

AVIATION WEEK

Brazing Symposium 2014

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Active braze filler metal modification and optimization

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U.S. DEPARTMENT OF
ENERGY



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Acknowledgements

Tom Crenshaw

Alice Kilgo

Carl Smith

Shelly Williams

Outline

Background: Brazing, metal-nonmetal brazing considerations

“Standard” active brazing filler metals

Thin-film active element additions & calculations

Examples

Test samples

Results

Analysis

Discussion / Conclusions

Background: Brazing, metal-nonmetal brazing considerations

Brazing is a method of joining materials.

- 1) Takes place $\geq 450^{\circ}$ C
- 2) Parent substrate materials are not melted
- 3) Braze filler metal is drawn into and held within the braze joint by capillary attraction

Generally, standard brazing practices do not work on non-metallic (oxide, carbide, nitride) surfaces, failing to meet the 3rd requirement shown above. (filler metal must be or preplaced into the braze joint)

Brazing to a nonmetallic surface requires 1) surface modification* or 2) the use of non-standard braze filler metals.

Coefficient of Thermal Expansion (CTE) mismatch is especially important when brazing metals to nonmetals.

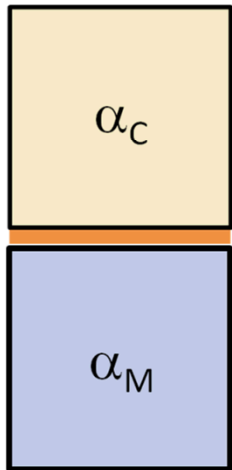
*direct brazing methods are an exception

Brazing metals to nonmetals = residual stress

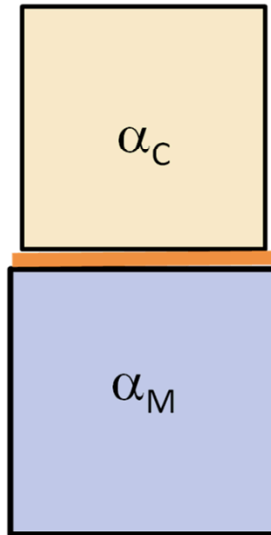
CTE Metal > CTE Ceramic, ($\alpha_M > \alpha_C$)

Unconstrained Substrates

Room
Temperature

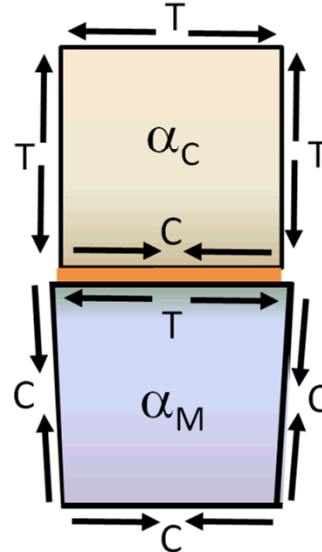


Joining
Temperature



Constrained Substrates

Post-Joining,
Room Temperature



94% - 97% Al_2O_3
brazed at $\sim 1000^\circ\text{C}$

Cu = $0.014''/1.000''$ mismatch
SS304 = $0.012''/1.000''$
Kovar = $0.004''/1.000''$
Niobium = $0.001''/1.000''$
Mo = $-0.002''/1.000''$
W = $-0.004''/1.000''$

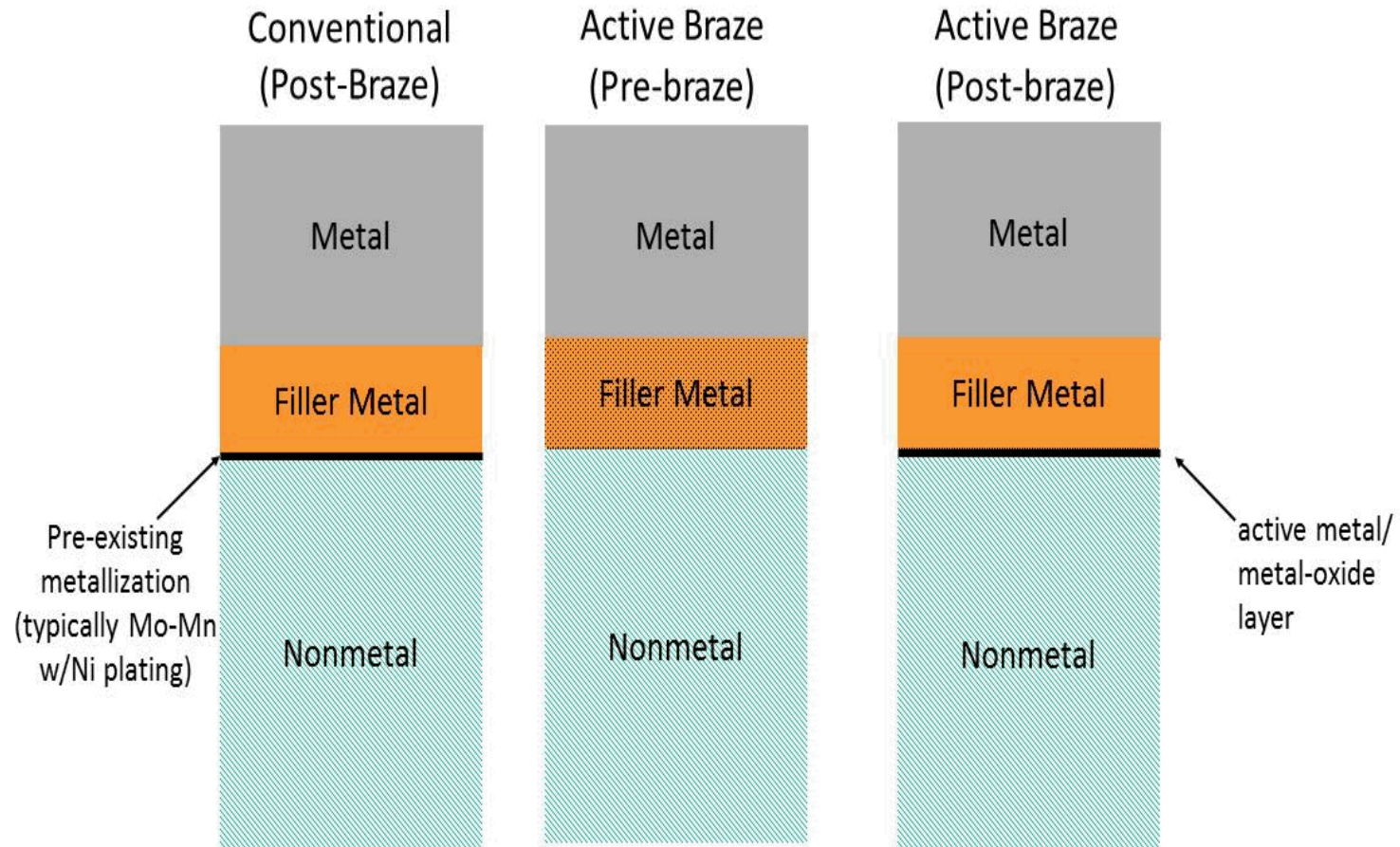
α_C = CTE Ceramic, α_M = CTE Metal
T = Tension, C = Compression,
 = Ceramic Substrate
 = Metal Substrate
 = Brazing Filler Metal
 = Stress Direction

Active brazing filler metals

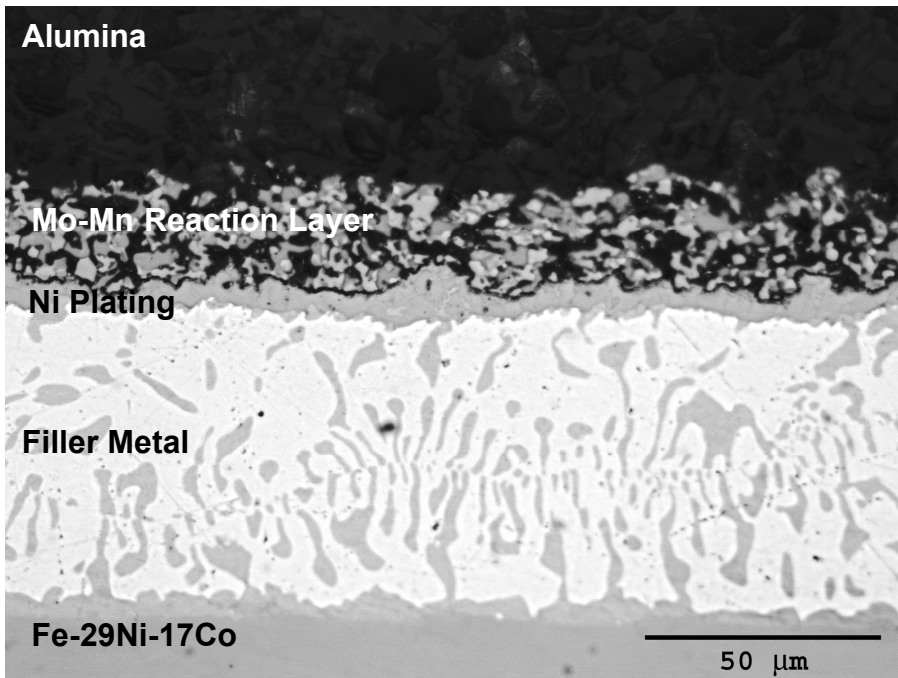
- Active brazing uses a filler metal that has been modified by the addition of a small amount (typically 1-2 wt. %) of a Group IVA element, such as: Ti, Zr, or Hf.
- These elements form strong oxides, that are very stable at the liquidus temperature ranges of braze filler metals.
- Commercially available active filler metal compositions are limited and generic. The result is a few compositions must be made to work with many applications and materials systems.
- For this study, two commercially* produced active filler metals were used as a baseline: 98Ag-2Zr and 62Cu-35Au-2Ti-1Ni (weight percent).

* Morgan Technical Ceramics
Wesgo Metals
Hayward, CA

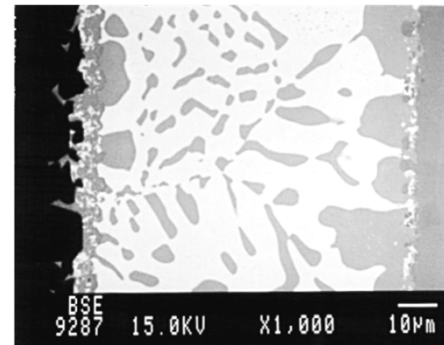
Conventional vs. active brazed metal-nonmetal assembly



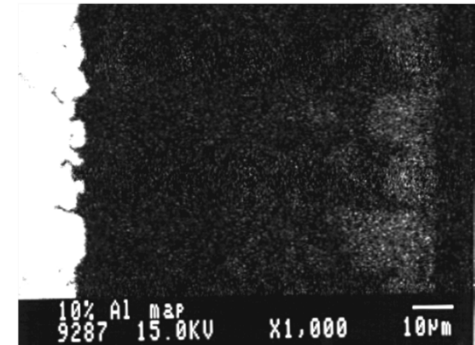
Conventional vs. active brazed metal-nonmetal assembly



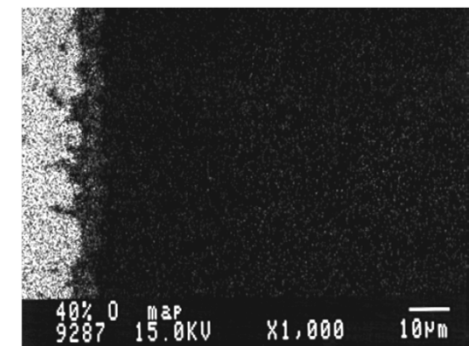
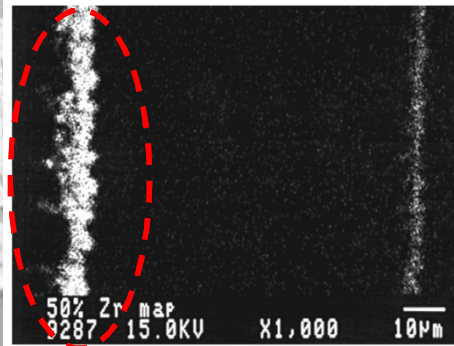
72Ag-28Cu, 810C - 3 min, dry H₂ AL-500 with Mo/Mn Metallization and Ni Plate, 500X



Alumina Braze Fe-29Ni-17Co



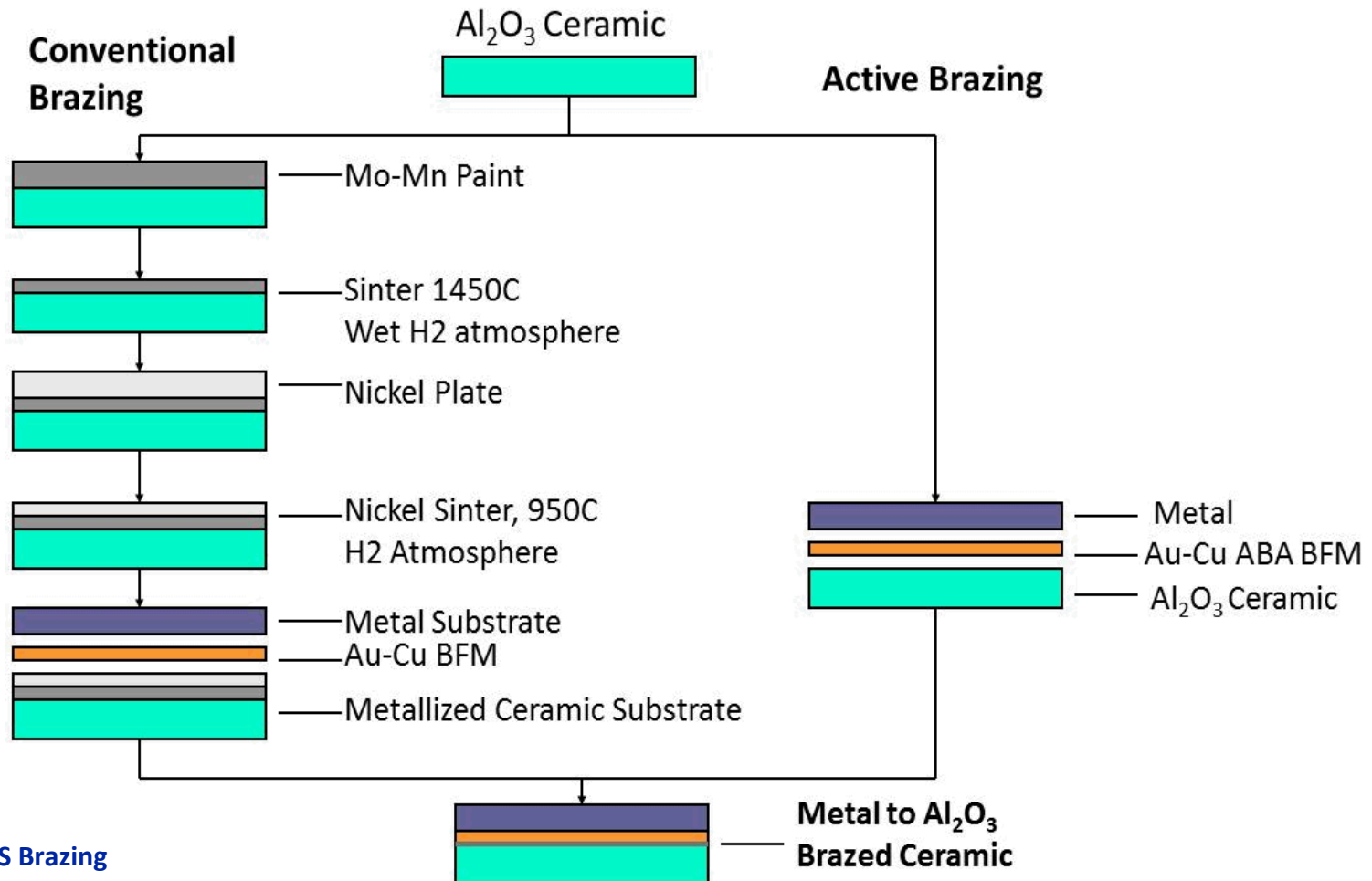
Alumina Braze Fe-29Ni-17Co



BSE and Probe Maps for a sample brazed with 97Ag-1Cu-2Zr at 950°C, 5 min in a 12 torr argon partial pressure atmosphere

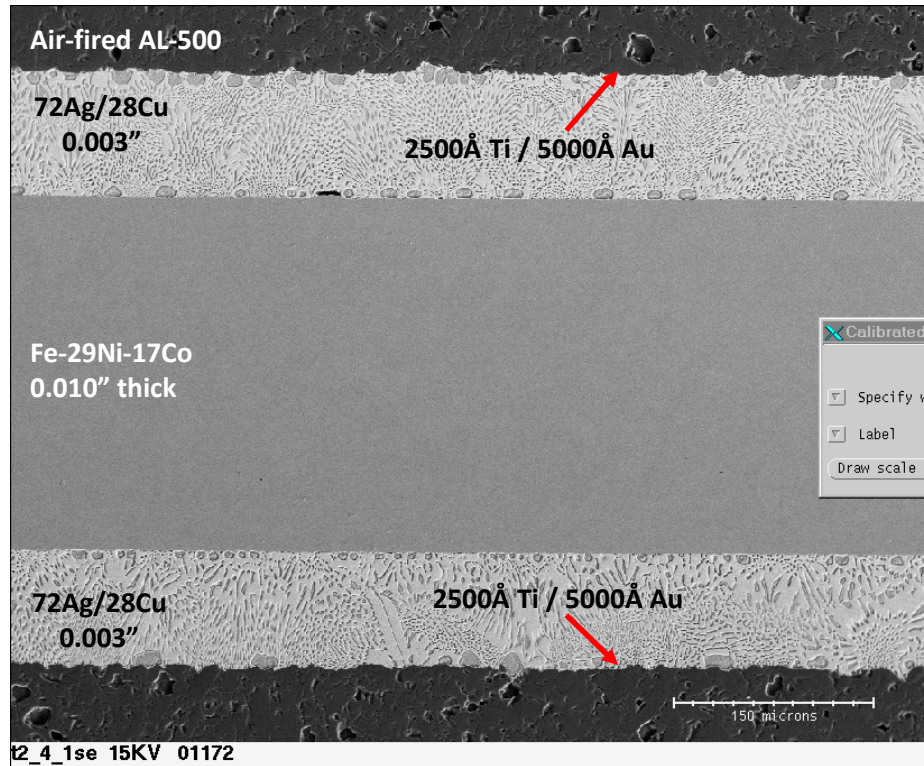
Note that the Zr has successfully migrated to the oxide ceramic surface to allow the filler metal to bond properly.

Comparison between conventional and active brazing

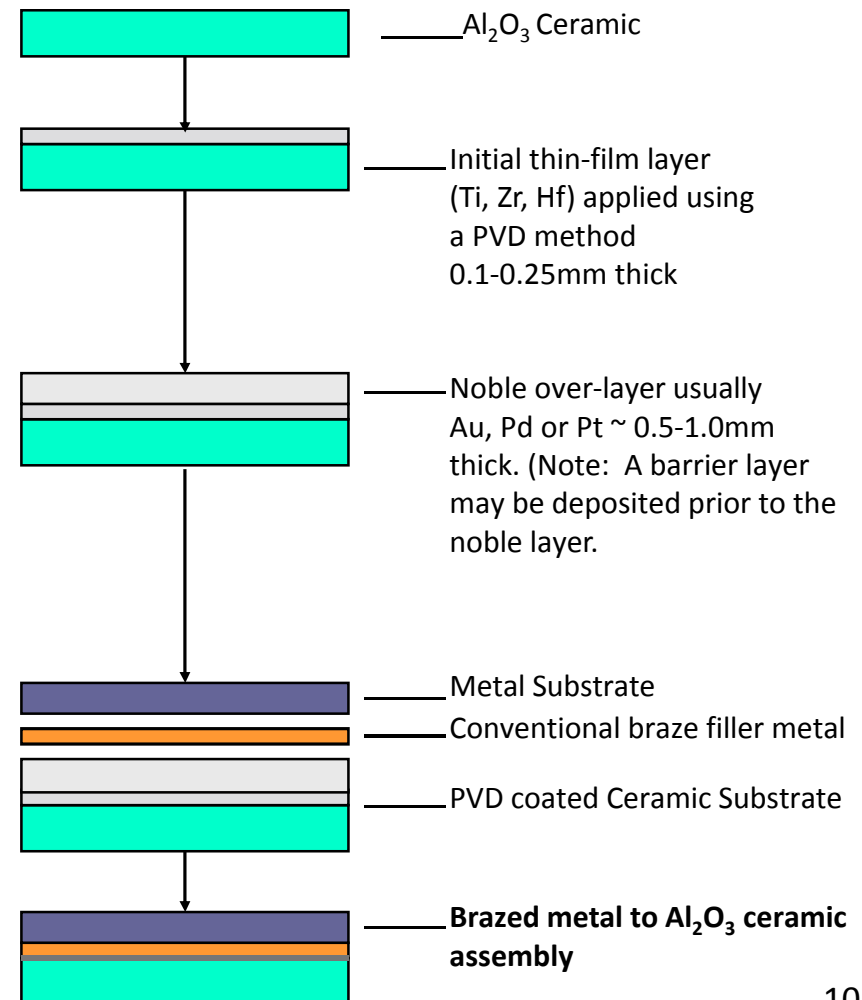


Adapted from AWS Brazing
Handbook, 5th Edition, Page 463

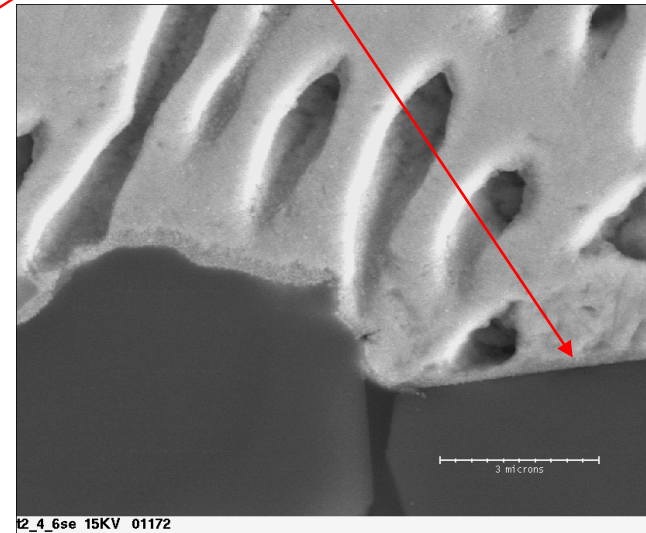
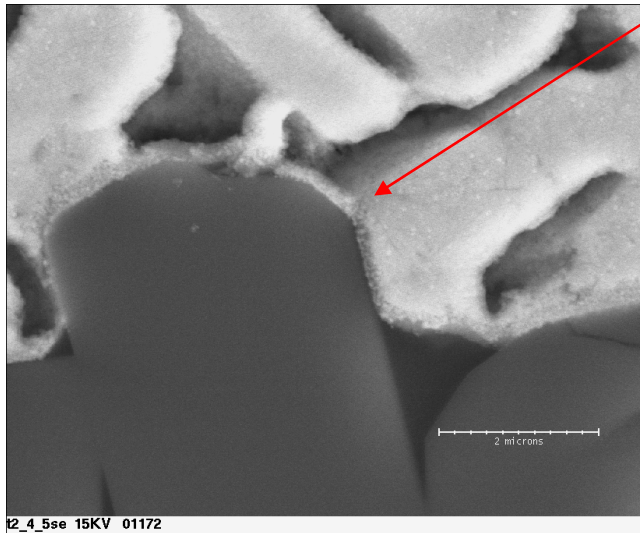
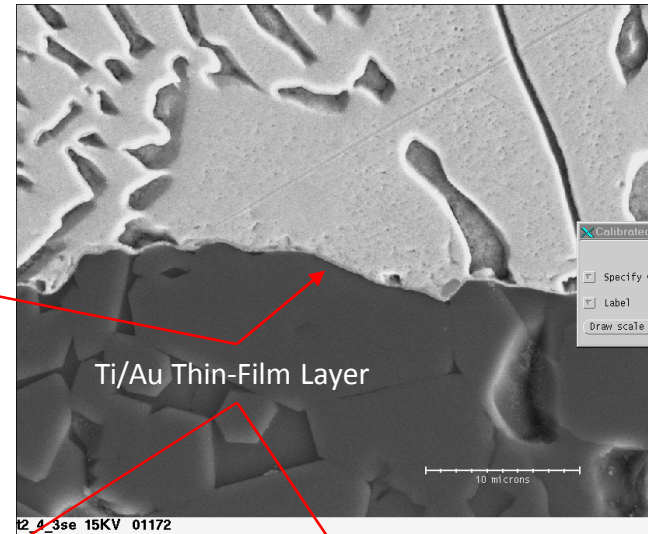
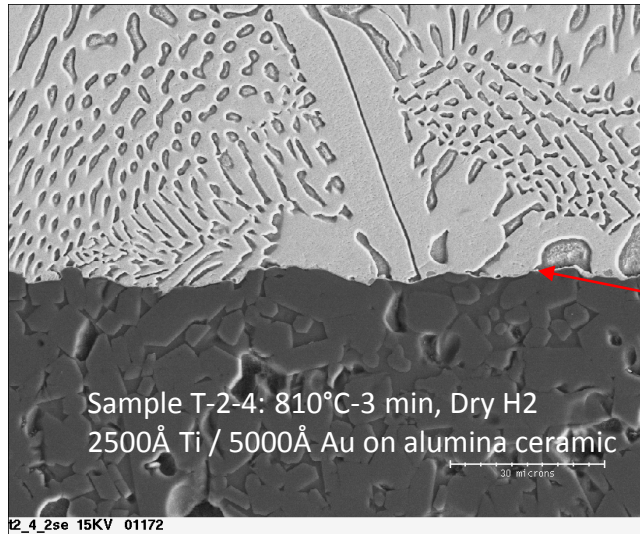
Comparison between conventional and active brazing



Thin-Film Metallization



Comparison between conventional and active brazing



Outline

Background: Brazing, metal-nonmetal brazing

“Standard” active brazing filler metals

Thin-film active element additions & calculations

Examples

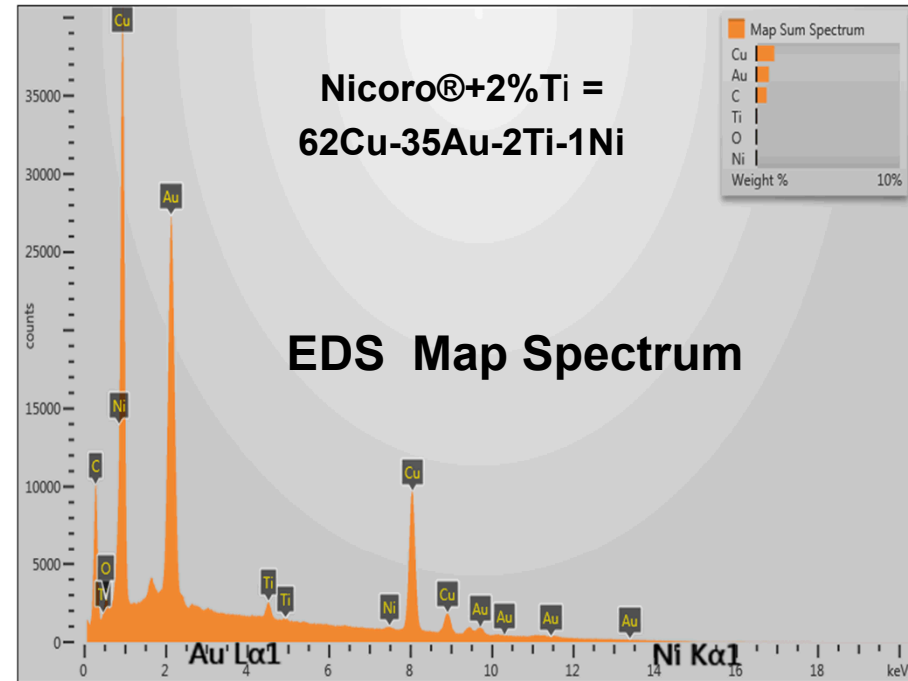
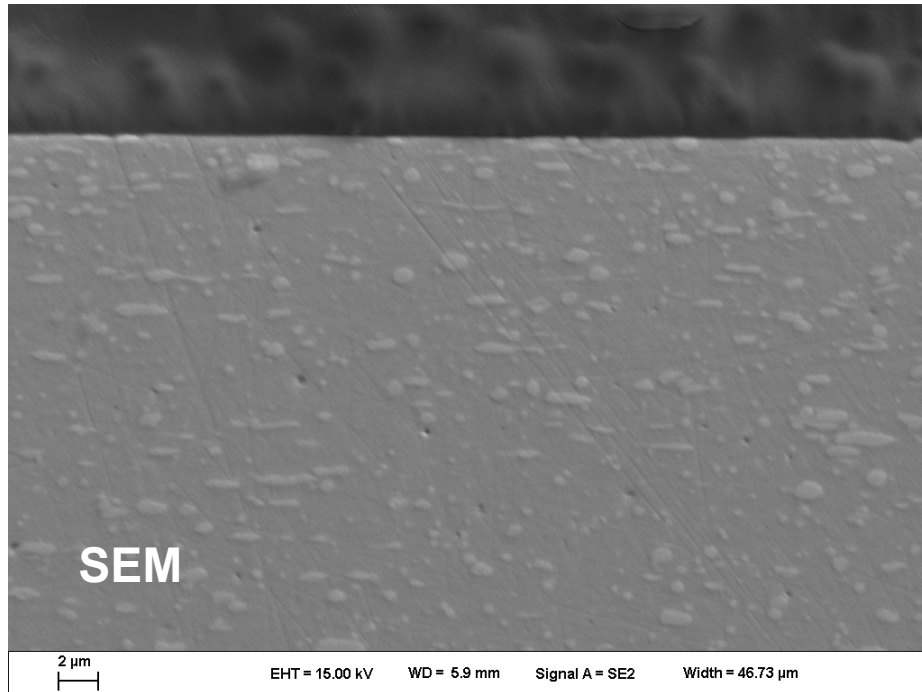
Test samples

Results

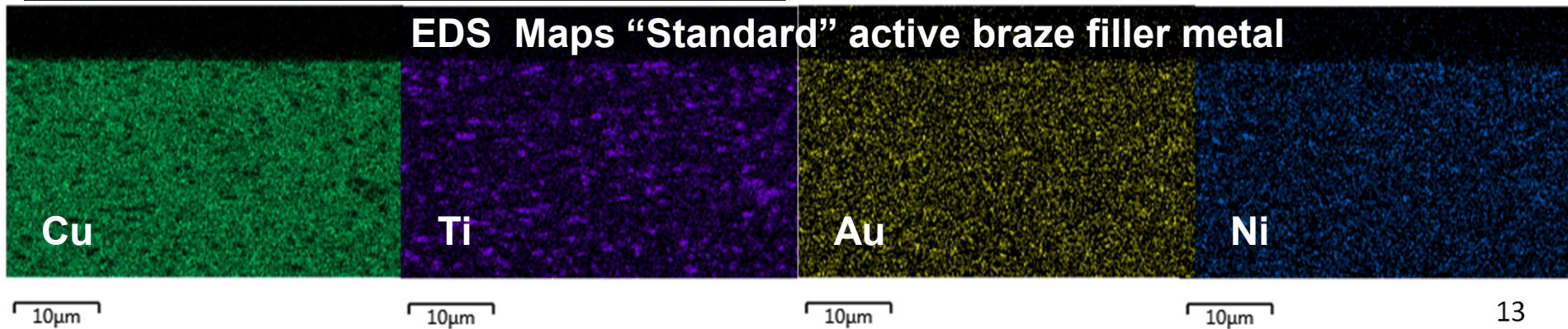
Analysis

Discussion / Conclusions

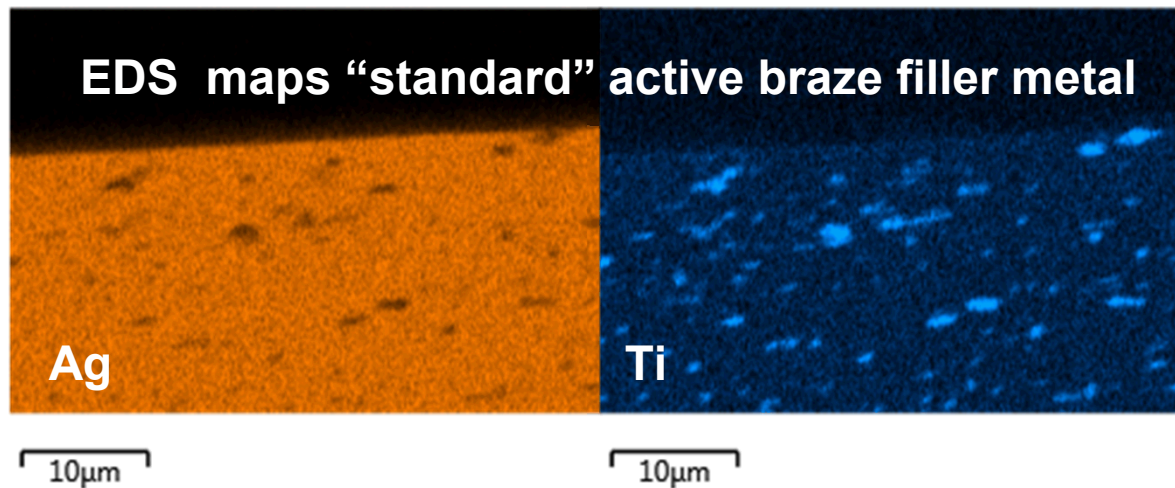
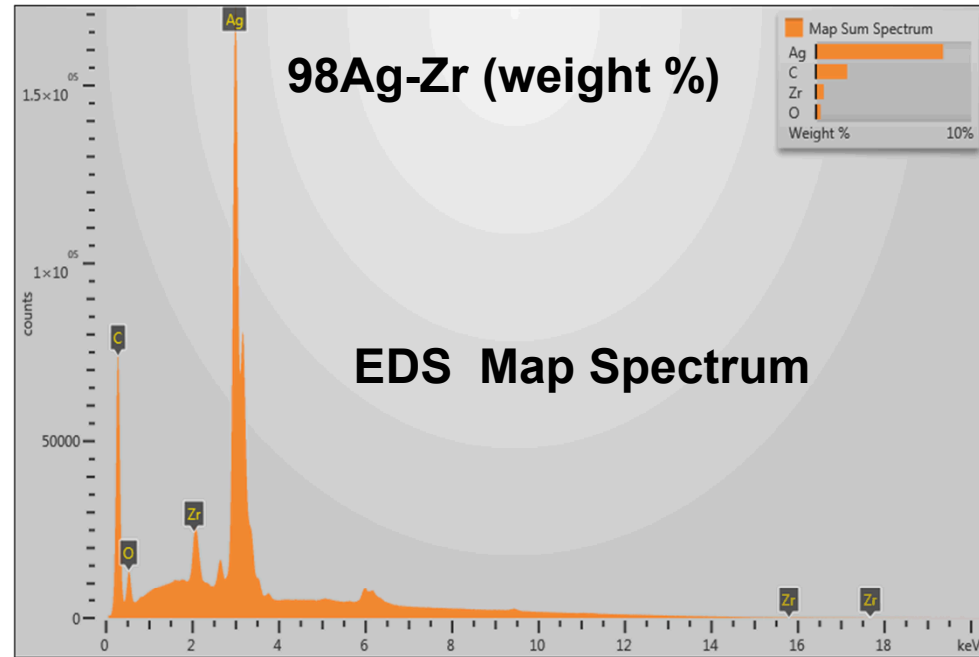
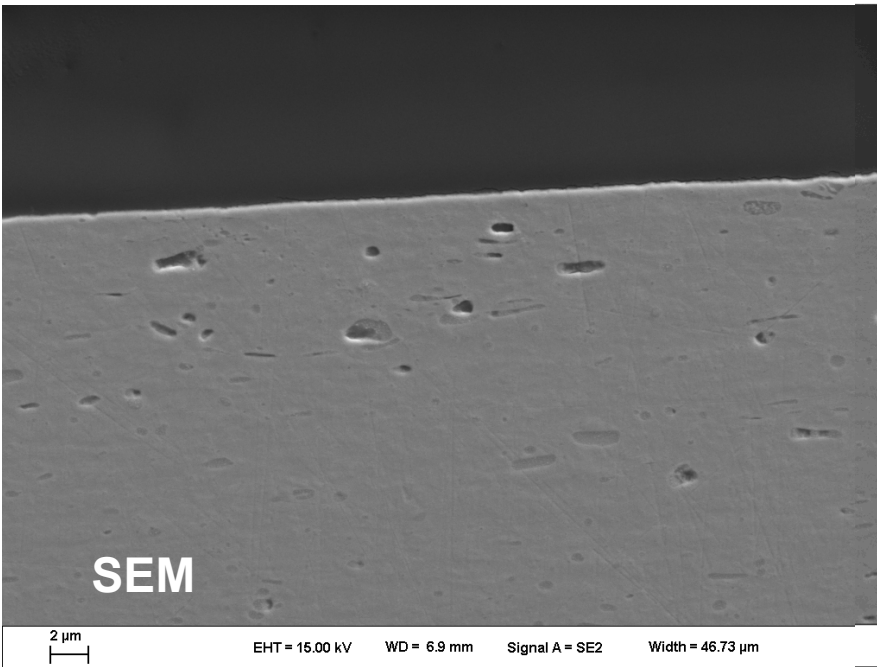
Nicoro[®]+2%Ti active braze filler metal



EDS Maps "Standard" active braze filler metal



98Ag-2Zr active braze filler metal



Outline

Background: Brazing, metal-nonmetal brazing

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Braze Filler Metal Samples

Test samples

Results

Analysis

Discussion / Conclusions

Active element thickness & equivalent weight calculations

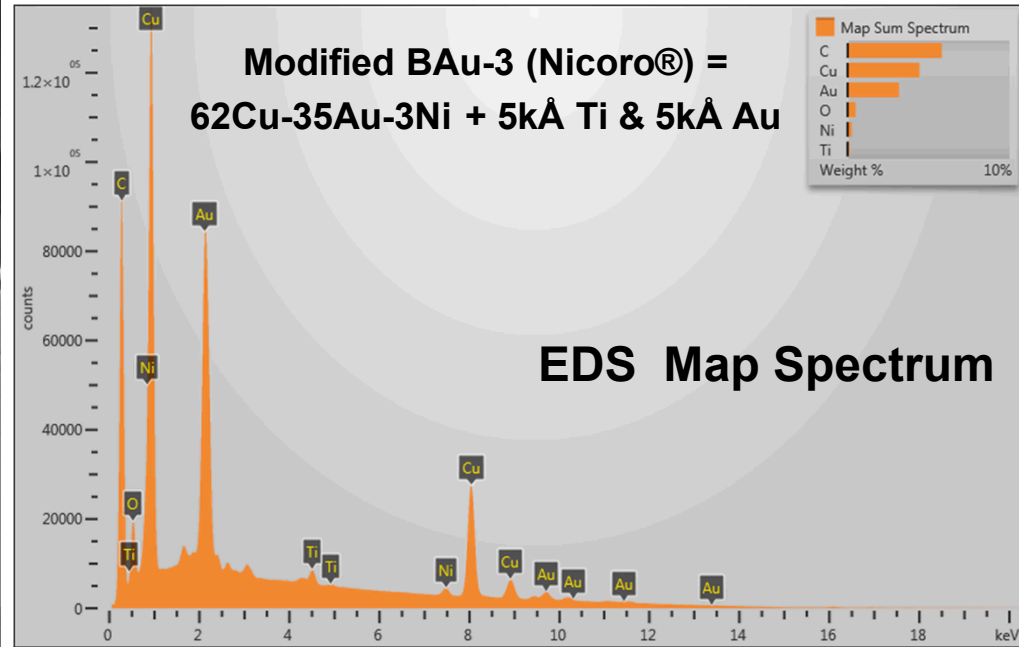
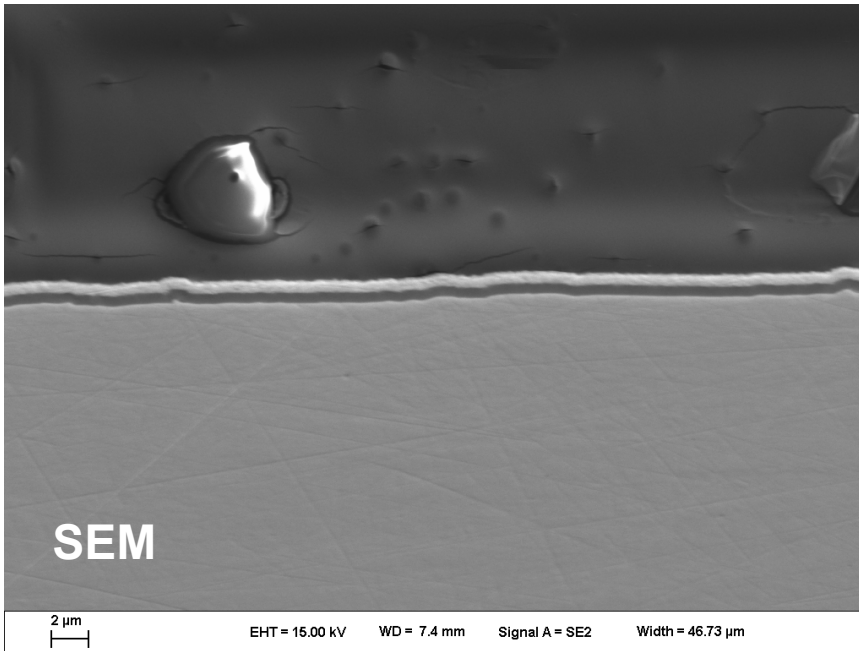
- The two tested commercial active filler metals contain 2 wt. % active element (Ti or Zr).
- Weight percent calculation formulas assumed sputtered layers are fully dense.
- Previous studies and results have shown that a 2500-5000Å active element layer with a similar thickness noble element protective layer.
- 5000Å (0.5μm) layers were chosen for the initial trials.

Active element sputtered thickness & equivalent weight %

Brazing Filler Metal	Thickness inches (μm)	Weight percentage of active element	Sputtered layer equivalent, (μm)
A - Silver+0.626Zr	0.002 (50.8)	0.63	0.50
B - Silver+0.4Zr	0.003 (76.2)	0.4	0.50
C - BAu-3+0.35Ti	0.002 (50.8)	0.4	0.50
D - BAu-3+0.235Ti	0.003 (76.2)	0.24	0.50
E - 97Ag-1Cu-2Zr	0.002 (50.8)	2.0	1.61
F - 97Ag-1Cu-2Zr	0.003 (76.2)	2.0	2.41
G - Nicoro [®] +2%Ti	0.002 (50.8)	2.0	2.90
H - Nicoro [®] +2%Ti	0.003 (76.2)	2.0	4.35

Assumption: Sputtered layers are ~ fully dense.

Nicoro®+5kÅ Ti & 5kÅ Au filler metal



Cu K α 1

Ti K α 1

Au L α 1

Ni K α 1

EDS maps sputtered layer active braze filler metal

Cu

Ti

Au

Ni

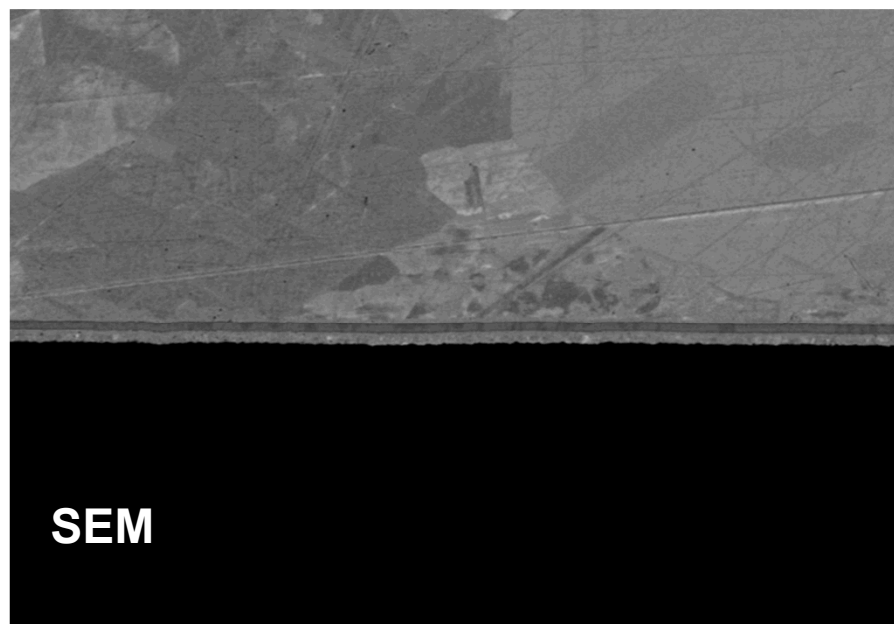
10 μm

10 μm

10 μm

10 μm

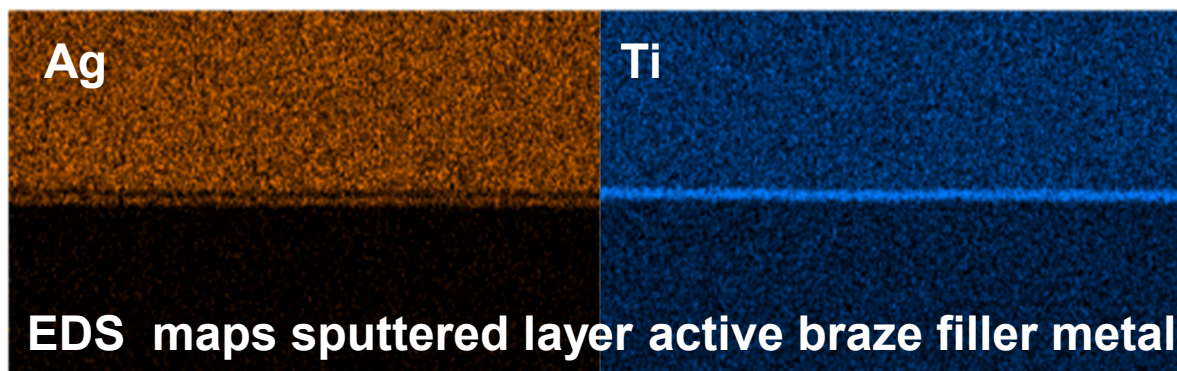
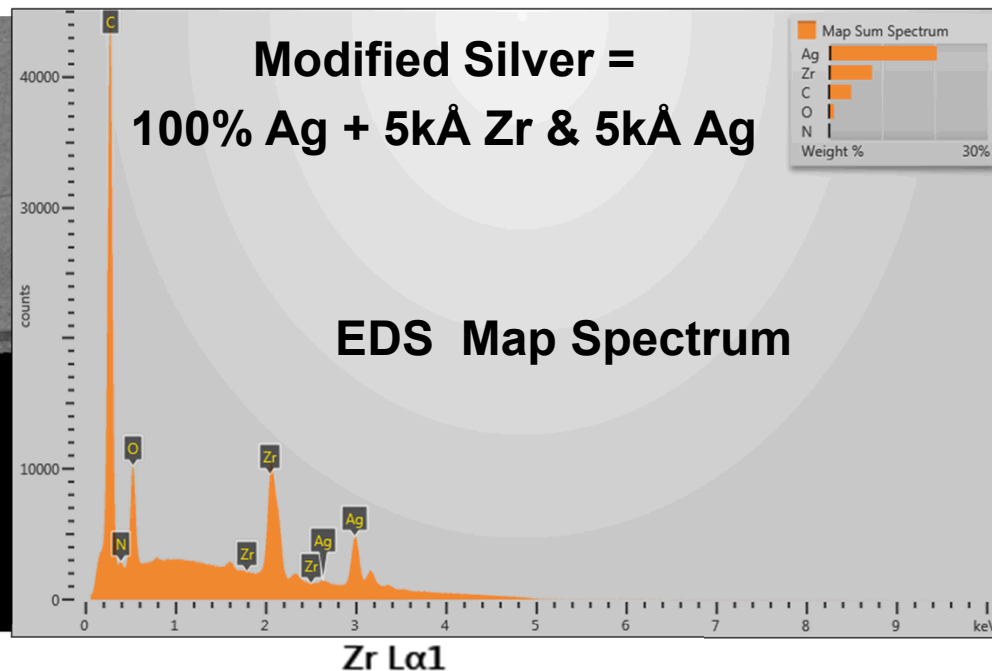
100%Ag + 5kÅ Zr & 5kÅ Ag filler metal



SEM

10µm

Ag Kα1



10µm

10µm

Topics to be addressed

Background: Brazing, metal-nonmetal brazing

“Standard” active brazing filler metals

Thin-film active element additions & calculations

Test samples: ASTM-F19 Tensile Buttons

***Results
Analysis***



Discussion / Conclusions

ASTM-F19 Ceramic-Ceramic tensile button results

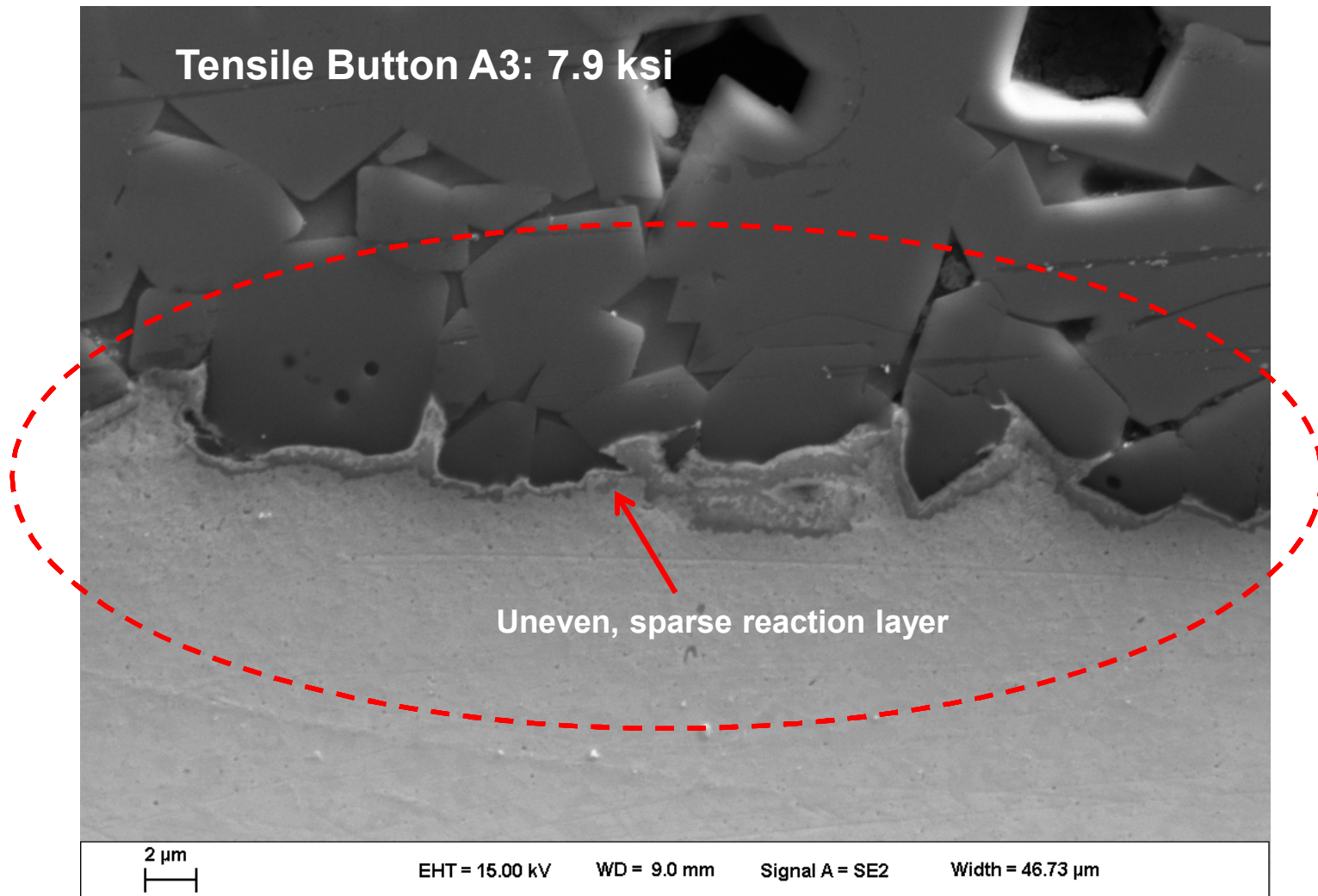
Filler Metal ID	Interlayer	BFM thickness (mils)	Pass/Total	Tensile Load (lbf)	Average Tensile Stress (psi)	Notes:
A	None	2	0/4	595	3323	99.375Ag-0.625Zr (5kÅ Zr followed by 5kÅ Ag on Ag foil)
B	None	3	0/4	158	1077	99.6Ag-0.4Zr (5kÅ Zr followed by 5kÅ Ag on Ag foil)
C	None	2	0/4	782	4368	Nicoro [®] +0.35%Ti (5kÅ Ti followed by 5kÅ Au on Nicoro [®] foil)
D	None	3	0/4	594	3320	Nicoro [®] +0.235%Ti (5kÅ Ti followed by 5kÅ Au on Nicoro [®] foil)
E	None	2	1/4	740	4137	98Ag-2Zr, Standard ABA BFM
F	None	3	2/4	1901	10619	98Ag-2Zr, Standard ABA BFM
G	None	2	0/4	1567	8752	Nicoro [®] +2%Ti, Standard ABA BFM
H	None	3	2/4	2155	12041	Nicoro [®] +2%Ti, Standard ABA BFM

More about this later...



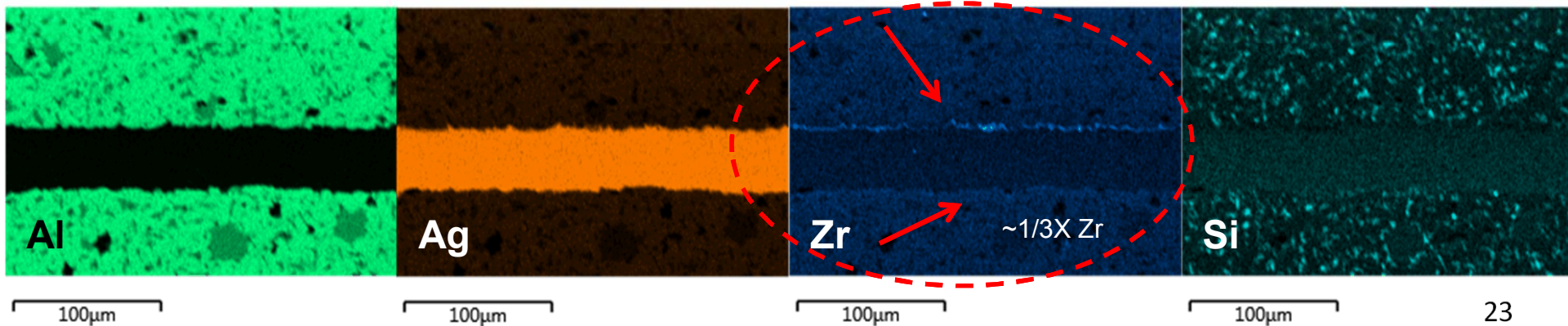
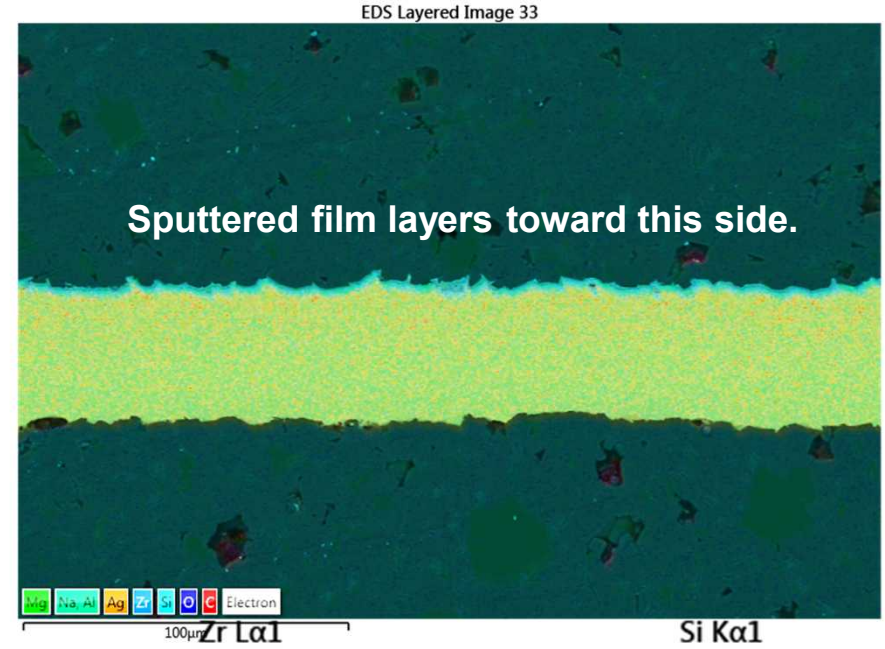
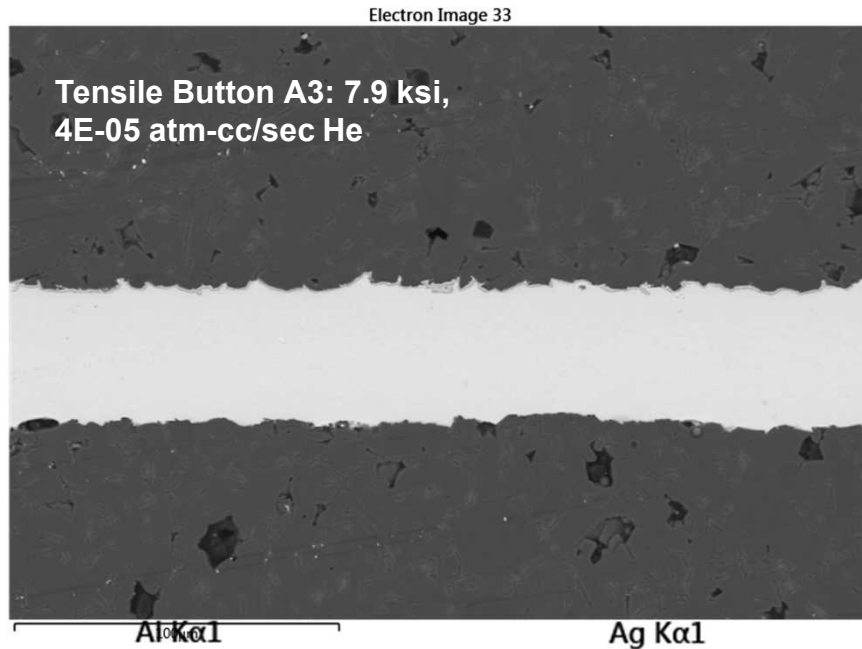
Sample Analysis: ASTM F-19 Tensile Buttons

A3 - 99.375Ag-0.625Zr (5kÅ Zr followed by 5kÅ Ag on 50µm Ag foil)



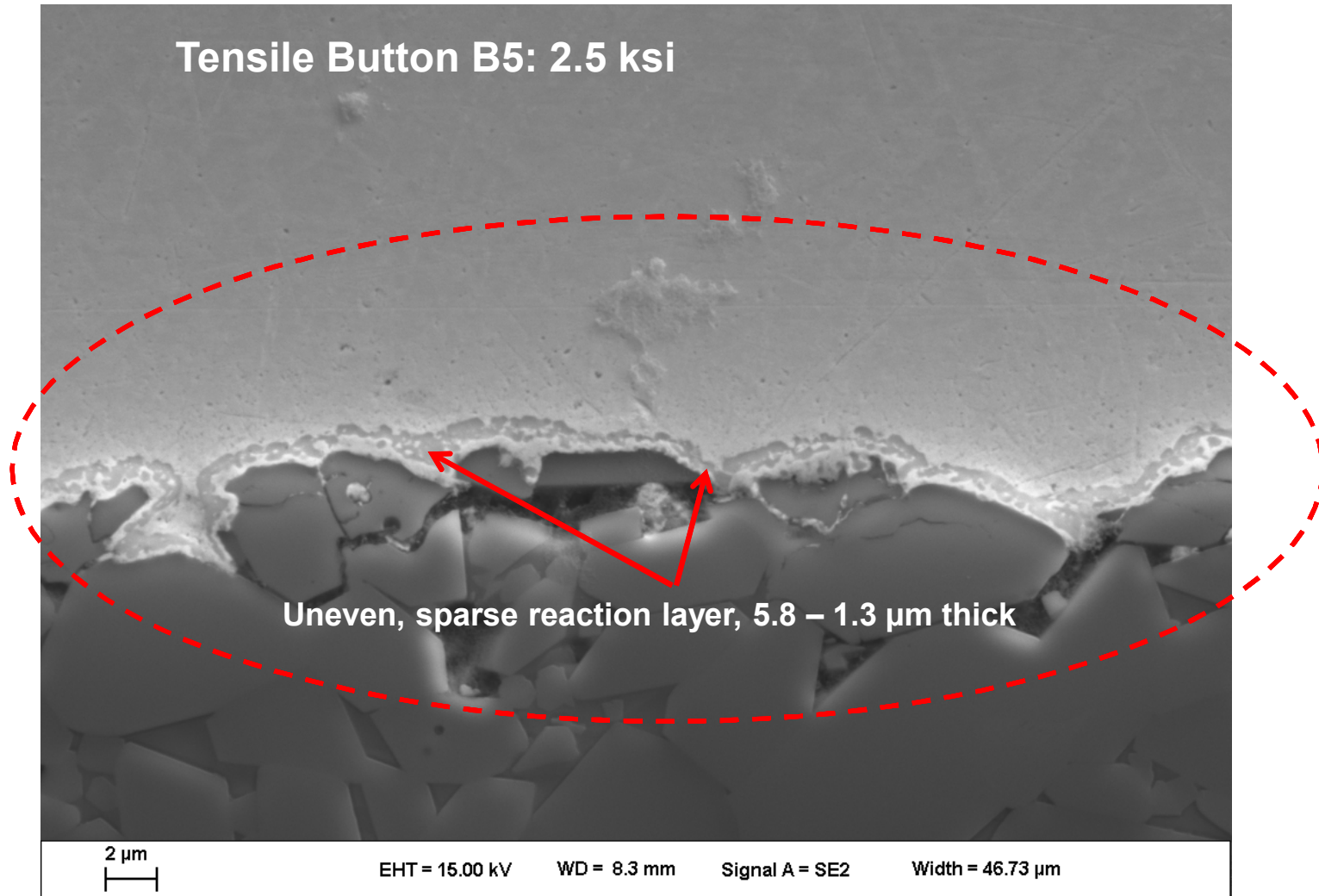
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Sample Analysis: ASTM F-19 Tensile Buttons

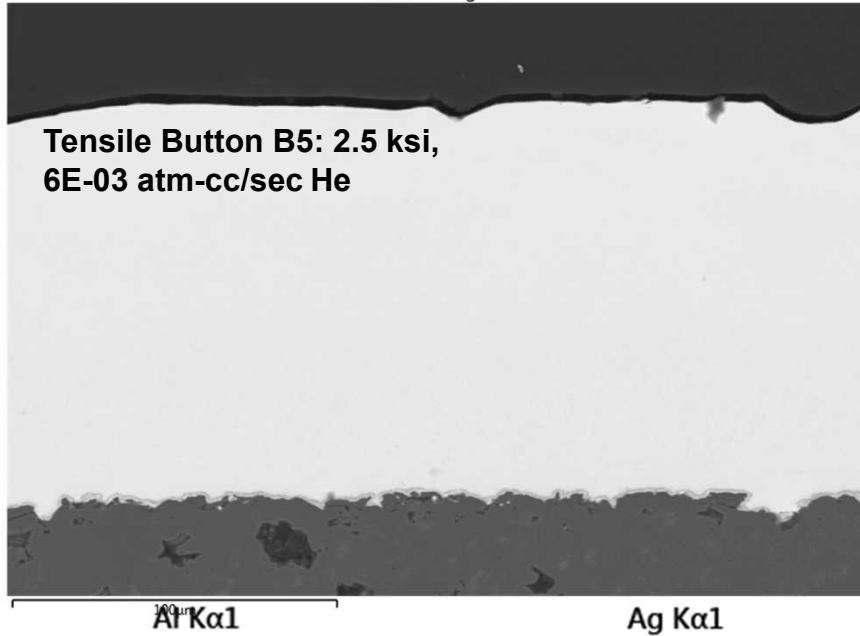
B5 - 99.6Ag-0.4Zr (5kÅ Zr followed by 5kÅ Ag on 75µm Ag foil)



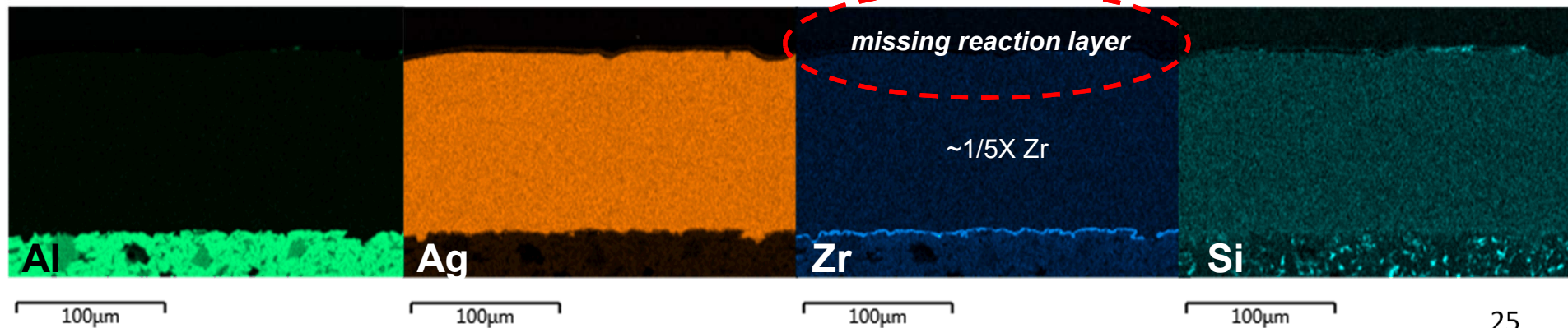
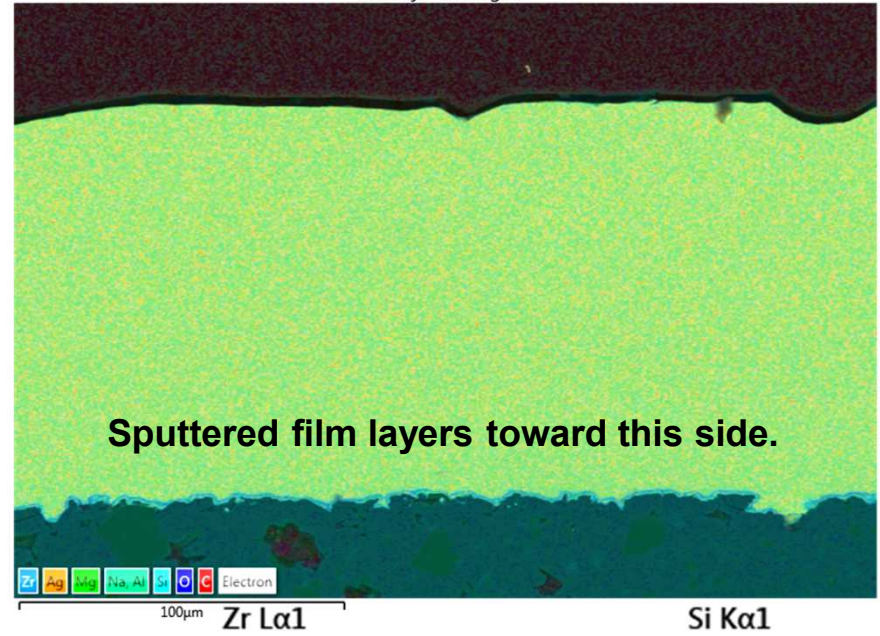
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Electron Image 37

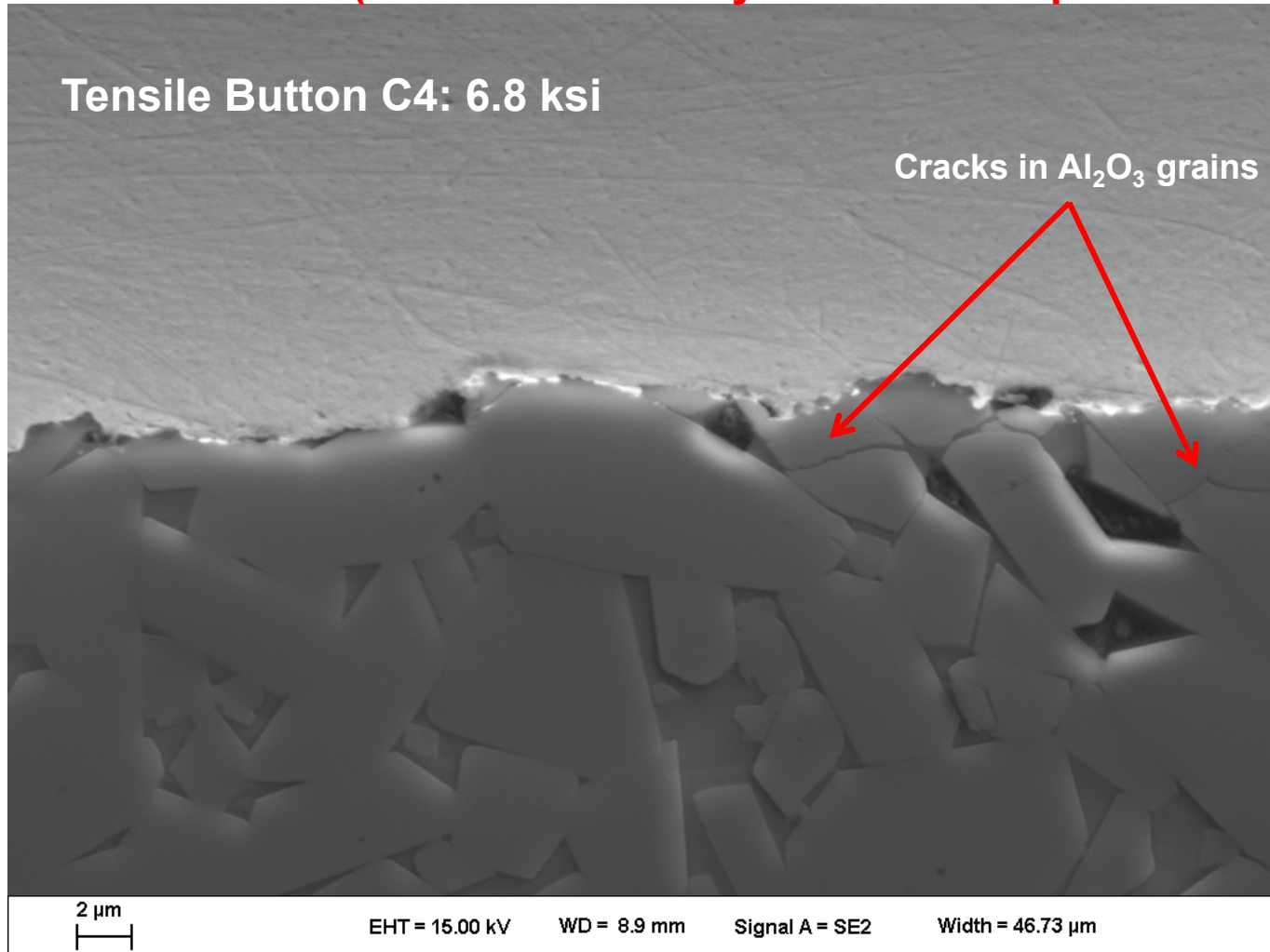


EDS Layered Image 37



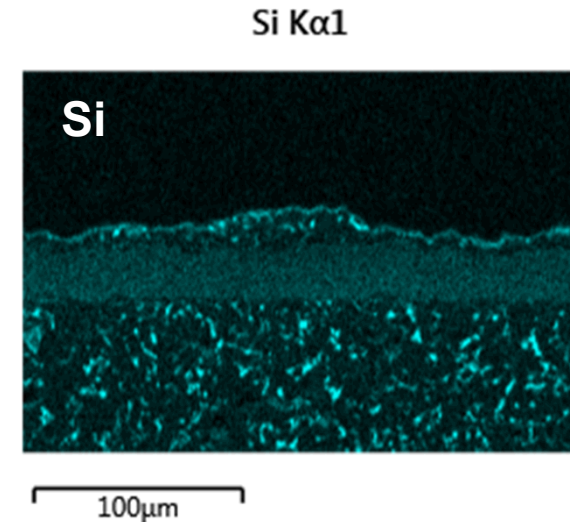
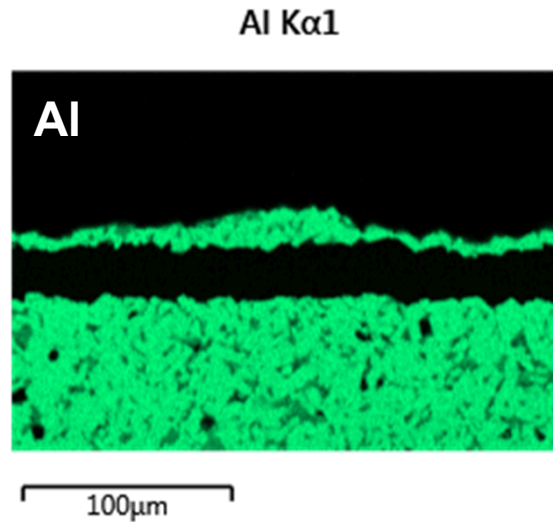
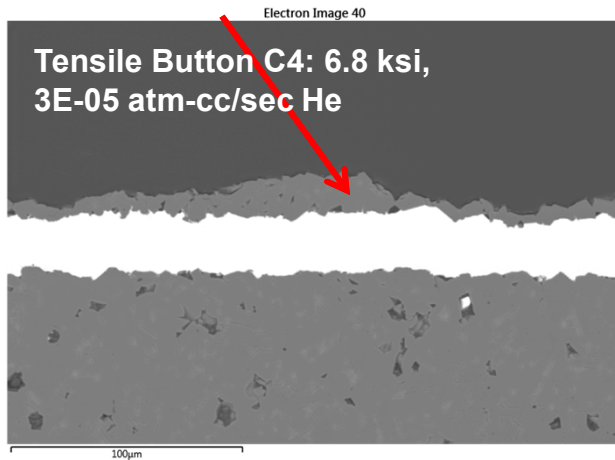
Sample Analysis: ASTM F-19 Tensile Buttons

C4 - Nicoro®+0.35%Ti (5kÅ Ti followed by 5kÅ Au on 50µm Nicoro® foil)

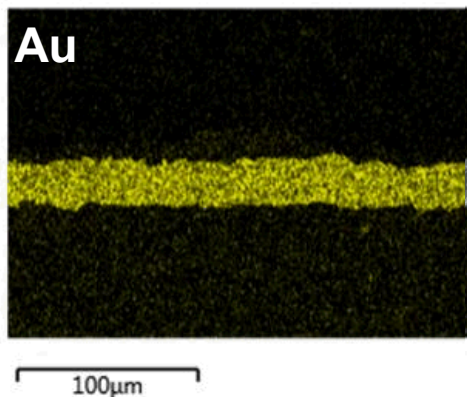


Sample Analysis: ASTM F-19 Tensile Buttons

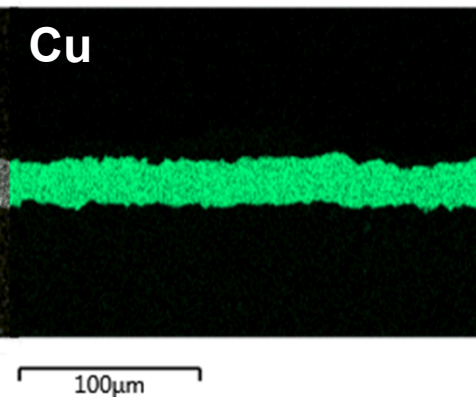
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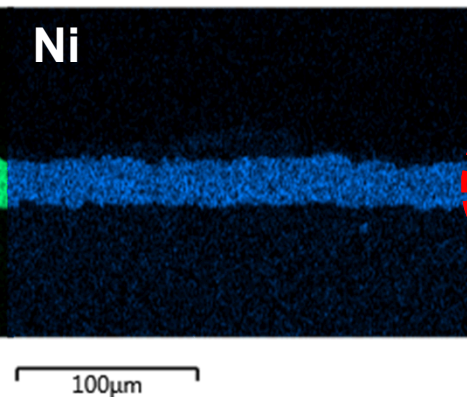
Au Lα1



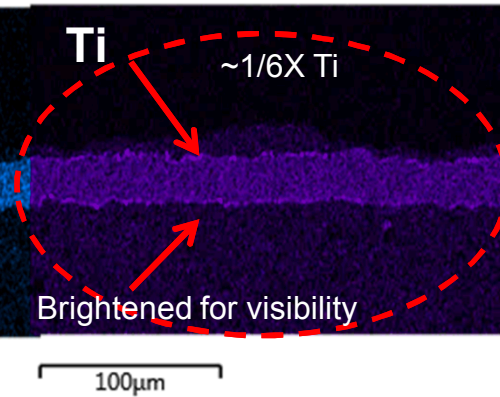
Cu Kα1



Ni Kα1

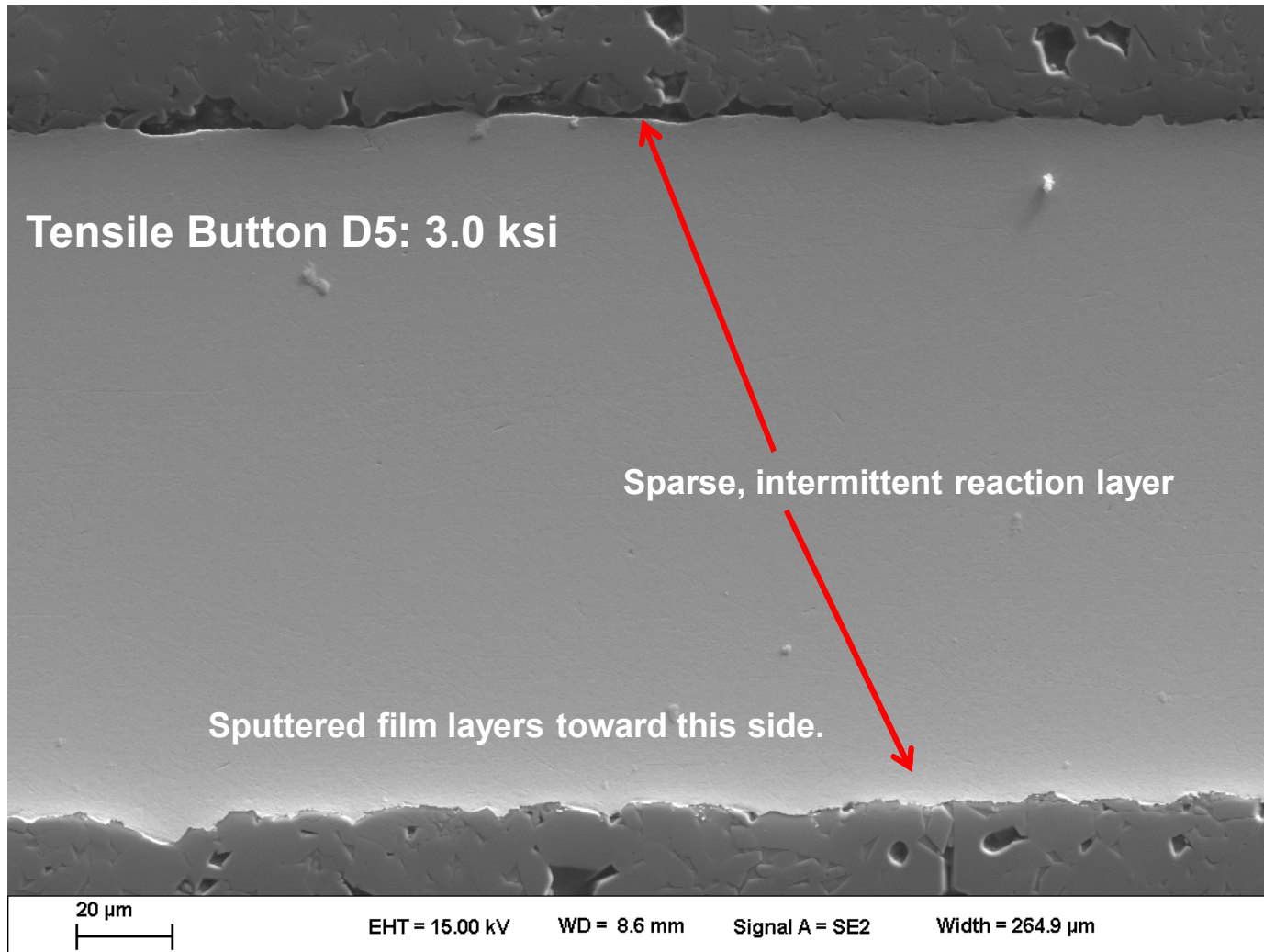


Ti Kα1



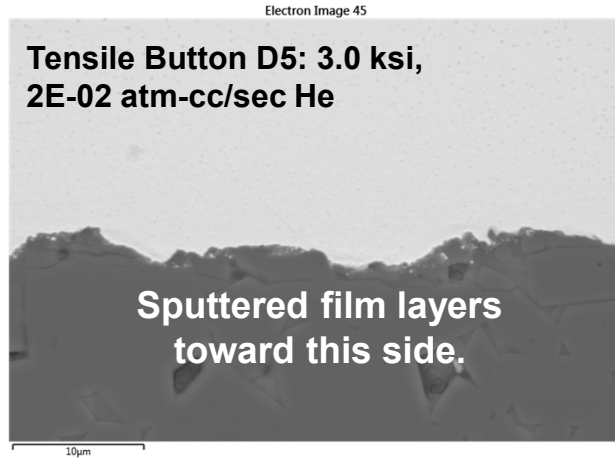
Sample Analysis: ASTM F-19 Tensile Buttons

D5 - Nicoro®+0.235%Ti (5kÅ Ti followed by 5kÅ Au on 75µm Nicoro® foil)

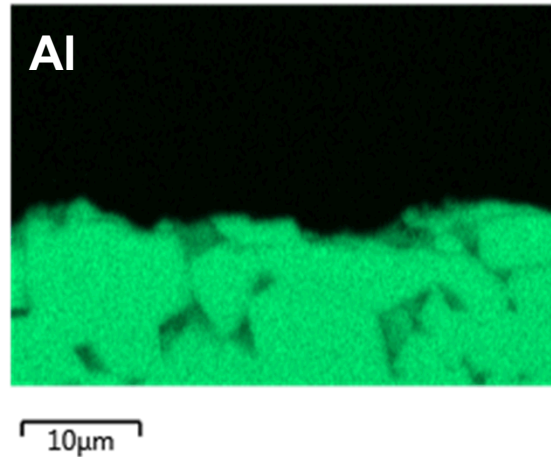


Sample Analysis: ASTM F-19 Tensile Buttons

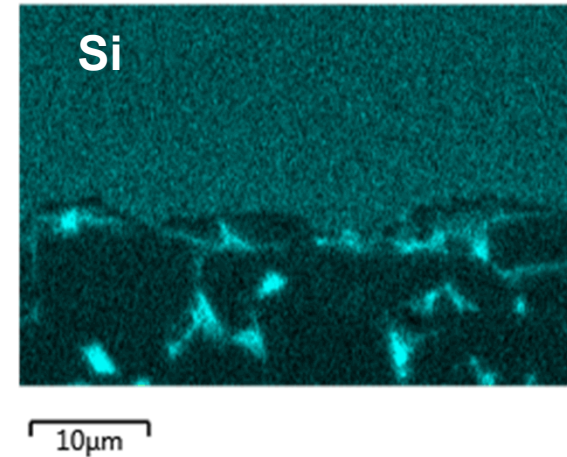
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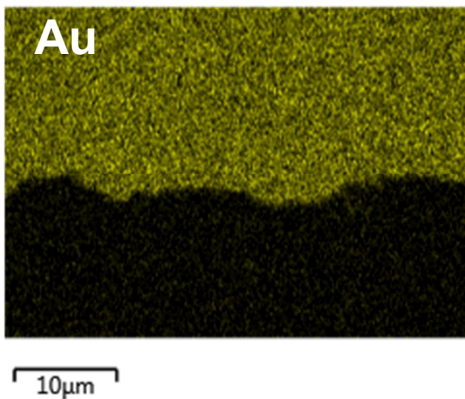
Al Kα1



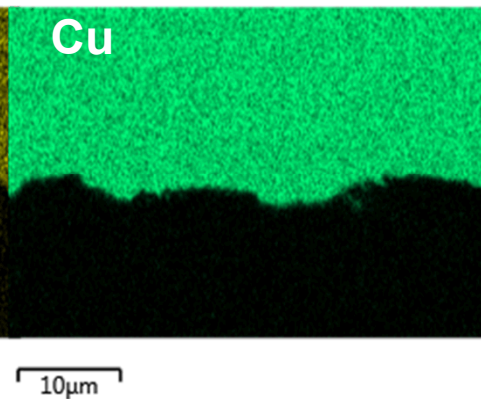
Si Kα1



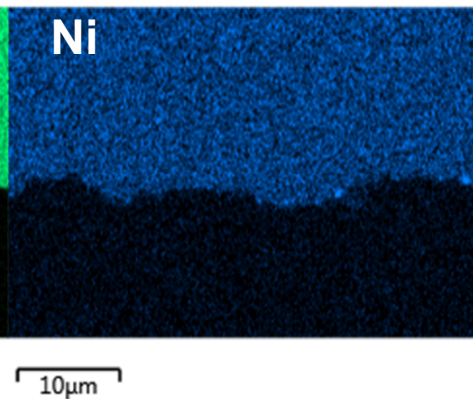
Au Lα1



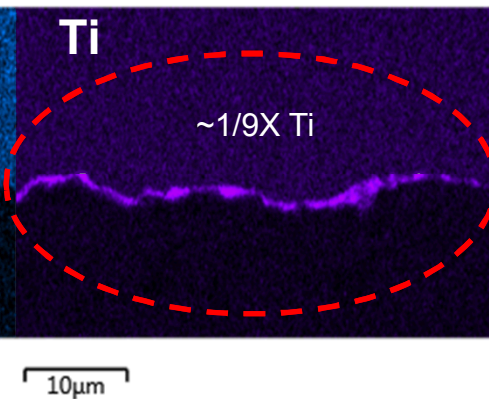
Cu Kα1



Ni Kα1

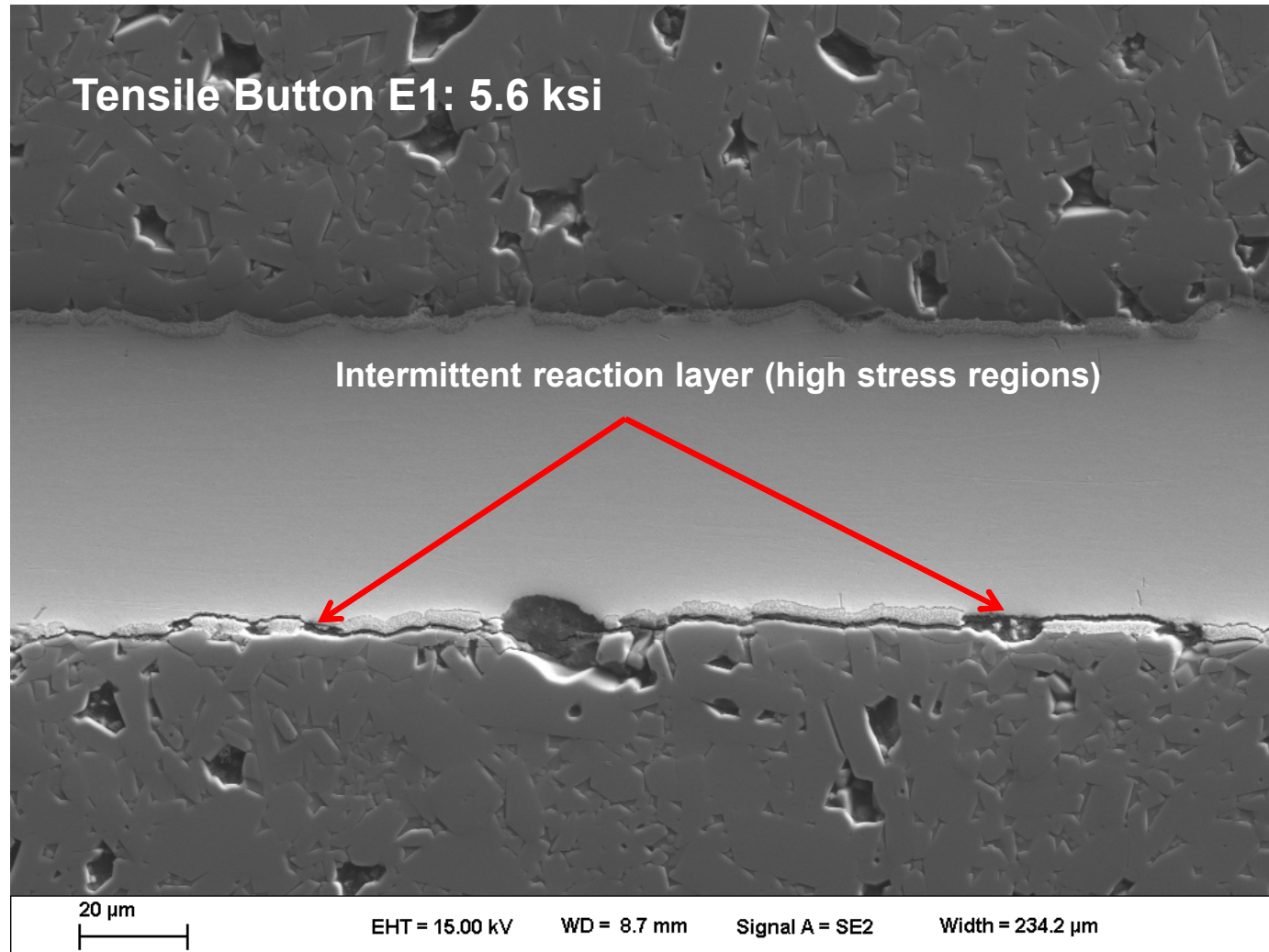


Ti Kα1



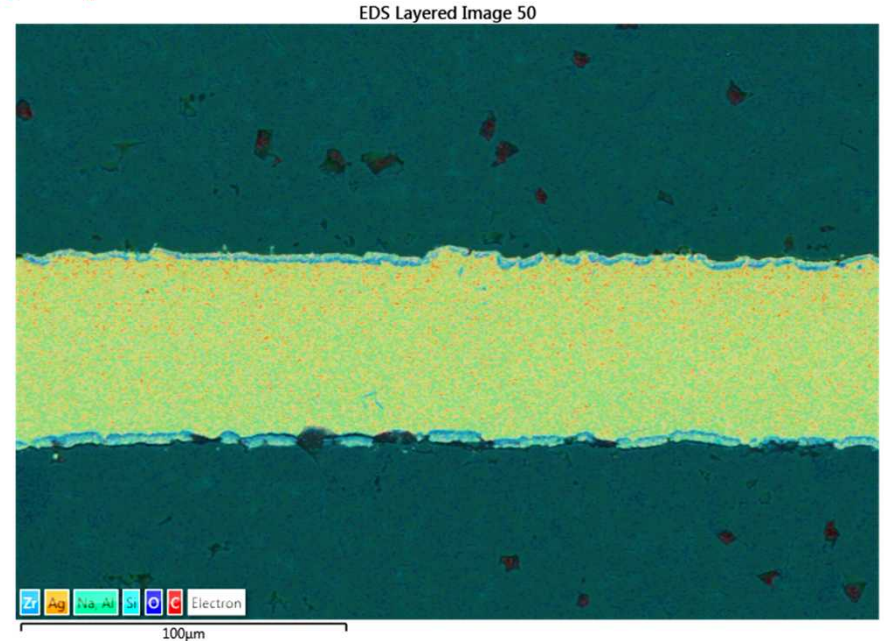
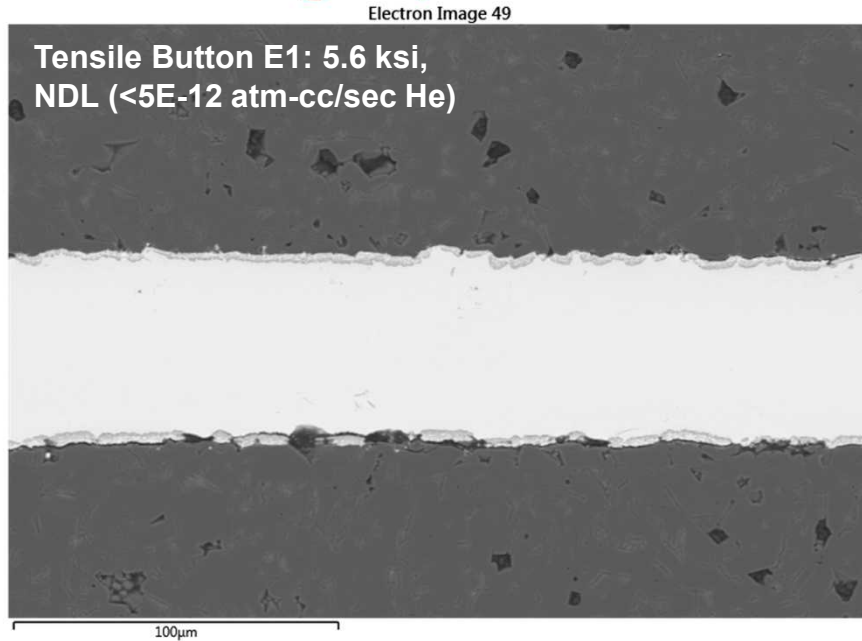
Sample Analysis: ASTM F-19 Tensile Buttons

E1 - 98Ag-2Zr, Standard ABA BFM, 50 μ m thick



Sample Analysis: ASTM F-19 Tensile Buttons

E1 - 98Ag-2Zr, Standard ABA BFM, 50 μ m thick

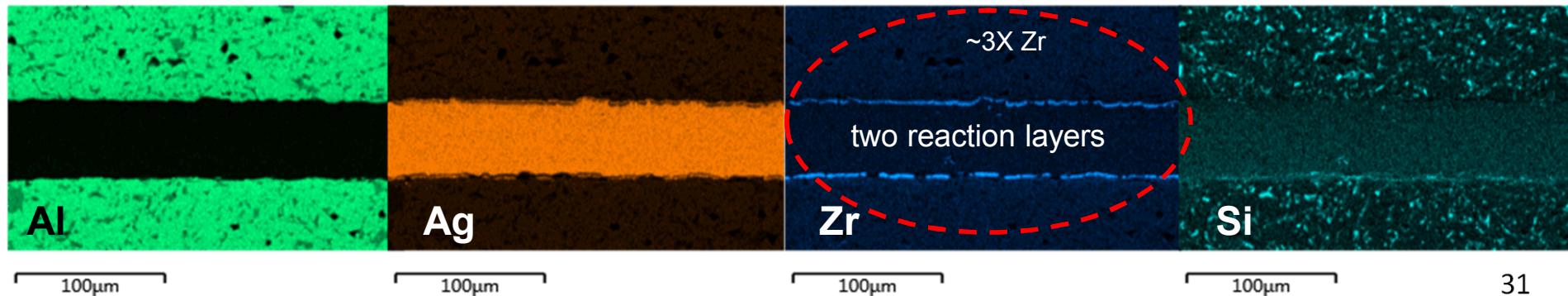


Al K α 1

Ag K α 1

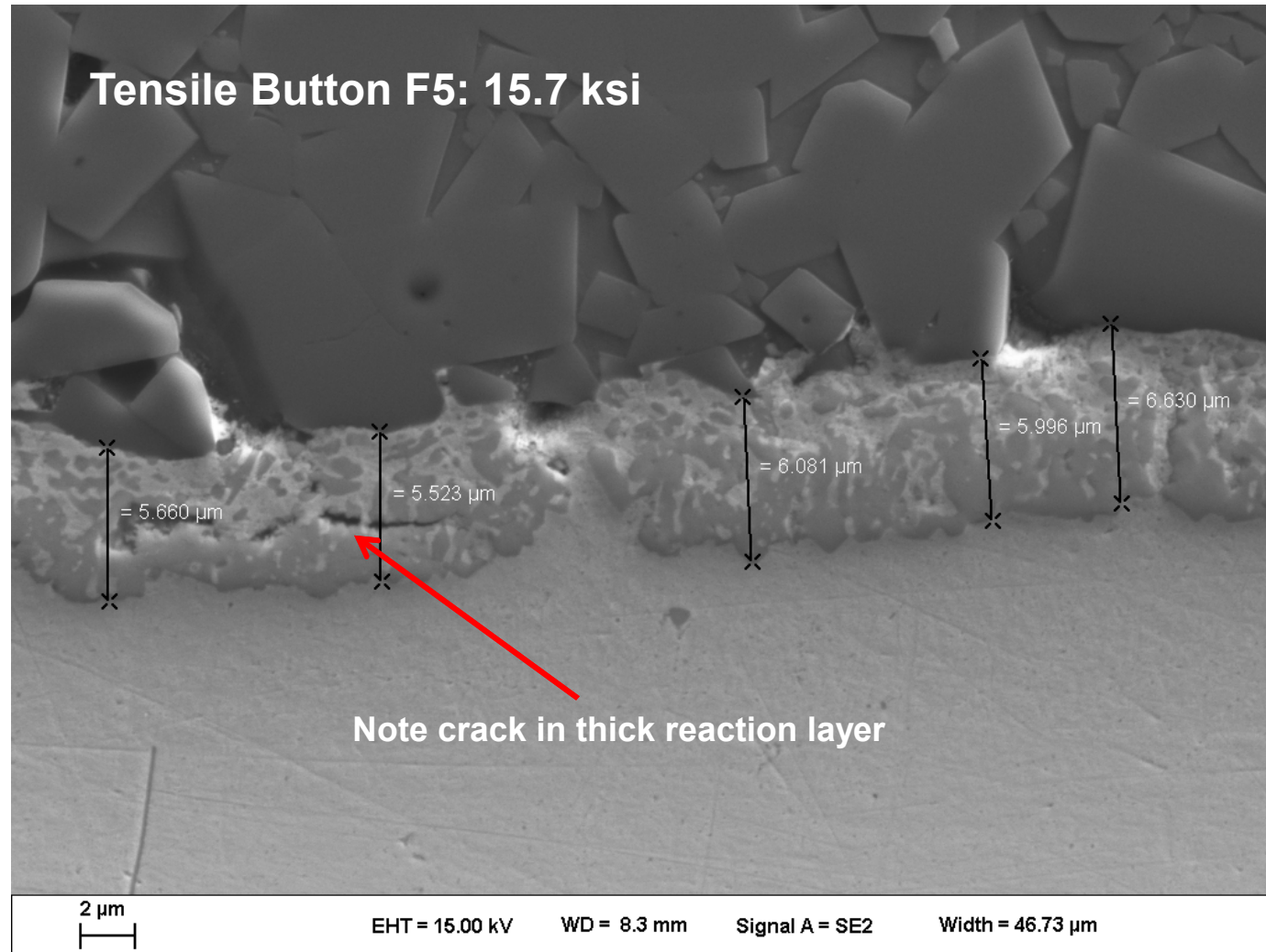
Zr L α 1

Si K α 1



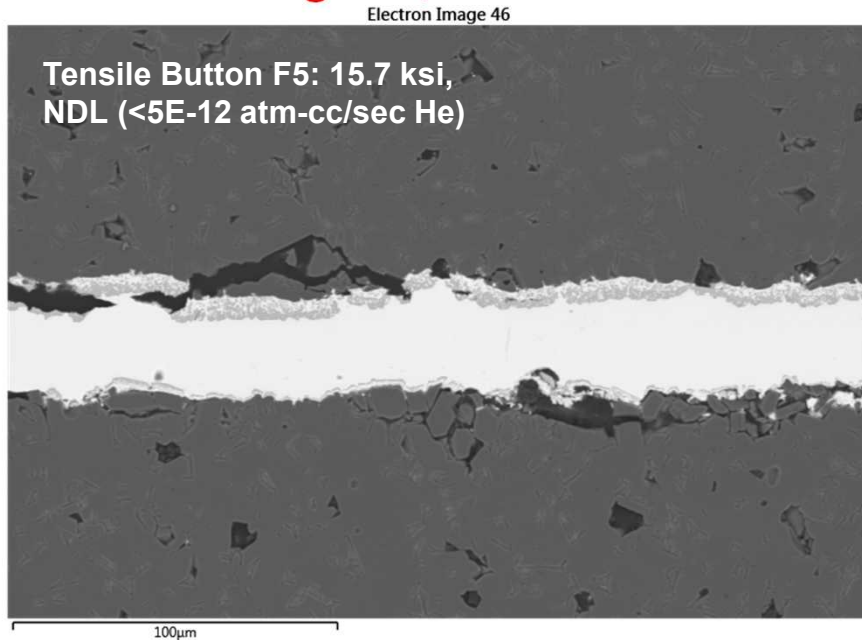
Sample Analysis: ASTM F-19 Tensile Buttons

F5 - 98Ag-2Zr, Standard ABA BFM, 75 μ m thick



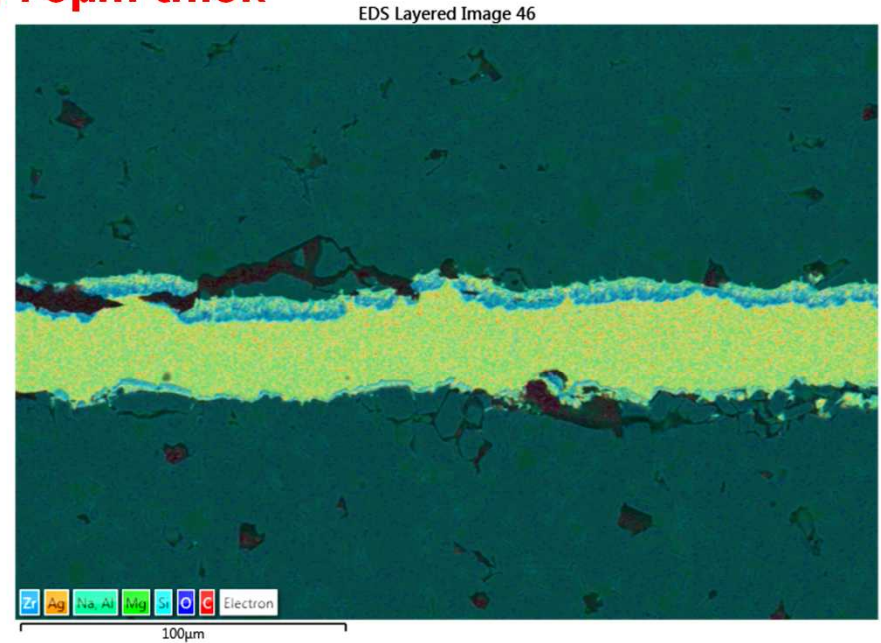
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F5 - 98Ag-2Zr, Standard ABA BFM, 75μm thick



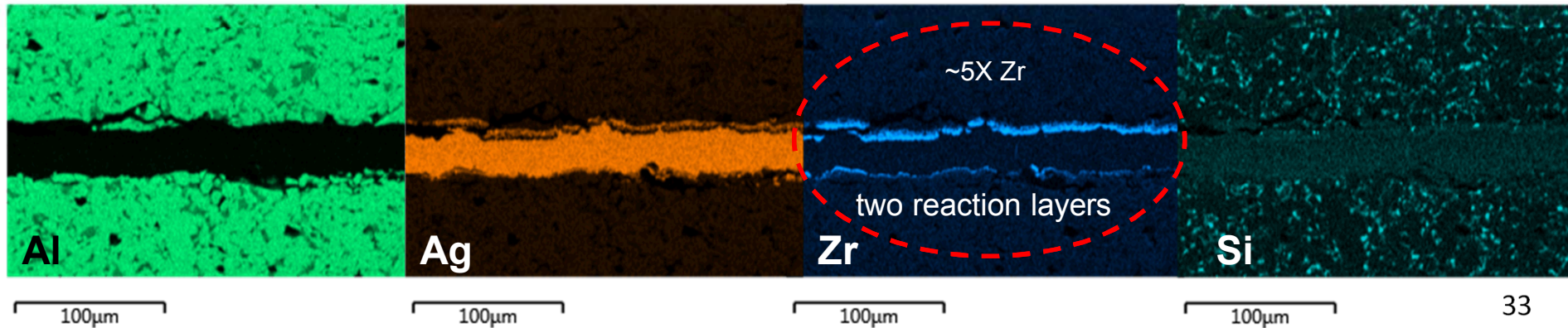
Al Kα1

Ag Kα1



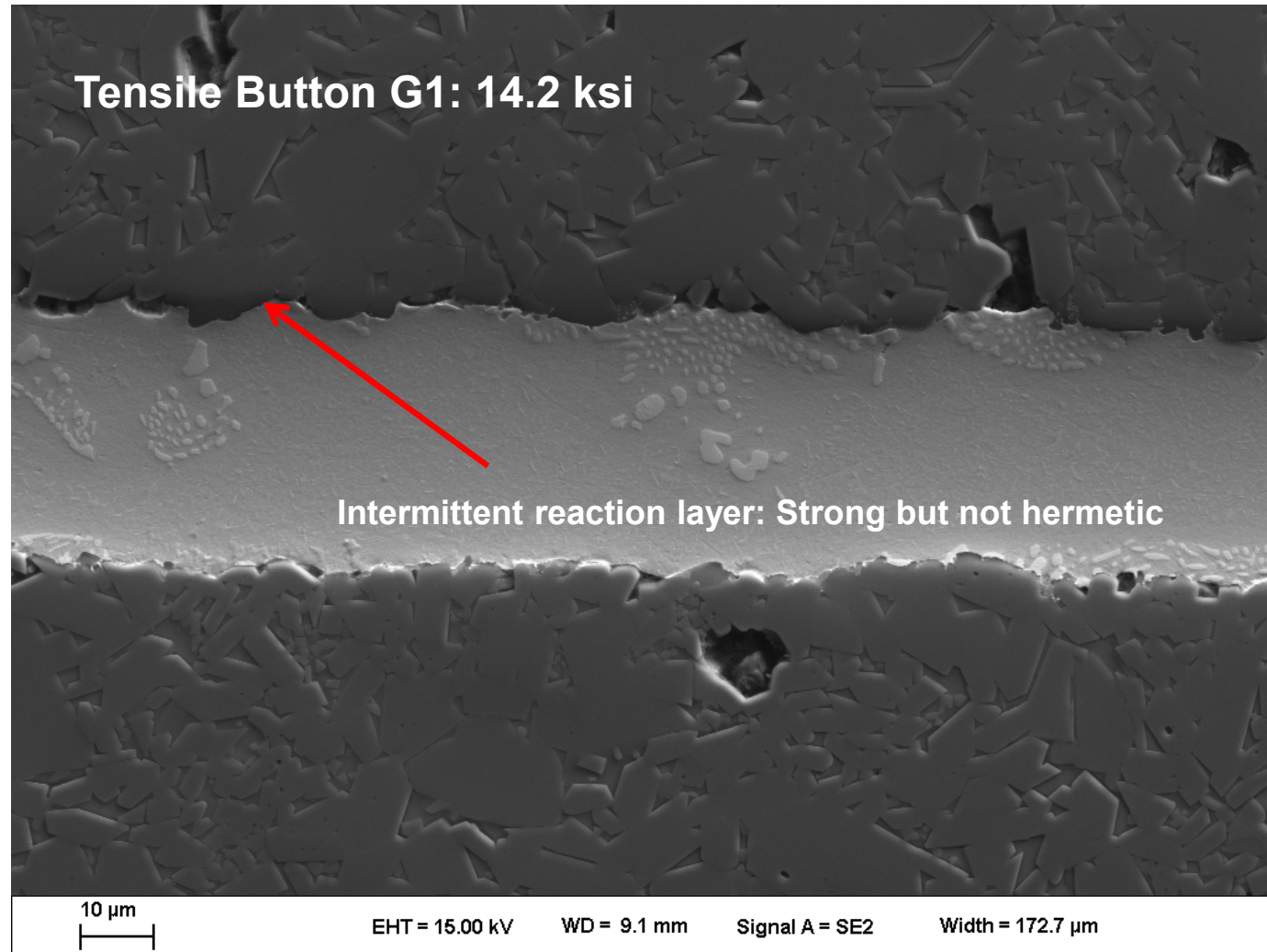
Zr Lα1

Si Kα1



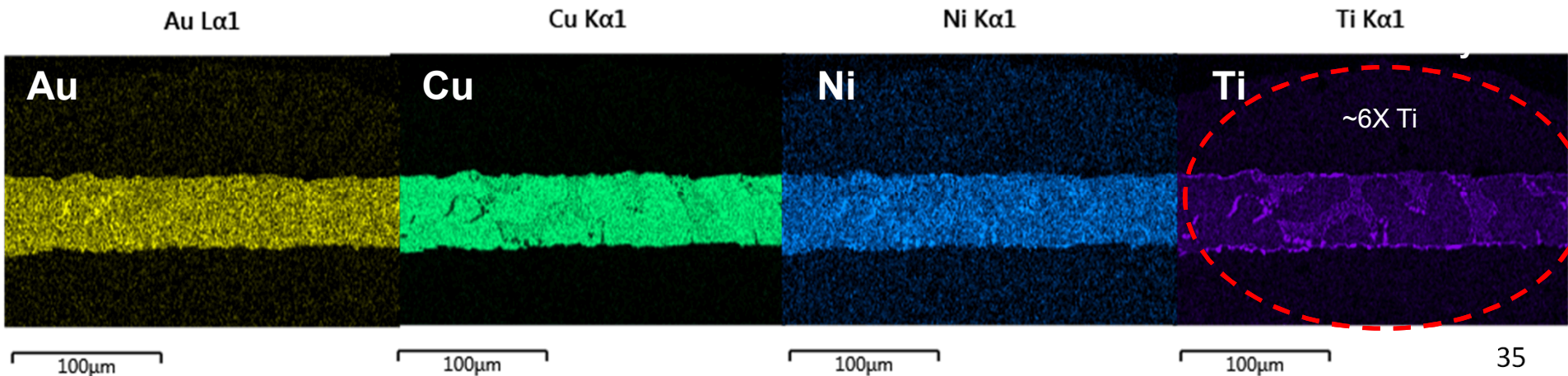
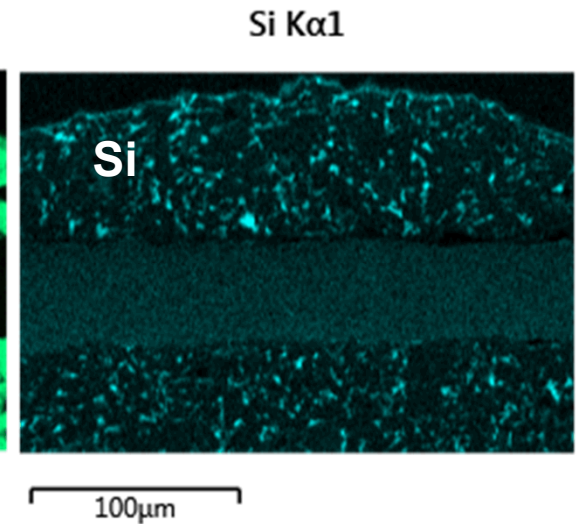
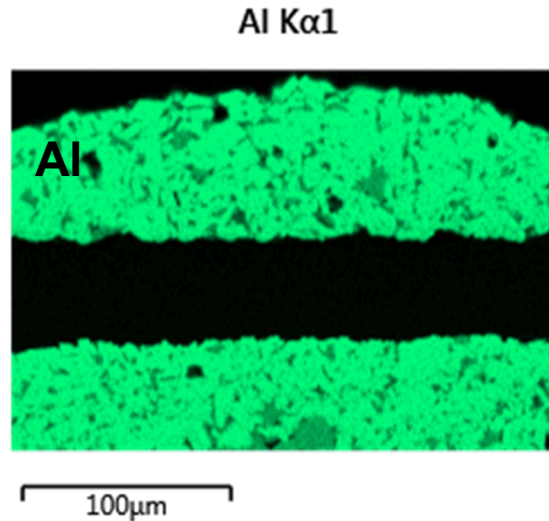
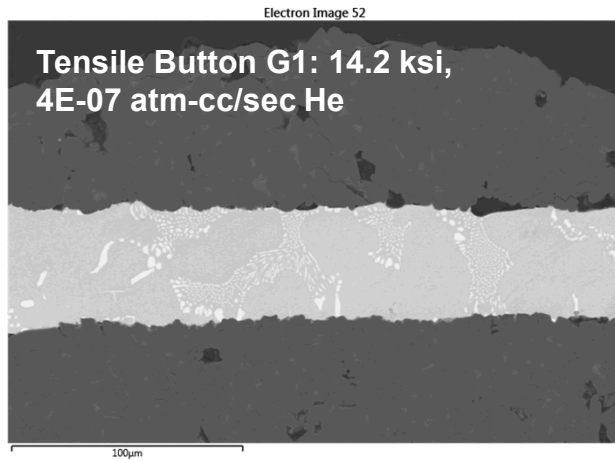
Sample Analysis: ASTM F-19 Tensile Buttons

G1 - Nicoro®+2%Ti, Standard ABA BFM , 50μm thick



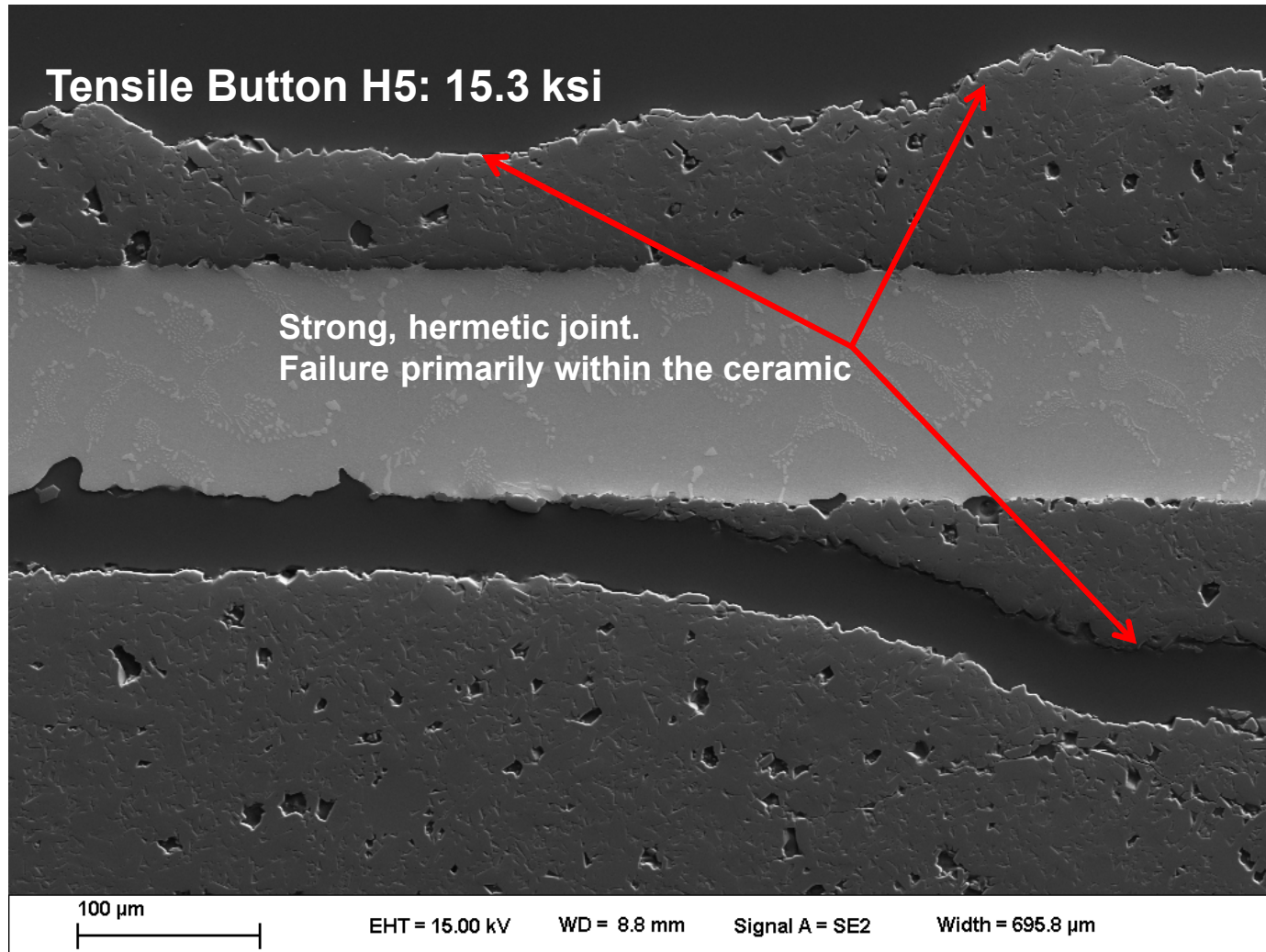
Sample Analysis: ASTM F-19 Tensile Buttons

G1 - Nicoro®+2%Ti, Standard ABA BFM , 50μm thick



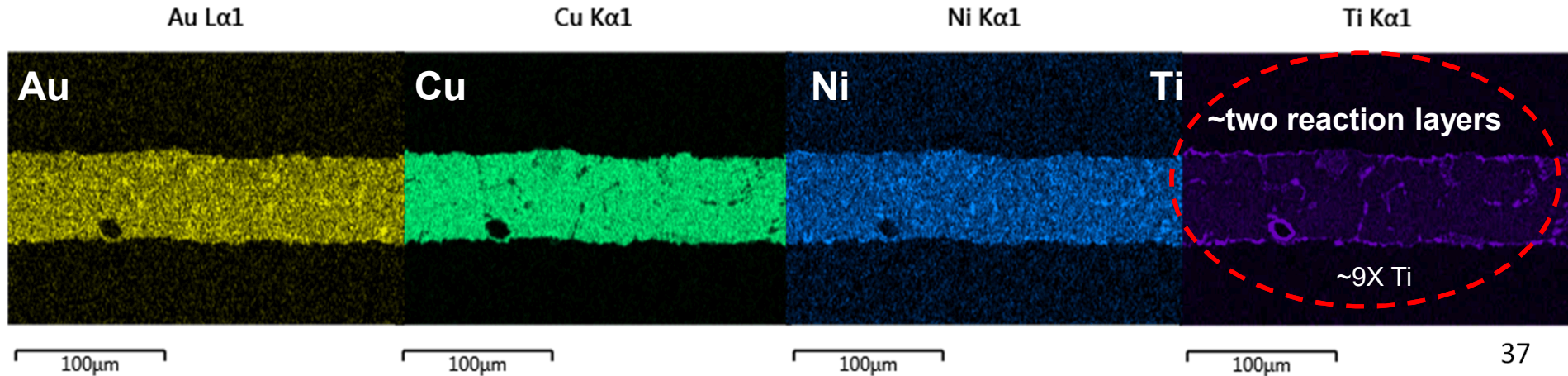
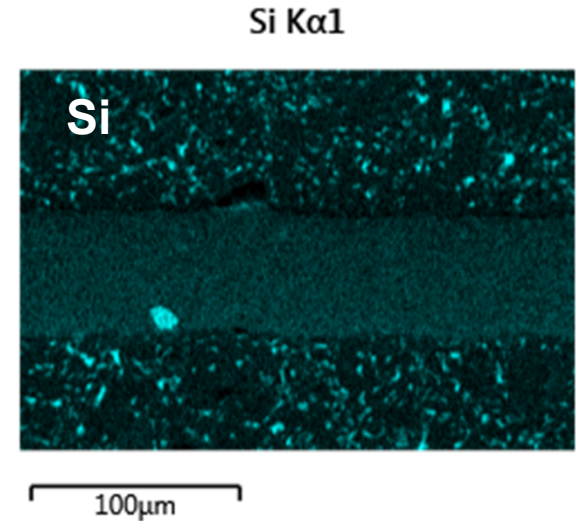
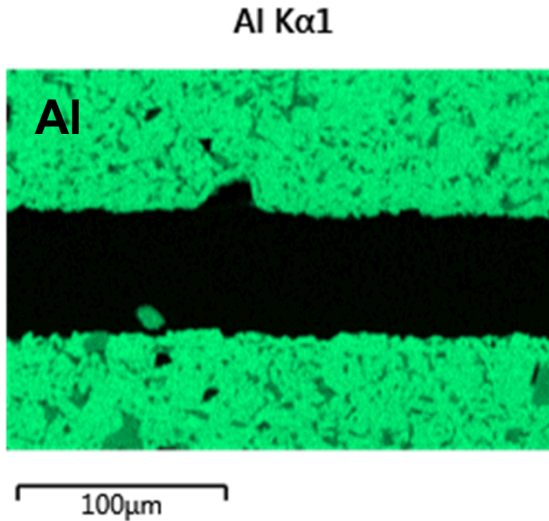
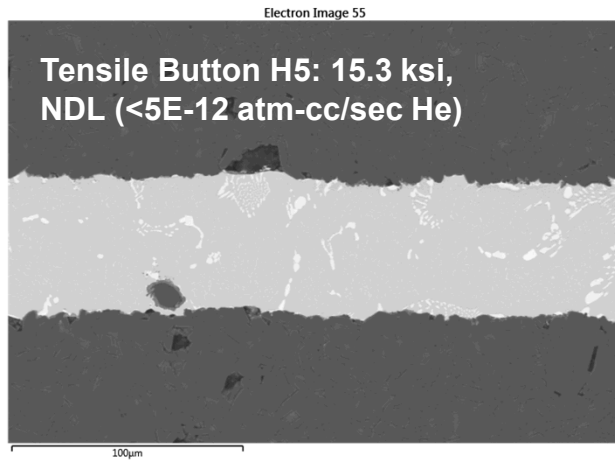
Sample Analysis: ASTM F-19 Tensile Buttons

H5 - Nicoro®+2%Ti, Standard ABA BFM , 75μm thick



Sample Analysis: ASTM F-19 Tensile Buttons

H5 - Nicoro®+2%Ti, Standard ABA BFM , 75μm thick



Outline

Background: Brazing, metal-nonmetal brazing

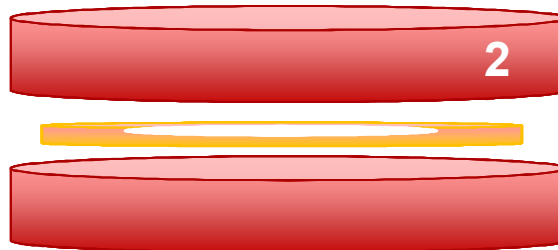
“Standard” active brazing filler metals

Thin-film active element additions & calculations

Test samples: Ceramic-Ceramic Discs (Al-500)

Results

Analysis

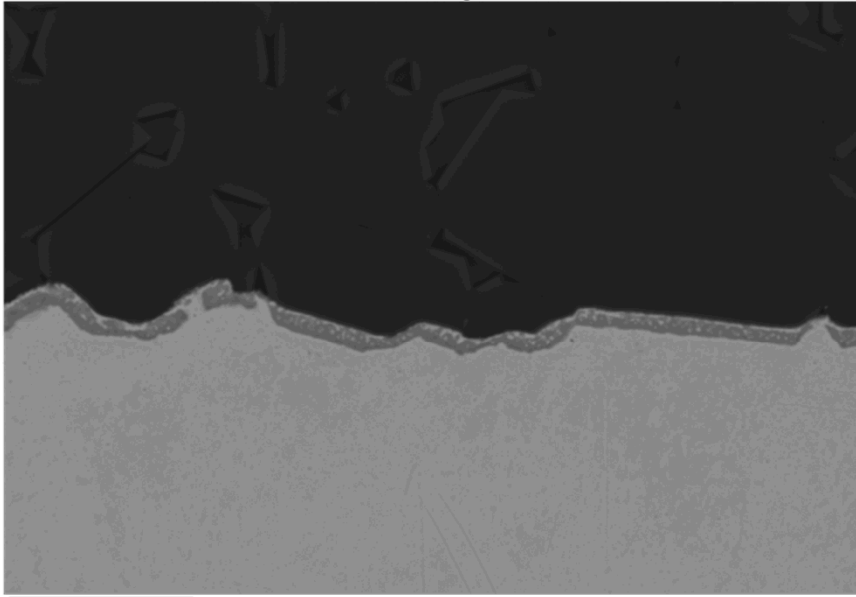


Discussion / Conclusions

Sample Analysis: Ceramic-Ceramic Discs

Disc A – 50 μ m thick 100% Ag + 5kÅ Zr & 5kÅ Ag (99.375%Ag – 0.625%Zr)

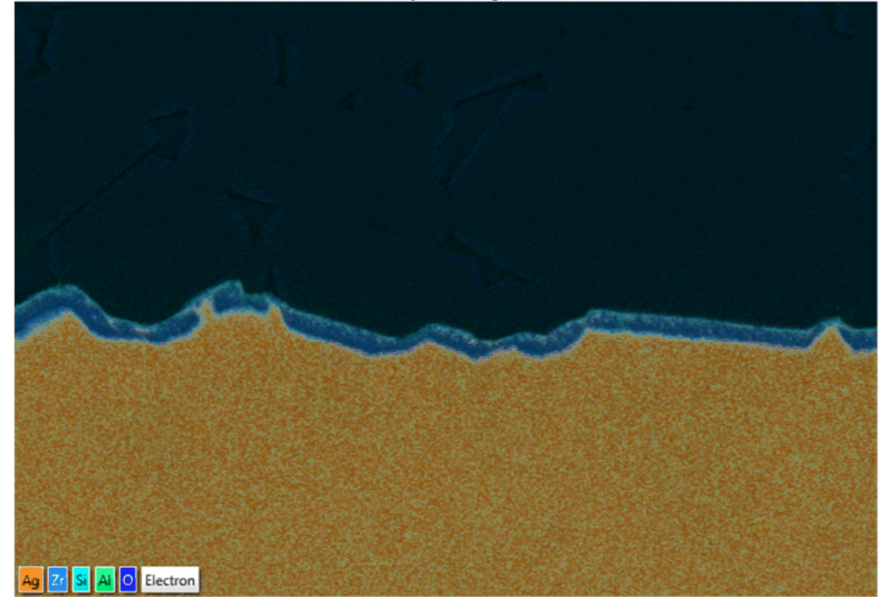
Electron Image 13



Al K α 1

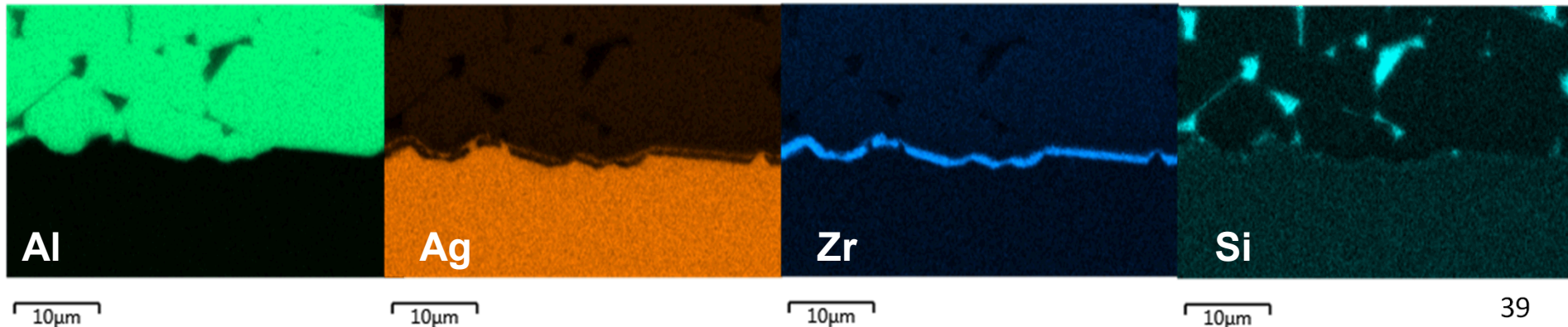
Ag K α 1

EDS Layered Image 13



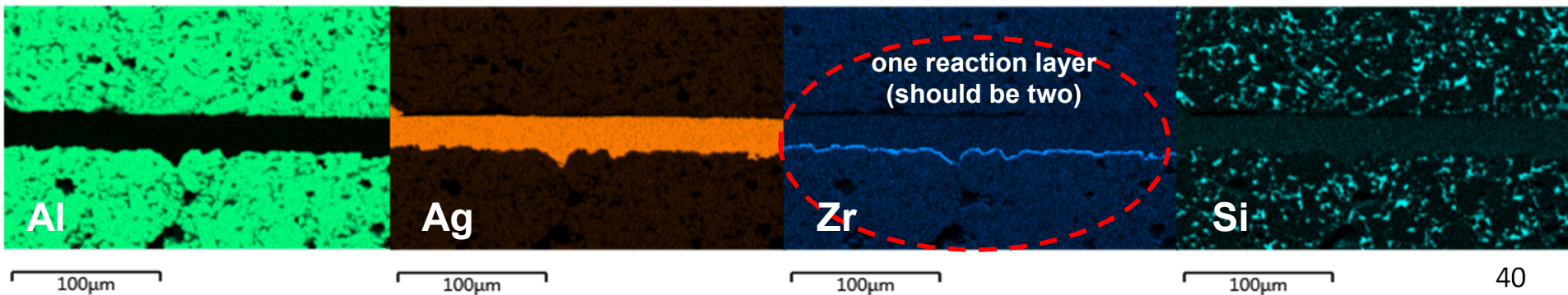
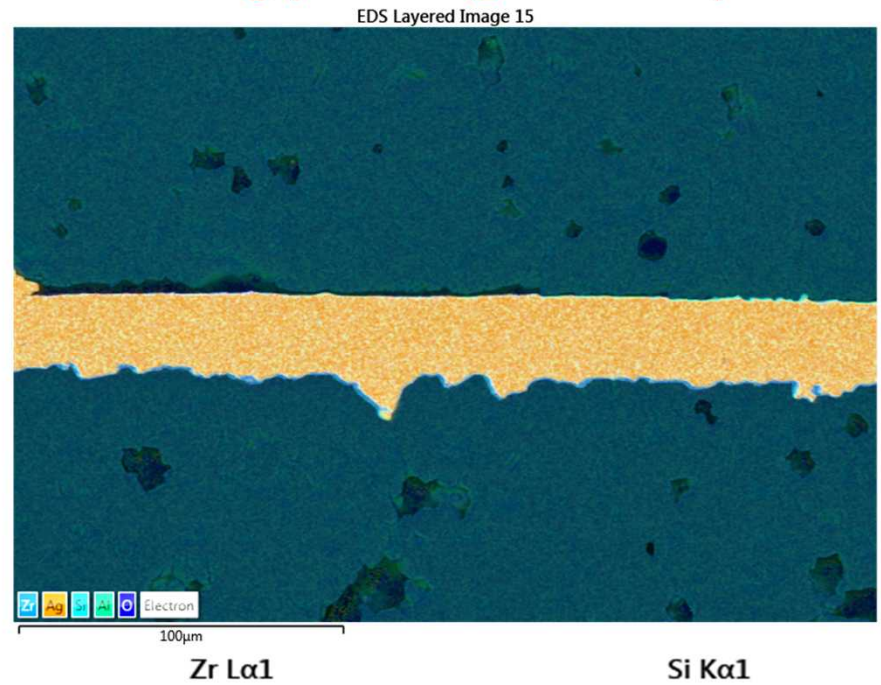
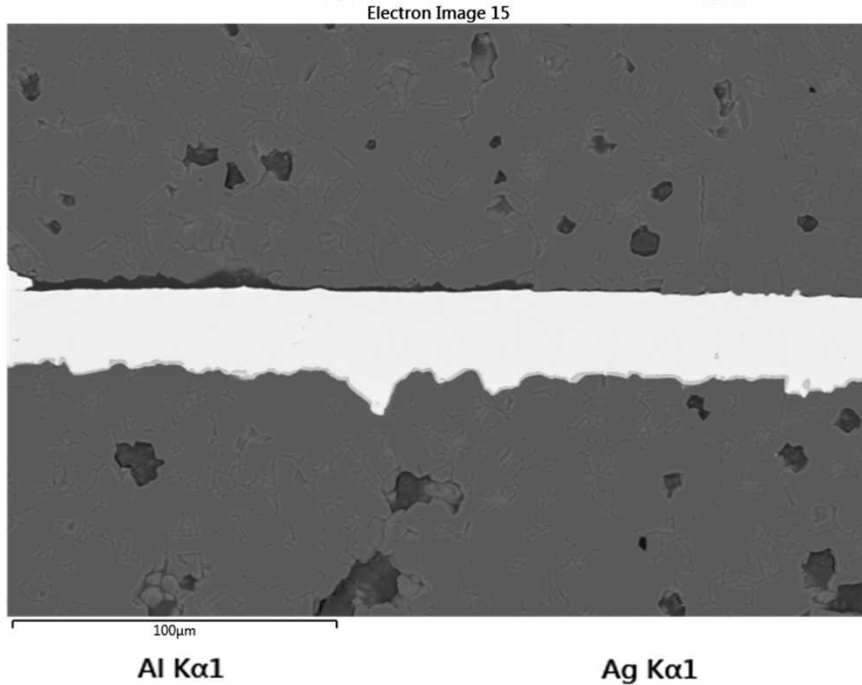
Zr L α 1

Si K α 1



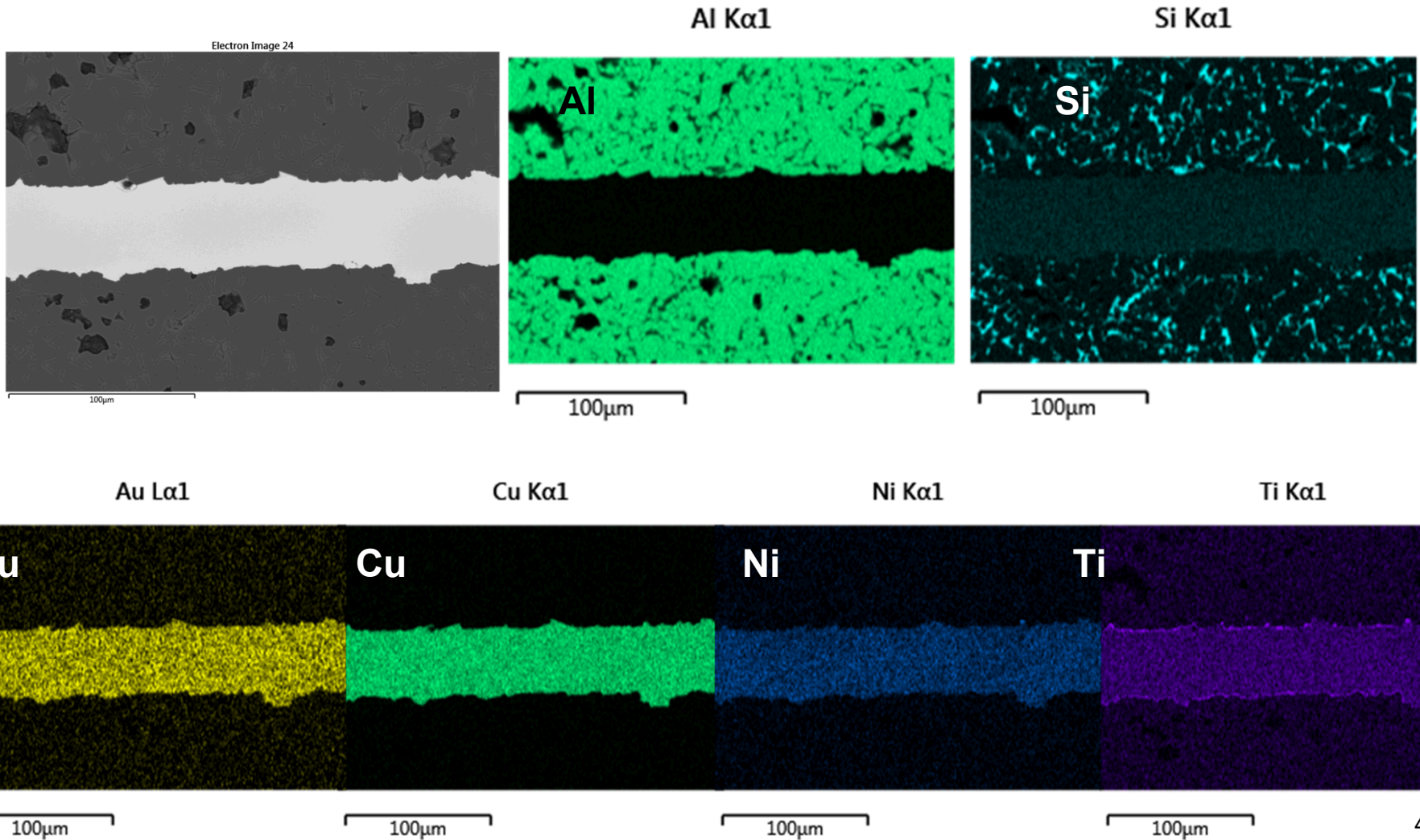
Sample Analysis: Ceramic-Ceramic Discs

Disc B – 75 μ m thick 100% Ag + 5kÅ Zr & 5kÅ Ag (99.6%Ag – 0.4%Zr)



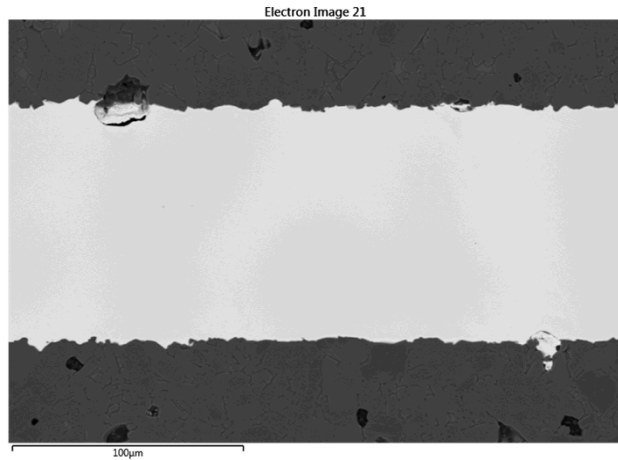
Sample Analysis: Ceramic-Ceramic Discs

Disc C – 50 μ m thick Nicoro®+ 5kÅ Ti & 5kÅ Au (Nicoro®+0.35%Ti)

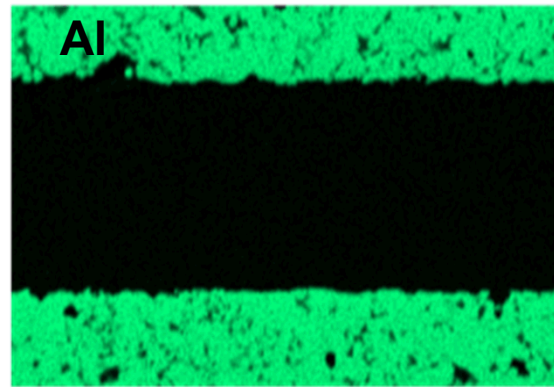


Sample Analysis: Ceramic-Ceramic Discs

Disc D – 75 μ m thick Nicoro®+ 5kÅ Ti & 5kÅ Au (Nicoro®+0.235%Ti)

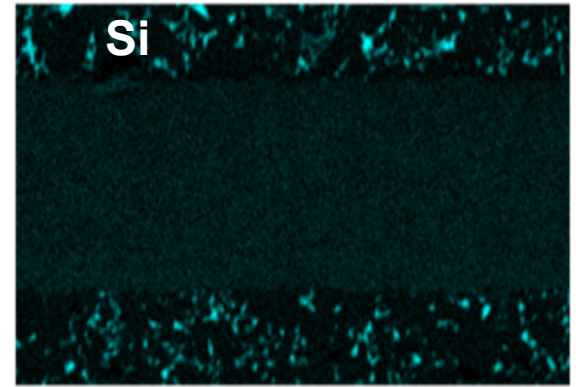


Al K α 1



100 μ m

Si K α 1



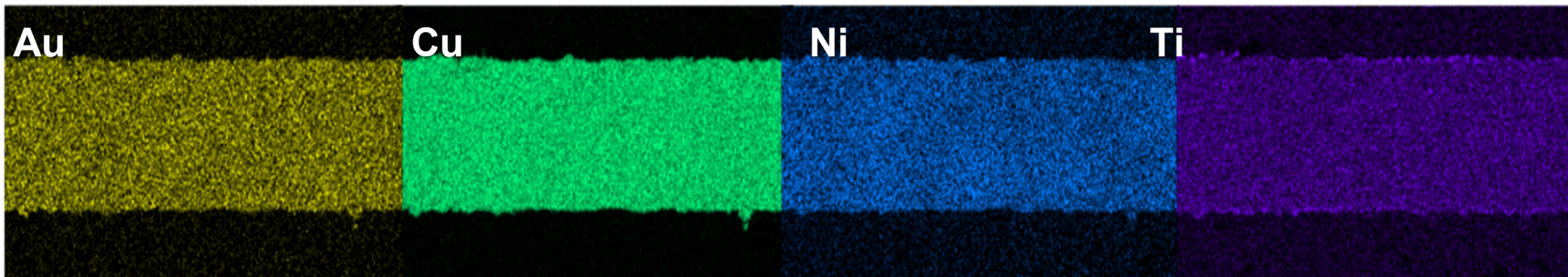
100 μ m

Au L α 1

Cu K α 1

Ni K α 1

Ti K α 1



100 μ m

100 μ m

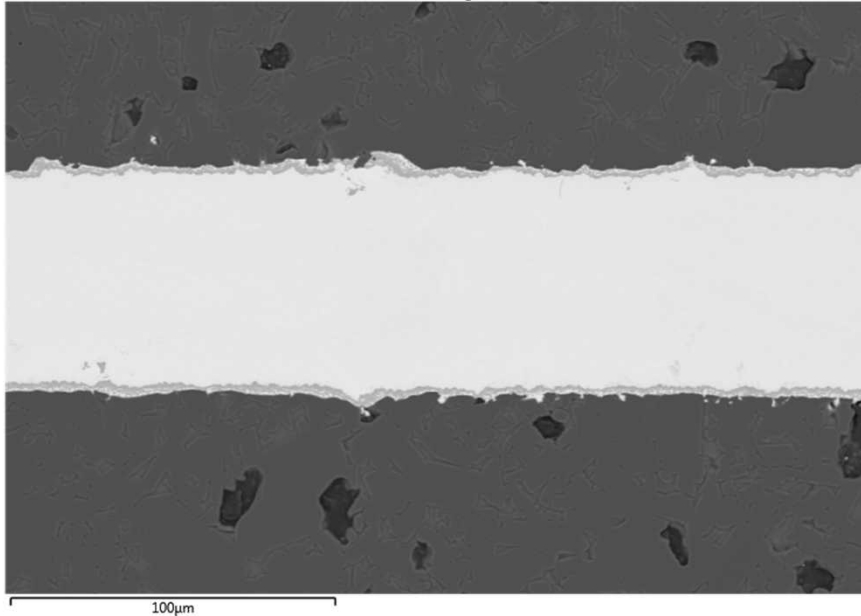
100 μ m

100 μ m

Sample Analysis: Ceramic-Ceramic Discs

Disc E – 50 μ m thick 98% Ag + 2%Zr

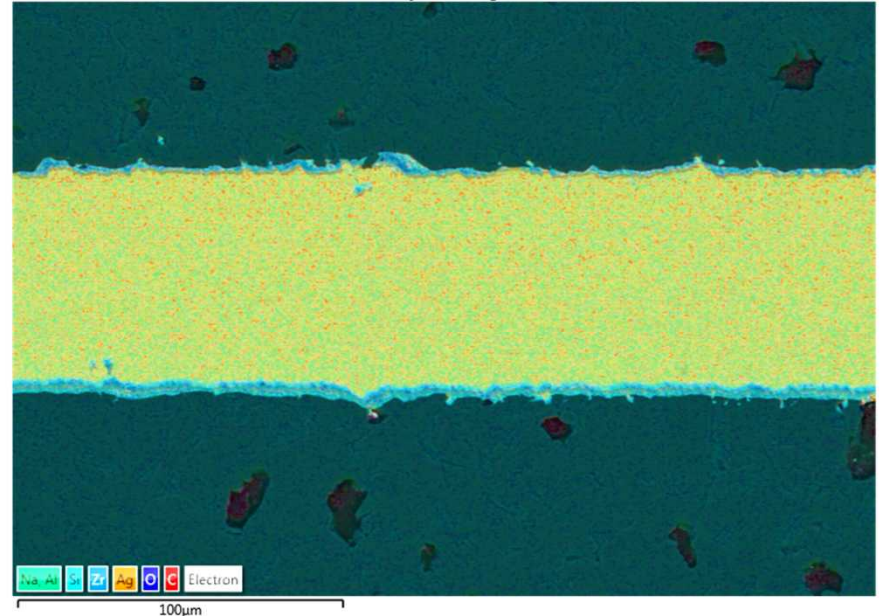
Electron Image 30



Al K α 1

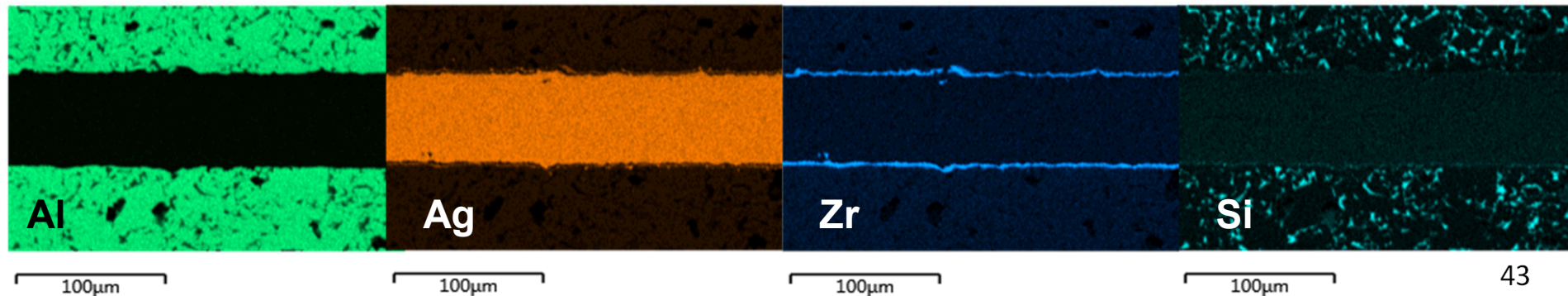
Ag K α 1

EDS Layered Image 30



Zr L α 1

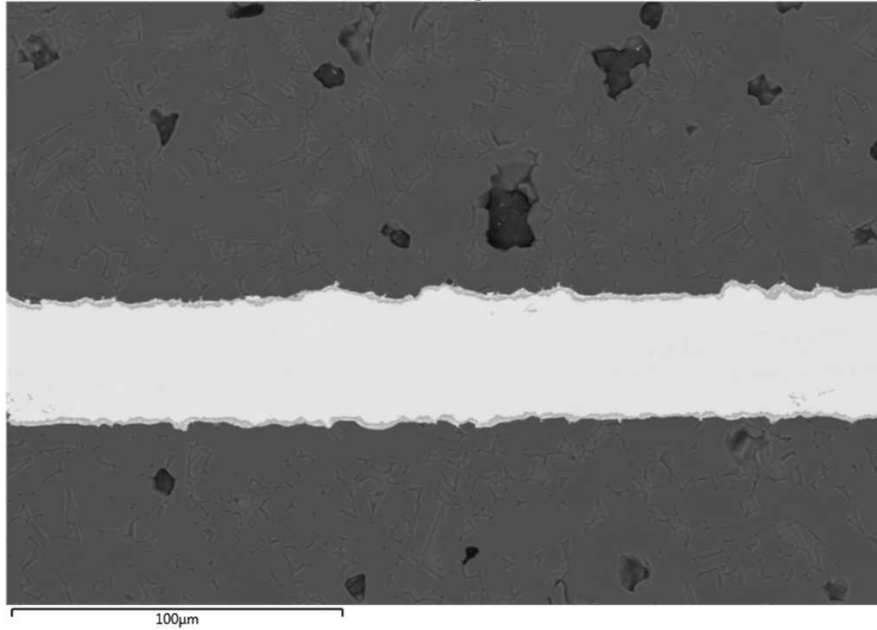
Si K α 1



Sample Analysis: Ceramic-Ceramic Discs

Disc F – 75 μ m thick 98% Ag + 2%Zr

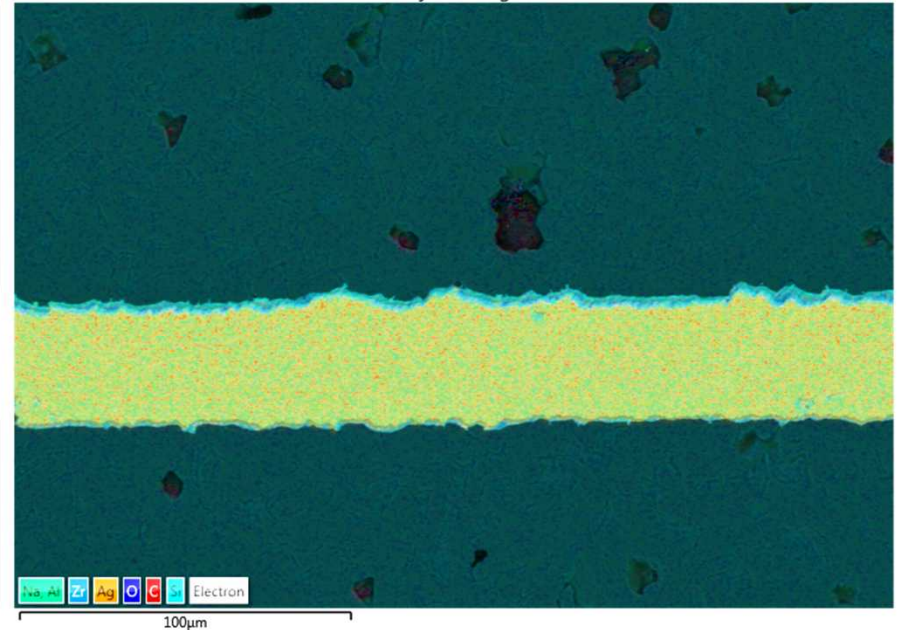
Electron Image 27



Al K α 1

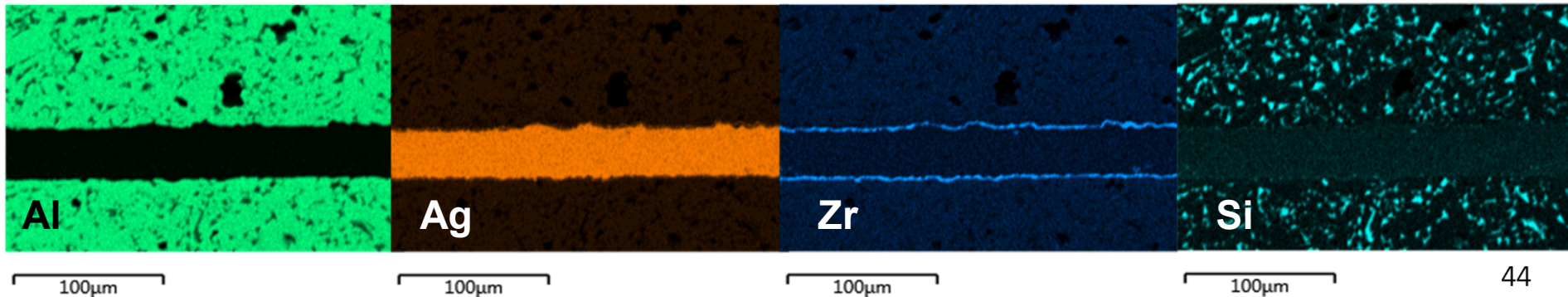
Ag K α 1

EDS Layered Image 27



Zr L α 1

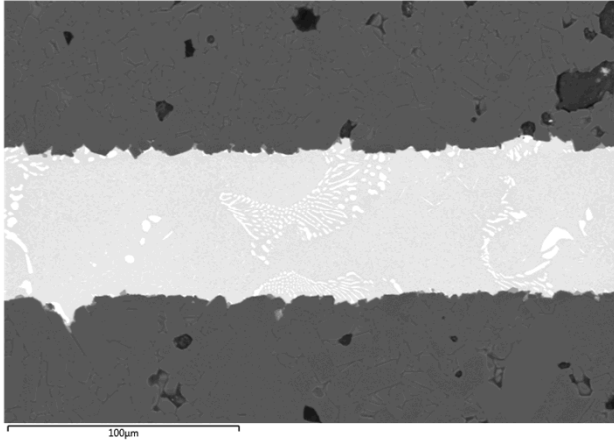
Si K α 1



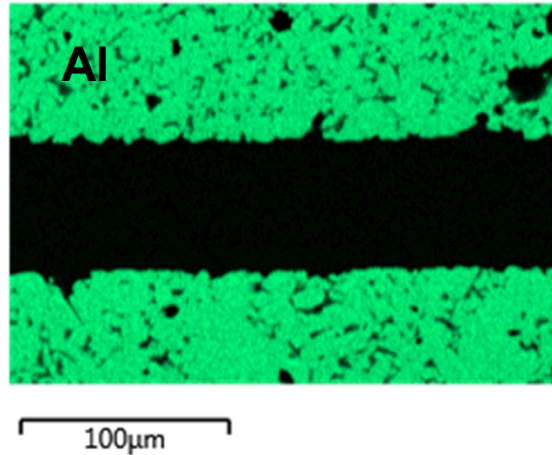
Sample Analysis: Ceramic-Ceramic Discs

Disc G – 50 μ m thick Nicoro®+ 2%Ti

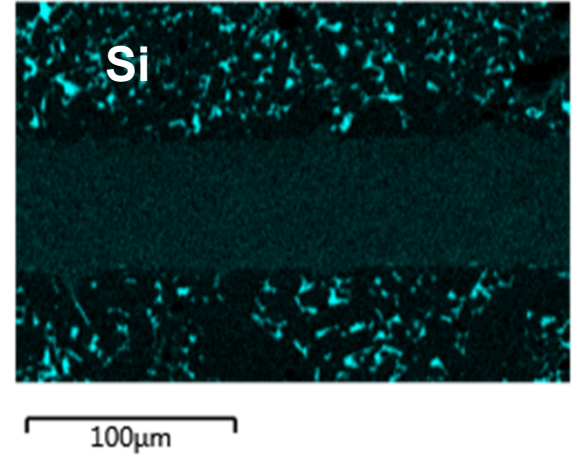
Electron Image 18



Al K α 1



Si K α 1

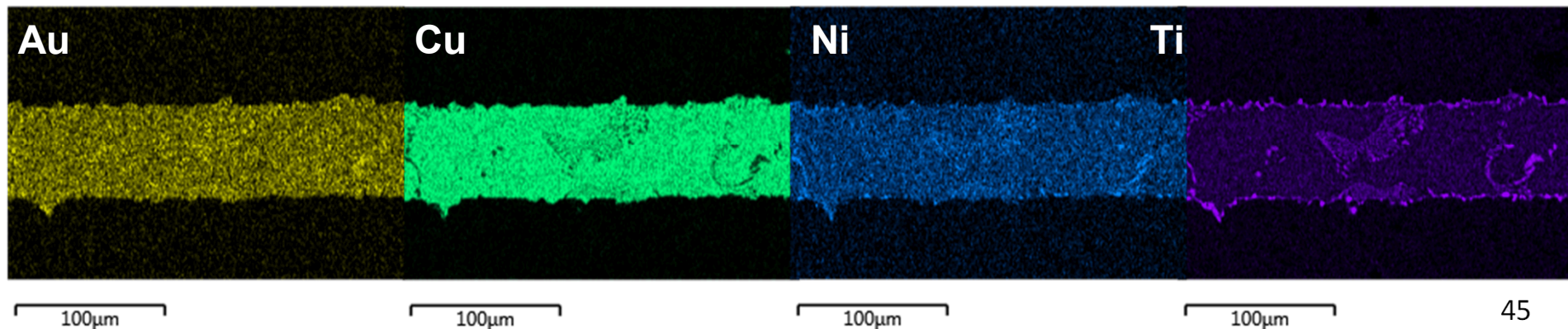


Au L α 1

Cu K α 1

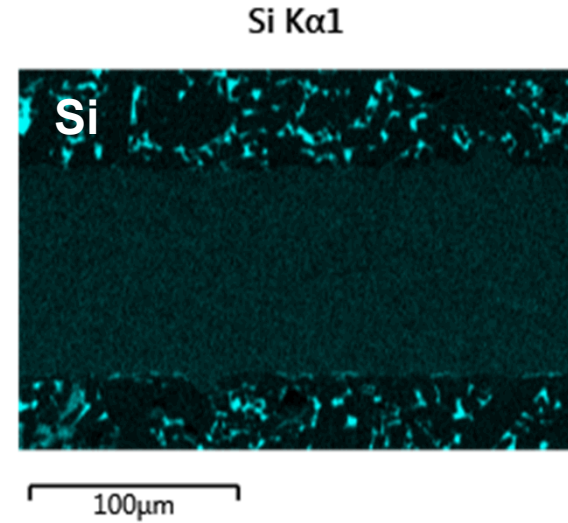
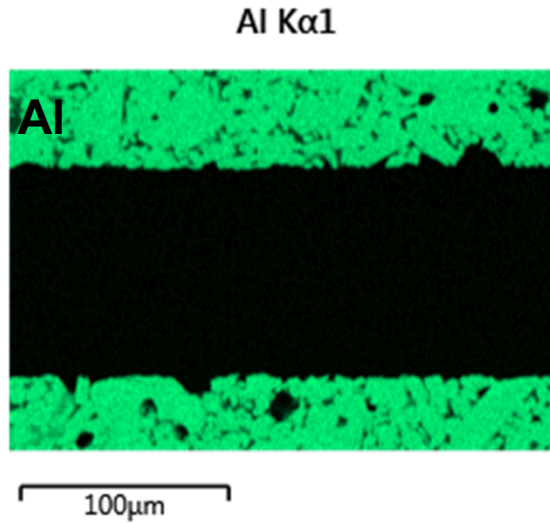
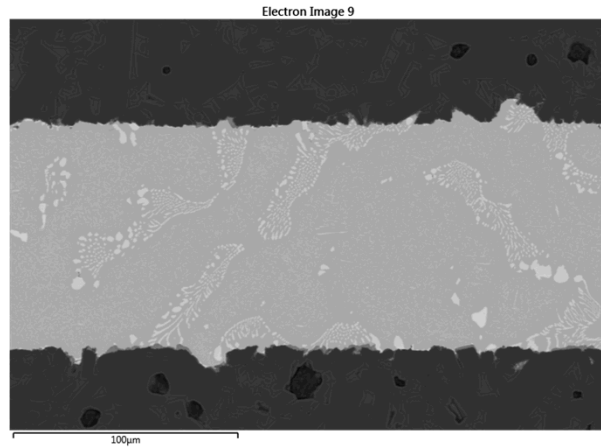
Ni K α 1

Ti K α 1



Sample Analysis: Ceramic-Ceramic Discs

Disc H – 75 μ m thick Nicoro®+ 2%Ti

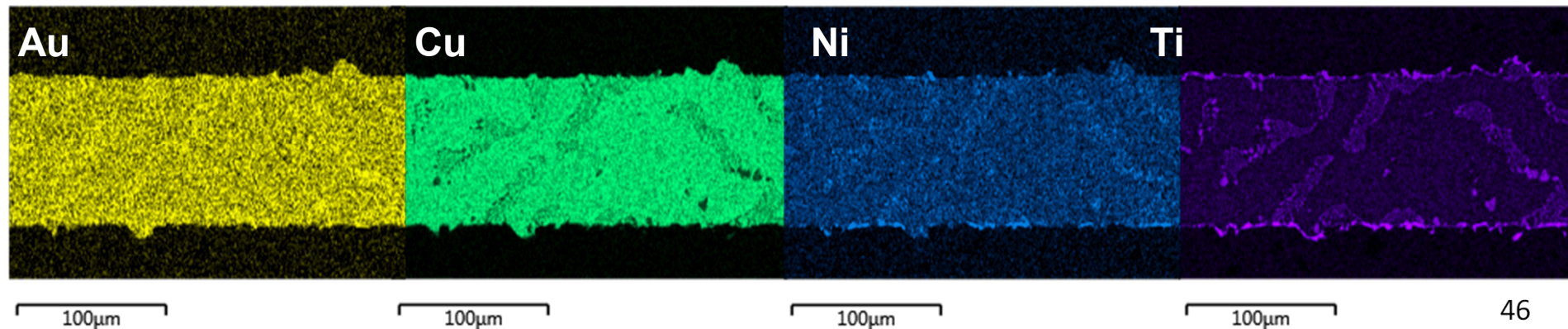


Au L α 1

Cu K α 1

Ni K α 1

Ti K α 1



Outline

Background: Brazing, metal-nonmetal brazing

“Standard” active brazing filler metals

Thin-film active element additions & calculations

Test samples: Ceramic cylinders-Metal Washers (Nb & Kovar®)

Results

Analysis

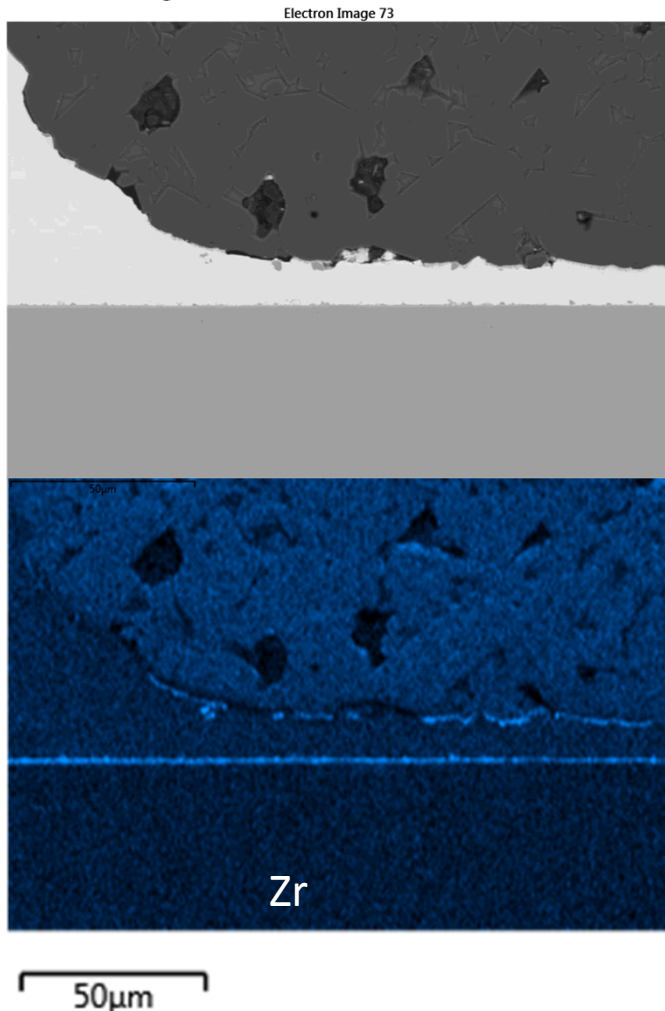


Discussion / Conclusions

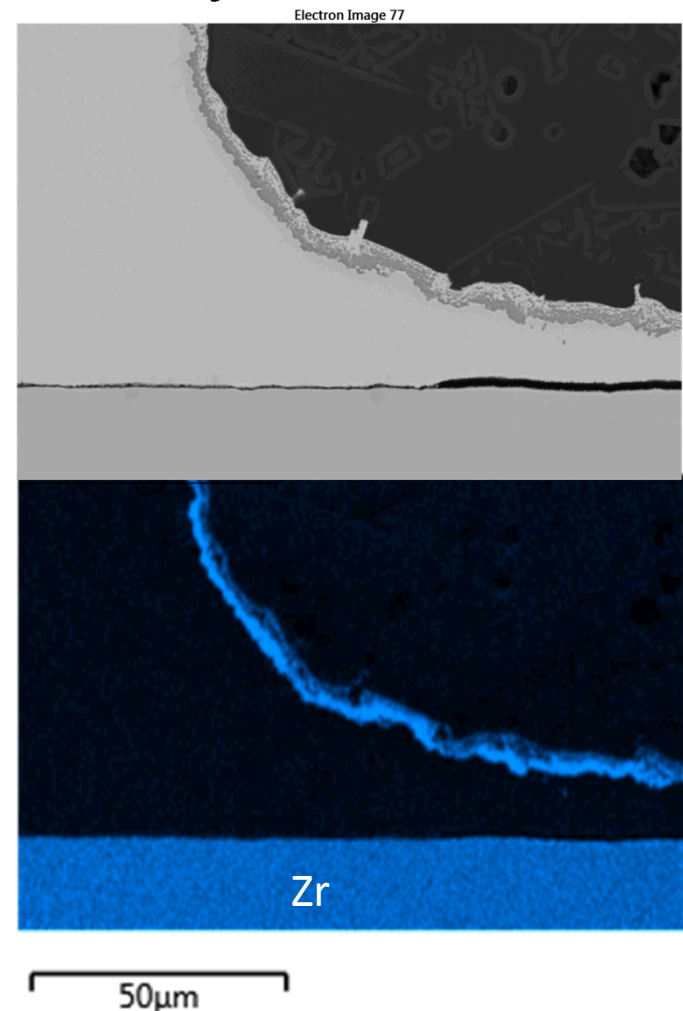
Sample Analysis: Ceramic-Metal Washers

Disc A – 50 μ m thick 100% Ag + 5kÅ Zr & 5kÅ Ag (99.375%Ag – 0.625%Zr)

Ceramic cylinder - Kovar® Washer



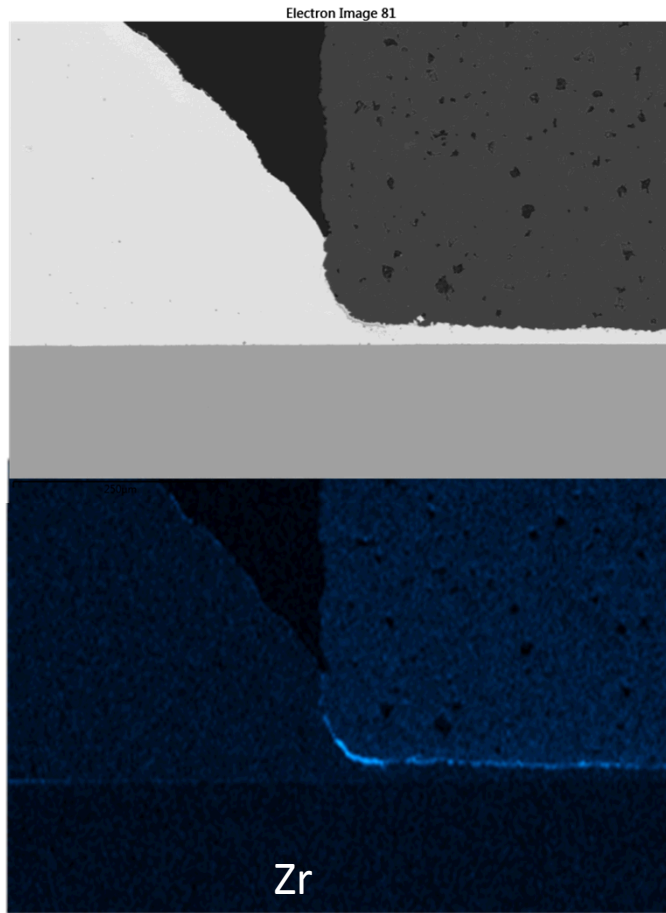
Ceramic cylinder - Niobium Washer



Sample Analysis: Ceramic-Metal Washers

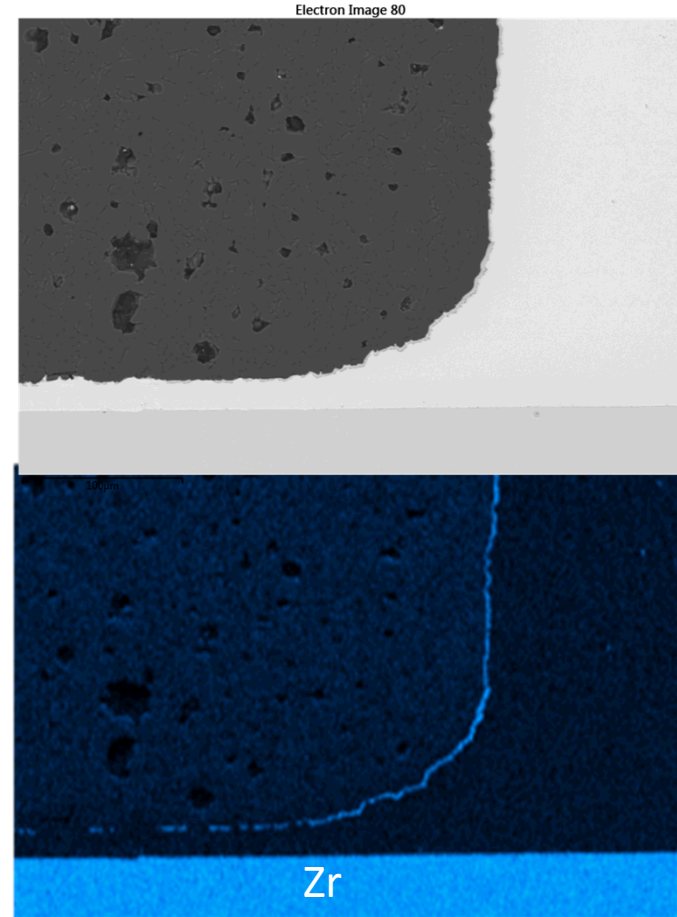
Disc B – 75 μ m thick 100% Ag + 5kÅ Zr & 5kÅ Ag (99.6%Ag – 0.4%Zr)

Ceramic cylinder - Kovar® Washer



250 μ m

Ceramic cylinder - Niobium Washer



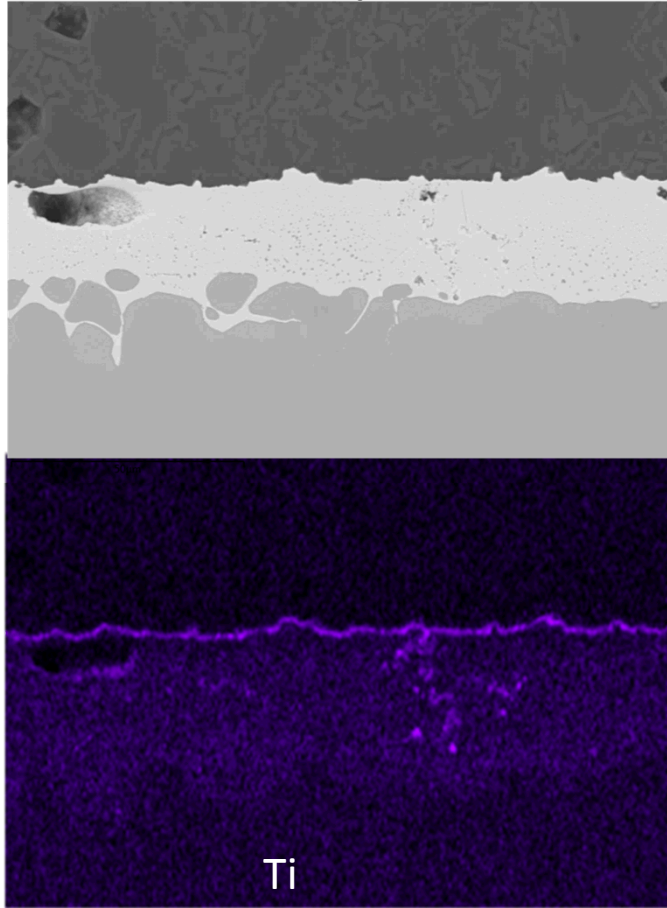
100 μ m

Sample Analysis: Ceramic-Metal Washers

Disc C – 50 μ m thick Nicoro®+ 5kÅ Ti & 5kÅ Au (Nicoro®+0.35%Ti)

Ceramic cylinder - Kovar® Washer

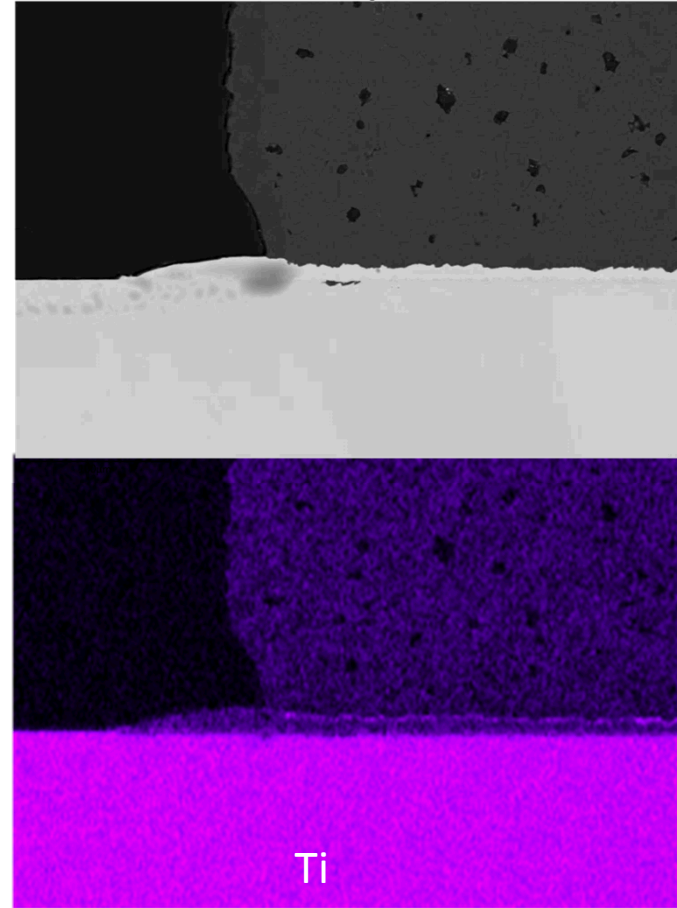
Electron Image 106



50 μ m

Ceramic cylinder - Niobium Washer

Electron Image 86



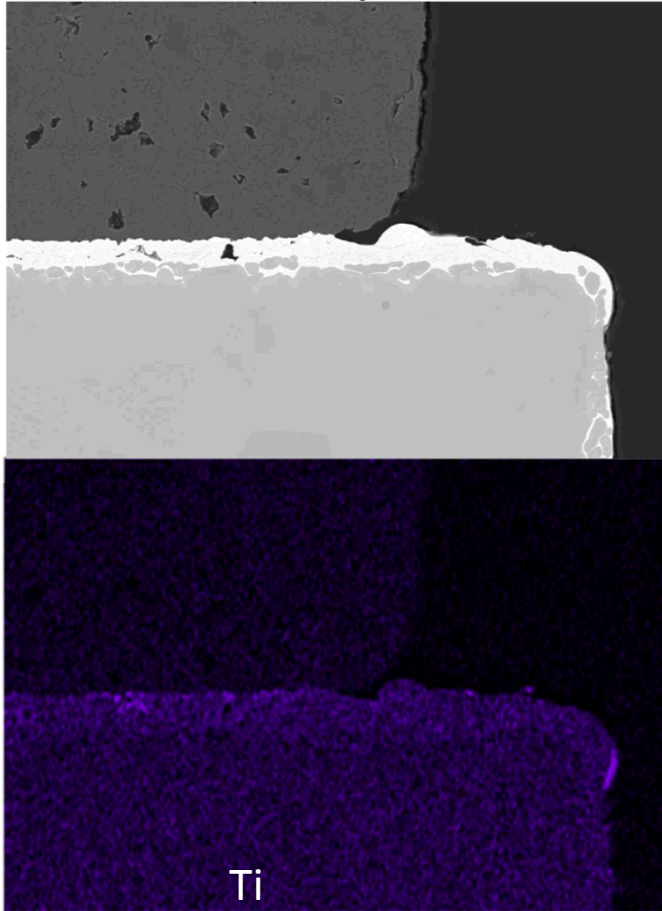
100 μ m

Sample Analysis: Ceramic-Metal Washers

Disc D – 75 μ m thick Nicoro®+ 5kÅ Ti & 5kÅ Au (Nicoro®+0.235%Ti)

Ceramic cylinder - Kovar® Washer

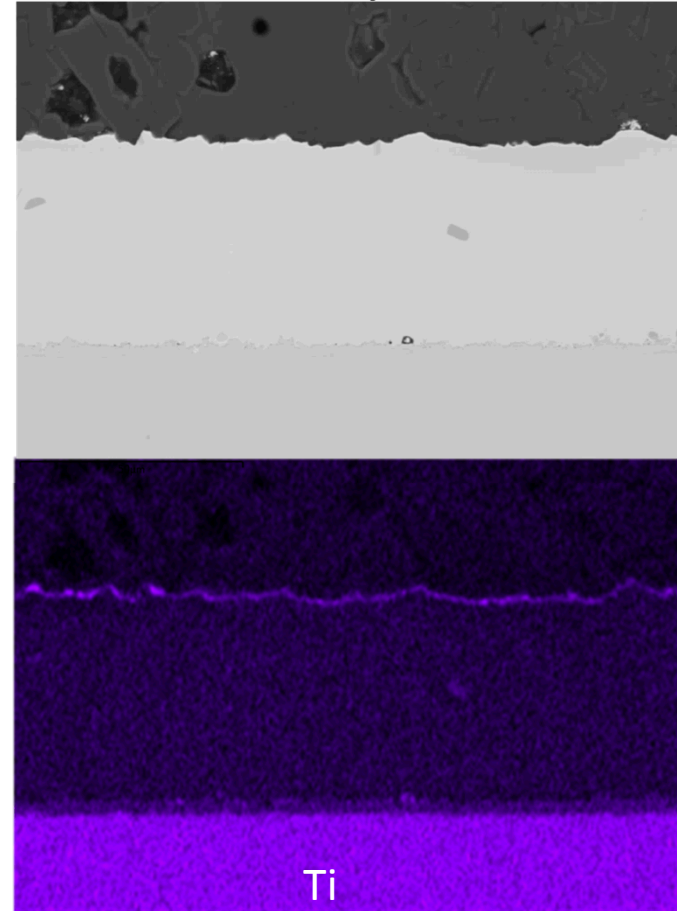
Electron Image 98



100 μ m

Ceramic cylinder - Niobium Washer

Electron Image 92

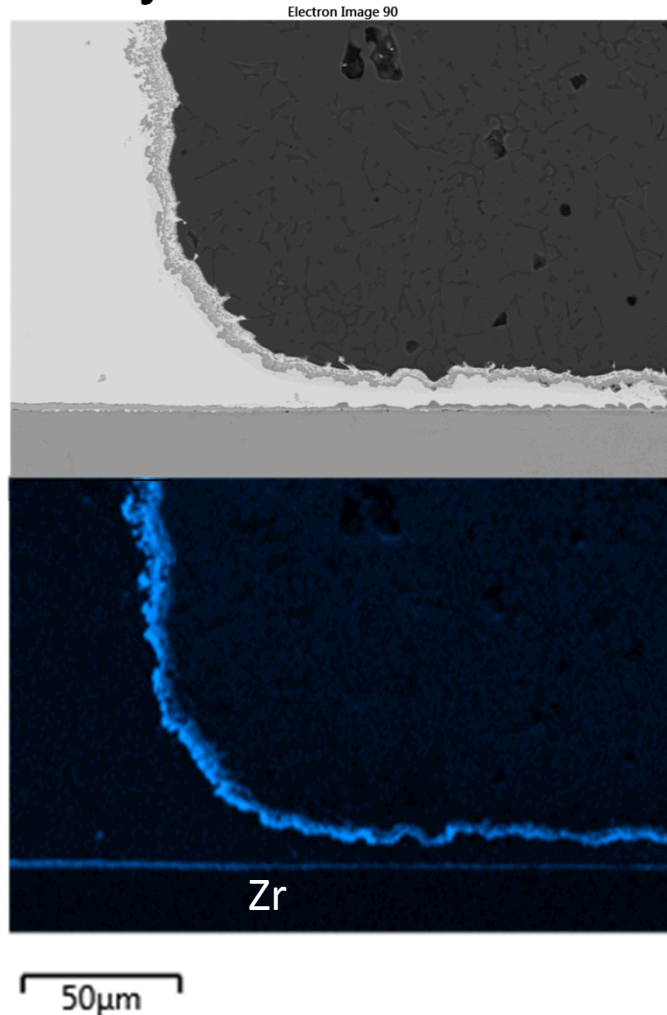


50 μ m

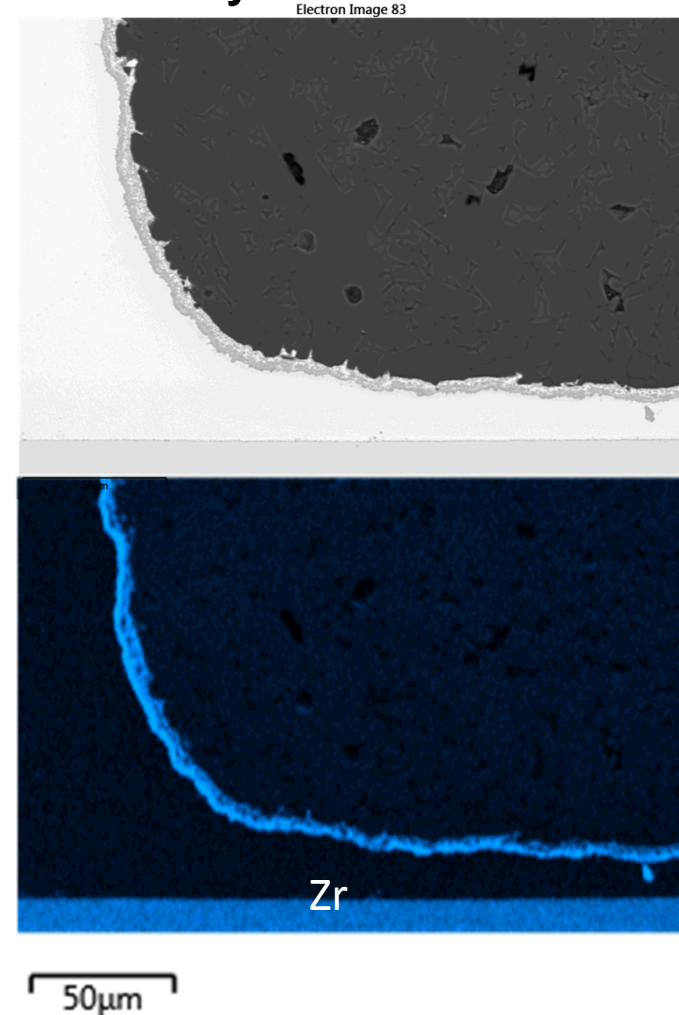
Sample Analysis: Ceramic-Metal Washers

Disc E – 50μm thick 98% Ag + 2%Zr

Ceramic cylinder - Kovar® Washer



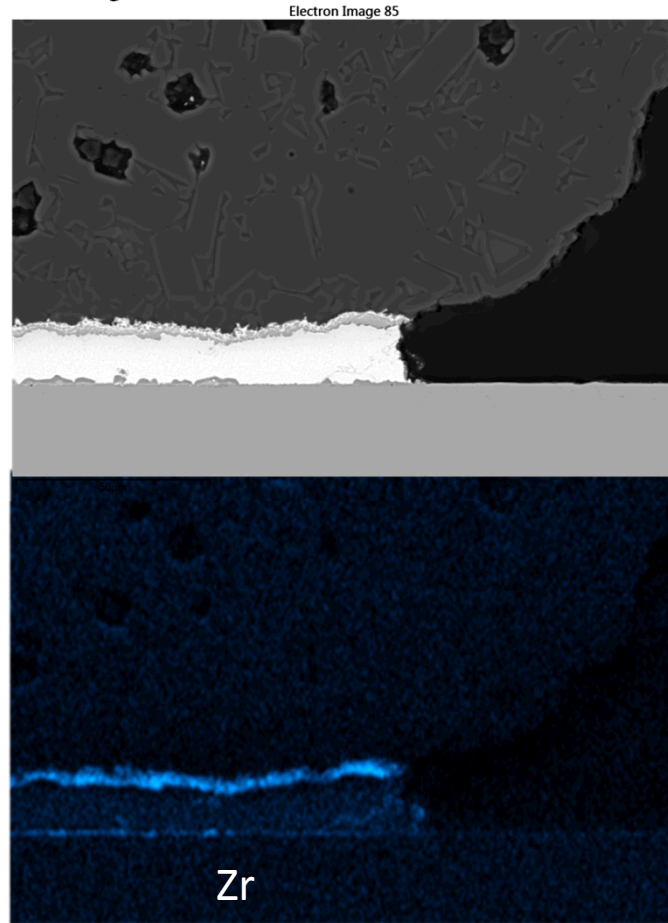
Ceramic cylinder - Niobium Washer



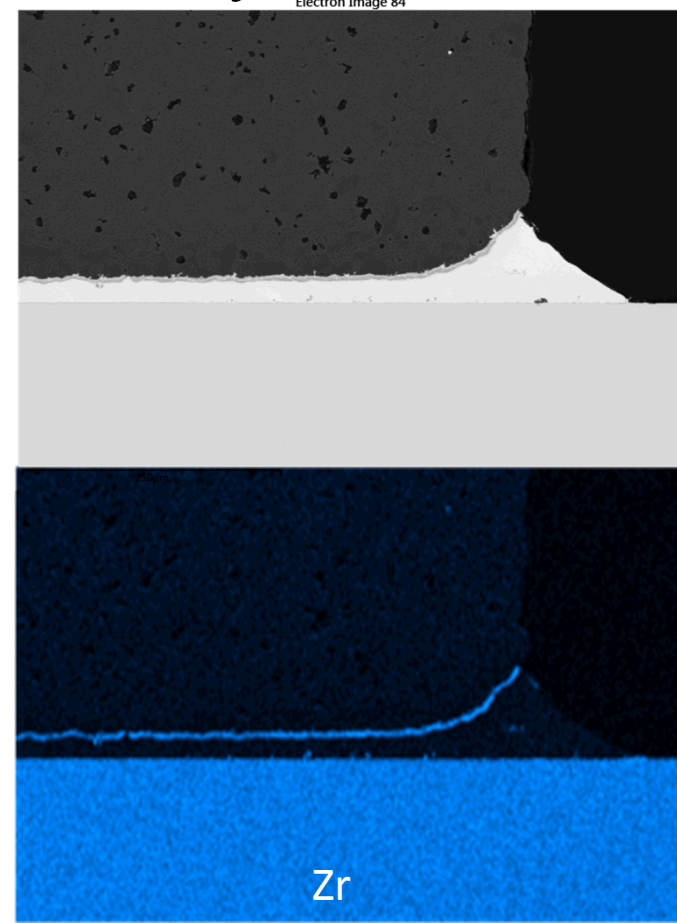
Sample Analysis: Ceramic-Metal Washers

Disc F – 75 μ m thick 98% Ag + 2%Zr

Ceramic cylinder - Kovar® Washer



Ceramic cylinder - Niobium Washer

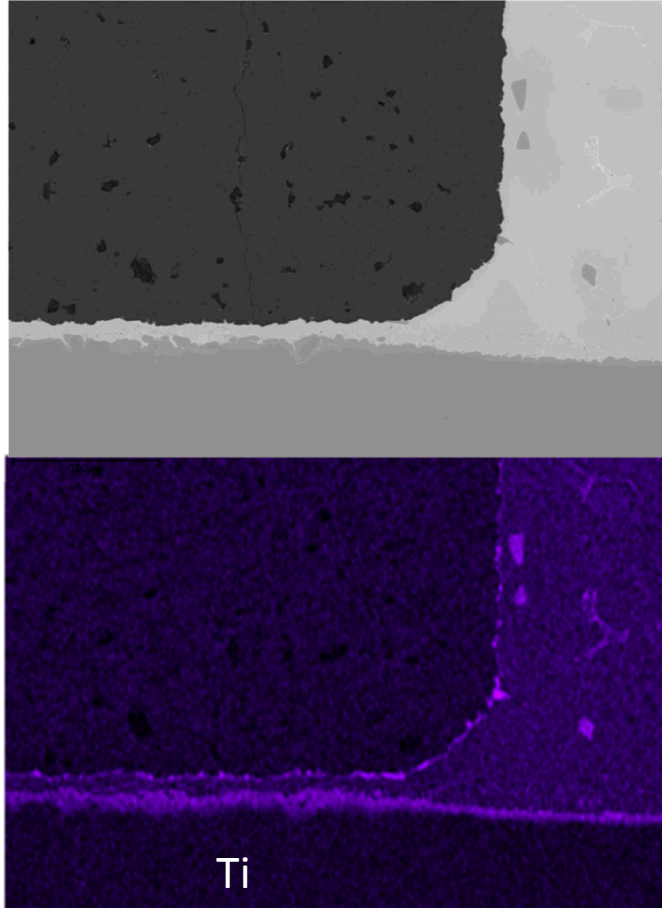


Sample Analysis: Ceramic-Metal Washers

Disc G – 50 μ m thick Nicoro®+ 2%Ti

Ceramic cylinder - Kovar® Washer

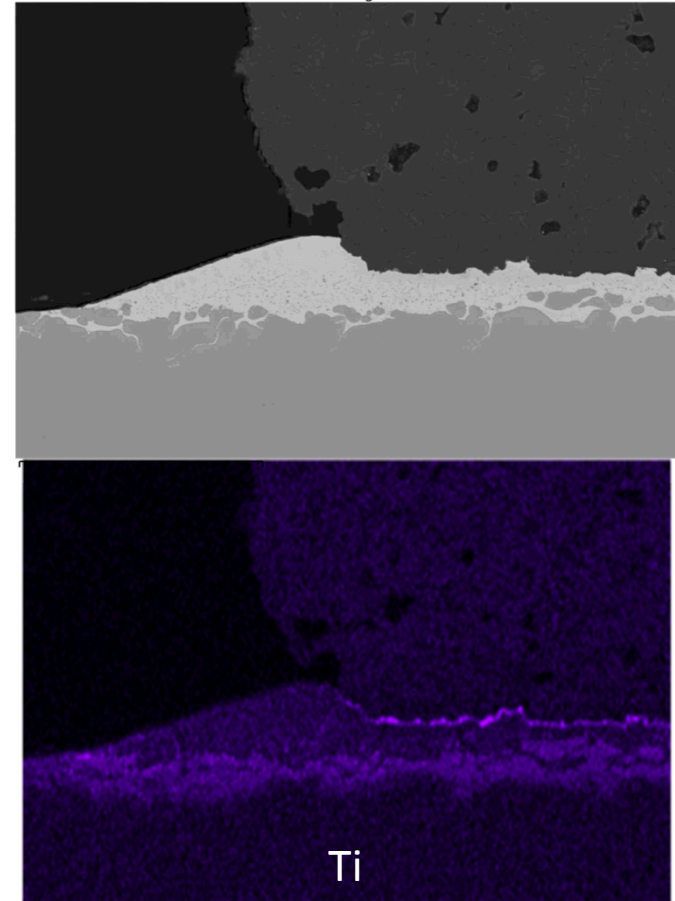
Electron Image 101



100 μ m

Ceramic cylinder - Niobium Washer

Electron Image 103



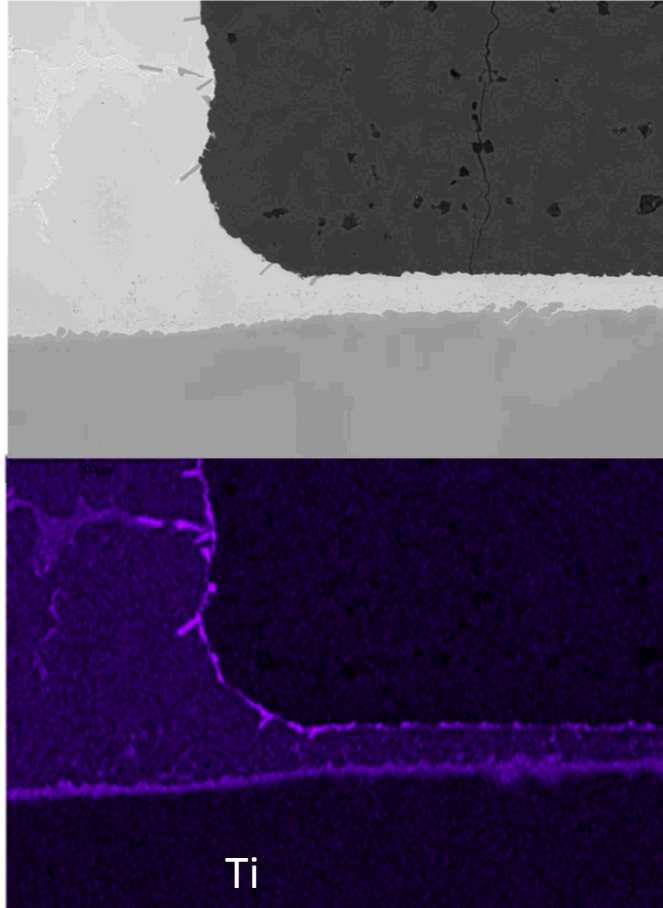
100 μ m

Sample Analysis: Ceramic-Metal Washers

Disc H – 75 μ m thick Nicoro®+ 2%Ti

Ceramic cylinder - Kovar® Washer

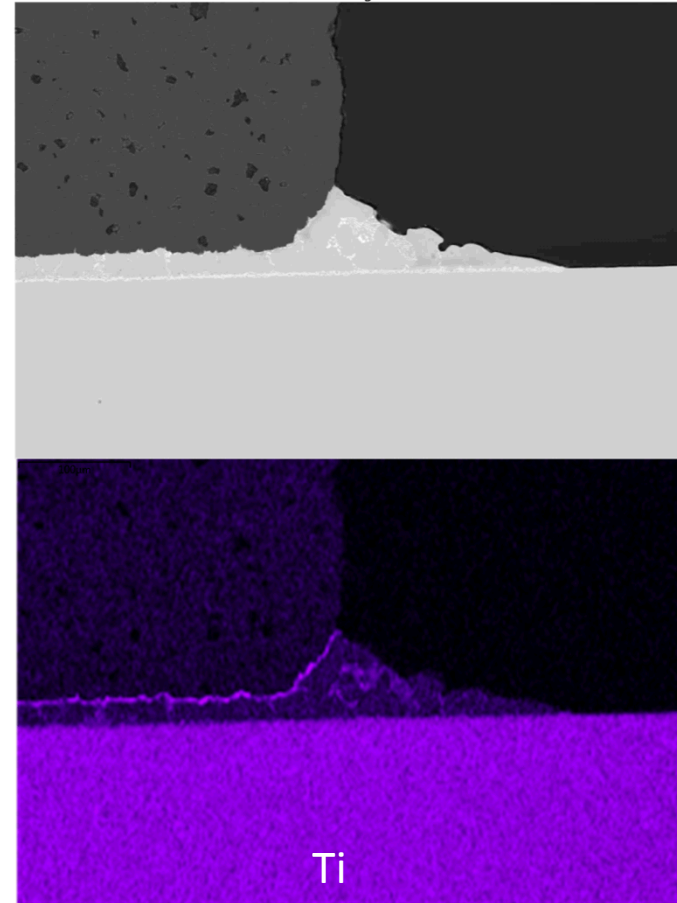
Electron Image 100



100 μ m

Ceramic cylinder - Niobium Washer

Electron Image 104



100 μ m

Outline

Background: Brazing, metal-nonmetal brazing

“Standard” active brazing filler metals

Thin-film active element additions & calculations

Test samples: Ceramic cylinders-Metal Washers (Nb & Kovar[®])

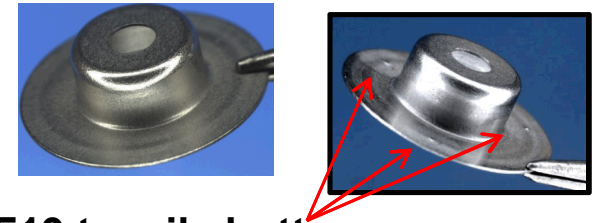
Results

Analysis

Discussion / Conclusions

Discussion

- Metal-Ceramic braze joint tensile strengths are typically in the range of 14-24 ksi (15-165Mpa), and hermetic (helium leak rates < 5E-12 atm-cc/sec)
- In this study the average tensile strengths of the ceramic-ceramic samples fabricated with lower than standard active element content were much less than is typically observed, ranging from 0.8 – 4.3ksi (slide 30)
- The strengths of the ceramic-ceramic samples made with the commercial active braze filler metals were also lower than anticipated, with more variation in the data.
- Further investigation revealed that different ceramic firing cycles and atmospheres had been used.



Typical average strengths of metal-ceramic ASTM-F19 tensile buttons (dimpled interlayers)

Filler Metal	Non-metal Substrate	Metal Substrate	Brazing Temperature	Furnace Atmosphere	Tensile Strength ksi (MPa)
97Ag-1Cu-2Zr	94% Alumina	Fe-29Ni-17Co	963°C, 3 minutes above liquidus	Partial pressure Ar	21 ± 3 (147 ± 21)
97Ag-1Cu-2Zr	96% Alumina	Fe-29Ni-17Co (dimpled, 0.002")	963°C, 3 minutes above liquidus	Partial pressure Ar	24 ± 4 (165 ± 21)
62Cu-35Au- 2Ti-1Ni	94% Alumina	Niobium	1030°C – 2 minutes	Vacuum	14 ± 3 (94 ± 21)
62Cu-35Au- 2Ti-1Ni	94% Alumina	Niobium (dimpled, 0.0015")	1030°C – 2 minutes	Vacuum	17 ± 3 (114 ± 21)

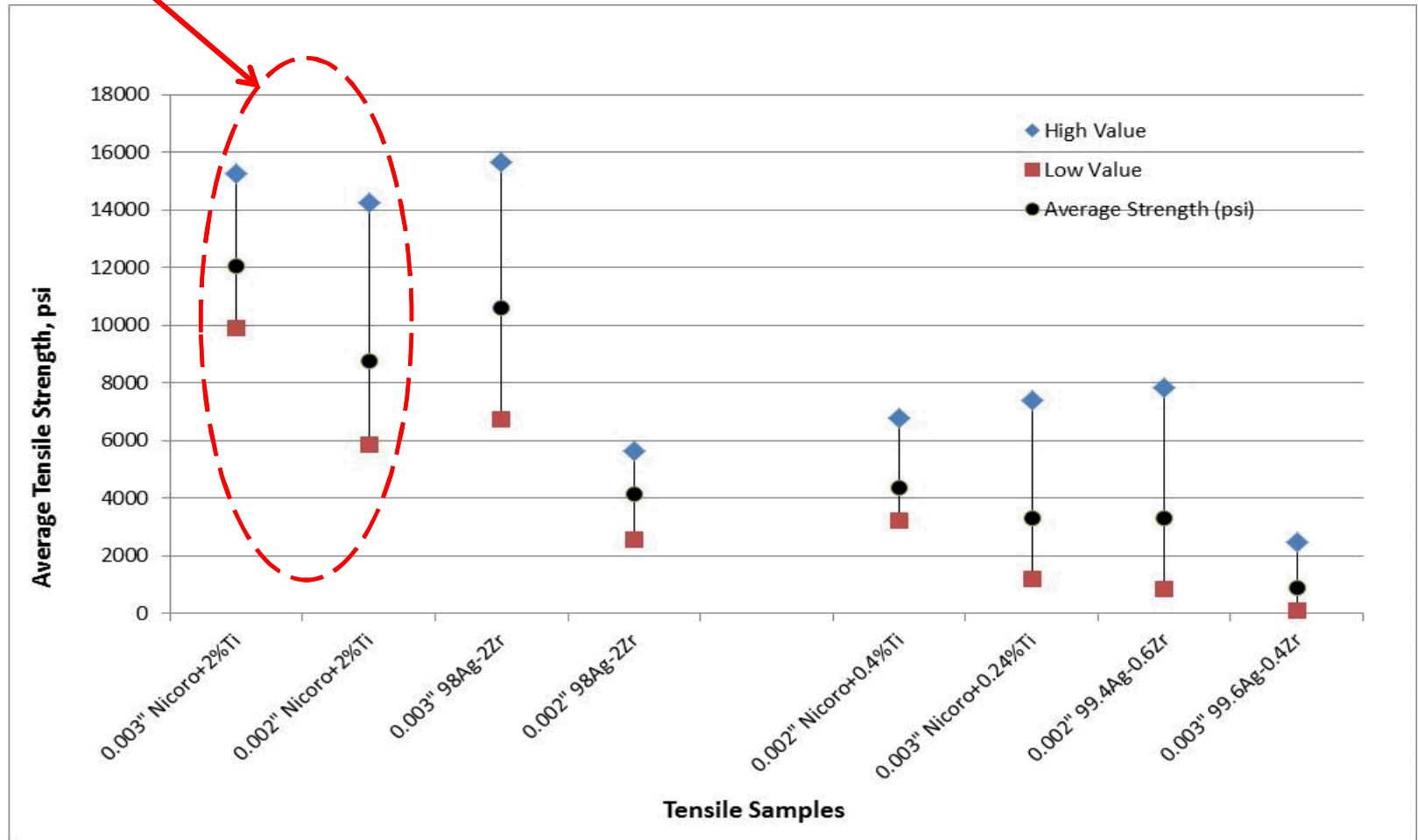
MTS crosshead speed = 8.38E-03 mm/sec)

Helium leak rates <5E-12 atm-cc/sec)

Different resintering
atmospheres &
temperatures within
the groups
discovered.

Discussion

Ceramic-Ceramic ASTM-F19 Tensile Strengths



Discussion

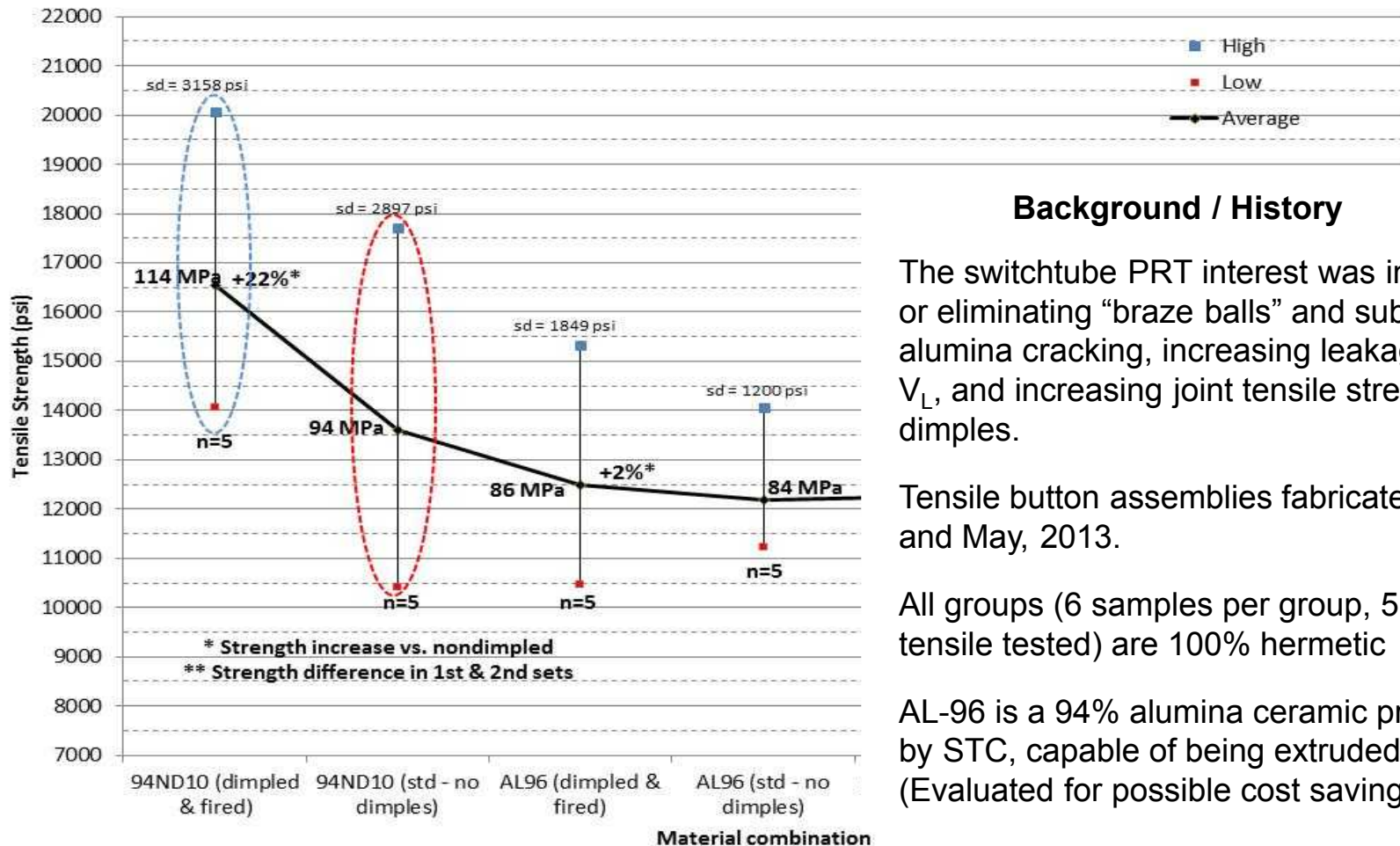
Ceramic-Ceramic ASTM-F19 Tensile Strengths

Discovered
differences in
resintering
atmospheres
and
temperatures.

Run ID	No Interlayer	BFM thickness (mils)	He Leak Rate (atm-cc/sec)	Tensile Load (lbf)	Tensile Stress (psi)	Notes:				
06042014F402-1		2	NDL	1008.1	5632	98Ag-2Zr, Standard ABA BFM				
06042014F402-2		2	2.00E-06	760.6	4249	98Ag-2Zr, Standard ABA BFM				
06042014F402-3		2	1.00E-03	455.8	2546	98Ag-2Zr, Standard ABA BFM				
06042014F402-4		2	6.00E-07	737.3	4119	98Ag-2Zr, Standard ABA BFM				
				Avg Stress =	4137	1261.952				
06042014F402-5		3	NDL	2804.4	15667	98Ag-2Zr, Standard ABA BFM				
06042014F402-6		3	5.00E-08	1725.3	9639	98Ag-2Zr, Standard ABA BFM				
06042014F402-7		3	NDL	1864.5	10416	98Ag-2Zr, Standard ABA BFM				
06042014F402-8		3	7.00E-08	1209.3	6756	98Ag-2Zr, Standard ABA BFM				
				Avg Stress =	10619	3715.235				
Run ID	No Interlayer	BFM thickness (mils)	Helium Leak Rate (atm-cc/sec)	Tensile Load (lbf)	Tensile Stress (psi)	Notes:				
06052014F401-1		2	4.00E-07	2549.8	14245	Nicoro [®] +2%Ti, Standard ABA BFM				
06052014F401-2		2	2.00E-05	1529.8	8546	Nicoro [®] +2%Ti, Standard ABA BFM				
06052014F401-3		2	7.00E-05	1047.2	5850	Nicoro [®] +2%Ti, Standard ABA BFM				
06052014F401-4		2	4.00E-04	1139.3	6365	Nicoro [®] +2%Ti, Standard ABA BFM				
				Avg Stress =	8752	3844.071				
06052014F401-5		3	NDL	2734.9	15279	Nicoro [®] +2%Ti, Standard ABA BFM				
06052014F401-6		3	NDL	2149.8	12010	Nicoro [®] +2%Ti, Standard ABA BFM				
06052014F401-7		3	2.00E-06	1770.7	9892	Nicoro [®] +2%Ti, Standard ABA BFM				
06052014F401-8		3	2.00E-07	1965.7	10982	Nicoro [®] +2%Ti, Standard ABA BFM				
				Avg Stress =	12041	2325.507				

Historical Data: ASTM F-19 Tensile Buttons

Tensile Strength: AL96 vs. 94ND10 Nicoro+2%Ti braze filler metal (std & dimpled niobium interlayers)



Background / History

The switchtube PRT interest was in reducing or eliminating “braze balls” and subsequent alumina cracking, increasing leakage voltage, V_L , and increasing joint tensile strength using dimples.

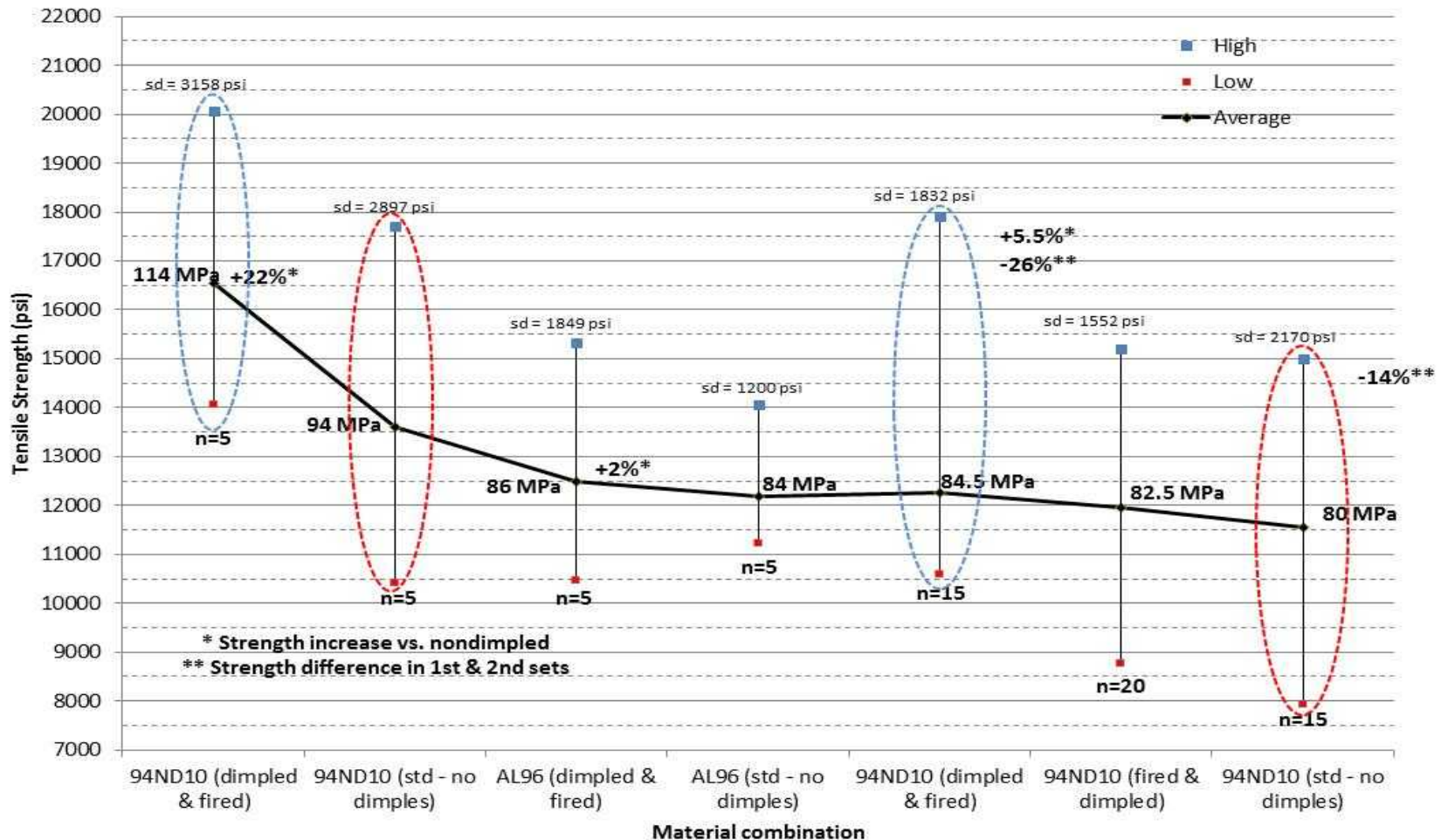
Tensile button assemblies fabricated April and May, 2013.

All groups (6 samples per group, 5 samples tensile tested) are 100% hermetic

AL-96 is a 94% alumina ceramic produced by STC, capable of being extruded. (Evaluated for possible cost savings.)

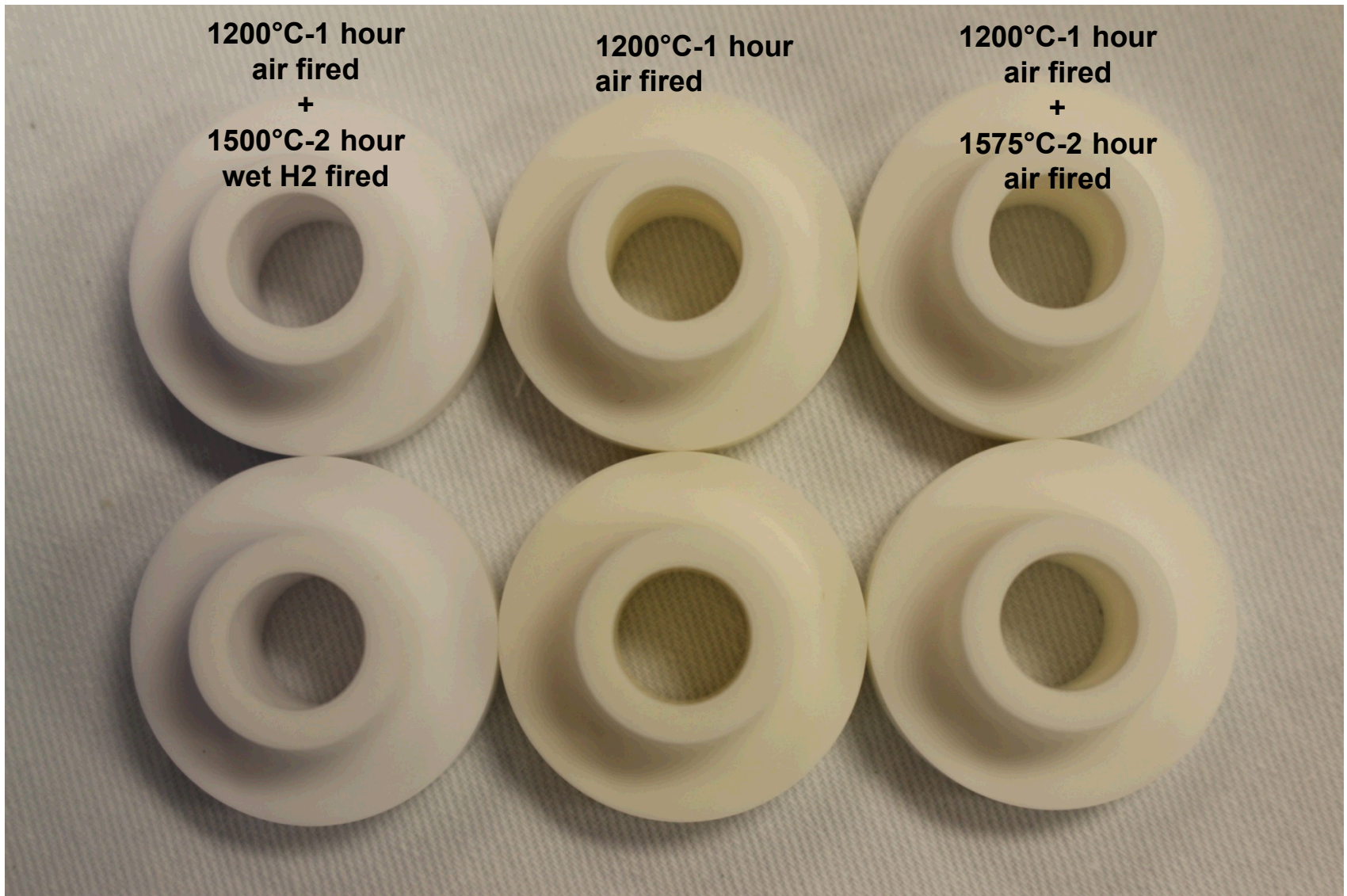
Historical Data: ASTM F-19 Tensile Buttons

Tensile Strength: AL96 vs. 94ND10 Nicoro+2%Ti braze filler metal (std & dimpled niobium interlayers)



Discussion

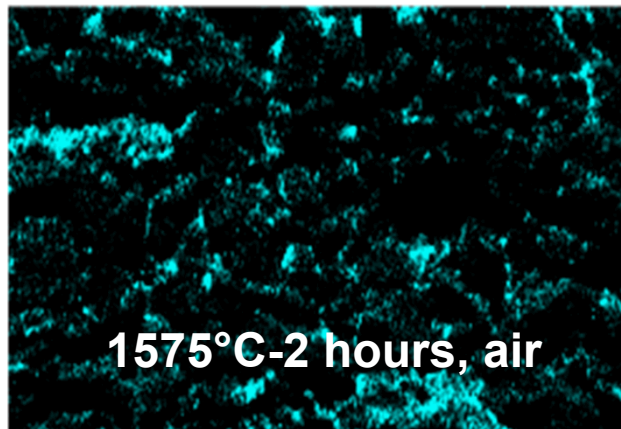
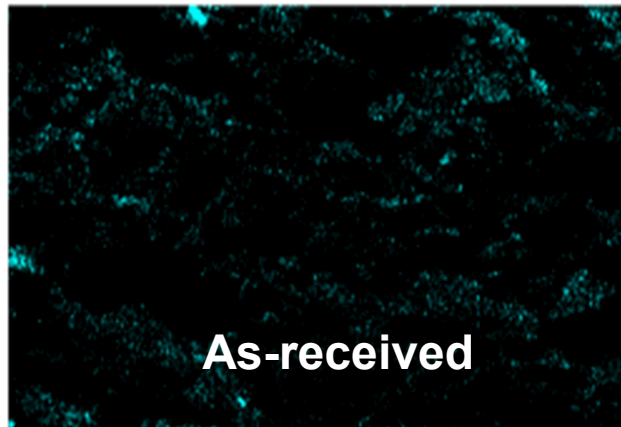
94ND10 tensile buttons



Discussion

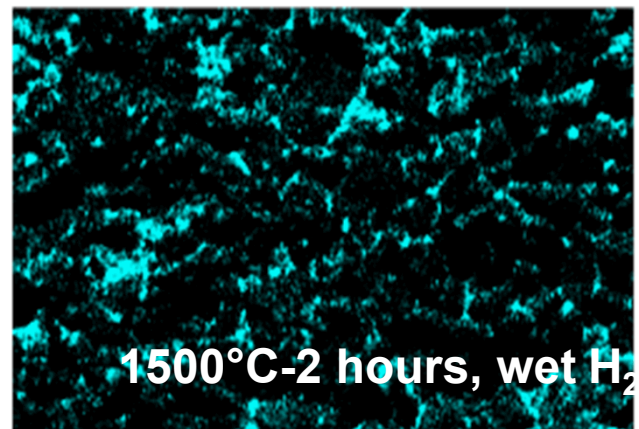
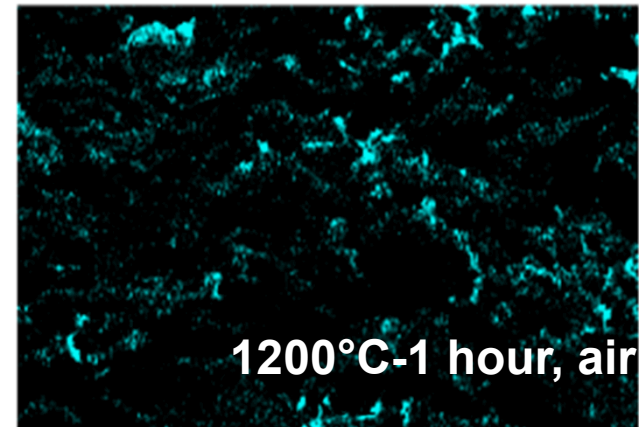
Ceramic Firing Differences: EDS Si spectra comparison

Si K α 1



25 μ m

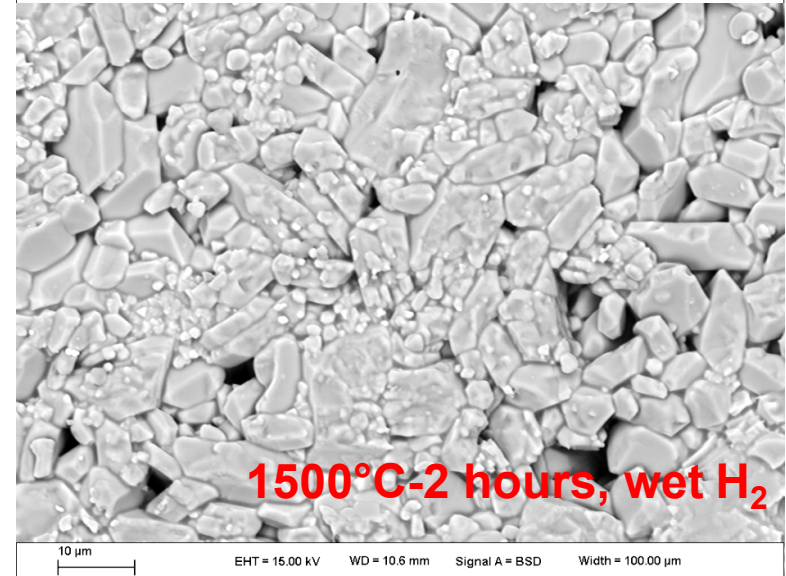
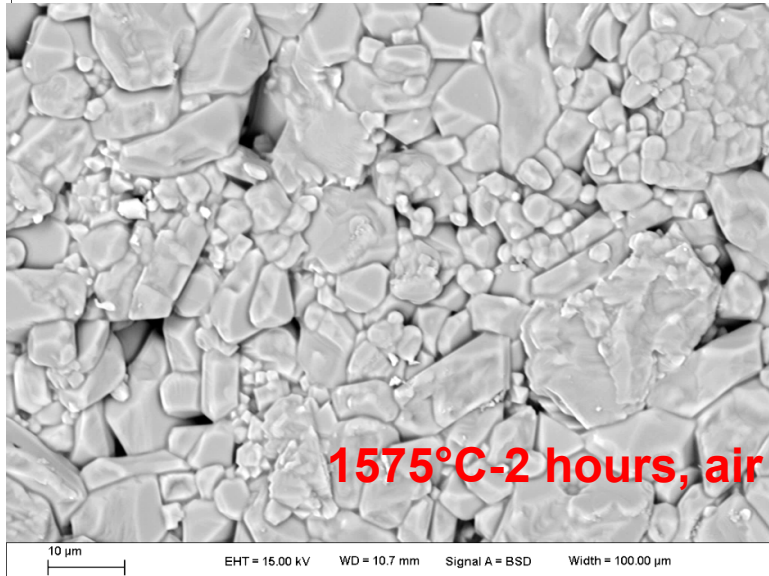
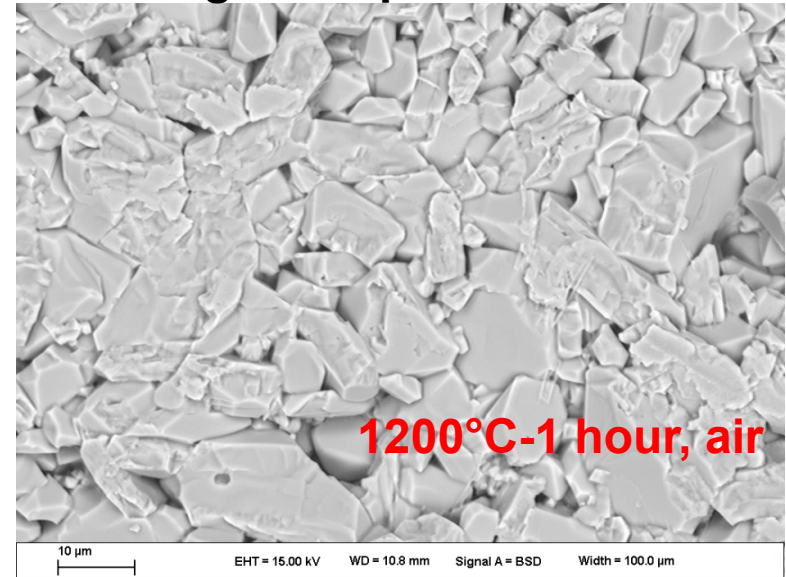
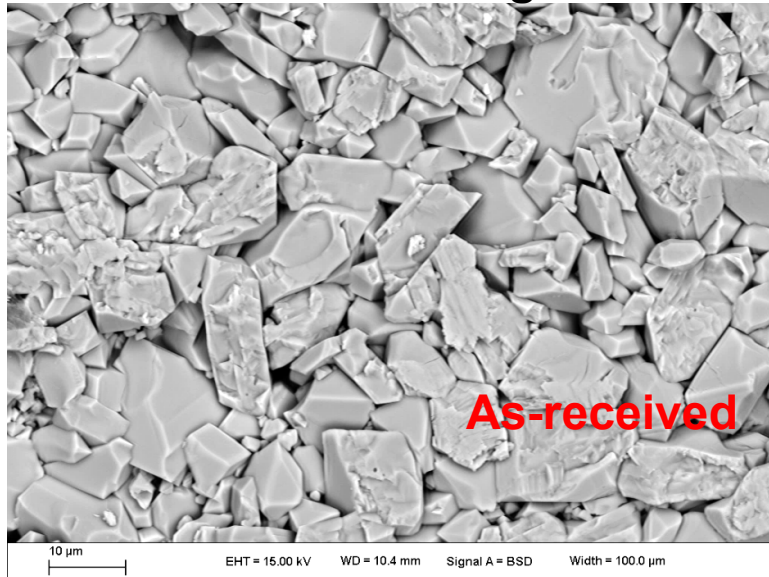
Si K α 1



25 μ m

Discussion

Ceramic Firing Differences: SEM Image Comparison



Conclusions

- This effort has confirmed that it is possible to create active element containing brazing filler metals that can be used to braze both ceramic-ceramic and metal-ceramic assemblies.
- Work to date has focused on filler metals with significantly lower active element additions (0.235 – 0.625 weight percent) than the commercially available counterparts (2 weight percent).
- The reduced active element filler metals performed significantly better (created a more continuous reaction layer on the nonmetal member) when a single reaction layer assemblies were fabricated: i.e. a metal-ceramic joint was made versus a ceramic-ceramic joint.
- Future work will:
 - 1) focus on increasing the amount of active element required to create strong, hermetic assemblies for one and two-reaction layer assemblies.
 - 2) Characterize ceramic firing conditions necessary to reflow glassy phase/matrix without causing devitrification and formation of the undesired Ca-Al-Si-O (anorthite) phase.

Thank you...

Questions?