Effect of Thermal Aging on Solderability of ENEPIG Surface Finish Used in Printed Circuit Boards

PURDUE MATERIALS ENGINEERING

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Medtronic seeks to quantify the thermal aging limits of electroless Ni-electroless Pd-immersion Au (ENEPIG) surface finishes to determine how aggressive the silicon burn-in process can be without loss of solderability. Silicon burn-in (power testing at elevated temperature) is used to eliminate early field failures, critical for device reliability. Thermal aging due to burn-in or annealing causes causes Ni and Pd diffusion to and oxidation on the surface. Surface oxides limit wetting of the PbSn solder, affecting electrical connectivity of components soldered after burn-in. Isothermal aging of two ENEPIG surface finishes was performed at 75°C- 150°C for 100hrs - 1500hrs to test the thermal aging limits and identify how loss of solderability occurs. Surface characterization by SEM and XRD and solderability studies to determine loss of wettability as a function of thermal aging were completed as part of the analysis. An overall decrease in solderability (increase in contact angle) with increased thermal aging time was observed. Unpredictable wetting behavior and irregular wetting profiles caused the variability and uncertainty in the measurement method, but gave insight into the behavior of the PbSn preforms as well as the changing wettability of the aged surfaces.

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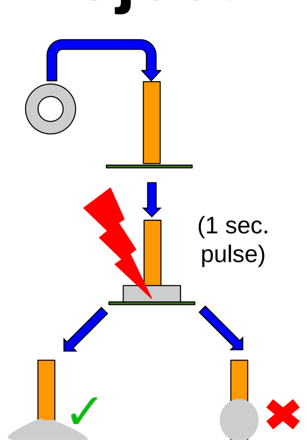
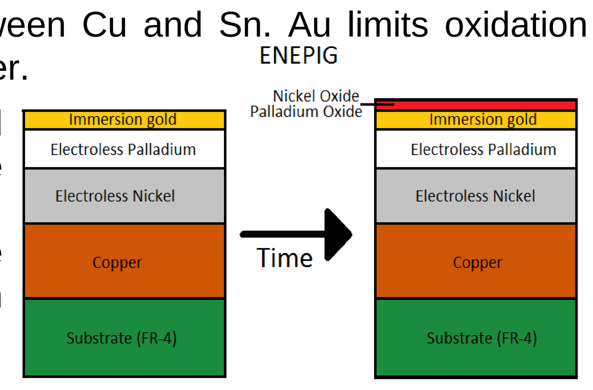
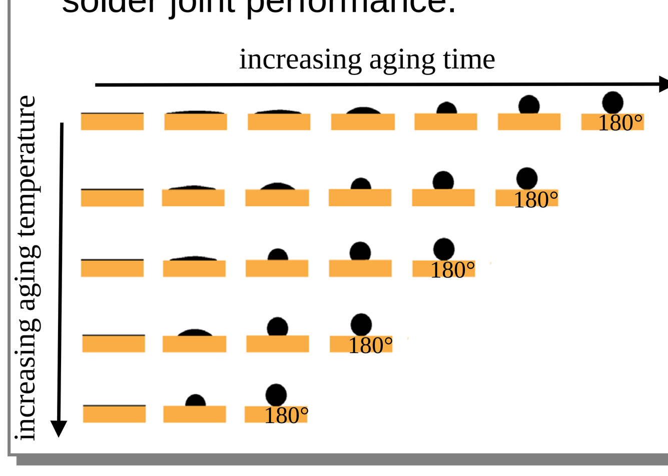
Project Background



- Medtronic is a leading company in medical electronics innovation.
- Laser selective soldering is used to form a few solder joints after burn-in (left).
- Occasionally the solder preform will not wet the pad (indicated by red x) during soldering, resulting in scrapped circuit boards.
- Purpose of this project is to determine the effects and limits of thermal aging of electroless Ni-electroless Pd-immersion Au (ENEPIG) as it relates to burn-in, the process suspected of causing occasional loss of solderability.
- ENEPIG: is a common surface finish that is deposited onto the Cu pad in layers as seen in the schematic below. Ni and Pd act as diffusion barriers to prevent reaction between Cu and Sn. Au limits oxidation and promotes wetting of the solder.



- Thermal aging causes nickel and palladium to diffuse to the surface and oxidize in air [1]
- Oxidation of the ENEPIG surface finish can significantly diminish the solderability and therefore, solder joint performance.



Hypotheses (schematic left):

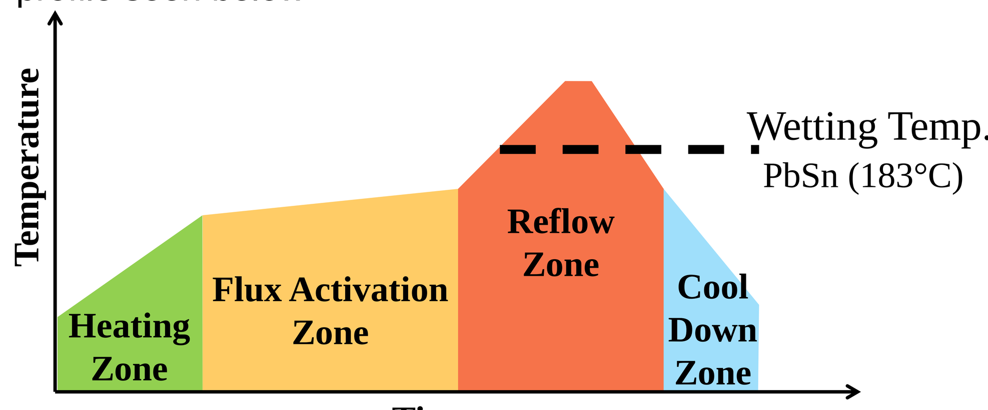
- As aging time increases, solderability will decrease.
- At each temperature a critical (non-wetting) point will be reached.
- As aging temperature increases, time to reach the critical point will decrease.

Experimental Procedure

- Two versions of ENEPIG (V1 – thin Pd (X nm), V2 – thick Pd (Y nm) were thermally aged according to the experimental matrix below

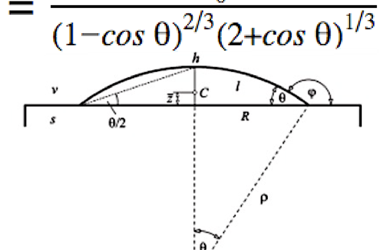
Times (Hours)	Room Temp	75°C	100°C	125°C	150°C
As received	1	Initial Preconditioning for all samples prior to aging			
2x reflow (Assembly)	2				
125°C - 24 hours (Initial Burn In)	3				
100		4	5	6	7
200		8	9	10	11
500		12	13	14	15
1000		16	17	18	19
1500		20	21	22	23

- Surface characterization of ENEPIG surfaces
  - SEM and EDS – surface topography and chemical composition
  - XRD – change in phases and amorphous fraction over time
- Solderability was characterized by measuring dimension changes and areas of spread characteristic of the wetting patterns. Contact angle was calculated assuming that the area of spread is a hemispherical droplet of fixed volume.
  - Prefluxed PbSn solder preforms with hollow cylindrical geometry (dimensions) were placed on surface finished pads of the printed circuit boards and soldered using a reflow oven following the reflow profile seen below



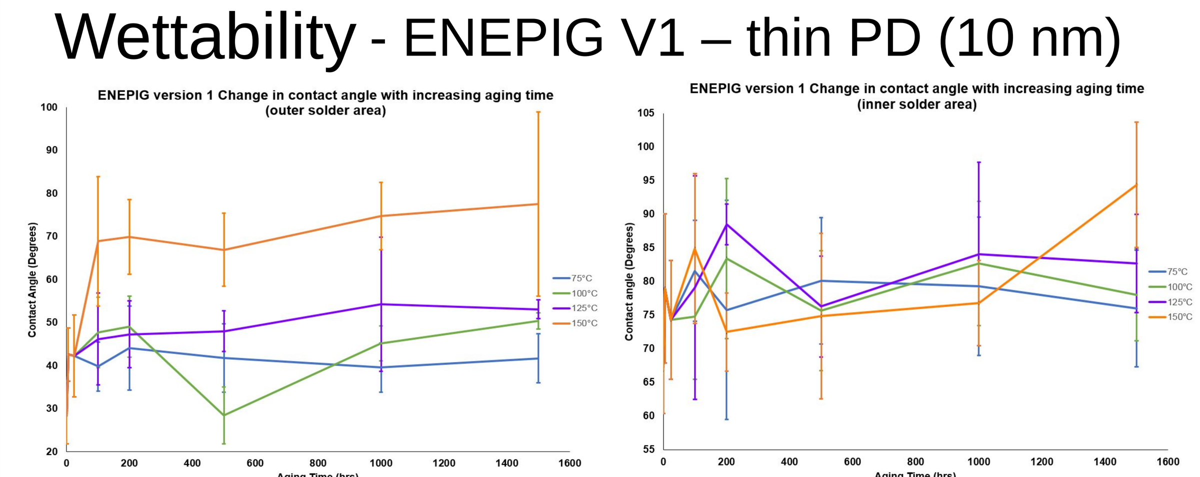
- Optical images were taken of each pad and areas of spread were measured using ImageJ image analysis software
- Area of spread was measured in two locations; outer solder area (red) and inner solder area (purple)
- Contact angle was determined mathematically from the measured area of spread using the equation below R_0 = radius of the original solder volume, R = area of spread radius, θ = contact angle

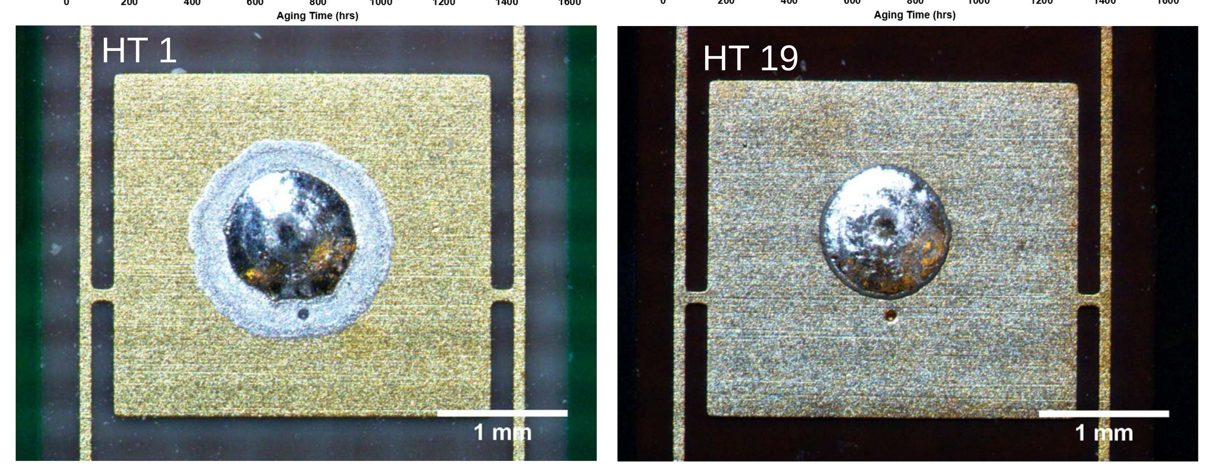
$$R = \frac{2^{2/3} R_0 \sin\theta}{(1-\cos\theta)^{2/3}(2+\cos\theta)^{1/3}}$$



Results And Discussion

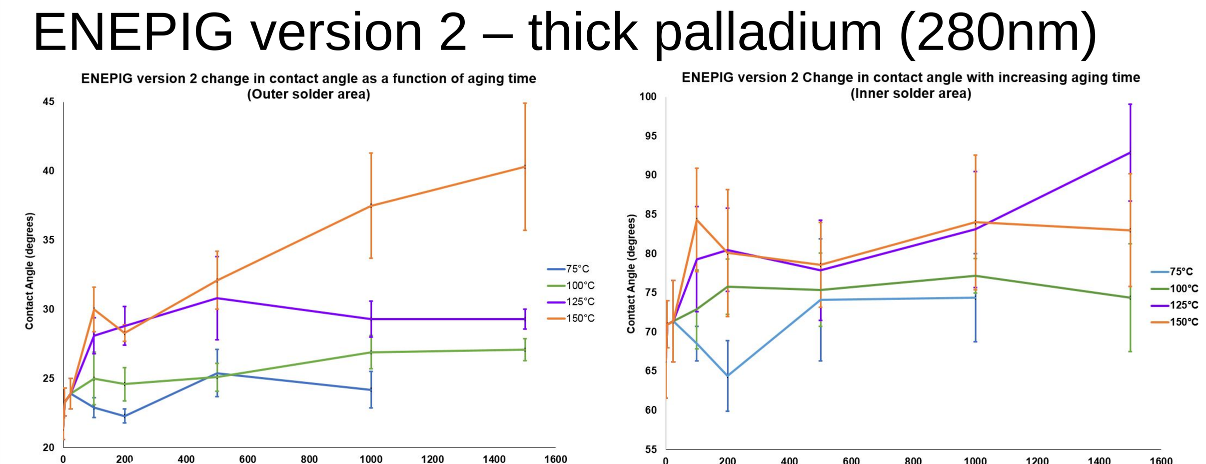
Wettability - ENEPIG V1 – thin PD (10 nm)

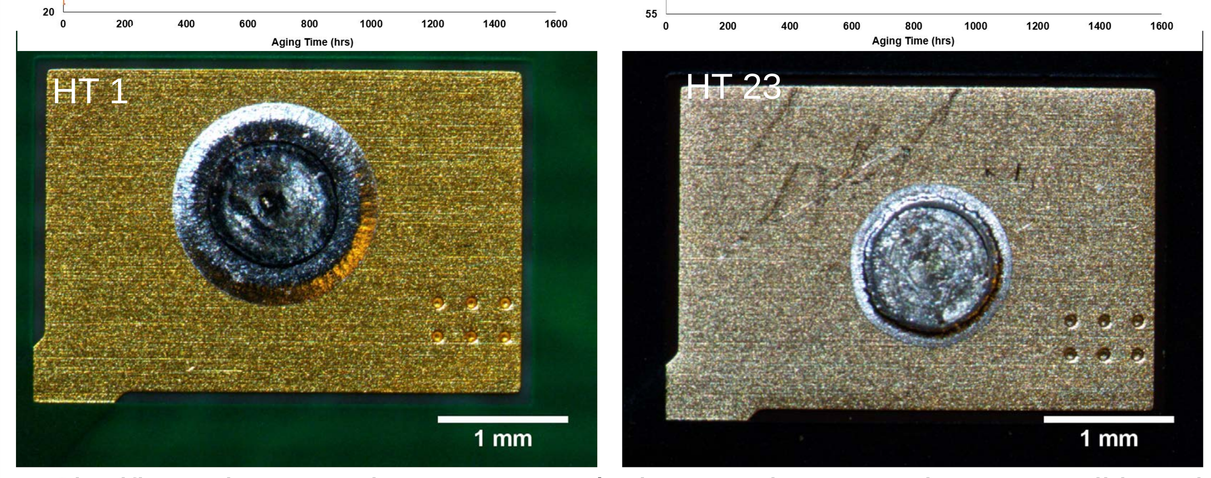




- Significant increase in contact angle seen between preconditioned boards (HT 3) and most aggressively aged boards (HT 23)
  - HT 3 had average inner and outer contact angles of 74° and 42°, HT 23 had average inner and outer contact angles of 94° and 78° respectively
- Calculated contact angle (inner ring) was highly variable throughout the aging process for all temperatures due to complex wetting behavior of preform-surface finish
  - Area of spread measurement is based on the entire solder volume coalescing into a hemispherical shape during reflow
  - Outer ring surrounding the solder likely caused by dewetting of the solder during reflow, ring contains intermetallics (IMC) which consume Sn, reducing the solder volume.
  - Since the outer ring area changes with aging time, extent of wetting/dewetting and amount of Sn consumed by IMC is also changing with annealing temperature and time, but not in a simple way. Leads to significant uncertainty in solder volume and wetting sequence
- Previous research shows similar ring behavior in cases of dewetting – solder initially wet entire area and then dewet to the final hemispherical cap with a ring of intermetallics surrounding the solder.
  - Dewetting is typically caused by spalling of intermetallics from the solder-substrate interface during the reflow process.
- Lack of IMC rings with more aggressive aging indicates the presence of an oxide layer that prevents initial wetting, IMC formation, and dewetting

ENEPIG version 2 – thick palladium (280nm)



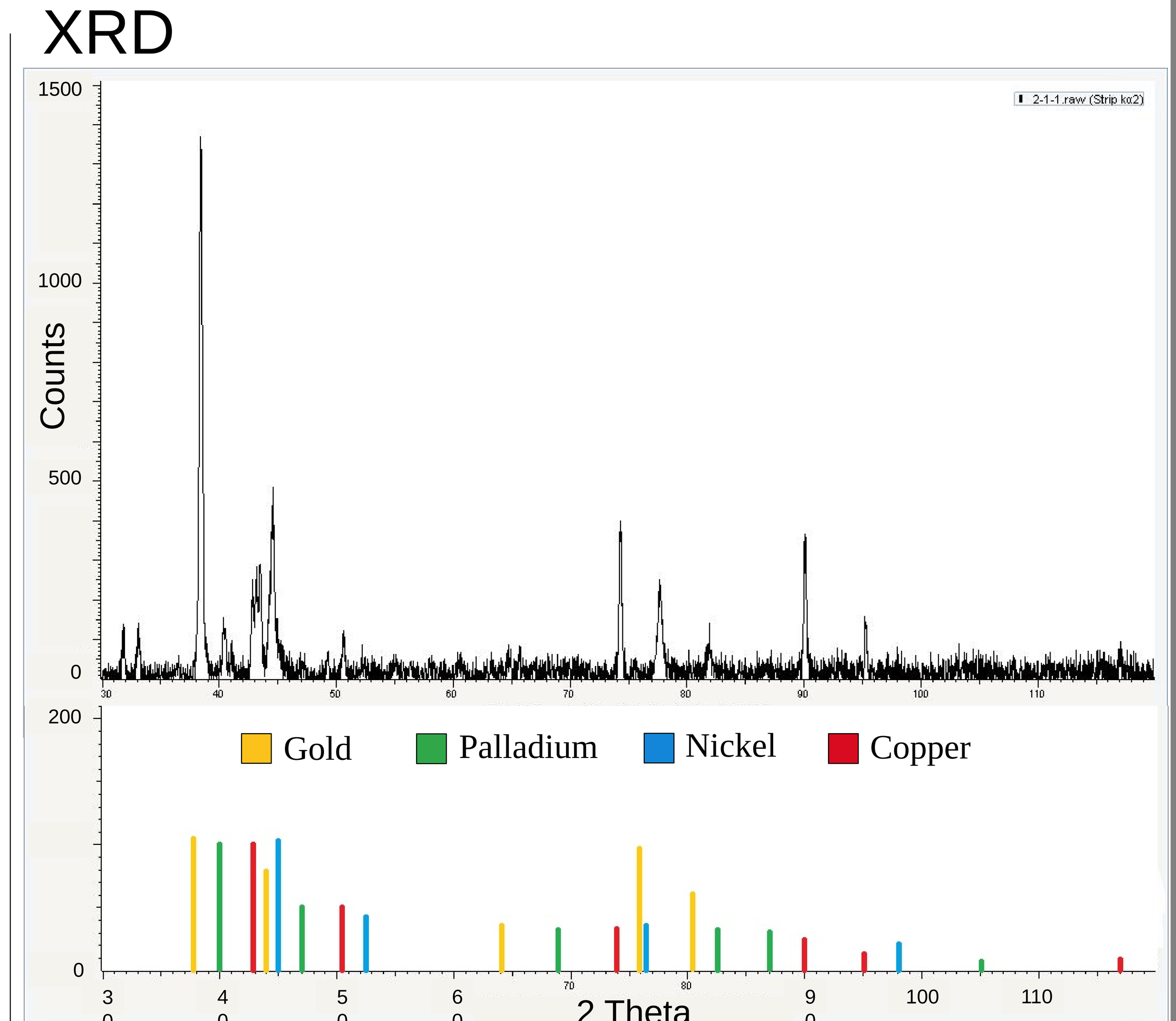


- Significant increase in contact angle is seen between the preconditioned boards (HT 3) and the most aggressively aged boards (HT 23)
  - HT 3 had average inner and outer contact angles of 71° and 24°, HT 23 had average inner and outer contact angles of 83° and 40°, respectively
- Contact angle was highly variable throughout the aging process for all temperatures due to the wetting behavior not following the same hemispherical cap assumption seen in V1 – but more extreme
  - Solder did not form a hemisphere of any kind and visually looks like the outer ring rises above or is the same height as the center.
- Previous assumption of wetting behavior is too simple causing large variation and uncertainty in contact angle measurements; a definitive critical non-wetting time cannot be determined with these experiments.
- Lack of dewetting in V2 with thicker Pd aligns closely with results seen in *"Role of wetting front in dewetting of liquid solder drop on Cu thin films"*[2].
  - Thicker Cu layer caused surface energy of the system to prevent dewetting when intermetallics spalled off the solder-substrate interface
  - Thinner Cu layer could not overcome loss of energy caused by spalling and dewetting occurred
  - Direct parallels cannot be drawn to this work without further investigation using profilometry and evaluating the two cases in cross-section to determine if V1 shows dewetting or some other phenomenon and if intermetallic spalling is present in both cases

[1] C.K. Chung, Y.J. Chen, C.C. Li, C.R. Kao; *The critical oxide thickness of Pb-free reflow soldering on substrate*, Thin Solid Films 520 (2012) 5346-5352

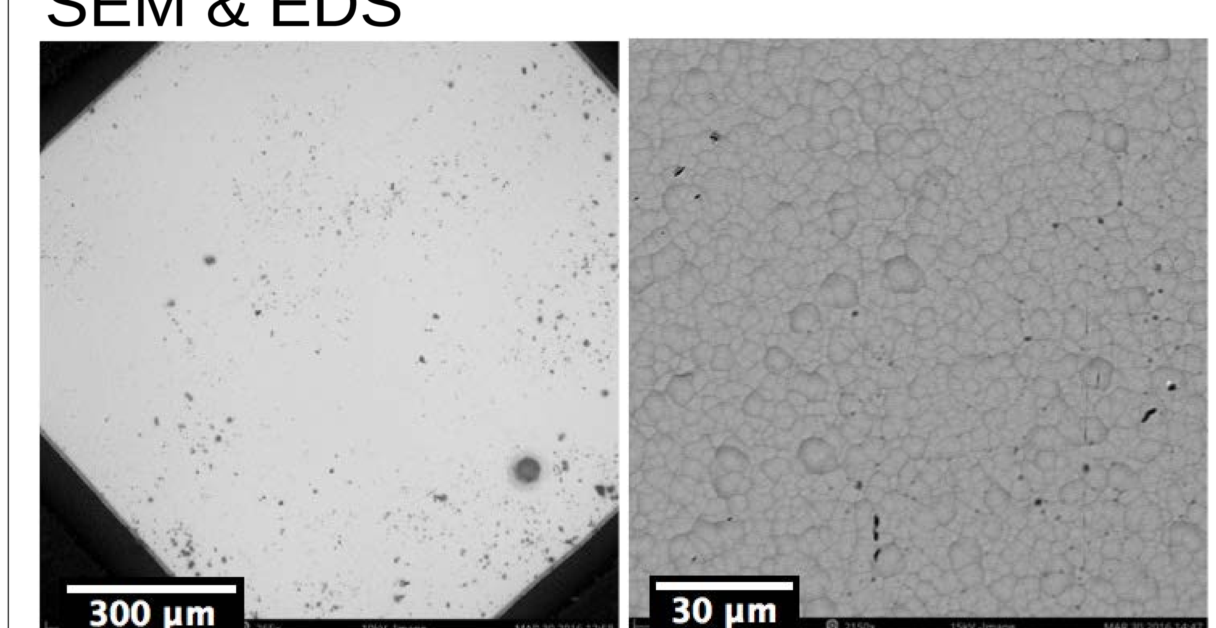
[2] W. Liu, L. Zhang, J.K. Shang. Role of Wetting Front in Dewetting of Liquid Solder Drop on Cu Thin Films, Journal of Materials Science and Technology, (2010), 26(3), 200-205

XRD



- XRD diffraction slit was open to minimum allowed width of 1 mm
- Area of the surface finished pads is approximately 0.5 mm^2
- XRD could not capture a scan of the pad without scanning the surrounding epoxy circuit board.
- Noisy peaks with a large amorphous region was seen in the XRD scan
- A few peaks match separately, however, there is no definitive match between the PDFs of the surface finish elements and the XRD.
- XRD provided no reliable characterization data due to interference from the circuit board material.

SEM & EDS



SEM micrographs of surface finish bond pads with (a) overall pad view and (b) surface grain structure features

- SEM used to understand wetting profile for soldered samples to determine contact angle measurements
- EDS not able to detect changes in underlying composition. Variability seen in composition and in elements other than Au, Ni, Pd, and Cu meant that this was not a useful characterization tool.

Conclusions

- Both versions of ENEPIG (V1-thin Pd and V2-thick Pd) showed an overall increase in contact angle from the preconditioned boards to the boards that were thermally aged most aggressively (longest time at the highest temperature) but were highly variable.
- Wetting behavior of the solder did not follow the hypothesized hemispherical drop profile making the contact angle measurements themselves not useful for assessing the effect of thermal aging
- Measurements of the wetting-dewetting profiles were useful.
- V1 ENEPIG showed solder wetting and then dewetting during the reflow, V2 ENEPIG did not
- V2 showed an unusual solder droplet shape after reflow.
  - This could be due to the different palladium thicknesses seen in the two different surface finishes, but requires more characterization to identify a mechanism
- XRD and EDS were not found to be useful in understanding the effects of aging on diffusion and oxidation

Recommendations

- Further investigation needed into the causes of wetting-dewetting phenomenon seen during reflow. Analyses of the reaction fronts (outer and inner rings) and the wetting droplet profiles are needed using profilometry, cross-sectional SEM and optical microscopy.
- High speed, in-situ monitoring of solder melting, wetting, dewetting needed
- Auger depth profiling of the surface finish needed to characterize oxidation as a function of thermal aging