Cross-Sectional imaging of Soft Indium Solder Thermal Interface Materials with Thermal Cycling

Jeff Kurish, Piyush Kulkarni,Zechen Zhang

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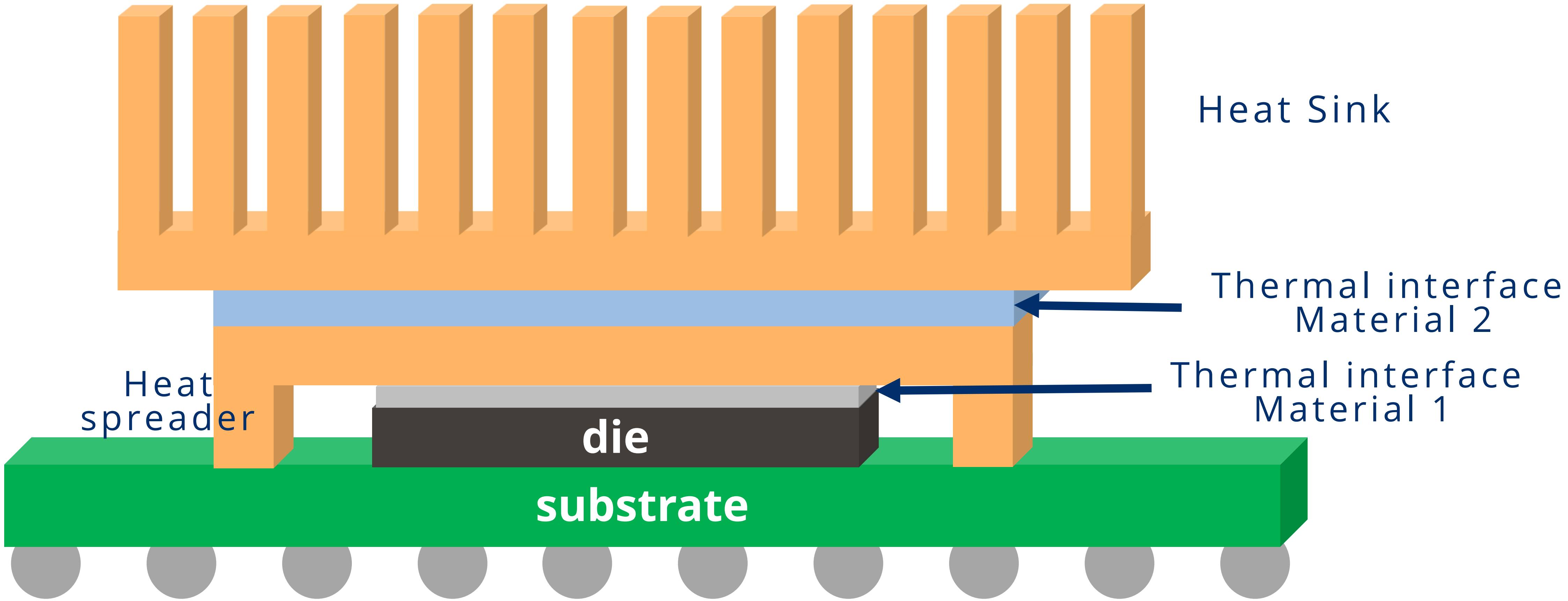
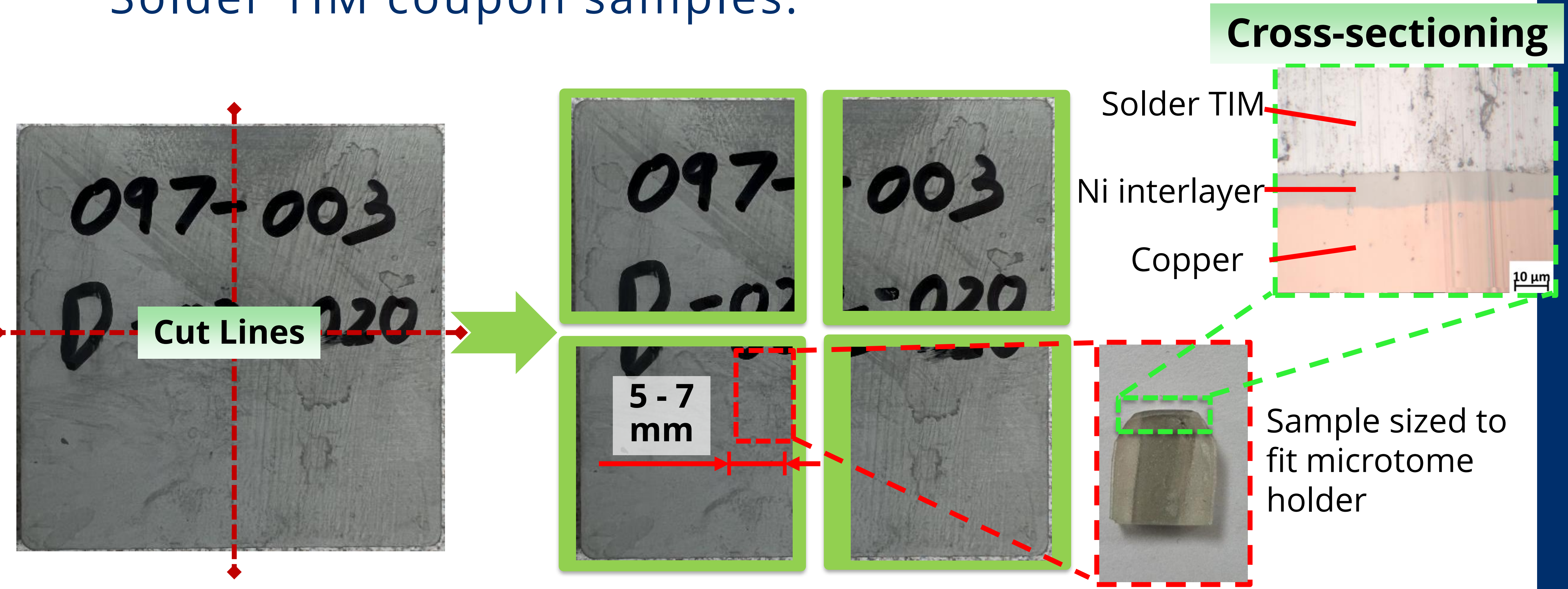
High quality metrology in the thermal interface material (TIM) industry is necessary for research and assembly



This work is mostly an update on cross sections for Solder TIM coupon samples.



Cross-sectioning

Cut Lines

5 - 7 mm

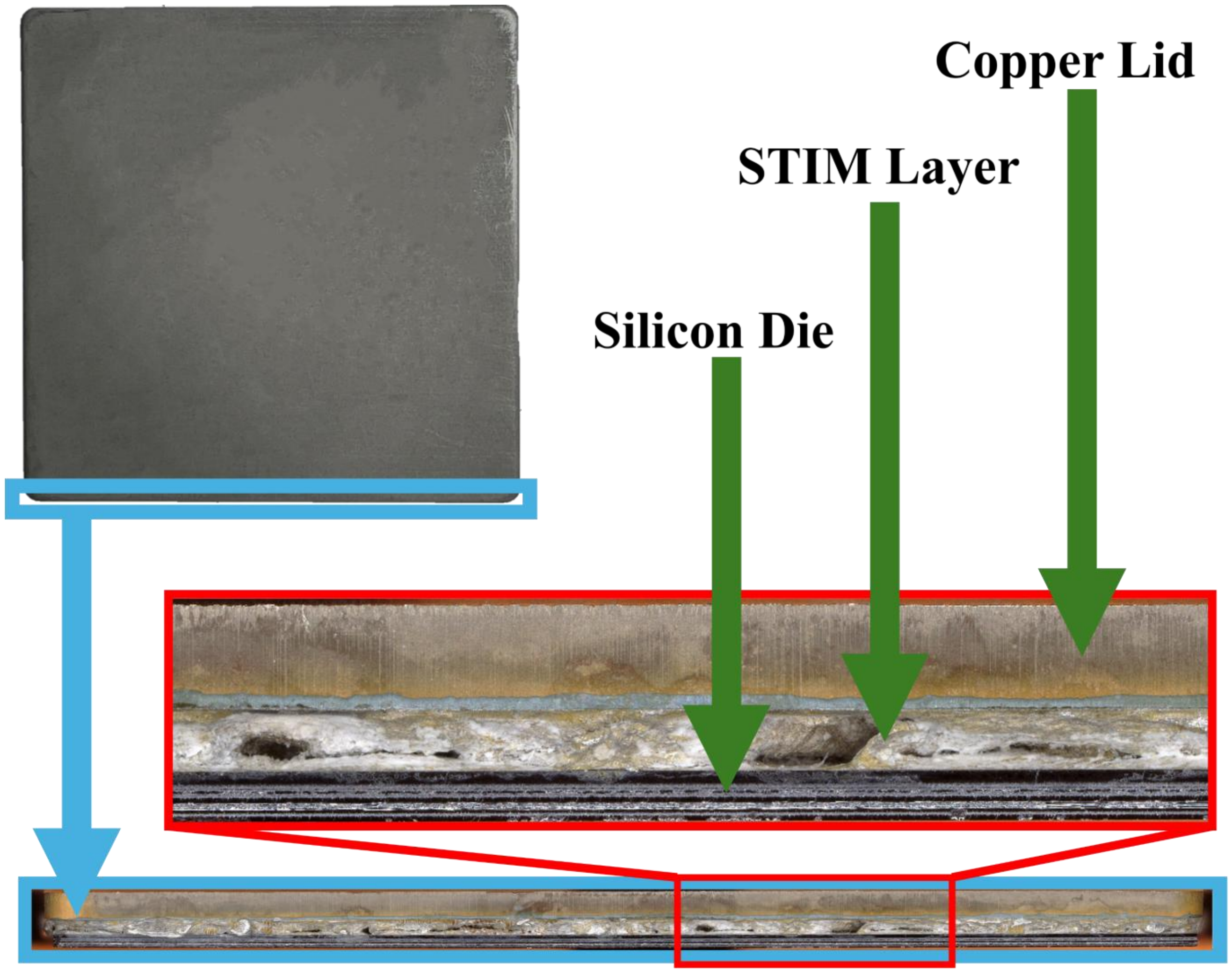
Solder TIM

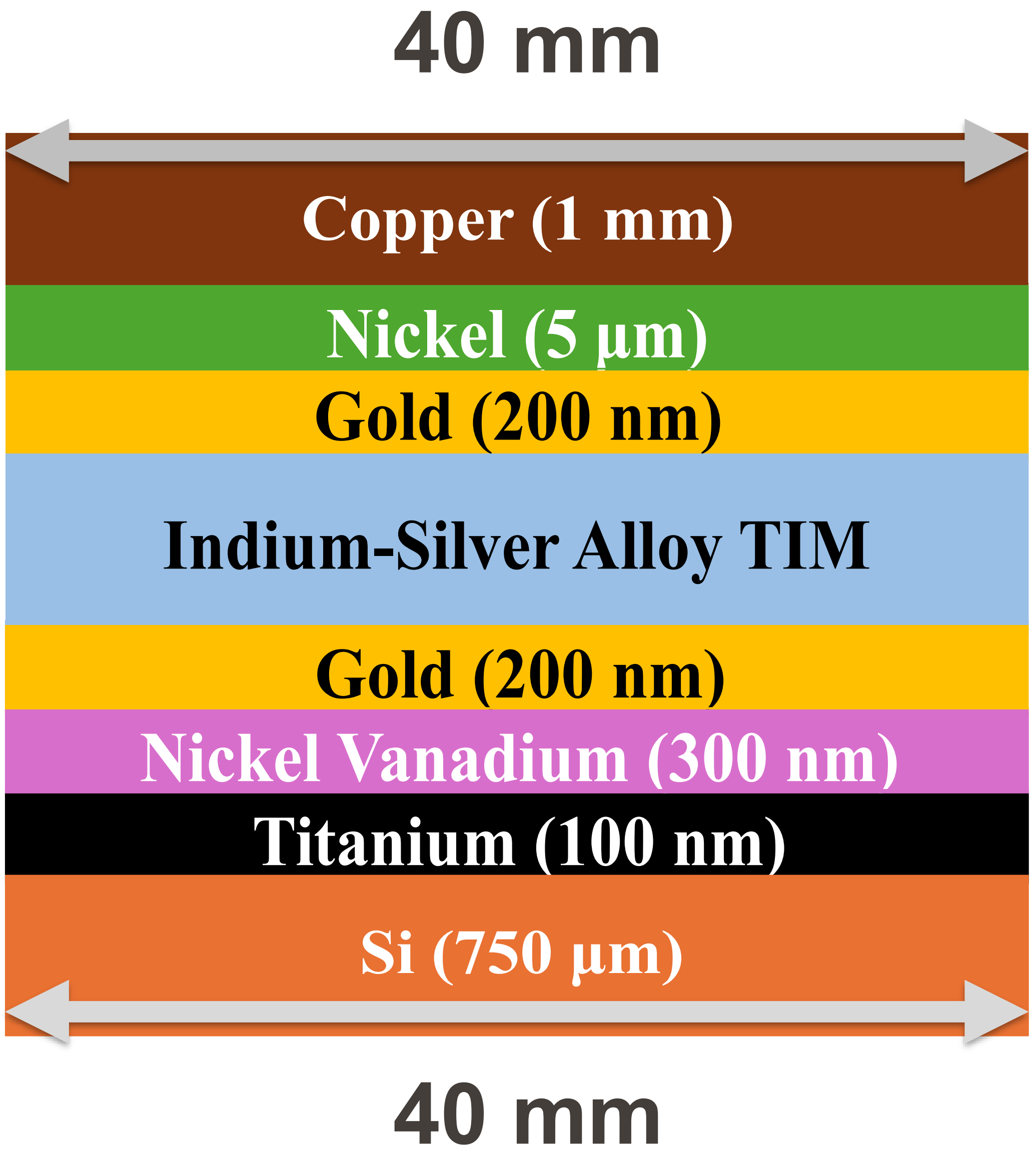
Ni interlayer

Copper

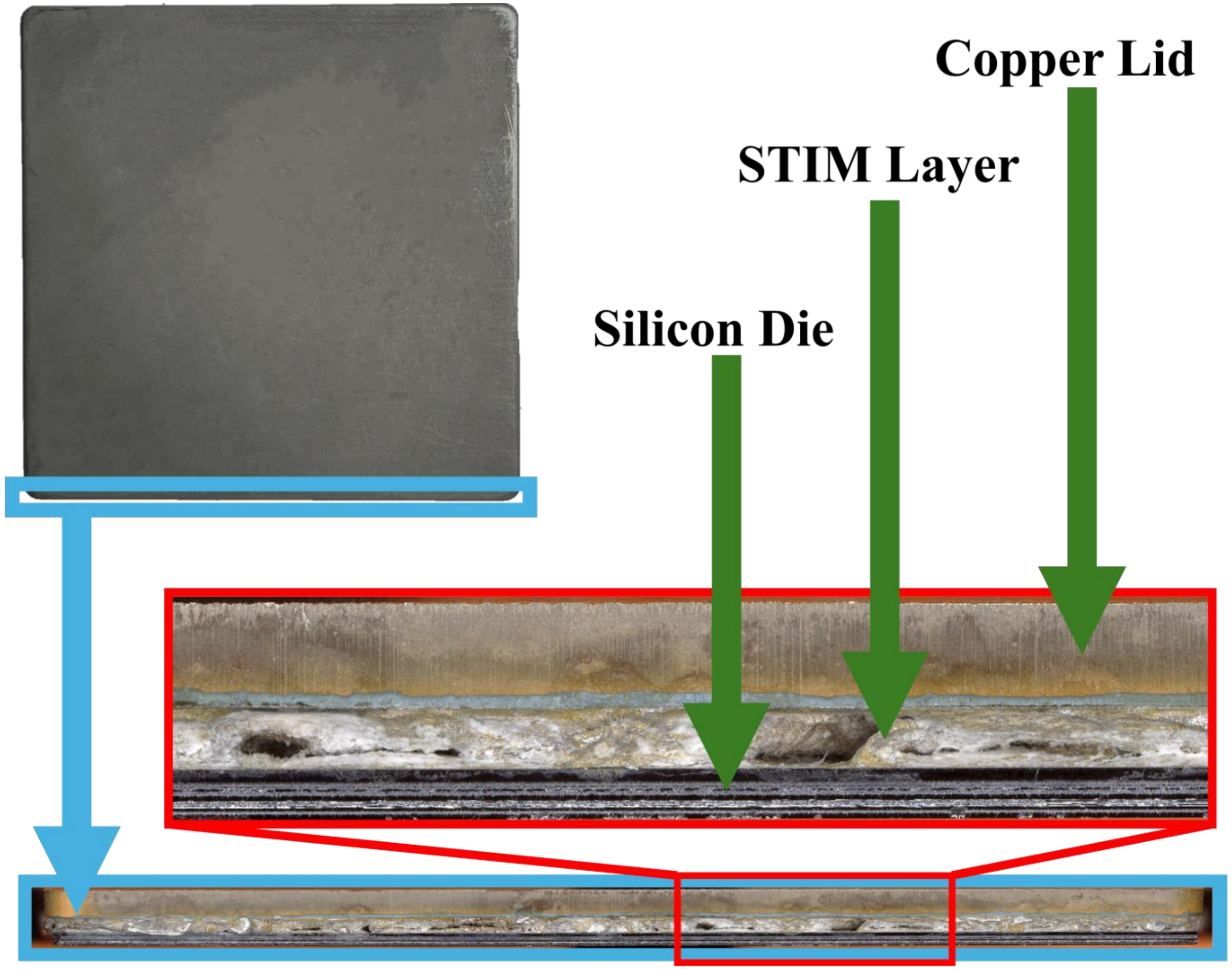
Sample sized to fit microtome holder

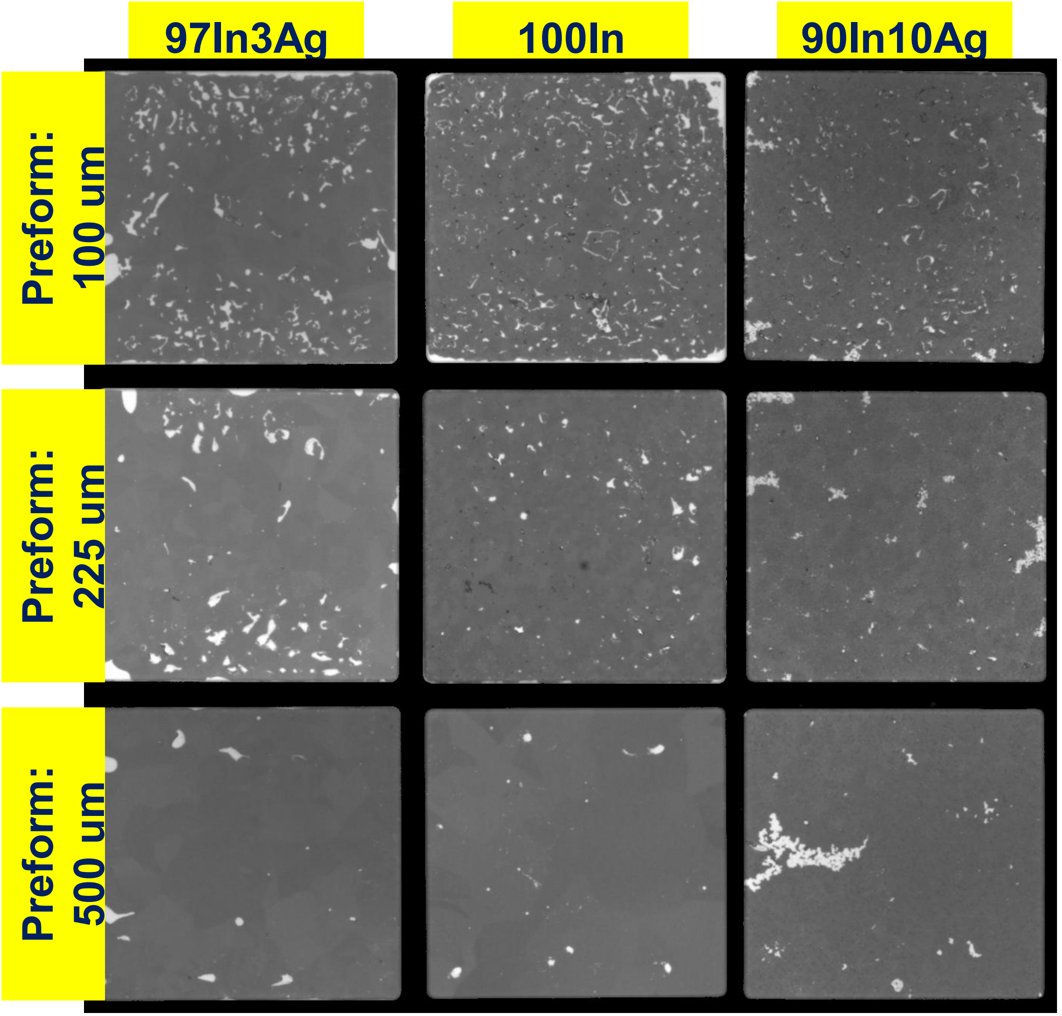
Full stack metallization involve many layers.





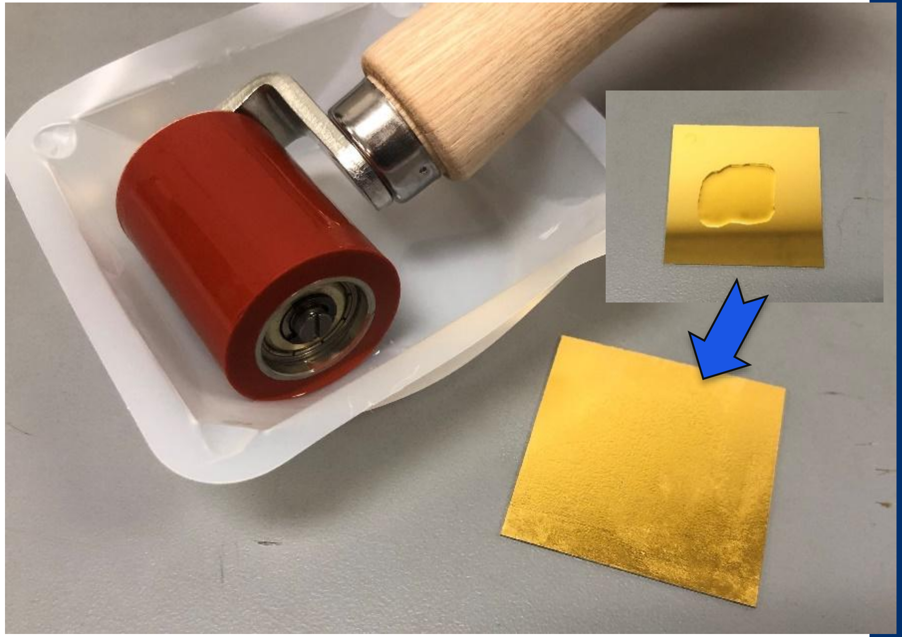
A ~7 fold higher thermal conductivity in solder TIMs allows viability of larger bond line thicknesses.

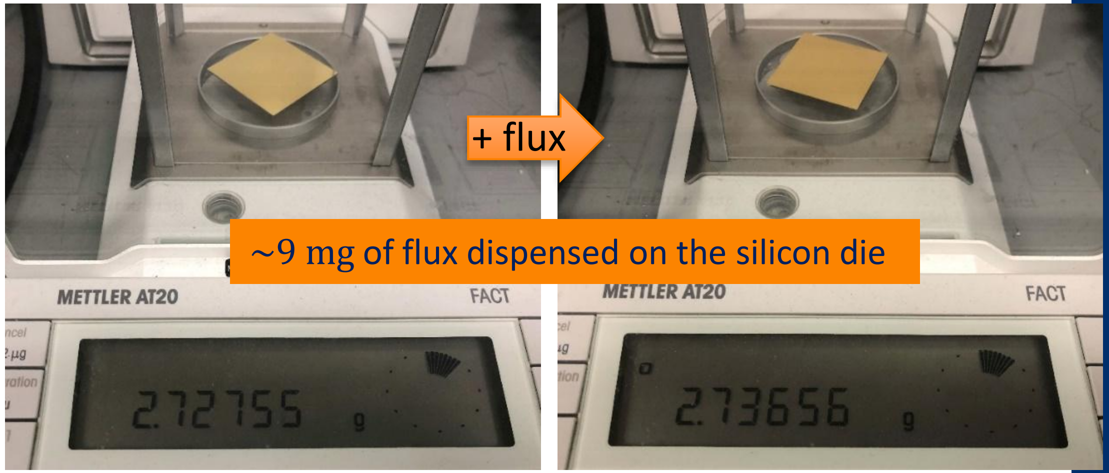




Flux was a requirement of the study and is roller deposited.

- Metalized silicon piece, and Au-plated copper piece are thoroughly cleaned.
- Flux is **manually spread** over the Si die surface.
  - A controlled volume of flux is dispensed on the surface using a micro-pipette (recommended **5mg/** $\mathbf{in^2}$).
  - A soft silicone rubber roller is used to spread out the flux to cover the entire surface.
- Preform is picked and placed on the fluxed surface of the die.
- Another layer of flux is deposited on top of the preform.
- Cu lid is placed on top to complete the sandwich structure.
- Samples are baked in box oven at **125° C for 60 minute**.

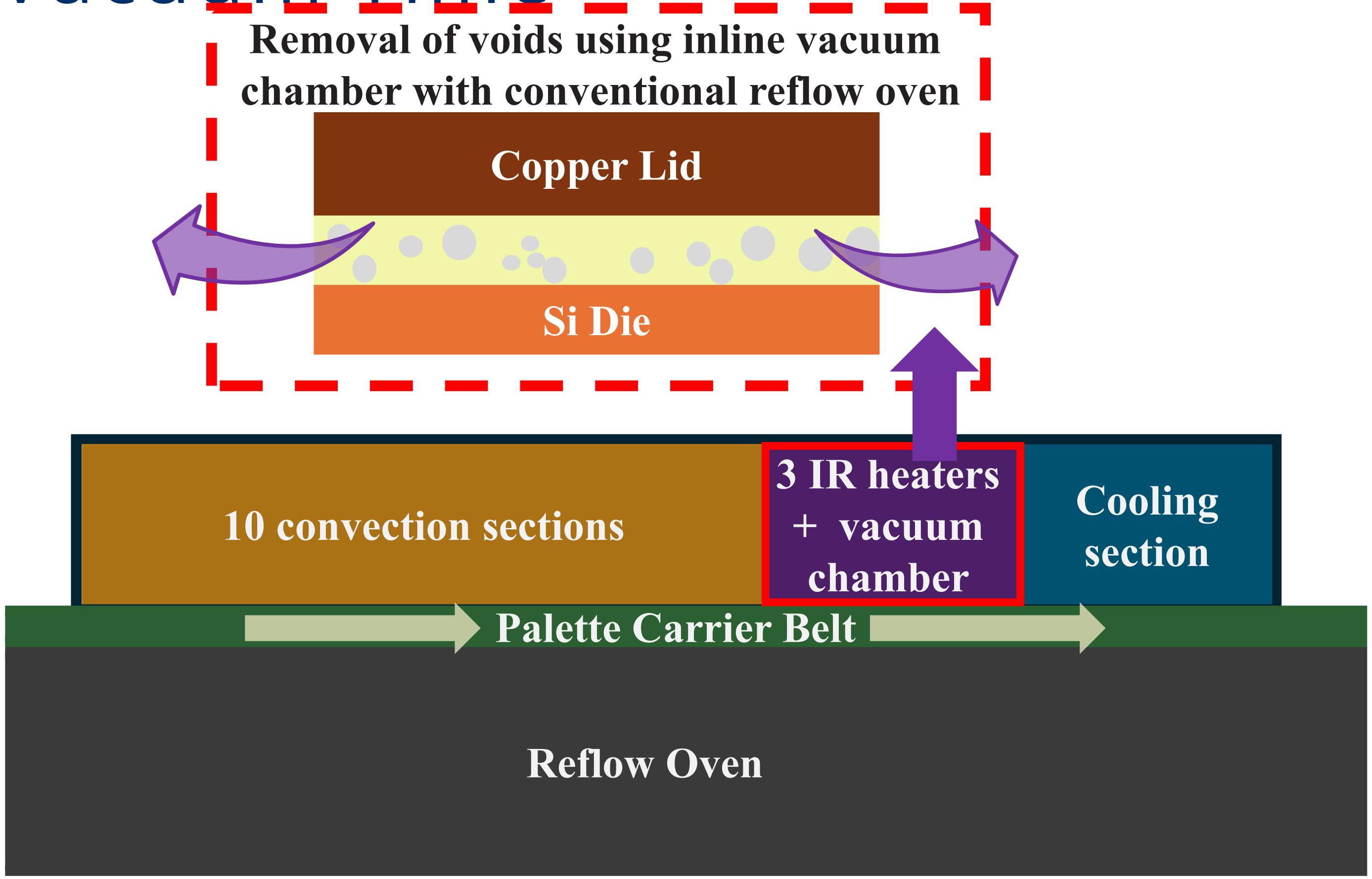




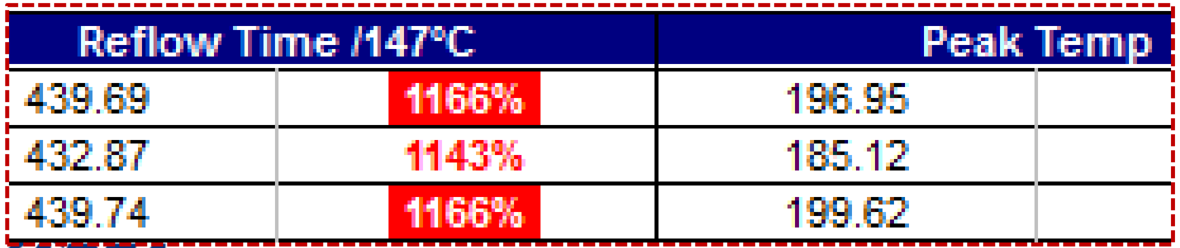
+ flux

~9 mg of flux dispensed on the silicon die

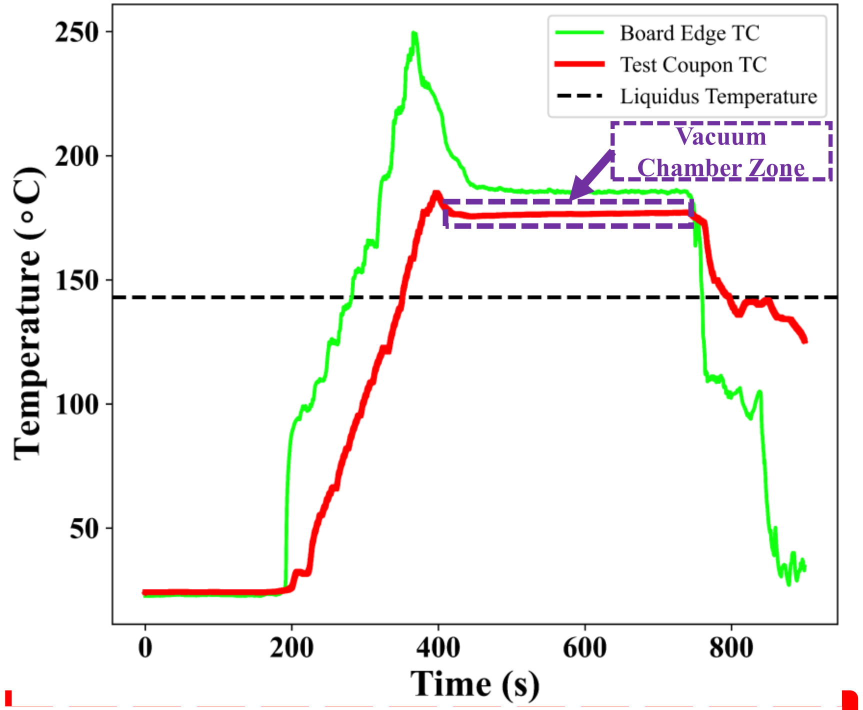
Example of Vacuum Reflow Profiles with Extended Vacuum Time

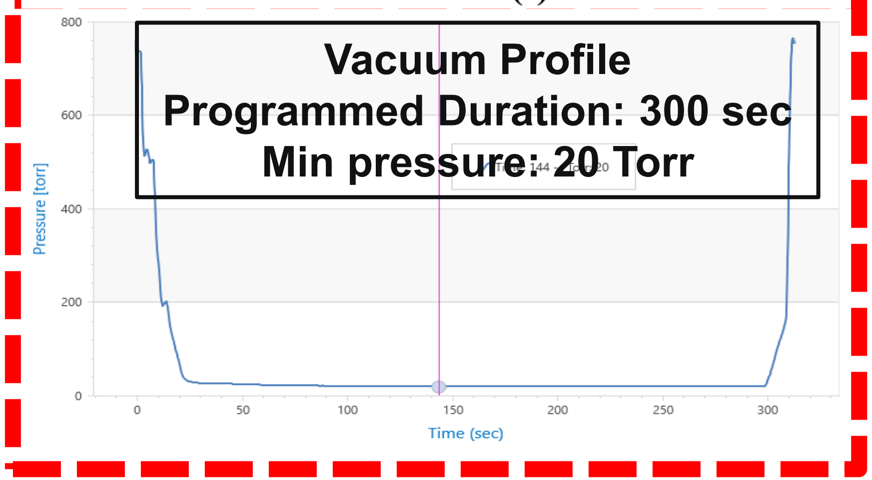


Removal of voids using inline vacuum chamber with conventional reflow oven



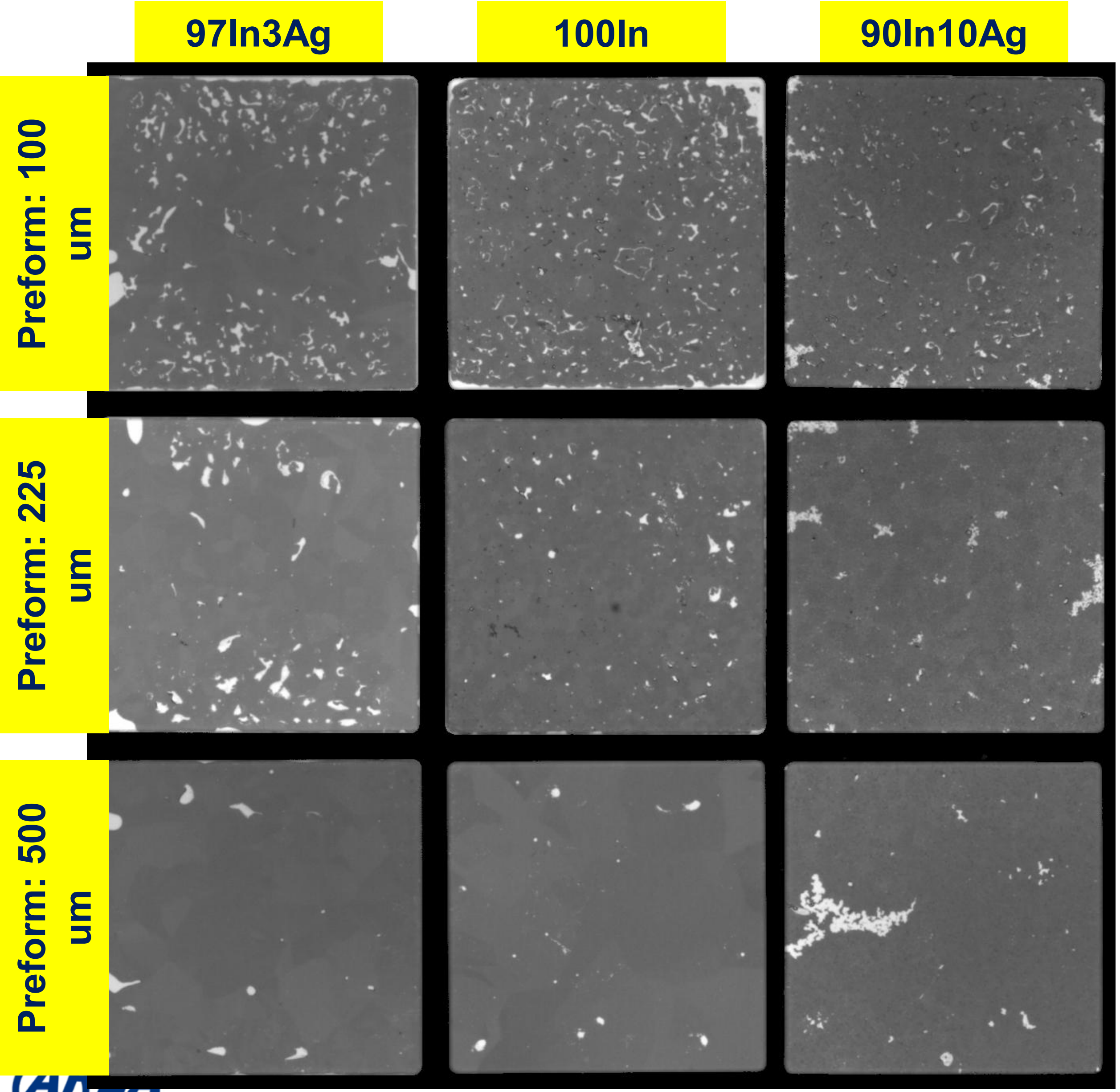
Reflow Time /147°C		Peak Temp	
439.69	1166%	196.95	
432.87	1143%	185.12	
439.74	1166%	199.62	

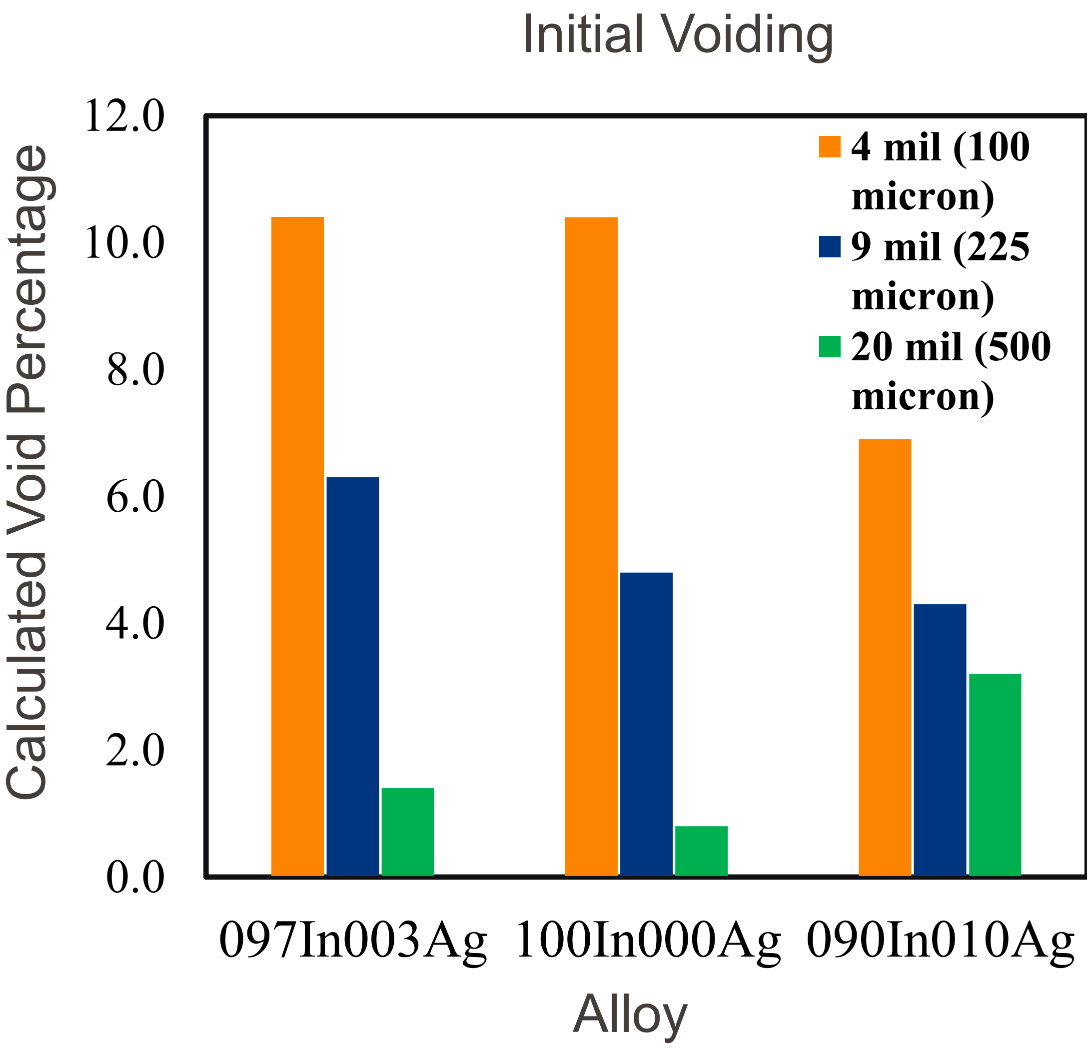




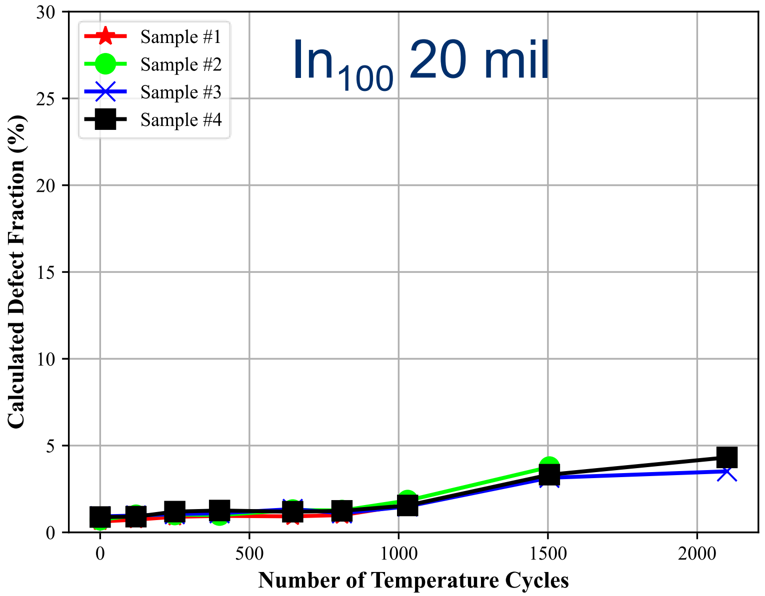
Vacuum Profile
Programmed Duration: 300 sec
Min pressure: 20 Torr

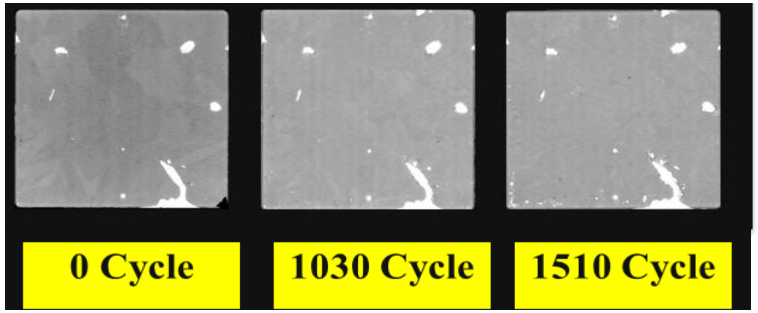
CSAM easily reveals difference between thicknesses.

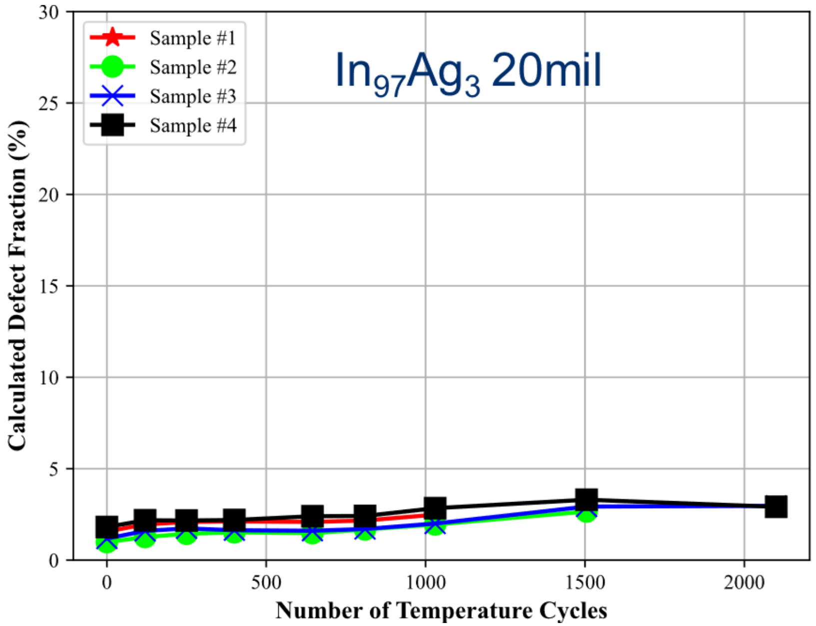


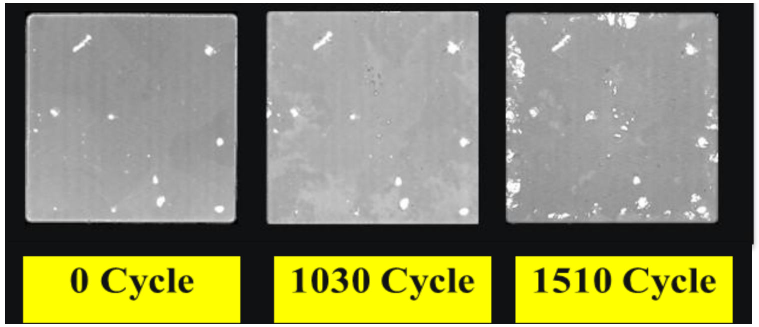


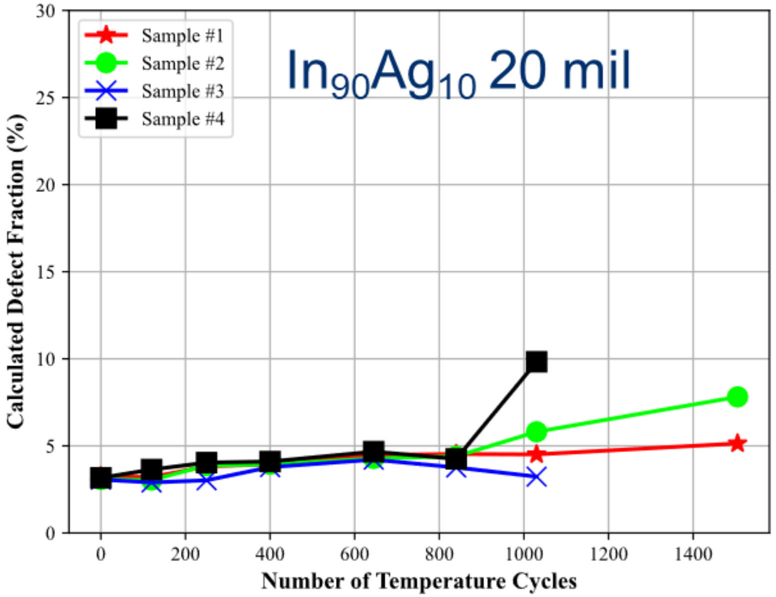
20 mil: All samples are profoundly stable.

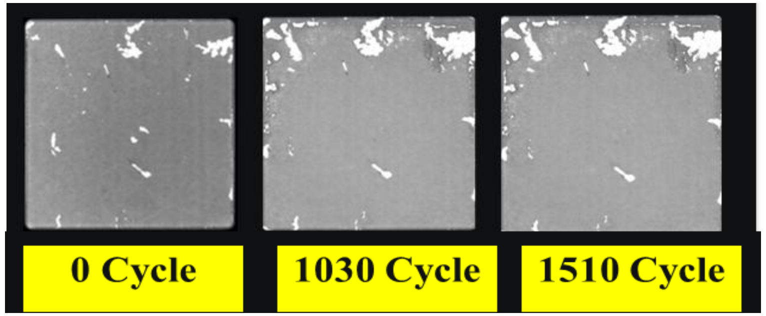




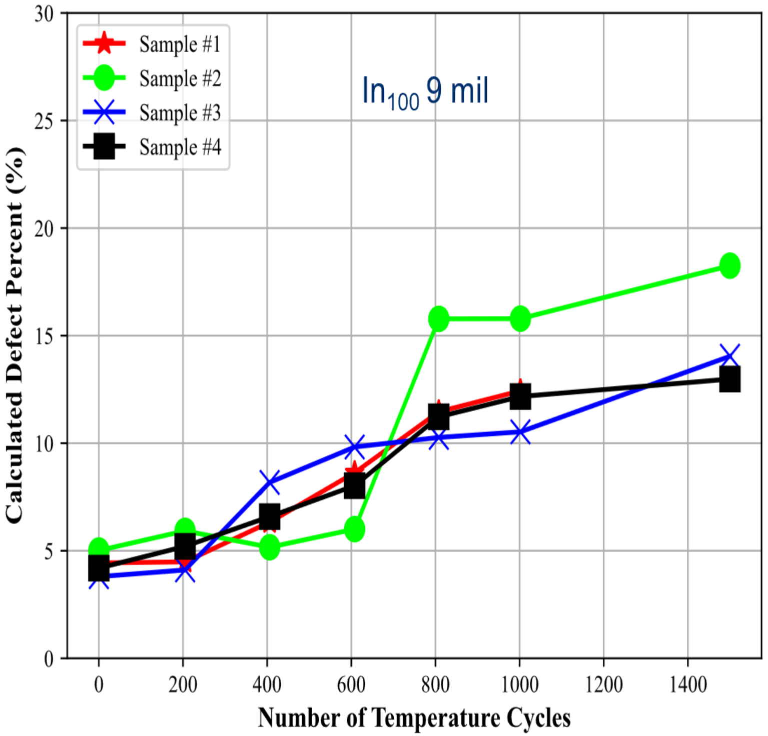


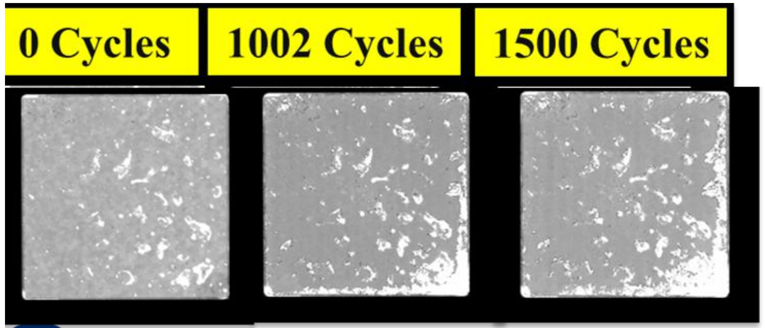


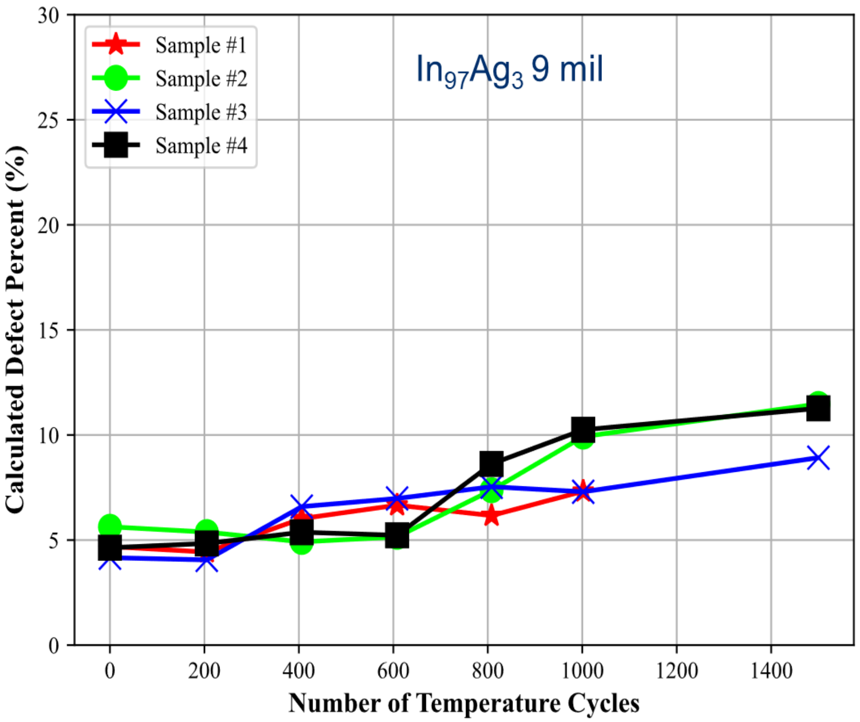


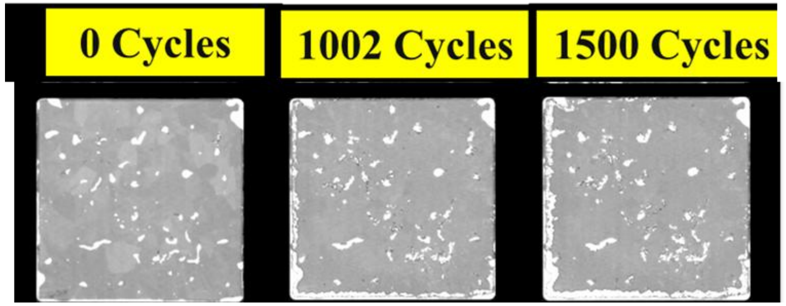


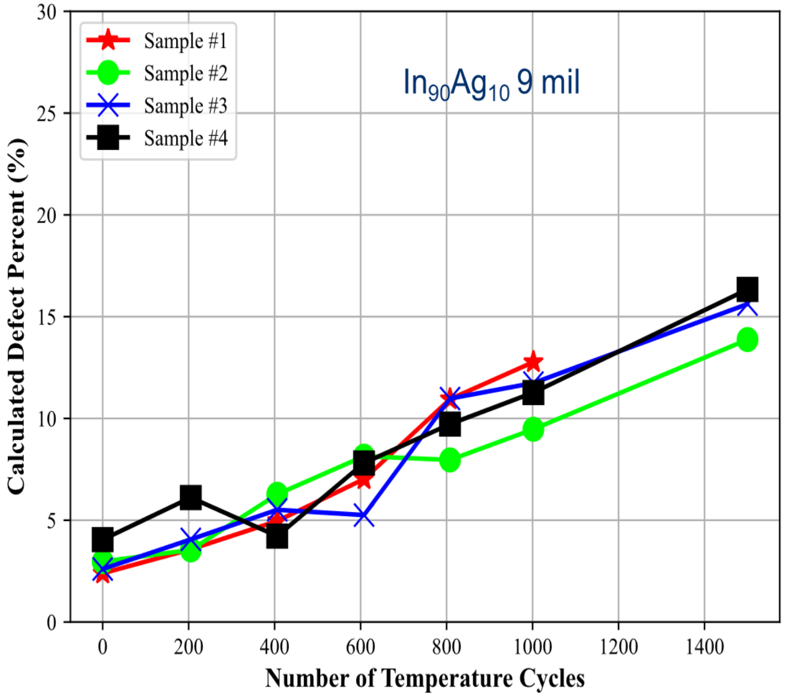
9 mil : All samples follow similar trend.

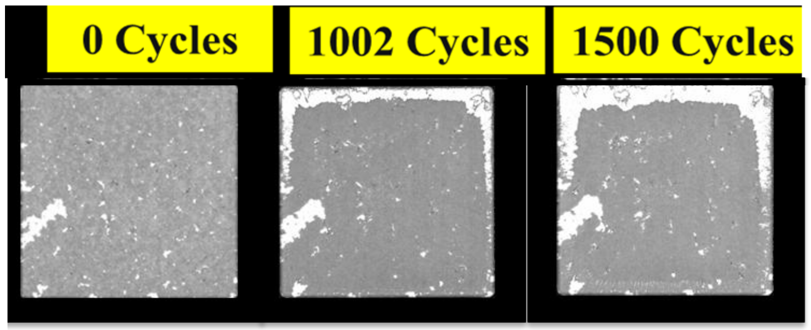




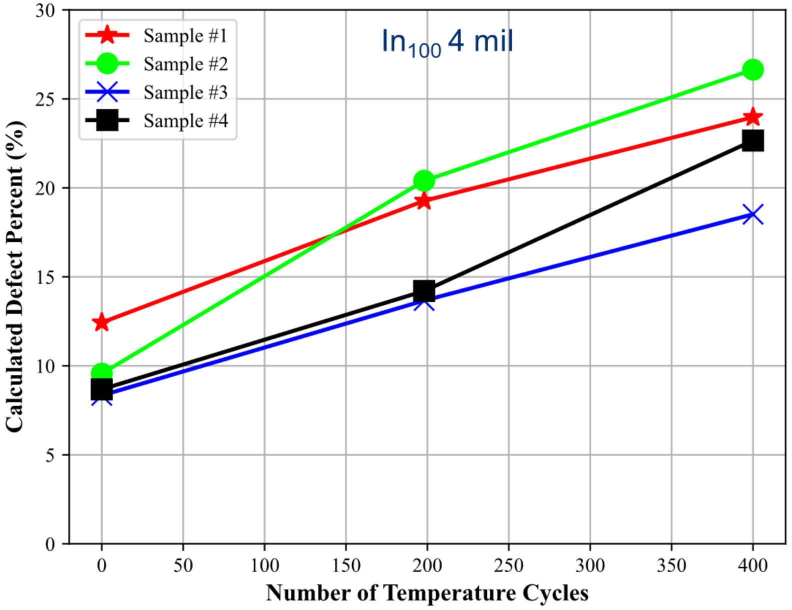


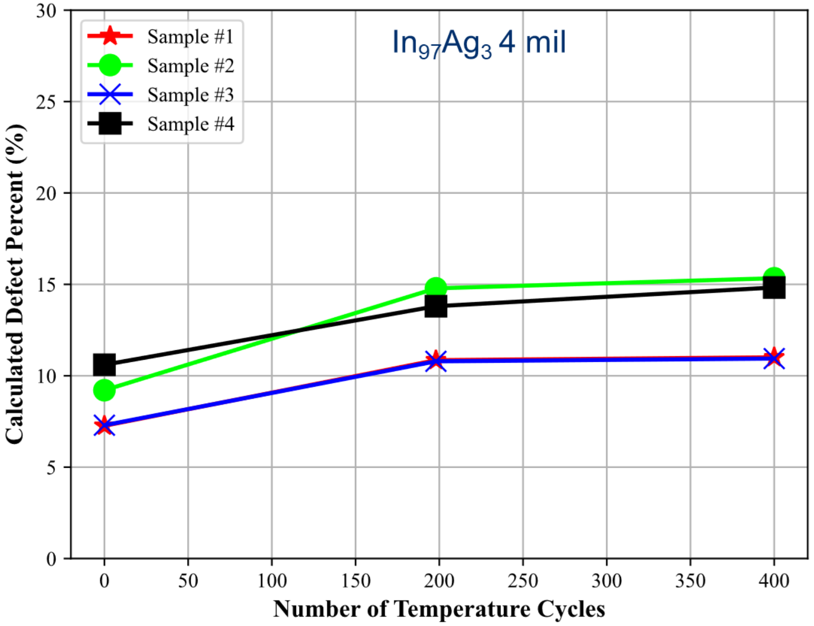


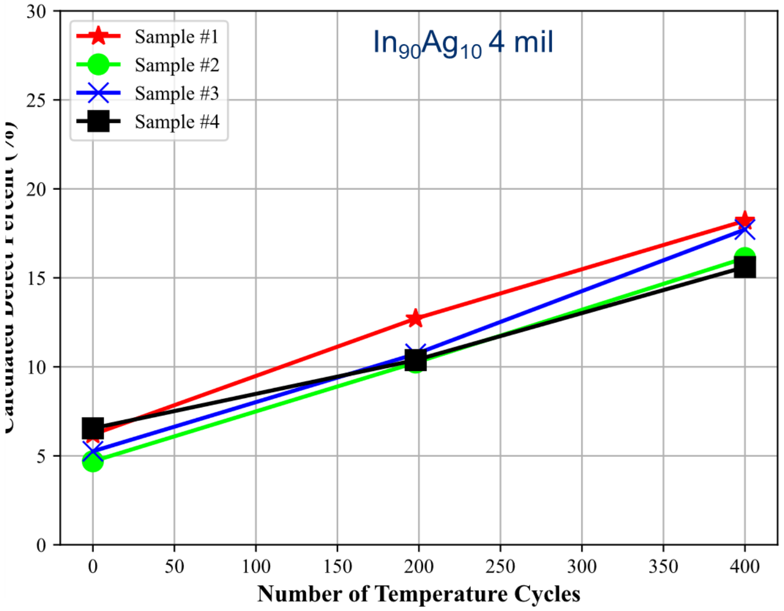


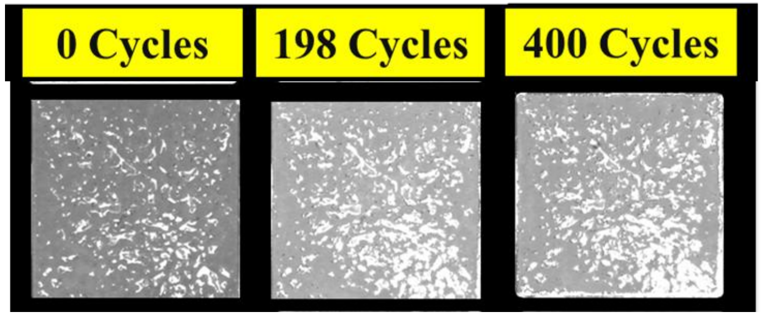


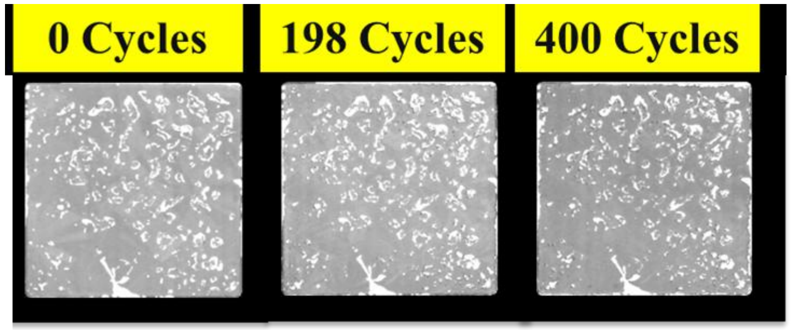
4 mil: Study is young but showing clear trends

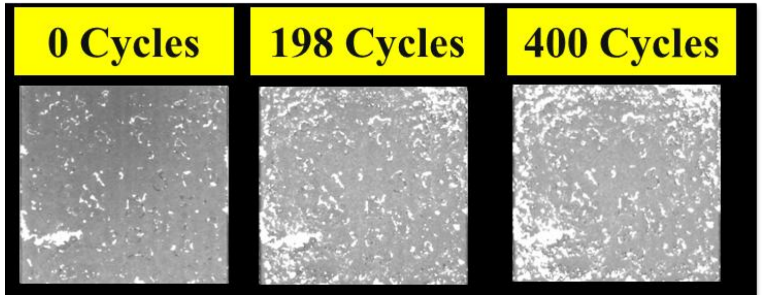












Comparisons between thicknesses are straight forward.

500 µm

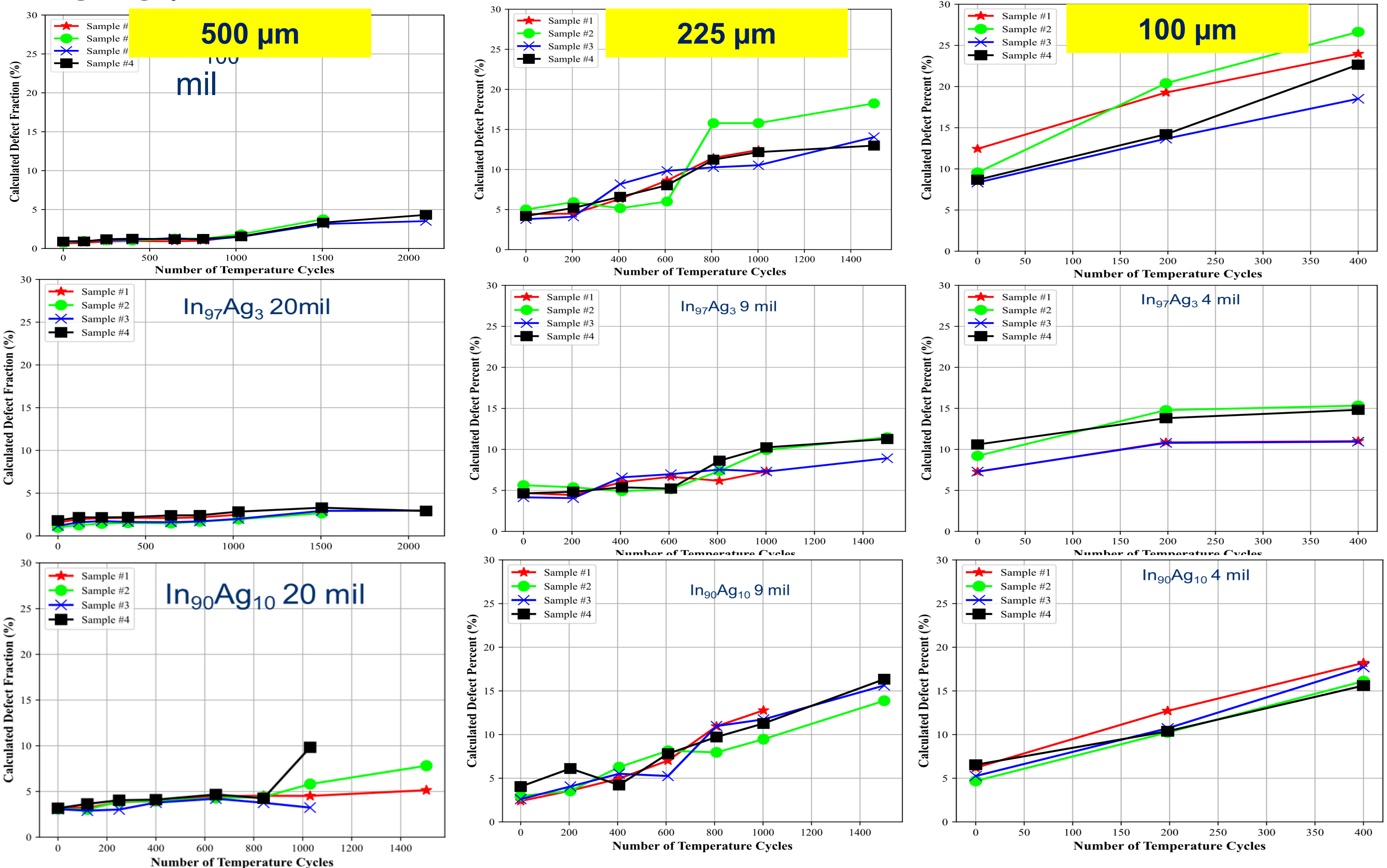
225 µm

100 µm

100In

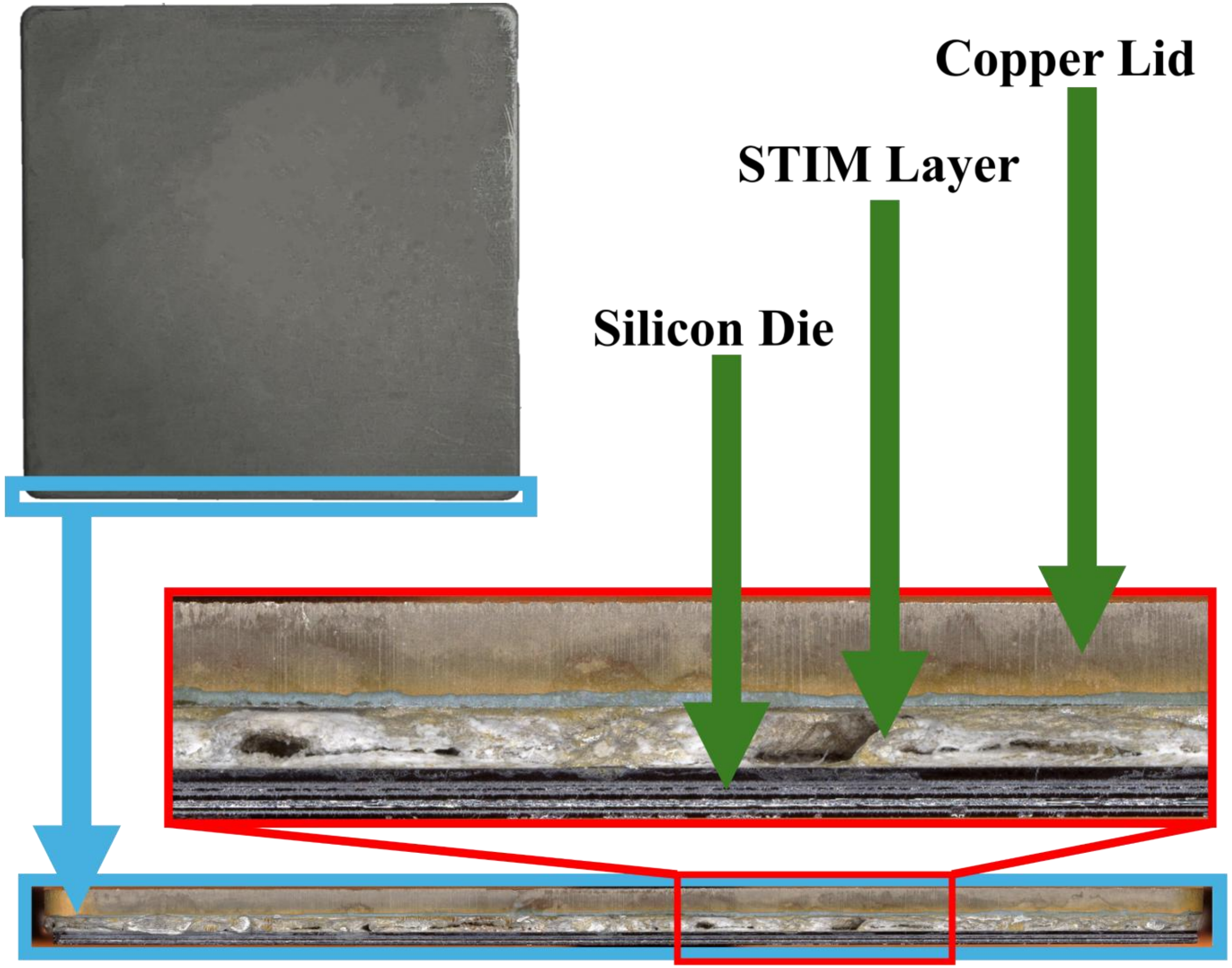
97In3Ag

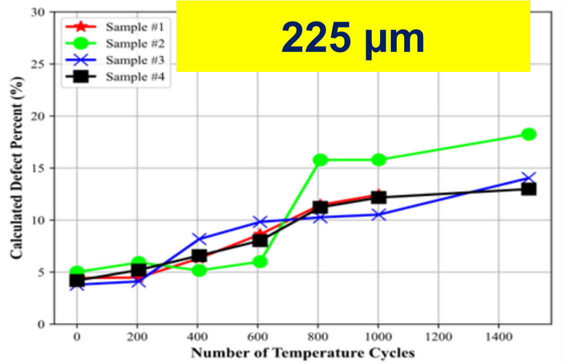
90In10Ag

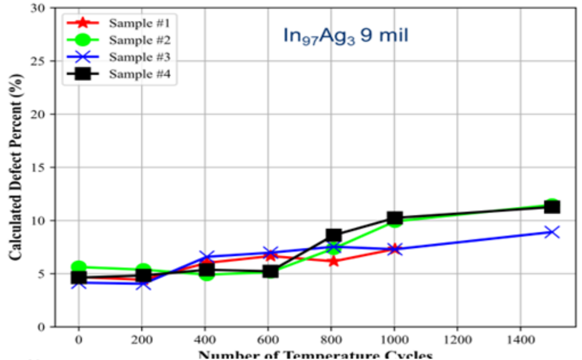


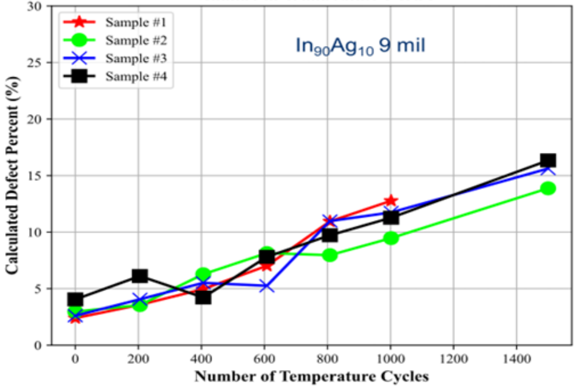
AREA CONSORTIUM

Alloy level understanding requires cross sectioning.





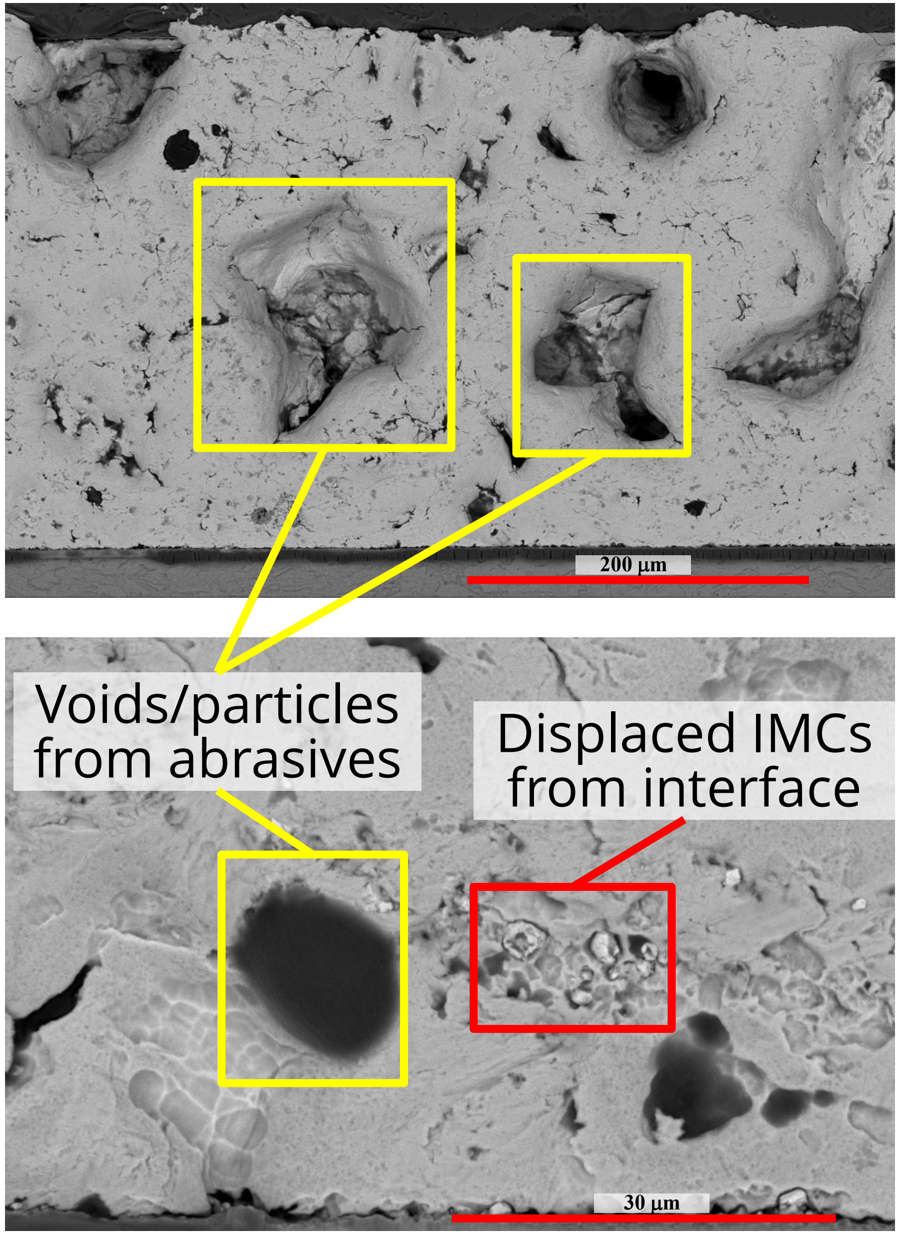




2025 24th IEEE ITHERM Conference. Indium Solder TIM Stability under Temperature Cycling

Particle embedment is primary challenge for cross-sectioning indium-based STIM test coupons

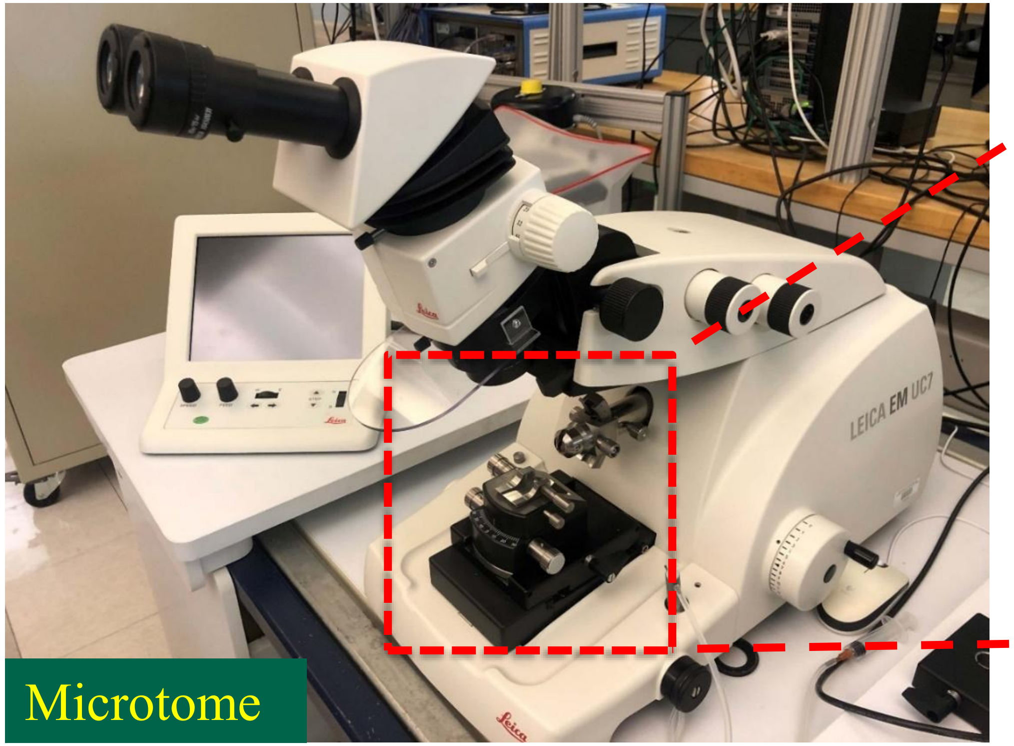
- Cross-sectioning of In solder TIMs is challenging
  - Indium is soft → smearing & embedment of Si/abrasive particles
  - Conventional methods damage the solder surface and cause artifacts
- Different grinding and polishing recipes, combining SiC paper, ultrasonic bath, and chemical etching (HNO3), were tried.
- Alternative cross-sectioning & polishing
  - abrasive-free, avoiding damage to solder TIMs

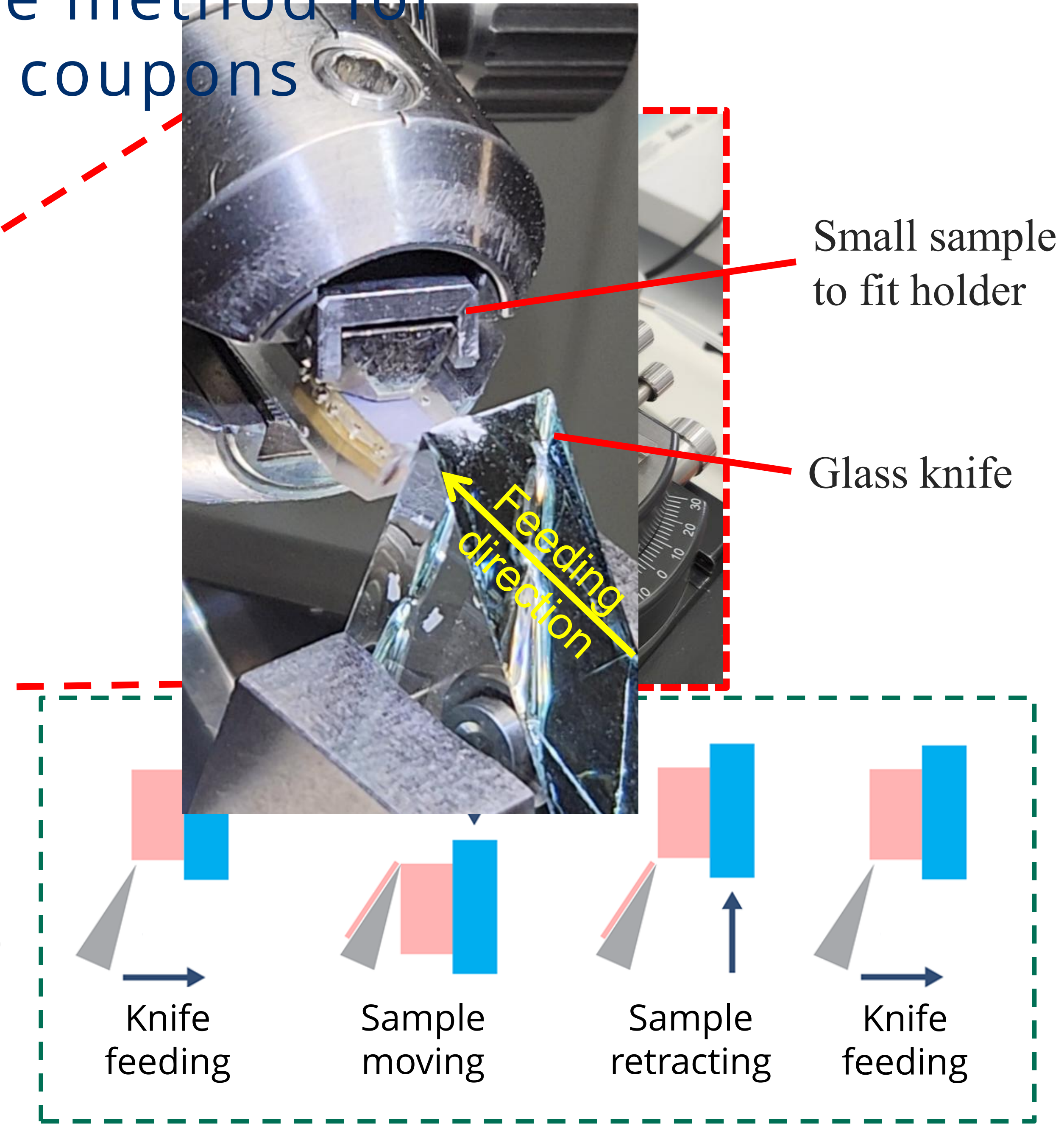


Voids/particles from abrasives

Displaced IMCs from interface

Microtome is an alternative method for cross-sectioning STIM test coupons



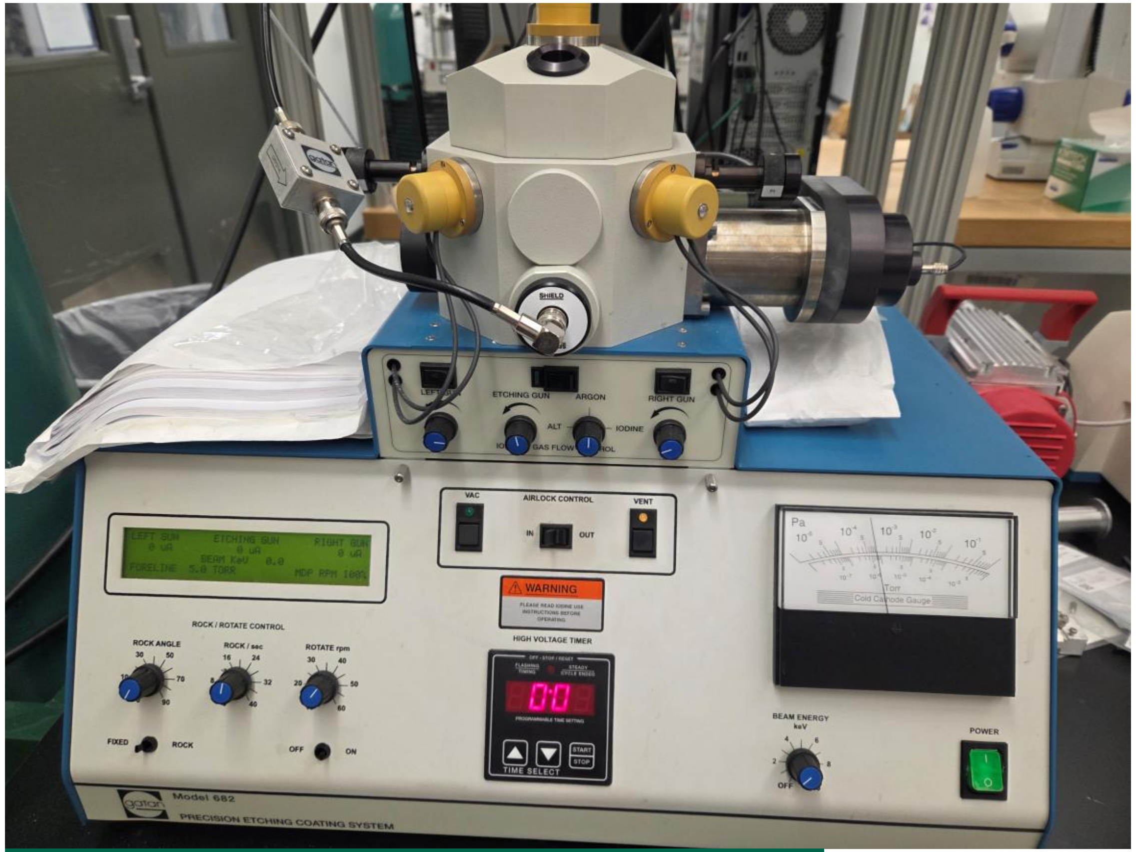


Main controlling parameters:

- Specimen moving speed (mm/s)
- Knife feeding distance (nm)

Doi: 10.1109/ECTC51529.2024.00247

Ion milling provides much more refinement.

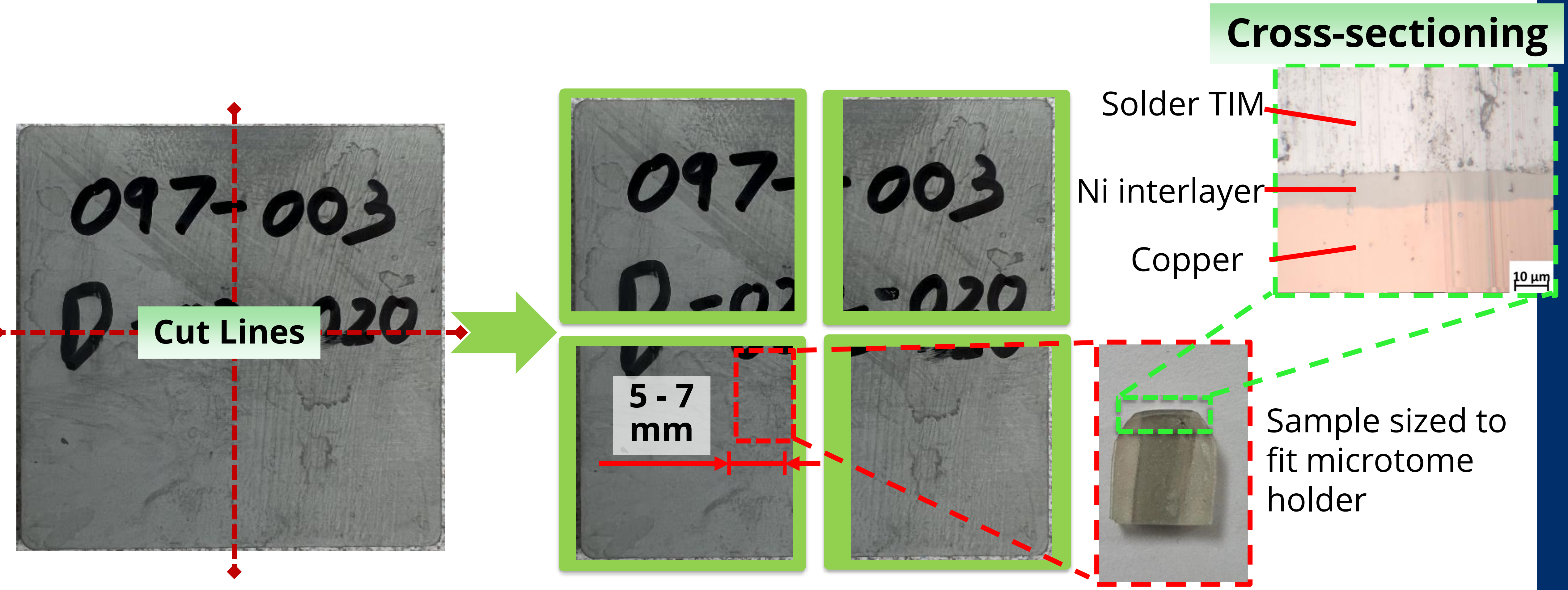


Precision etching coating system

	In/Ag – 97/3	In – 100
Voltage	2-3keV	2-3keV
Time	30 minutes	30 minutes
Angle	80°	80°

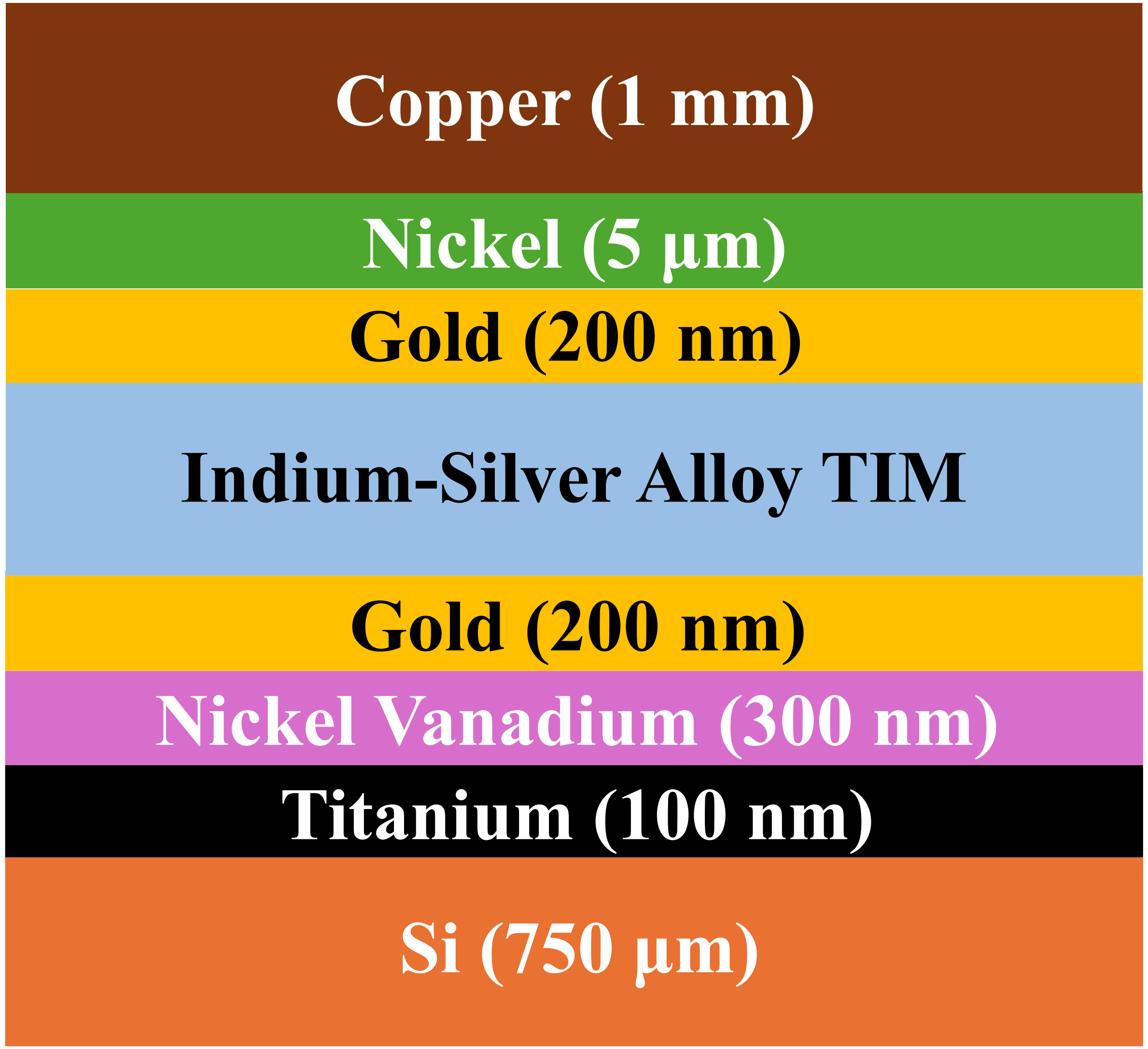
- Etching time
  - On for 1 minute, off for 1 minute
  - Sheet includes accumulated working time
- Angle is between the beam and the sample surface

Preparing parts for microtome sectioning



- Part cut into four pieces for different tests
- Section of interest cut out and potted in epoxy

Due to restrictions on the microtome the silicon side is ground away and not visualized.



Copper (1 mm)

Nickel (5 µm)

Gold (200 nm)

Indium-Silver Alloy TIM

Gold (200 nm)

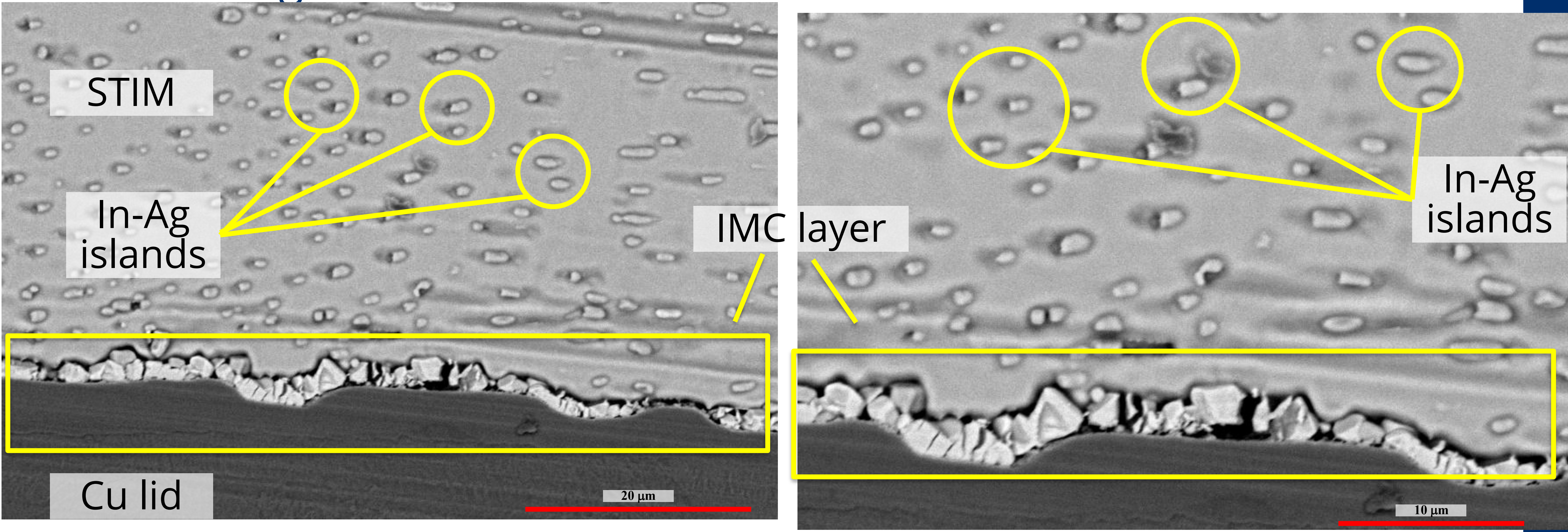
Nickel Vanadium (300 nm)

Titanium (100 nm)

Si (750 µm)

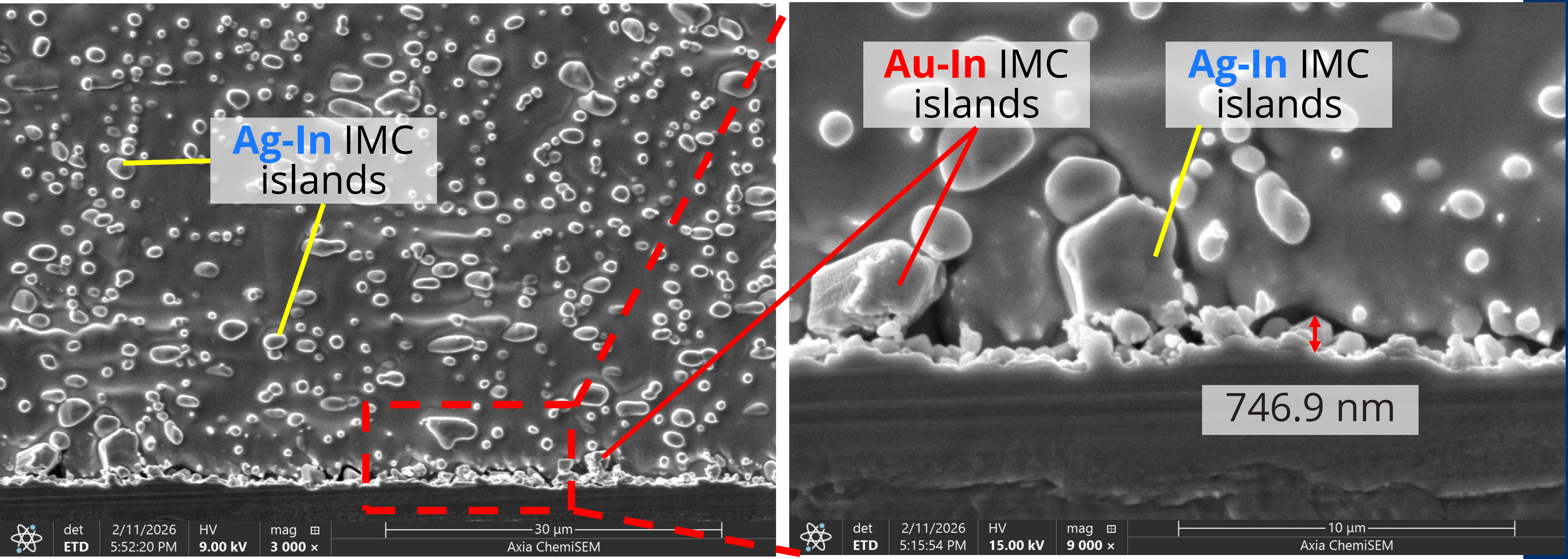
$In_{97}Ag_3$

Earlier image shows time zero $In_{97}Ag_3$ is dominated by small AgIn intermetallic islands



- In/Ag – 97/3 alloy preform, 508 µm (20 mil)
- High quality of sections with no embedded particles
- Clear structures of an IMC layer near the Ni interlayer
- Significant IMC island areas scattered across the solder region

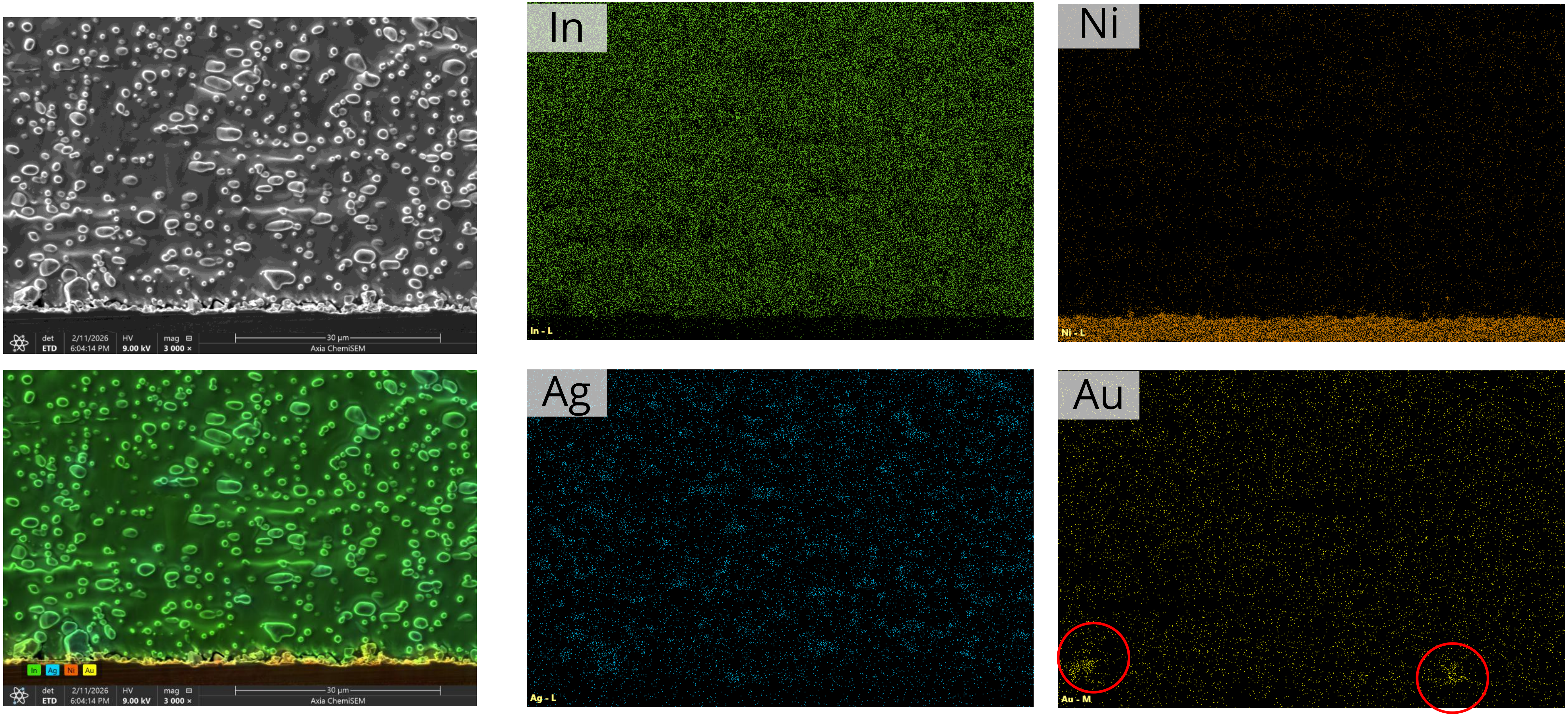
New Images show Au-In IMC islands clustered near In-Ni IMC layer.



- In/Ag – 97/3 alloy preform, 508 µm (20 mil)
- IMC islands of Au-In and Ag-In
- Ion mill for 30 minutes

$In_{97}Ag_3$

In/Ag – 97/3 alloy – Time Zero



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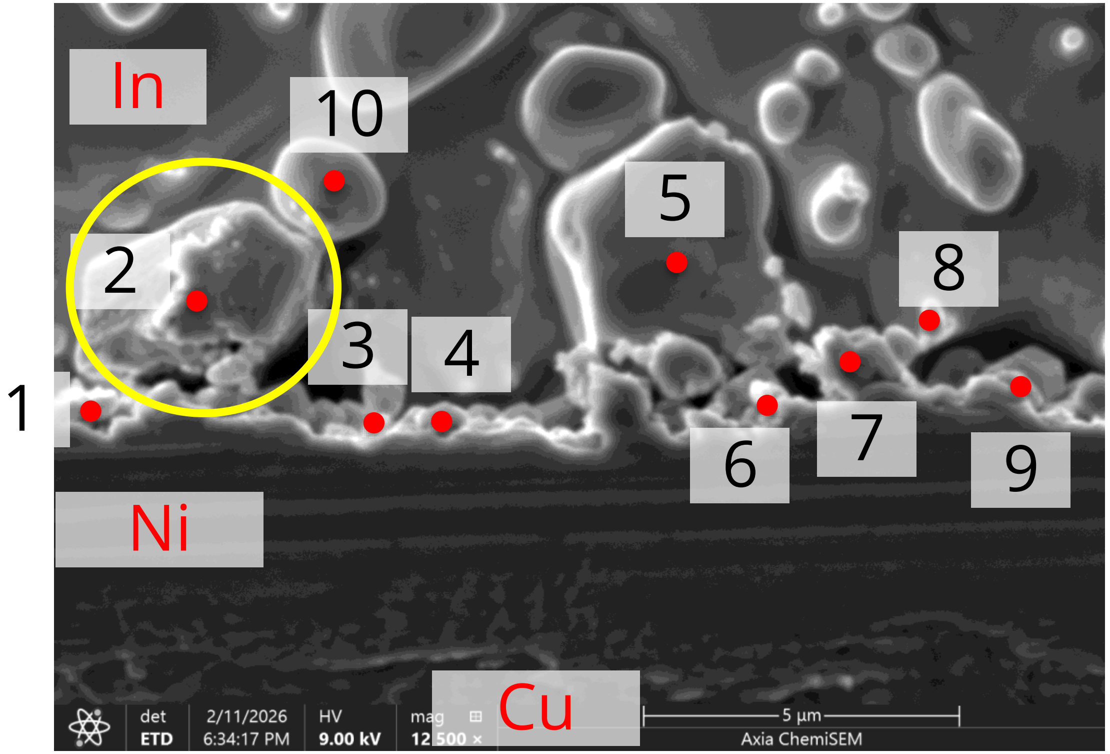
$In_{97}Ag_3$

In/Ag – 97/3 alloy – Time Zero

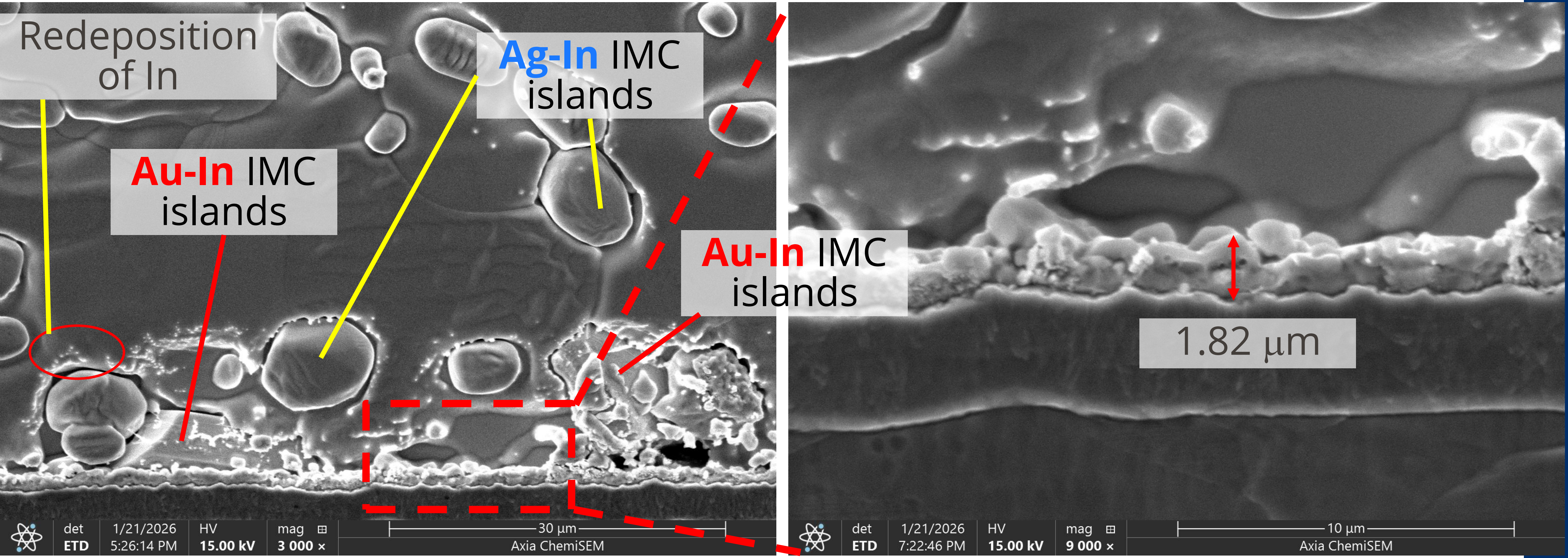
- $AgIn_2$
  - P5, P8, P10
  - IMC islands: size range from submicron to ~5 μm
- $AuIn_2$
  - P2, IMC island: ~4 μm
  - A small amount of Au in Ni-In IMC layer
- Ni-In
  - $NiIn_2$ or $Ni_{28}In_{72}$



	Atomic %									
Element	**Point 1**	**Point 2**	**Point 3**	**Point 4**	**Point 5**	**Point 6**	**Point 7**	**Point 8**	**Point 9**	**Point 10**
Ni	29.6	0.0	32.7	33.2	0.0	24.1	24.9	0.0	30.0	0.0
In	67.0	72.1	64.3	63.9	67.2	75.9	71.6	75.8	66.8	68.2
Au	3.4	27.9	3.0	2.9	0.0	0.0	3.5	0.0	3.2	0.0
Ag	0.0	0.0	0.0	0.0	32.8	0.0	0.0	24.2	0.0	

$In_{97}Ag_3$

1000 cycles. Newer images reveal thicker IMC and gold islands mainly near surface.



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$In_{97}Ag_3$

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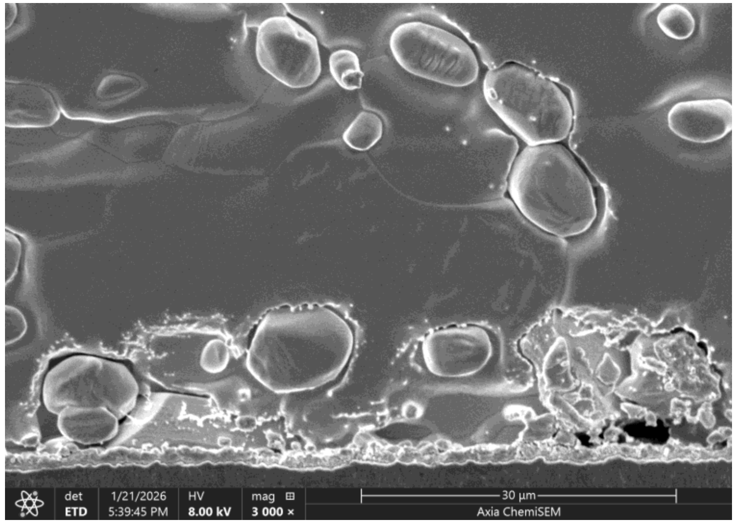
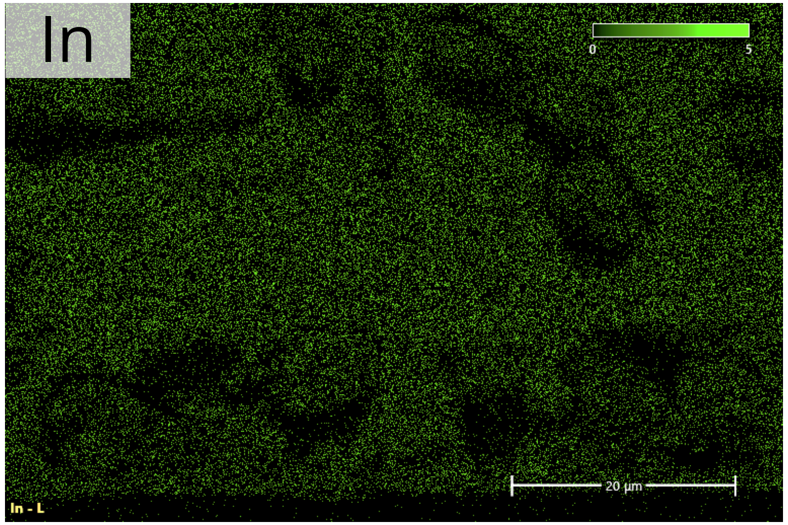
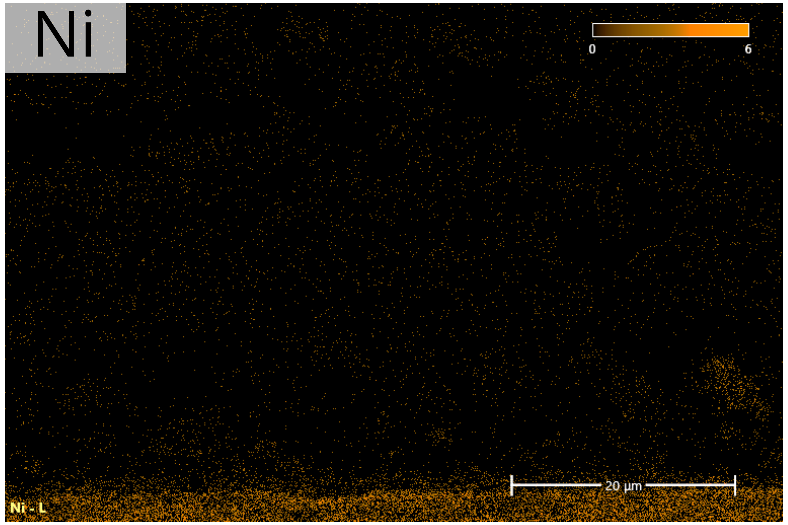
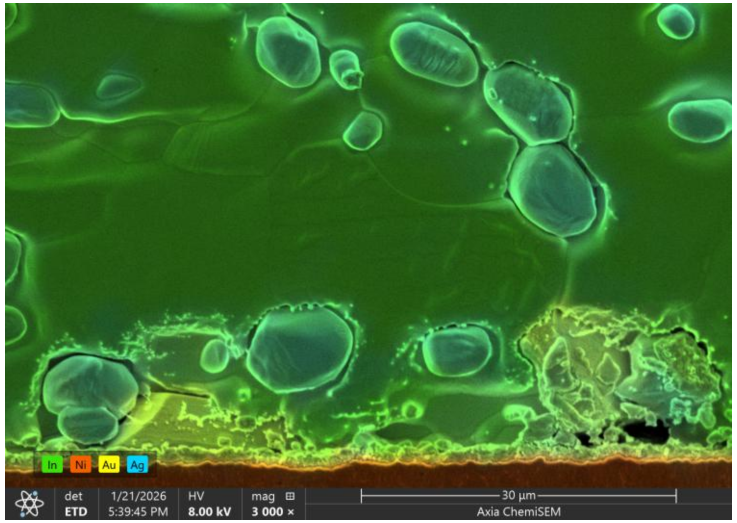
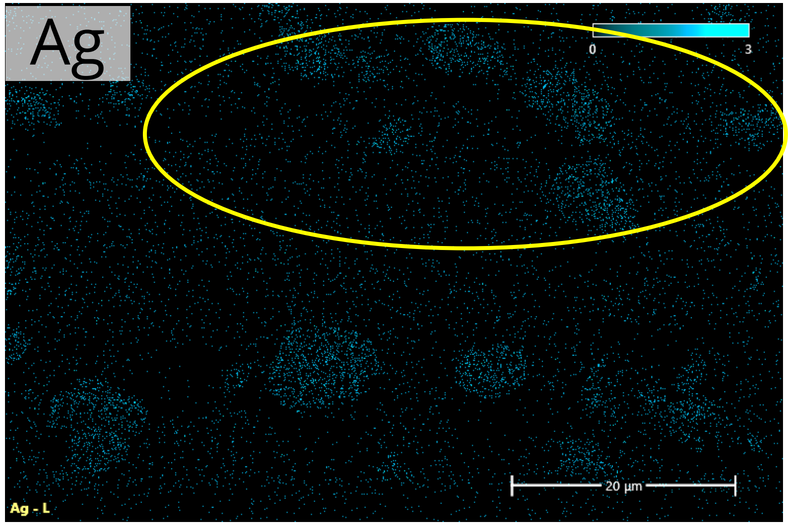
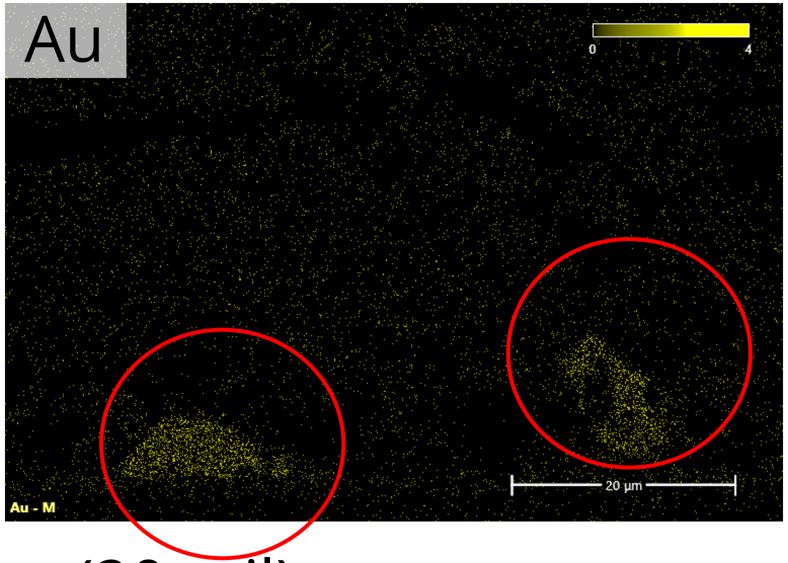








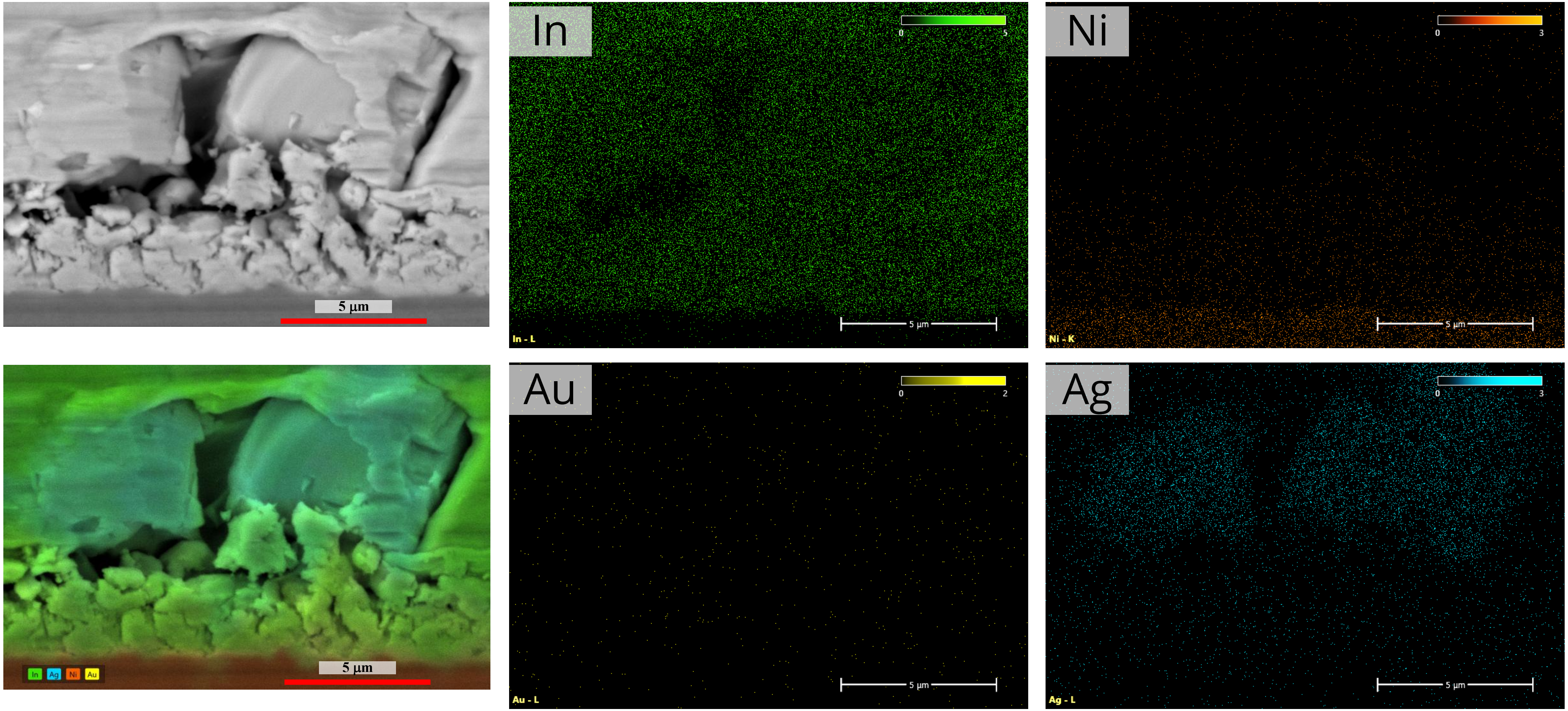




- In/Ag – 97/3 alloy preform, 508 µm (20 mil)

$In_{97}Ag_3$

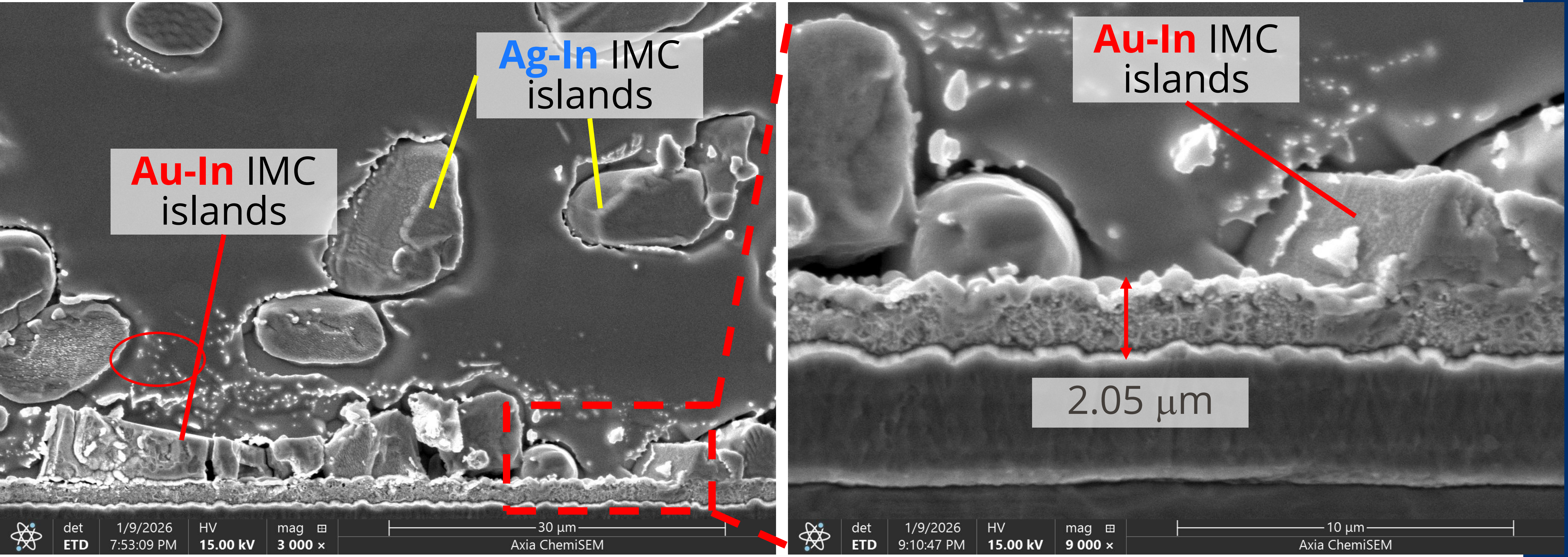
1500 cycles: Earlier result at IMC did not reveal gold islands



- In/Ag – 97/3 alloy preform, 508 µm (20 mil)

$In_{97}Ag_3$

1500 Cycles: Gold islands are found.



- In/Ag – 97/3 alloy preform, 508 µm (20 mil)
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AREA CONSORTIUM

$In_{97}Ag_3$

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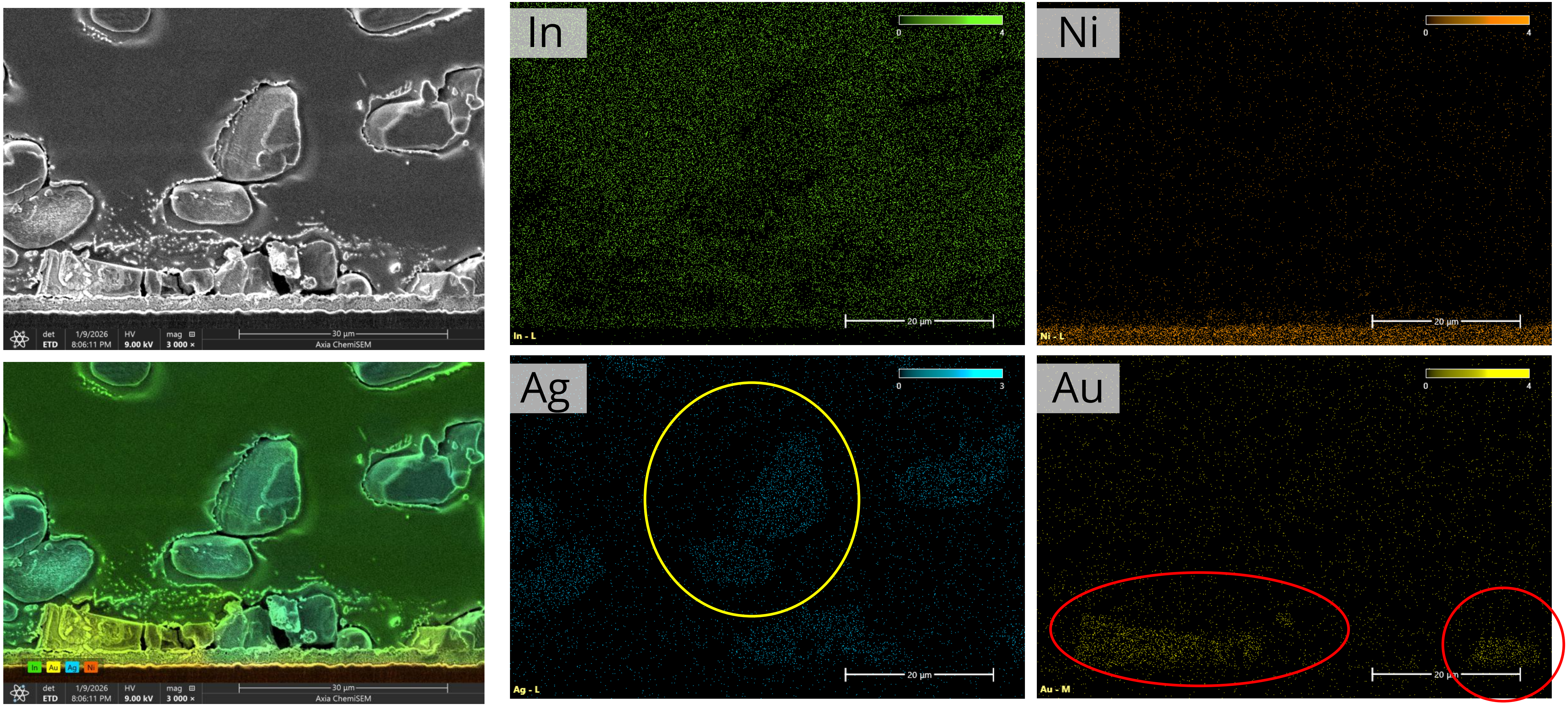
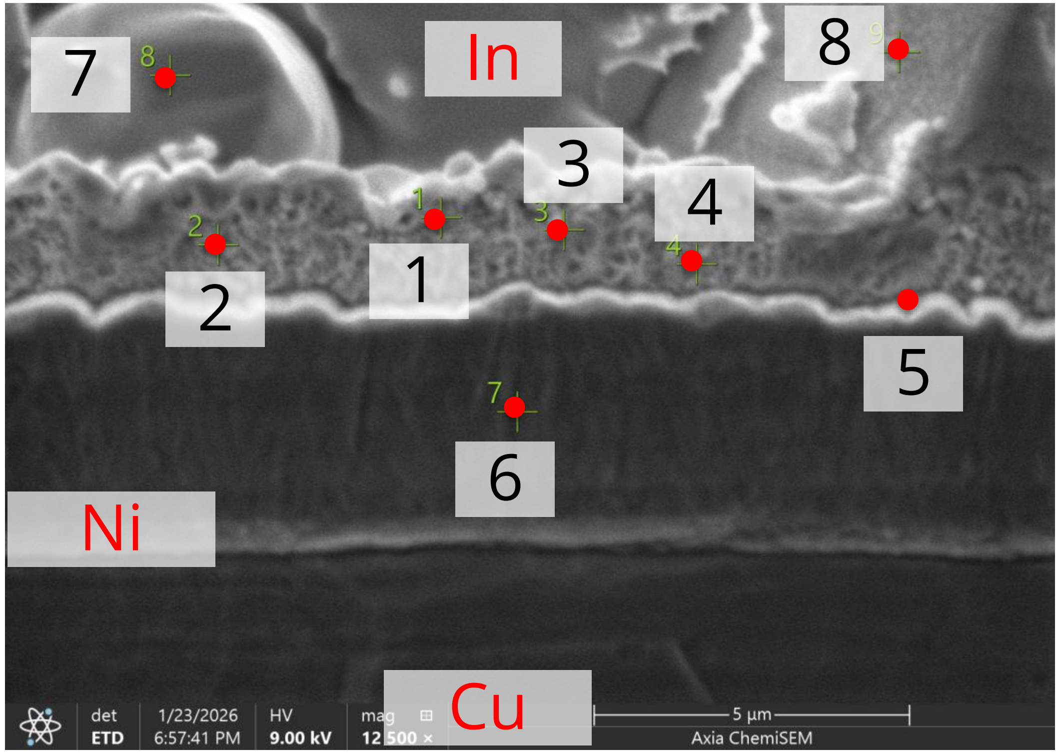


- In/Ag – 97/3 alloy preform, 508 µm (20 mil)

$In_{97}Ag_3$

1500 Cycles: Gold islands are found.

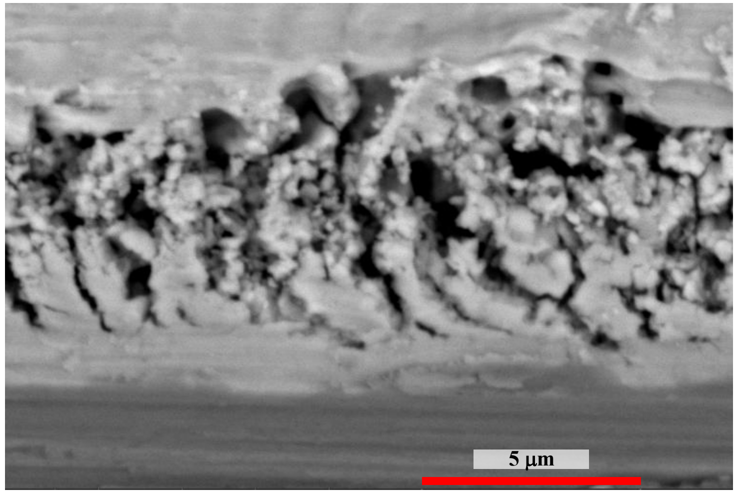
- $AgIn_2$
  - P7
- $AuIn_2$
  - P8, IMC island
  - A small amount of Au in Ni-In IMC layer
- Ni-In
  - $NiIn_2$ or $Ni_{28}In_{72}$
  - From P1 to P5, Ni concentration increases as getting closer to Ni layer

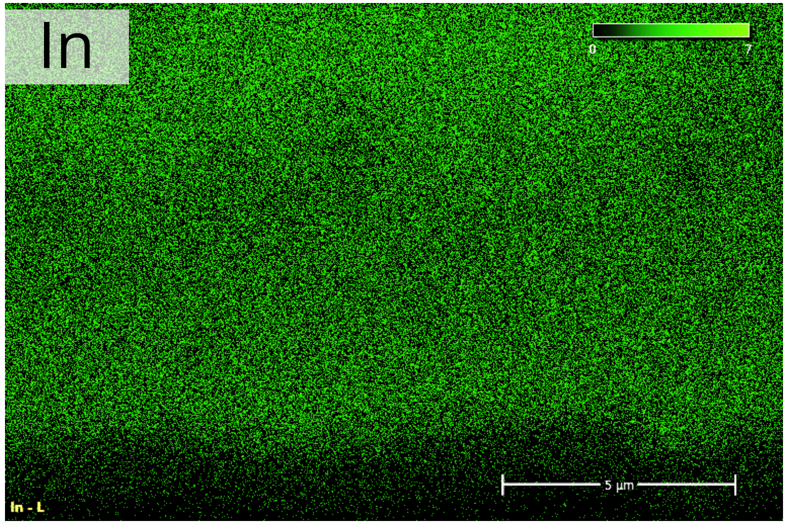


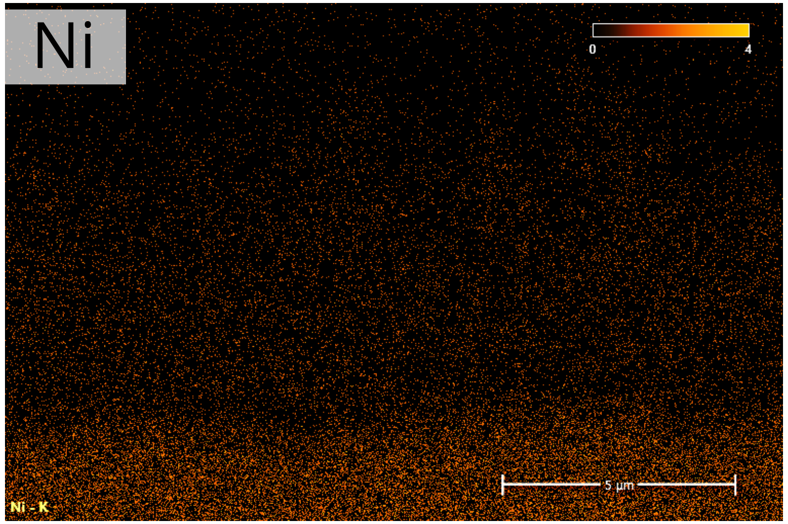
	Atomic %							
Element	**Point 1**	**Point 2**	**Point 3**	**Point 4**	**Point 5**	**Point 6**	**Point 7**	**Point 8**
Ni	25.2	30.5	35.6	35.7	76.7	100.0	0.0	0.0
In	71.5	69.5	64.4	64.3	23.3	0.0	69.6	70.4
Au	3.3	0.0	0.0	0.0	0	0.0	0.0	29.6
Ag	0.0	0.0	0.0	0.0	0	0.0	30.4	0.

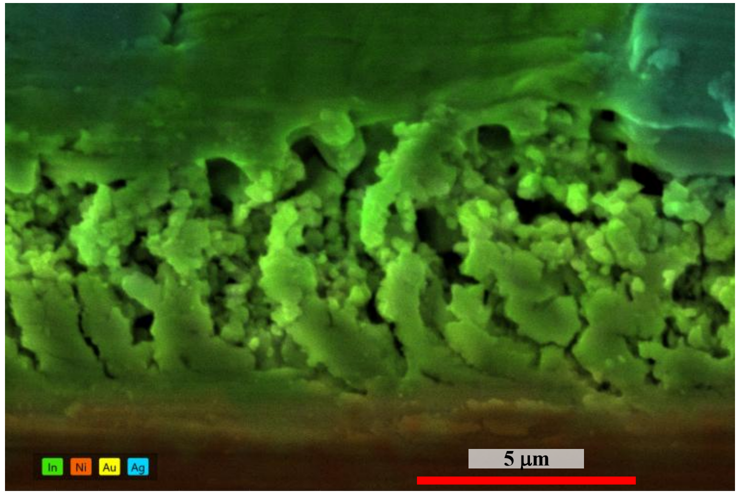
$In_{97}Ag_3$

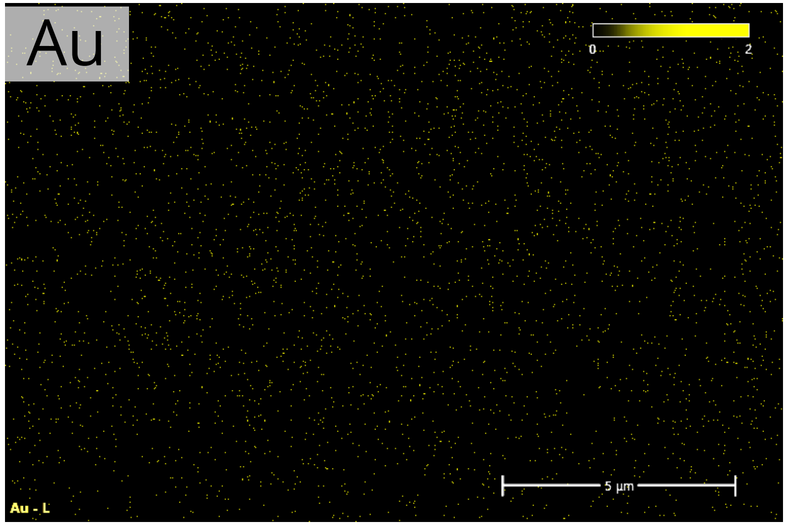
2000 Cycles Old image: IMC Vertical cracking; No gold.

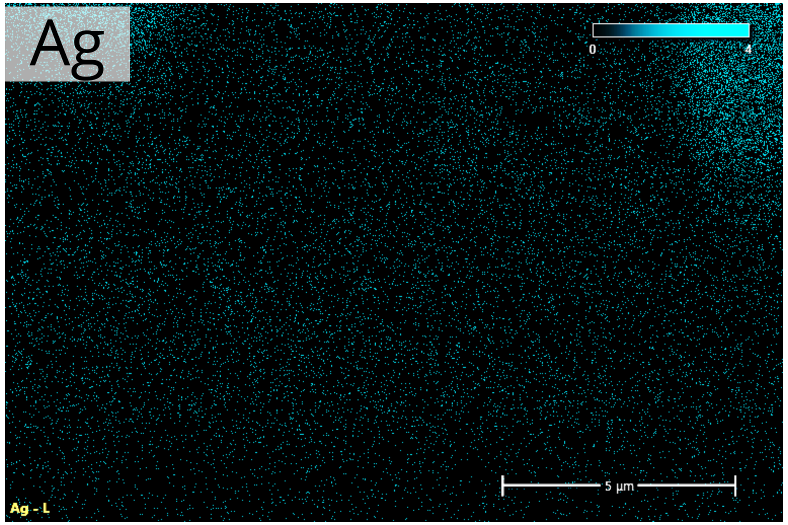








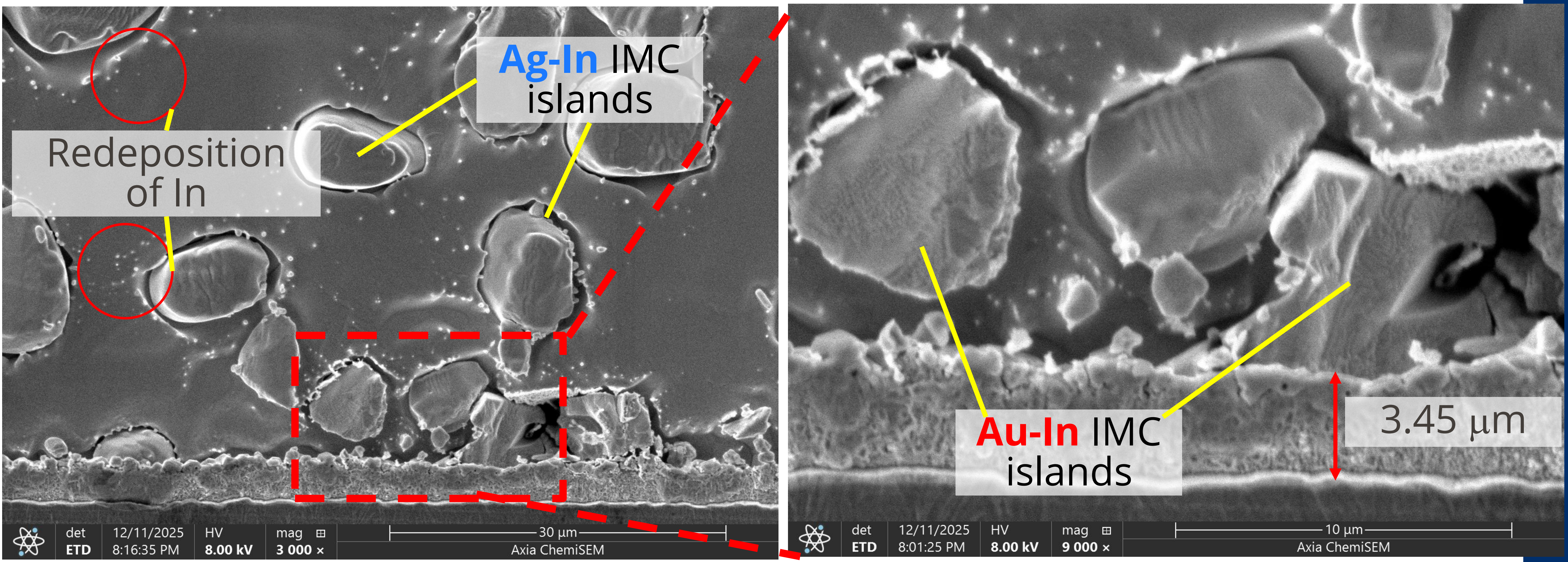




- In/Ag – 97/3 alloy preform, 508 µm (20 mil)

$In_{97}Ag_3$

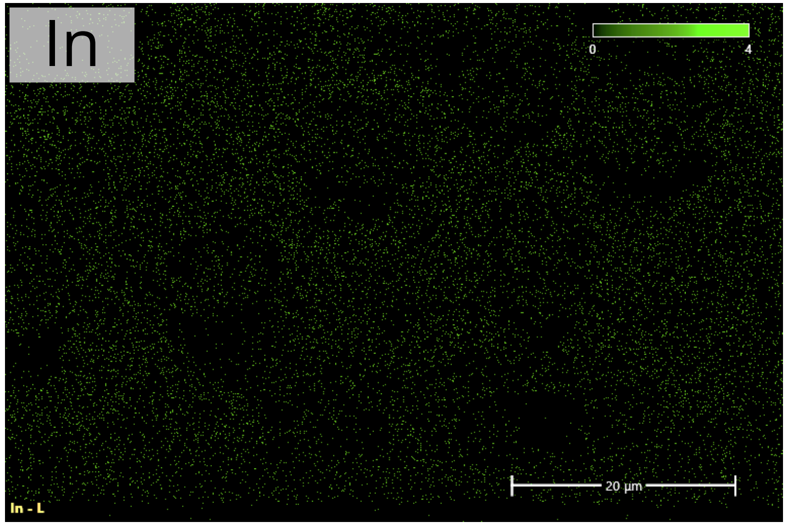
2000 Cycles: Vertical cracking is not observed

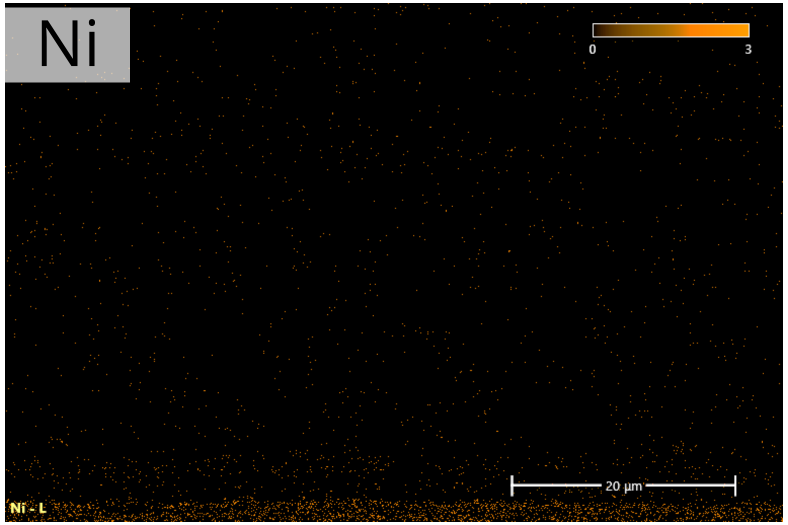


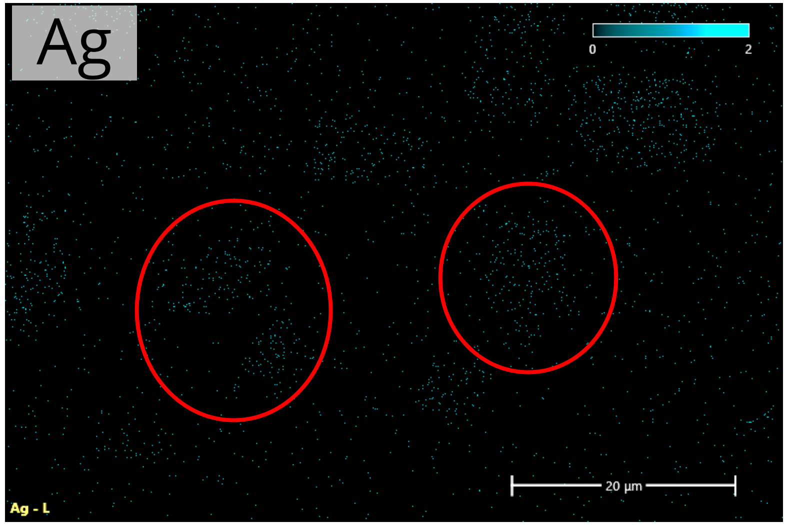
- In/Ag – 97/3 alloy preform, 508 µm (20 mil)
- IMC islands of Au-In and Ag-In
- Ion mill for 30 minutes

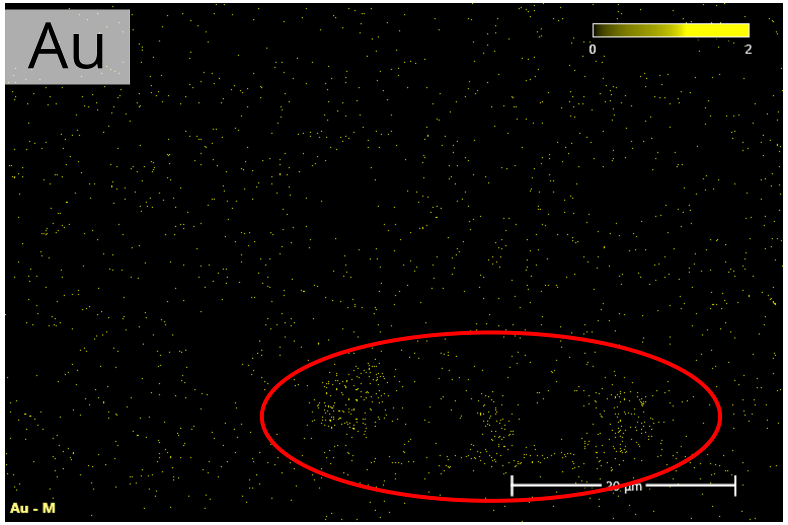
$In_{97}Ag_3$

In/Ag – 97/3 alloy – 2000 Temperature Cycles







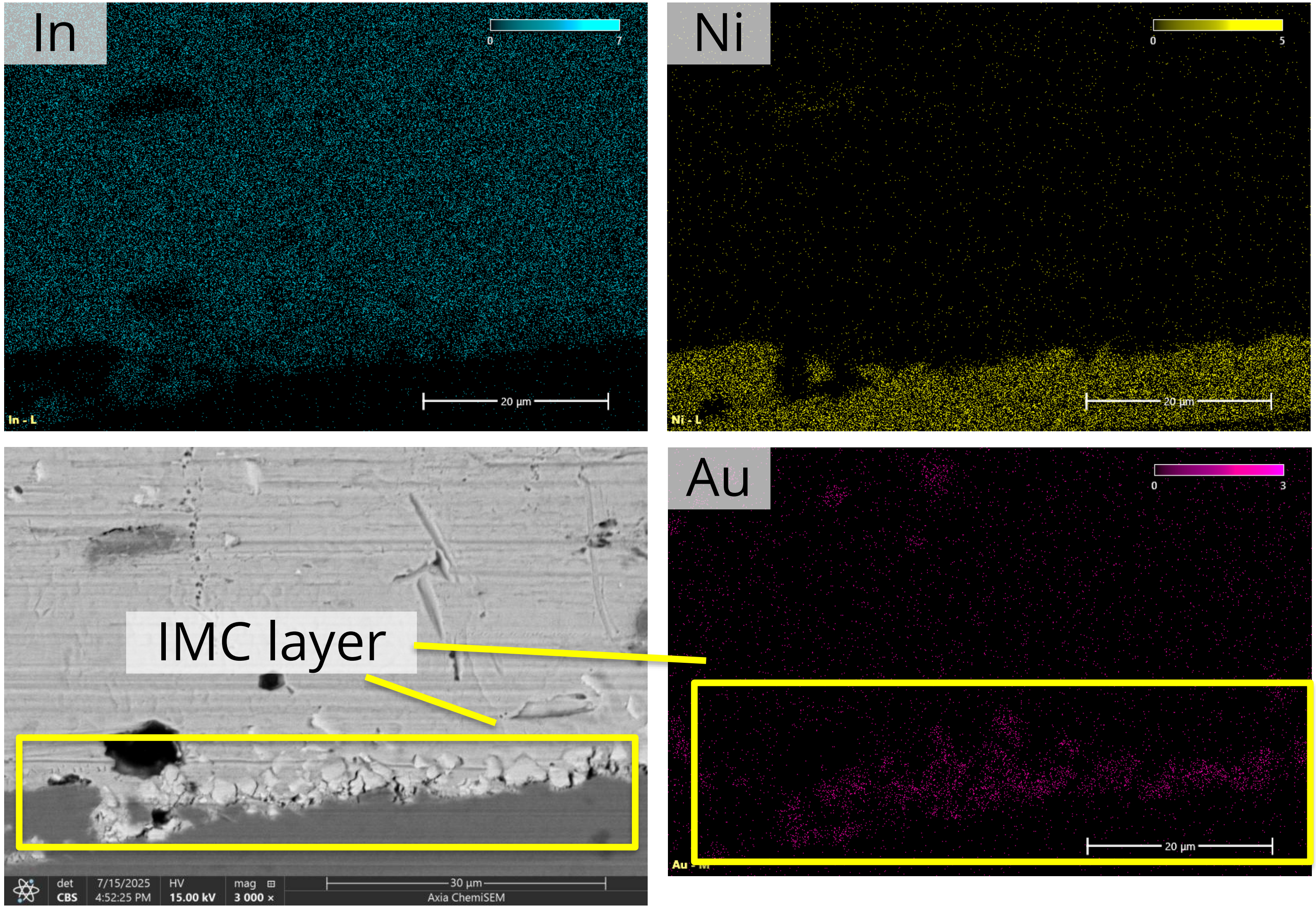


- In/Ag – 97/3 alloy preform, 508 µm (20 mil)

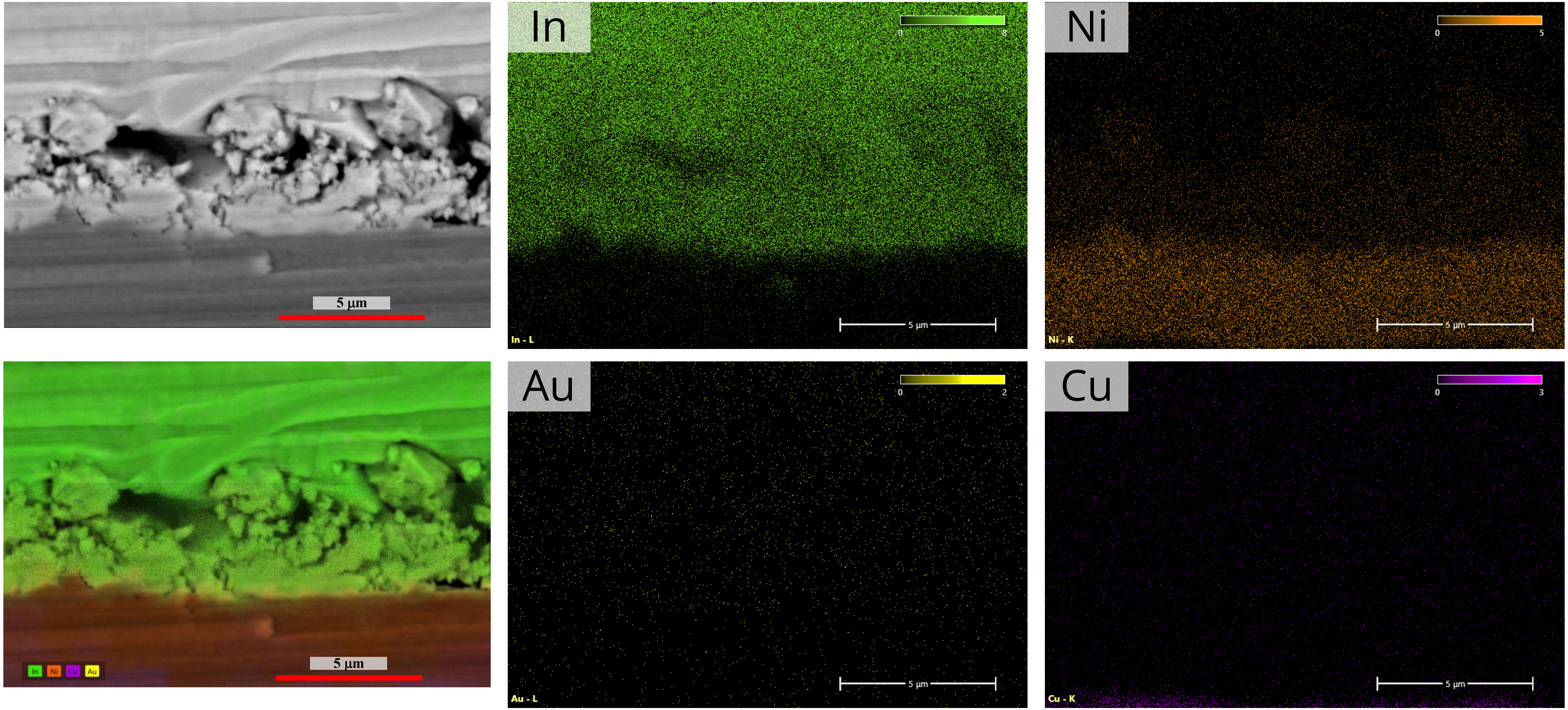
$In_{97}Ag_3$

In_{100}

T_0 Samples show overabundance of gold near IMC

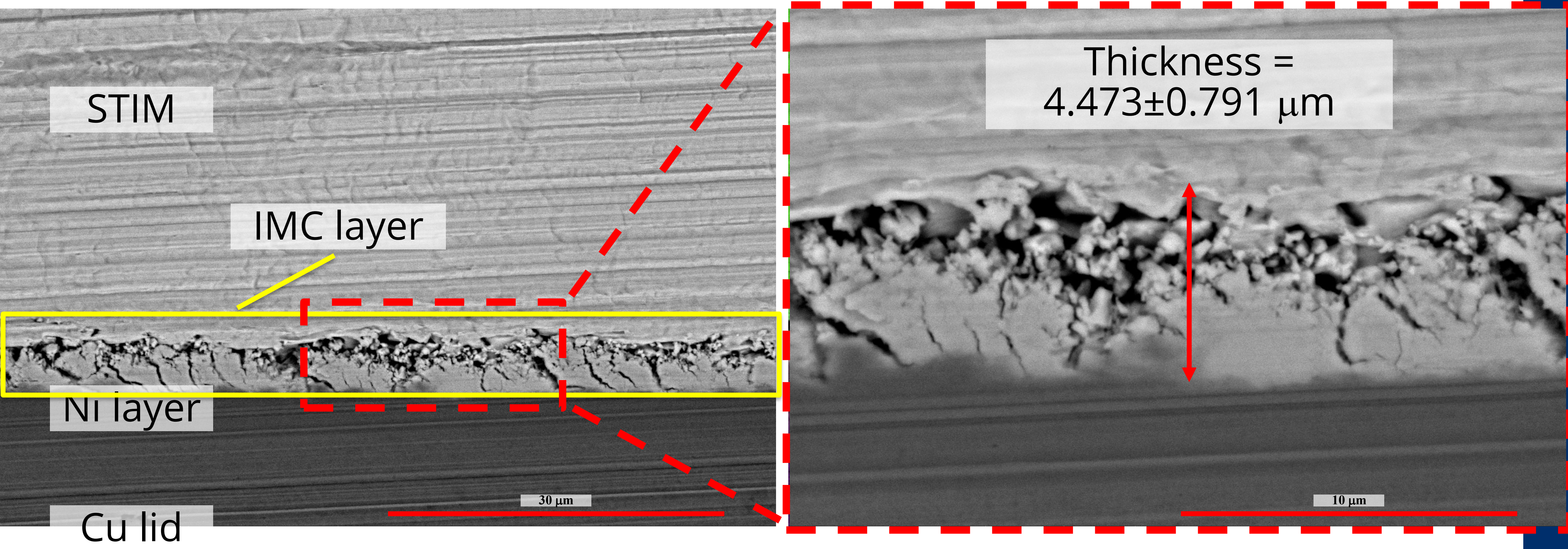


1000 Cycles: Largely dispersed gold



- In – 100 alloy preform, 508 µm (20 mil)

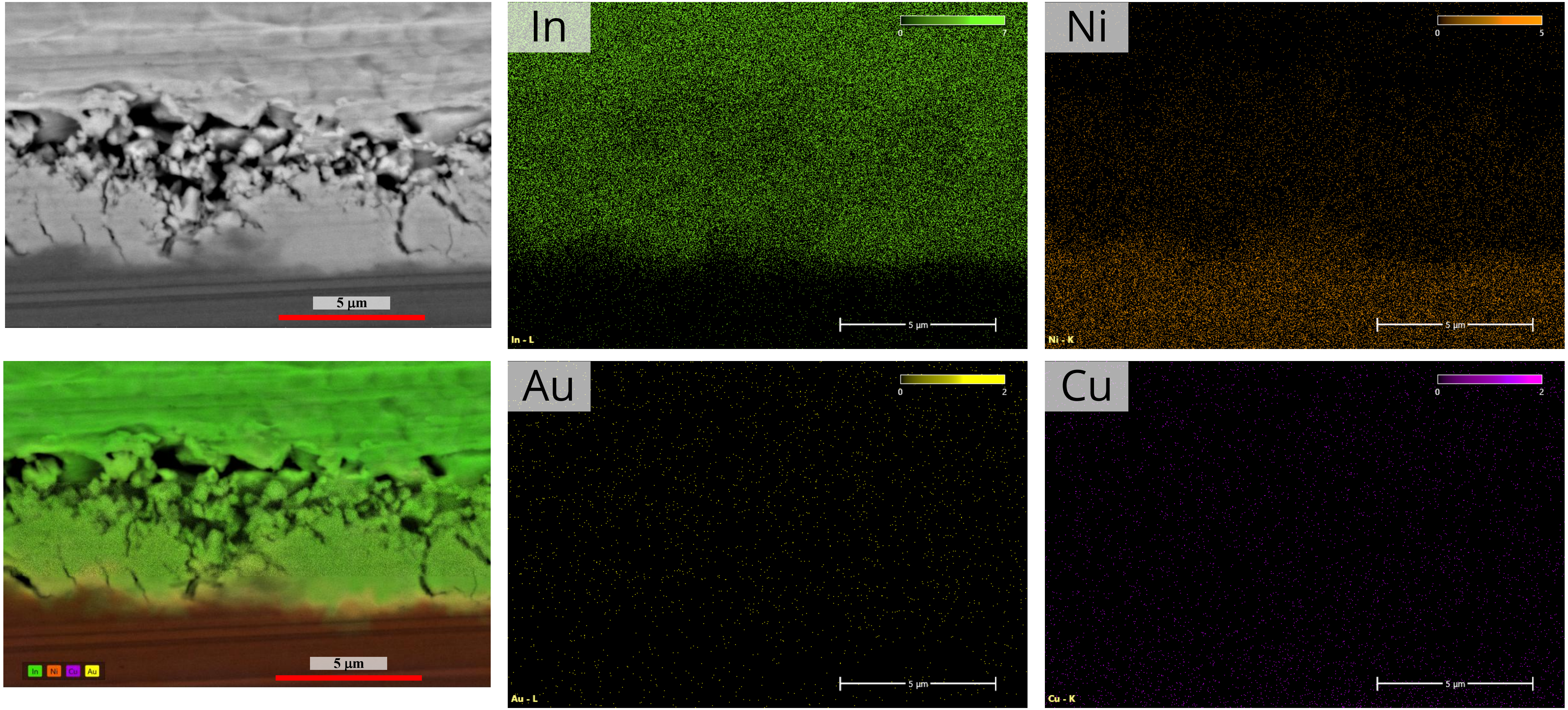
SEM analysis of prepared section at 1500 cycles



Thickness = 4.473±0.791 µm

- In – 100 alloy preform, 508 µm (20 mil)
- **Ni-In** IMC layer keeps growing at Cu/solder interface

EDX mapping of prepared section at 1500 cycles

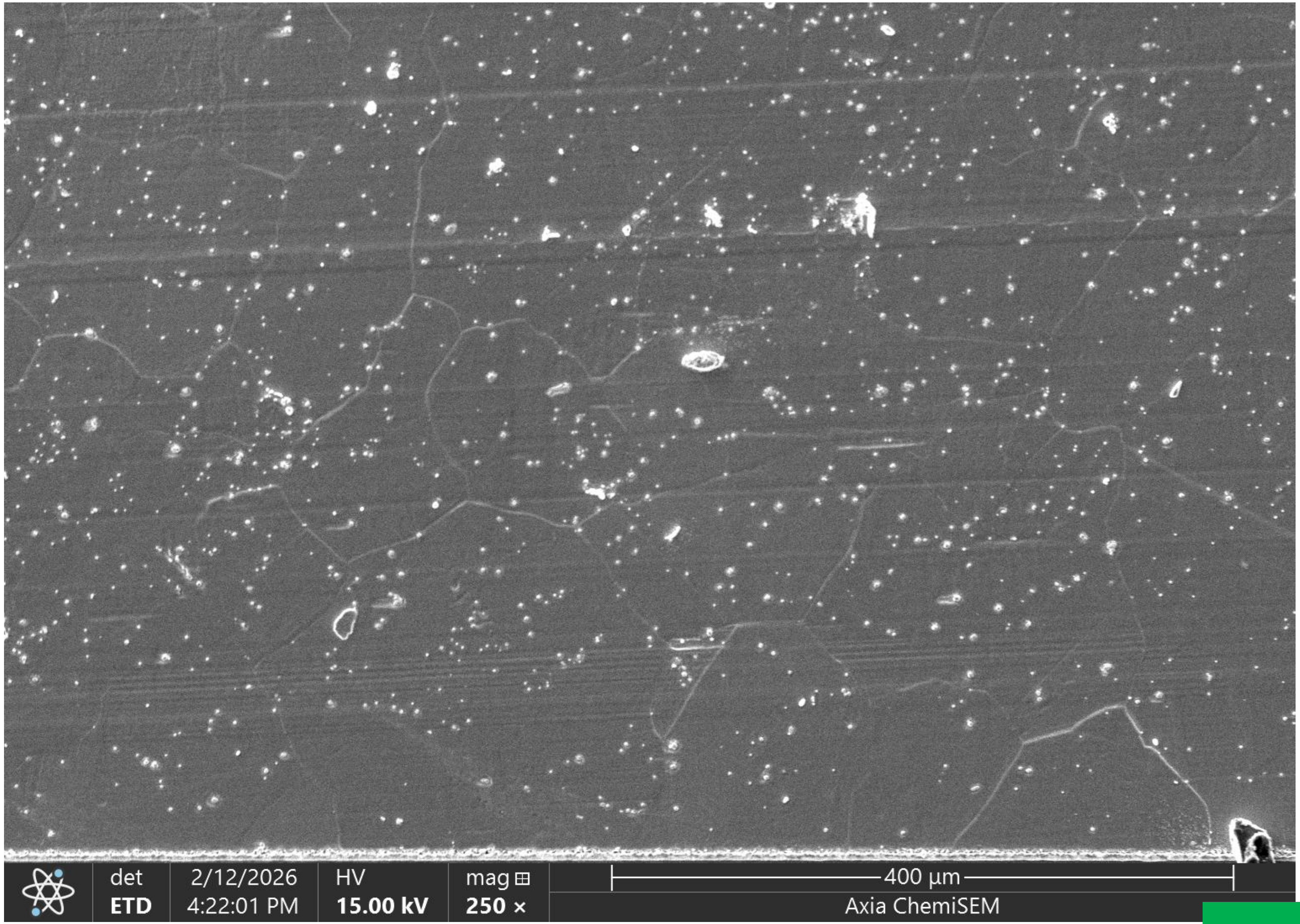


- In – 100 alloy preform, 508 µm (20 mil)
- No Au layer was detected

In$_{100}$

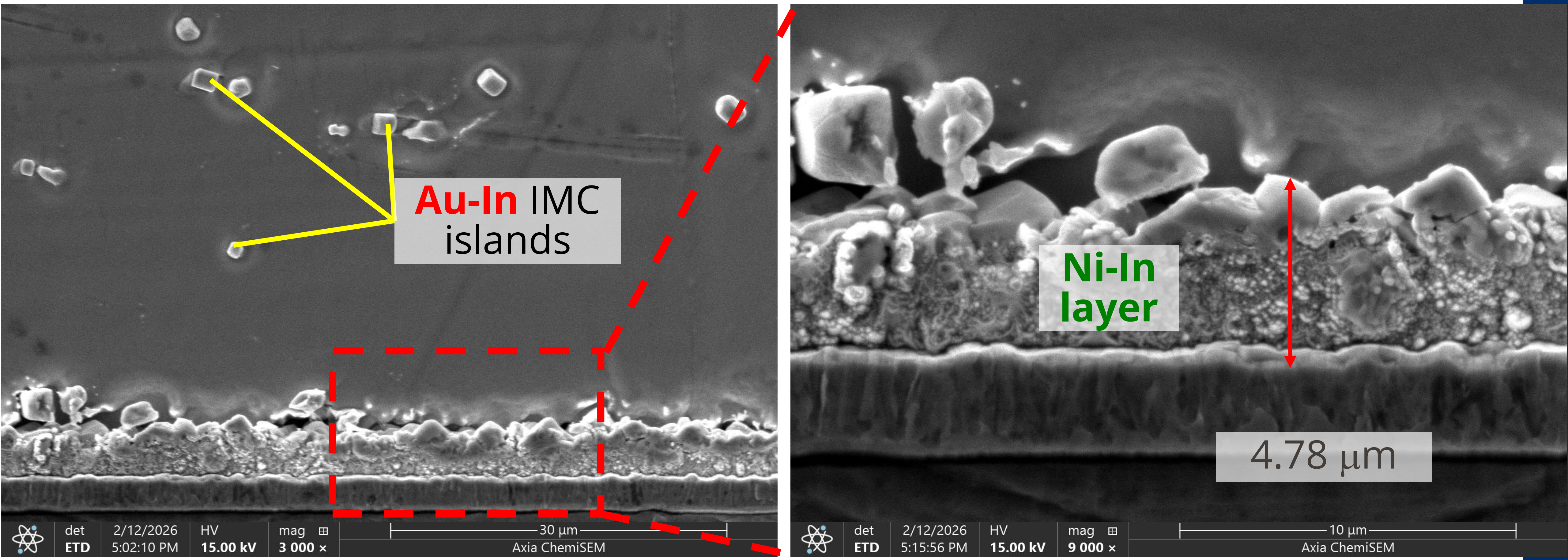
1500 Cycles: New images show gold throughout sample

- In – 100 alloy preform, 508 µm (20 mil)
- The cross-section is covered by Au-In particles



In_{100}

1500 Cycles: New images show gold throughout sample

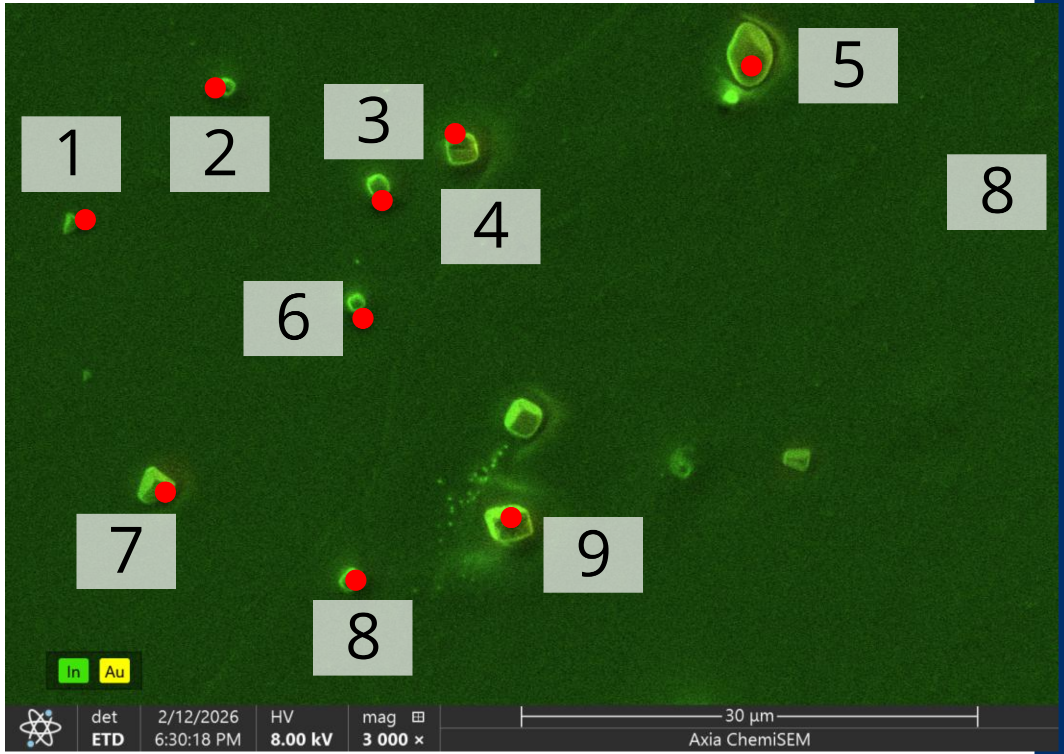


- In/Ag – 100/0 alloy preform, 508 µm (20 mil)
- IMC islands of Au-In
- Ion mill for 30 minutes

In$_{100}$

1500 Cycles: New images show gold throughout sample

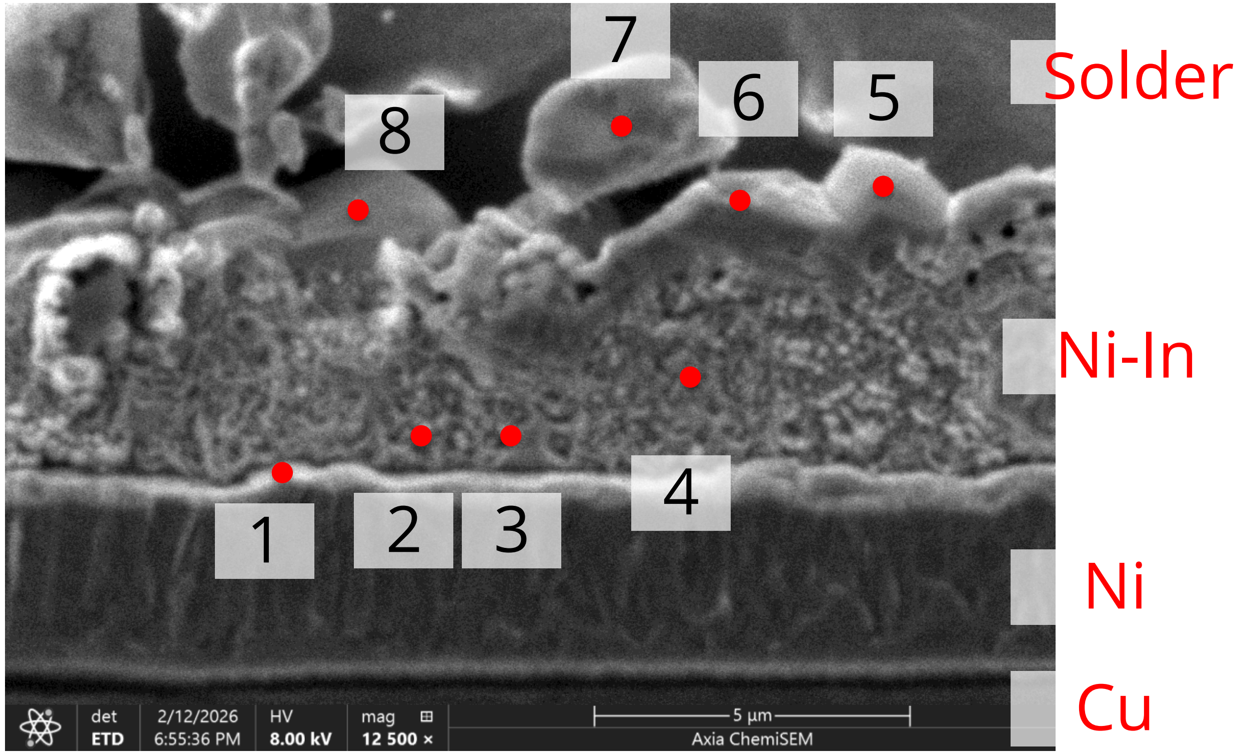
- In – 100 alloy preform, 508 µm (20 mil)
- Most of the particles are **Au-In** IMC (**$AuIn_2$**)



	Atomic %								
Element	**Point 1**	**Point 2**	**Point 3**	**Point 4**	**Point 5**	**Point 6**	**Point 7**	**Point 8**	**Point 9**
In	71.3	73.3	71.5	70.7	70.7	71.4	73.0	71.2	70.7
Au	28.7	26.7	28.5	29.3	29.3	28.6	27.0	28.8	29.3

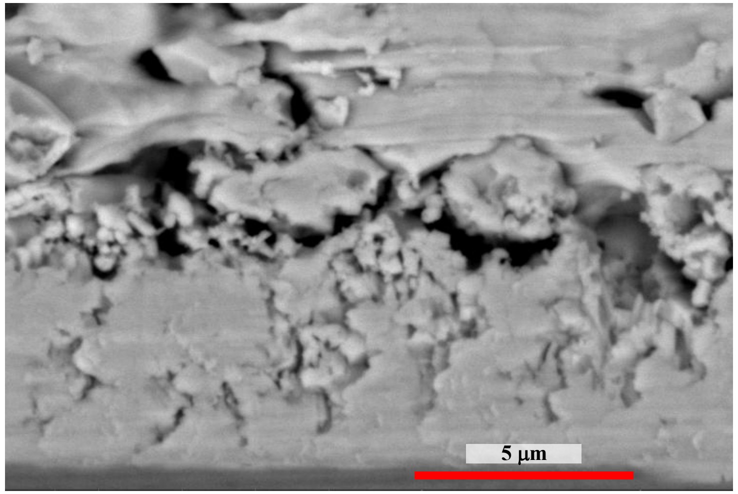
1500 Cycles: gold persists in interfacial IMC, not as specific gold-indium intermetallics

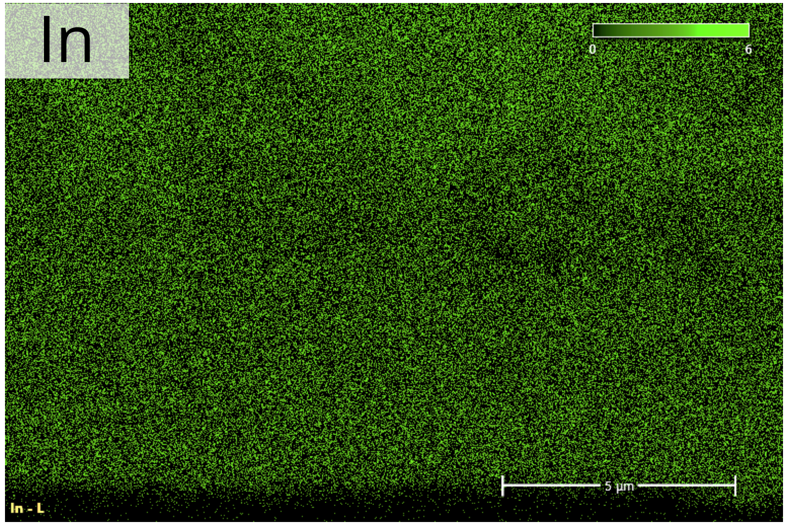
- $AuIn_2$
  - A small amount of Au in Ni-In IMC layer
- Ni-In
  - $NiIn_2$ or $Ni_{28}In_{72}$

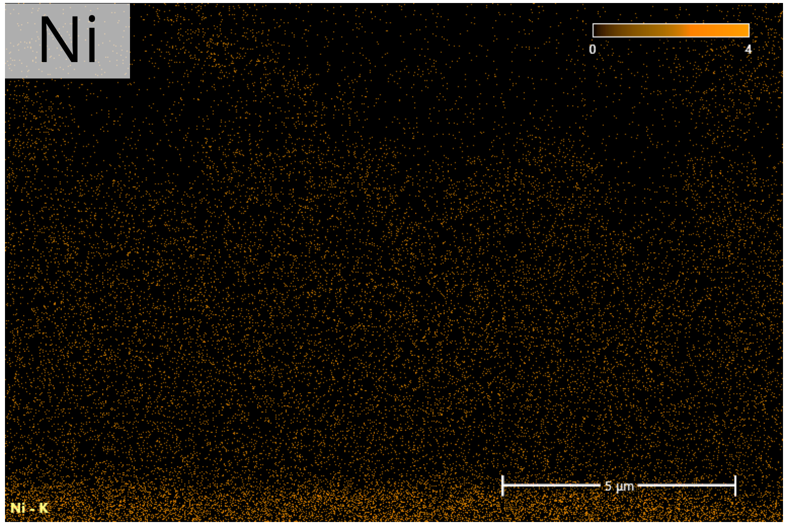


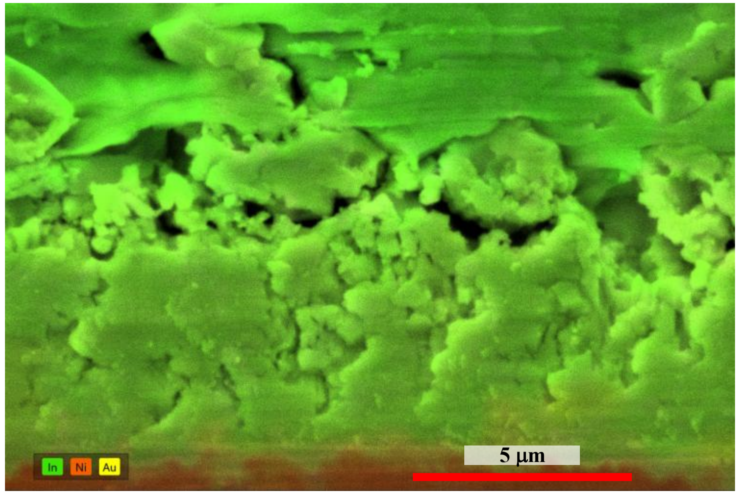
	Atomic %							
Element	**Point 1**	**Point 2**	**Point 3**	**Point 4**	**Point 5**	**Point 6**	**Point 7**	**Point 8**
Ni	43.1	32.6	33.7	16.0	28.8	28.7	27.9	29.3
In	56.9	67.4	66.3	84.0	66.0	66.7	67.6	66.1
Au	0	0	0	0	5.2	4.6	4.5	4.6

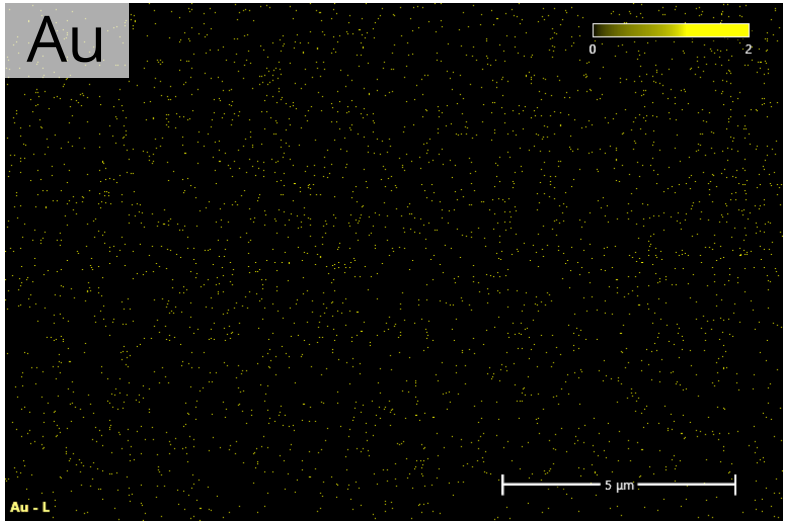
2000 Cycles low levels of gold





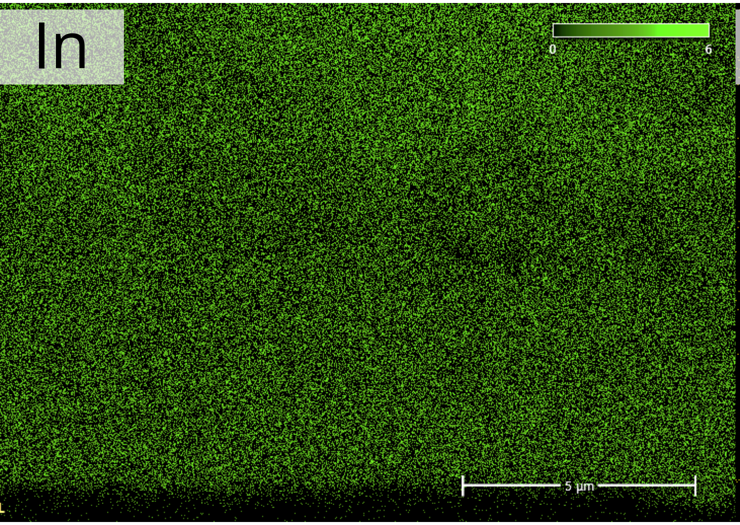


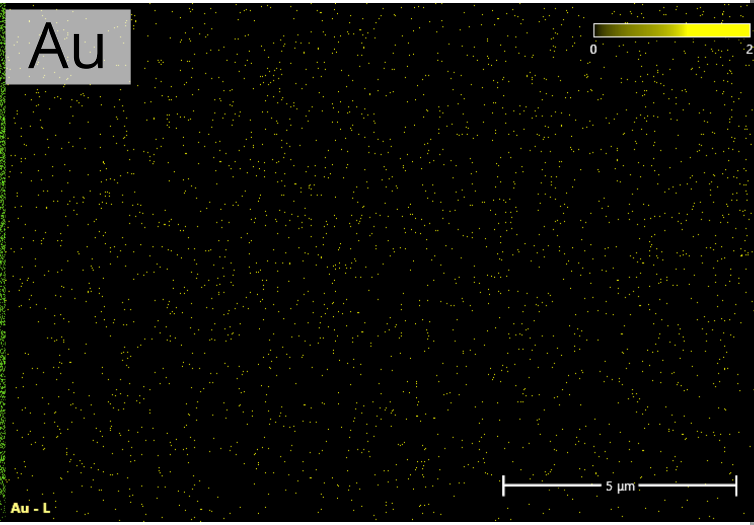


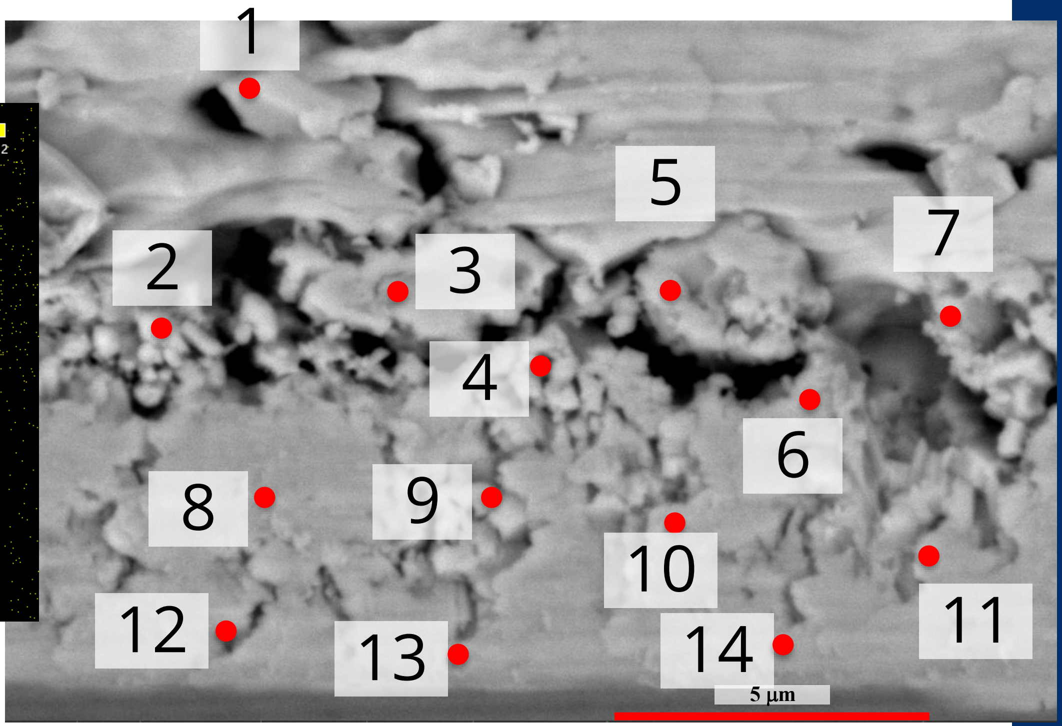


- In – 100 alloy preform, 508 µm (20 mil)

Spot analysis of prepared section at 2000 cycles



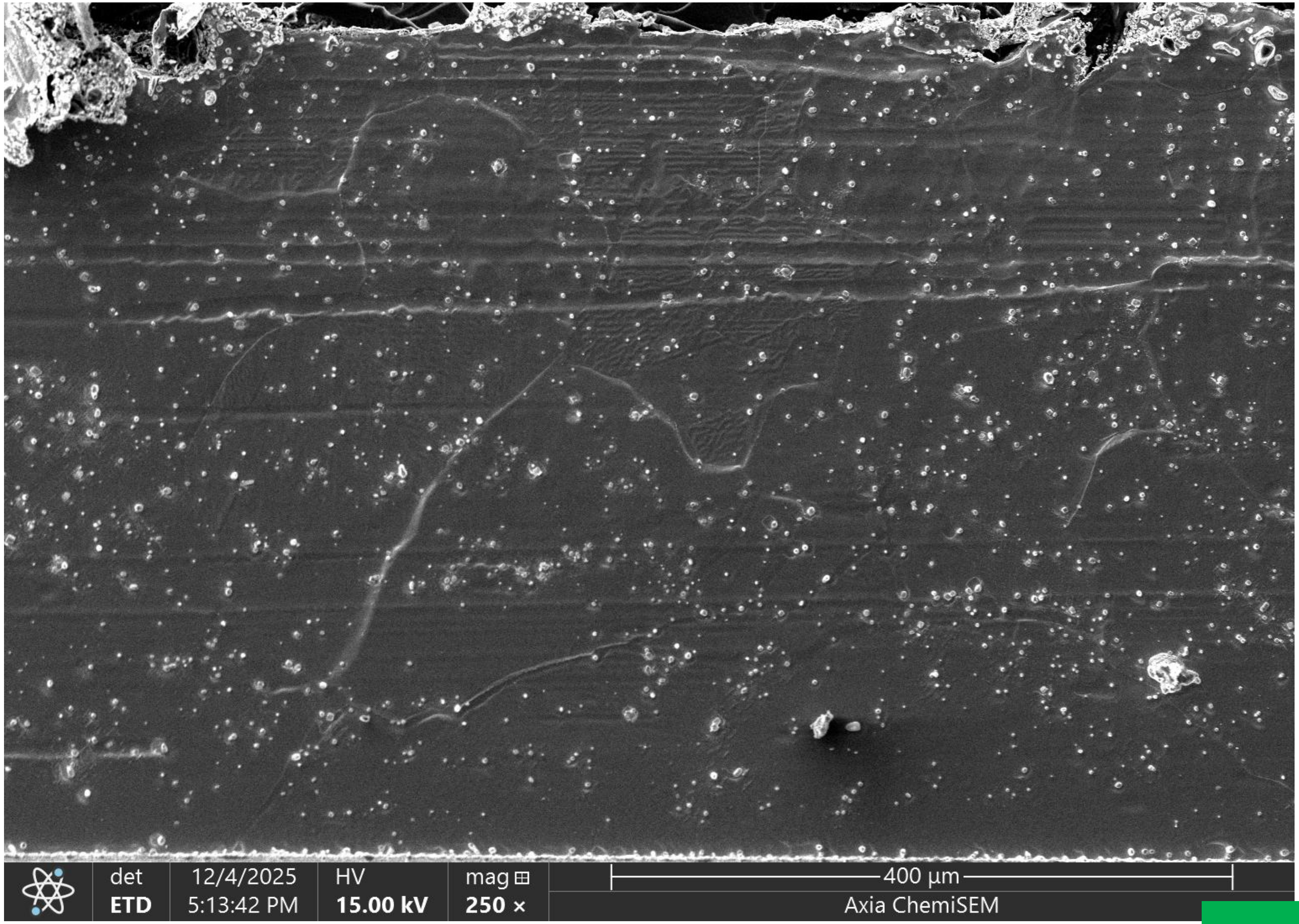




	Atomic %													
Element	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	Point 7	Point 8	Point 9	Point 10	Point 11	Point 12	Point 13	Point 14
Ni	22.0	23.1	22.8	25.6	23.8	27.0	23.6	25.5	27.5	28.7	26.0	31.0	39.1	33.4
In	73.0	71.6	73.6	73.5	72.0	73.0	72.4	74.5	72.5	71.3	74.0	69.0	60.9	66.6
Au	5.0	5.3	3.6	0.9	4.2	0.0	4.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

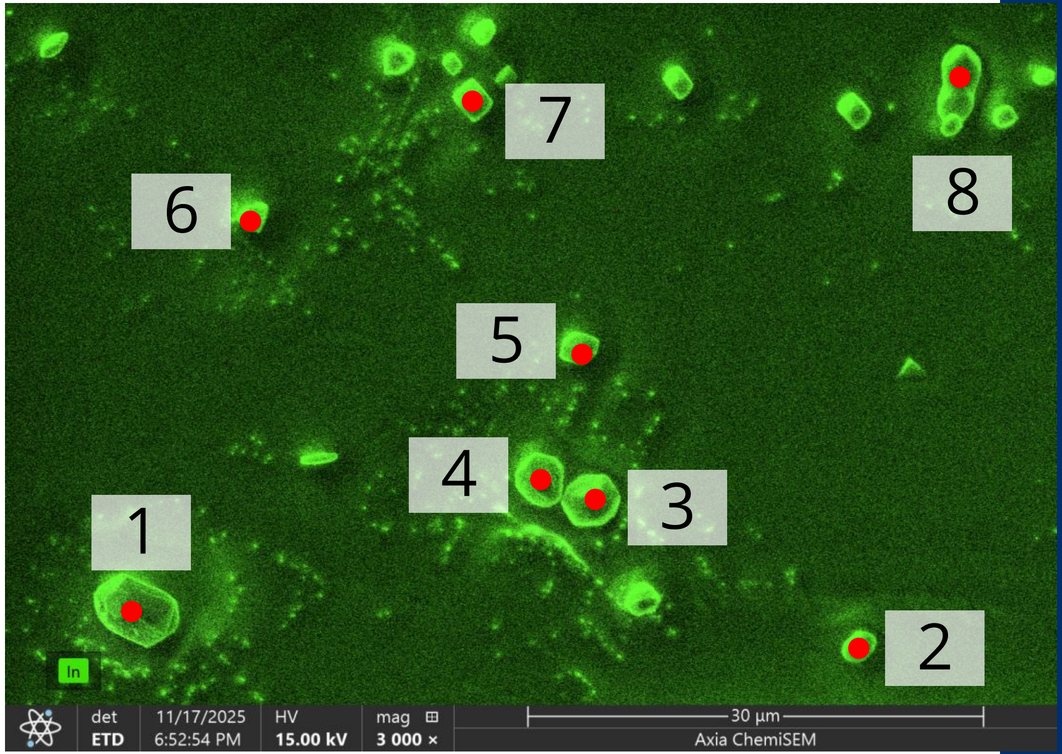
2000 Cycles/New Images: Gold Islands throughout sample.

- In – 100 alloy preform, 508 µm (20 mil)
- The cross-section is covered by Au-In particles
  Ion mill for 30 minutes



2000 Cycles: Gold islands throughout

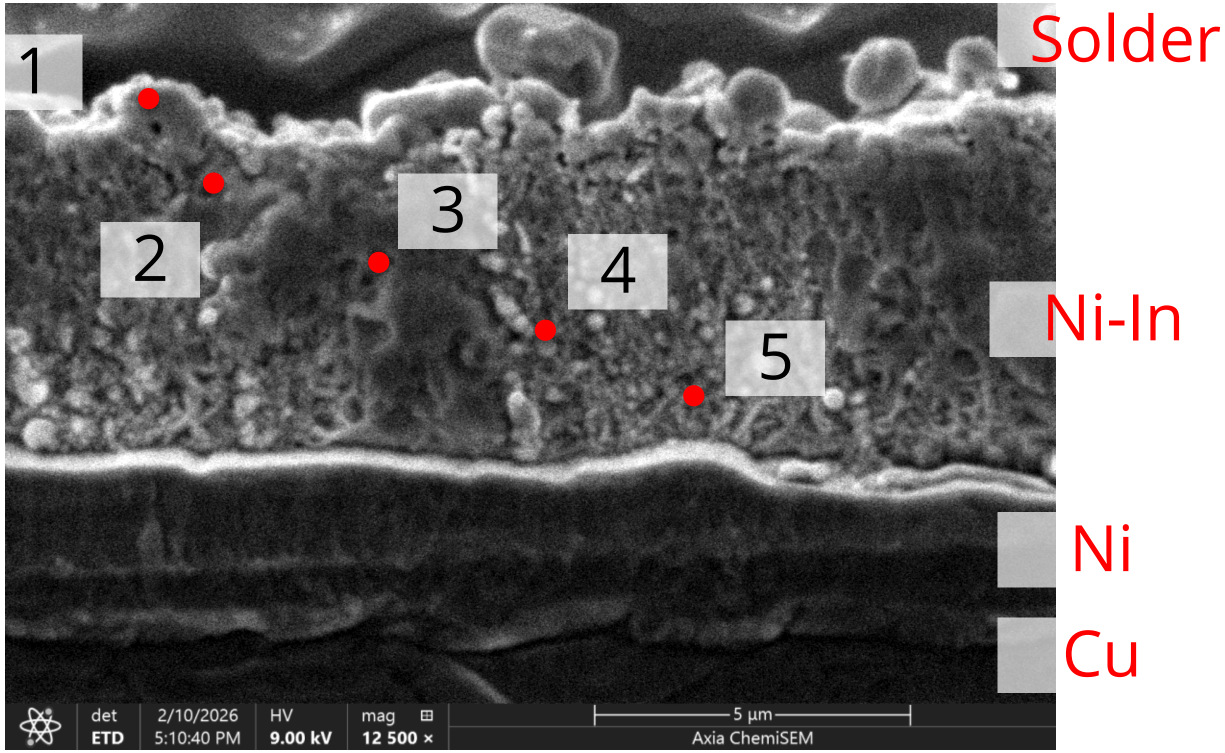
- In – 100 alloy preform, 508 µm (20 mil)
- Most of the particles are **Au-In** IMC (**$AuIn_2$**)



	Atomic %							
Element	**Point 1**	**Point 2**	**Point 3**	**Point 4**	**Point 5**	**Point 6**	**Point 7**	**Point 8**
In	72.4	67.3	71.0	73.4	72.9	78.0	71.5	76.1
Au	27.6	32.7	29.0	26.6	27.1	22.0	28.5	23.9

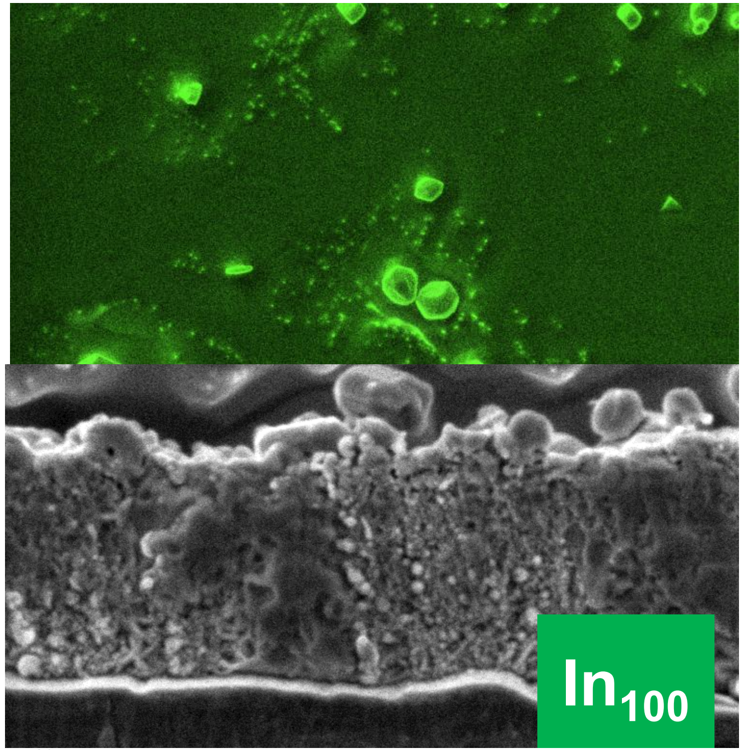
2000 Cycles: Trace gold at interface

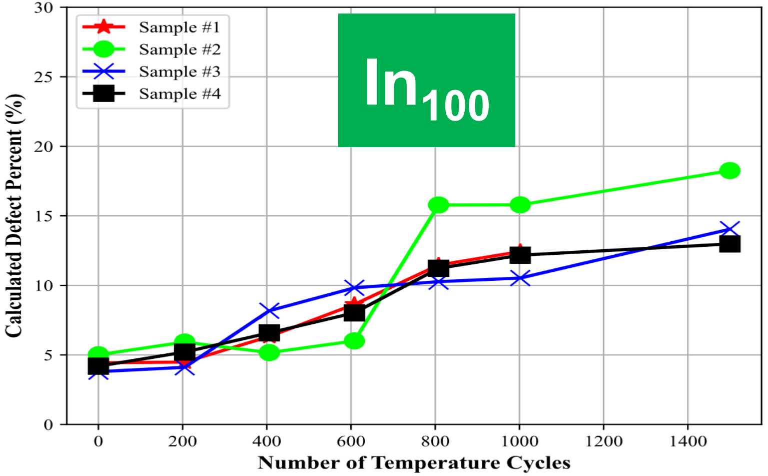
- $AuIn_2$
  - A small amount of Au in Ni-In IMC layer
- Ni-In
  - $NiIn_2$ or $Ni_{28}In_{72}$

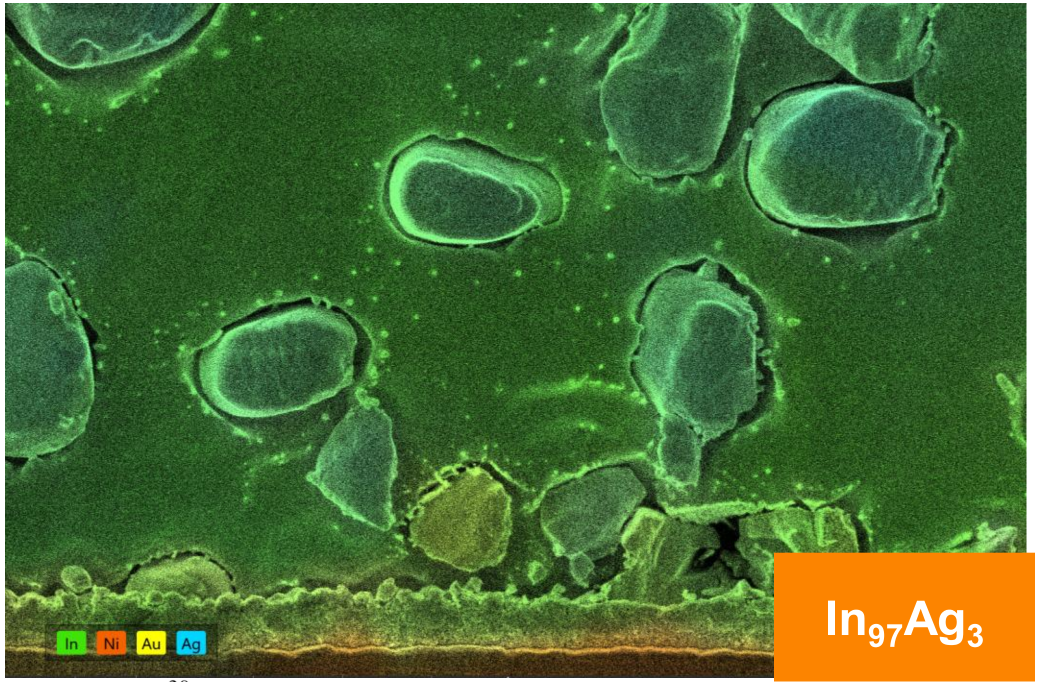


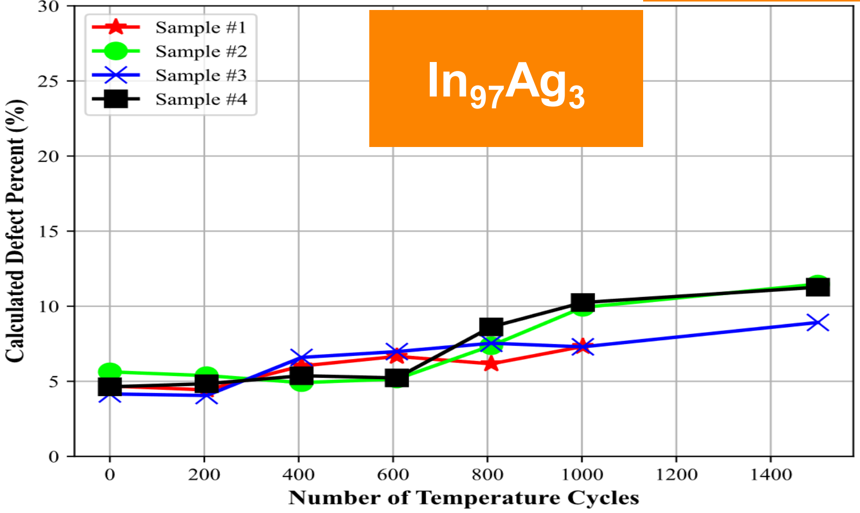
	Atomic %				
Element	**Point 1**	**Point 2**	**Point 3**	**Point 4**	**Point 5**
Ni	24.5	28.8	29.7	35.0	33.0
In	70.3	69.6	69.4	64.4	66.8
Au	5.2	1.6	0.9	0.6	0.2

Relative locations of gold intermetallics could impact reliability.



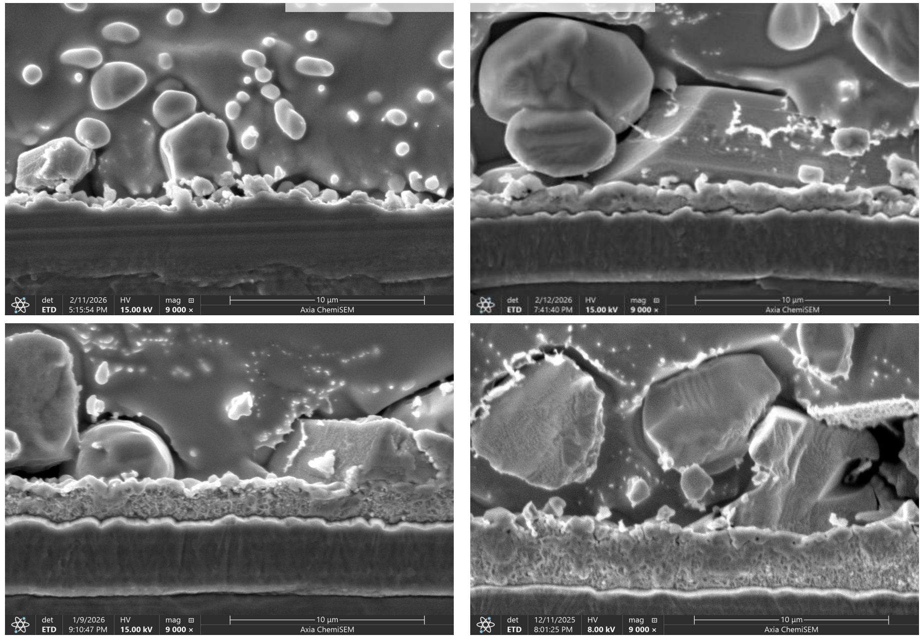




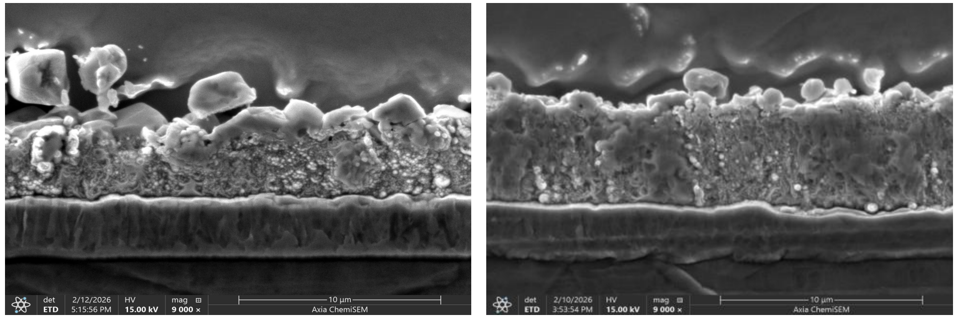


Much more work needs to be done.

97-3



In



Initial

1000

90-10

Missing

Thanks for listening, questions?