

ACCELERATED AGING AND TESTING OF THIN FILMS ON LTCC TEST VEHICLES

**P. Sarobol, P. Vianco, K. Peterson, J. Rejent, M. Grazier,
C. Profazi, A. Kilgo, B. Mckenzie, and A. Wolf***

Sandia National Laboratories, Albuquerque, NM

***Kansas City Plant (operated by Honeywell), Kansas City, MO**

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

We are grateful for KCP's support/PDRD funding.

**The Rio Grande Symposium on Advanced Materials
October 6th, 2014.**



**Sandia
National
Laboratories**

P. Sarobol, Org. 1832 SNLA

Motivation

Objective

- ◆ Explore accelerated aging of thin films with presence of Sn-Pb solder on 9K7 low temperature co-fired ceramic (LTCC).

Goal

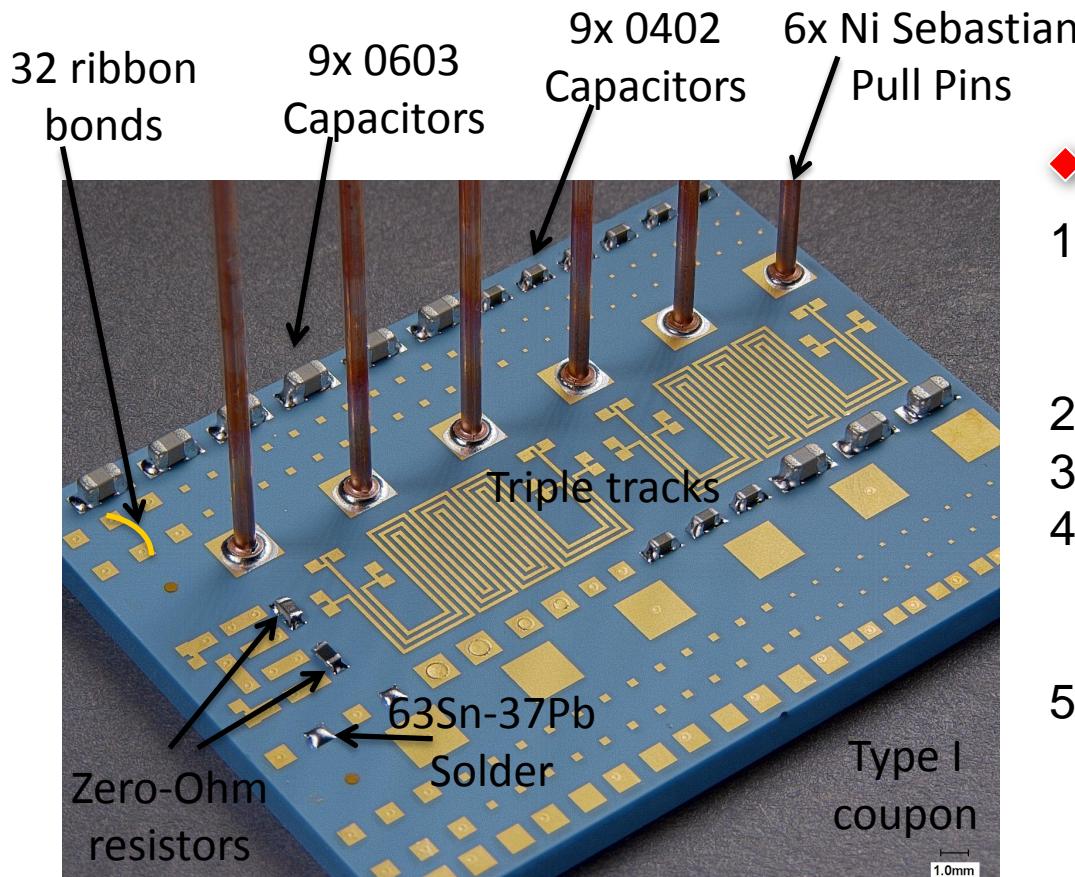
- ◆ Establish a relationship between microstructures, mechanical behaviors and accelerated aging conditions of thin films on 9K7 LTCCs. Special attention paid to:
 - 0.2Ti, 4Cu, 2Pt, 0.375Au
 - 0.2Ti, 1Cu, 2Pt, 0.375Au
 - 4Cu, 2Pt, 0.375Au

Approach

- ◆ Determine wire bonds and soldered joints integrity via mechanical testing (ribbon pulling and pin pulling). Determine solid-state interface reactions of different thin film stacks in multiple accelerated aging conditions.
 - Room Temperature (baseline)
 - 10, 25, and 100 days at 170°C
 - 500 and 1000 Thermal Cycles
 - -55°C to +125°C at 1 cycle/hr per JEDEC22-A104-D

Approach

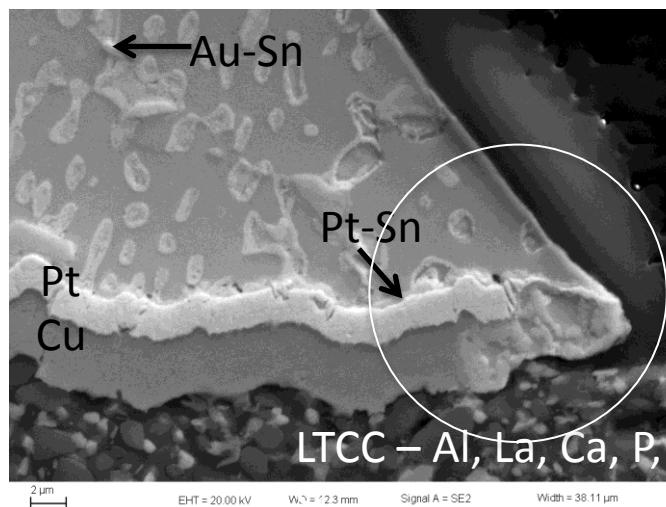
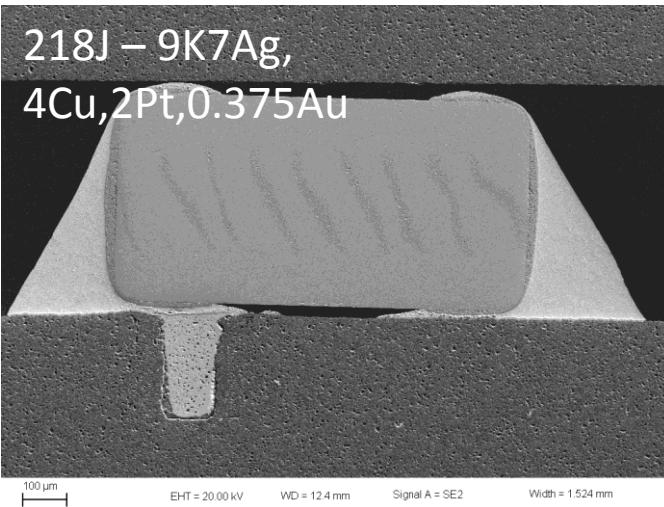
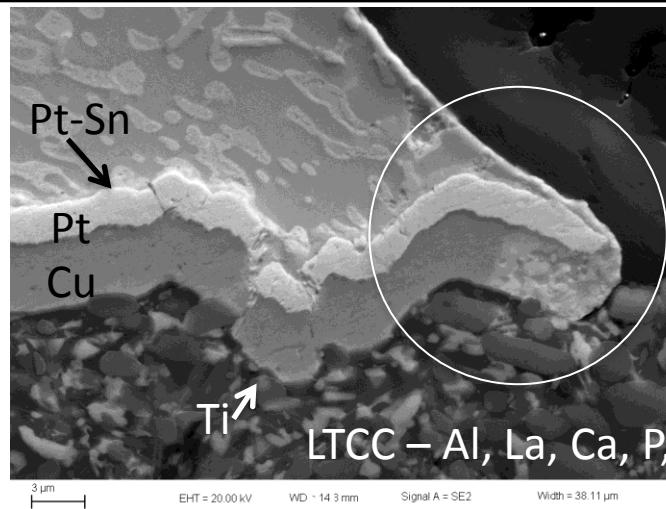
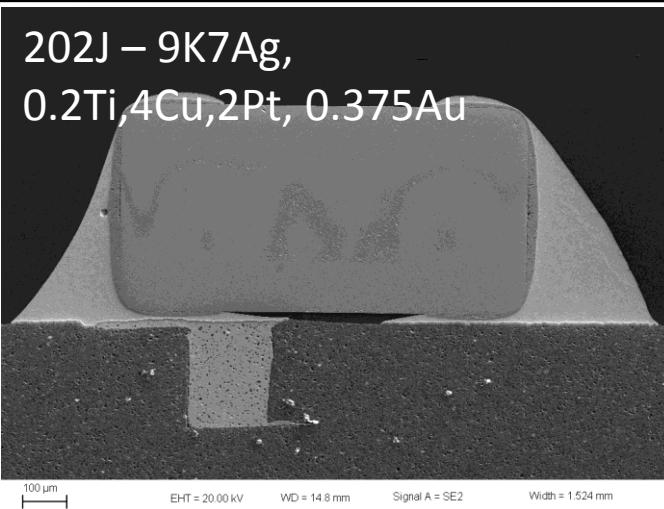
- ◆ The long-term reliability of the LTCC is determined by potential degradation modes resulting from solid-state interface reactions (thin films - solder) that occur at elevated temperature environments.



◆ Testing and Analysis

1. Mechanical testing:
 - a) Ribbon pull
 - b) Pin pull
2. Evaluate failure modes.
3. Cross section aged samples.
4. Use the SEM images to measure the thicknesses of the Ti, Cu, Pt, and IMC layers.
5. Use SEM/EDS to determine inter-diffusion and elements in IMC layers.

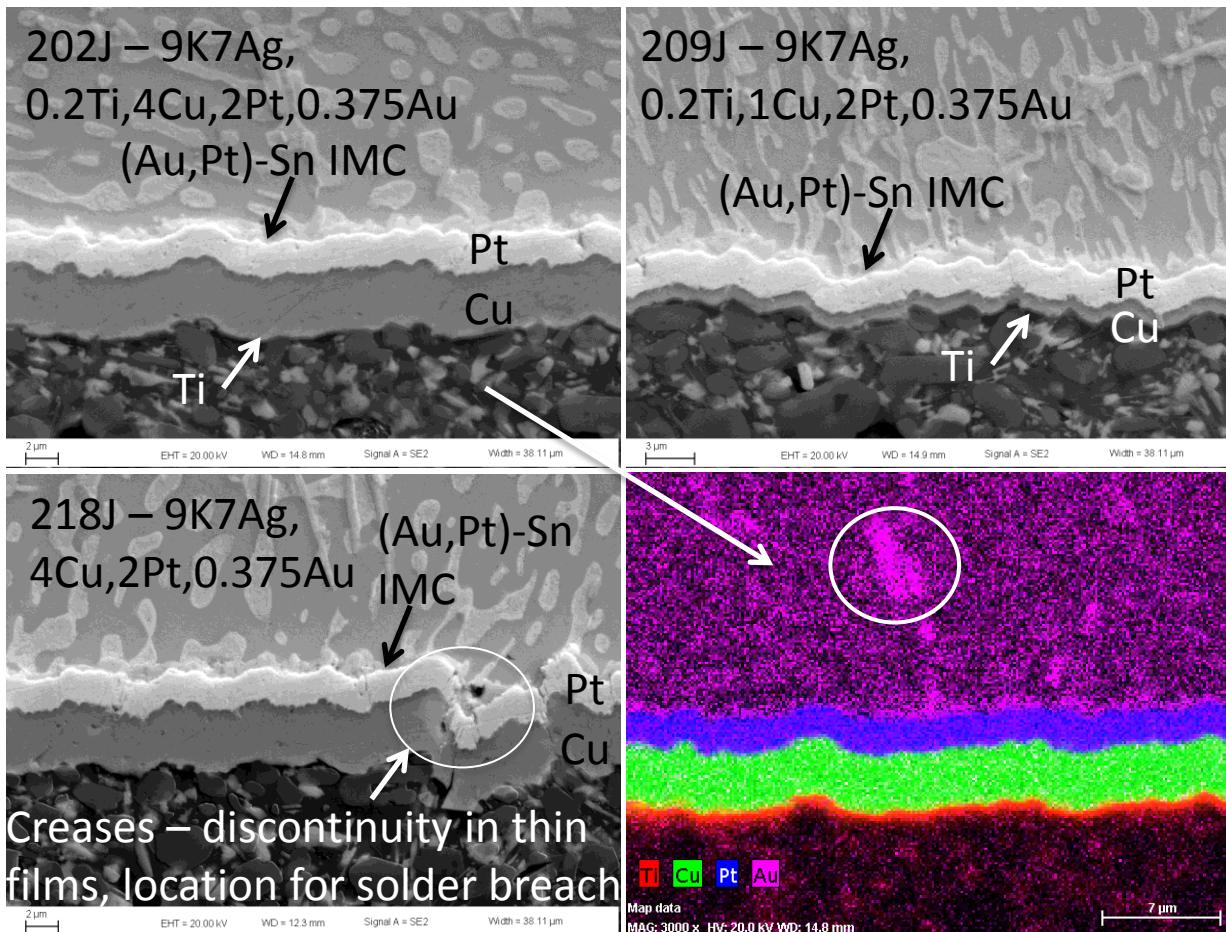
RT Baseline



Solder wetted pad corners during fabrication and continues to react with thin film layers.
Without Ti adhesion layer, solder can undermine the Cu layer.

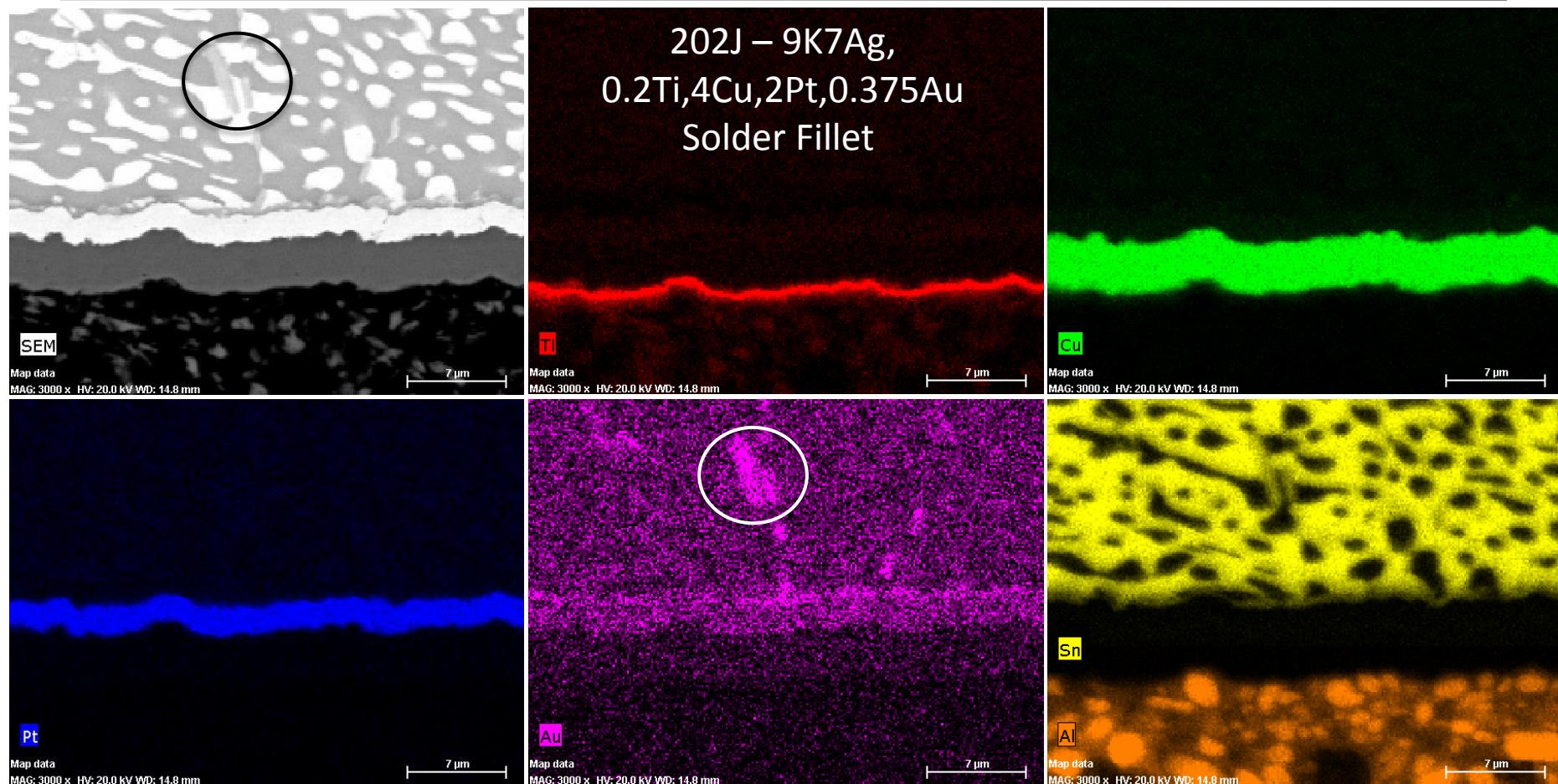
These accelerate aging degradation.

RT Baseline



- Images taken at the solder fillet. Dispersed Pb-rich particles & Au-Sn IMC laths in Sn-Pb solder.
- 9K7 surface roughness provides creases in the film stacks.
- Substrate surface topography can cause **thinned regions** and other **discontinuity defects** in the films *that accelerate aging degradation*.

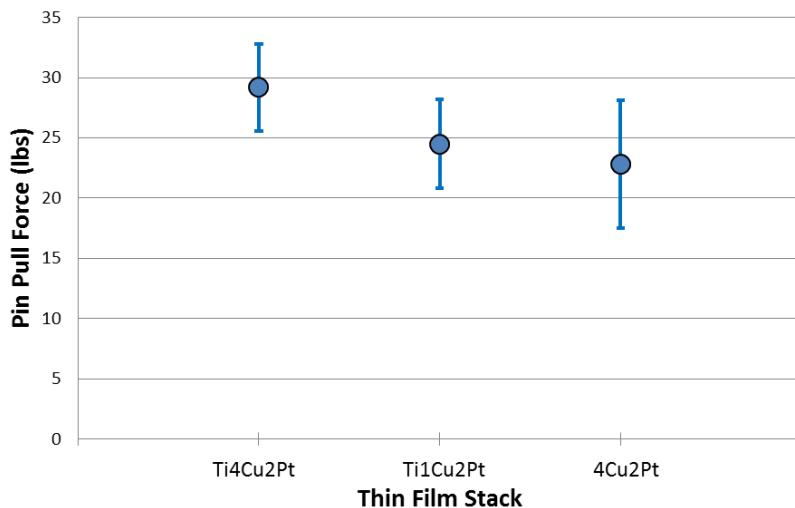
RT Baseline



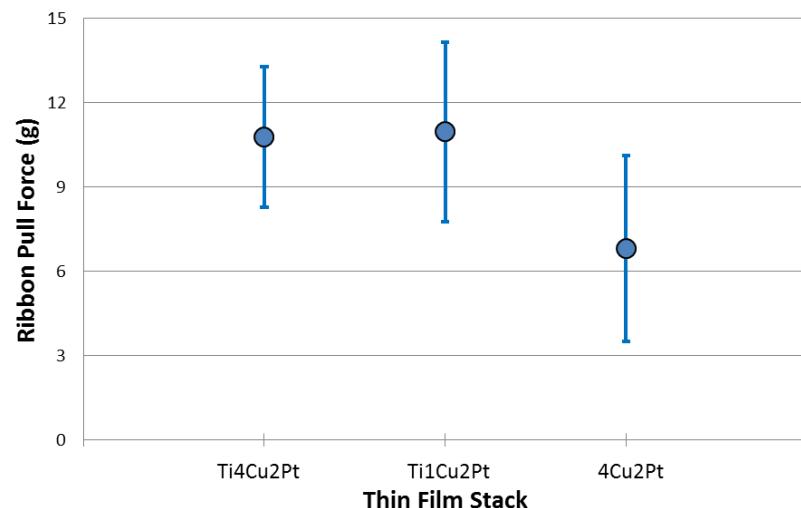
- Au-Sn IMCs found in Sn-Pb solder fillet. Very thin Au-Sn IMC layer above the Pt layer.
- Discrete layers (LTCC → Solder) ~0.2um Ti, ~0.4um Cu, ~0.2um Pt, and ~0.5um Au-Pt-Sn IMC.
- Au and Pt peaks almost overlap.

RT Baseline – Pin/Ribbon Pull Forces

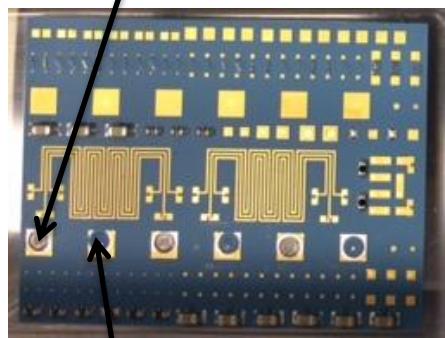
Pin Pull Forces, Room Temp Aging



Ribbon Pull Forces, Room Temp Aging



Solder Failure



Thin film/LTCC separation

Pin Pull

- With Ti → failed in solders
- Without Ti → thin film/LTCC separation

Ribbon Pull

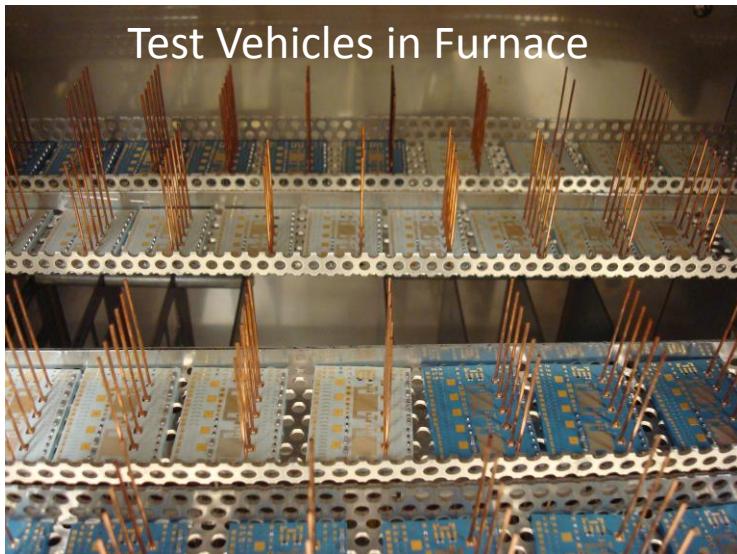
- All samples failed in the ribbons.

Remarks

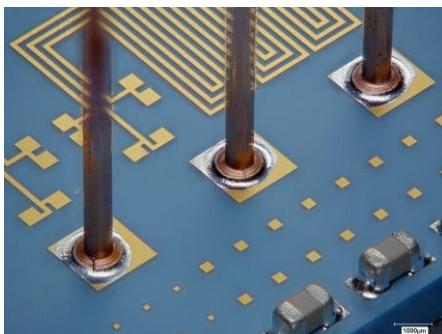
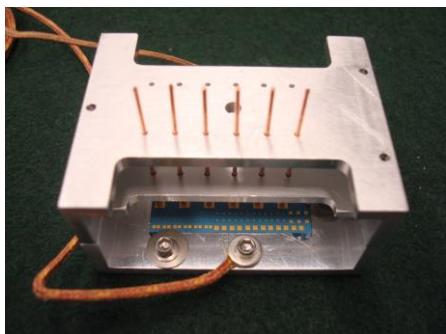
- Ti layer needed for higher pin pull and ribbon pull forces.

LTCC Solid-State Aging

- ◆ The long-term reliability of the LTCC is determined by potential degradation modes resulting from solid-state interface reactions that occur at elevated temperature environments.

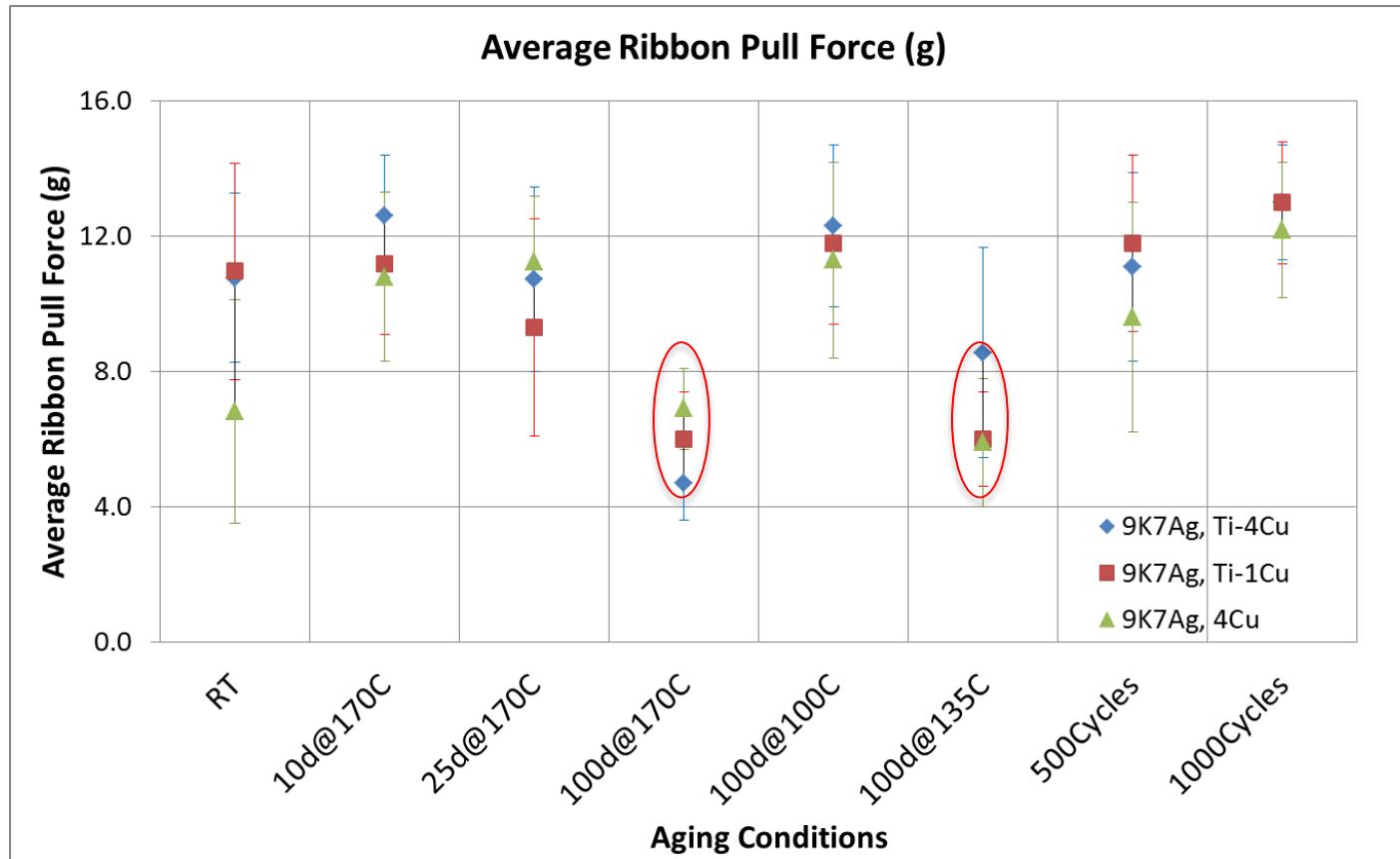


- ◆ Examine the following samples in detail:
 - 0.2Ti, 4Cu, 2Pt, 0.375Au
 - 0.2Ti, 1Cu, 2Pt, 0.375Au
 - 4Cu, 2Pt, 0.375Au
- ◆ Perform mechanical Testing (ribbon & pin pull) for the following aging conditions:
 - 10, 25, 100 days at 170°C
 - 500 and 1000 thermal cycles
- ◆ Examine microstructure (solder/thin film interaction) of the selected samples.



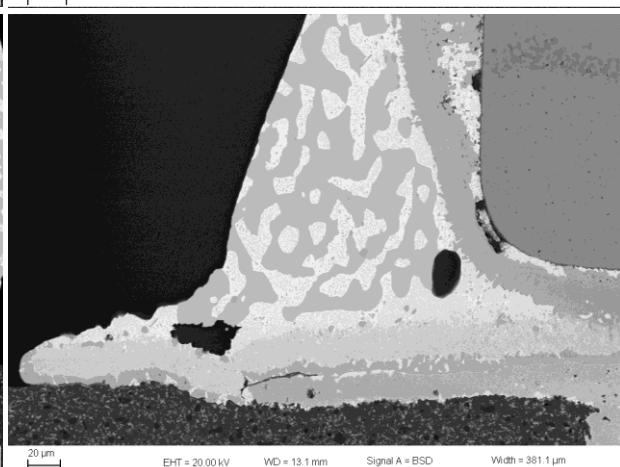
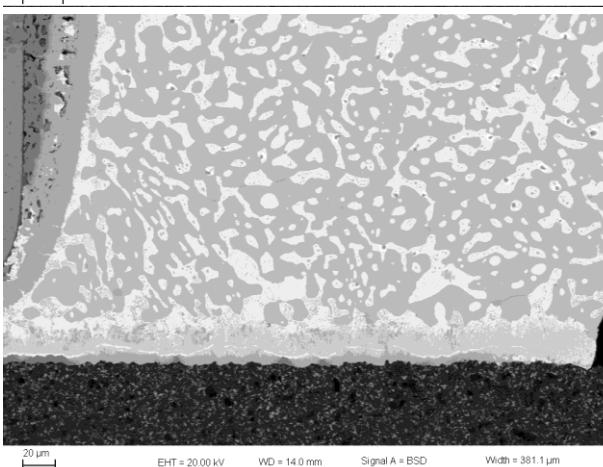
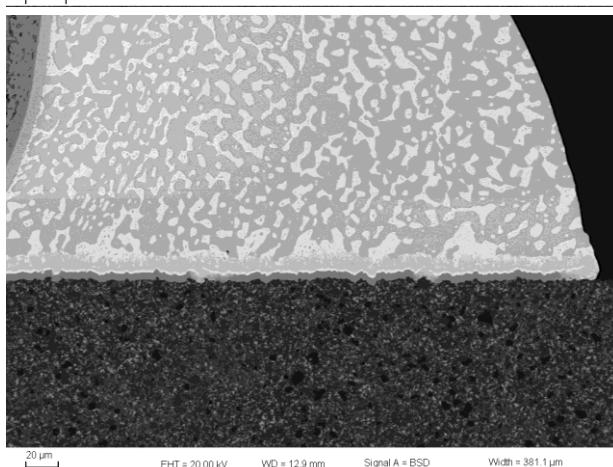
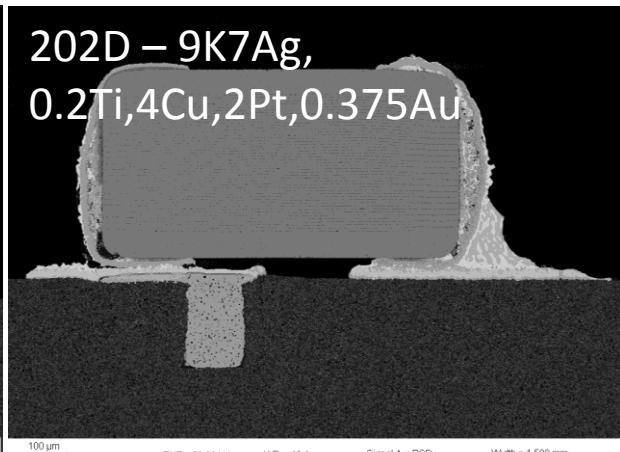
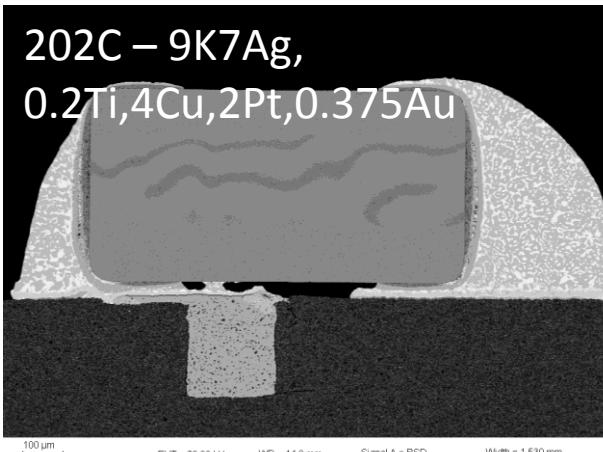
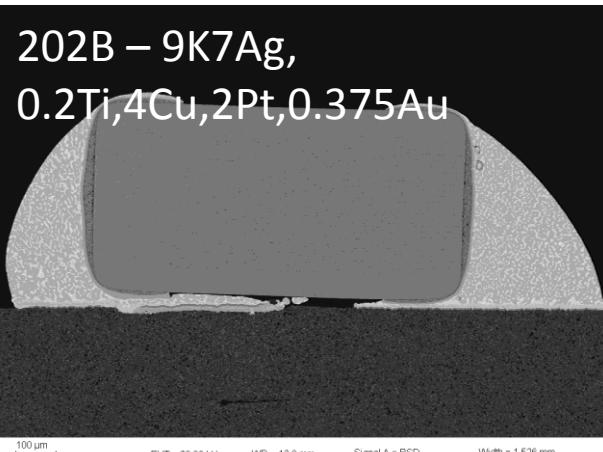
LTCC Solid-State Aging – Ribbon Pull Forces

◆ Average Ribbon Pull Forces



- All failures were in the ribbons.
- Annealed ribbons have lower strengths (100days @ 135°C and 170°C).

LTCC Solid-State Aging – 10, 25, 100 days at 170°C



10 days

25 days

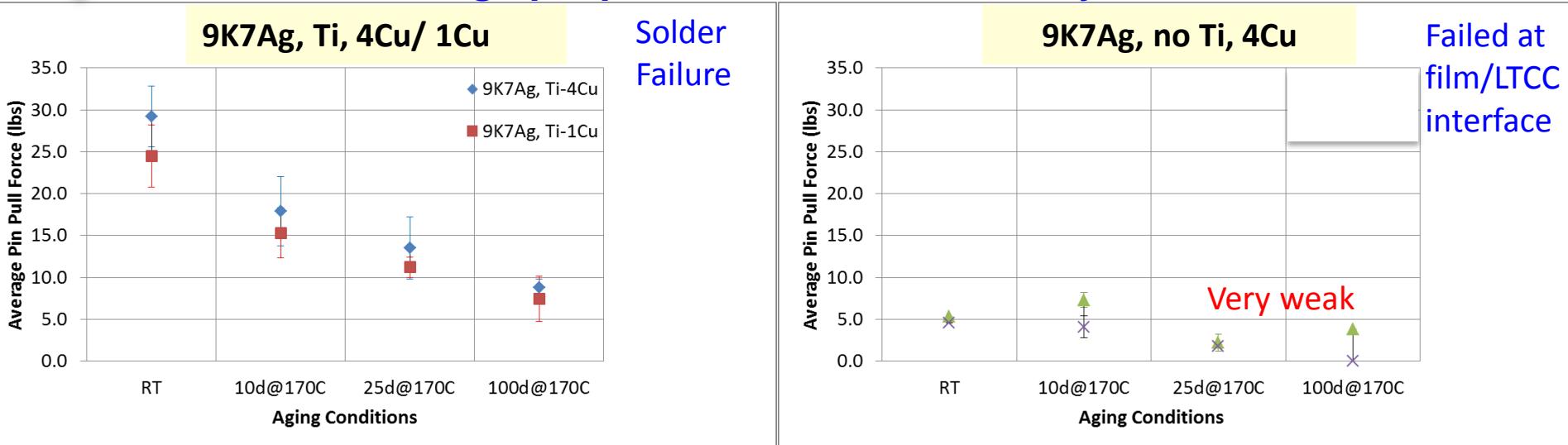
100 days

Solder interaction with thin films → increasing intermetallic compound (IMC) formation.
Pb-rich particles coarsened in the solder fillets.

LTCC Solid-State Aging – 10, 25, 100 days at 170°C



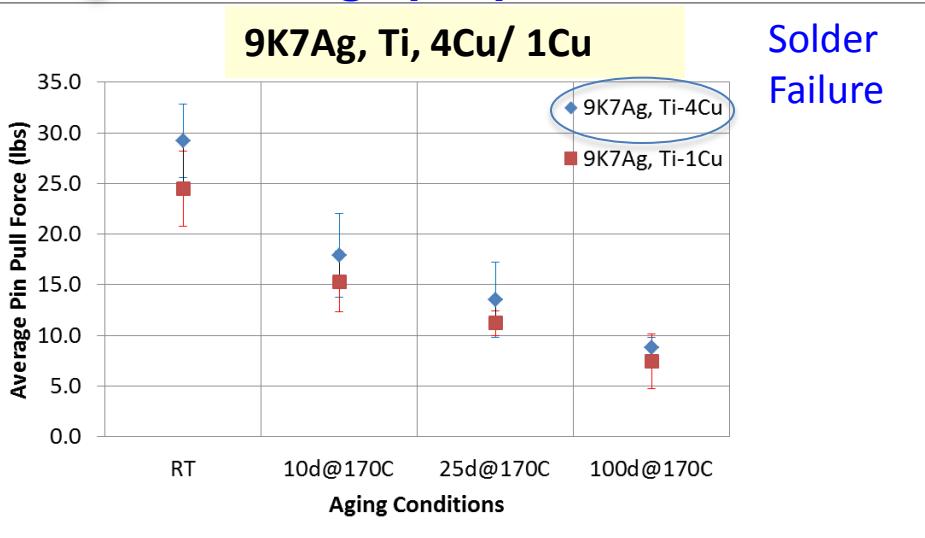
Average pin pull forces, 10, 25, 100 days at 170°C



- Pin pull forces:
 - Little effects from Cu thickness
 - Large effects from presence/absence of Ti
- Pin pull forces decreased from RT → 100d due to increased intermetallic compounds (IMC), brittle phases.
 - 9K7Ag, Ti, Cu dropped > 75%
 - 9K7Ag, no Ti, Cu, poor adhesion, dropped 100% to zero

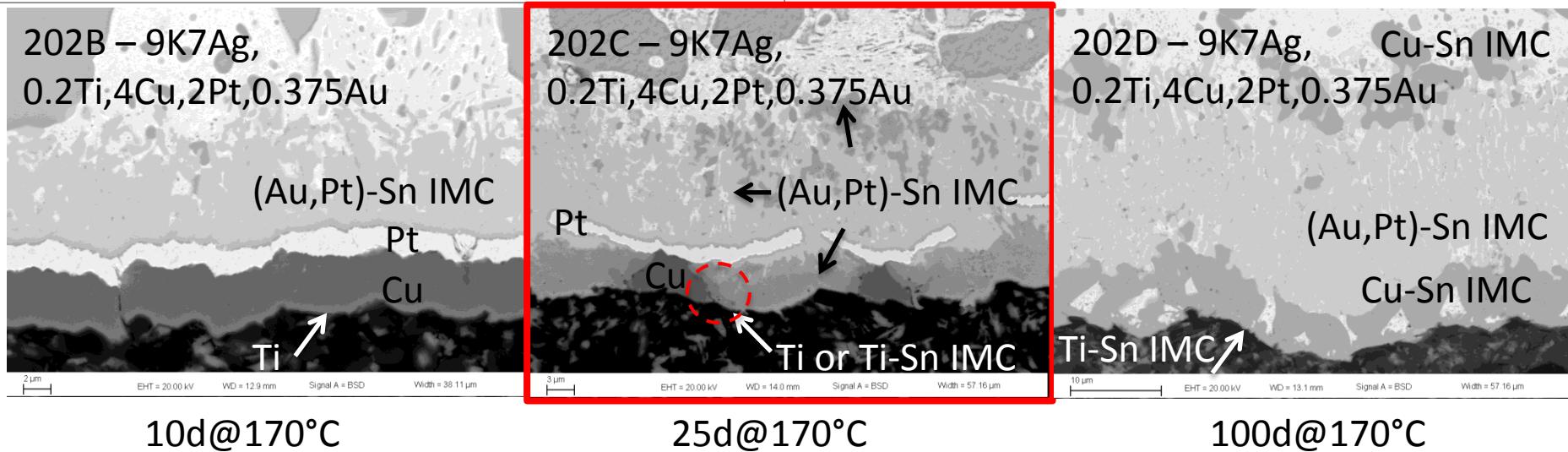
LTCC Solid-State Aging – 10, 25, 100 days at 170°C

Average pin pull forces, 10, 25, 100 days at 170°C, 9K7Ag w/ Ti & 4Cu.

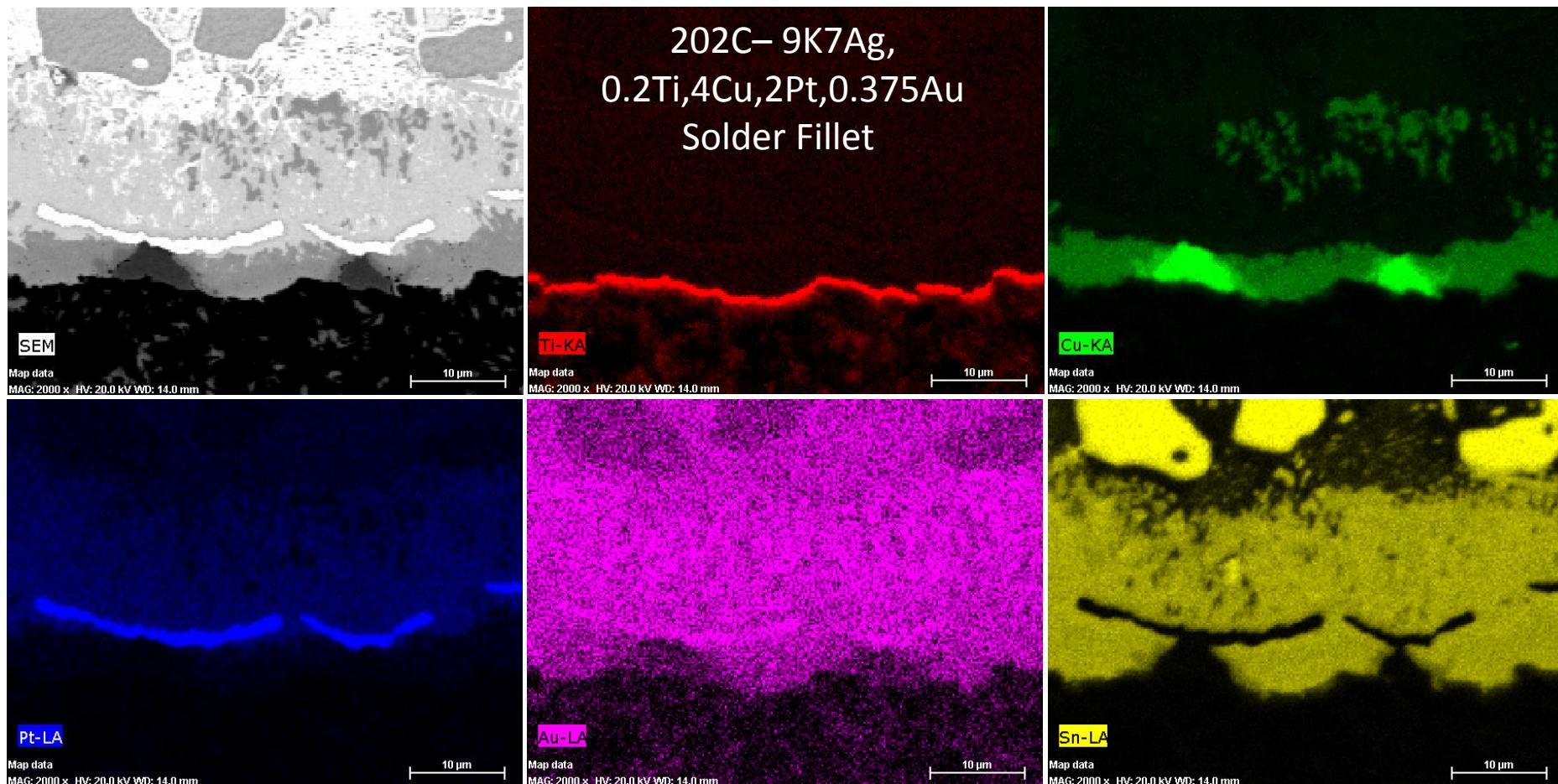


Pin pull forces decreased from RT → 100d.

- **10d**: IMCs formed above Pt layer and Pb-rich particles coarsened.
- **25d**: Solder breached Pt layer and reacted with Cu and Ti layers.
- **100d**: significant IMCs, Solder reacted with all thin film layers. Solder volume decreased → >75% drop in force w/ respect to at RT.



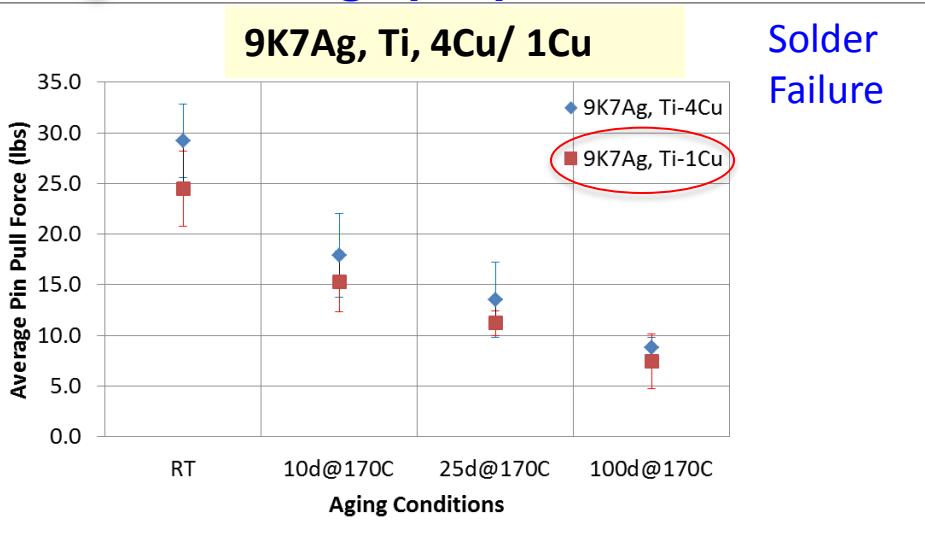
LTCC Solid-State Aging – 25days at 170°C



Solder breached through Pt layer from top.
Sn reacted with all elements- Au, Pt, Cu, Ti-in film stack.
Islands of unreacted Cu in film stack.

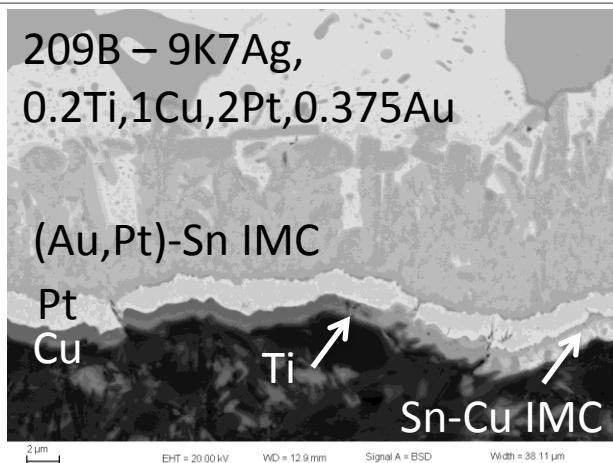
LTCC Solid-State Aging – 10, 25, 100 days at 170°C

Average pin pull forces, 10, 25, 100 days at 170°C, 9K7Ag w/ Ti & 1Cu.

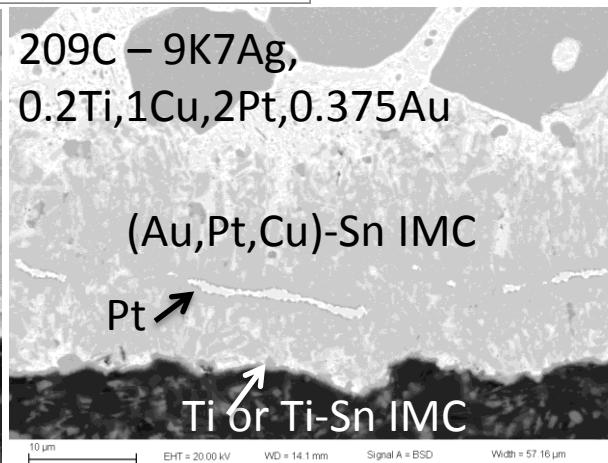


Pin pull forces decreased from RT → 100d.

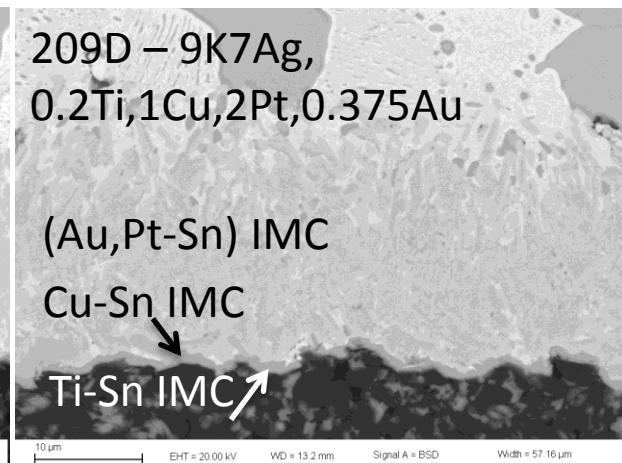
- **10d**: IMCs formed above/below Pt layer. Cu layer was compromised.
- **25d**: Pt and Cu almost completely consumed. Significant IMC in solder.
- **100d**: significant IMCs, Solder reacted with all thin film layers. Solder volume decreased → >75% drop in force w/ respect to at RT.



10d@170°C



25d@170°C

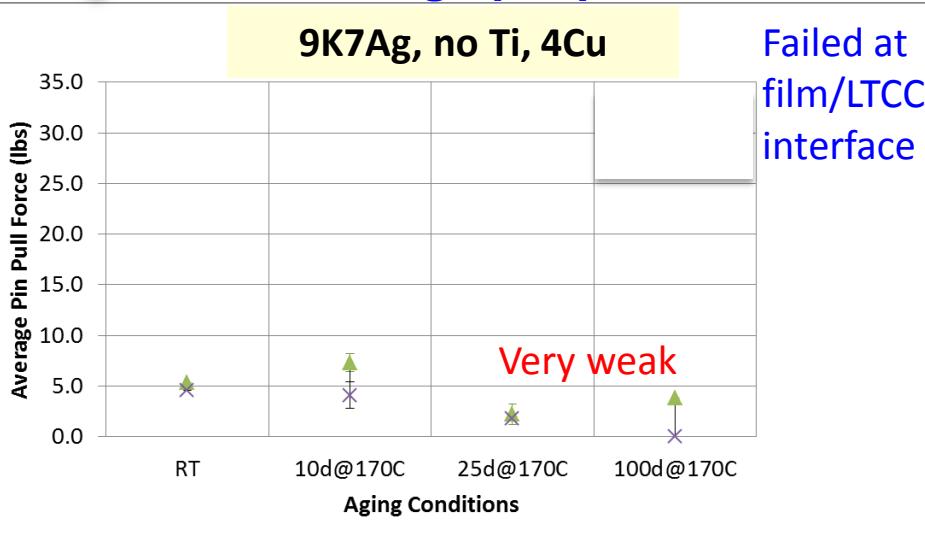


100d@170°C

LTCC Solid-State Aging – 10, 25, 100 days at 170°C

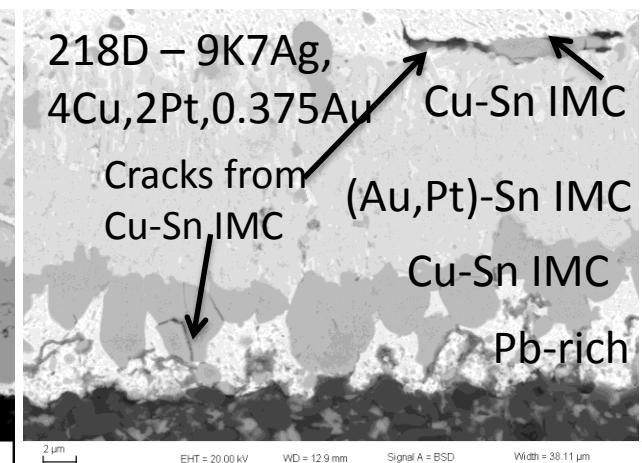
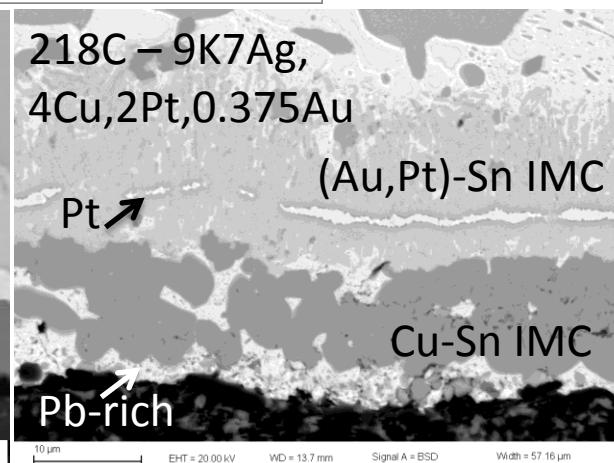
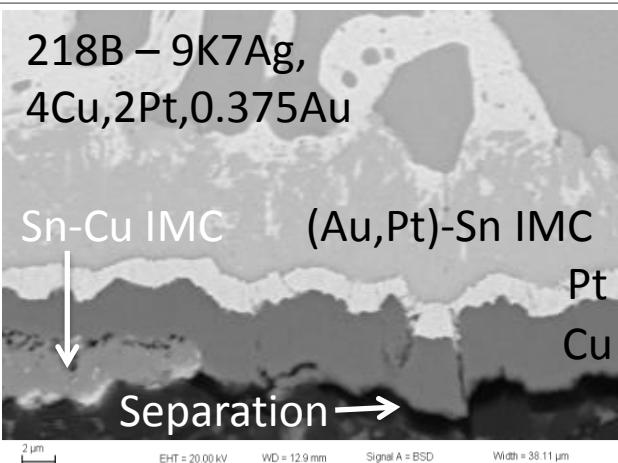


Average pin pull forces, 10, 25, 100 days at 170°C, 9K7Ag 4Cu.



*Separation between film stack/LTCC at RT.
Pin pull forces were weak.

- **10d**: IMCs formed above/below Pt layer. Cu layer was compromised.
- **25d**: Pt and Cu almost completely consumed. Significant IMC in solder.
- **100d**: significant IMCs, Solder reacted with all thin film layers. Cracks found in IMCs.



10d@170°C

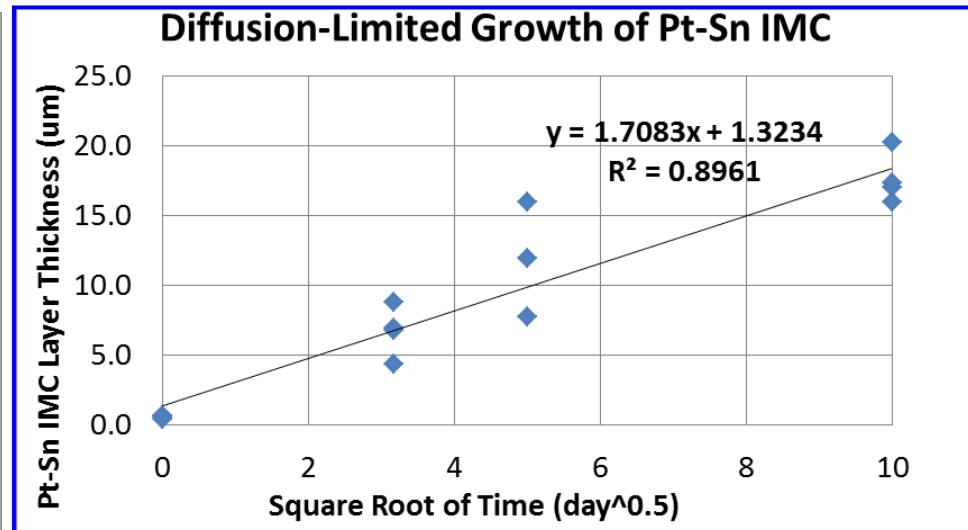
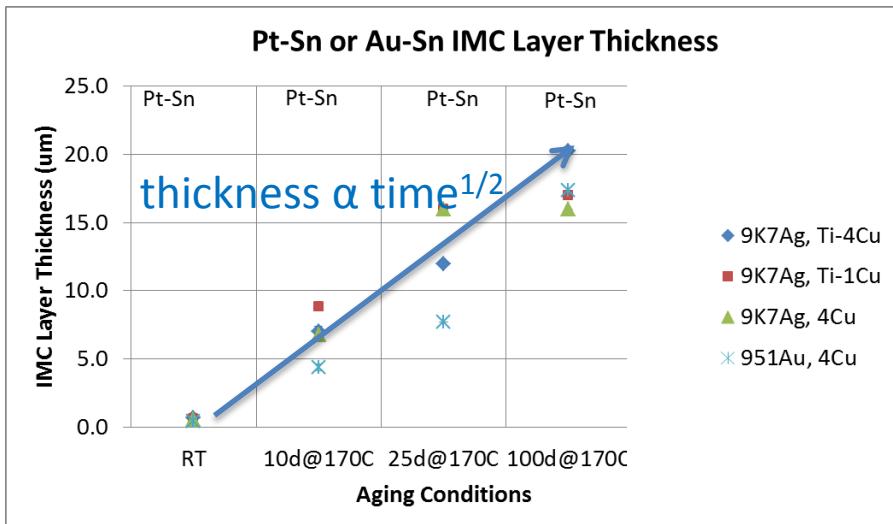
25d@170°C

100d@170°C

LTCC Solid-State Aging – 10, 25, 100 days at 170°C

◆ Thin Film Layer Thicknesses

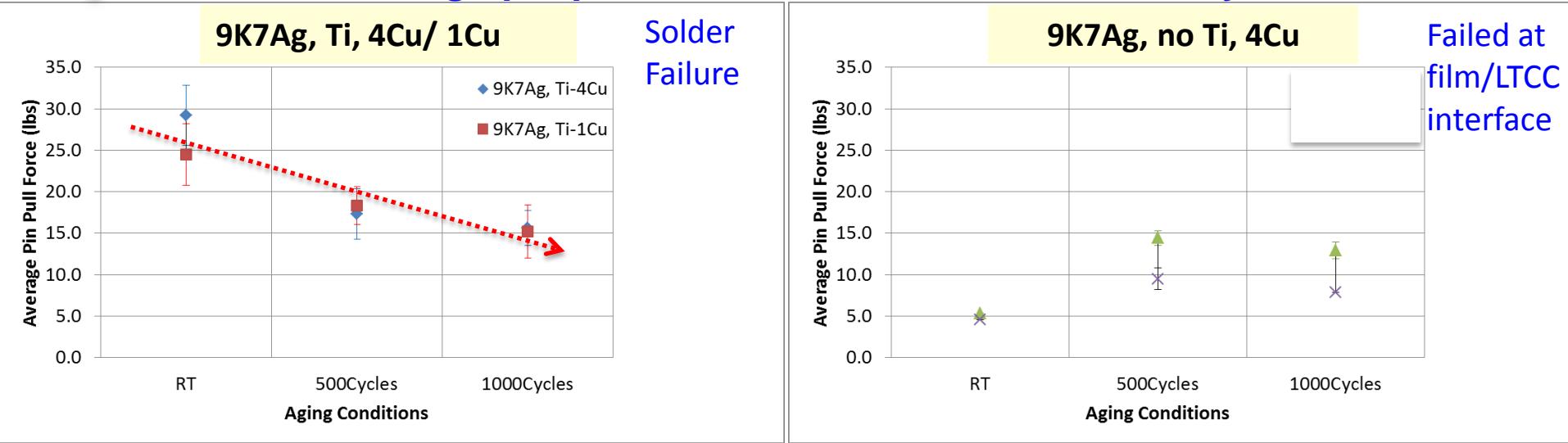
- Pt dissolution, (Au,Pt)-Sn IMC in 170°C aging
- Pt-Sn IMC growth was diffusion limited as thickness \propto time^{1/2}
 - Abundance source of Pt and Sn atoms.
 - As IMC layer thickens, it takes longer time for Pt and Sn to diffuse through IMC layer to react with each other.
- At 100 days, Pt was completely consumed in all samples.



LTCC Solid-State Aging – 500 & 1000 Thermal Cycles



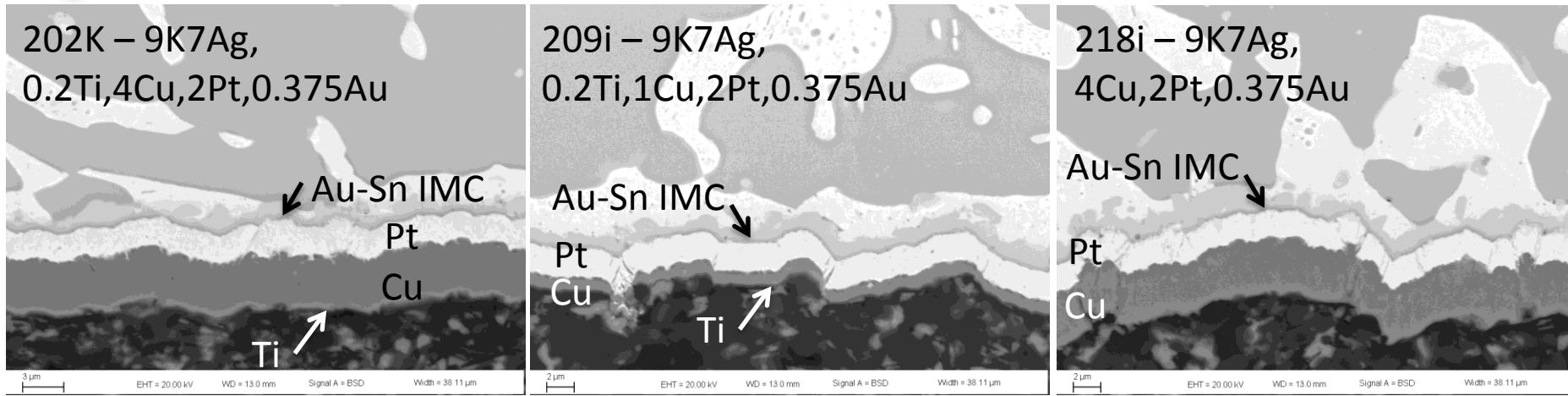
Average pin pull forces, 500 and 1000 thermal cycles.



- Pin pull forces:
 - Little effects from Cu thickness
 - Large effects from presence/absence of Ti
- Thermal cycling induces thermomechanical fatigue (TMF) between the pin, solder, and 9K7 LTCC caused drop in pin pull forces.
- Without Ti, adhesion between thin film and LTCC is poor. Little effects from thermal cycling on pin pull forces were observed.

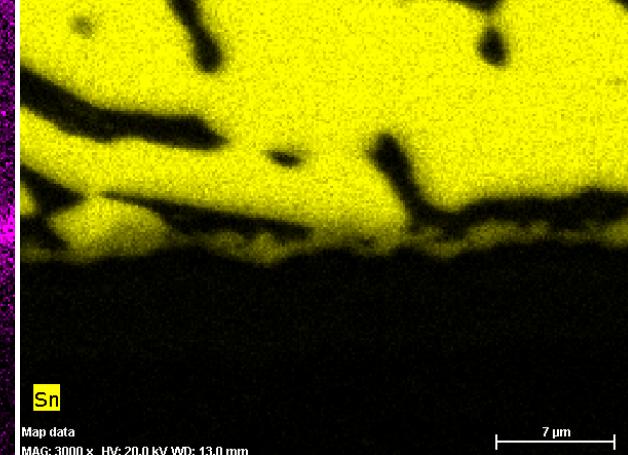
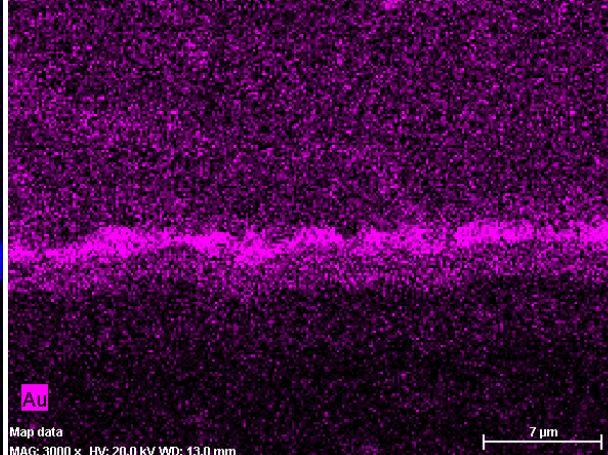
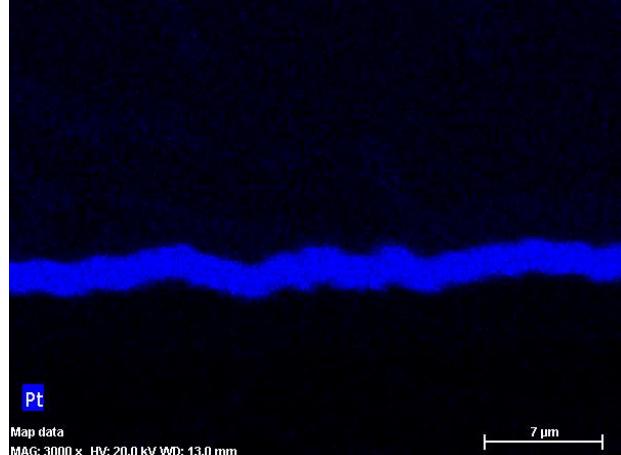
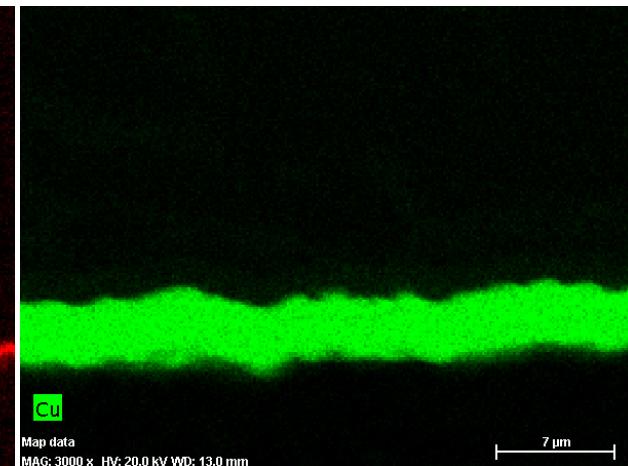
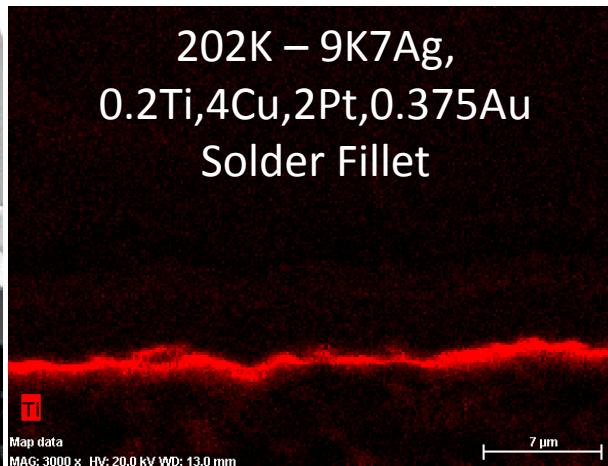
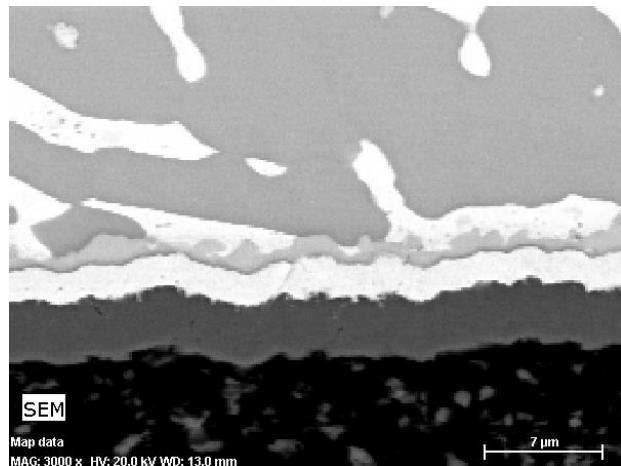
LTCC Solid-State Aging – 500 & 1000 Thermal Cycles

Microstructures at 1000 thermal cycles.



- Microstructural examination of 1000 thermal cycle samples revealed:
 - Pt and Cu layers showed NO signs of dissolution.
 - Au-Sn IMC formed above Pt layer. This IMC layer helped retard solder – Pt interaction and decreased likelihood of solder breaching/interaction with thin film stack.
- Due to low IMC volumes, pin pull forces did not appear to decrease much with thermal cycling.

LTCC Solid-State Aging – 1000 Thermal Cycles



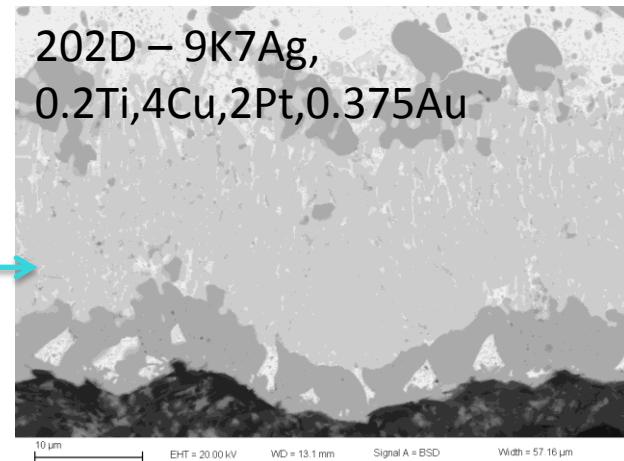
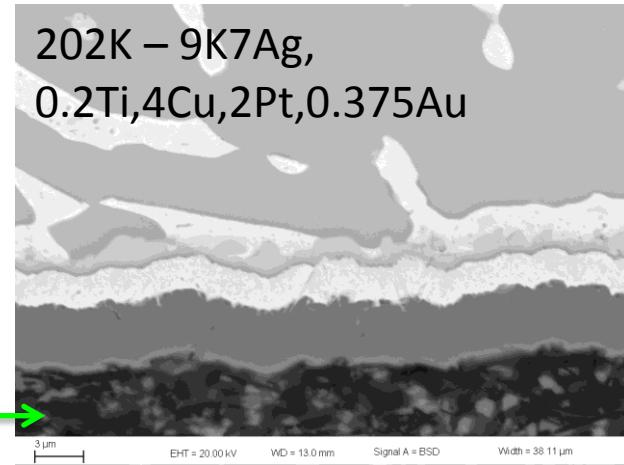
Discrete layers (LTCC → Solder) Ti, ~0.4um Cu, ~0.2um Pt and [~1.7um Au-Sn IMC](#).
Pt and Cu layers appeared to be undisturbed.

Summary: LTCC Solid-State Aging

◆ Observations from Mechanical Testing:

- Ribbon pull test showed failure in ribbons
- Pin pull forces
 - decreased slightly after 1000 cycles.
 - decreased >75% at 100 days at 170°C.
 - with Ti >> without Ti.
 - with Ti, failed in solder.
 - without Ti, Failed at thin film/LTCC interface.

Au-Sn IMC layer formed above Pt layer, providing a barrier layer between solder and Pt. Thermomechanical fatigue from thermal cycling reduced pin pull forces.



Solder breached and interacted completely with all thin film layers, forming large volume of IMCs (brittle phases), degrading the mechanical integrity of solder joins (>75% drop in pin pull forces).

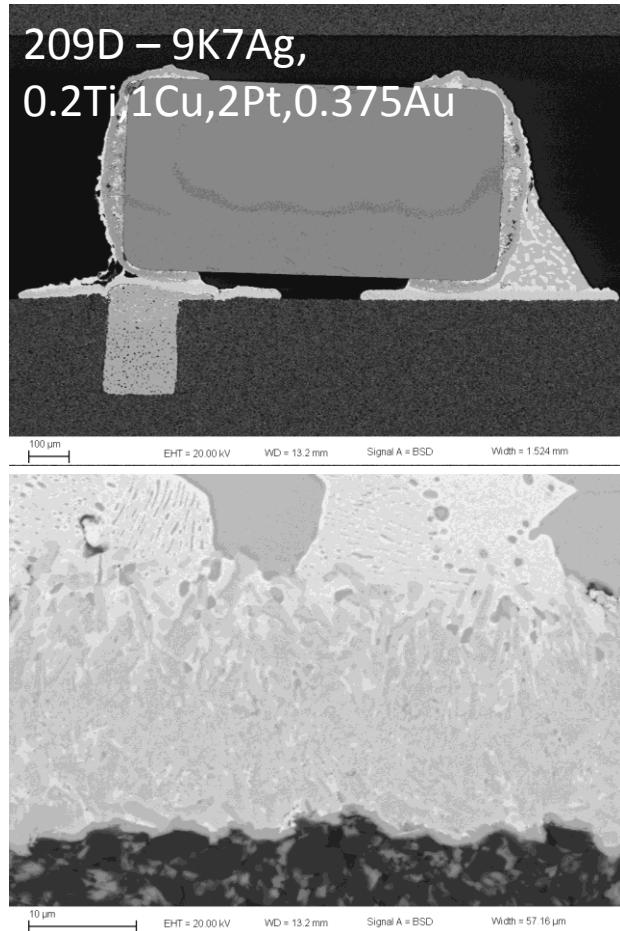
Summary: LTCC Solid-State Aging

◆ Observations from Mechanical Testing:

- Ribbon pull test showed failure in ribbons.
- Pin pull forces
 - decreased slightly after 1000 cycles.
 - decreased >75% at 100 days at 170°C.
 - with Ti >> without Ti.
 - with Ti, failed in solder.
 - without Ti, Failed at thin film/LTCC interface.

◆ Observations from Cross-sections:

- For 170°C – not much solder left at 100 days
 - Significant growth of Pt-Sn IMC with time (diffusion-limited).



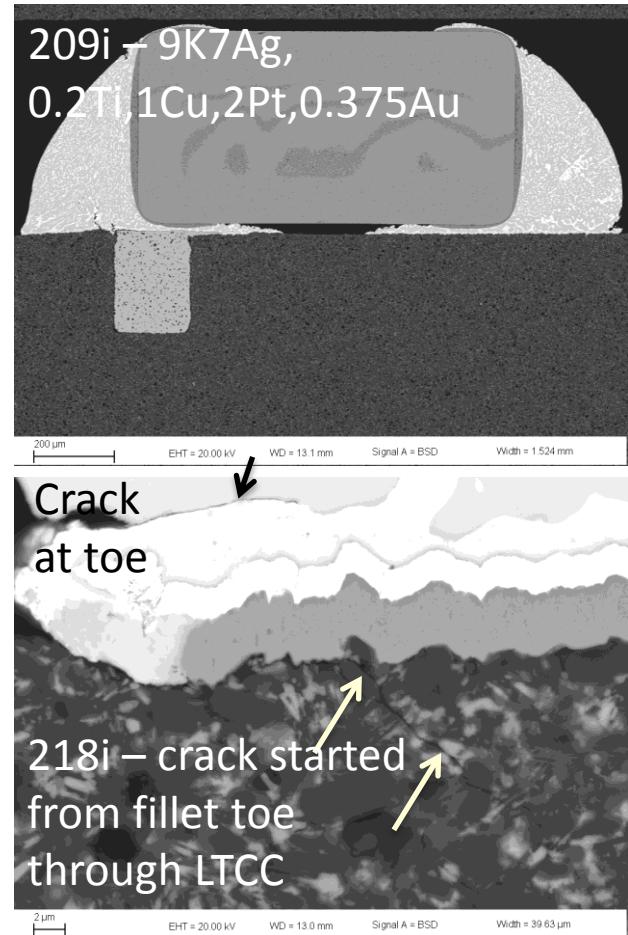
Summary: LTCC Solid-State Aging

◆ Observations from Mechanical Testing:

- Ribbon pull test showed failure in ribbons.
- Pin pull forces
 - decreased slightly after 1000 cycles.
 - decreased >75% at 100 days at 170°C.
 - with Ti >> without Ti.
 - with Ti, failed in solder.
 - without Ti, Failed at thin film/LTCC interface.

◆ Observations from Cross-sections:

- **For 170°C – not much solder left at 100 days**
 - Significant growth of Pt-Sn IMC with time (diffusion-limited).
- **For 1000 thermal cycles.**
 - Long cracks in LTCC under solder fillet
 - Large separation between Cu film and LTCC
 - Discrete Au-Sn IMC form above Pt layer.
 - No significant Pt-Sn IMC formed.



Summary

- ◆ Mechanical testing (ribbon/pin pulling) results showed excellent adhesion between the accelerated-aged thin films with Ti and LTCC in general.
- ◆ Absence of Ti layer contributed to lower pin pull forces.
- ◆ With Ti, pin pull forces on the aged samples were not affected by Cu layer thickness (1 μ m or 4 μ m).

Important Observations

- The thin film/LTCC interface is the “weak link”. Ti layer is important !
- Solder breaching thin film layers started during the soldering process.
- Pt-Sn IMC growth is diffusion limited ($\propto t^{1/2}$) at 170°C.
- Cracks in LTCC under solder fillets occurred in all examined cross-sectioned samples aged at 1000 thermal cycles.

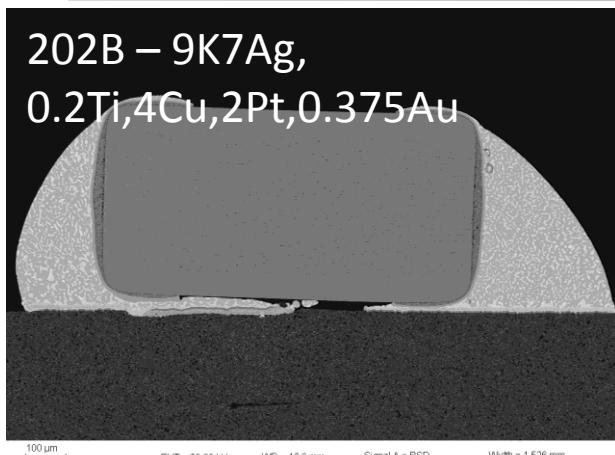


Back up Slides

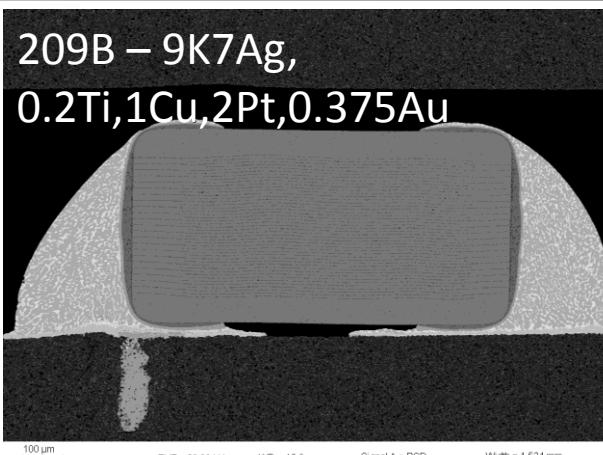
Microstructure Examination

LTCC Solid-State Aging – 10days at 170°C

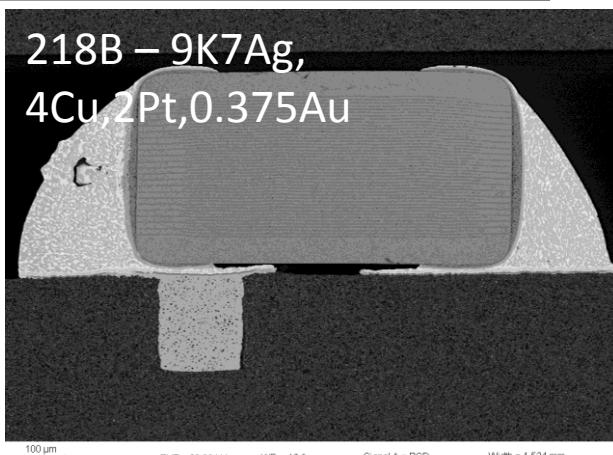
202B – 9K7Ag,
0.2Ti,4Cu,2Pt,0.375Au



209B – 9K7Ag,
0.2Ti,1Cu,2Pt,0.375Au



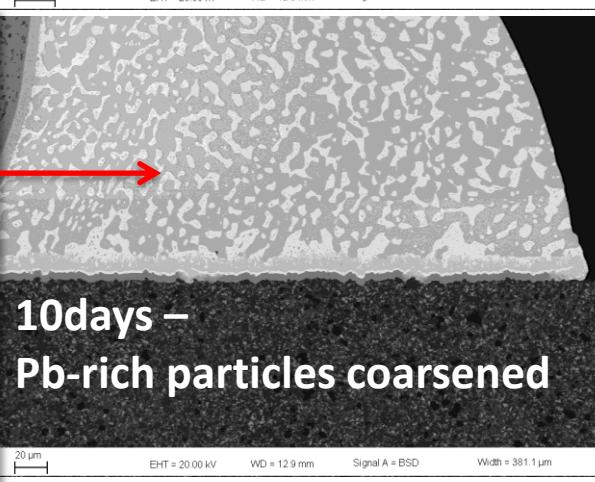
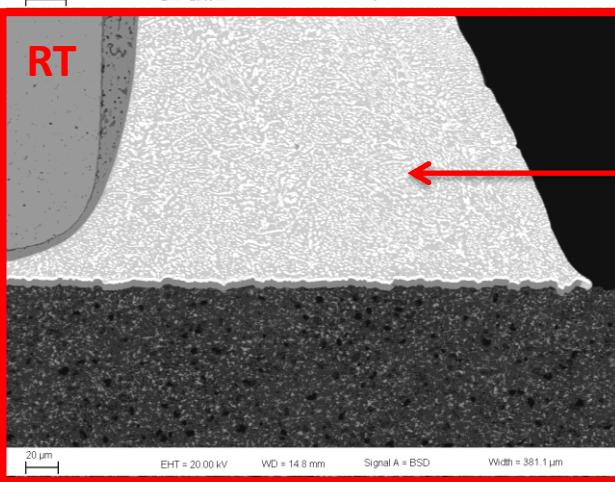
218B – 9K7Ag,
4Cu,2Pt,0.375Au



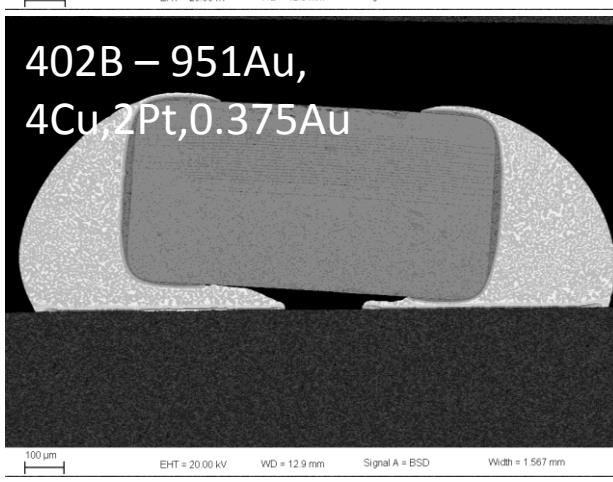
RT



10days –
Pb-rich particles coarsened



402B – 951Au,
4Cu,2Pt,0.375Au



Solder fillets are intact.
Pb-rich particles coarsened.
Pt-Sn IMC layer above Pt layer is prevalent.

Microstructure Examination

LTCC Solid-State Aging – 10days at 170°C

202B – 9K7Ag,
0.2Ti,4Cu,2Pt,0.375Au

(Au,Pt)-Sn IMC
Pt
Cu

Ti

2 μm EHT = 20.00 kV WD = 12.9 mm Signal A = BSD Width = 38.11 μm

209B – 9K7Ag,
0.2Ti,1Cu,2Pt,0.375Au

(Au,Pt)-Sn IMC
Pt
Cu

Ti

Sn-Cu IMC

2 μm EHT = 20.00 kV WD = 12.9 mm Signal A = BSD Width = 38.11 μm

218B – 9K7Ag,
4Cu,2Pt,0.375Au

Sn-Cu IMC (Au,Pt)-Sn IMC
Pt
Cu

Separation →

2 μm EHT = 20.00 kV WD = 12.9 mm Signal A = BSD Width = 38.11 μm

202-Solder breached to LTCC

(Au,Pt)-Sn IMC
(Au,Pt,Cu)-Sn IMC
Pt
Cu

2 μm EHT = 20.00 kV WD = 12.9 mm Signal A = BSD Width = 38.11 μm

209-Solder breached
to cover pad

20 μm EHT = 20.00 kV WD = 12.9 mm Signal A = BSD Width = 285.8 μm

402B – 951Au,
4Cu,2Pt,0.375Au

(Au,Pt)-Sn IMC
Pt
Cu

2 μm EHT = 20.00 kV WD = 12.9 mm Signal A = BSD Width = 38.11 μm

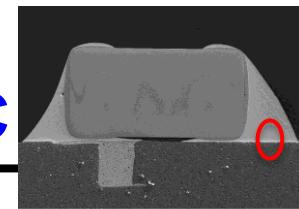
In solder region, prevalent growth of (Au,Pt)-Sn IMCs and Au-Sn IMC laths disappeared?

Top surface of Pt layer showed signs of dissolution (roughened).

Observed solder breached through (creases in) Pt layer, interacted with Cu → Cu-Sn IMC.

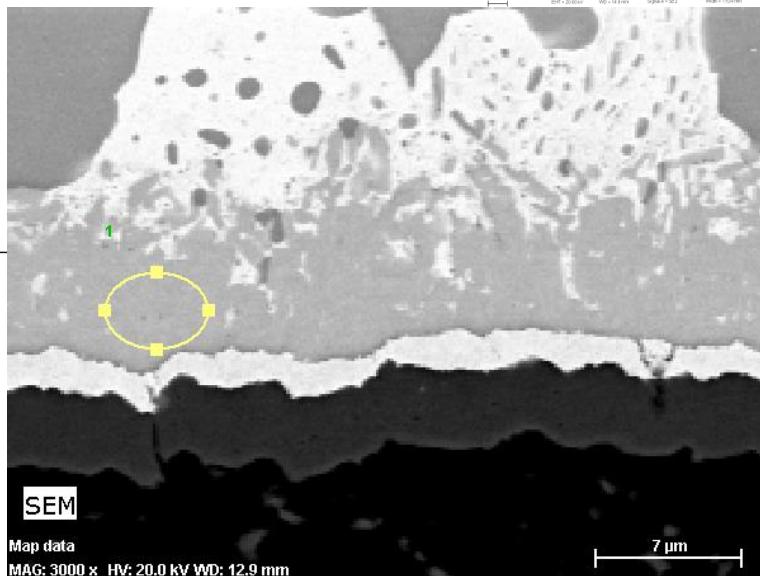
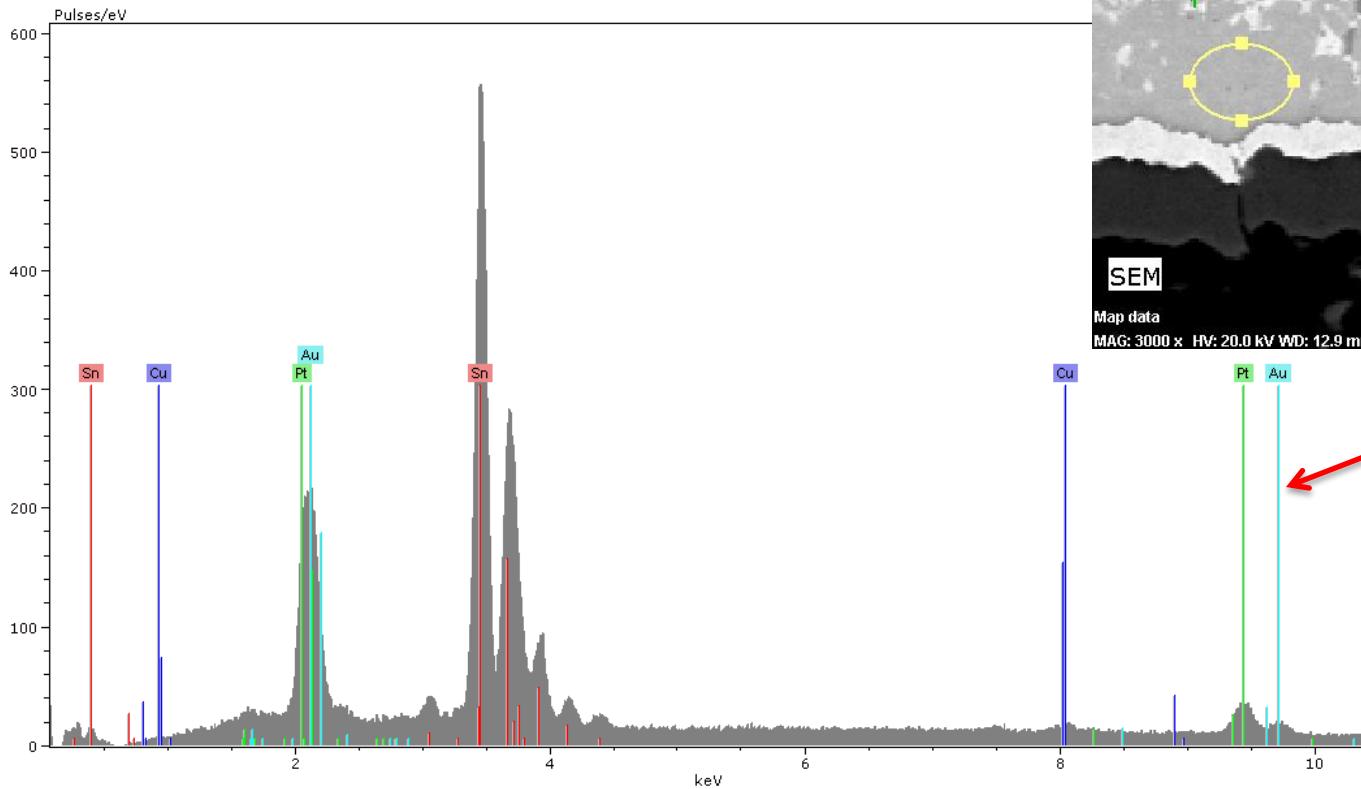
Observed solder breached through cover pad and interacted with via materials.

Microstructure Examination LTCC Solid-State Aging – 10days at 170°C



small-cap_10d_170C_202_C1_map1

EDS spectrum taken within circled area

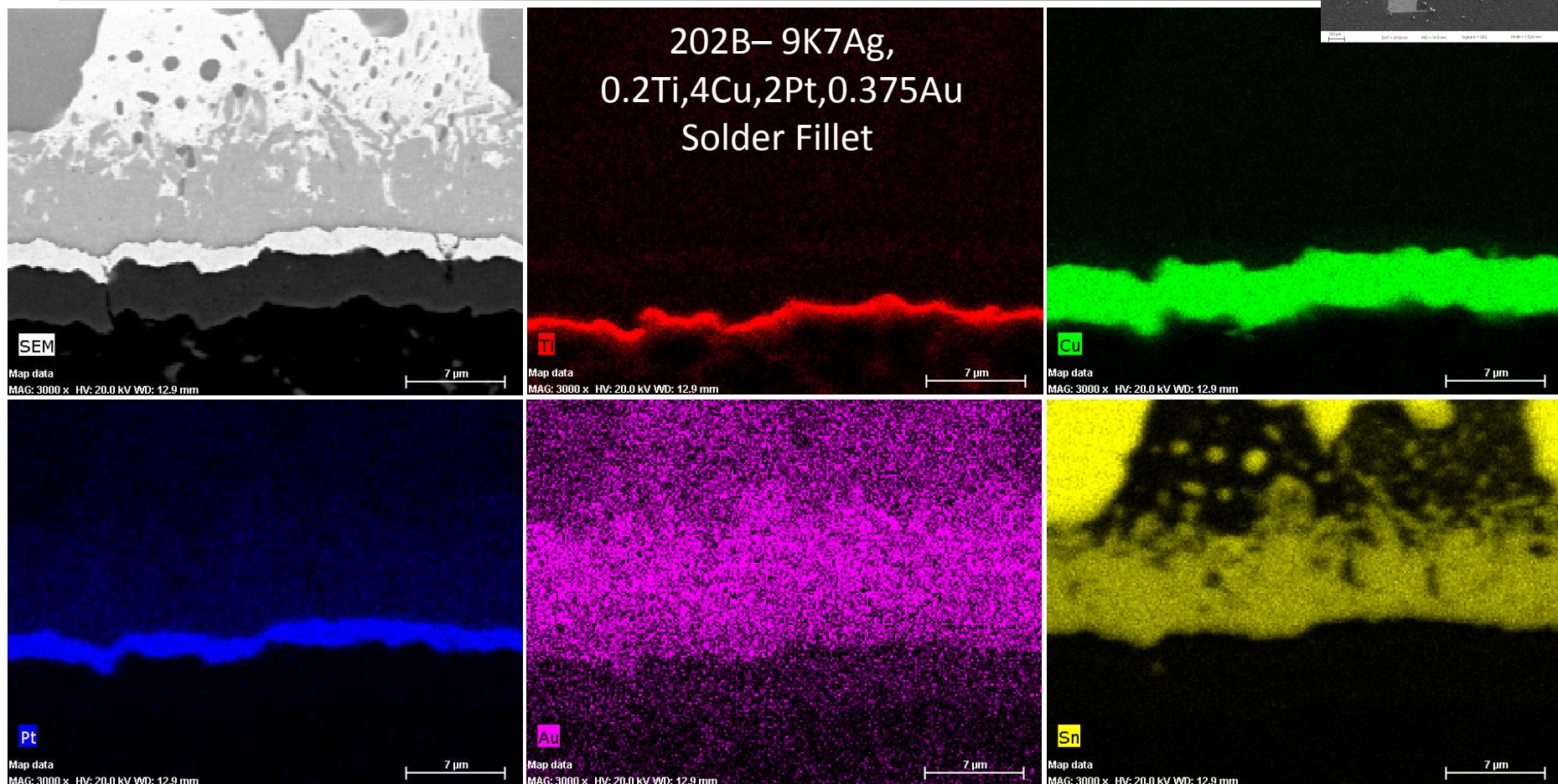


(Au,Pt)-Sn

- Pt-Sn IMC
- Au-Sn IMC
- Au-Pt mix

Microstructure Examination

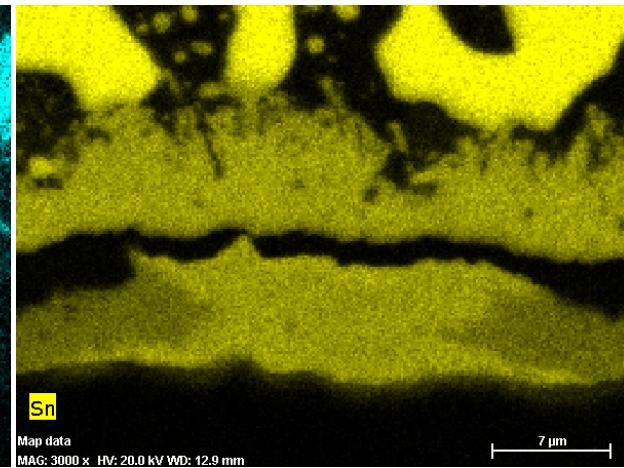
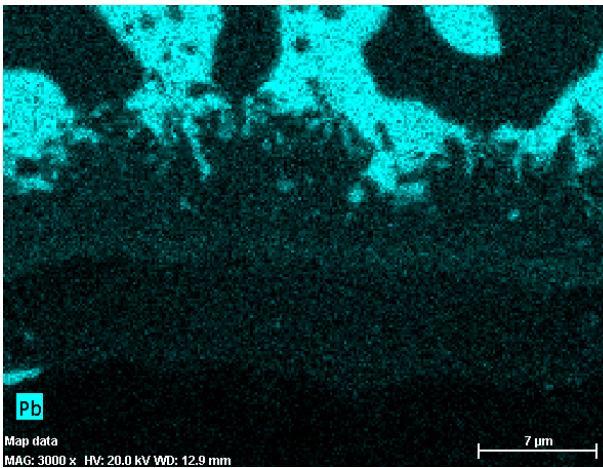
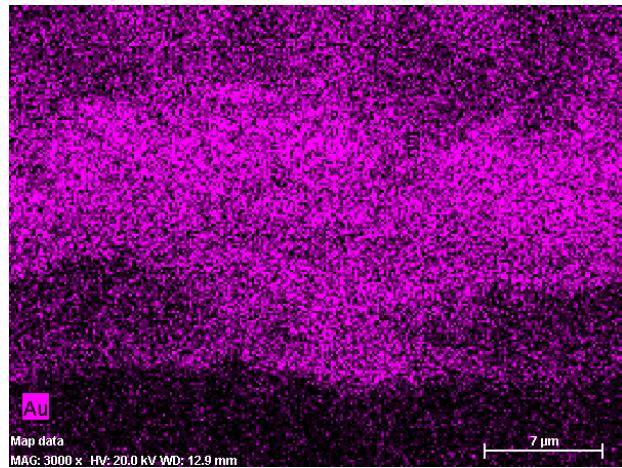
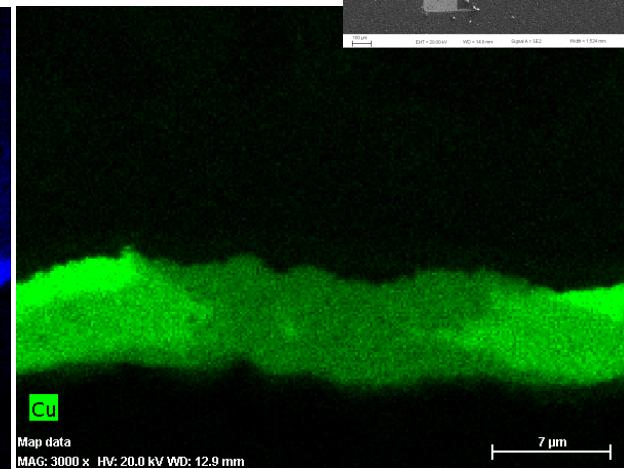
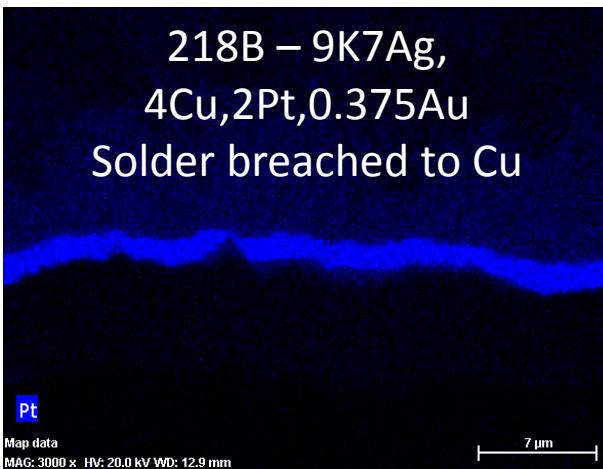
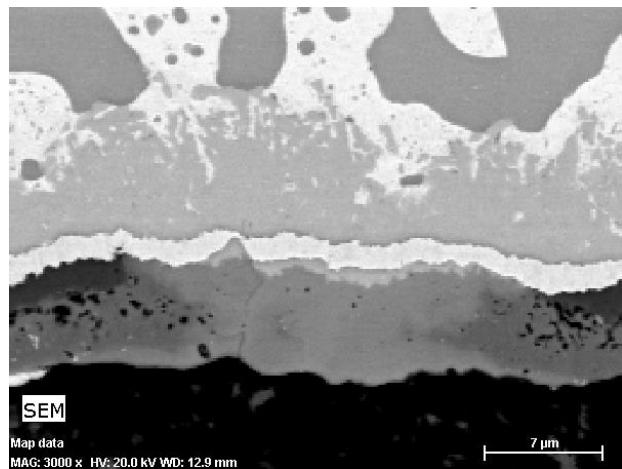
LTCC Solid-State Aging – 10days at 170°C



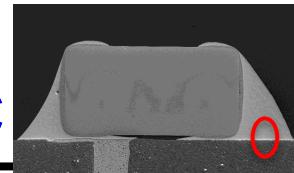
Discrete layers (LTCC → Solder) ~0.2um Ti, ~0.4um Cu, ~0.2um Pt.
Pt-Sn IMC above the Pt layer. Cu layer was not disturbed.
Note that Au and Pt peaks almost overlap.

Microstructure Examination

LTCC Solid-State Aging – 10days at 170°C

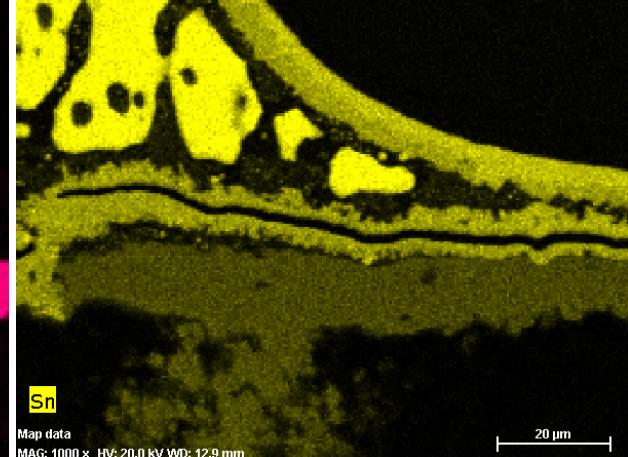
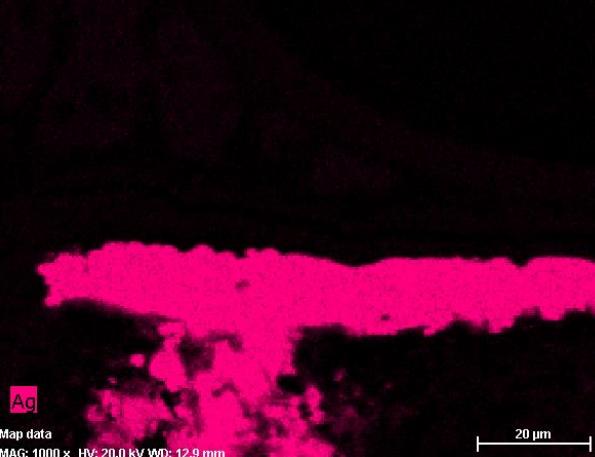
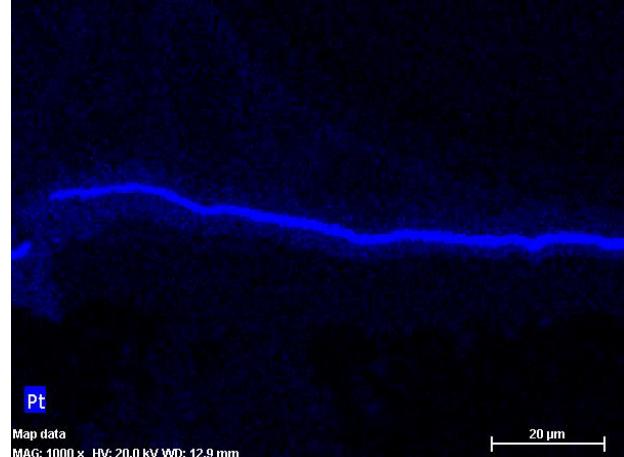
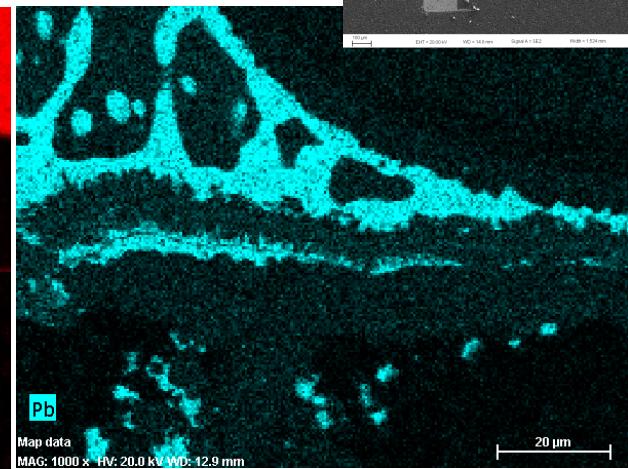
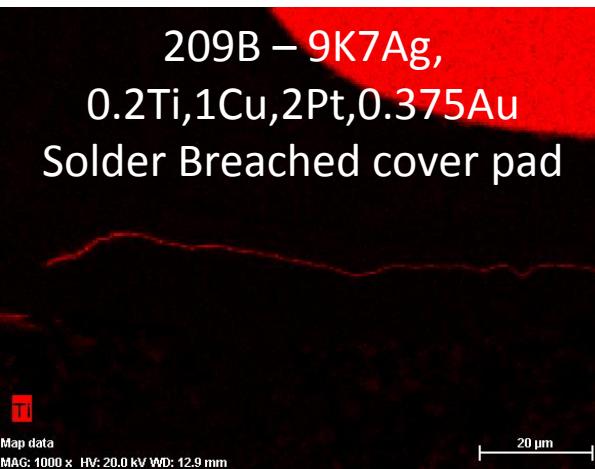
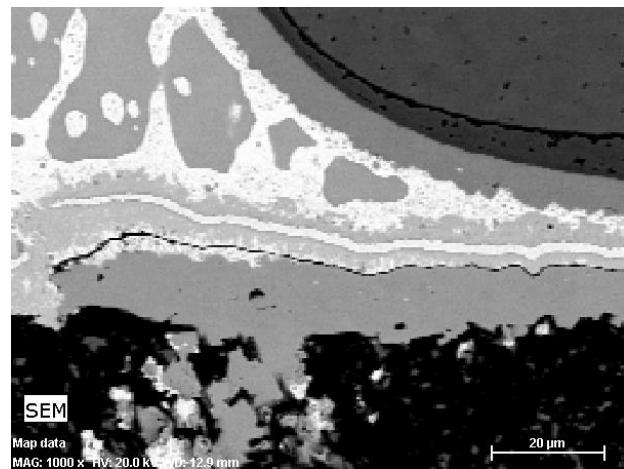


Solder breached through creases in Pt to LTCC.
Pt-Sn, Au-Sn, Cu-Sn IMC present.



Microstructure Examination

LTCC Solid-State Aging – 10days at 170°C

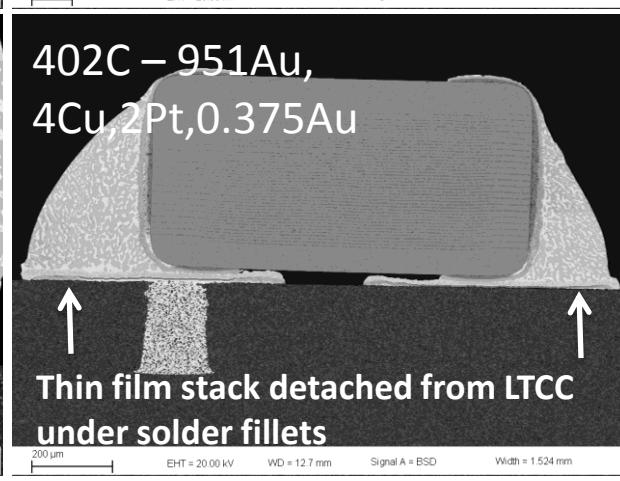
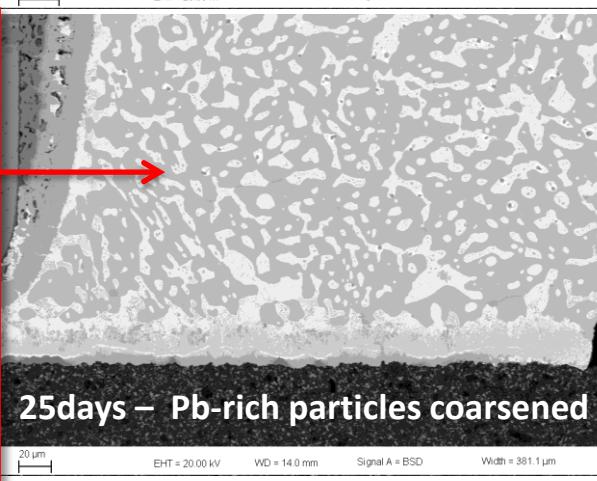
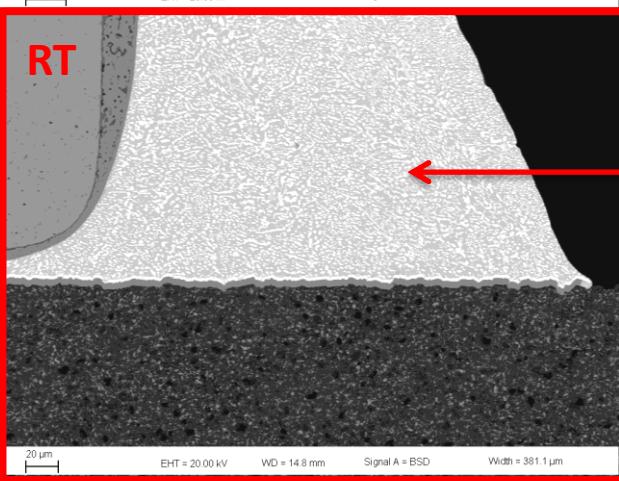
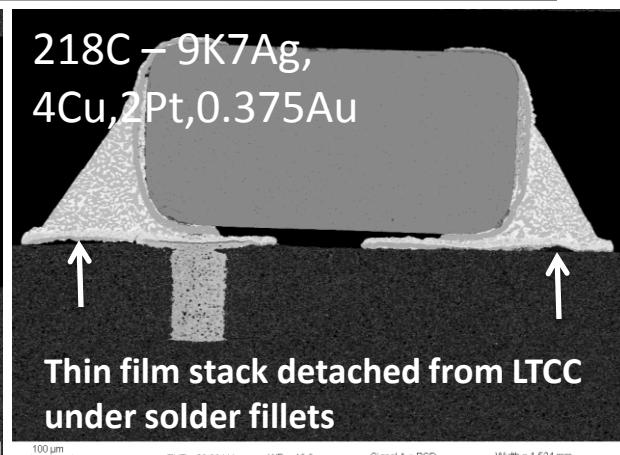
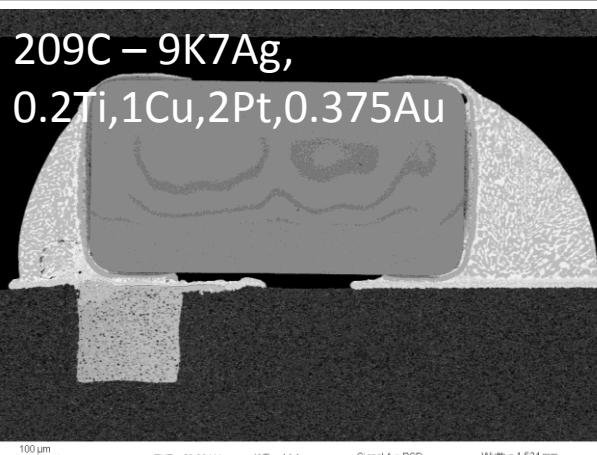
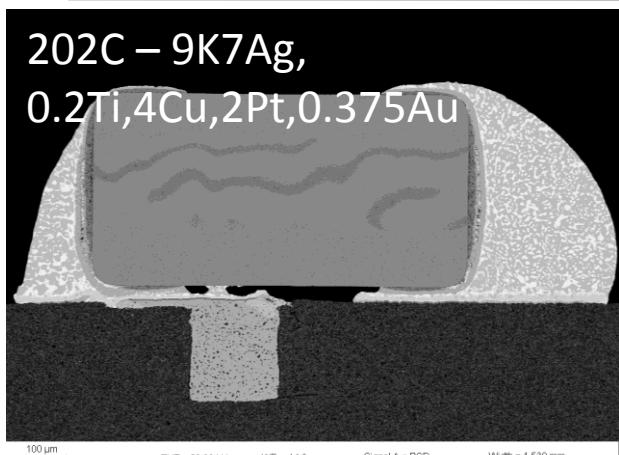


Ni-Sn IMC under capacitor Ni outer layer.
Ti layer lifted. Cu-Sn IMC not seen?

Via breached. Pb-rich particles segregated in Ag. Presence of Ag-Sn IMC and Sn-Pt IMC.

Microstructure Examination

LTCC Solid-State Aging – 25days at 170°C

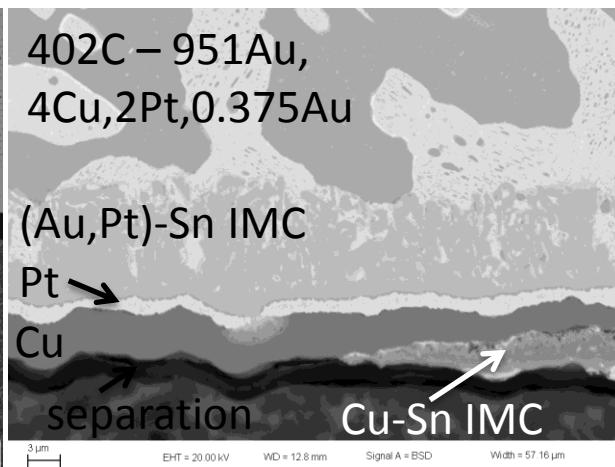
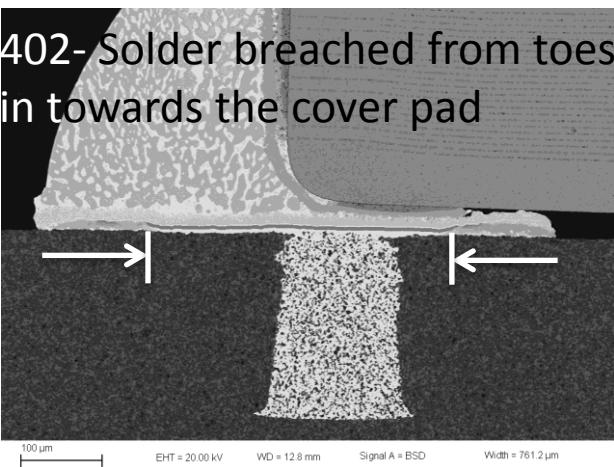
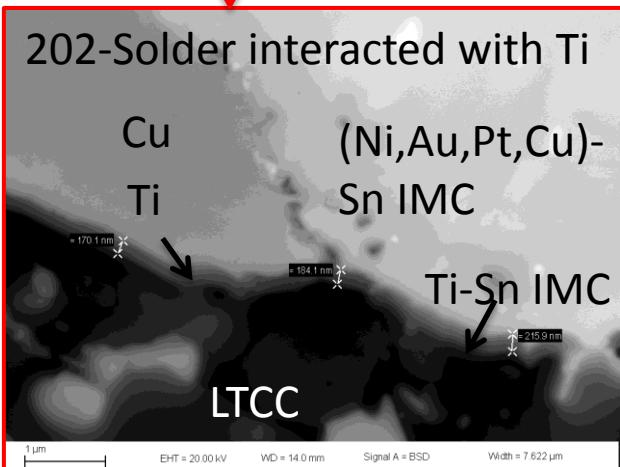
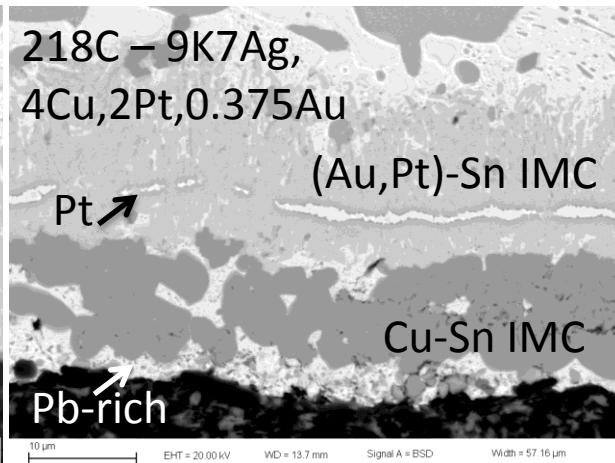
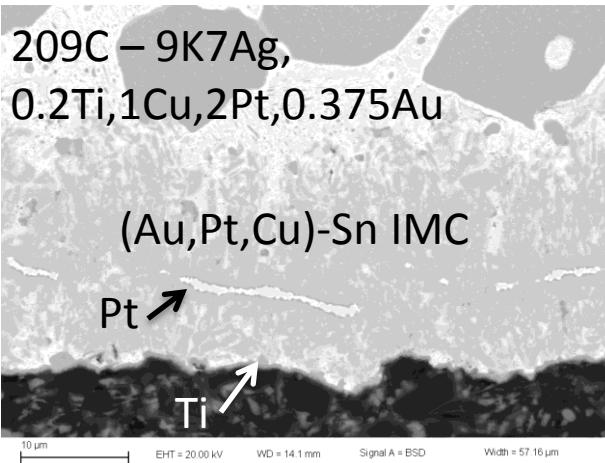
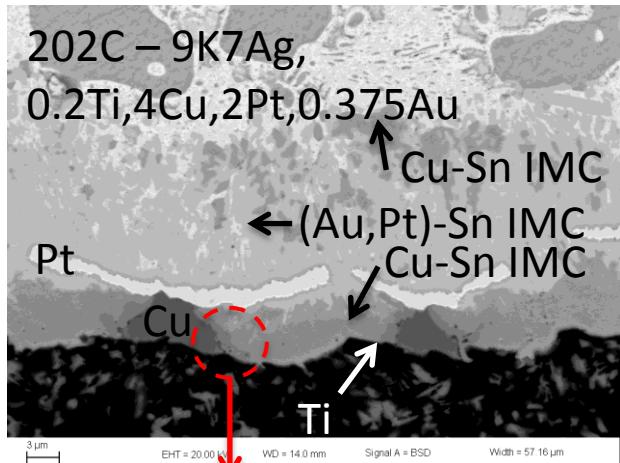


Solder fillets are intact.
Pb-rich particles coarsened.

Without Ti adhesion layer, thin films delaminated from LTCC under solder fillets.
Solder breached and interacted with Pt and Cu almost completely at the fillets.

Microstructure Examination

LTCC Solid-State Aging – 25days at 170°C

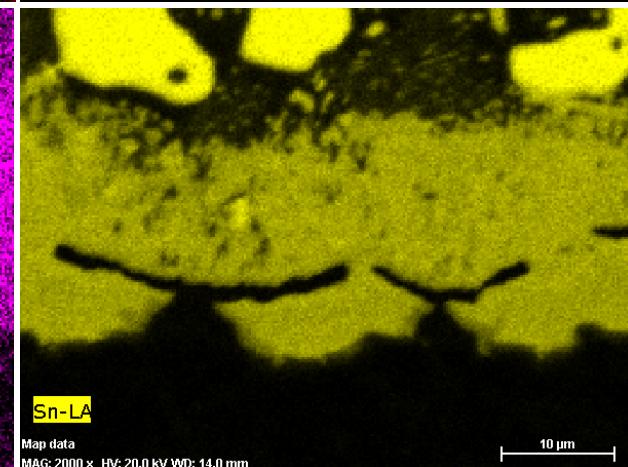
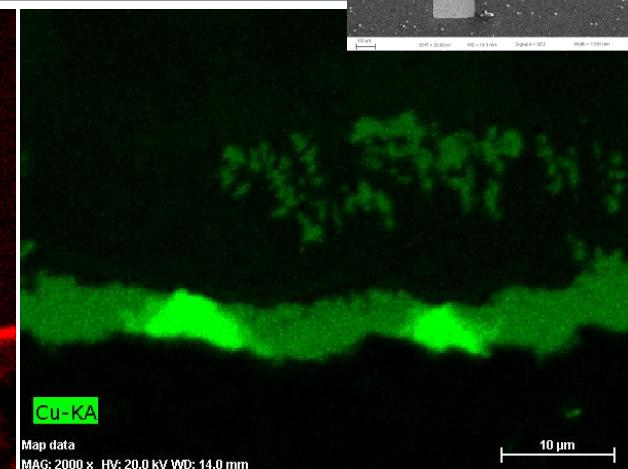
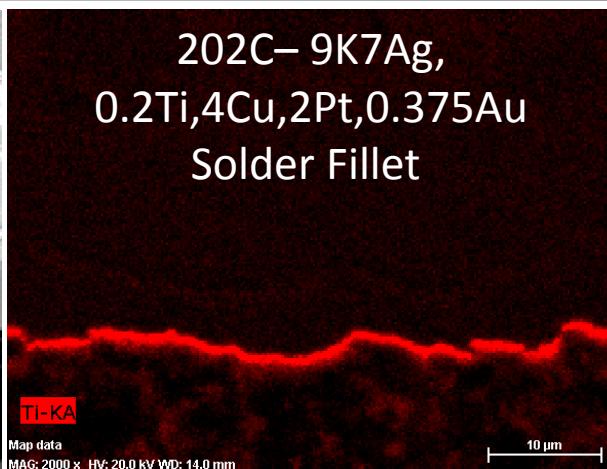
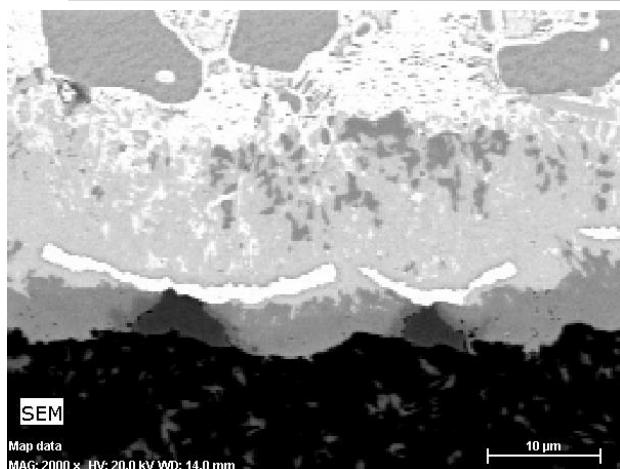


Solder reacted with Pt, breached through and reacted with Cu to great extent
402B appeared to have the least Sn-Cu interaction.

Observed signs of Sn reacted with Ti.

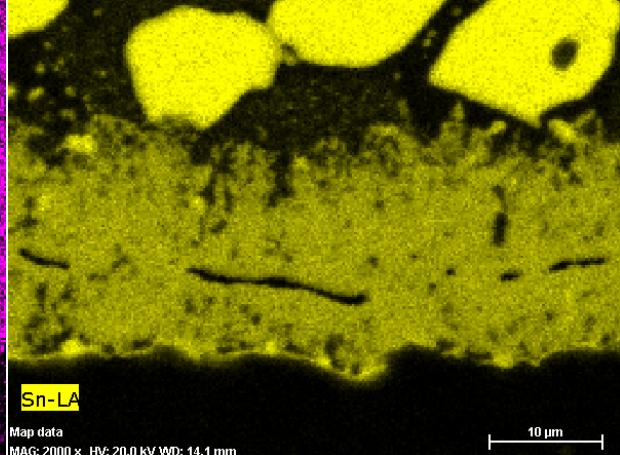
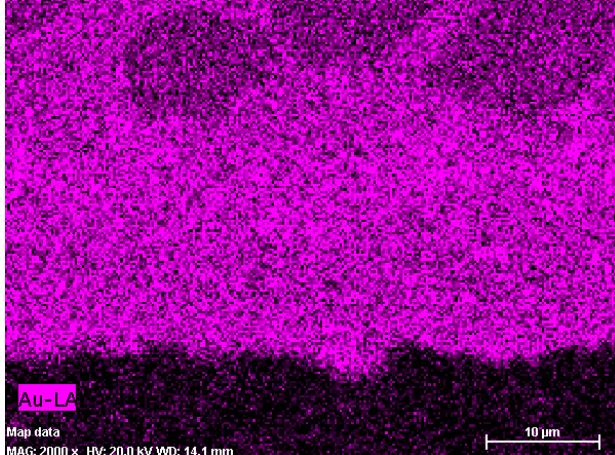
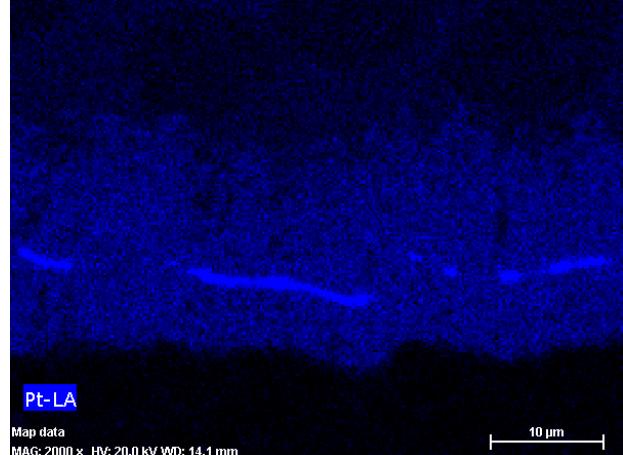
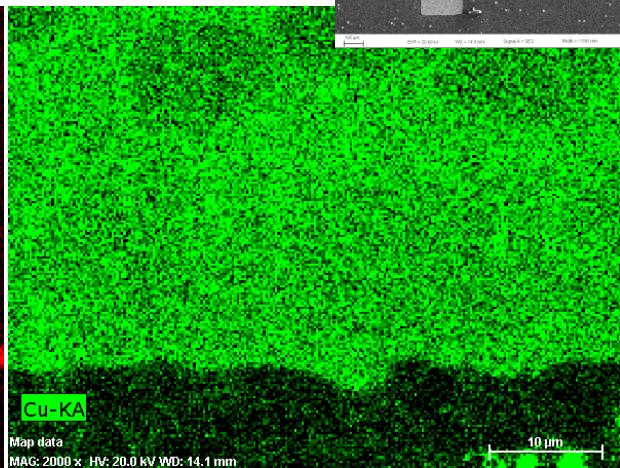
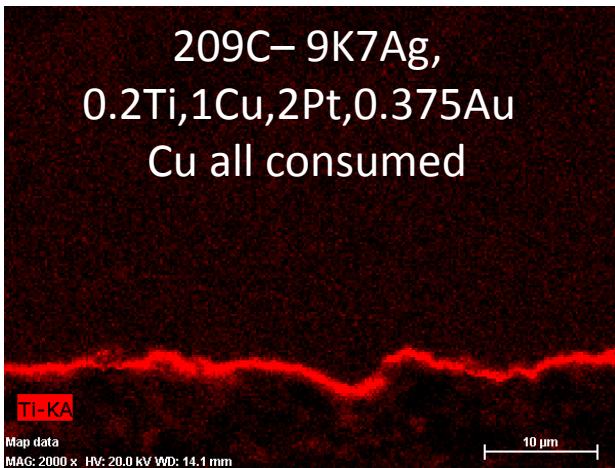
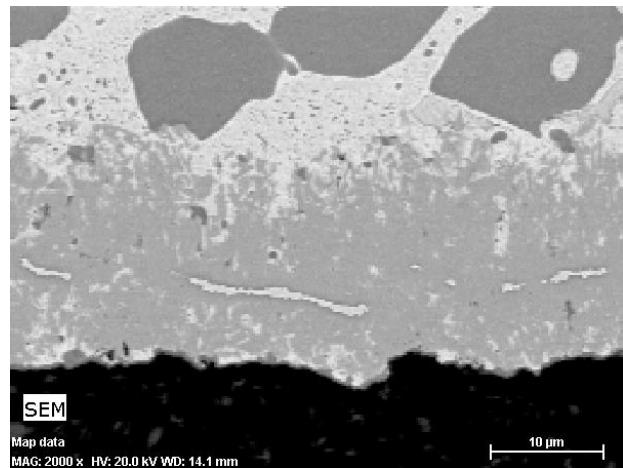
Microstructure Examination

LTCC Solid-State Aging – 25days at 170°C



Solder breached through Pt layer from top.
Sn reacted with all elements- Au, Pt, Cu, Ti-in film stack.
Islands of unreacted Cu in film stack.

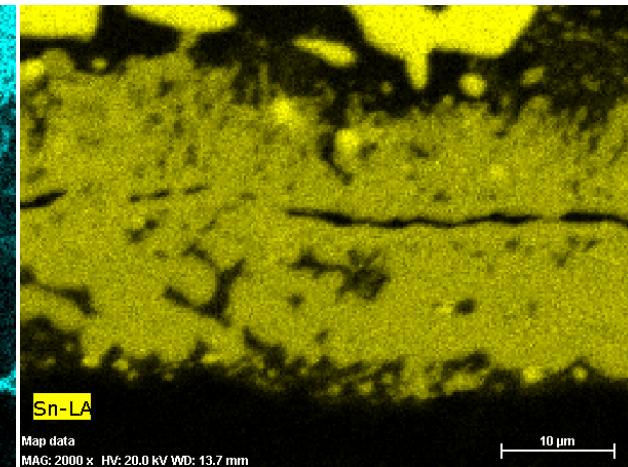
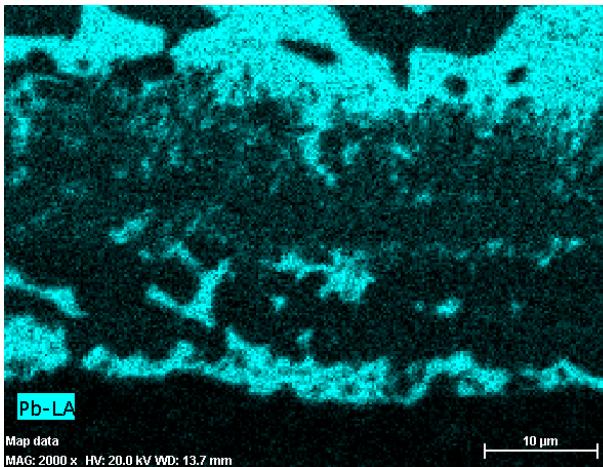
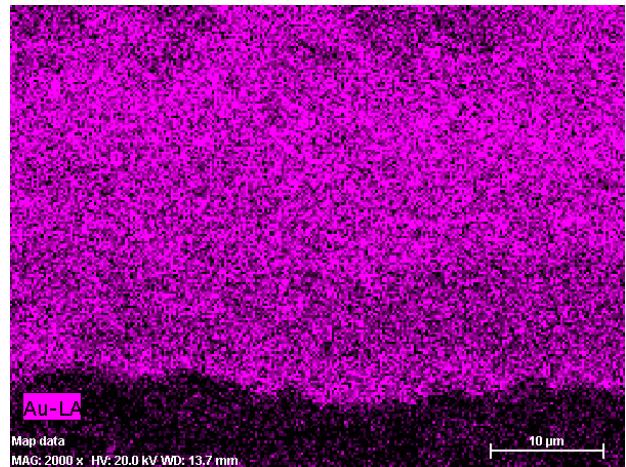
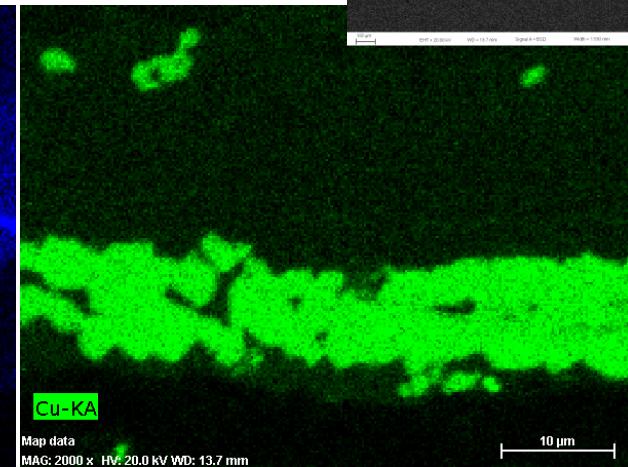
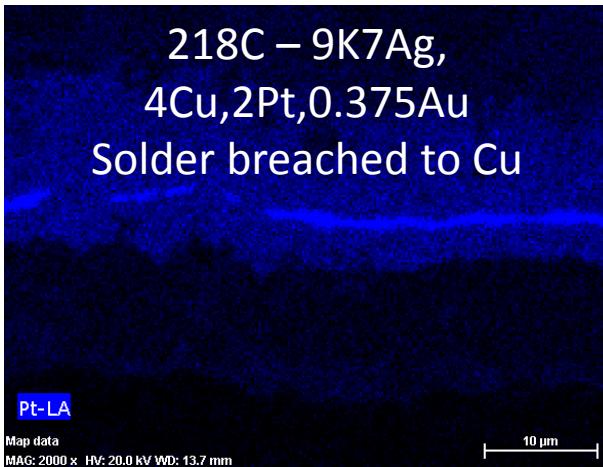
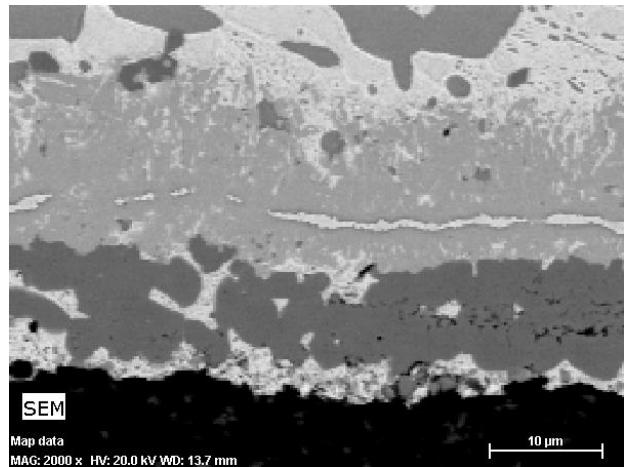
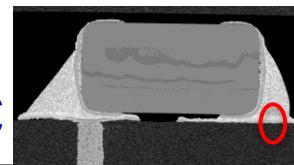
Microstructure Examination LTCC Solid-State Aging – 25days at 170°C



Sn reacted with all elements in thin film stack - Au, Pt, Cu, Ti.
All Cu in the film stack is consumed. Pt in the film stack is almost all consumed.

Microstructure Examination

LTCC Solid-State Aging – 25days at 170°C



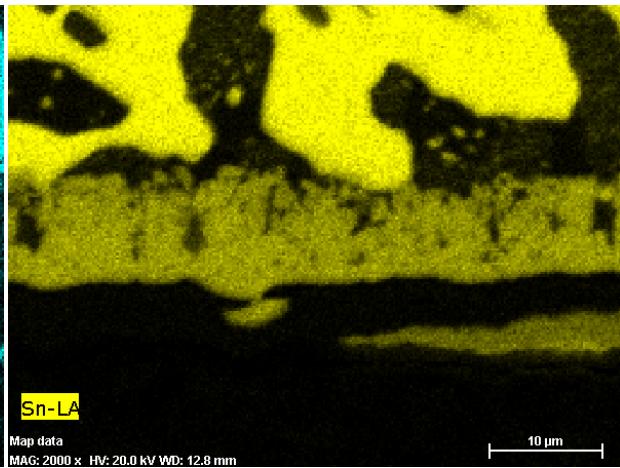
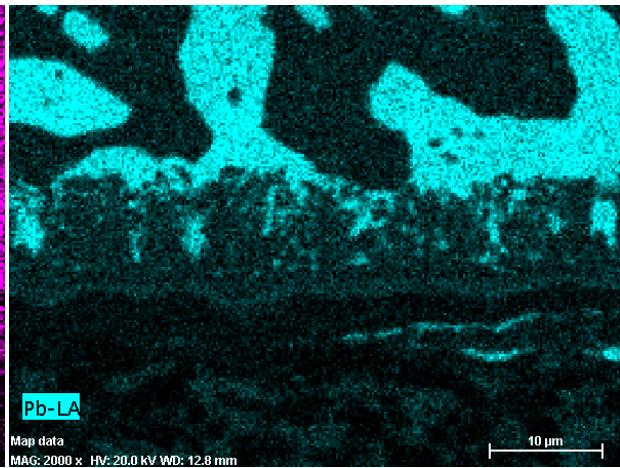
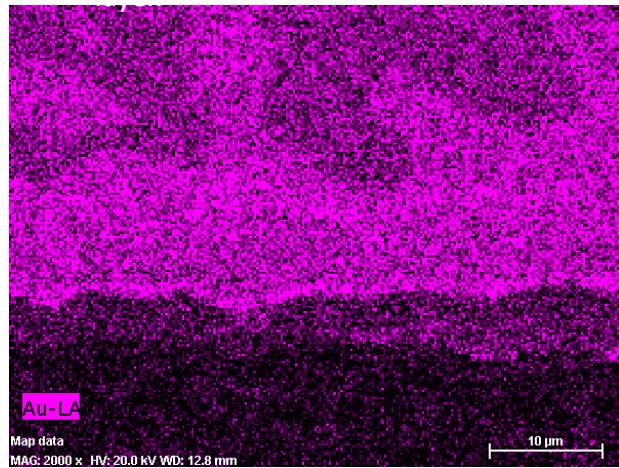
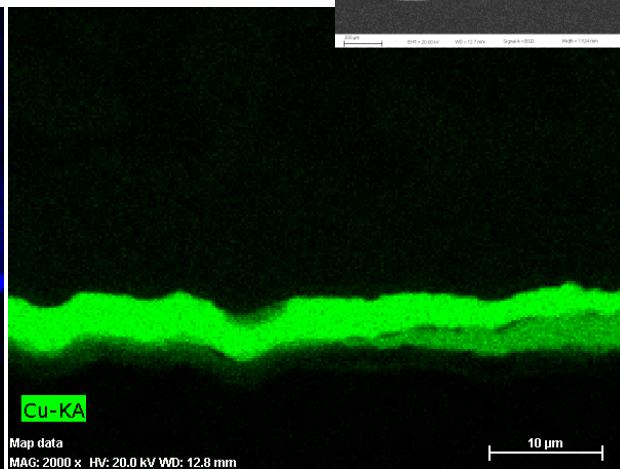
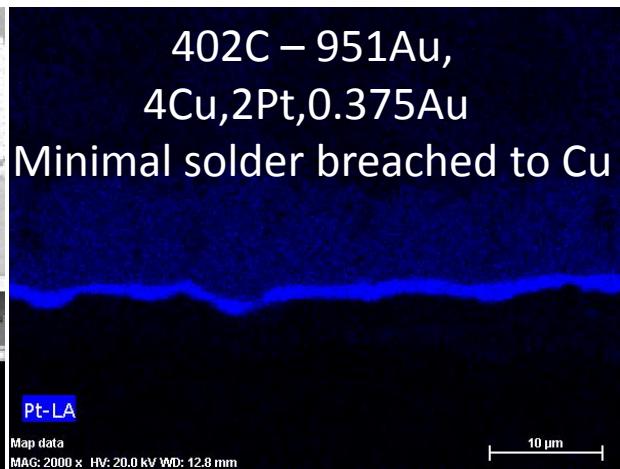
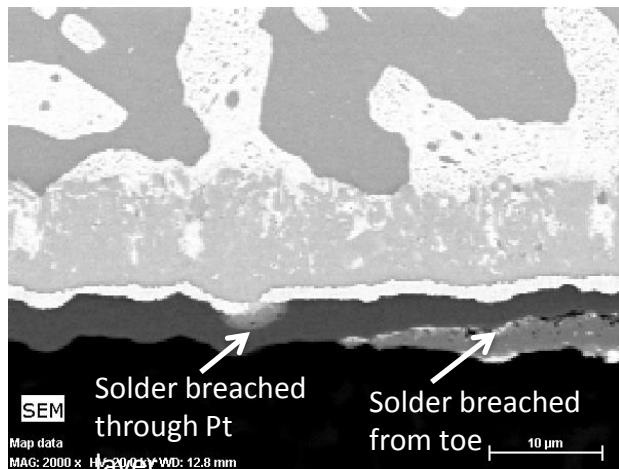
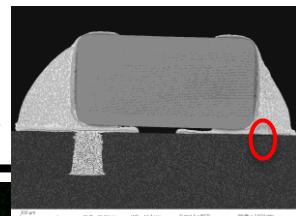
Sn reacted with all elements in thin film stack - Au, Pt, Cu and in the capacitor shells – Ni, Cu.

All Cu in the film stack is consumed. Pt in the film stack is almost all consumed.

Pb rich particles found at the thin film/LTCC interface were possibly due to molten solder undermining film stack during the time the joint was made because there was no Ti adhesion layer.

Microstructure Examination

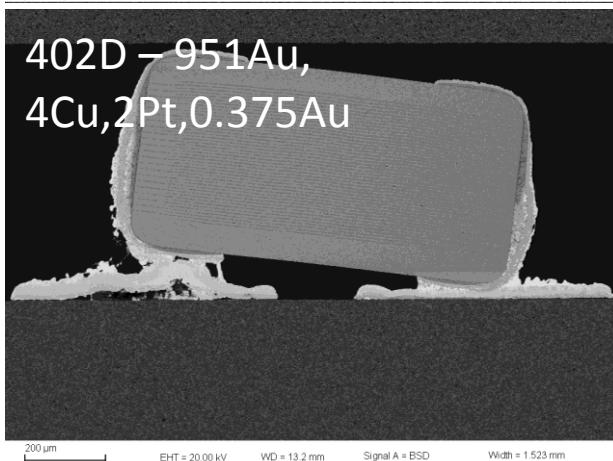
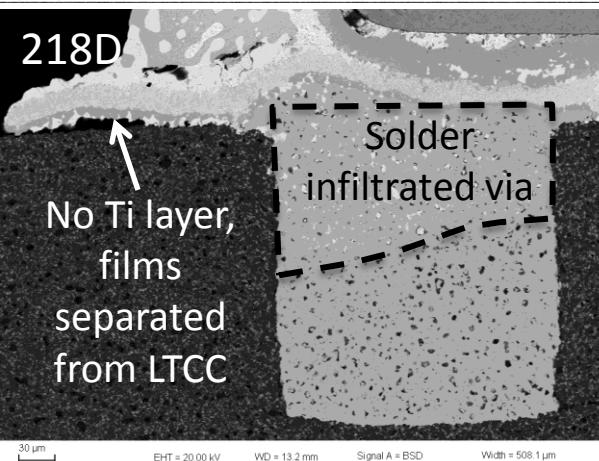
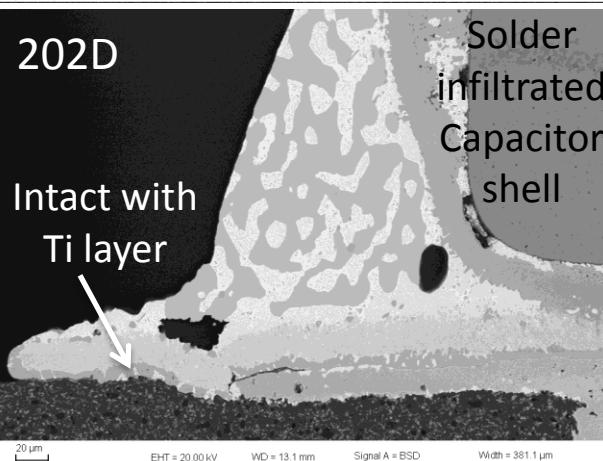
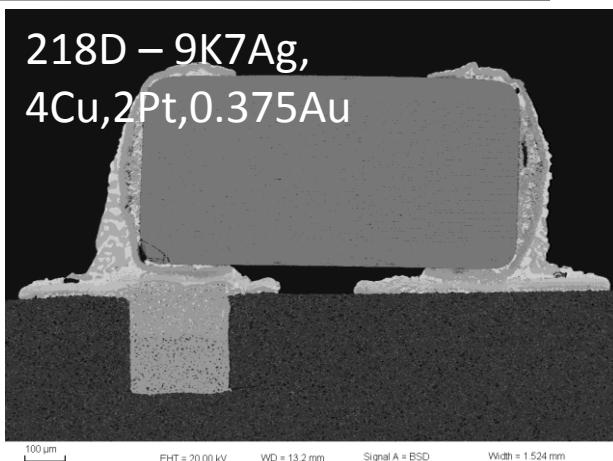
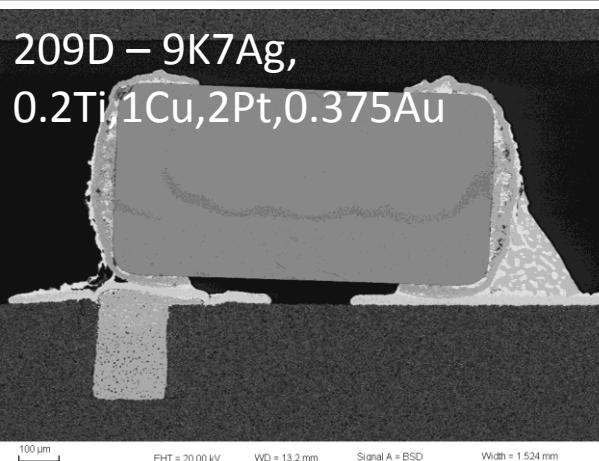
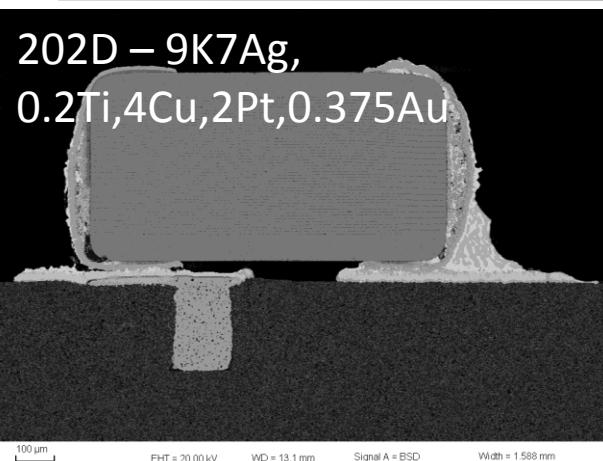
LTCC Solid-State Aging – 25days at 170°C



Solder breached from toe, reacted with Cu layer more quickly than solder breached through Pt from top.
Pt layer is mostly intact.

Microstructure Examination

LTCC Solid-State Aging – 100days at 170°C

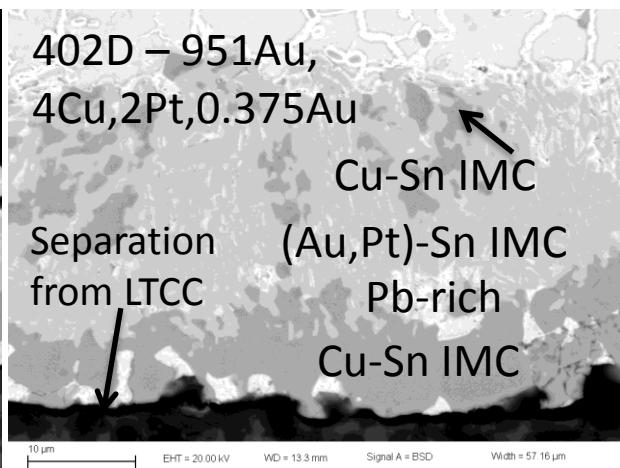
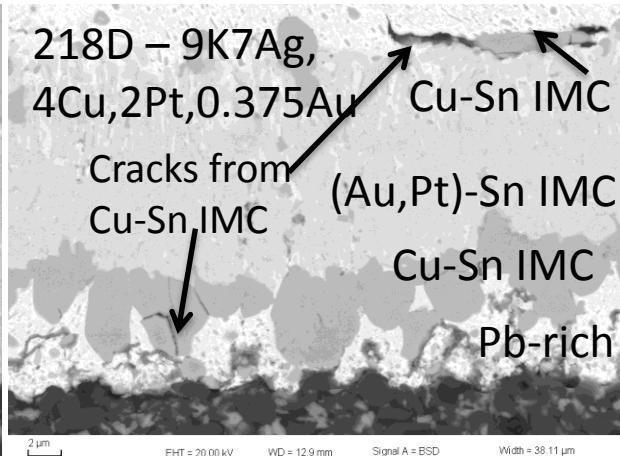
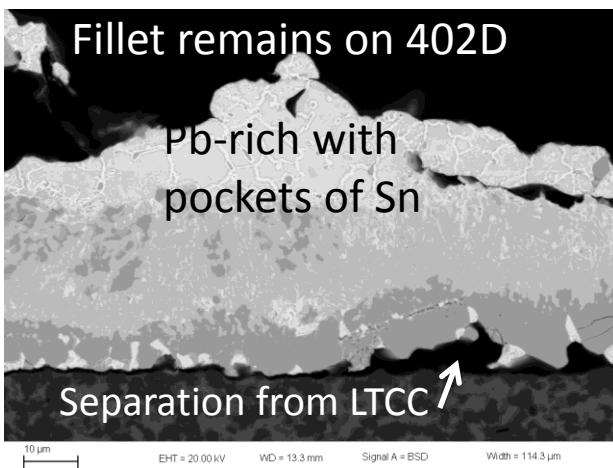
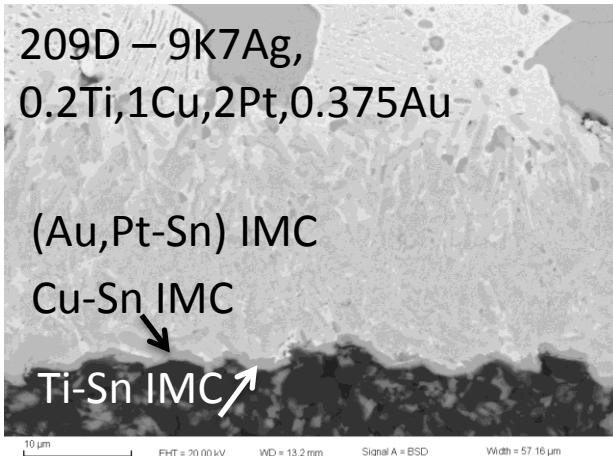
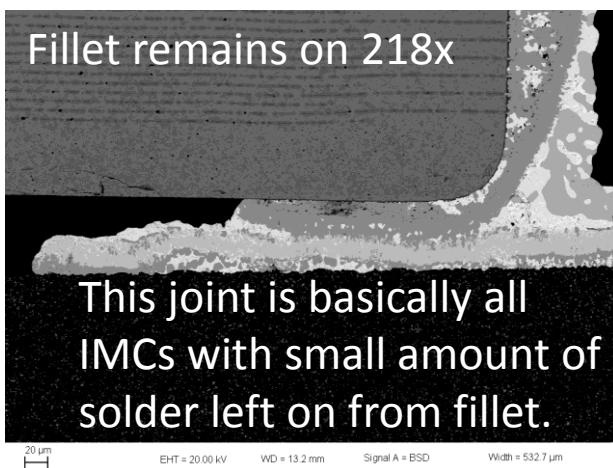
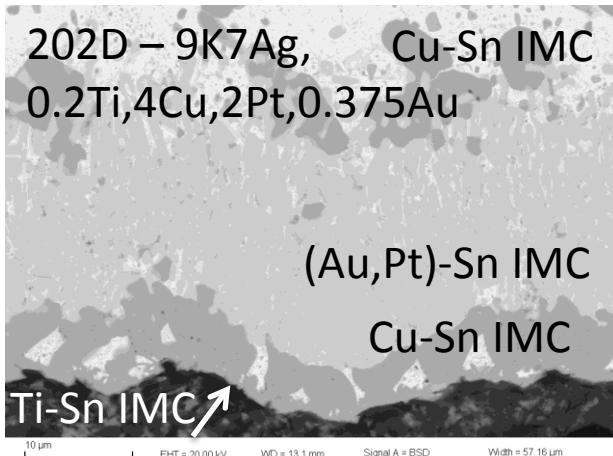


Where did the solder go???

Vias and Ni/Cu shell on capacitors were infiltrated with solder.
Pb-rich particles coarsened. No discrete Ti or Cu layers.
Significant growth of Pt-Sn and Cu-Sn IMCs.

Microstructure Examination

LTCC Solid-State Aging – 100days at 170°C

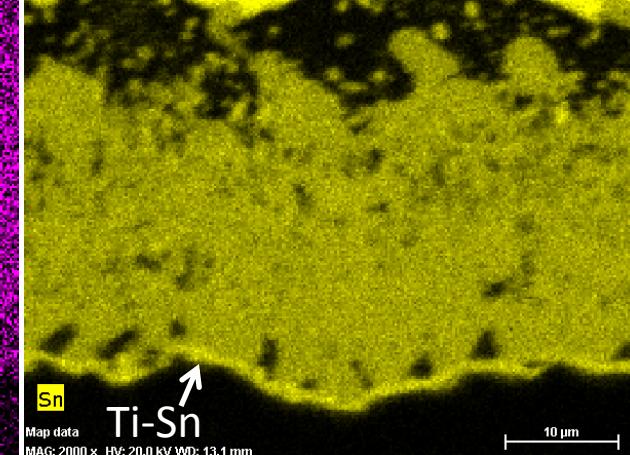
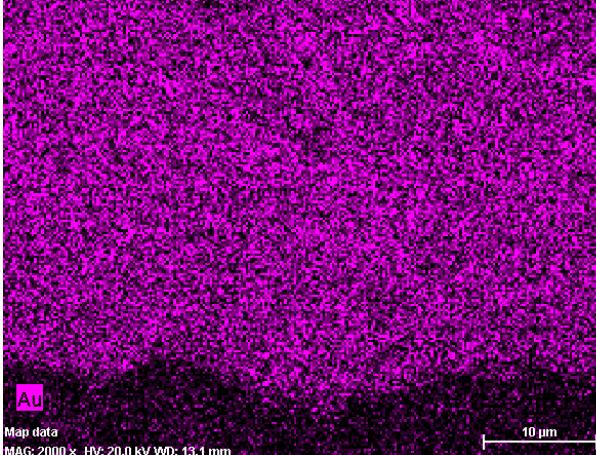
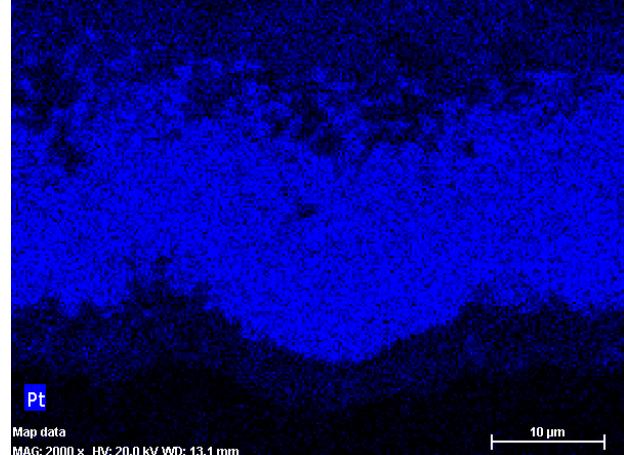
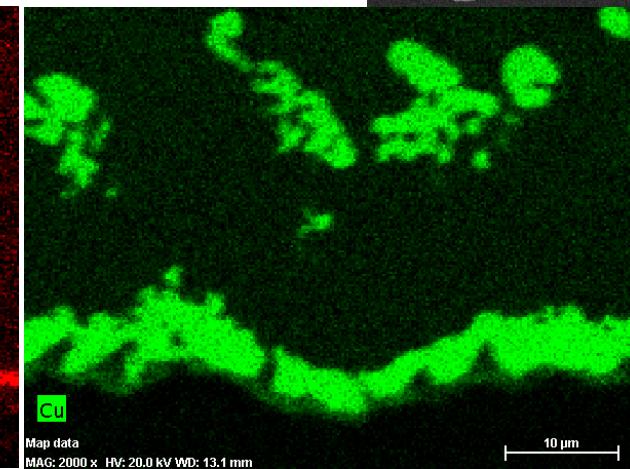
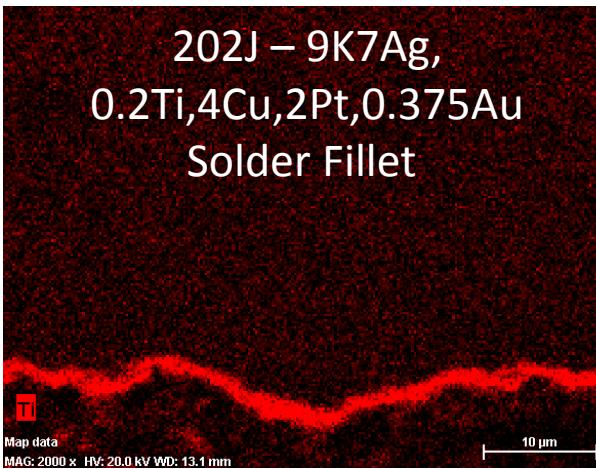
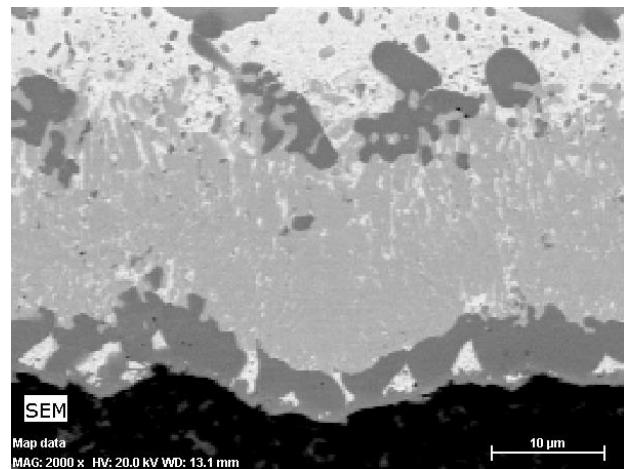
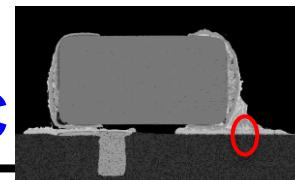


Where did the solder go???

All joints composed of Pt-Sn, Sn-Cu, and Ti-Sn IMCs with many segregated Pb-rich particles.
Micro-cracks in regions near or in Cu-Sn IMCs.
Clear separations from LTCC.

Microstructure Examination

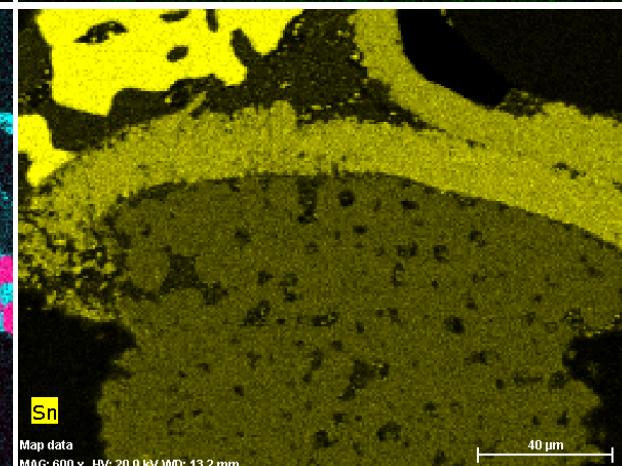
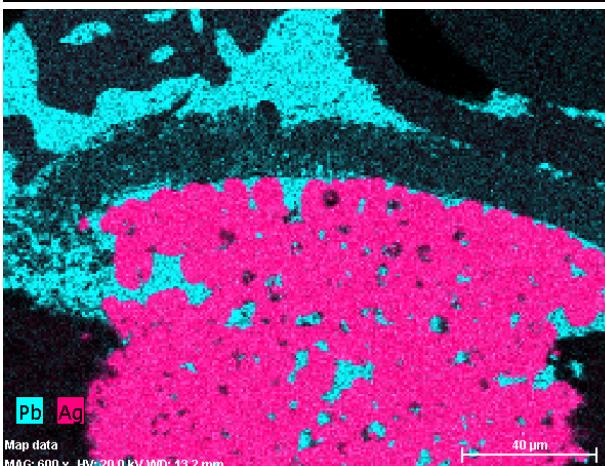
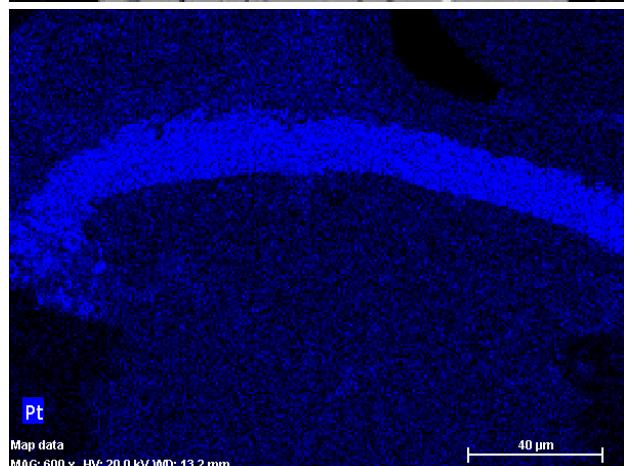
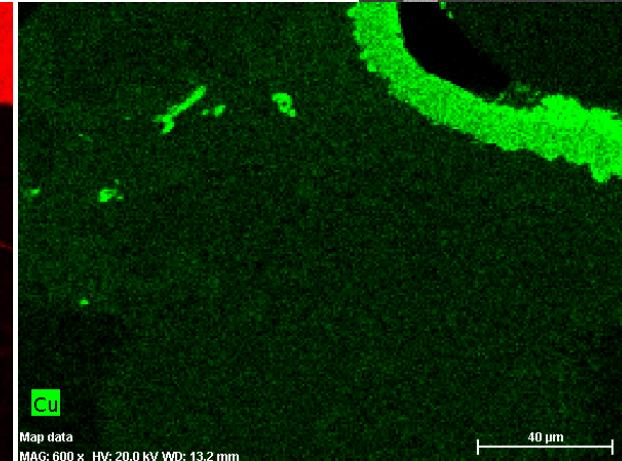
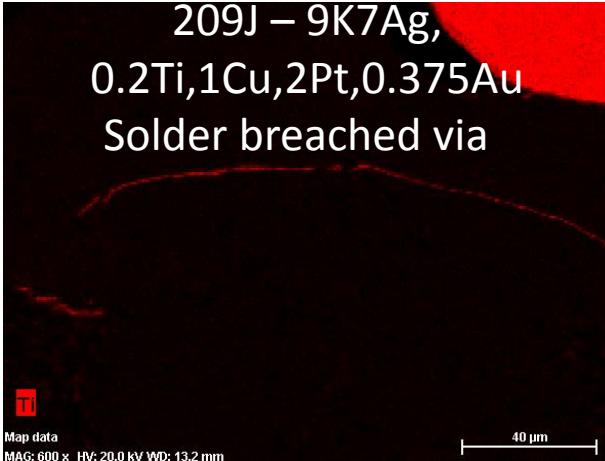
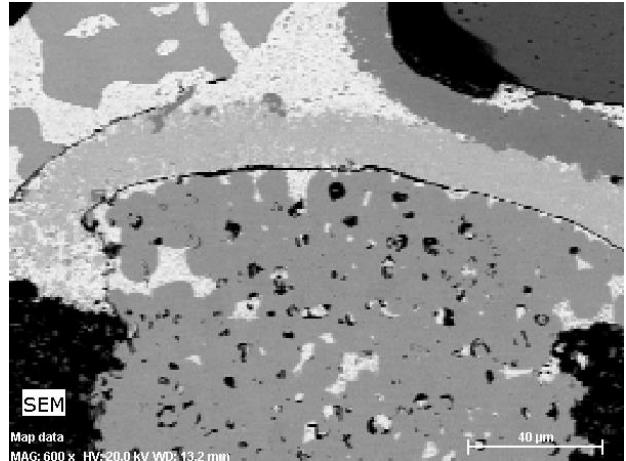
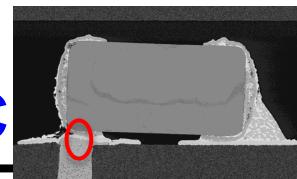
LTCC Solid-State Aging – 100days at 170°C



Sn reacted with all elements in thin film stack - Au, Pt, Cu, Ti.
The Cu-Sn IMCs appeared scalloped with Pb-rich pockets between them.

Microstructure Examination

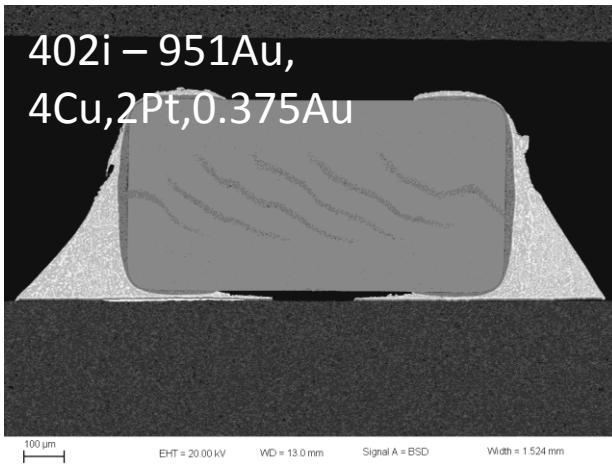
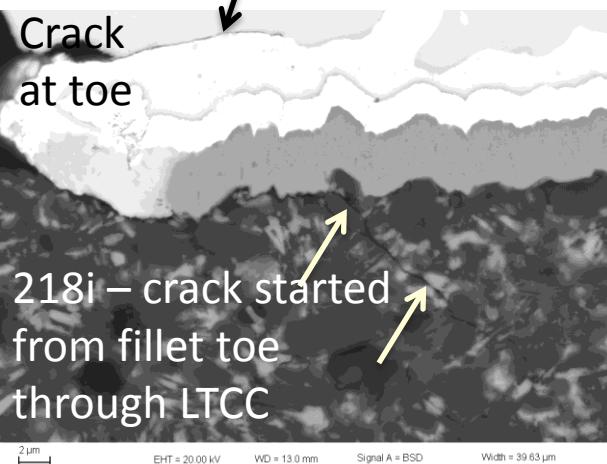
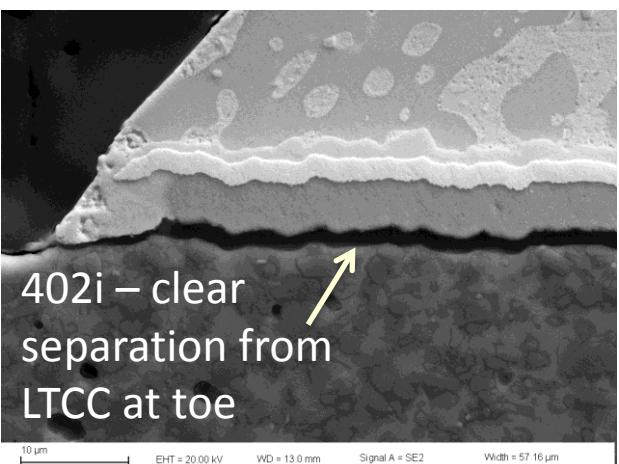
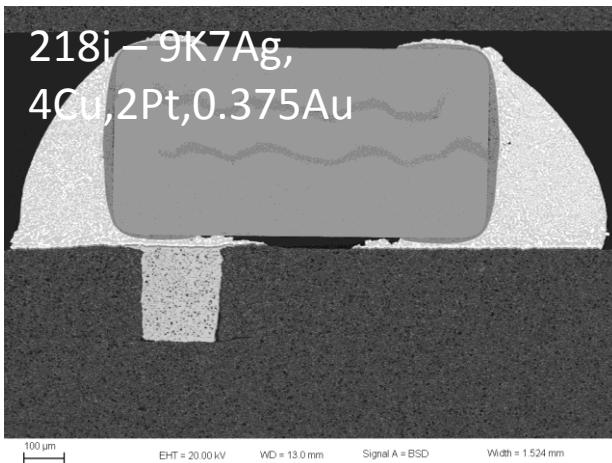
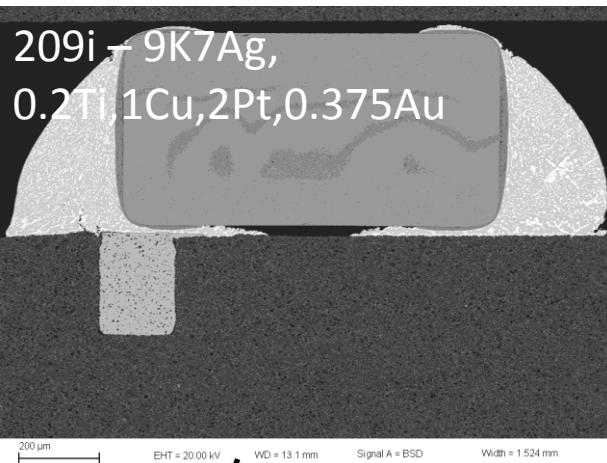
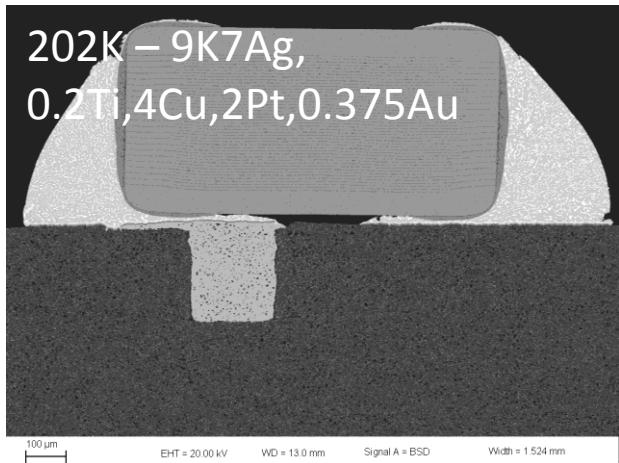
LTCC Solid-State Aging – 100days at 170°C



Ti layer lifted. Cu-Sn IMC at capacitor shell location and pockets in the solder.
Via breached. Pb-rich particles segregated in Ag. Presence of Ag-Sn IMC and Sn-Pt IMC.

Microstructure Examination

LTCC Solid-State Aging – 1000 Cycles



Solder fillets are intact. Films adhered to LTCC in samples with Ti layers.

All samples have long cracks starting from fillet toes in LTCC.

Pb-rich particles coarsened.

Discrete Au-Sn IMC layer above Pt layer, less laths dispersed in solder as in RT samples.

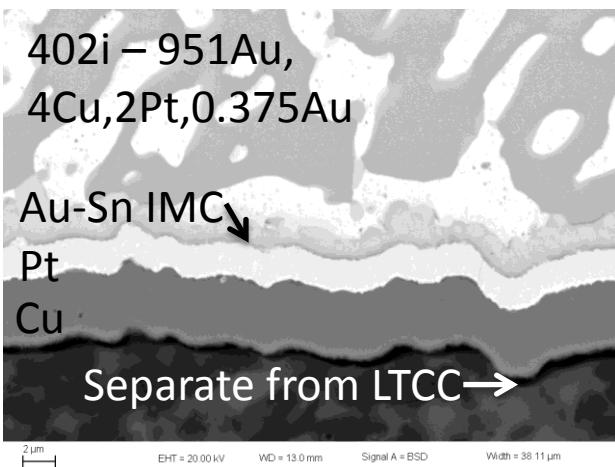
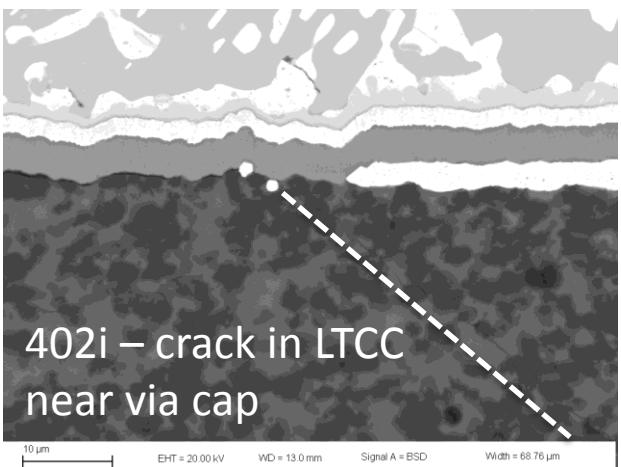
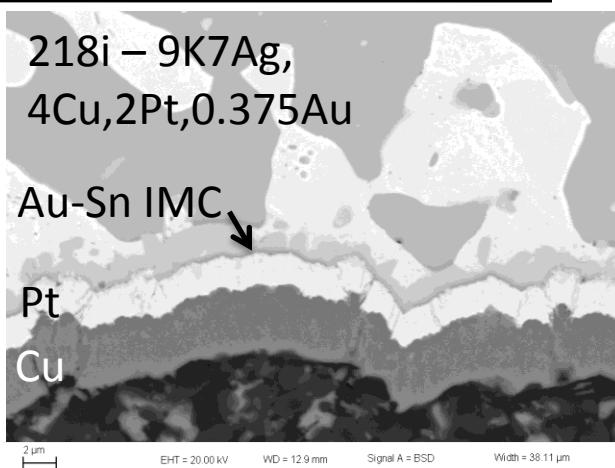
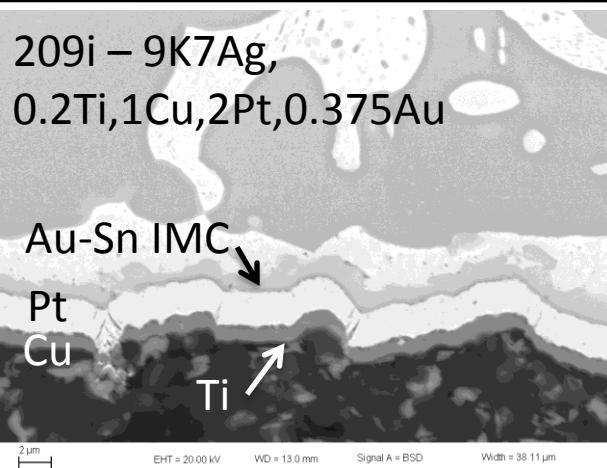
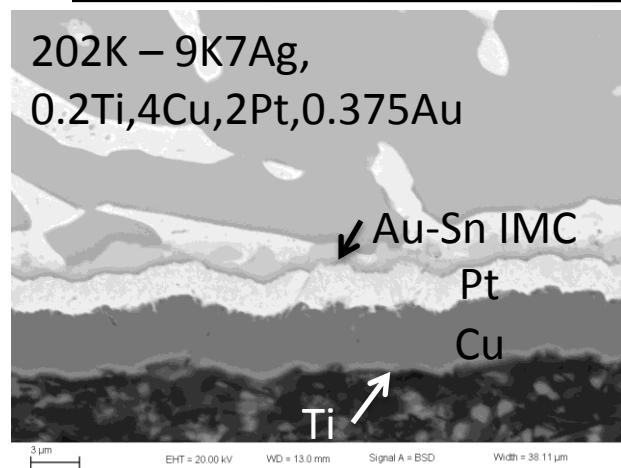
8/12/2014

P. Sarobol, Org. 1832 SNLA

42

Microstructure Examination

LTCC Solid-State Aging – 1000 Cycles

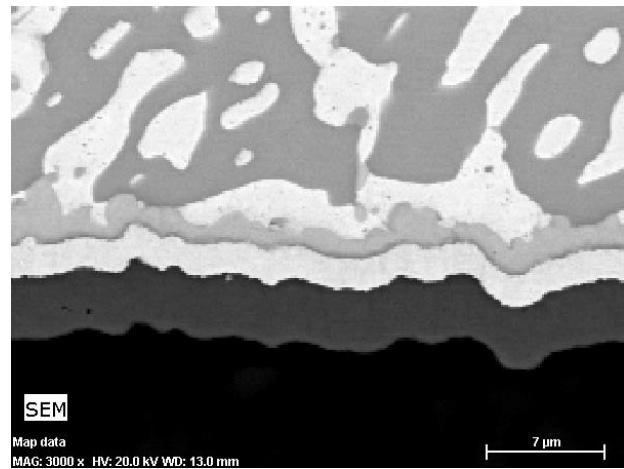
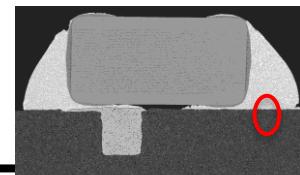


In solder region, Au-Sn IMC laths and discrete layer above Pt layer.
Pt and Cu layers showed NO signs of dissolution. There are creases.

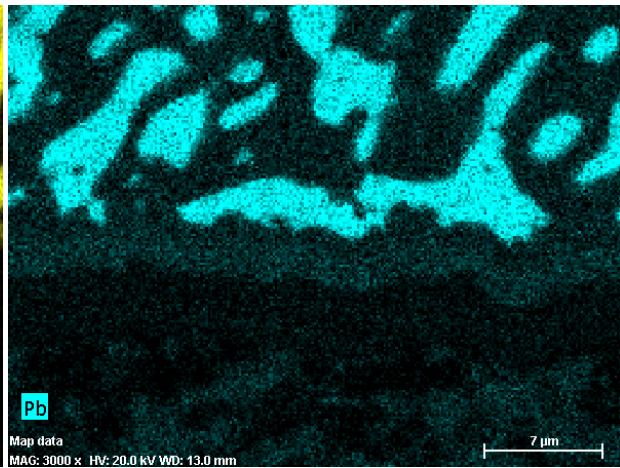
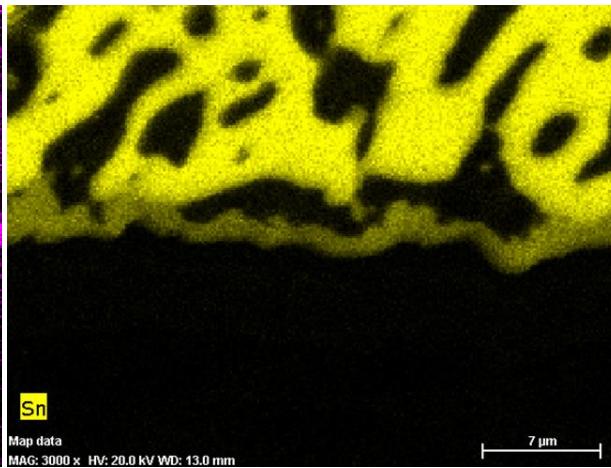
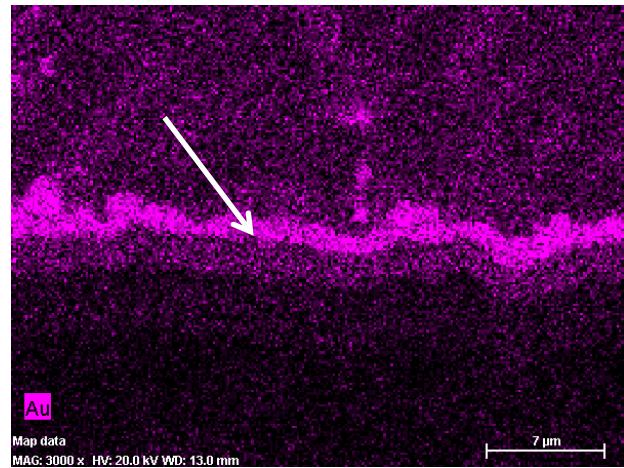
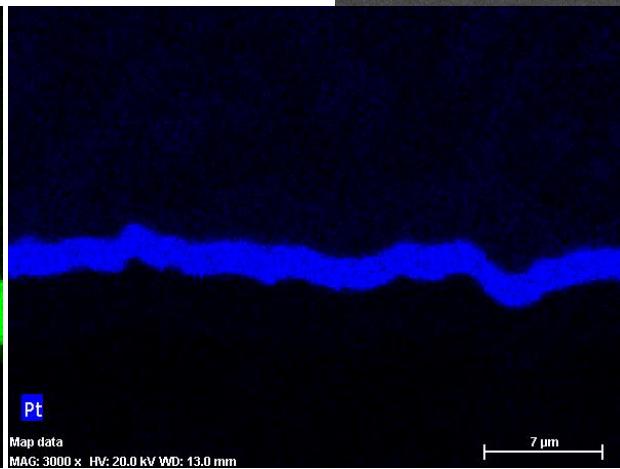
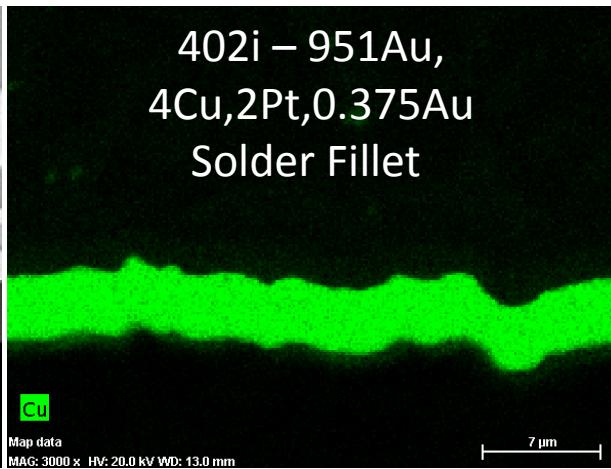
Au-Sn IMC layer prevented solder breach to thin films, via caps, and via materials.

*Cracks in LTCC near fillet toes and cover pad. Clear separation of Cu layer from LTCC in 402i.

Microstructure Examination LTCC Solid-State Aging – 1000 Cycles

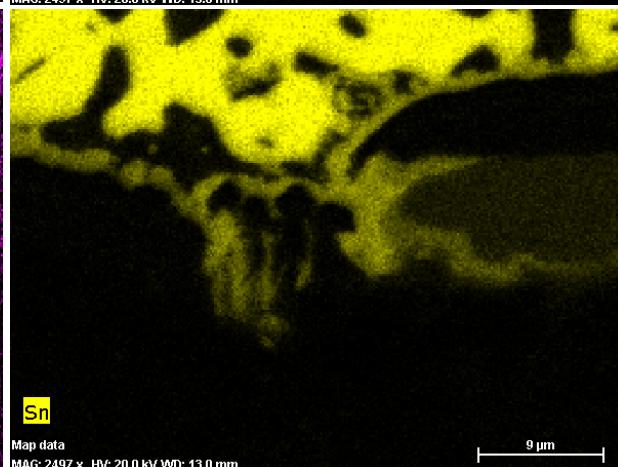
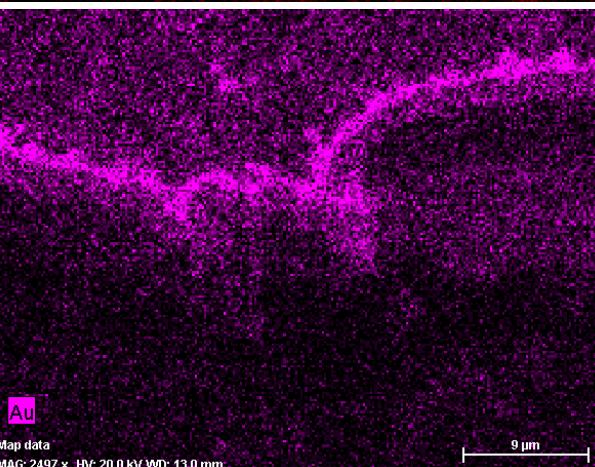
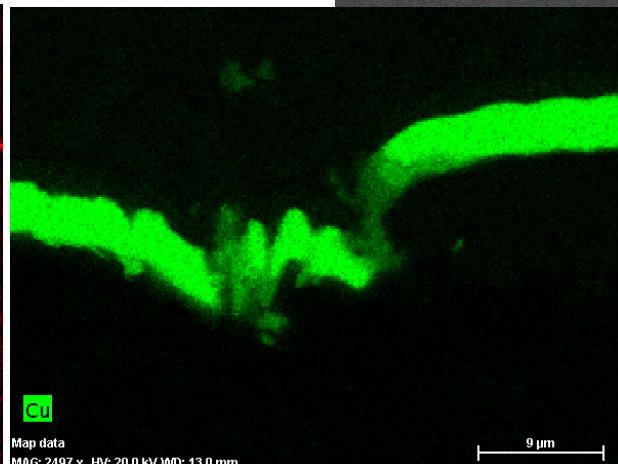
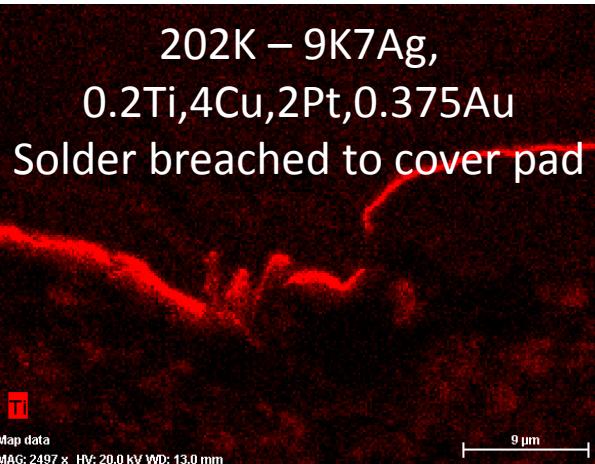
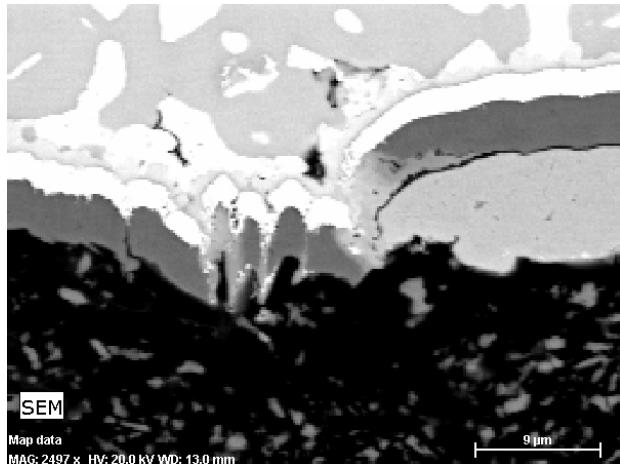
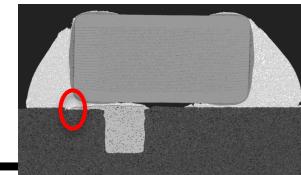


402i – 951Au,
4Cu,2Pt,0.375Au
Solder Fillet



Discrete layers (LTCC → Solder) ~0.4um Cu, ~0.2um Pt and ~1.7um Au-Sn IMC.
Sn only reacted slightly with Pt layer.
Cu layer appeared to be undisturbed.

Microstructure Examination LTCC Solid-State Aging – 1000 Cycles



Creases in thin film stack provided discontinuity in the Pt.
Sn reacted with Pt, Cu, Ti and Au to form IMCs.
Via breached. Sn reacted with Ag via material to form Ag-Sn IMC.