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13. SUPPLEMENTARY NOTES

14. ABSTRACT

The overall objective of this effort was to provide state of the art hardware to the Compact Power Group at the Army Research Laboratory (ARL) in Adelphi, MD, for use in their development of a quiet, fuel-efficient, compact, power generator based on thermophotovoltaic (TPV) power conversion. As part of this program, novel emitters were designed, fabricated, and tested. A thermal system model was developed using ray tracing. We also developed a procedure for assembling individual emitter tiles into a more complex shape.

15. SUBJECT TERMS
thermophotovoltaic, generator, selective emitter, thermal system model

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Joint Development of Thermophotovoltaic Concepts

Contract No. W911NF-18-2-0142
Final Report

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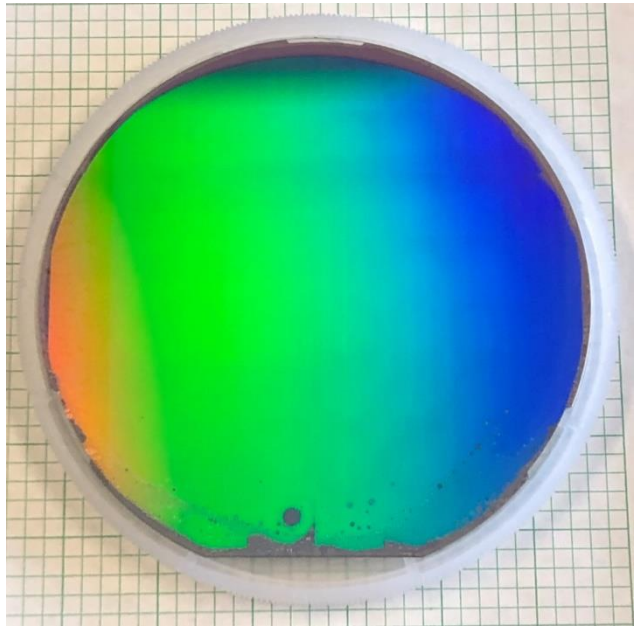
Summary

- Through modeling and experiments, we evaluated parameters for the fabrication of selective emitters based on the Pt/Al_2O_3 material system
- We evaluated metamaterial emitters and planar film-based emitters up to 1200°C,
- Accelerated lifetime testing on the best performing emitters show a lifetime of over 10,000 minutes (a week) at 900 C and over 24 hrs 1000°C
- Experimental matrix revealed some trends with lifetime, but did not find a global lifetime maximum. Design/materials still can be improved
- Developed ray tracing approach to model overall system
- Developed assembly procedure for holding 8 flat emitter panels in an octagonal assembly

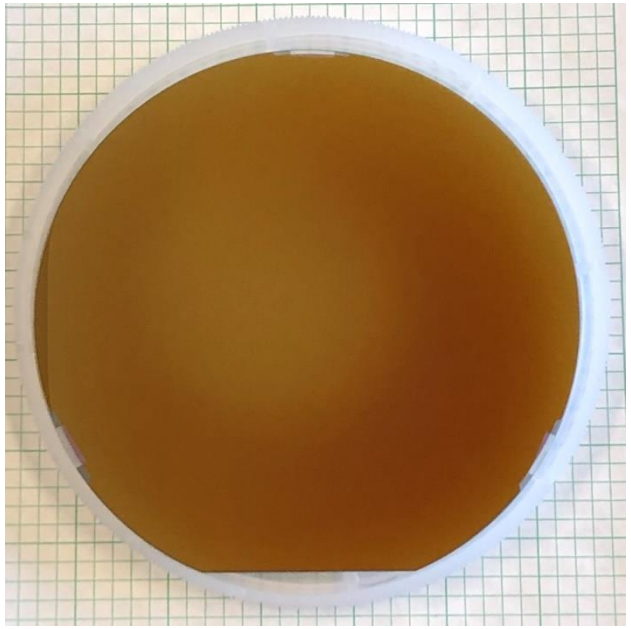
New Emitters Fabricated NILT Wafers

- **Wafers with dot pattern (6)**
- **Un-patterned wafers (2)**

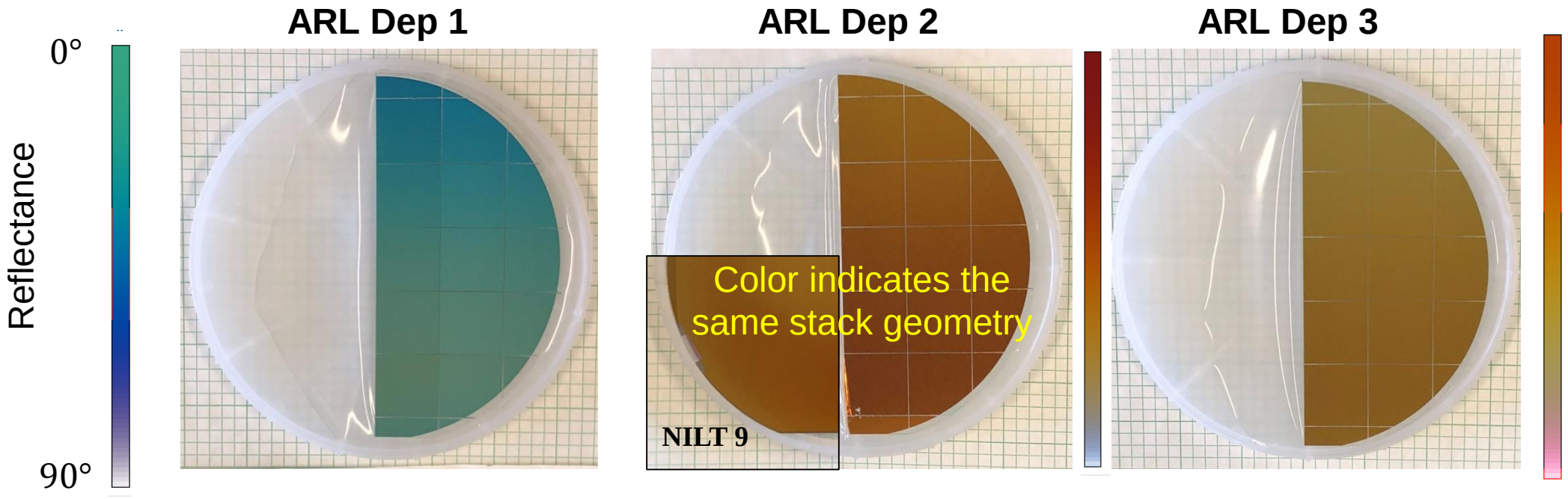
NILT Wafer 1 (metasurface)



NILT Wafer 9 (no metasurface)

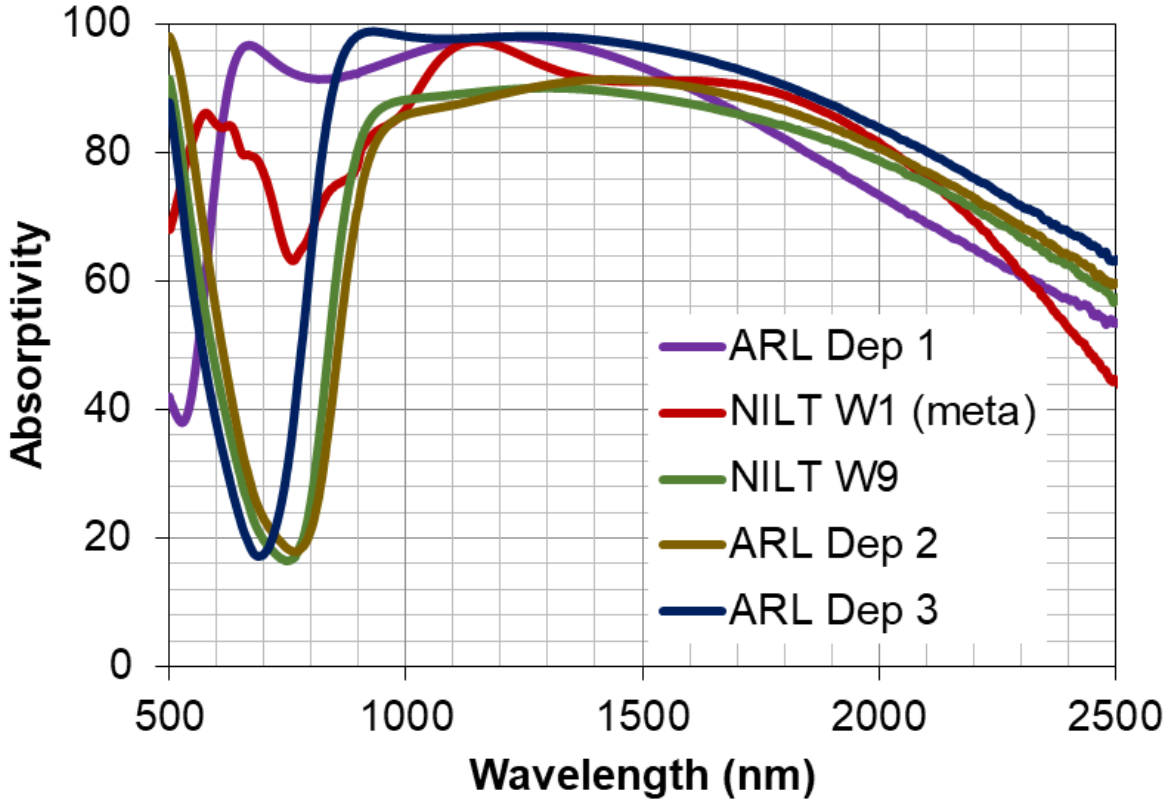


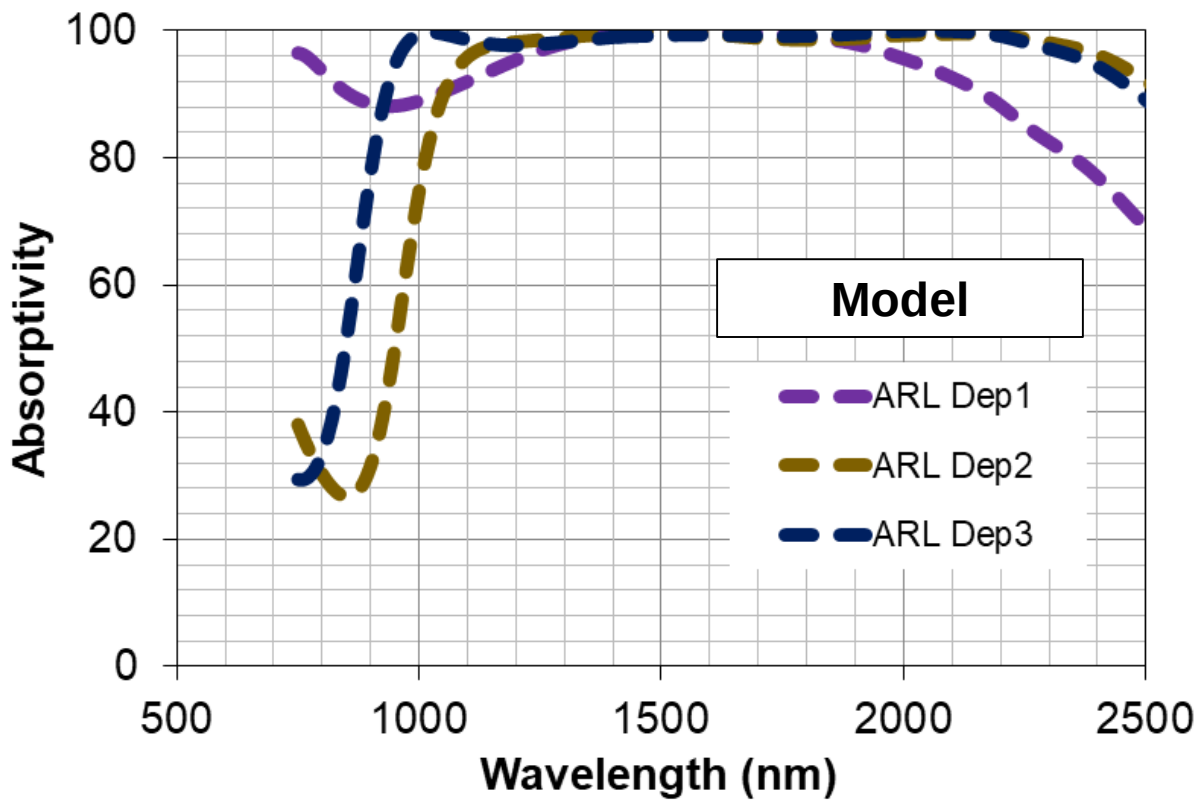
New Emitters Fabricated
ARL Planar Deposition



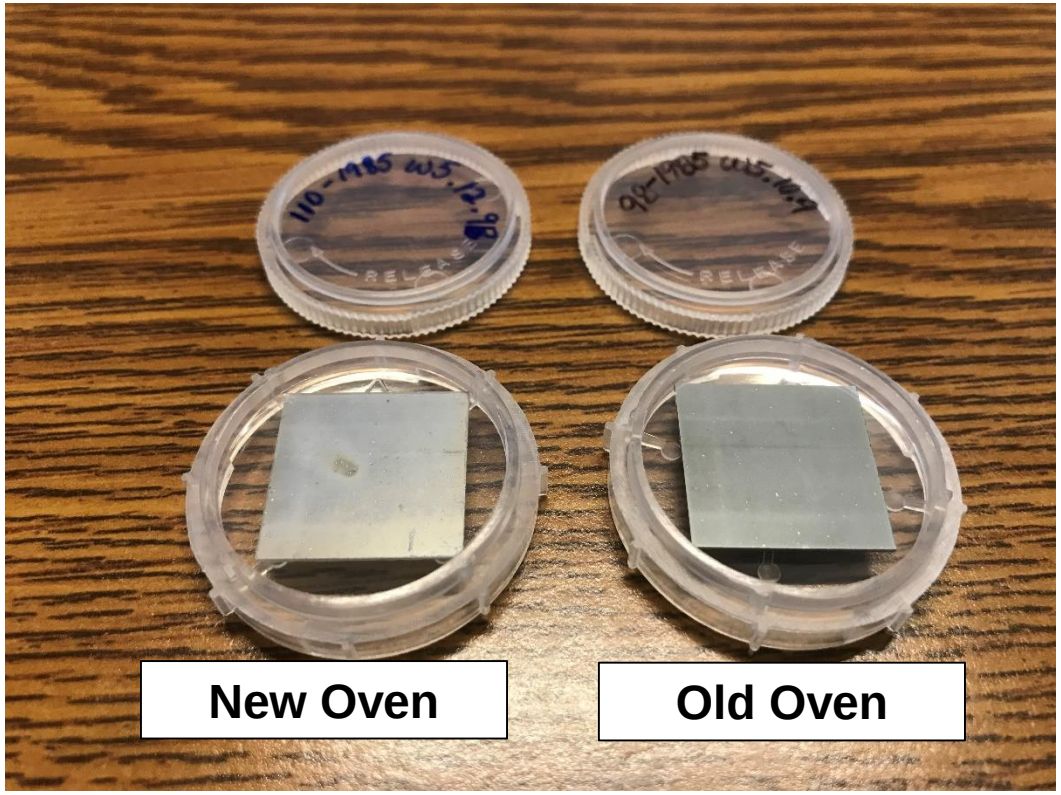
Spectral Comparison of Selective Emitters

- **All emitters, including metasurface and non-metasurface, have high emissivity between 1-2 microns.**
  - The metasurface (**red**) has the lowest emission at 2.5 microns
  - "Planar Design#3" (ARL dep 3, **blue**) has the highest emissivity between 1-2 microns
  - Results match model pretty well, with some disagreement between 2 and 2.5 microns



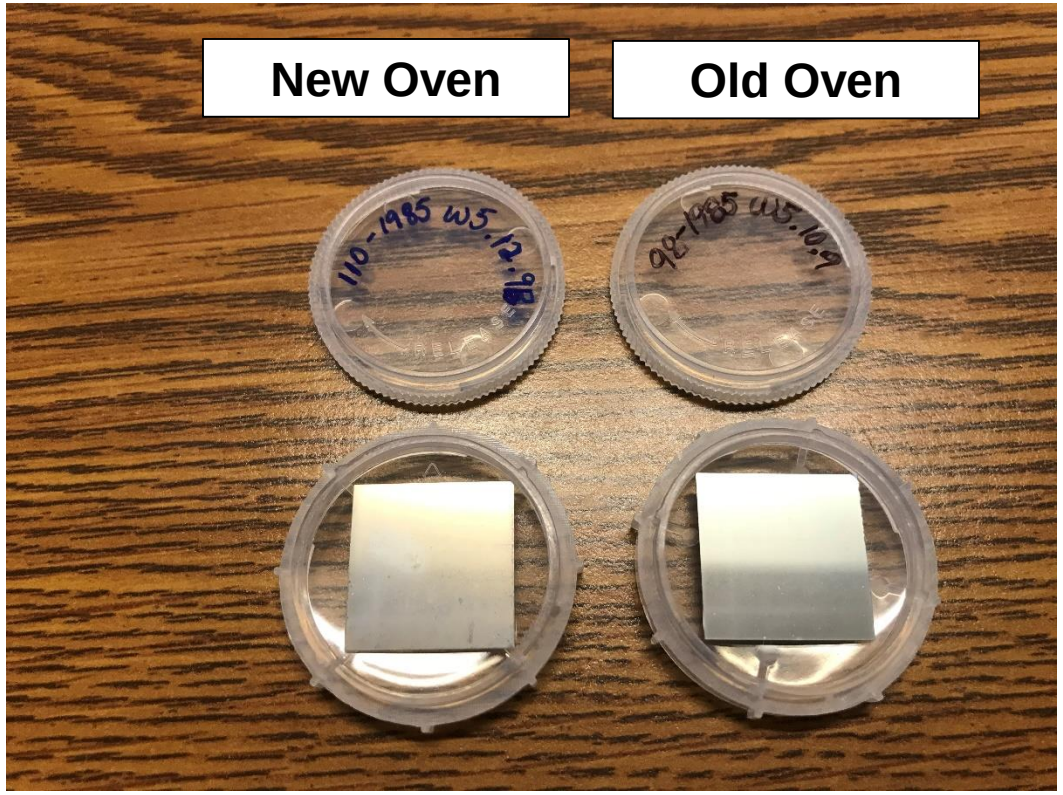


Evaluation of Furnace-to-Furnace Differences – None seen!



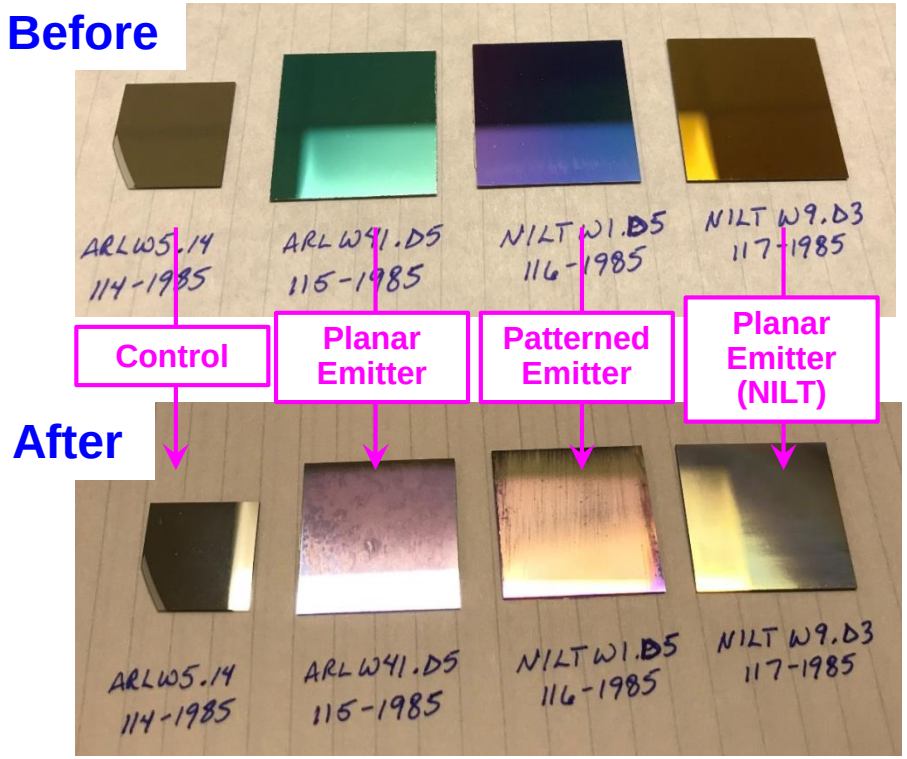
- 110-1985 W5.12.9b – New oven
- 98-1985 W5.10.9 – Old (tube furnace) oven

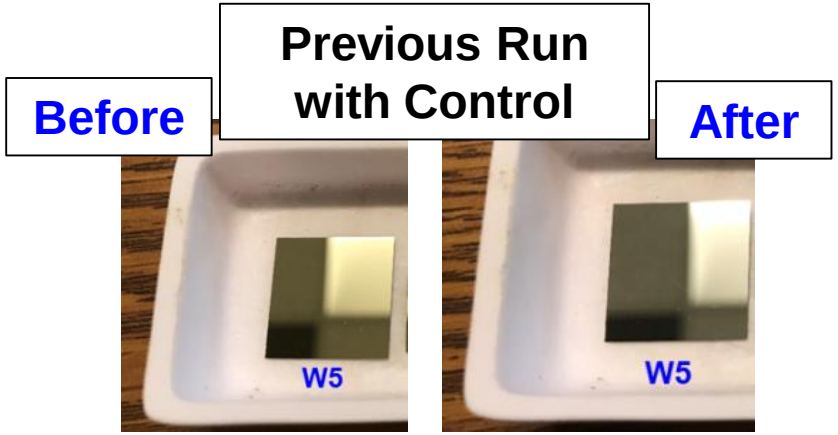
- A new furnace was required to evaluate the new samples, due to a failure of the old furnace.
- Upon visual inspection, no difference between the samples processed in different furnaces for the same time and temperature



Latest Lifetime Testing Results – 300 min @ 1000°C

- **Process 2: 10°C/min ramp to 1000°C (from '0°C'), 300min soak, 10°C/min to '0°C'**
- **Samples**
  - Control: 114-1985 W5.12.3b (sapphire)
    - **A die from this wafer was previously processed in the old furnace under same process conditions**
  - Planar Emitter: 115-1985 W41.D5.3b (ARL Dep 1)
  - Patterned Emitter: 116-1985 W51.D5.3b (NILT W1)
  - Planar Emitter: 117-1985 W59.D3.3b (NILT W9)
- **Evaluation criteria**
  - Specular reflectivity, patina, uniformity.
  - *Not looking at absolute color change, which is not correlated with IR spectrum*
- **Observations**
  - **Before**: All samples mirror-like
  - **After:**
    - Planar and Patterned samples hazy and dull. Relatively uniform of color.
    - Control sample (W5) looks fairly unaffected, as it was in previous run (images at right)
      - **This implies that the thin ~10 nm thick Pt layer is less stable than the 45 nm thick layer in W5**





Latest Lifetime Testing Results – 300 min @ 1000°C (additional images)

-

Before

Control

Planar Emitter

Patterned Emitter

Planar Emitter (NILT)

After

Latest Lifetime Testing Results – 300 min @ 1000°C (additional images)

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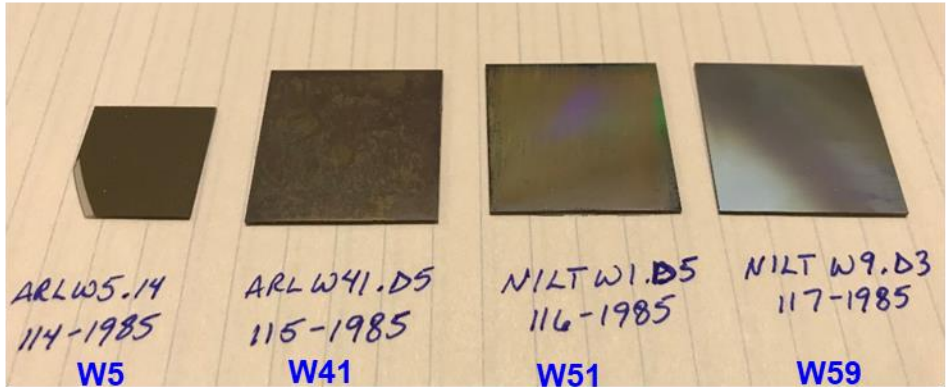
-

Ceiling background



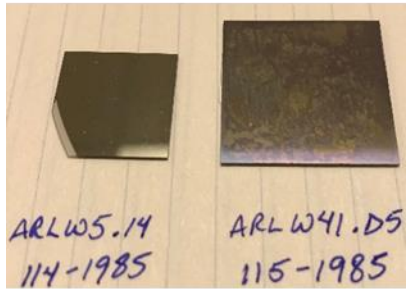
W5 (ARL) — Control

W41 (ARL Dep1) — Planar Emitter

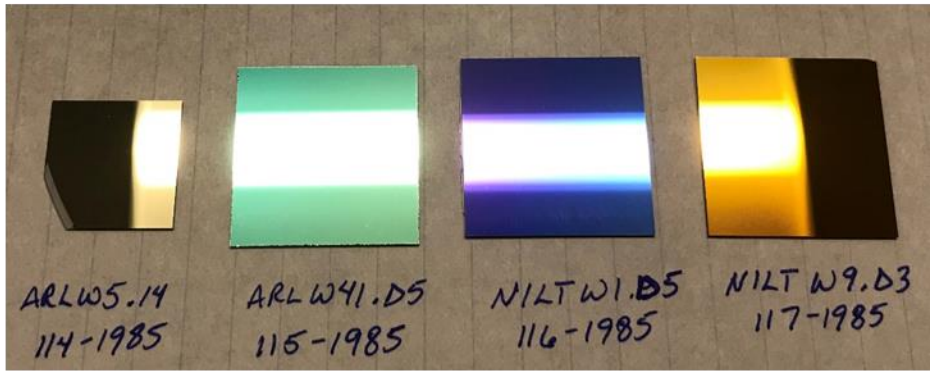
W51 (NILT W1) — Patterned Emitter

W59 (NILT W9) — Planar Emitter (NILT)

Close ups



Room lamp background (before)



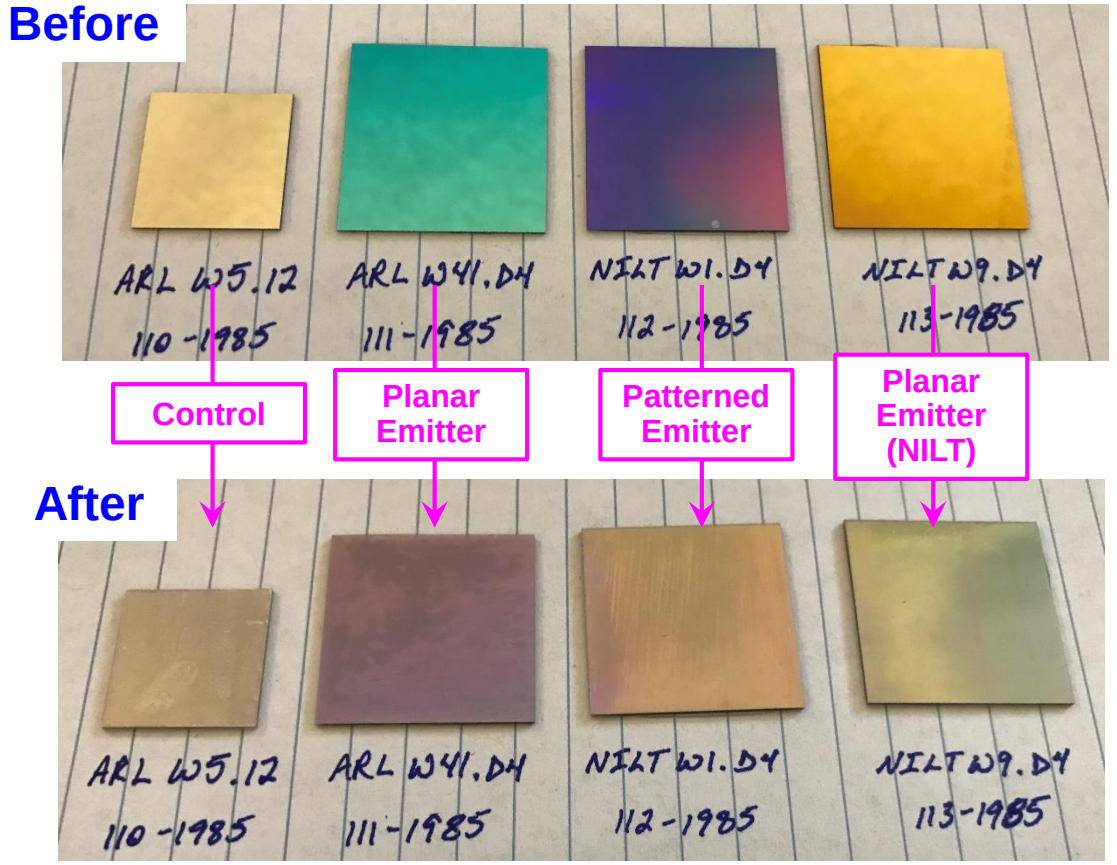
Control | Planar Emitter | Patterned Emitter | Planar Emitter (NILT)

Room lamp background (after)

Latest Lifetime Testing Results – 300 min @ 1200°C

- **Process 9b: 10°C/min ramp to 1200°C, 300min soak, 10°C/min to '0°C'**

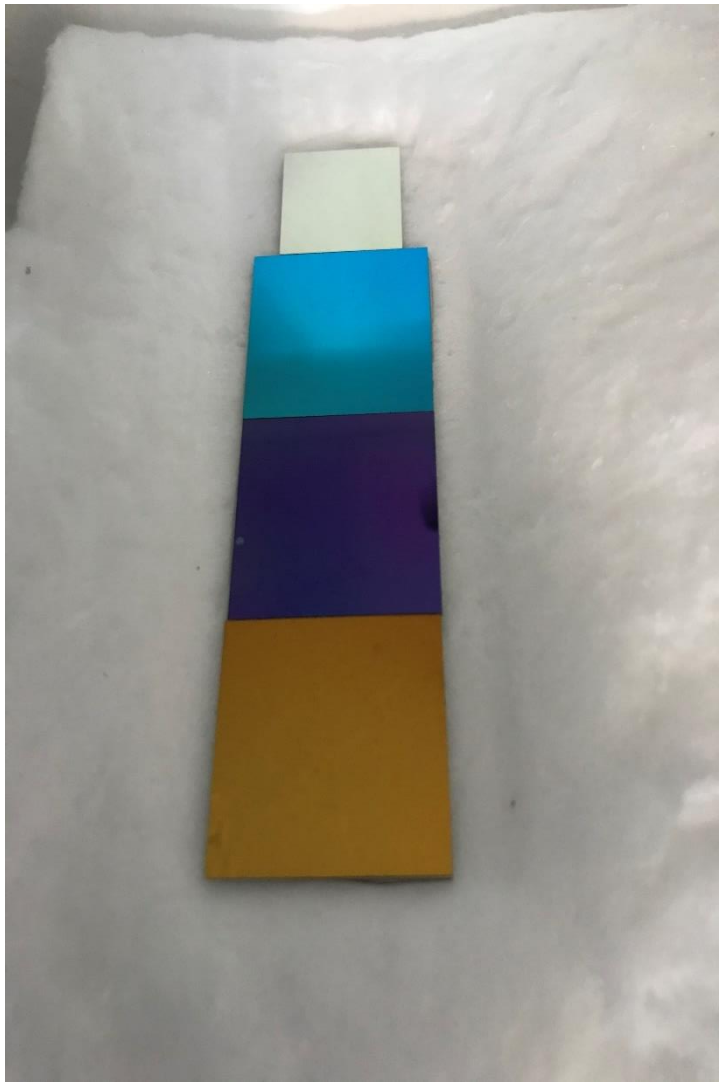
- **Samples:**
  - Control: 110-1985 W5.12.9b (sapphire)
    - **A die from this wafer was previously processed in the old furnace under same process conditions**
  - Planar Emitter: 111-1985 W41.D4.9b (ARL Dep 1)
  - Patterned Emitter: 112-1985 W51.D4.9b (NILT W1)
  - Planar Emitter: 113-1985 W59.D4.9b (NILT W9)
- **Evaluation criteria**
  - Specular reflectivity, patina, uniformity.
  - *Not looking at absolute color change, which is not correlated with IR spectrum*
- **Observations:**
  - Before: All are mirror-like
  - After:
    - All samples pretty hazy and dull. Relatively uniform of color.
    - All samples are slightly semi-transparent, indicating mass loss



Latest Lifetime Testing Results – 300 min @ 1200°C (additional images)

Physical Sciences Inc.

Before



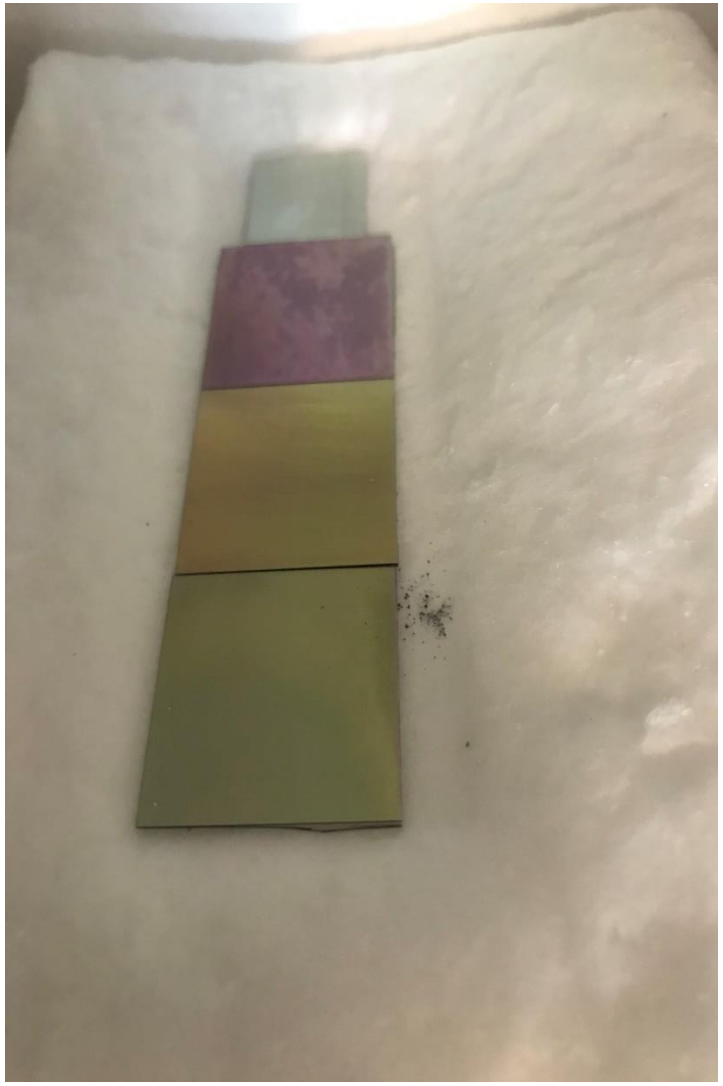
Control

Planar Emitter

Patterned Emitter

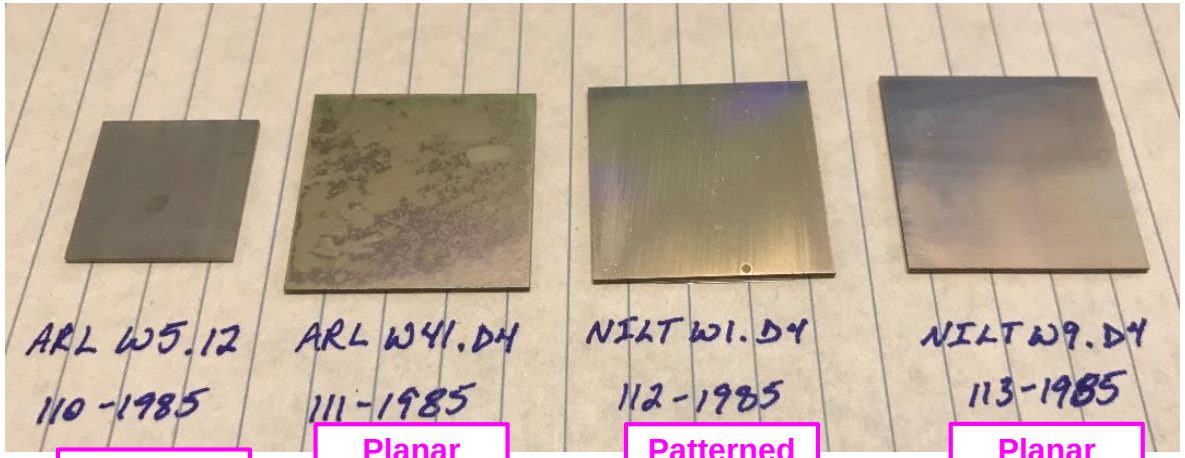
Planar Emitter (NILT)

After

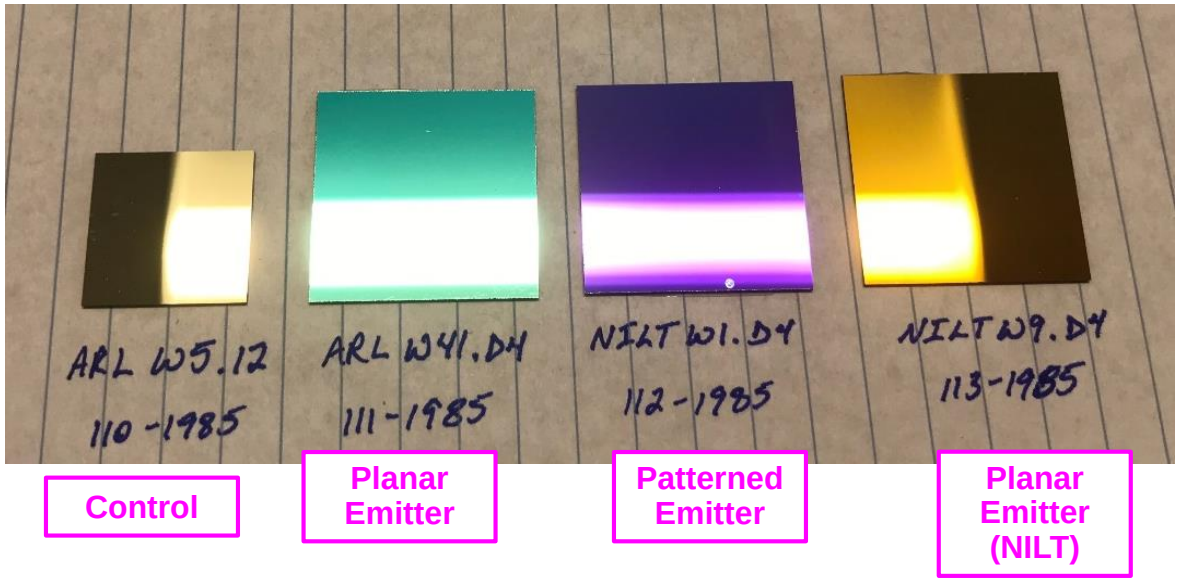


Latest Lifetime Testing Results – 300 min @ 1200°C

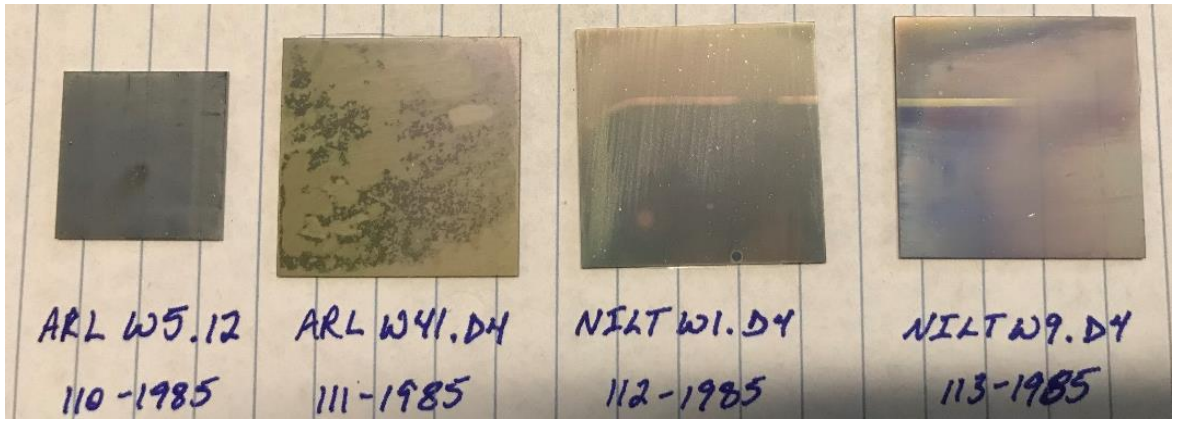
Ceiling background



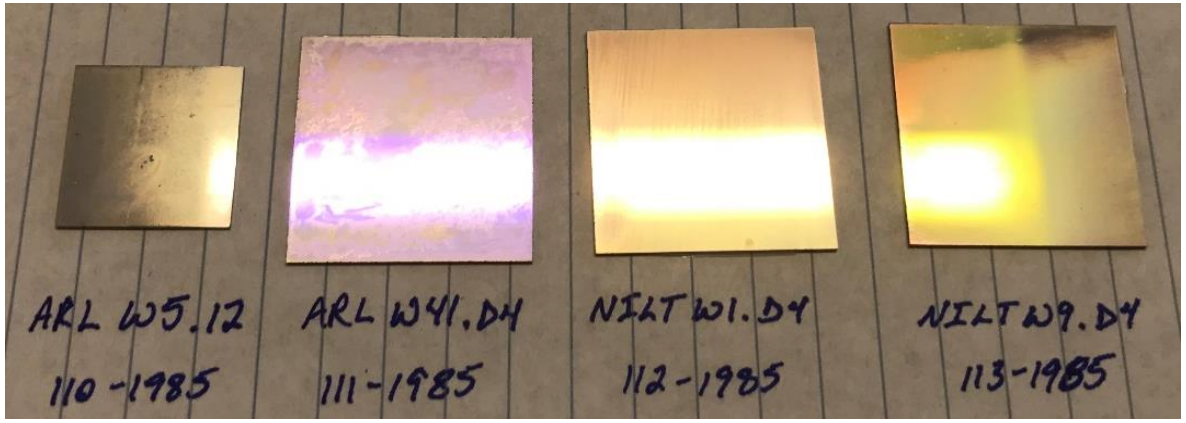
Room lamp background (before)



Near normal angle



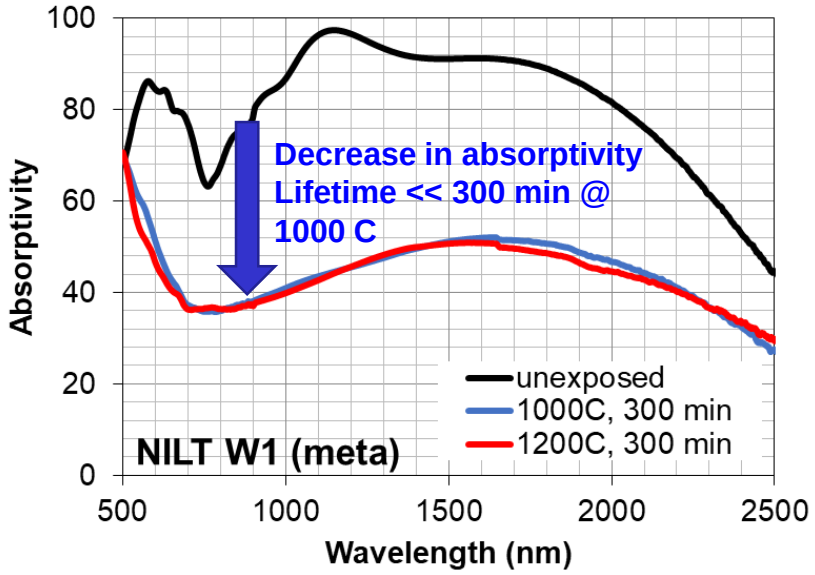
Room lamp background (after)

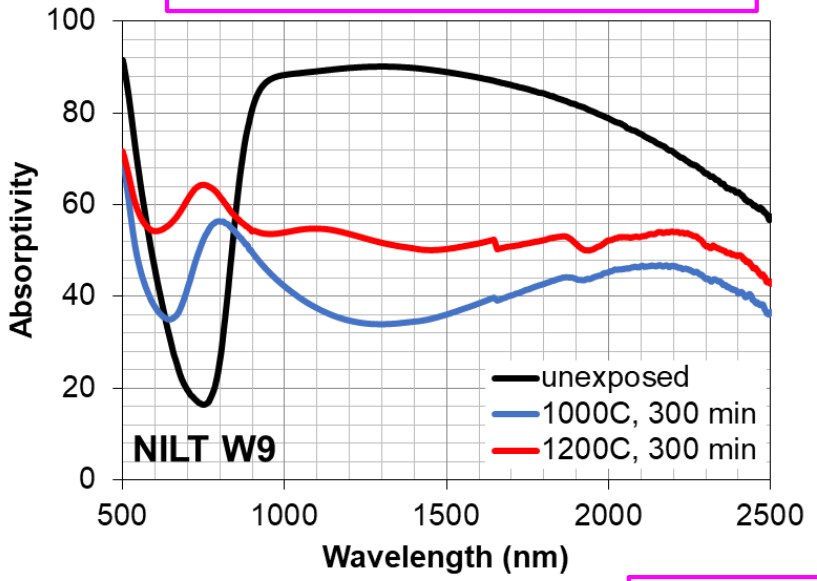


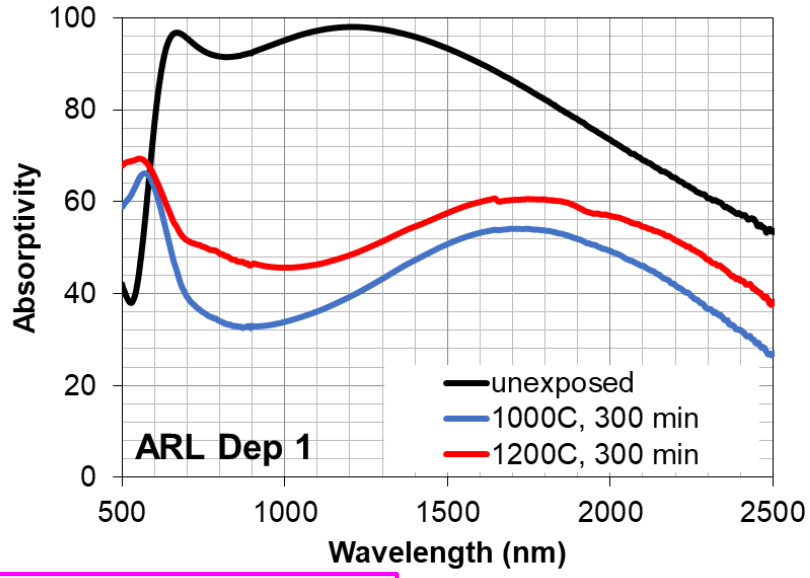
Spectrophotometer Measurements of Samples

- **All samples showed similar amounts of degradation after 300 minutes at 1000 C and 1200 C**

2nd Generation Emitters

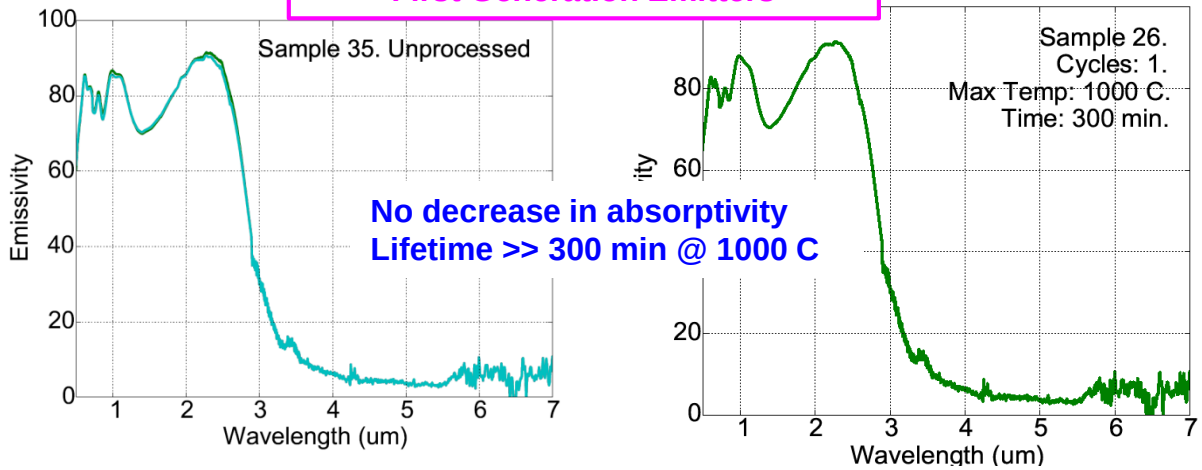






- **Spectra of samples from beginning of program showed less degradation at 1000 C for 300 minutes**
- **Some aspect of deposition or processing conditions led to worse lifetimes in the 2nd generation emitters**

First Generation Emitters



Lifetime Analysis and Discussion

- **Old emitters evaluated in both ovens appeared the same after visual inspection**

- **Old emitters in old furnace showed better lifetime than new emitters in new furnace**

Parameter	Unit	Spec	Sample no	Avg
Ta thickness	nm	5-10	1-8	10
Pt thickness	nm	200 ± 30	1-8	192
Al203 thickness	nm	68 ± 10	1-8	75
Pt dot diameter	nm	320 ± 48	1-8	317
Pt dot pitch	nm	550 ± 82	1-8	553
Pt dot thickness	nm	45 ± 7	1-8	49.9
Al203 sealing layer	nm	182 ± 27	1-8	184

Old device parameters	Spec
Cr thickness	5 nm
Pt thickness	200 nm
Al2O3 thickness	90 nm
Pt dot diameter	300 nm
Pt dot pitch	650 nm
Pt dot thickness	45 nm
Al2O3 sealing layer	150 nm

Why do old devices seem to show better lifetime?

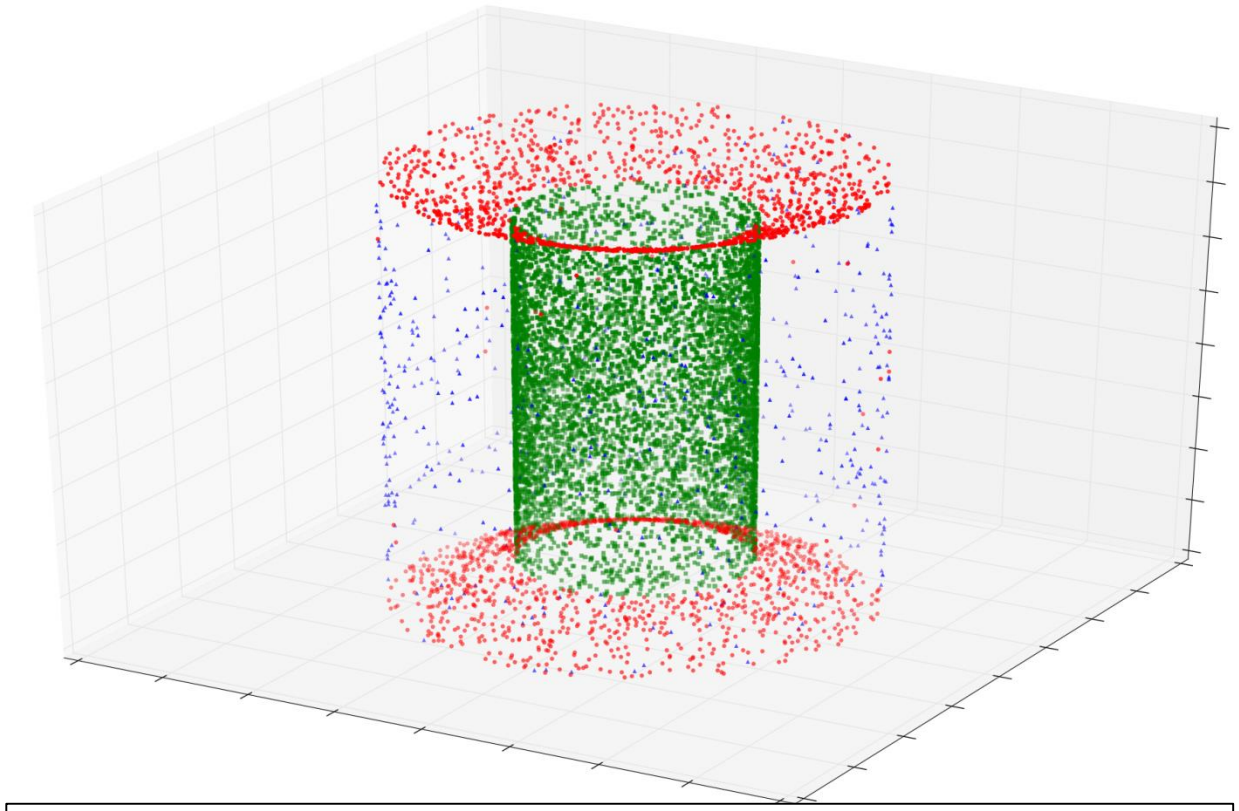
- Cr instead of Ta
  - (against what literature says)
- Thicker Al_2O_3
  - (plausible, but the difference is not large)
- Different deposition parameters
  - (possible, though vendor was asked to use the same processes)

- **Thinner planar films with good emissivity had the same lifetime as the new metamaterial films**

- **Test matrix to evaluate different substrates (SiC vs Al_2O_3), adhesion layers (Ta vs Ti vs Cr), Metals (Pt vs Pt/Rh) revealed trends in thin film lifetime, but did not result in an optimum set of parameters**

Ray Tracing Model

- **PSI built a 3D ray tracing model with the following features:**
  - Ability to build cavity out of arbitrary shapes with as many elements as desired
    - Spheres, cones, planes, cylinders, polygons
  - Code can handle materials with $E(x), A(x), R(x), T(x)$ where $x = (\Theta, T, \lambda)$
  - Code collects location, wavelength, and on **what surface an emitted photon is absorbed**
  - Code determines PV's Voc, FF, and heat sink based on required PV temperature
  - Includes (analytically) thermal losses from conduction
- **Determined that in a 3D TPV cavity**
  - Cold side filtering does not improve efficiency, when multiple reflections and R(θ) taken into account
  - Conduction is negligible for gaps > 1-2 cm (i.e. energy transfer is radiation dominant)



Plot of **recycled photons**, **converted photons**, and **lost photons**, revealing concentric cylinder geometry with conic end caps. Absorptivity values adjusted to better render surfaces.

Emitter Assembly

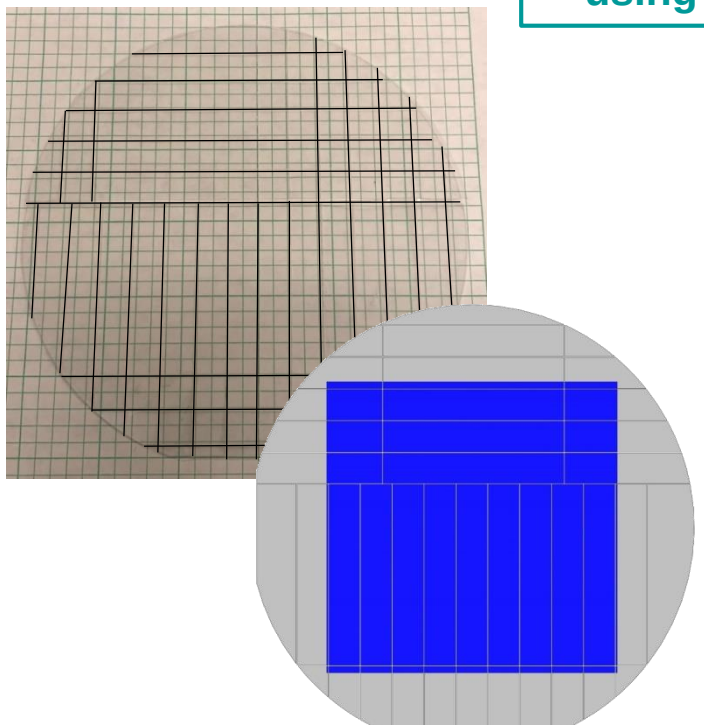
- **Developed process for forming flat emitter pieces (tiles) into a more complex assembly: octagonal representation of a cylinder**
  - High temperature oxide (primarily alumina) foams(Zircar) and adhesives (Aremco)

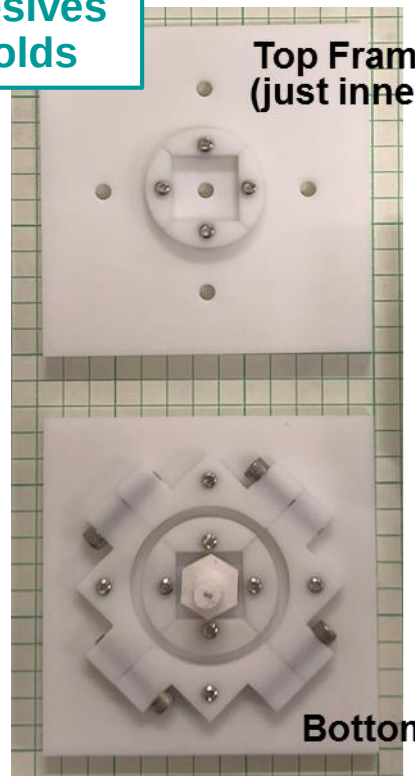
Dice emitter wafers

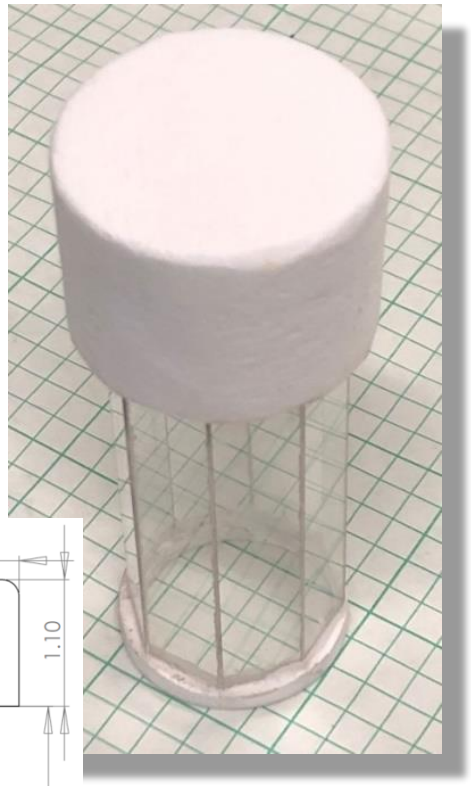
Shape alumina foam preforms

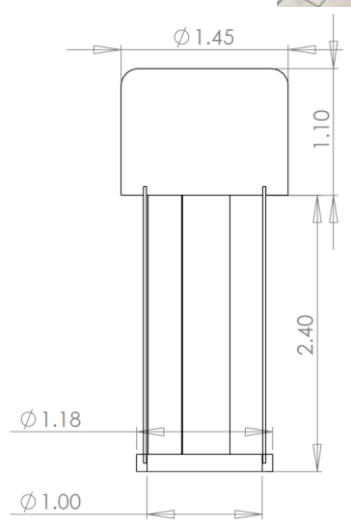
Cast adhesives using molds

Cure Anneal Bake









Next Steps

- **Improve lifetime**
  - In the most recent batch of samples, made with the same set of materials, no difference in lifetime was seen between metamaterial and non-metamaterial samples
    - Allows for a less expensive parameter matrix, all materials to be deposited at the same time
  - Compare sticking layers (Cr, Ti, Ta)
  - Compare deposition methods (ALD, Sputtering, E-beam)
  - Different material combinations (HfO_2, W)

- **Assemble and Test Prototype Converter**
  - Combustor or temporary electrical heater?
  - Backside cooling standalone or (labbench) temporary?

  - What service offices / groups would fund this?
  - Who should we talk to? (together)