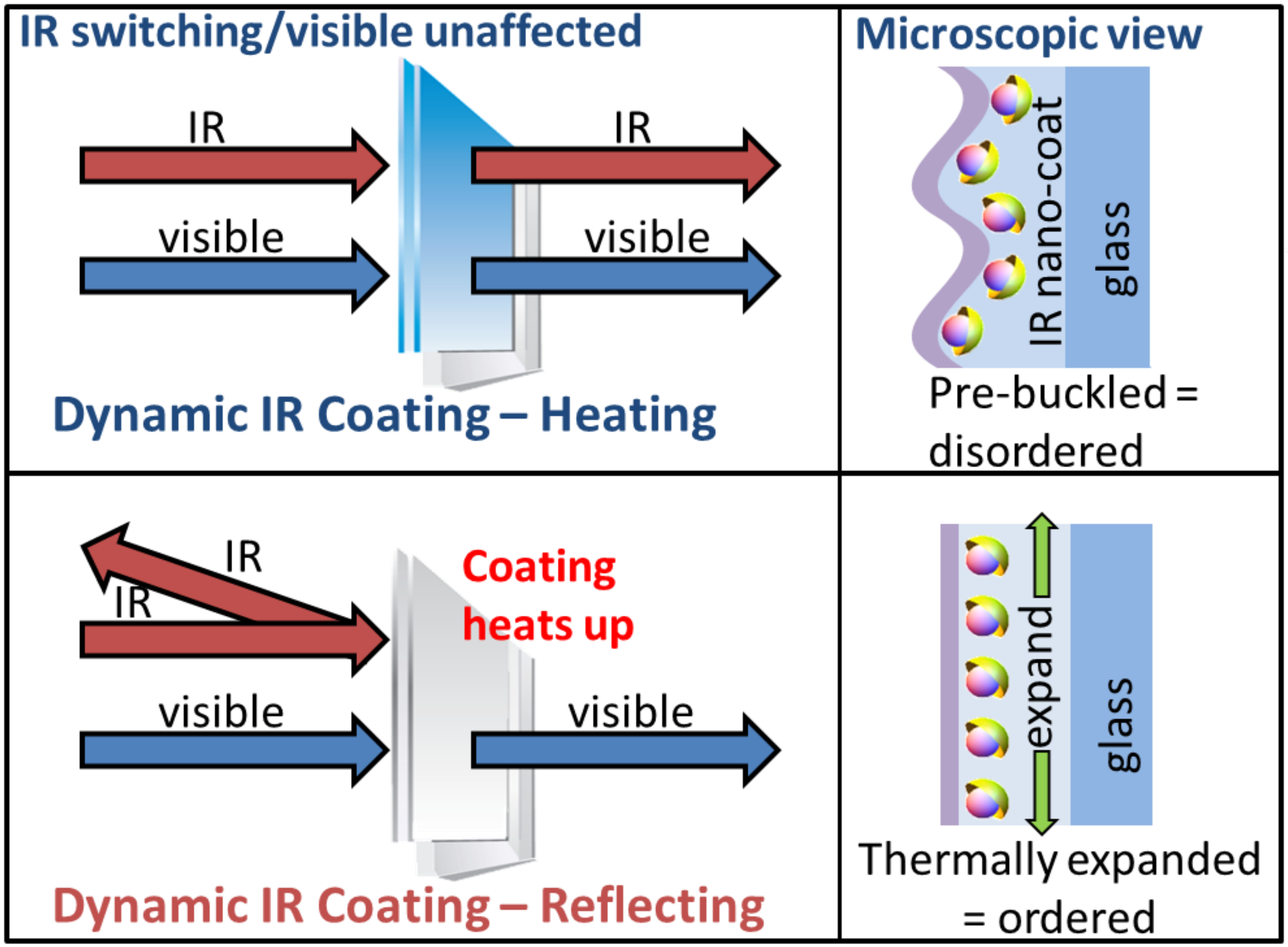
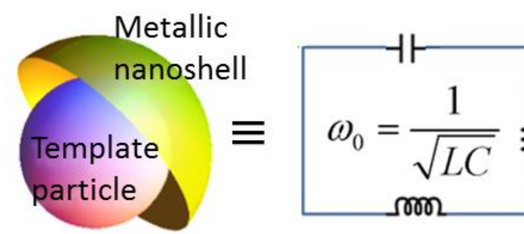
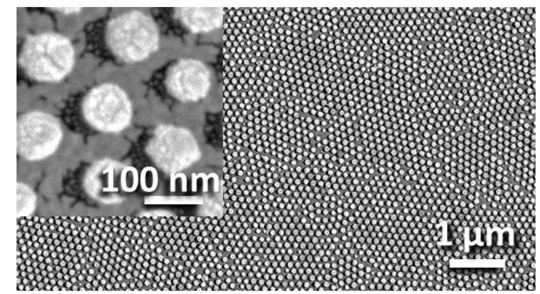
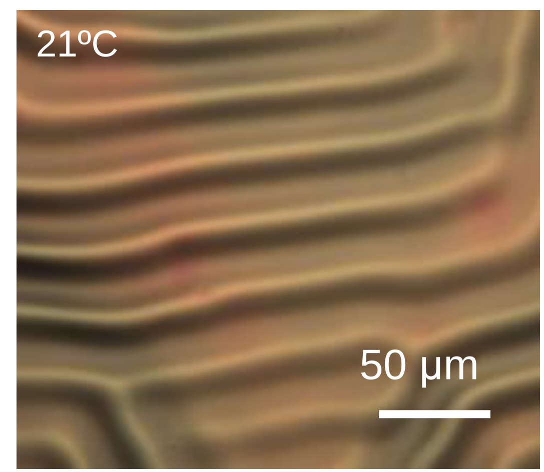
Dynamically Responsive Infrared Window Coatings

2015 Building Technologies Office Peer Review









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Pacific Northwest National Laboratory

Project Summary

Timeline:

Start date: 10/1/2013

Planned end date: 6/31/16

Key Milestones

1. (6/30/15) Demonstrate reversible buckling within a temperature window target of 30 C to 90 C over 5-10 cycles (buckle/unbuckle) on a 6” scale – completed February 2015
2. (8/15/15) PPG will complete initial installation, testing, and debug of their directional metallization source.
3. (3/31/16) – Integrate buckling and subwavelength films at the 6” scale. Performance targets of 10-15% near infrared (NIR) Δ and visible transmission (VT) ≥ 20%, with temperature switch range of 30-90°C

Key Partners:

PNNL	PPG

Budget:

Total DOE $ to date: $750K

Total future DOE $: 0

Target Market/Audience:

Windows Coatings for commercial and residential. Both new and retrofit markets.

Project Goal:

To develop a low-cost, energy-saving, passively switchable dynamic IR coating by integrating a IR reflective sub-wavelength nanostructures in a buckling layer. Both lab scale prototypes and intermediate scale-up will be addressed.

Purpose and Objectives

Problem Statement: Current electrochromic and thermochromic window technology costs as much as 16 x double glazing and blocks daylight. This significantly reduces market penetration and subsequent energy savings. This project addresses both cost and a means to allow daylighting.

Target Market and Audience: Commercial and Residential Windows – both new construction and retrofit. Technology will result in 30/20% primary heating/cooling energy savings, while allowing daylighting for potentially as low as \$5-8/ft^2 cost (PNNL projection).

Impact of Project: If successful, switchable IR window coating technology has technical potential to save up to 2.24 Quad/yr in heating, cooling, and lighting.

1. Project will develop lab-scale prototype and address intermediate level scale-up.
2. Metrics for success
    a. Lab scale prototype film development & intermediate level scale-up
    b. R&D on durability, aesthetics, cost (1-3yr after project)
    c. Pilot scale testing (beyond current (3+yr after)

Approach

Approach: Combine a scalable nanostructured coating with a passive thermally switchable buckling coating to create a dynamic infrared (IR) window film. Laboratory scale proof-of-concept will be done in phase 1 (complete) and then intermediate scale-up will be the focus of phase 2 (in progress).

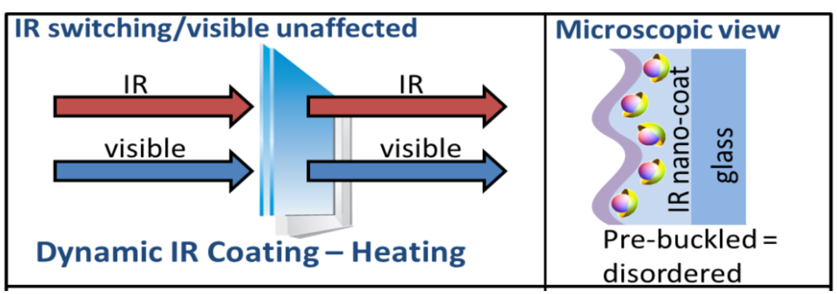
Key Issues:

- Tailorable subwavelength nanostructured coatings to control visible transmission and infrared transmission separately
- Buckling effect used for passive thermal switching

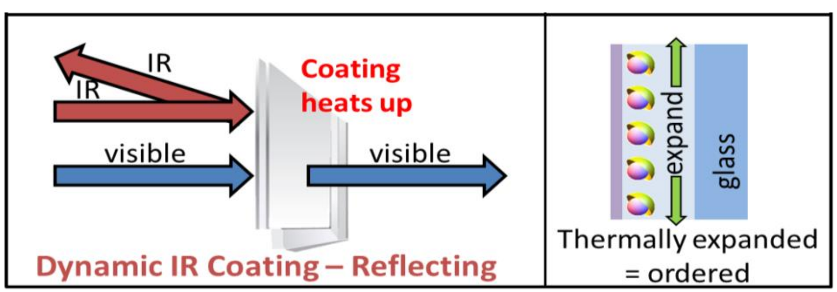
Distinctive Characteristics:

Tailored IR response: Subwavelength features allow for tailored IR response
Buckling Films: Allows passive switching controlled by materials & processing conditions
Scale-up: Intermediate level scale-up is a critical challenge and will be the major focus of Year 2 – current phase

Cold State



Hot State



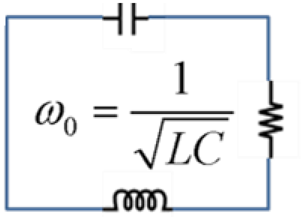
Approach – Nanostructured Coatings

Subwavelength Structures:

- Window coatings based on nanostructures smaller than wavelength of light
- Coatings contain an ordered, oriented array of the metallic nanoshells
- Open-ring resonator (ORR) nanoshells that are optically responsive
  - Resonance tuned in the visible/IR by adjusting fabrication parameters
  - Structure has intrinsic capacitance and inductance to form analogous resonant LC circuit
  - Multiple sizes will be needed for longer wavelengths (future scope)

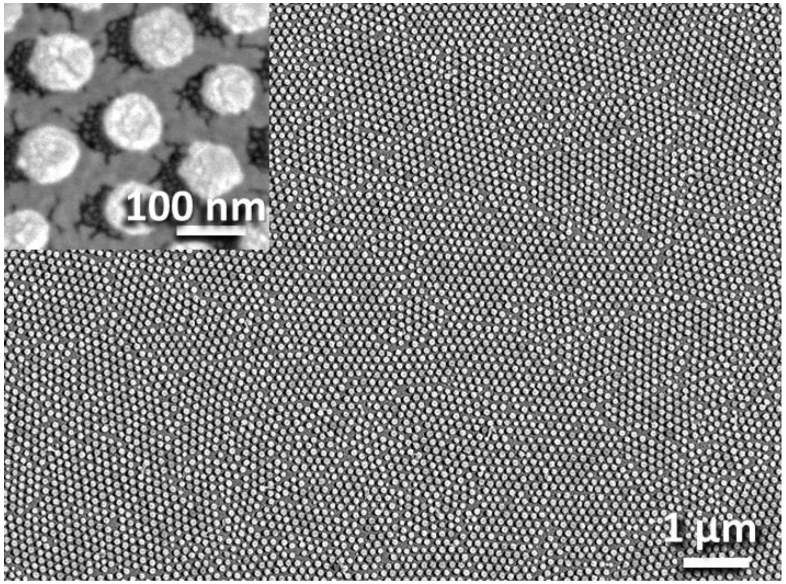
Metallic Nanoshell

Equivalent Resonant Circuit

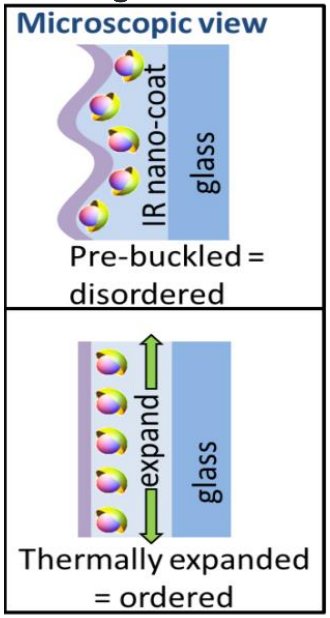


$$\omega_0 = \frac{1}{\sqrt{LC}}$$

Nanoshell Array



Integrated Film

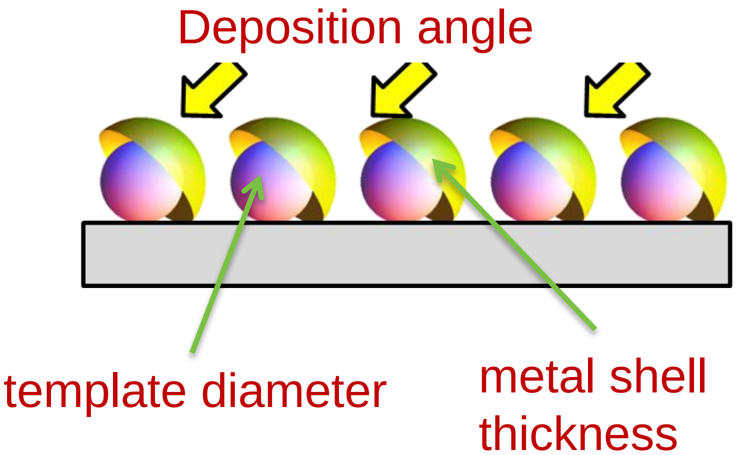


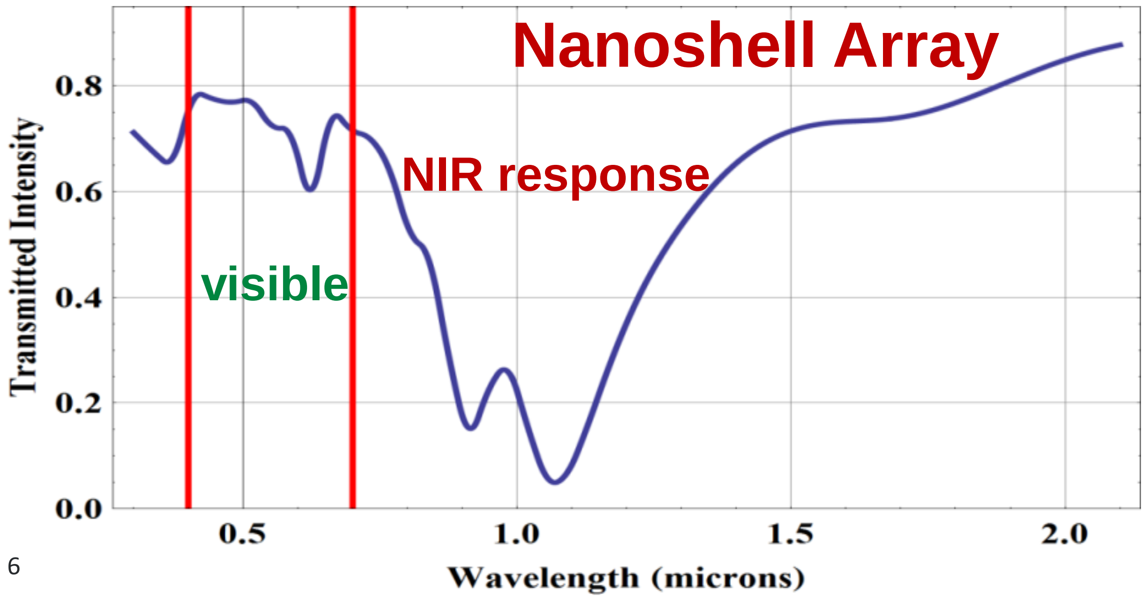
Mirin, *et al.*, Nano Letters **9**, 1255 (2009).
Alvine, *et al*., Appl. Phys. Lett. **102**, 201115 (2013)
Alvine, *et al*. SPIE Defense, Security, and Sensing. **8725**, 87252H (2013)

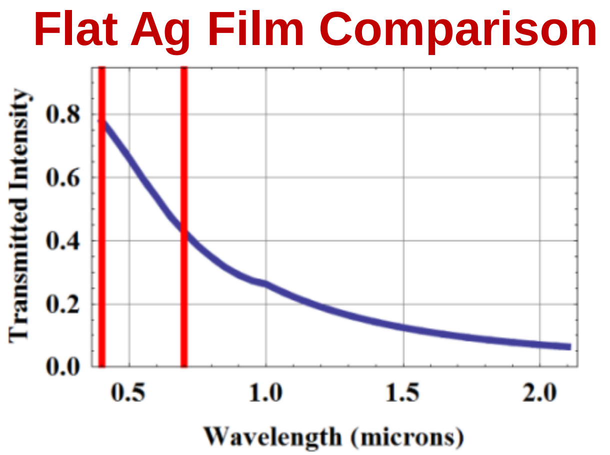
Approach – Subwavelength Structure Design

- PNNL Numerical modeling was used to determine optimal structures
- Finite Difference Time Domain modeling used – solving Maxwell's eq. at each point in a mesh defined in a CAD program
- Parameters varied: particle diameter, metal type, thickness, deposition angle
- Results target 70% Visible transmission and 50% NIR (750-900nm) reflection

Fabrication Parameters







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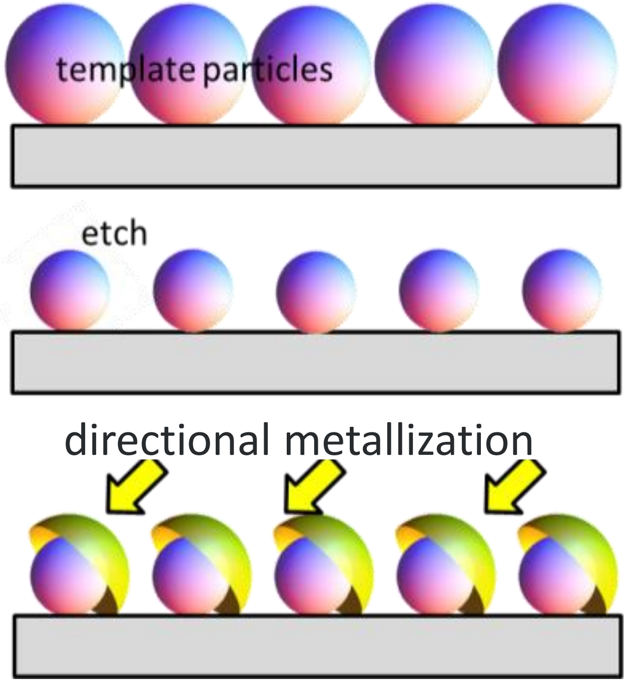
Approach – Subwavelength Array Fabrication (lab scale)

Template Process



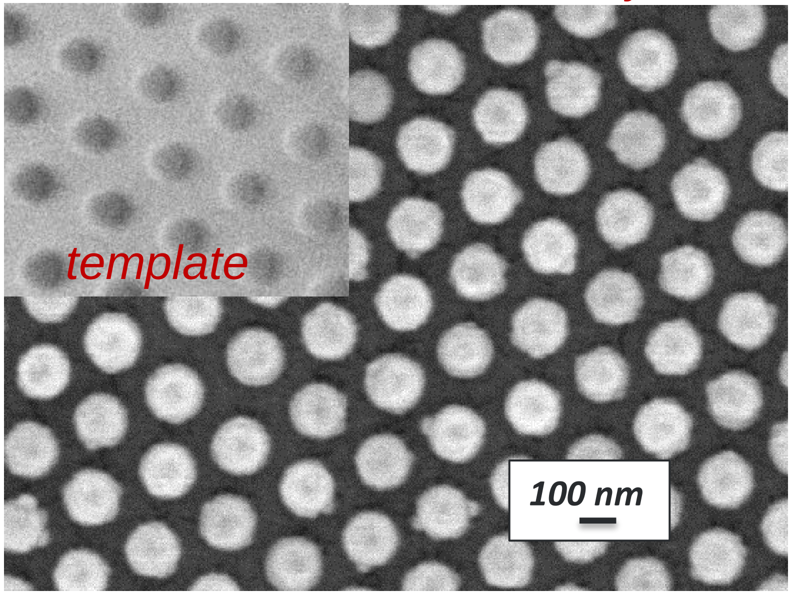
PNNL Fabrication Approach

- Scalable to large areas
- Wet deposition nanoparticle template
- Directional metallization to form nanoshells

Fabrication Parameters

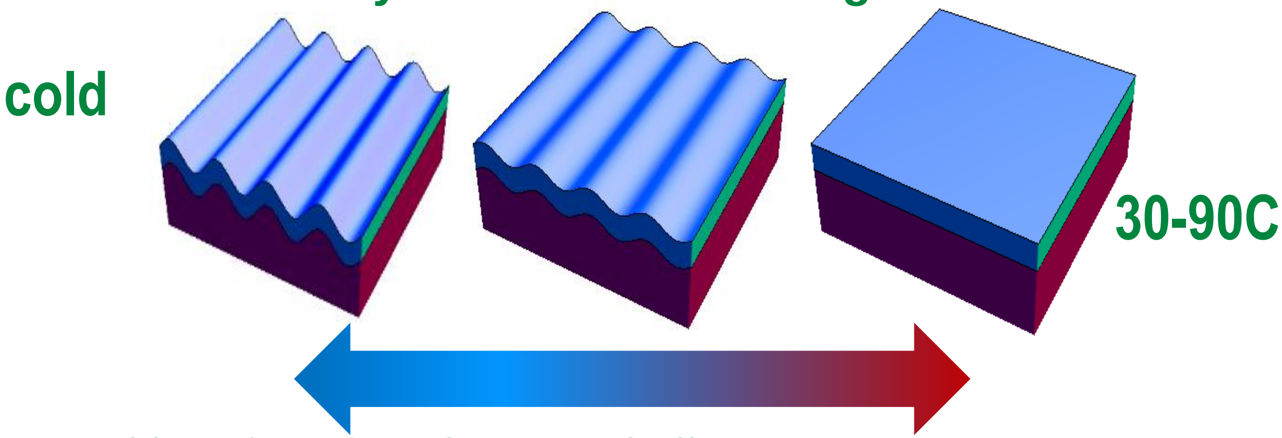
- Particle diameter
- Particle separation (linked)
- Metal thickness
- Metal deposition angle

Metal coated Nanoshell arrays



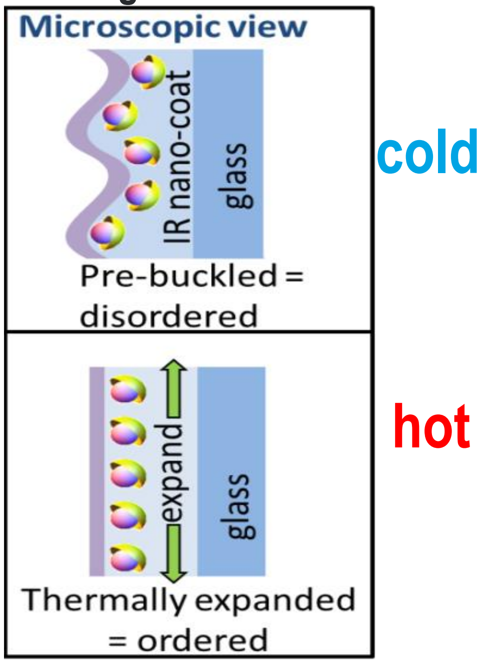
Approach – Reversible Buckling

Thermally Reversible Buckling



Buckling disorders the nanoshell array allowing IR transmission

Integrated Film



Buckling Geometry:

- Stiff film/soft film stack
- Pre-buckled at room temperature gives disordered nano-array
- Heating expands and flattens film – giving ordered nano-array

Buckling Equation

$$\lambda \sim t\left(E_f / 3E_s\right)^{1/3}$$

Tuning:

- Wavelength/Amplitude tuned by choice of material modulus (E), and thickness (t) and deposition
- Temperature response set by material choice (CTE) and processing conditions

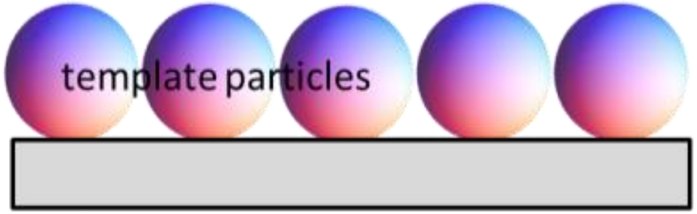
Approach – Scale-Up: Large Area Template Coatings

PPG Scale-Up of Template coatings

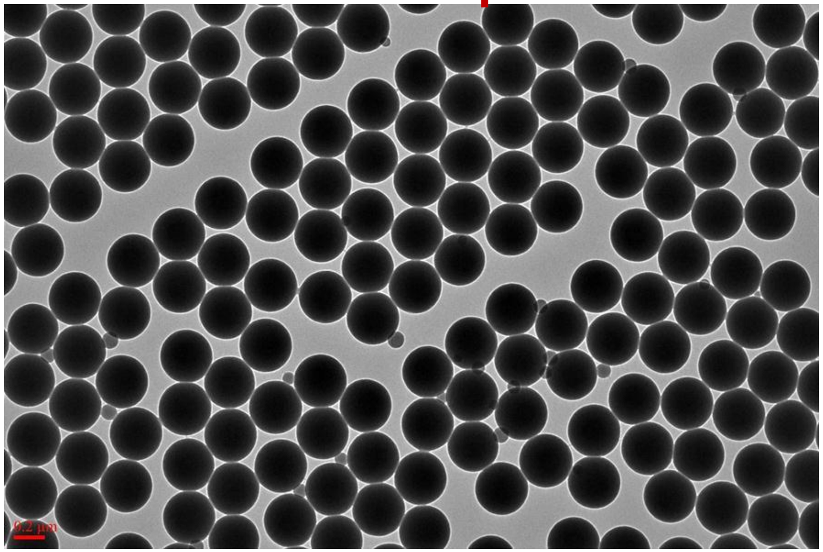
- PPG developing process to coat large areas
- Focus on intermediate level scale-up > 6” width

Fabrication Parameters

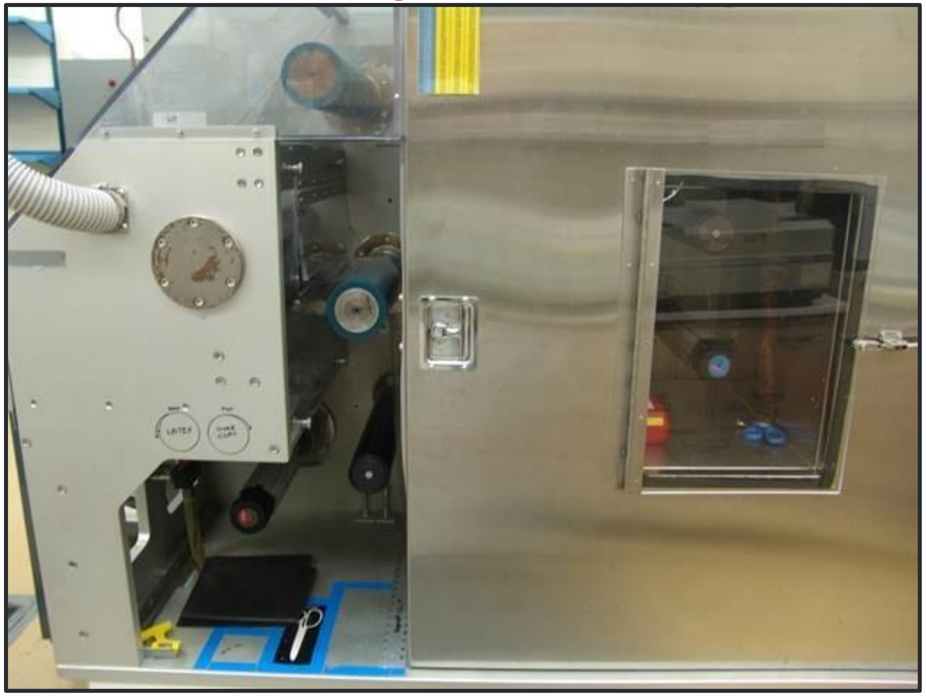
- Coatings of polymer nanoparticles
- Size determined by PNNL modeling
- High degree of monodispersity



TEM of PPG nanospheres



PPG large scale Template Coater



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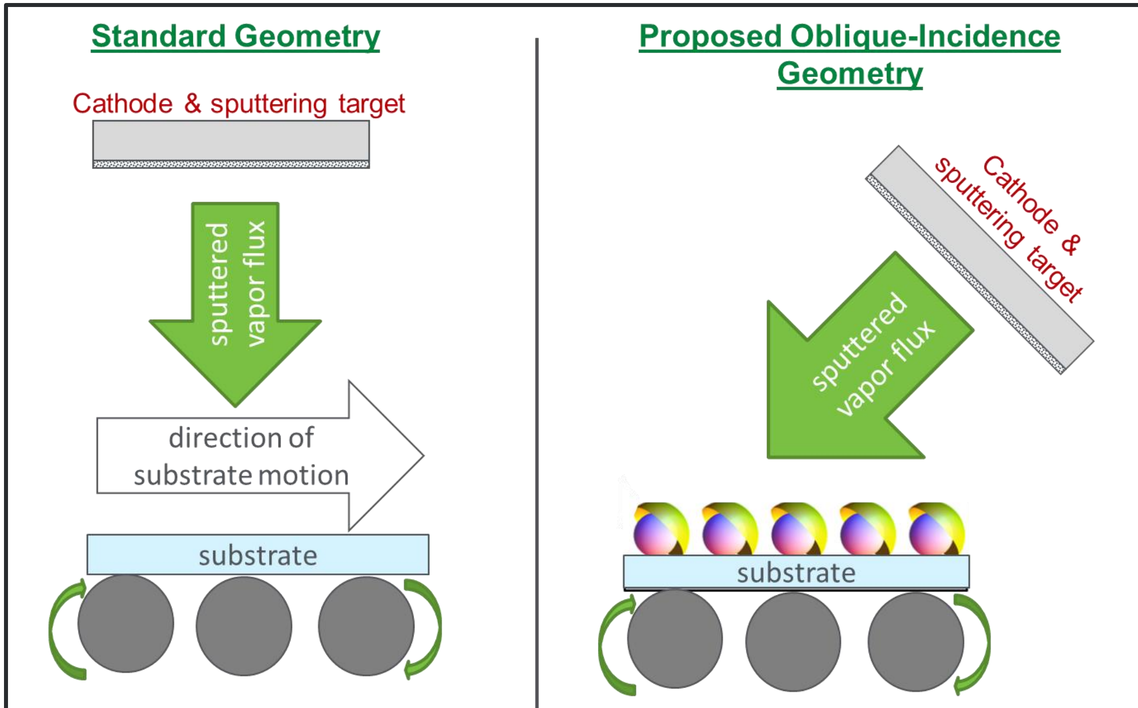
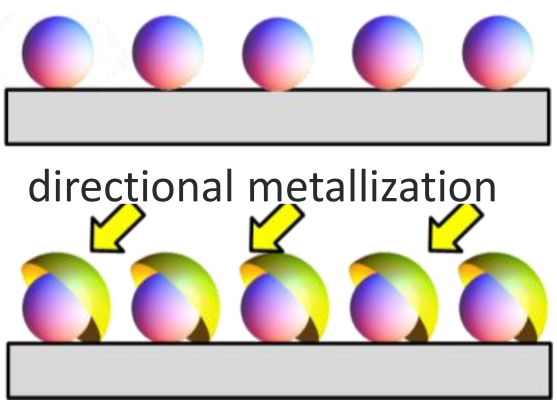
Approach – Scale-Up: Directional Metallization

PPG developing scaled up directional metallization

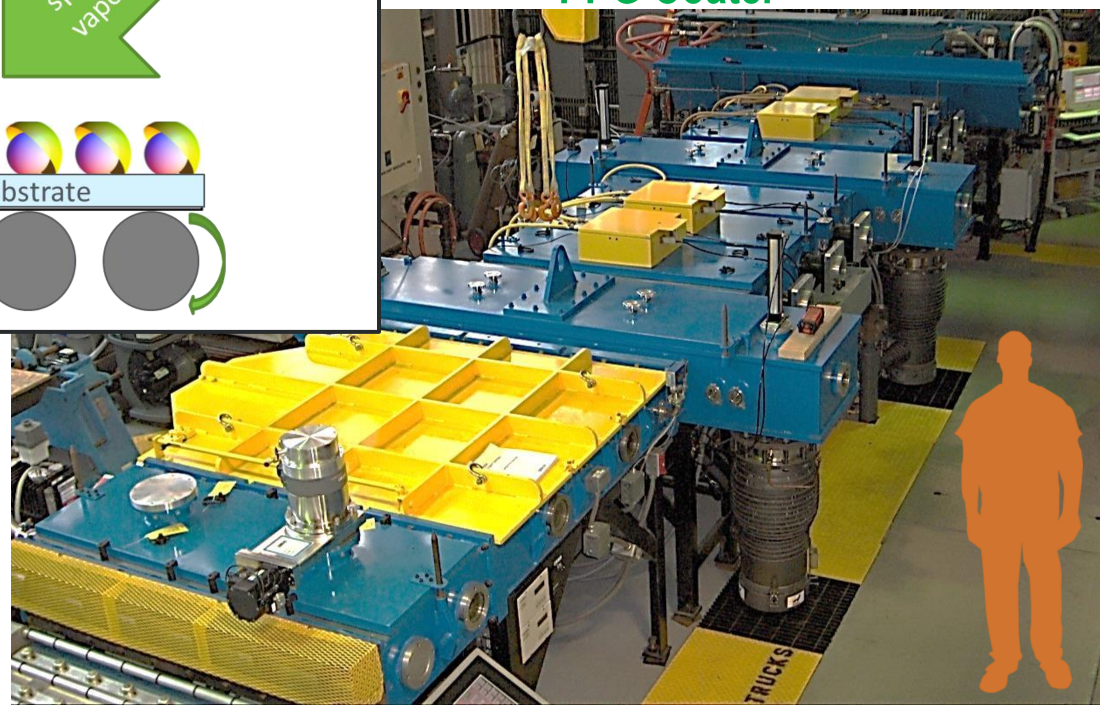




Scale-Up of Metallization

- PPG designing new head for large scale ≥6" coating
- Will be tested in pilot scale line with PPG made nanoscale templates

PPG Coater



Progress and Accomplishments

Lessons Learned: Buckling will require addition of an index matching layer to reduce spurious scattering due to the increased roughness of the interface in the cold (buckled) state. More work will be needed in this area.

Accomplishments: In the past year the project team was able to meet the go/no go to demonstrate proof-of-concept with the visible transmission and NIR delta (transmission vs blocked) targets. In addition, PPG has been able to demonstrate the ability to fabricate hundreds of feet of optimized nanoscale template material.

Market Impact: The target markets for this dynamic window coating are new and retrofit commercial and residential buildings. Technology will result in 30/20% primary heating/cooling energy savings, while allowing daylighting for potentially as low as \$5-8/ft^2 cost (PNNL projection). Savings in all climate zones are possible as the coating surface within the window (interior/exterior pane) may be varied based on climate and tailored to maximize energy savings.

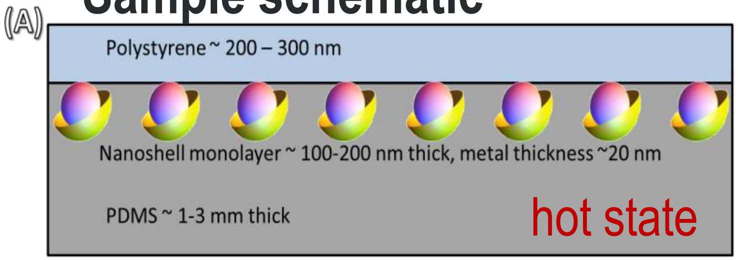
Awards/Recognition: Alvine, *et al.* "Subwavelength Films" OSA Renewable Energy and Environment, Tucson, AZ (2013) *invited talk*

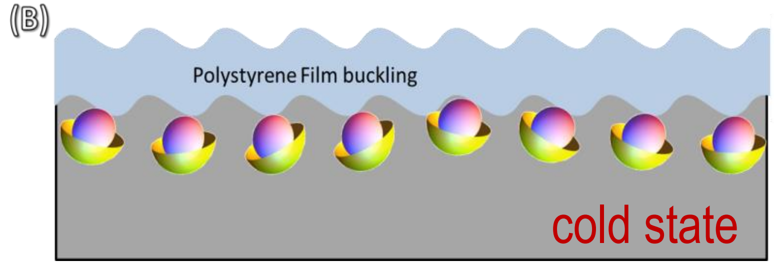
Progress and Accomplishments – Phase 1 Go/No Go

Demonstration of nanoshell arrays that meet milestone 22% VT (at 500 nm) and NIR delta of 50% (relative at 900 nm), switching between 30 and 90 C

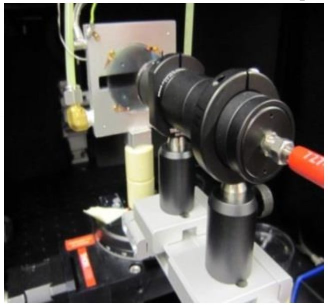
- In-plane hemispherical integration measurements for VT and NIR delta

Sample schematic

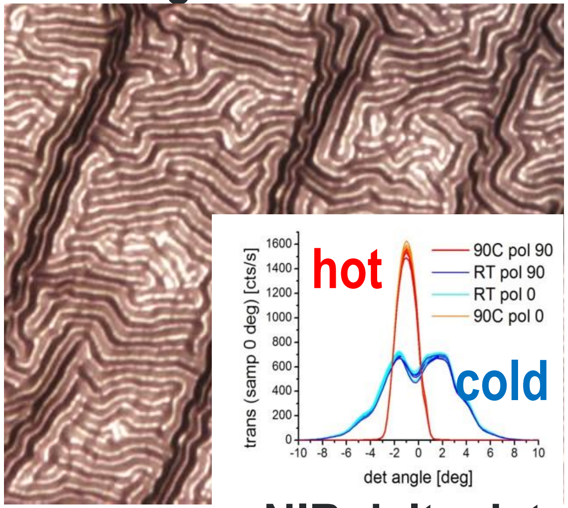




Measurement setup

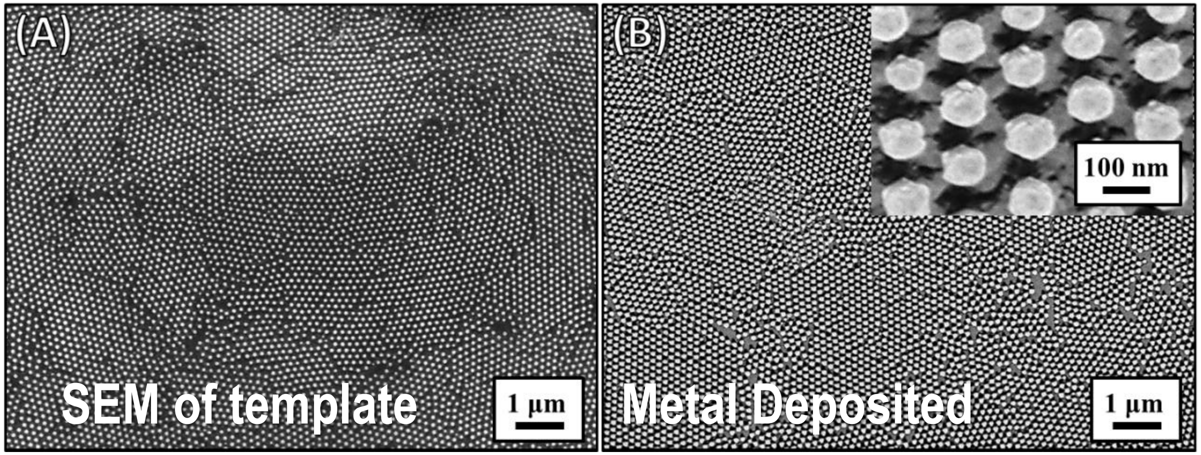


Buckling in the cold state



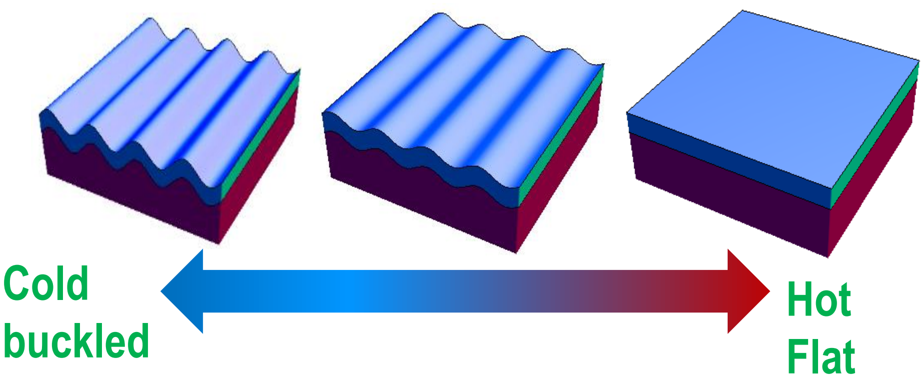
NIR delta data

Subwavelength Template



Progress and Accomplishments – Reversible Buckling at 6”

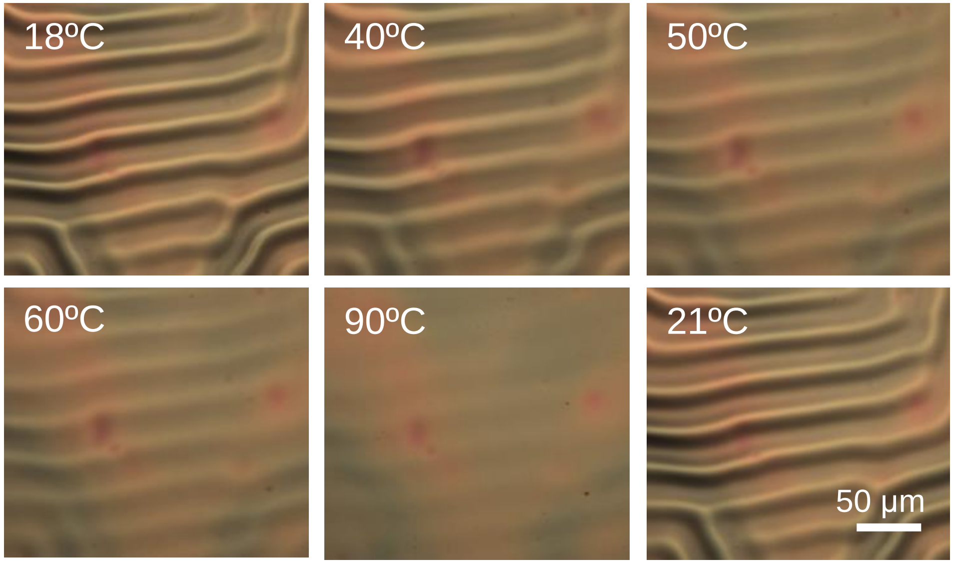
Thermally Reversible Buckling



- Fabricated and tested 6” transparent buckling bilayer film (PNNL)
- Demonstrated switching between microscopic buckling (cold) and flat (90C) over ten cycles
- Change in materials can change the switching temperature

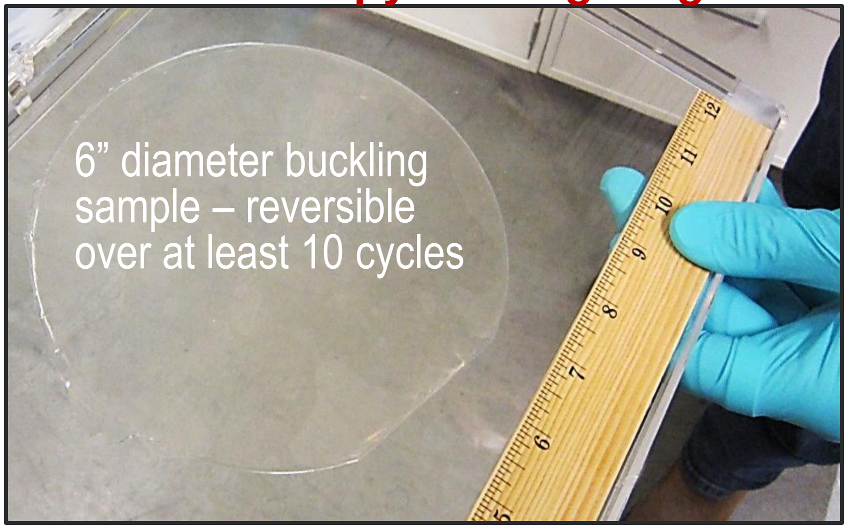
Reversible Buckling demonstrated >10 cycles

Effect is continuous change in amplitude with Temperature



Wavelength chosen to be easily visible with microscope – see continuous change with temperature

Measured with in-situ optical microscopy heating stage



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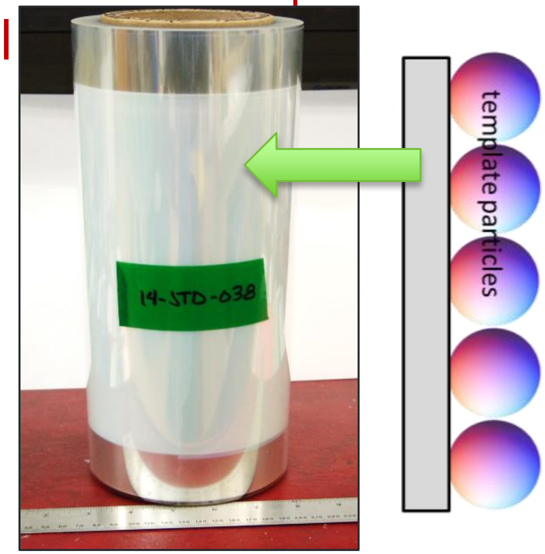
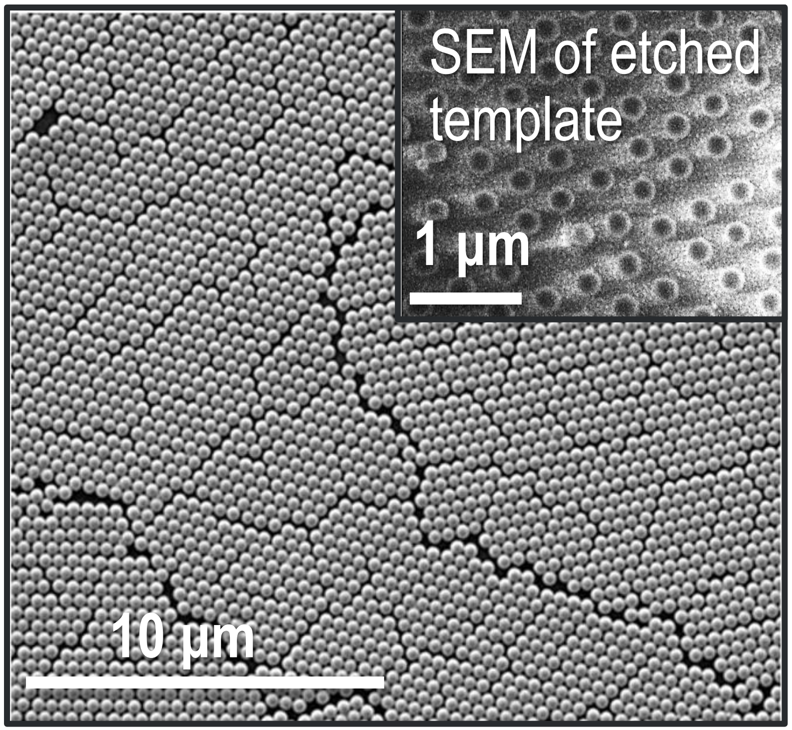
Subtask 4.1: Subwavelength Templates

Scale-Up of Template coatings

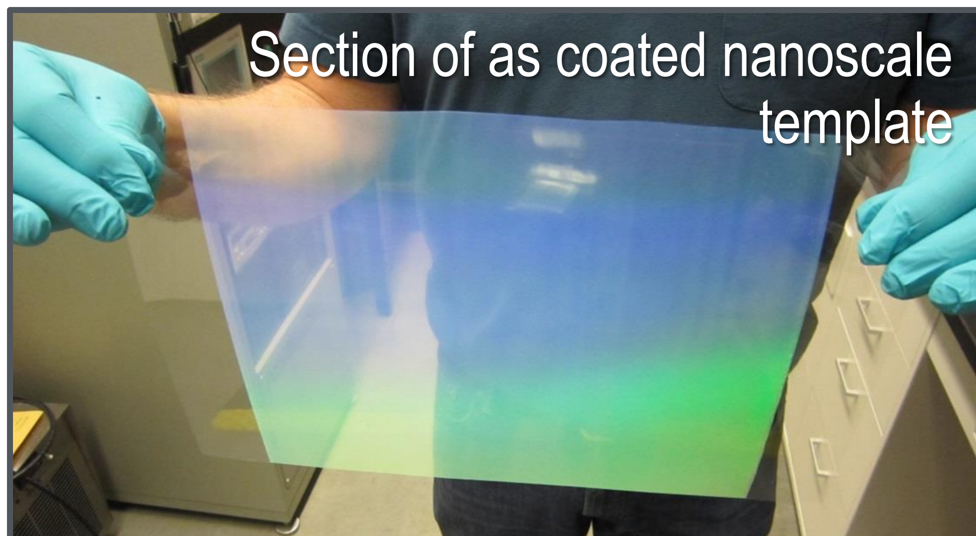
- PPG has developed a roll-to-roll process
- Hundreds of square feet of template coated
- Excellent overall quality, but some defects
- Initial particles showed bridging after etching
- Polymer cross linking was thought to be the problem
- 2nd Gen. particles w. no cross linking did not bridge

Roll of nanoscale template material





SEM of as coated template

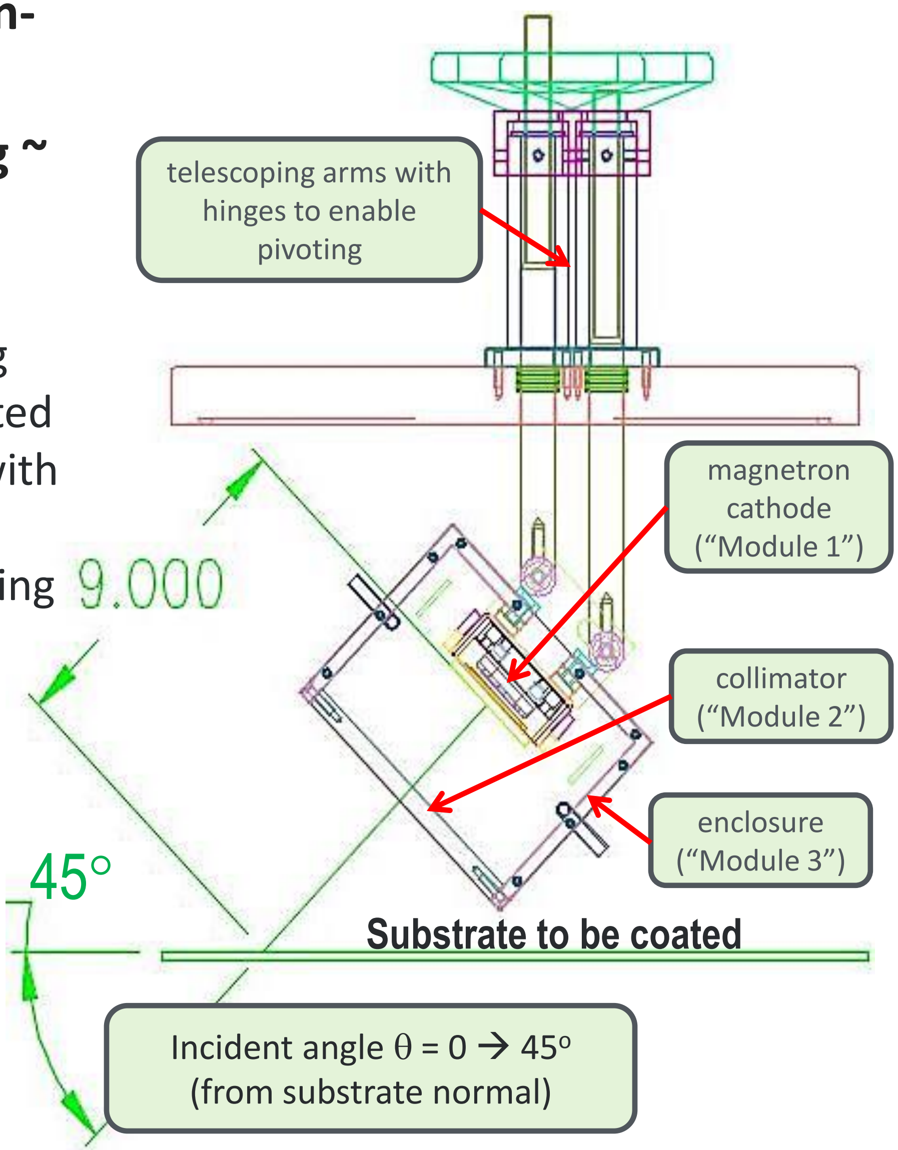


Section of as coated nanoscale template

Subtask 4.2: Directional Metallization

- **Design of Directional Sputter Deposition Source (DSDS) completed & construction in-progress**
- **Install DSDS in pilot coater and start debug ~ mid-to-late May 2015**
- **Experiment plan**
  - Deposit Ag on nanotemplated substrates using optimum deposition angle(s) (~ 30°) as predicted by PNNL-based modeling work (without and with collimation mask – "Module 2")
  - Deposit very thin passivation layer over Ag (using conventional, non-directional magnetron cathode)
  - Ship specimens to PNNL for measurement of optical spectra and other analytical characterization
  - Based on PNNL feedback, do follow up experiments as appropriate
  - Produce final metallized 6 inch-scale samples

DSS Design for ≥6" sample metallization



Project Integration and Collaboration

Project Integration: This is a joint project between PNNL and PPG, which accelerates market impact. Project team communicates through frequent email, monthly telecoms, quarterly reviews with the client, and yearly site visits and/or reviews.

Partners, Subcontractors, and Collaborators:
PNNL(lead): to develop laboratory scale dynamic IR films
PPG: to develop intermediate level scale-up of the dynamic IR films

Communications: Alvine, *et al.* “Subwavelength Films” OSA Renewable Energy and Environment, Tucson, AZ (2013) *invited talk*

“Thermally Reversible Buckling Thin Films,” K.J. Alvine – in progress.

Next Steps and Future Plans

Next Steps and Future Plans:

<u>Project Activities:</u>

- PPG will receive, install and debug directional metallization setup and begin metalizing large area nanotemplates made by PPG.
- PPG will provide metallized coatings to PNNL for integration with buckling films.

<u>Tasks for Possible Expansion:</u>

- Research into index matching layers will be required to reduce scattering
- Research into alternate dynamic switching routes to improve delta
- Optimization of films for better NIR and VT response

REFERENCE SLIDES

Project Budget

Project Budget: $750K Federal Funding, $78K proposed cost share from PPG. Full Federal Funding of $750K was authorized on 10/1/2013. Phase 2 was authorized 12/2014.

Variances: A no-cost extension into FY16 will be necessary based on the Go/ No Go decision shifting from Sept 2014 to Dec 2014.

Cost to Date: Cumulative spent as of 3/20/2015 = $310,651
Additional Funding: N/A other than cost share.

Budget History					
10/1/2013 – FY2014 (past)		FY2015 (current)		FY2016 – 6/30/16 (planned)	
DOE	Cost-share	DOE	Cost-share	DOE	Cost-share
$375K	$32K	$375K	$46K	*NOTE: remaining carryover funding will be used to complete work in FY16.*	

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Project Plan and Schedule

Project Plan:

- Go/No Go met in December of 2014 with extension – required extension of most Phase 2 MS.

Project Schedule

Project Start: 10/1/2013	Completed Work
Projected End: Extend to 6/31/16 as result of SOPO revision	Active Task (in progress work)
	Milestone/Deliverable (missed)
	Milestone/Deliverable (MS met actual)
	Milestone/Deliverable (MS origional planned or extension)
	Go/No Go Decision Point

	FY13	FY2014				FY2015				FY2016			
Task	Q1-4(Oct-Sept)	Q1 (Oct-Dec)	Q2 (Jan-Mar)	Q3 (Apr-Jun)	Q4 (Jul-Sep)	Q1 (Oct-Dec)	Q2 (Jan-Mar)	Q3 (Apr-Jun)	Q4 (Jul-Sep)	Q1 (Oct-Dec)	Q2 (Jan-Mar)	Q3 (Apr-Jun)	Q4 (Jul-Sep)
Past Work													
Q1 Milestone: IP agreement reached between all parties.													
Q1 Milestone:Technology to Market discussions held between PNNL/PPG – results reported to DOE during quarterly check-in													
Q1 Milestone:Add integrated dynamically responsive IR window coating to BTO prioritization tool based on final project targets													
Q1 Milestone: PNNL will complete an optimized fabrication design based on numerical modeling for IR subwavlength arrays with performance targets of 70% visible transmittance and 50% IR reflection of the oriented array in the NIR design window of 750 to 900 nm.													
Q1 Milestone: Complete down-selection of materials and geometries (thickness/stacking) for reversible buckling.													
Q2 Milestone: Complete fabrication of preliminary oriented array subwavelength films with at least 20% NIR reflection and at least 50% visible light													
Q2 Milestone: Demonstrate reversible buckling within a temperature window target of 30 C to 120 C over 5-10 cycles (buckle/unbuckle).													
Q2 Milestone:Complete down-selection of scale-up methods for subwavelength templates on 6” samples.													

Project Plan and Schedule

Task	FY13 Q1-4(Oct-Sept)	FY2014 Q1 (Oct-Dec)	FY2014 Q2 (Jan-Mar)	FY2014 Q3 (Apr-Jun)	FY2014 Q4 (Jul-Sep)	FY2015 Q1 (Oct-Dec)	FY2015 Q2 (Jan-Mar)	FY2015 Q3 (Apr-Jun)	FY2015 Q4 (Jul-Sep)	FY2016 Q1 (Oct-Dec)	FY2016 Q2 (Jan-Mar)	FY2016 Q3 (Apr-Jun)	FY2016 Q4 (Jul-Sep)
Q2 Milestone:Directional metallization source design completed.													
Q3 Milestone:Complete comparison testing of order/disorder delta based on NIR transmission for subwavelength arrays with at least 10% difference in NIR transmission, between 2 different films (ordered and disordered).													
Q4 Milestone: Go/No Go - Demonstrate lab-scale (1x1") dynamic IR responsive coating with NIR transmission delta of 20%, a visible transmission of at least 20% and a temperature switching window of 30 to 90 C.													
Q4 Milestone: Supply 6" subwavelength templates for all necessary team members													
Current/Future Work (FY15/FY16)													
Q4 FY15 Milestone: PPG-GBDC will complete initial installation, testing, and debug of their directional metallization source.													
Q3 FY15 Milestone: Strategy for receiving next scope of funding developed and documented in a straw man-type plan.													
Q3 FY15Milestone: Update integrated dynamically responsive IR window coating in BTO prioritization tool based on achieved technical targets and long-term performance/cost goals.													
Q3 FY15 Milestone: PNNL will revise and update subwavelength array design as needed based on lessons learned from fabrication efforts in year 1.													
Q3 FY15 Milestone: Demonstrate reversible buckling within a temperature window target of 30 C to 90 C over 5-10 cycles (buckle/unbuckle) on a 6" scale.													
Q1 FY16 Milestone: PPG-CIC will provide PPG-GBDC with optimized 6" subwavelength templates for directional metallization.													
Q4 FY15 Milestone: PPG-GBDC will provide PNNL with preliminary 6" metallized nanoshell array samples for integration of buckling layers and optical measurements.													
Q2 FY16 Milestone: Integrate buckling and subwavelength films at the 6" scale and optically characterize said coatings. Performance targets of NIR transmission delta of 10-15% (over 750-900 nm), with an visible transmission of at least 20%, and a temperature switching in the range of 30-90C.													
Q1 FY16 Milestone: Best available metallized 6" samples will be provided to PNNL for integration and optical characterization.													