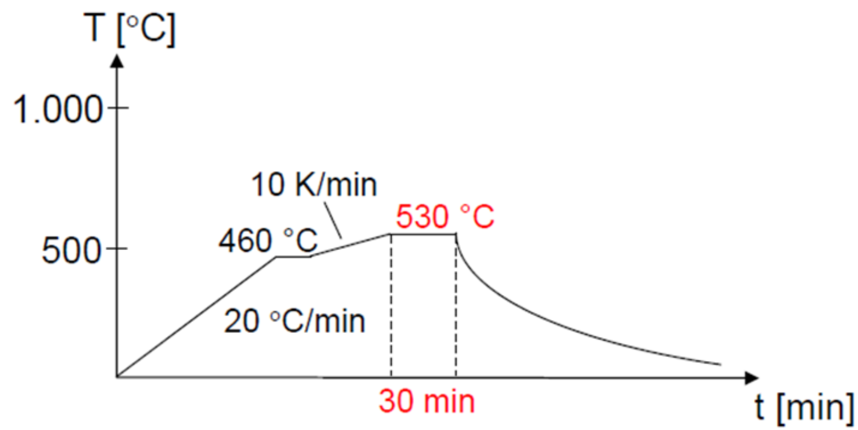
Transient Liquid Phase Bonding

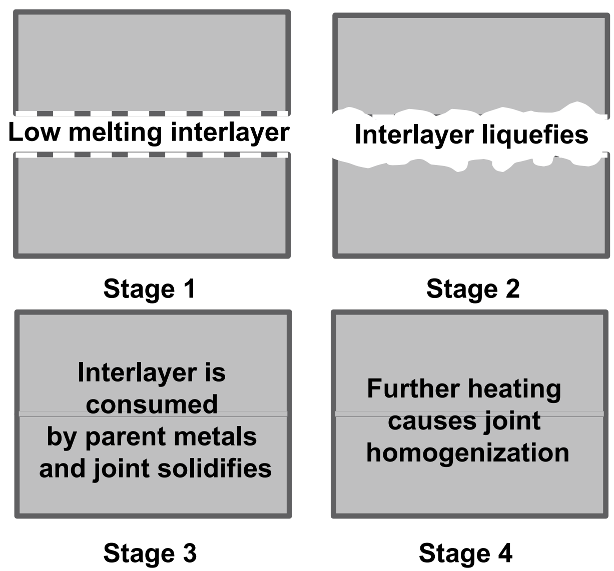
The Microstructure and mechanical properties of TLP bond approximately those of the substrate material

A bond can be formed at a temperature much lower than the melting point of the resulting joint.

Thin liquid interlayer helps to eliminate the high bonding or clamping force







AlSi7Mg0,3

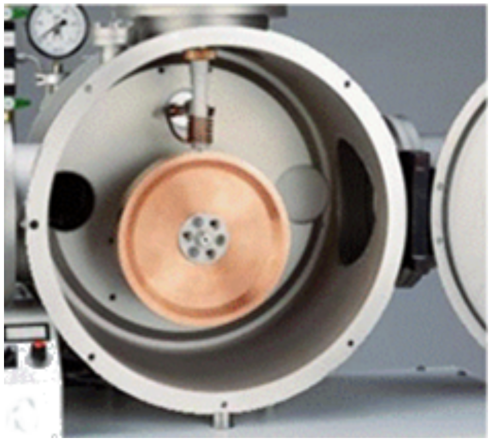
T_{eut} = 577 °C

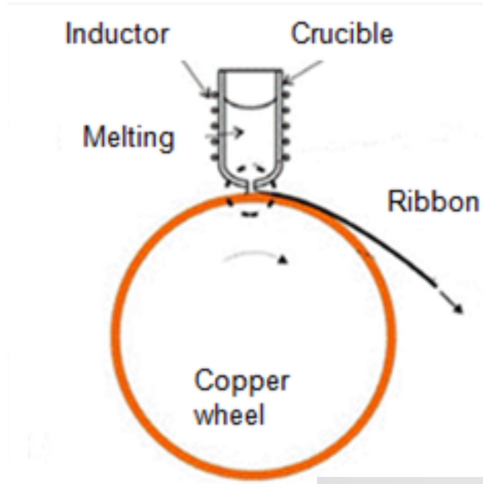
Process

T_{joint} = 530 °C

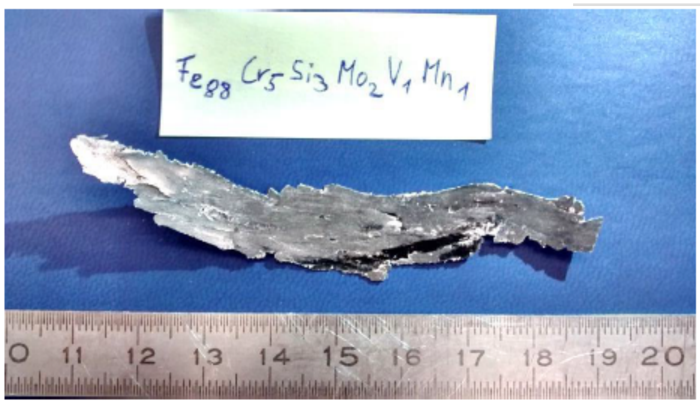
T_{joint} = 30 min

Melt Spinning Process





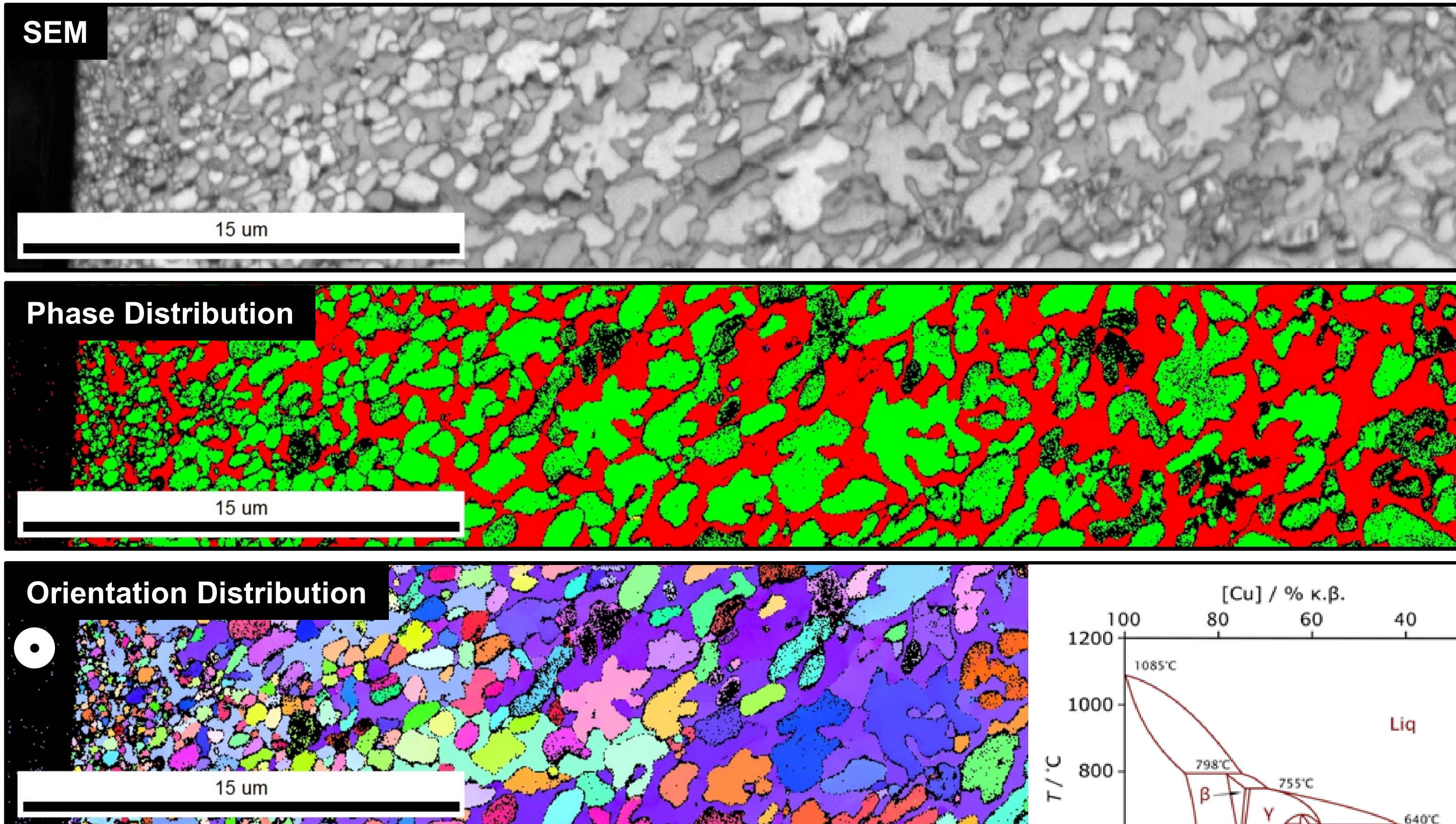
Process parameter	Value
Extrusion pressure	200 mbar
Rotation frequency of the copper wheel	30 Hz
Crucible nozzle width	0,5 cm
Crucible gap width	0,4 mm



- To thick (> 500 µm)
- In homogenous phase distribution
- Brittel an tends to fracture

- Thin (about 300 µm)
- Homogenous phase distribution
- Flexible

EBSD Analysis - SnCuGe Brazed Ribbon



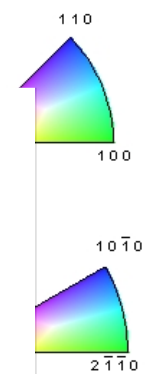
Gray Scale Map Type:<none>

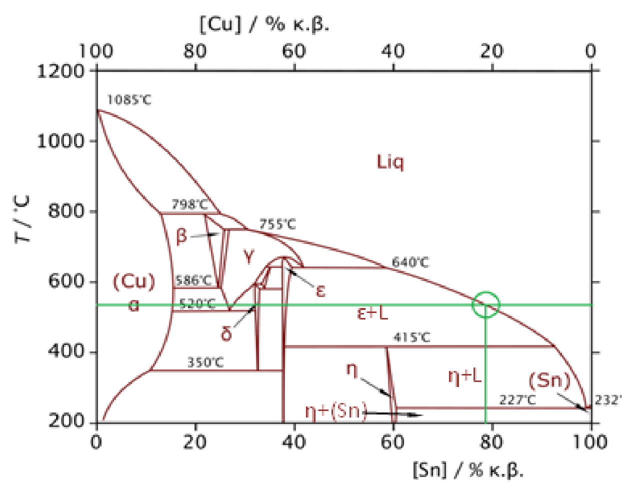
Color Coded Map Type: Phase

Phase	Total Fraction	Partition Fraction
Tin	0.356	0.472
29278	0.397	0.526
29279	0.000	0.001
57878	0.000	0.000
Germanium	0.000	0.000

Boundaries: <none>

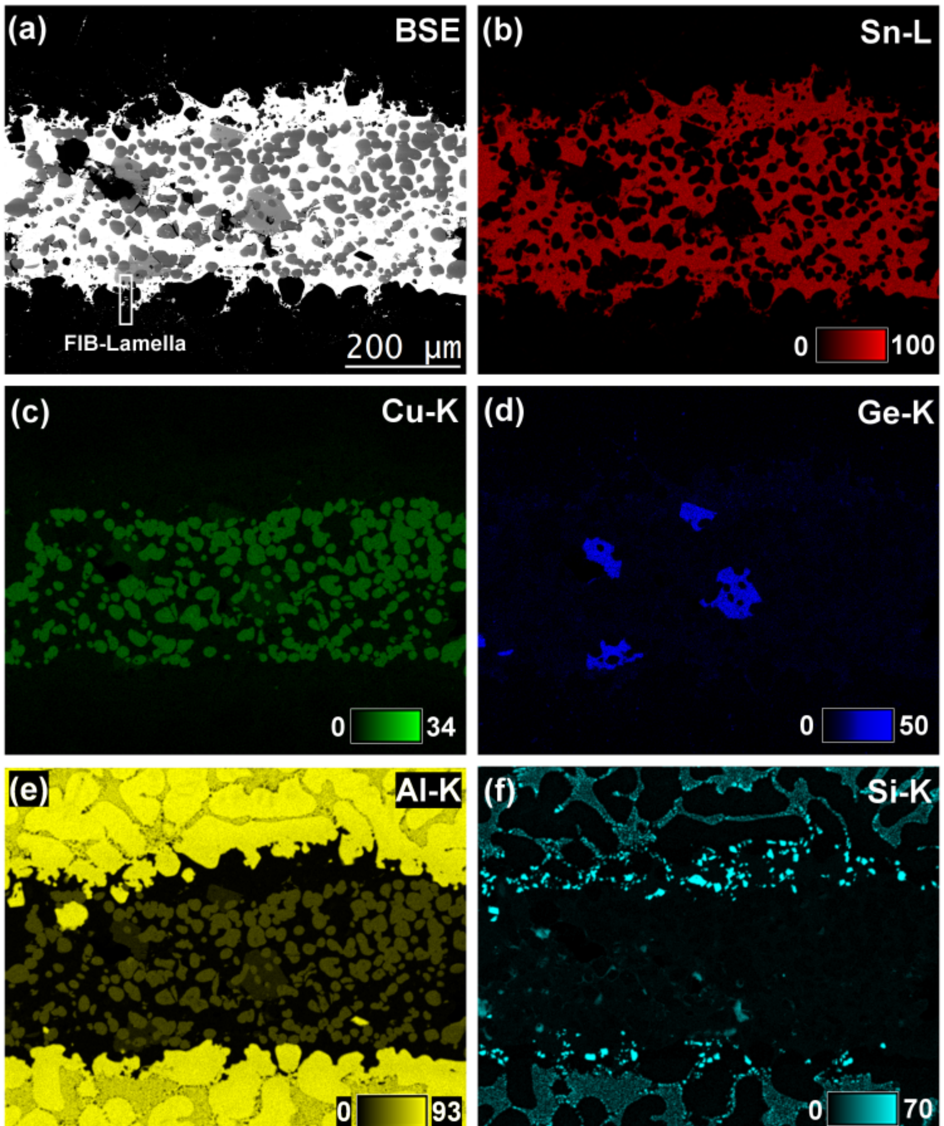
Tin

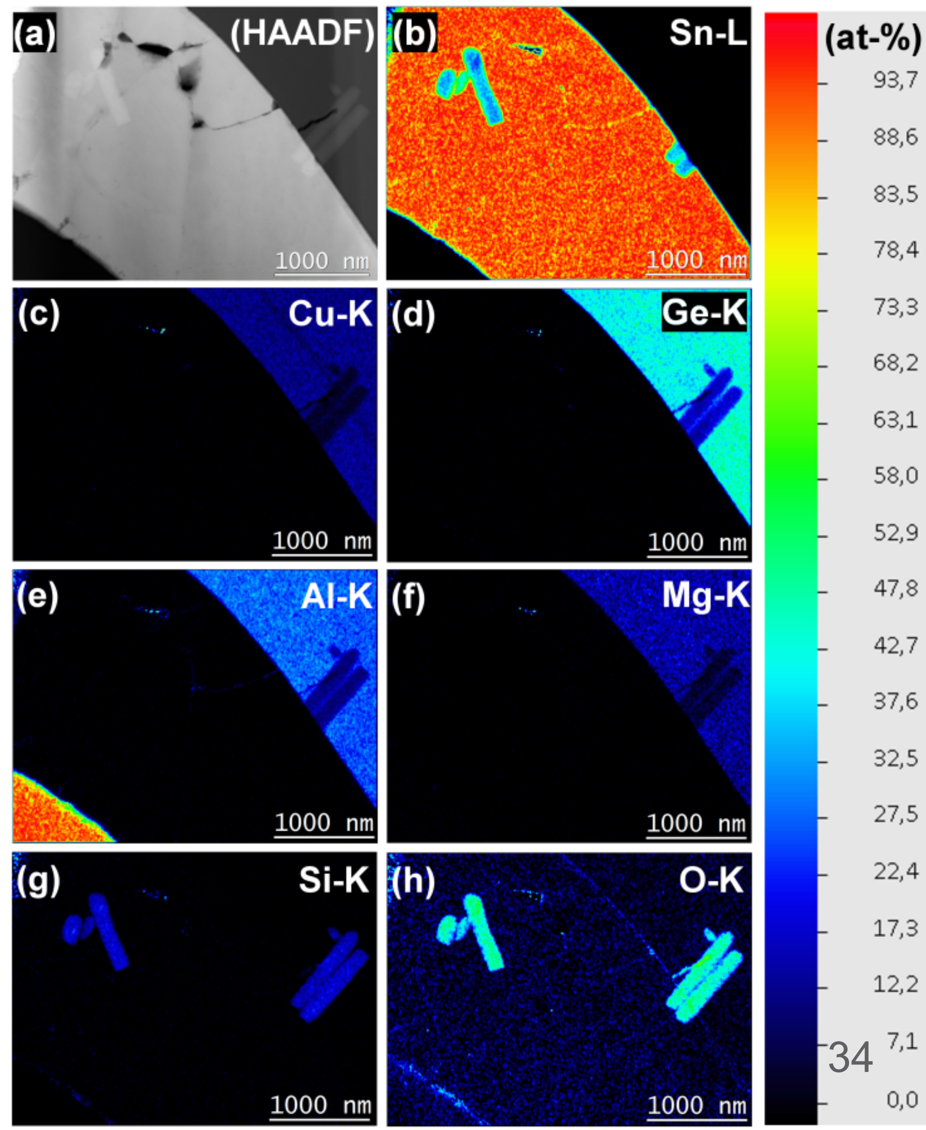




- The Sn phase has a preferred orientation in contrast
- Solidification of CuSn (η) phase in liquid matrix
- Solidification of Sn phase as a large crystals
- Different grain sizes indicates the solidification processes.

Analytical SEM and TEM SnCuGe on AlSiMg base material





SIGMA (σ)-PHASE FORMATION MECHANISMS IN HIGH CHROMIUM CONTENT IRON-CHROMIUM MODEL ALLOYS WITH SMALL ADDITION OF MOLYBDENUM FOR HIGH TEMPERATURE APPLICATION [10]

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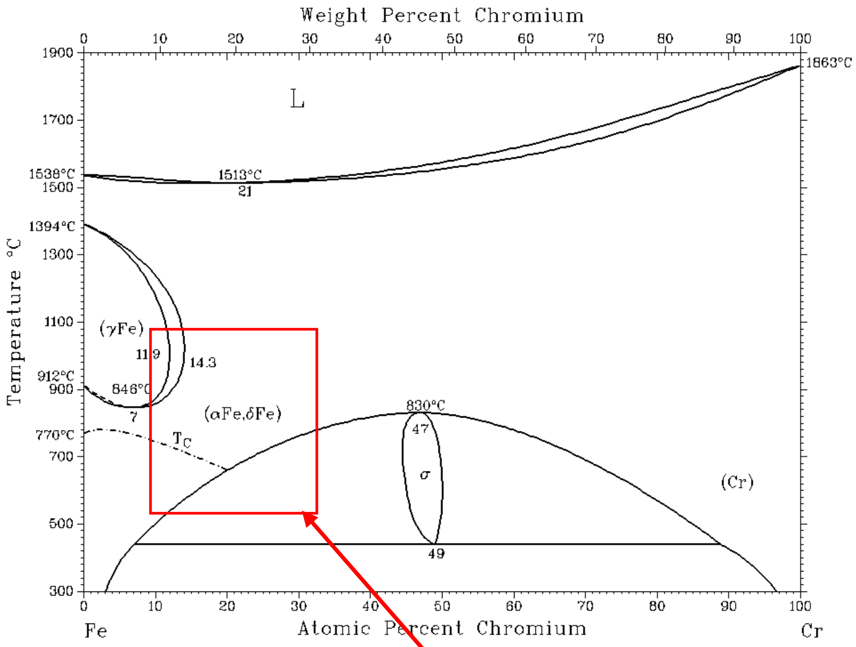
Sigma Phase

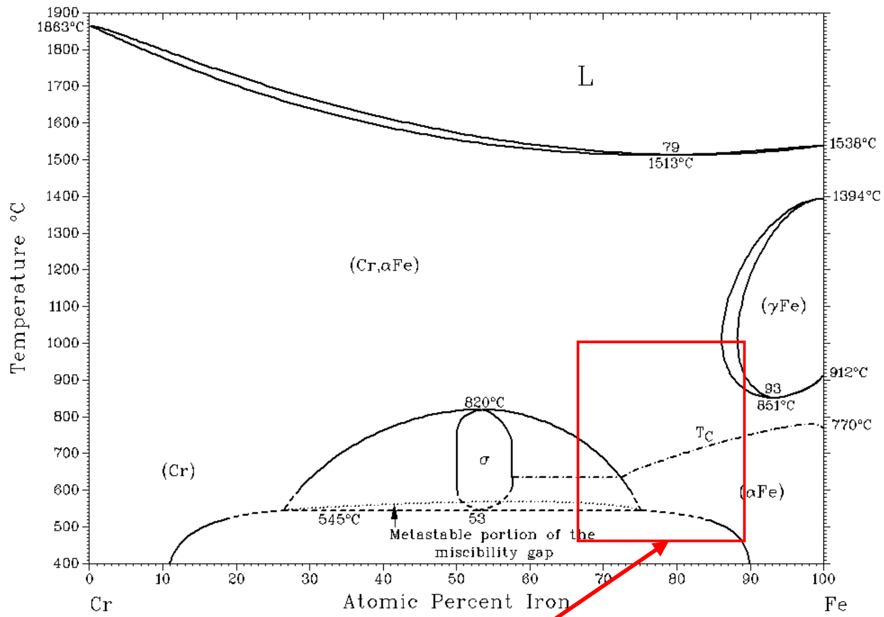
- Difficulties to accurately determining phase boundary of σ-phase due to [11]
  - Slowness of formation
  - Dependence of purity of alloys
  - Dependence of mechanical states
  - Dependence of temperature annealing
- For oxidation resistance
  - σ-phase on grain boundary consumes chromium and causes loss of oxidation resistance [12]
  - σ-phase stabilizing elements such as molybdenum

Old and New Fe-Cr Phase Diagrams

Old



New

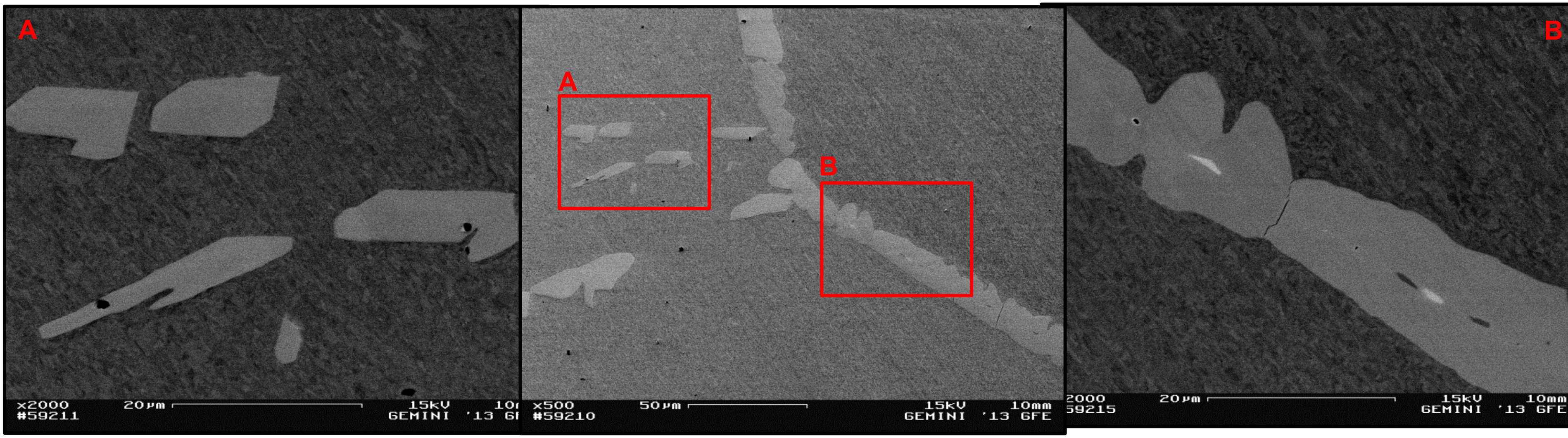


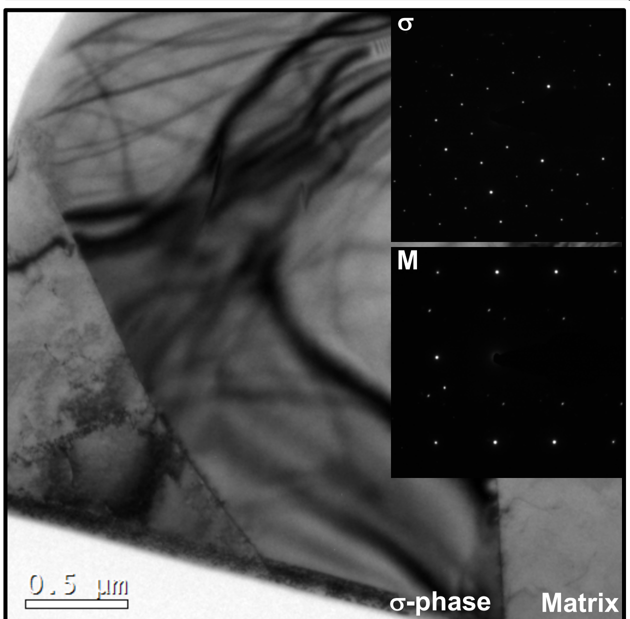
Range of Cr-contents for high temperature steels

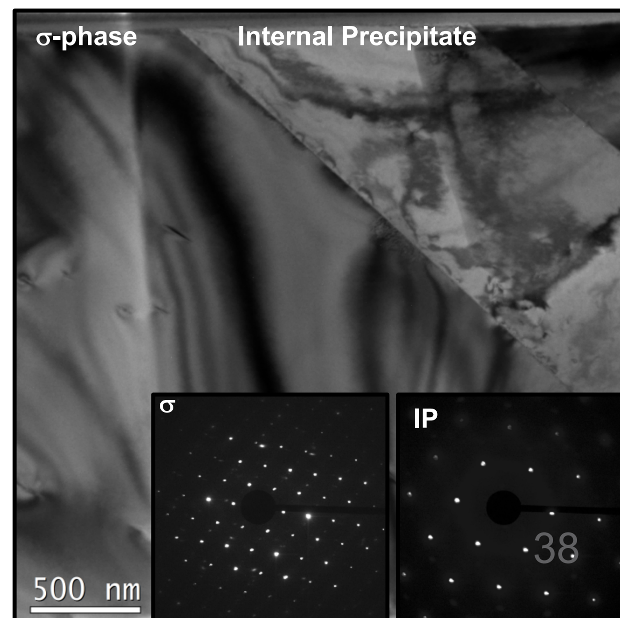
Aim:

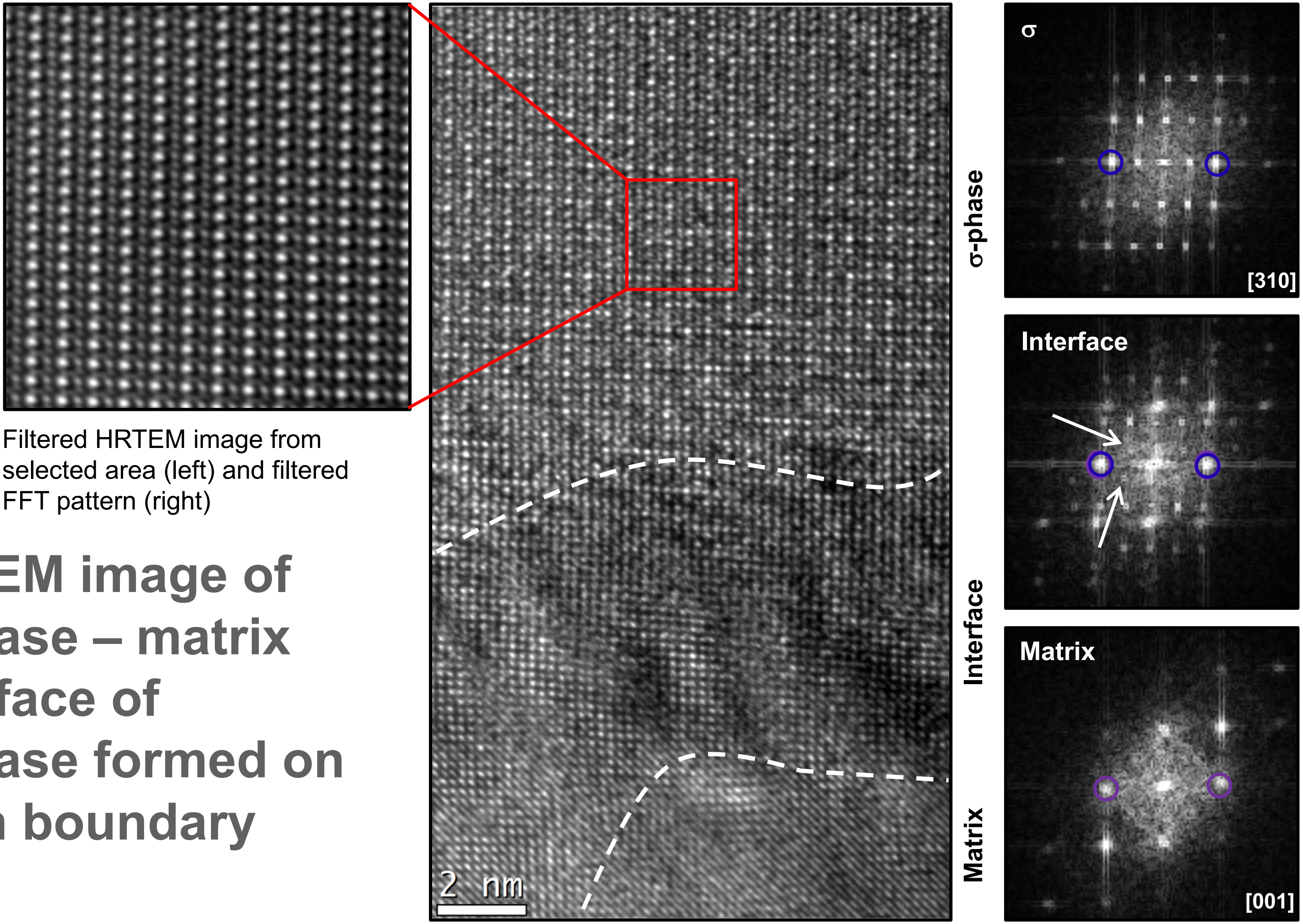
To understand the σ-phase formation caused by addition molybdemum based on the new phase diagram

SEM BSE and TEM BF Images



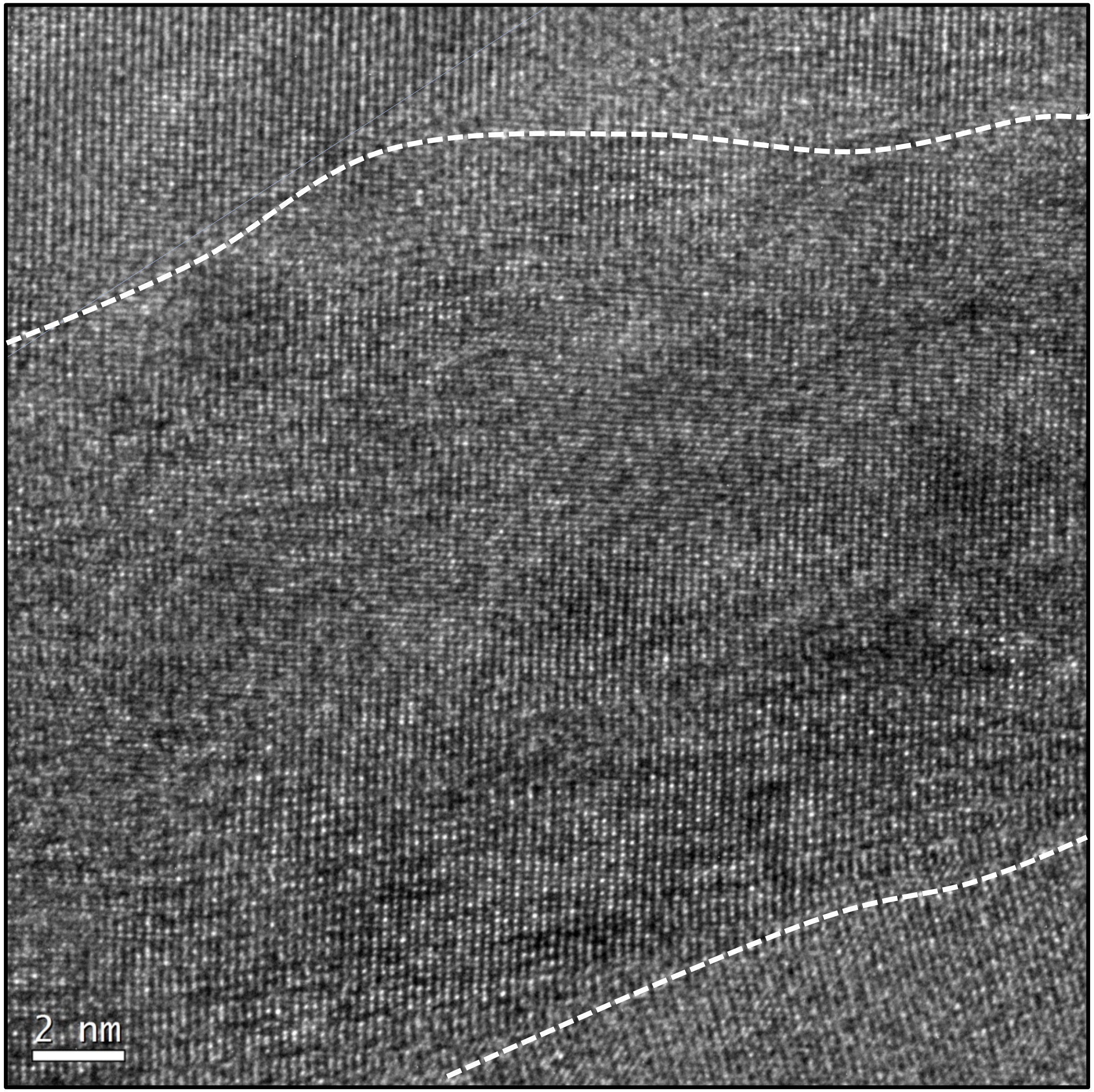


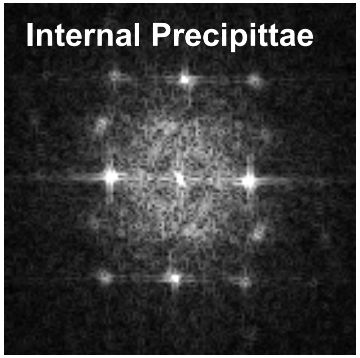


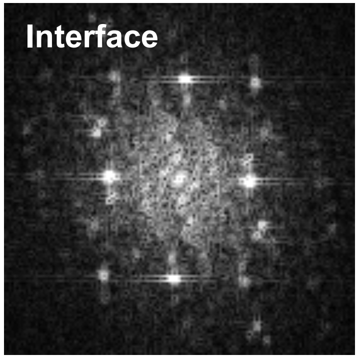


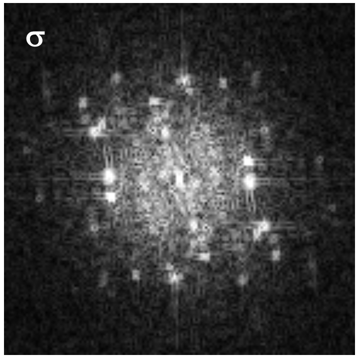
Filtered HRTEM image from selected area (left) and filtered FFT pattern (right)

HRTEM image of σ-phase – matrix interface of σ-phase formed on grain boundary









HRTEM of Sigma Matrix Interface Sigma formed inside grain

3D RECONSTRUCTION OF FIB FOR BIOMATERIAL APPLICATIONS

3D imaging of large biological samples by Dualbeam FIB [13,14]

Limitation of conventional method, ultramicrotomy and cryogenic ultramicrotomy:

- Inability to maintain true structural features
- Cellular structures are generally compressed in the direction of cutting
- Inability to choose specific sites of interest

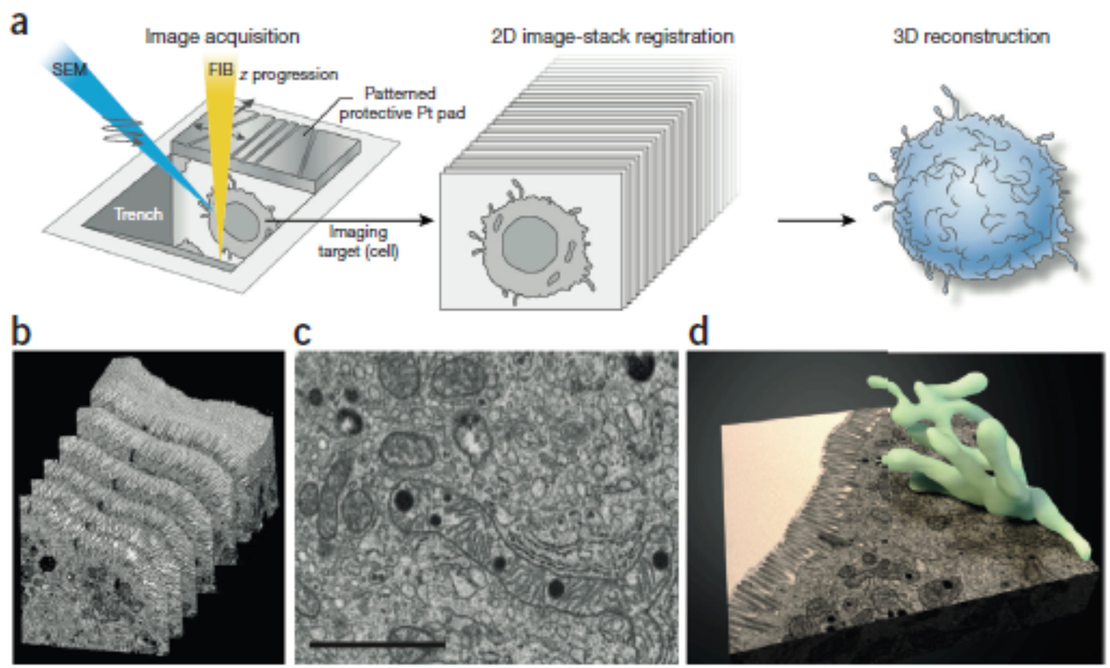


Figure 1 | 3D imaging of large biological samples by FIB-SEM. (**a**) Large biological samples that have been fixed either conventionally (by aldehydes) or cryogenically (by high-pressure freezing), stained by heavy metals, resin embedded and mounted are introduced into the FIB-SEM chamber. Here, chosen areas of the sample are 'trenched' to reveal the region of interest and then subjected to an iterative cycle of resin milling by the FIB (yellow beam) followed by SEM (blue beam) imaging of the newly revealed face to produce a 2D image stack. The patterned protective platinum (Pt) pad atop the sample to be imaged allows automatic beam tuning and slice-thickness control. The 2D image stack is then computationally converted to a 3D volume, aligned and segmented to reveal the 3D structure of interest. (**b–d**) A representative example of 3D tissue imaging using a mouse intestinal sample[109]. Shown are an image stack (**b**), a selected slice through the stack (**c**) and a segmented representation of an extensively branched mitochondrion present in the imaged volume (**d**). Scale bar, 1 μm. Panels **b–d** reprinted from *Encyclopedia of Cell Biology*, Vol. 2, Hartnell, L.M. *et al.*, "Imaging cellular architecture with 3D SEM," 44–50, Copyright 2016, with permission from Elsevier.

Summary

- FIB systems are similar to SEMs in many ways
- It uses ion instead of electron
- It can be used for SEM, TEM, STEM, AES, EDS sample preparation
- It’s very suitable to prepare specific area of the sample
- It can be used to prepare Biological and material science samples
- Limitless possibilities. Anything one can make, can be cut.


Thank you!

References

1. David B. Williams and C. Barry Carter, Transmission Electron Microscopy, Springer 2009
2. Gailly P, Collette J-P, Renson L, Tock JP (1999) Ion Beam Figuring of Small BK7 and Zerodur Optics: Thermal Effects. Proceedings of the SPIE 3739:124–131.
3. Cambridge Universitz Press, 978-0-521-83199-4, Focused Ion Beam Systems: Basics and Applications, Edt: Nan Yao
4. FEI, Focused Ion Beam Basics
5. John Melngailis, Focused ion beam technology and applications, Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics: Materials, Processing, Measurements, and Phenomena 5, 469, (1987)
6. M. W. Barsoum and T. El-Raghy, American Scientist, Volume 89, 2001
7. D.E. Hajas, et.al, Surface & Coatings Technology, 206 (2011) pp 591-598
8. R. Iskandar 2017 *J. Phys.: Conf. Ser.* 817012011
9. R. Iskandar et.al, Material Science and Engineering Technology, October 2017, Accepted
10. R. Iskandar et. al, International Microscopy Congress, Prague 2014
11. Stanislaw M. Dubiel and Jakub Cieslak, Sigma Phase in Fe-Cr nad Fe-V alloy systems and ist physical properties
12. N. Lopez, M. Cid, and M. Puiggali, Influence of σ-phase on mechanical properties and corrosion resistance of duplex stainless steels, Corrosion Science, vol. 41, no. 8, pp. 1615-1631, 1999
13. K. Grandfield and H. Engqvist, Focusedion beam in the study of biomaterials and biological Matter, Advances in Materials Science and Engineering, Vl 20112, Atcle ID 841961
14. K. Narayan and S. Subramanam, Focused ion beams in biology, Nature methods, Vol. 12. No 11, Nov 2015, 1021