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Structural integrity and thermal stability of low emissivity electroplated Au on 304L Stainless steel

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Outline

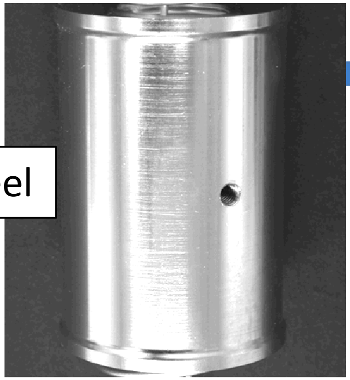
•Background and Motivation

•Materials and Experiments

•Experimental results

•Summary and conclusions

Design a low emissivity device for thermal management

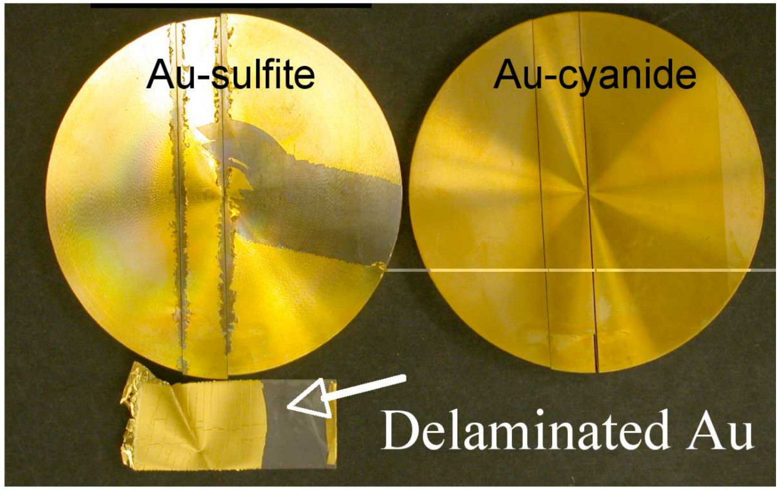


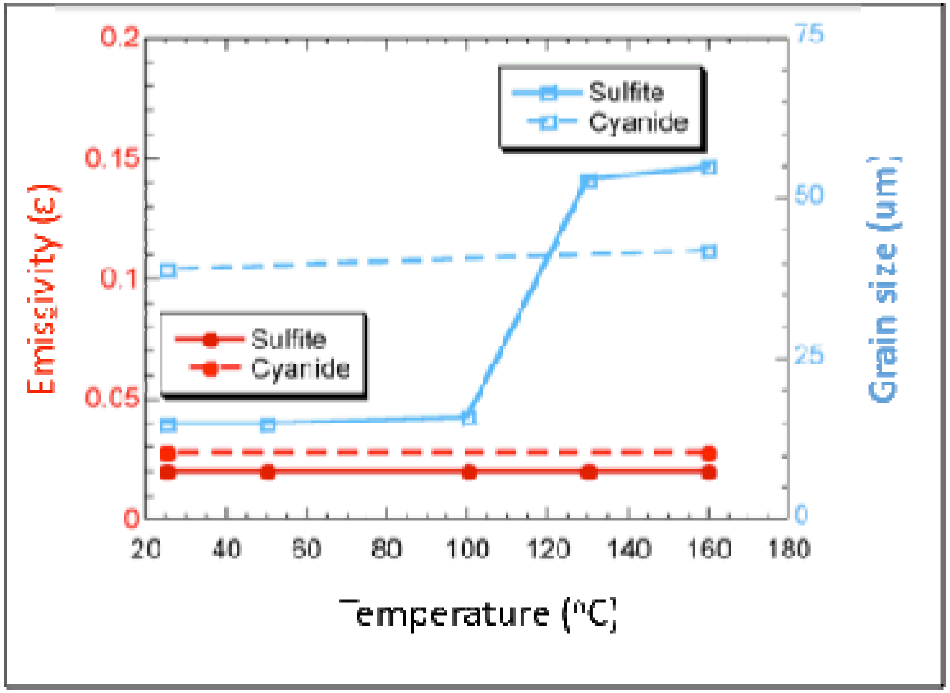
Lower thermal emissivity by applying metal coating

Metal	Exp. ε	Ideal ε
Stainless Steels	0.12 (SNL)	0.098
Nickel	0.04	0.033
Aluminum	0.02	0.019
Silver	0.015	0.016
Copper	0.027	0.015
Gold	0.02 (SNL)	0.007

Electroplate thin gold coating on 304L stainless steel

Developed an electroplating process for a Au coating on 304L with strong adhesion and stable microstructure





Courtesy from A. Morales

Au-sulfite exhibits weak adhesion and unstable microstructure at ≥100°C, not suitable for elevated temperature applications.

Down selected Au-cyanide process based on its good adhesion and thermally stable microstructure

1. Caustic clean 15 min
2. DI Water rinse
3. 30% by volume Nitric acid pickle dip 30 sec
4. DI Water rinse
5. Anodic etch in 30% sulfuric acid @ 150 ASF (Amps per Square Foot) for 3 minutes *
6. DI Water rinse
7. Woods Nickel strike @ 50 ASF for 5 minutes**
8. DI Water rinse
9. Gold plate in the Au-cyanide bath @ 1.5 ma/cm2 for 10 minutes = 1 µm
10. DI Water rinse
11. Blow dry with Dry Nitrogen

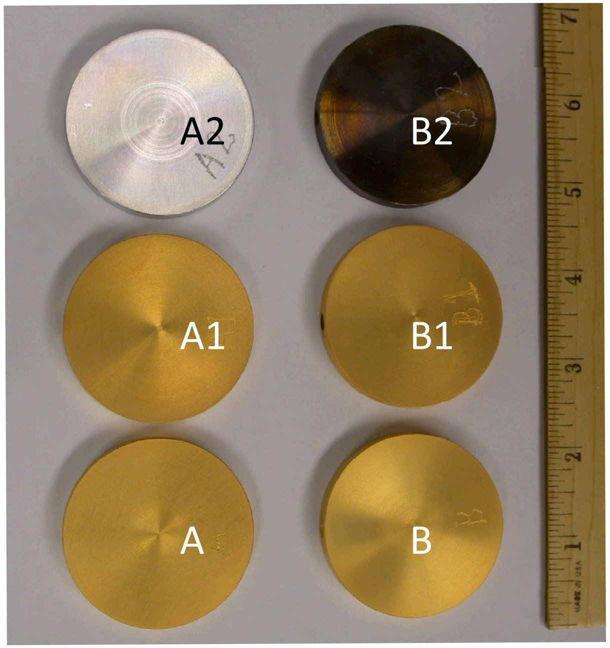
* The H_2SO_4 etch removes all foreign contaminants, including residues from air, mechanical handling and surface oxide from thermal annealing

** Wood Ni-strike serves as adhesion enhancer

Thermal aging experiment and test matrix

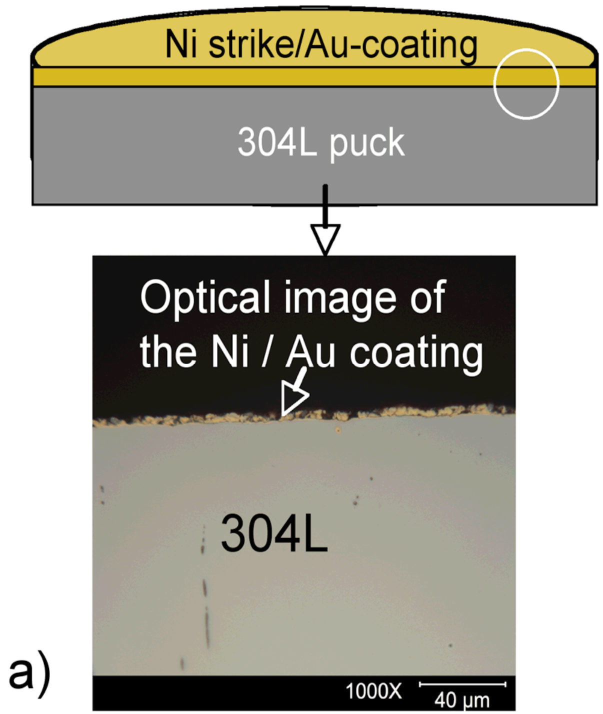
A2: 304L blank -As-received 304L puck	**B2: 304L blank** -Prebaked 304L puck at 400°C/5 days in air
A1: Au plated -As received 304L puck -Electroplated Au w/Ni strike	**B1: Au plated** -Pre-baked 304L puck at 400°C/5 days in air -Electroplated Au w/ Ni strike
A: Au plated - As-received 304L puck - Electroplated Au w/ Ni strike - Bake the entire component at: 100°C for 7 days at 10x-4 Torr	**B: Au plated** -Prebaked 304L puck at 400°C/5 days -Electroplated Au w/ Ni strike -Bake the entire component at: 100°C/7days at 10x-4 torr
A1+190°C: Au plated - As-received 304L puck - Electroplated Au w/ Ni strike - Bake the entire component at: 190°C for 7 days at 10x-4 Torr	**B1+190°C: Au plated** -Prebaked 304L puck at 400oC/5 days -Electroplated Au w/ Ni strike -Bake the entire component at: 190°C/7days at 10x-4 torr
A1+250°C Au plated - As-received 304L puck - Electroplated Au w/ Ni strike - Bake the entire component at: 250°C for 63 days at 10x-4 Torr	**B1+250°C Au plated** - As-received 304L puck - Electroplated Au w/ Ni strike - Bake the entire component at: 250°C for 63 days at 10x-4 Torr
A2+Ni strike -As-received 304L puck -Electroplated Ni-strike	**B2+Ni Strike** -Prebaked 304L puck -Electroplated Ni-strike

Sample geometry and dimension

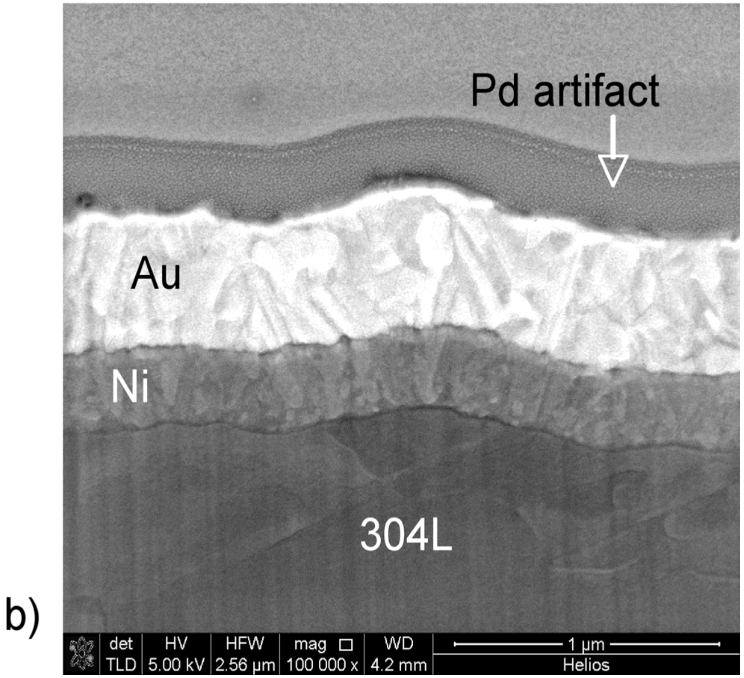


The annealing temperature and annealing duration are ambient to 250°C and 7days to two months, respectively

The electroplated Au and Woods Ni on 304L substrate system, fabricated at SNL-CA, meets the technical requirements



SEM image of the FIB cross section



•The system consists of thin electroplated Au (500nm) and Ni (300nm) on a 304L stainless steel substrate.

•Adhesion along the Au-Ni and Ni-304L interfaces appeared to be continuous

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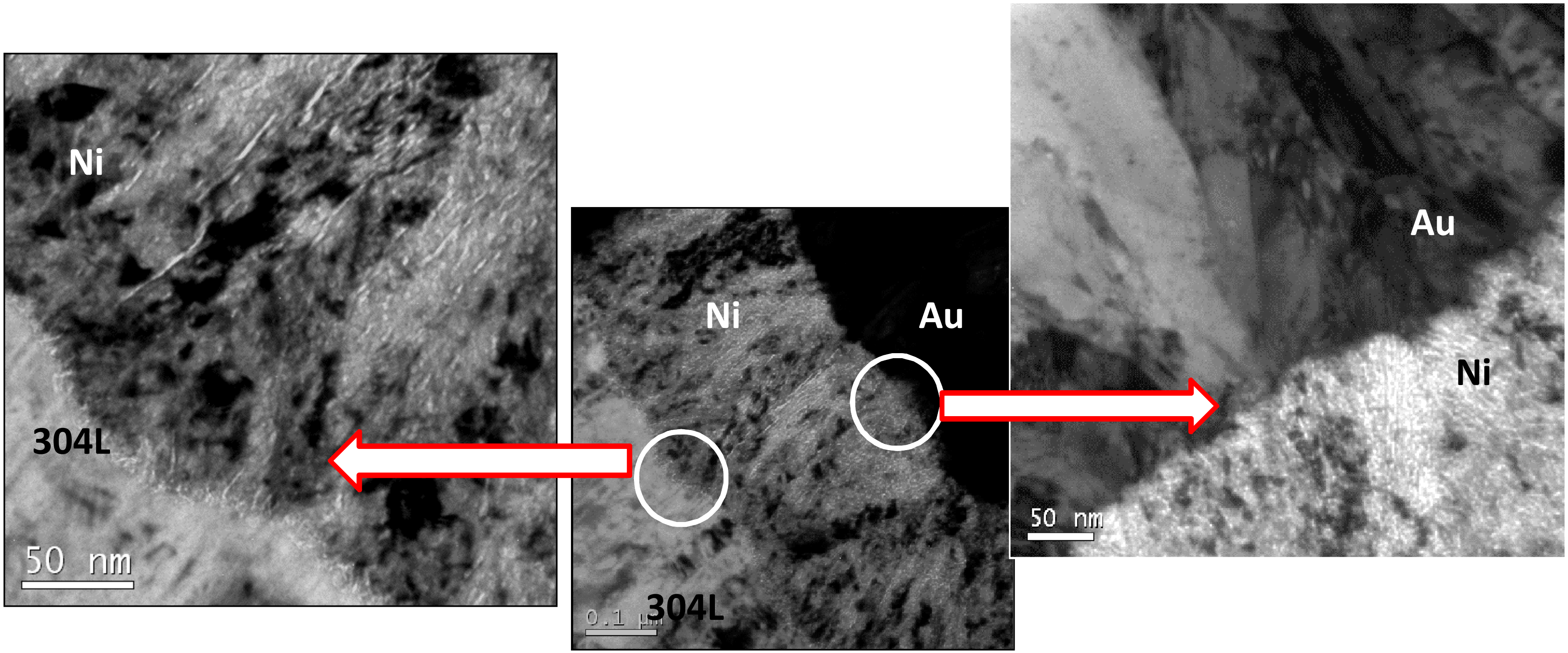
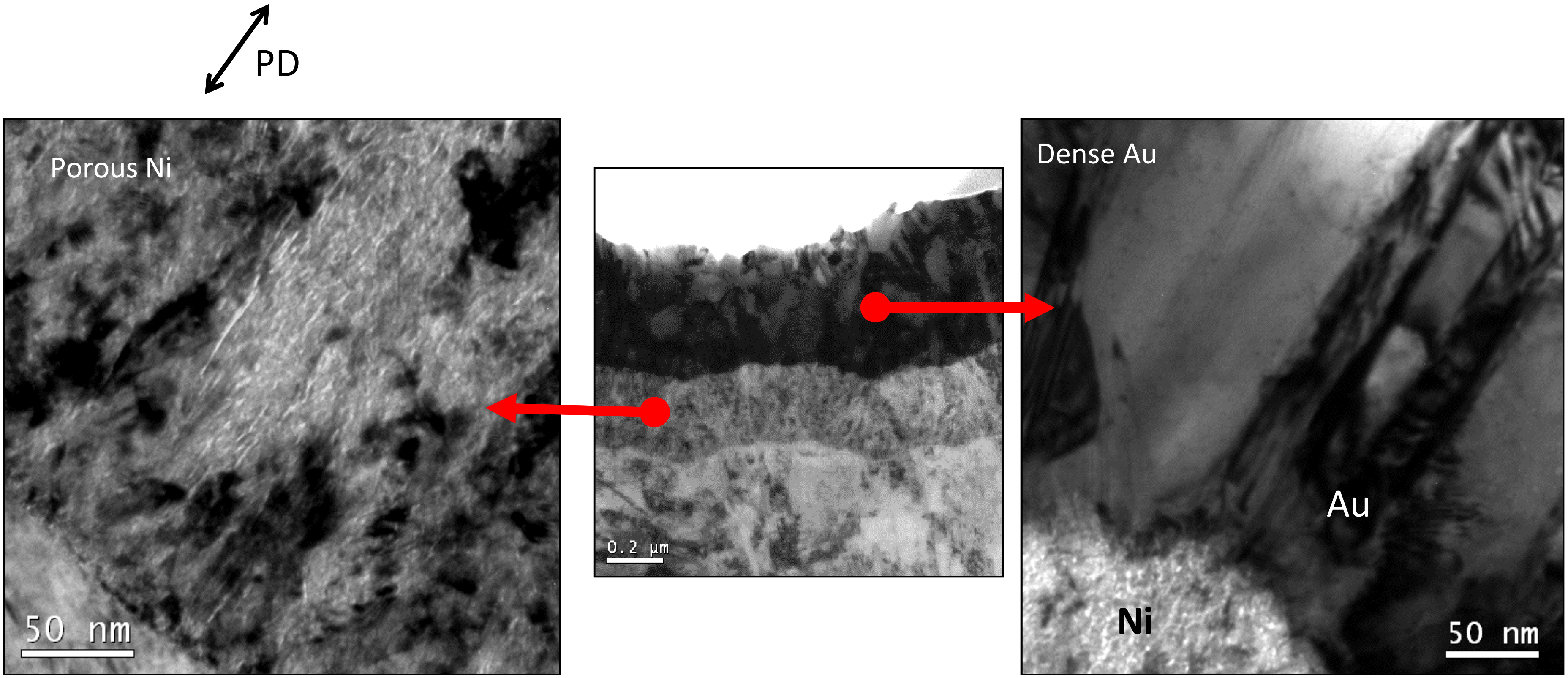
Good adhesion at the as-plated Au-Ni and Ni-304L interfaces was confirmed using TEM images



Good adhesion derived from the intertwined Au, Ni and 304L at the interfaces

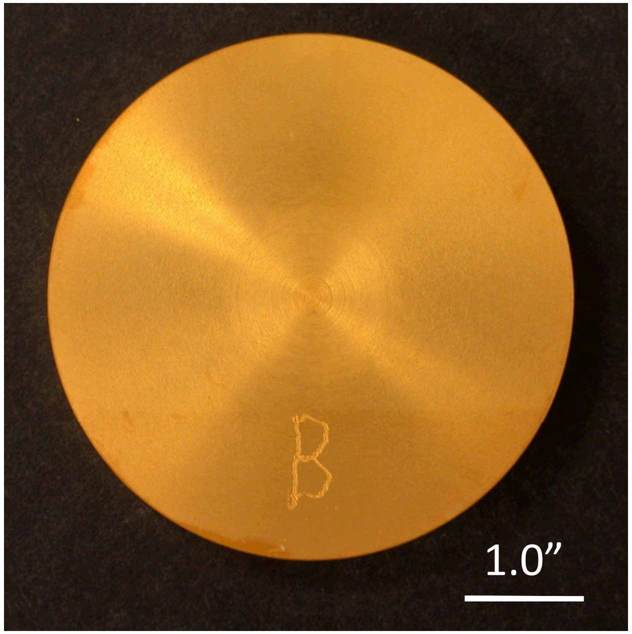
The Woods Ni-strike contains high density of nanopore stringers while the Au coating is fully dense



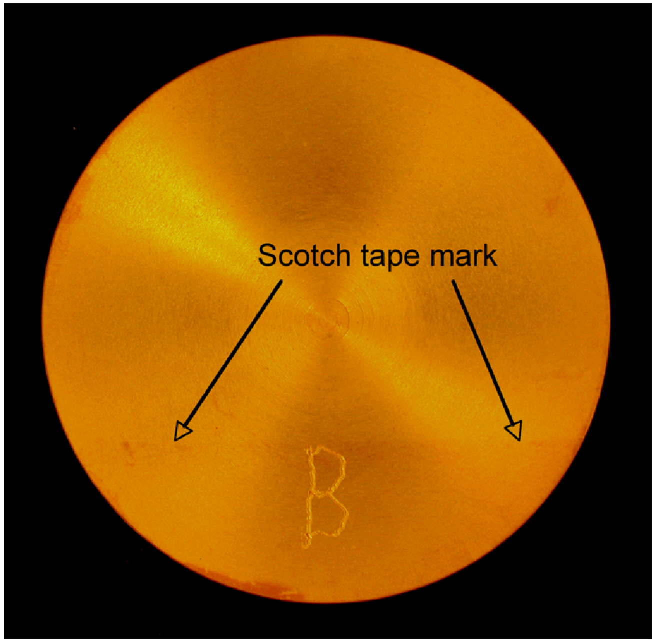
The long axis of nanopore stringers appears to follow the plating direction, along the columnar grain boundaries.

Qualitative Scotch tape test show good adhesion of the thermally annealed electroplated Au on Ni/304L

Before tape test



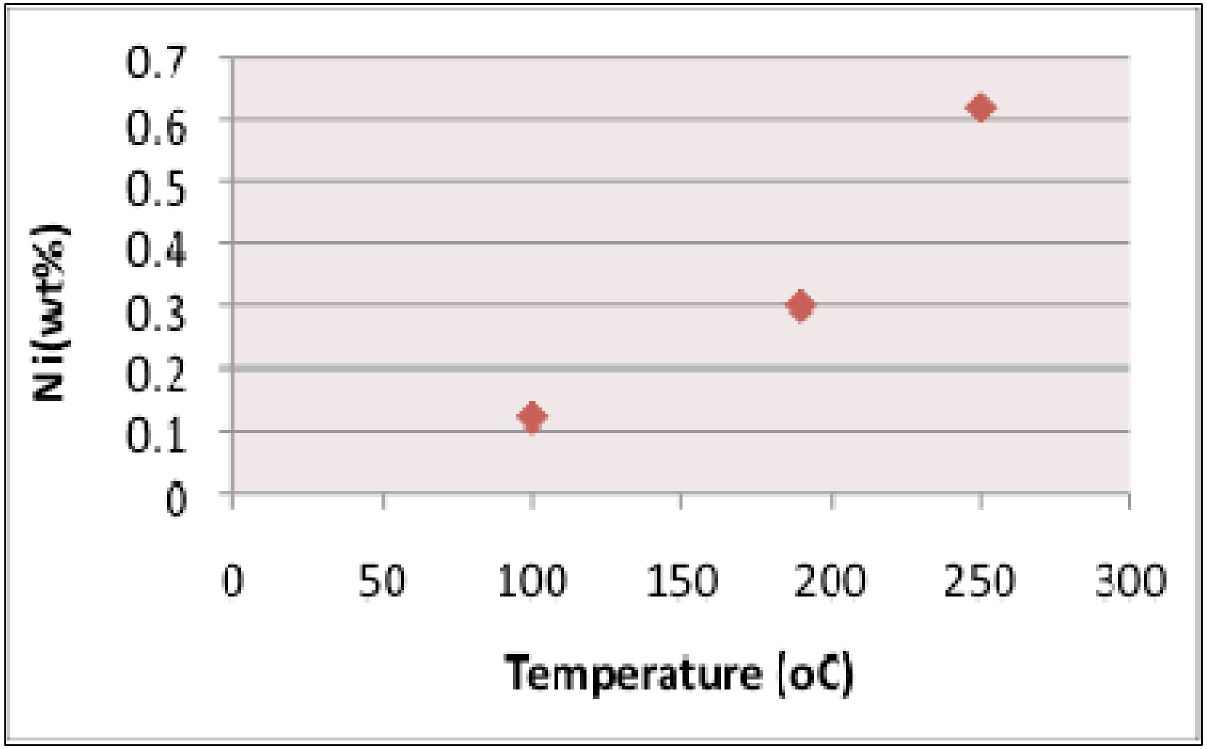
After tape test



The Scotch tape test shows the Au coating remains intact on Sample B, subjected to a 100^{o}C/7days annealing

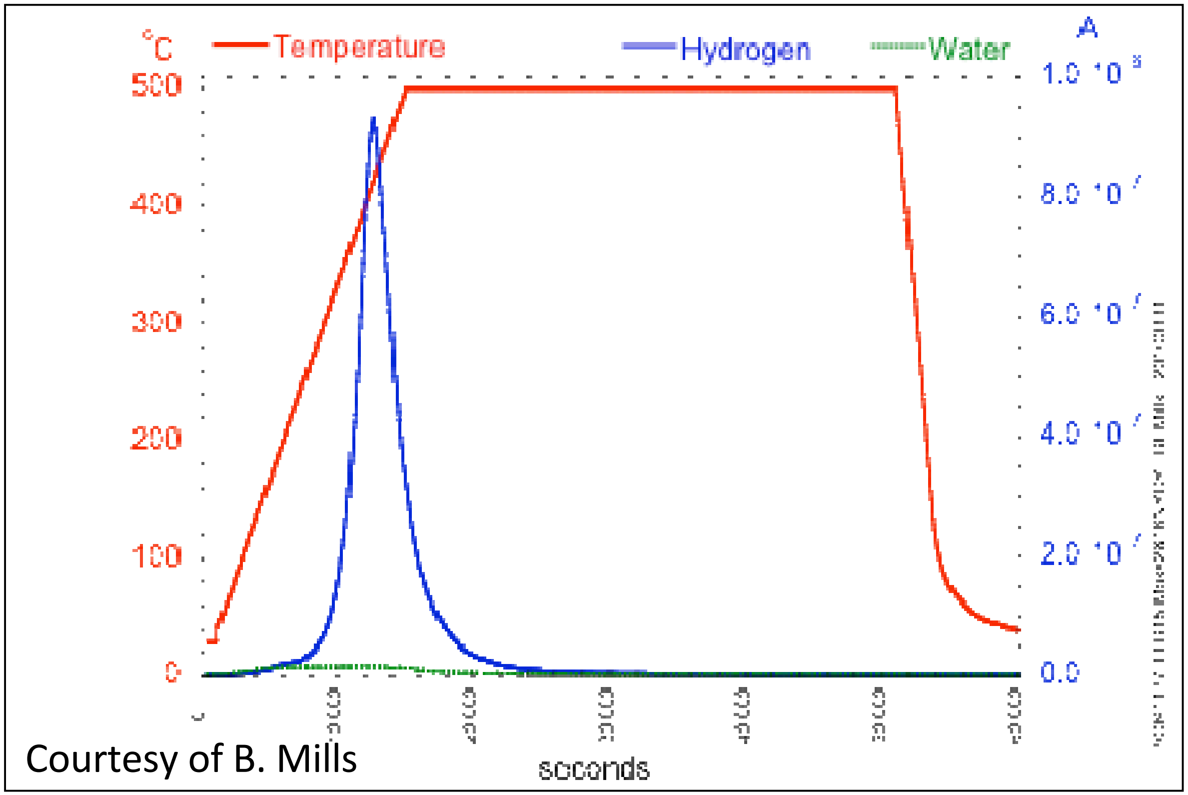
Thermal evolution of the Au/Ni/304L system and its implication to structural integrity

Ni diffusion to the electroplated Au coating is evident at ≥ 190^{o}C



Ni concentration in Au increases with annealing temperature

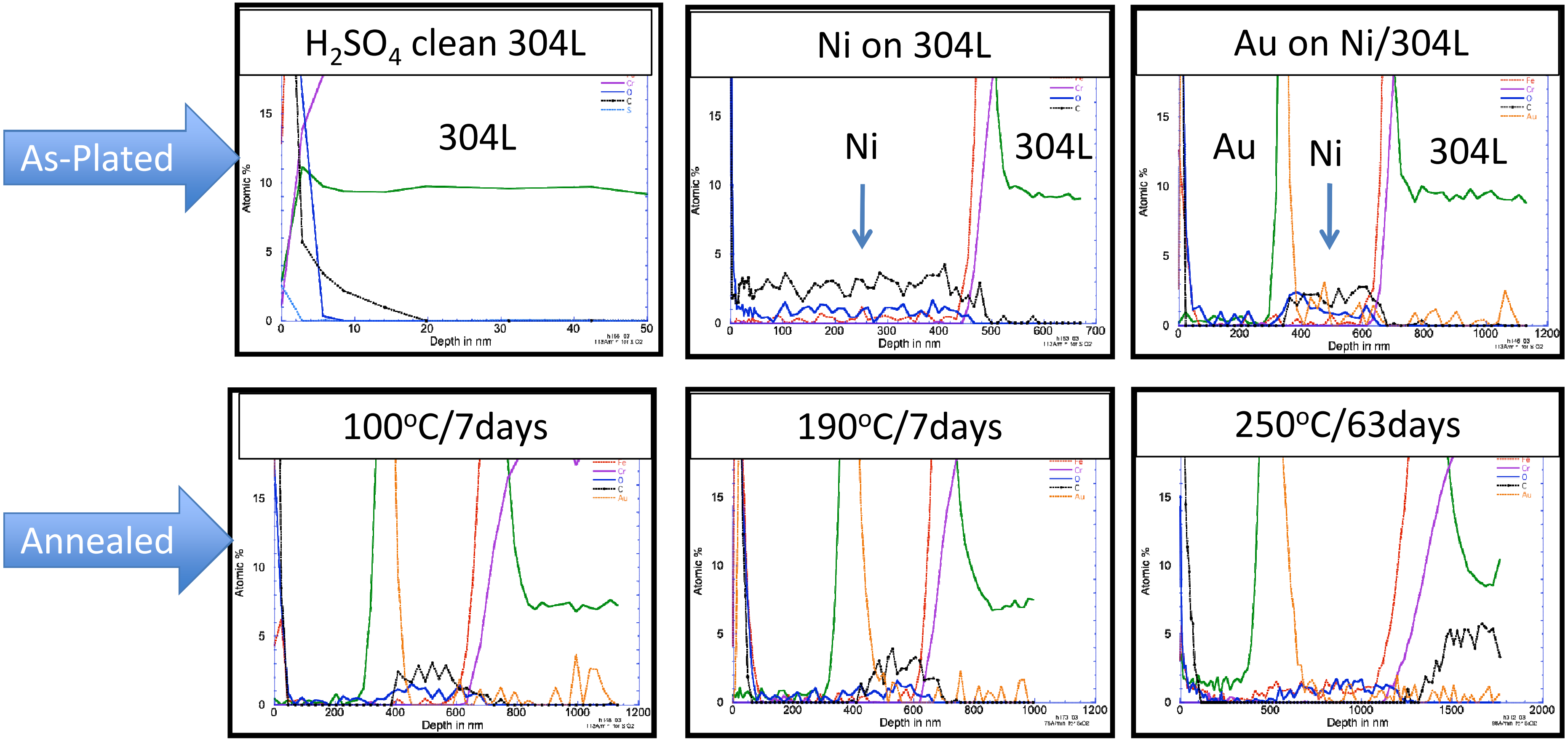
Mass spectrum shows the presence of hydrogen and water in the current Au/Ni/304L system



Courtesy of B. Mills

Water and hydrogen, entrapped during the electroplating process, outgas upon heating.

Auger depth profiles show elevation of C (black) and O (blue) in all the Ni layers except for the sample 250°C/63days

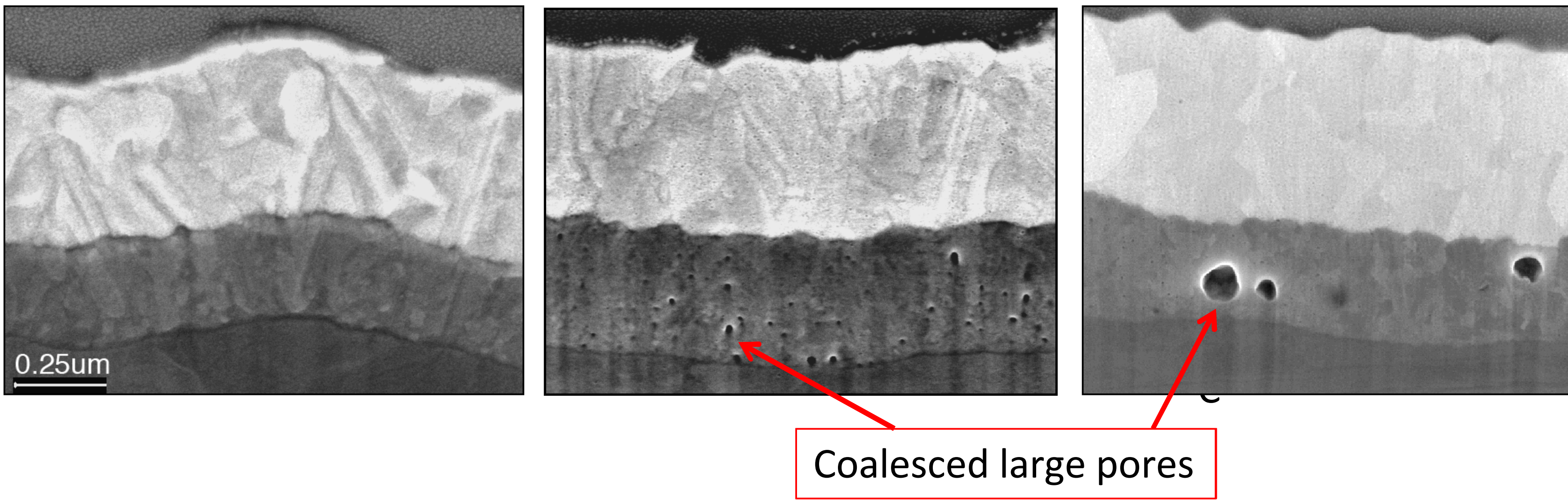


- Oxygen elevation observed in all samples.
- Carbon diffused back to 304L substrate at 250°C/63 days.
- H presence is unable to be verified by Auger technique

Implication to the strucure integrity?

FIB images show the size of the pores in the Woods Ni increases with annealing temperature

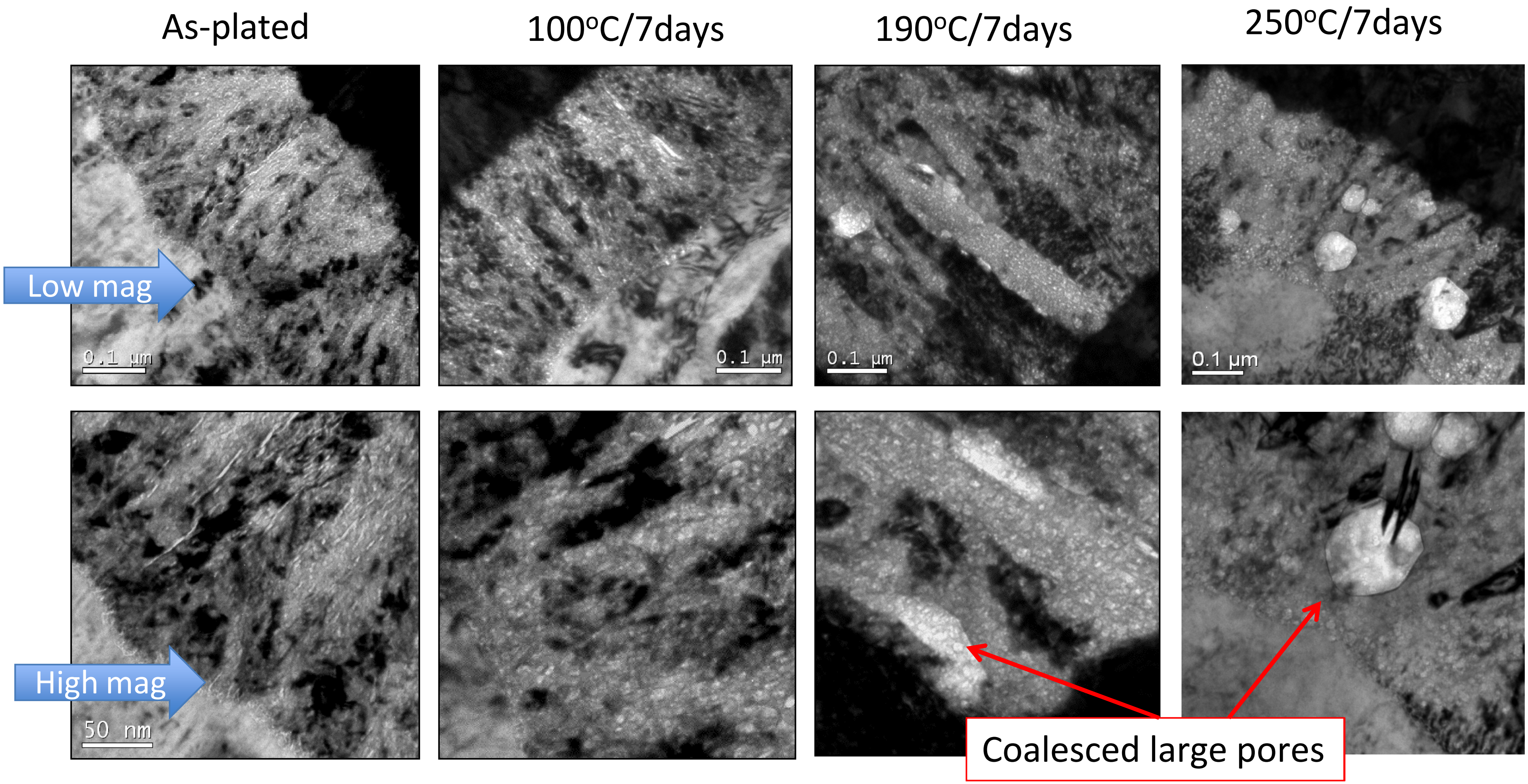
As-plated | 190^{o}C/7days | 250^{o}C/63 days



B

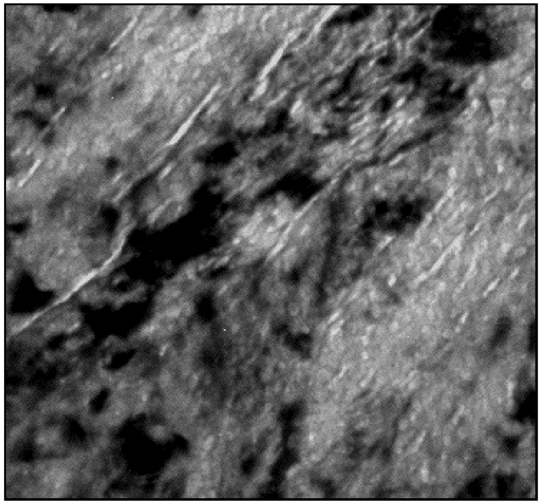
- The size of the large pores approaches one half of the Ni thickness at 250^{o}C/63 days
- The population of the visible pores decrease with annealing temperature suggesting Oswarld Ripening.

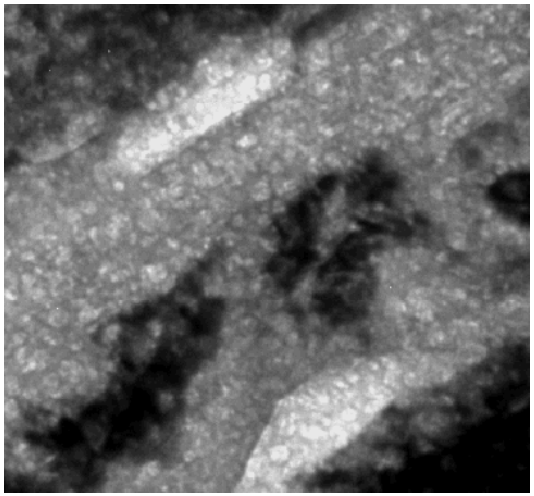
Thermal evolution of pore structure, size and morphology, are also evident in the TEM images.

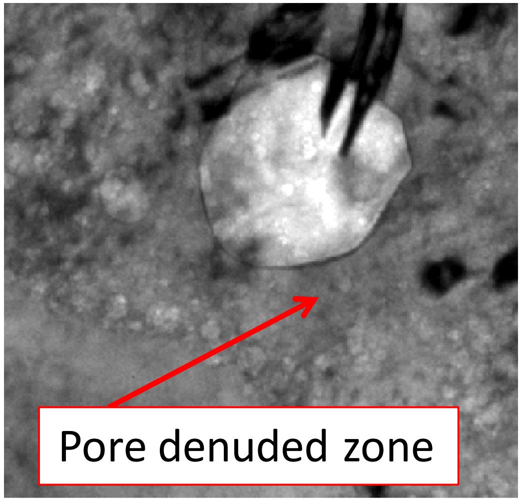


- Upon thermal annealing, the nanopore stringers spherodized and coalesced.
- Pore coalesces preferentially along the grain boundaries.

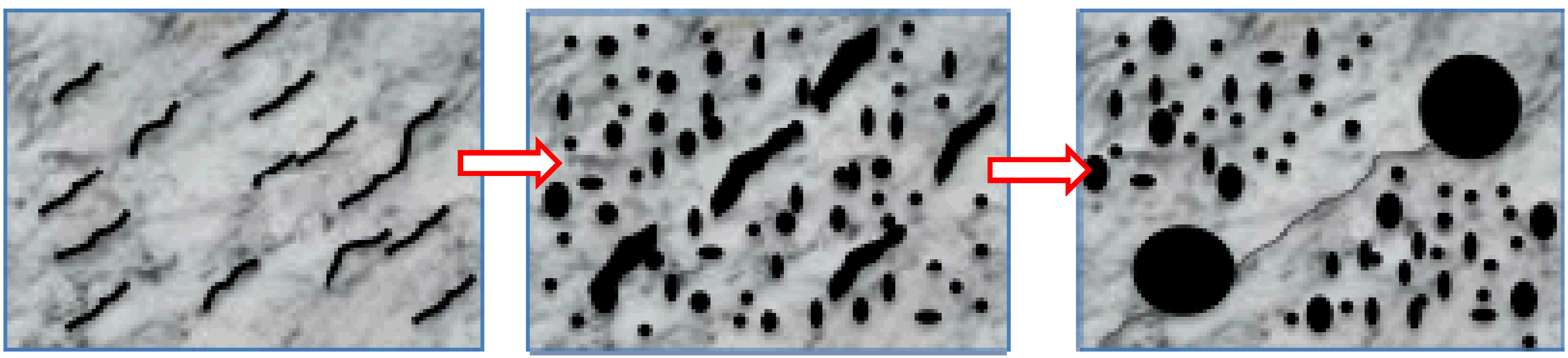
Schematic for thermal evolution of nanopore stringers







Schematic



Nanopore stringers | Pores spherodize & coalesce | Large pore w/ denuded zone

Summary and conclusions

•The Au/Ni/304L system exhibits good adhesion and the Au structure in general is thermally stable throughout all annealing cycles, up to 250°C/63 days.

•The electroplated Woods Ni contains nanopore stringers, water and H which are thermally unstable above 100°C/7 days . This thermal instability is related to the pores/H-bubbles diffusion, spherodization , coalescence and the outgassing of water and hydrogen.

• The thermally unstable Ni makes the system vulnerable to outgassing and loses structural integrity possibly leading to Ni delamination, therefore, premature system failure (see the Figure below).

•A post-Ni-plating prebaking, prior to Au plating, may be necessary to remove water and hydrogen in the Ni for outgasing mitigation.