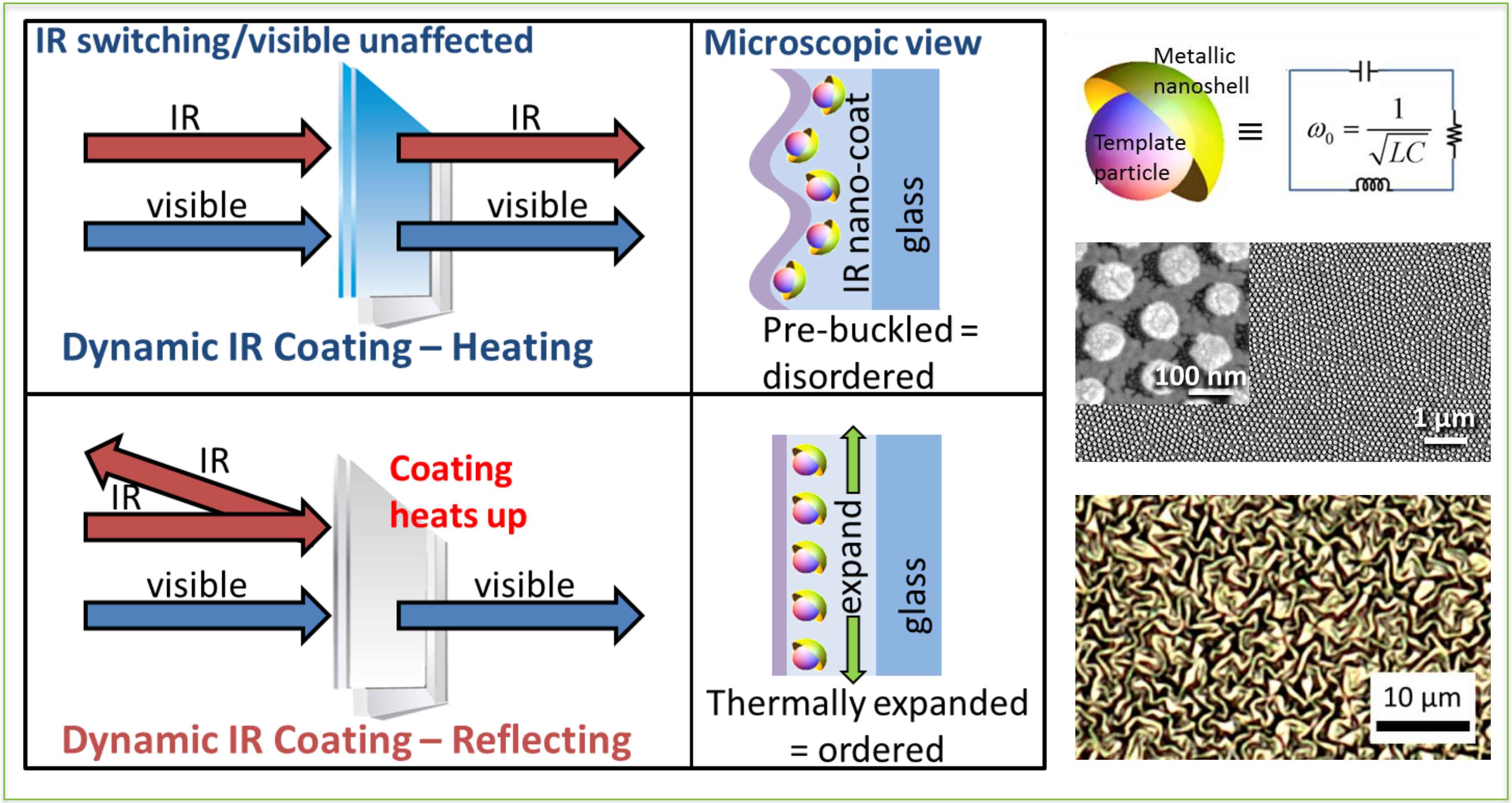
Dynamically Responsive IR Window Coatings

2014 Building Technologies Office Peer Review



U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy

Dr. Kyle J. Alvine, kyle.alvine@pnnl.gov
Pacific Northwest National Laboratory

Project Summary

Timeline:

Start date: 10/1/2013 (NEW PROJECT)

PPG subcontract start: 1/10/2014

Planned end date: 9/30/2015

Key Partners:

PNNL	PPG

Key Milestones

1. Milestone 1 (9/30/14) – Go/No Go to demonstrate lab scale dynamic IR responsive coating with 20% NIR Δ and average (visible transmittance) VT ≥ 50%, with T switch range of 30-90°C.
2. Milestone 2 (9/30/15) – Integrate buckling and subwavelength films at the 6” scale. Performance targets of 10-15% NIR Δ and average VT ≥ 50%, with T switch range of 30-90°C

Budget:

Total DOE $ to date: $375K/FY14

Total future DOE $: $375K/FY15

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 runs 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 - Summary

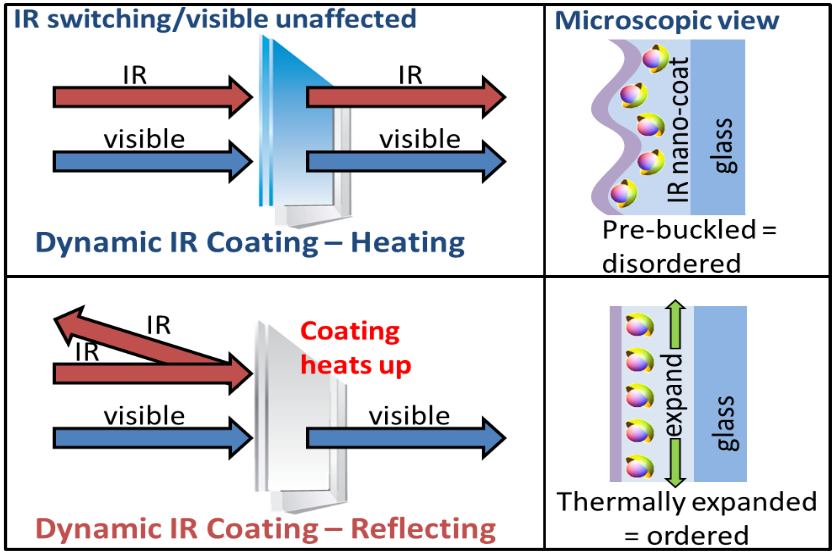
Approach: Combine a scalable nanostructured coating with a passive thermally switchable buckling coating to create a dynamic IR window film. Laboratory scale and then intermediate scale-up will be addressed.

Distinctive Characteristics:

- Tailorable subwavelength nanostructured coatings to control VT and IRT separately
- Buckling effect used for passive thermal switching

Key Issues:

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

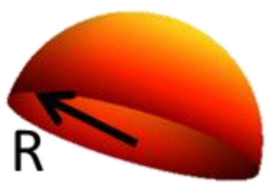


Approach – Nanostructured Coatings

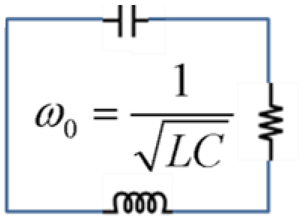
Subwavelength Structures:

- 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)

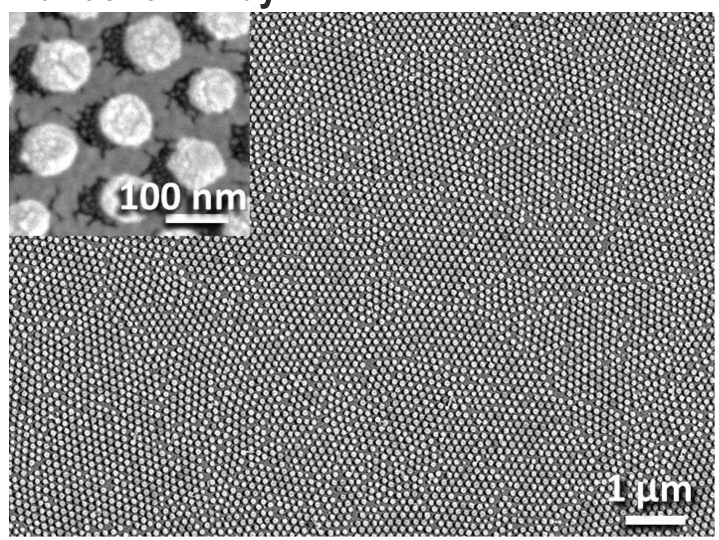
Metallic Nanoshell



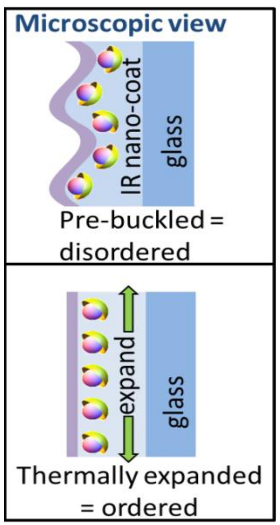
Equivalent Resonant Circuit



Nanoshell Array



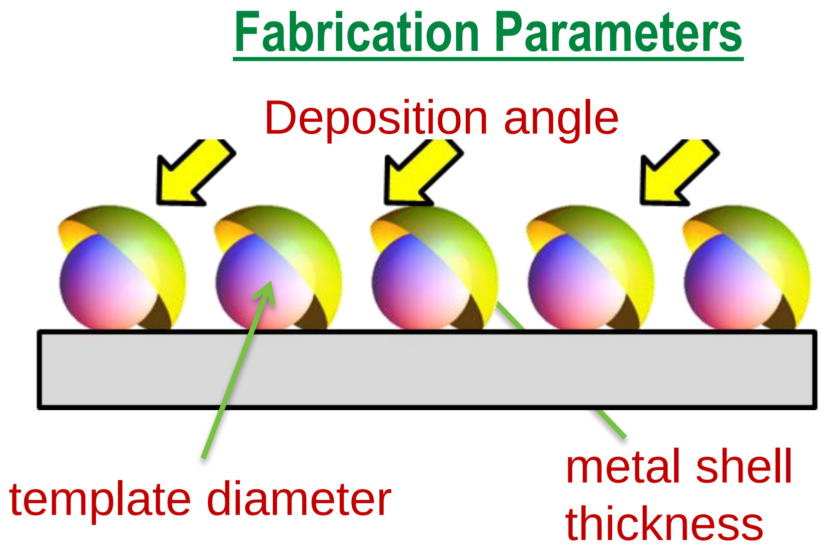
Integrated Film

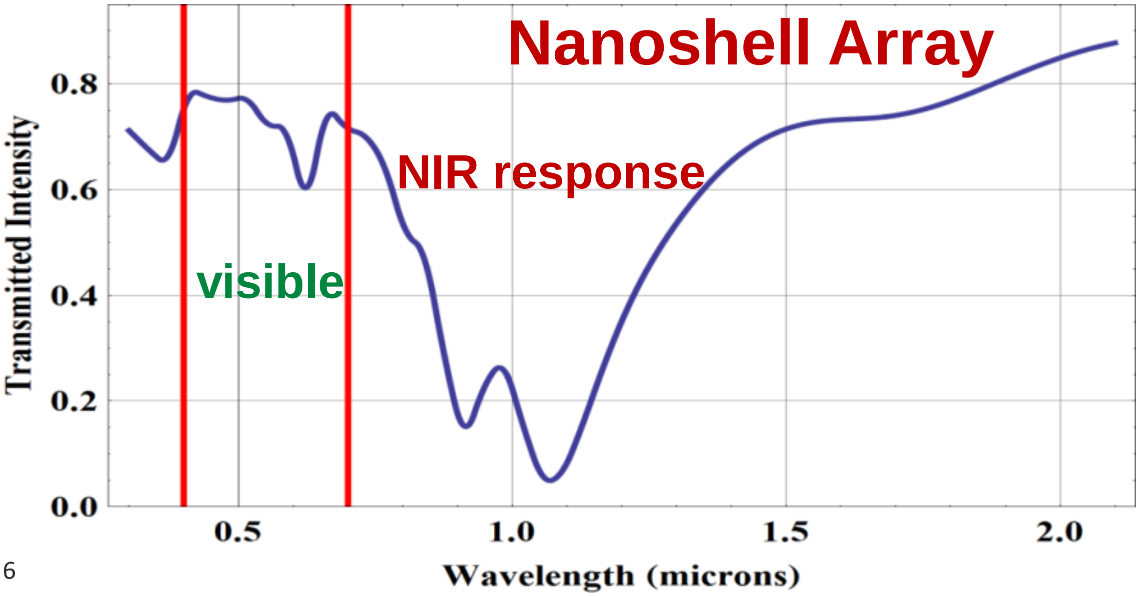


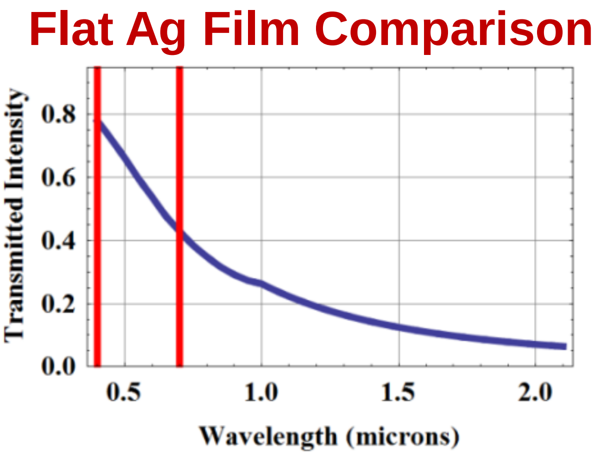
Mirin, *et al.*, Nano Letters **9**, 1255 (2009).
Alvine, *et al.*, Appl. Phys. Lett. **102**, 201115 (2013)
Alvine, *et al*. SPIE Defense, Security, and Sensing. **8725**, 87252H (2013)

Approach – Subwavelength Structure Design

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

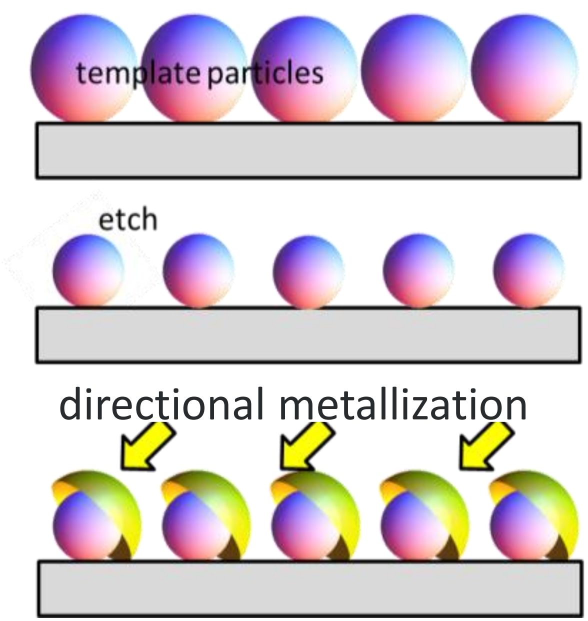






Approach – Subwavelength Array Fabrication (lab scale)

Template Process



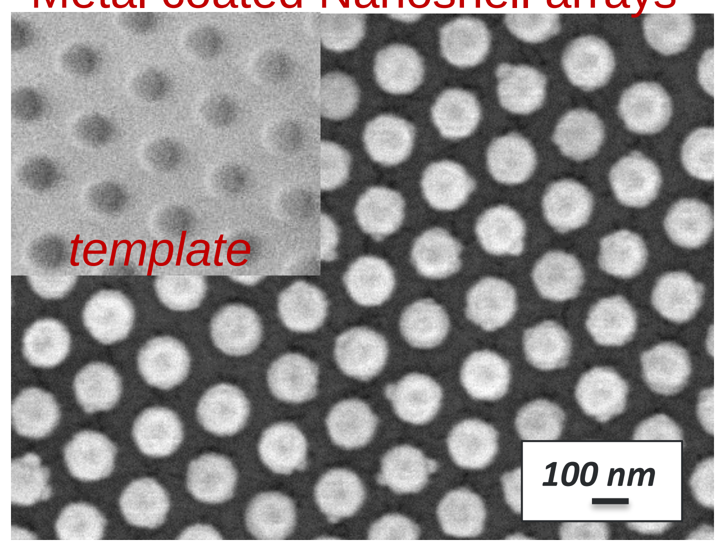
PNNL Fabrication Approach

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

Fabrication Parameters

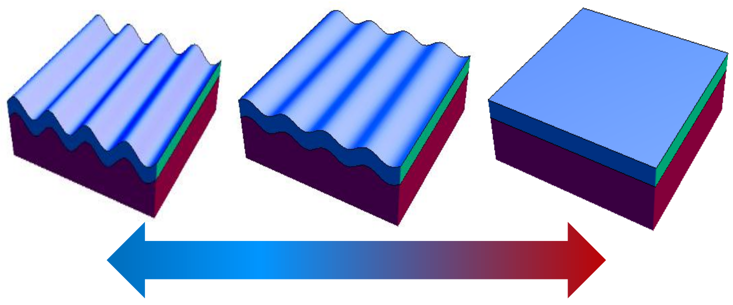
- Particle diameter
- Particle separation (linked)
- Metal thickness
- Metal deposition angle

Metal coated Nanoshell arrays



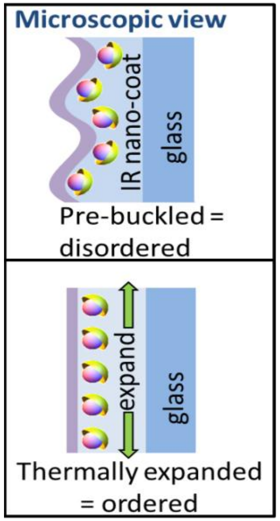
Approach – Reversible Buckling

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

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

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

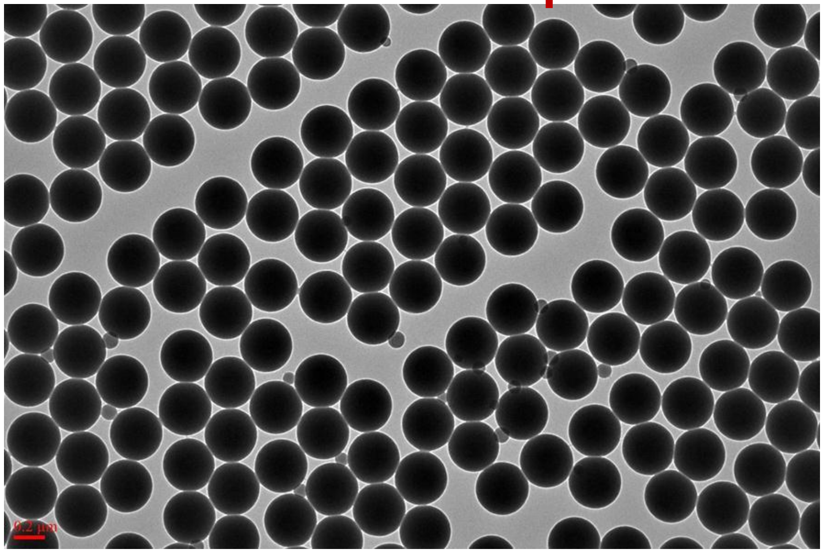
Scale-Up of Template coatings

- 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

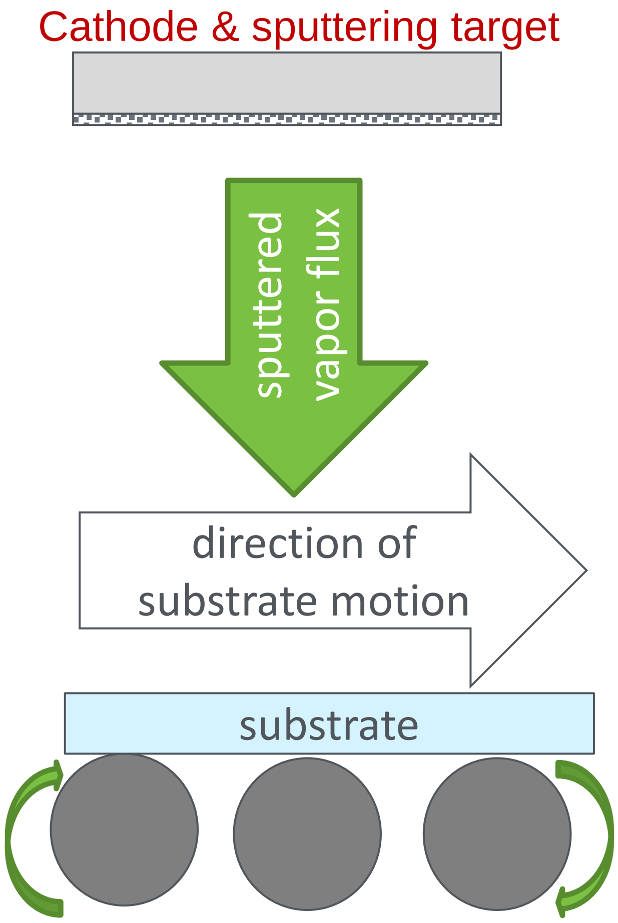


U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy

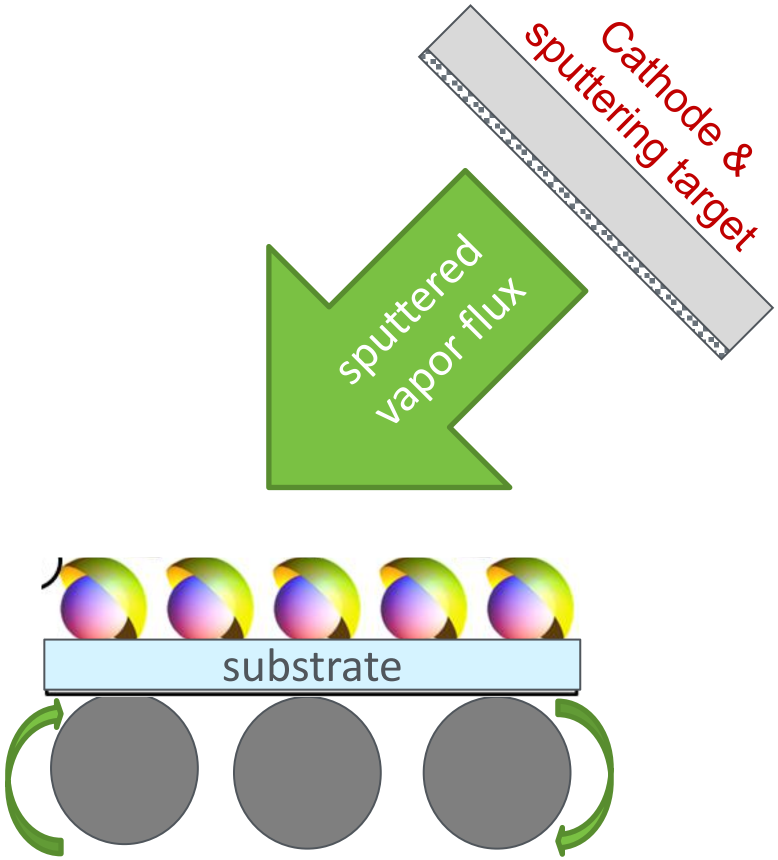
Approach – Scale-Up: Directional Metallization

PPG developing scalable directional metallization

Standard Geometry



Proposed Oblique-Incidence Geometry



Progress and Accomplishments

Lessons Learned: Metal oxidation of the nanoshells has become a potential issue upon migration to Ag. An additional protective coating was necessary, but requires adjustment to retain optimal optical properties.

Accomplishments:

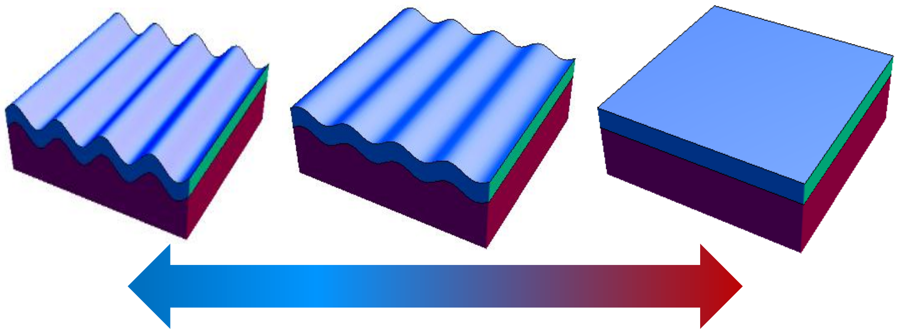
- Demonstration of reversible buckling
- Demonstration of IR specific subwavelength nano-arrays
- Demonstration by PPG of 200 ft. long x 10" wide templates

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: none at this time.

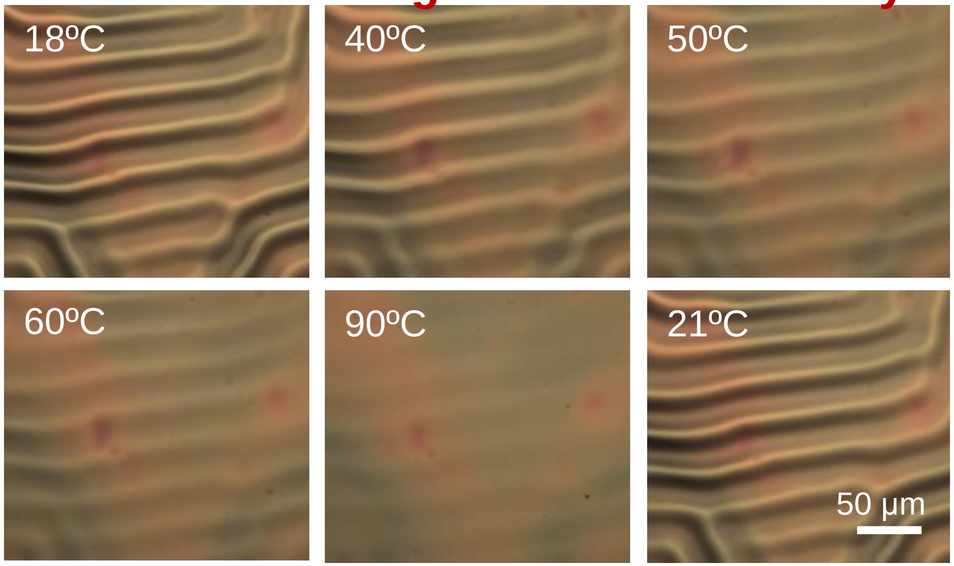
Progress and Accomplishments – Reversible Buckling

Thermally Reversible Buckling



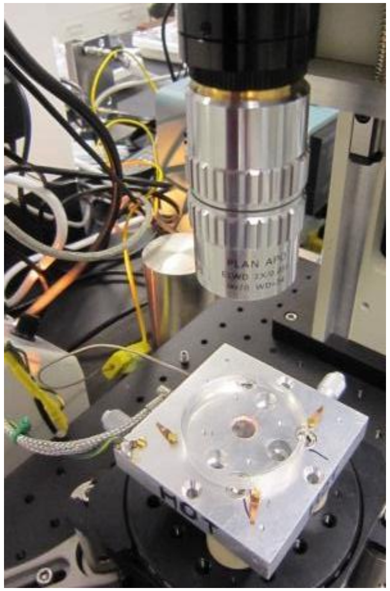
- Fabricated pre-buckled bilayer film
- Demonstrated reversible buckling over multiple cycles with optical microscopy

Reversible Buckling demonstrated >5 cycles



Wavelength chosen to be visible with microscope

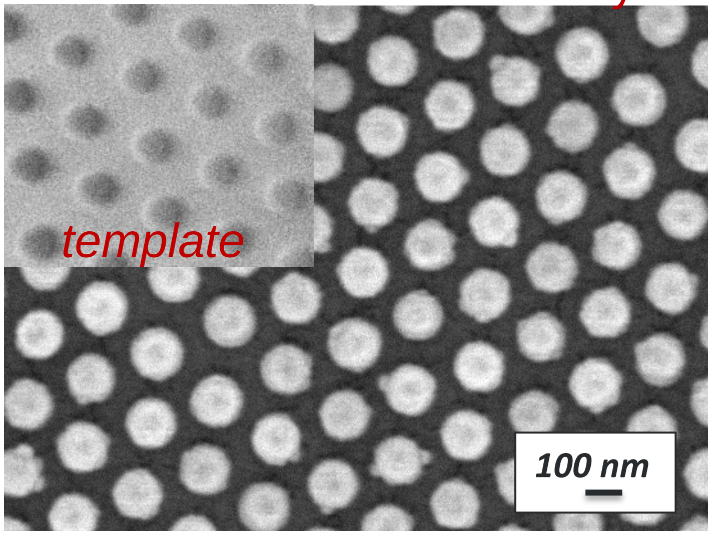
Measured with in-situ optical microscopy heating stage



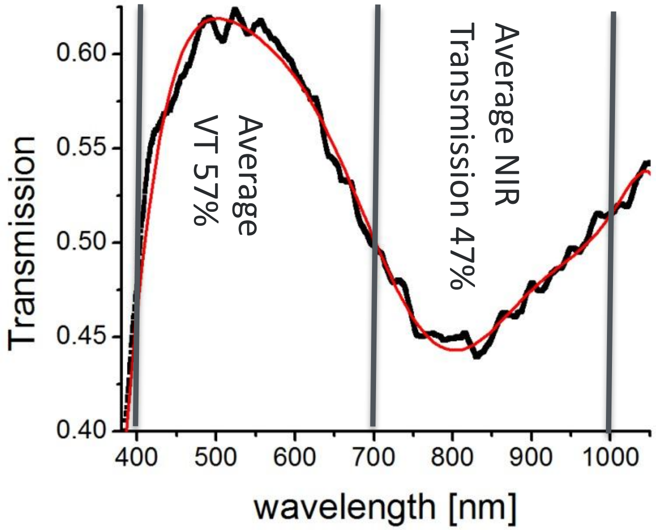
Progress and Accomplishments – Nanoshell Arrays

Demonstration of nanoshell arrays that meet milestone 50% average VT and block 53% of NIR

Metal coated Nanoshell arrays



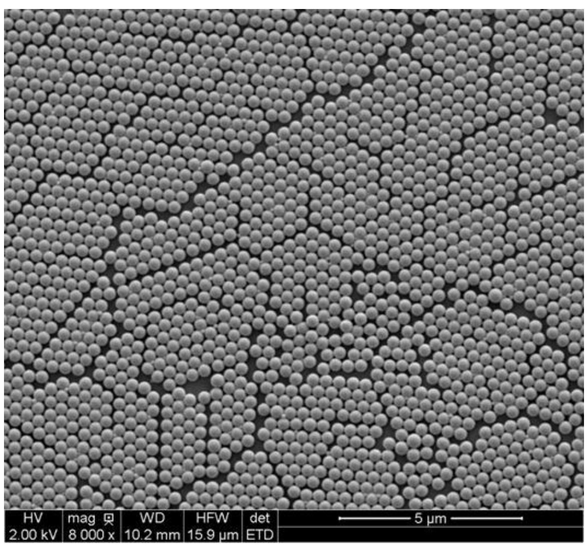
Preliminary Transmission Data



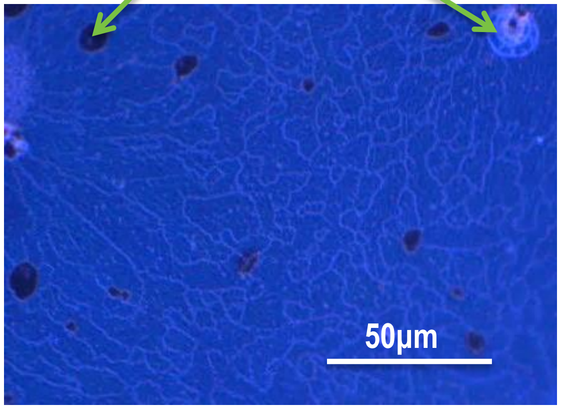
- Demonstration of lab-scale (2" diameter) subwavelength nano-arrays via the template and directional metallization approach.
- Using Ag as optimal metal for nanoshells due to cost and optical properties – still adjusting oxidation protective layer.
- Preliminary data shows >50% average visible transmission with clear dip in NIR transmission, down to 47%.
- Additional optimization of the structures, layers, and control of defects should lead to improved performance.

Progress and Accomplishments – Scale-Up Templates

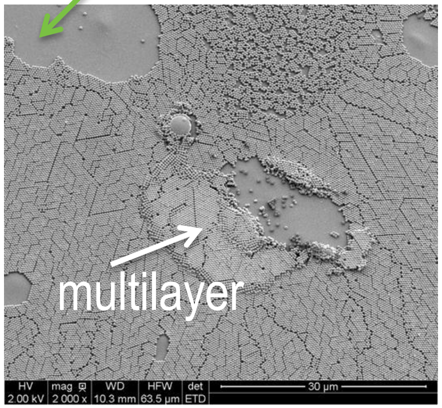
- More than 200ft of 10” wide template coating was made in first trial
- PPG synthesized optimum sized polymer nanospheres with ability to modify diameter if needed
- High degree of monodispersity
- Some defects are present – multilayer and voids in roughly equal amounts
- Opportunities for improvement by identifying and fixing source of repellencies leading to voids



SEM of well ordered monolayer



Optical micrograph of defects



SEM of defects

Progress and Accomplishments – Scale-Up Metallization

- Objective: deposit partial metallic nanoshells on nano-templated substrates supplied by PNNL and PPG
- Directional Sputter Deposition Source
  - Conceptual design in-place
  - Drawings and quotation in-preparation
  - 0-45^o incidence (from normal to substrate surface)
  - Possible need for mechanism to collimate vapor flux
  - Substrate rides on conveyer below source
- Preferred metal = Ag (currently used in Low-E)
- Preferred incident angle = 30^o

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*

Next Steps and Future Plans

Next Steps and Future Plans:

<u>Project Activities</u>:

Lab scale:

- Fabricate disordered subwavelength film
- Integrate buckling and subwavelength film to demonstrate IR switching

Scale-Up

- Evaluate and optimize scaled-up templates and etching
- Complete design and build directional metallization setup
- Fabricate subwavelength films and integrate with switching layer
- Demonstrate intermediate scale IR switching coating

Tasks for Possible Expansion

- Additional work on oxide getters/barriers – potential to improve durability and increase optical performance. Requires additional modeling and lab-scale testing of nanoshells.

REFERENCE SLIDES

Project Budget

Project Budget: $750K Federal Funding, $78K cost share from PPG.
Full Federal Funding of $750K was authorized on 10/1/2013
Variances: N/A.
Cost to Date: Cumulative spent as of 3/21/2014 = $127,281.
Additional Funding: N/A other than cost share.

Budget History					
FY2013 (past)		FY2014 (current)		FY2015 –9/30/2015 (planned)	
DOE	Cost-share	DOE	Cost-share	DOE	Cost-share
N/A	N/A	$375K	$32K	$375K	$46K

Project Plan and Schedule (1 of 3)

Project Plan:

- New Start: 10/1/2014 – 9/30/2015
- 4-6 week extensions granted for FY14 Q2 milestones due to needed repairs/oversubscription on sputter chamber and longer than expected subcontract negotiations. This is not expected to impact future milestones.
- Go/no-go decision point at end of FY14 – see schedule

Project Schedule		
Project Start: 10/1/2013 (New Start FY14)	(blue)	Completed Work
Projected End: 9/30/2015	(dark blue)	Active Task (in progress work)
	◆ (red)	Milestone/Deliverable (originally planned or granted extention)
	◆ (black)	Milestone/Deliverable (Actual)
	◆ (green)	Go/No Go Decision Point

	FY2013	FY2014				FY2015			
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)
Past Work	N/A	new start FY2014							
Current/Future Work (FY14)									
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.		◆							

Project Plan and Schedule (continued, 2 of 3)

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.
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 50% and a temperature switching window of 30 to 90 C.
Q4 Milestone: Supply 6" subwavelength templates for all necessary team members
Q1 FY15 Milestone: PPG-GBDC will complete initial installation, testing, and debug of their directional metallization source.
Q2 FY15 Milestone: Strategy for receiving next scope of funding developed and documented in a straw man-type plan.
Q2 FY15Milestone: Update integrated dynamically responsive IR window coating in BTO prioritization tool based on achieved technical targets and long-term performance/cost goals.
Q2 FY15Milestone: PNNL will revise and update subwavelength array design as needed based on lessons learned from fabrication efforts in year 1.
Q2 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.
Q2 FY15Milestone: PPG-CIC will provide PPG-GBDC with optimized 6” subwavelength templates for directional metallization.
Q3 FY15Milestone: PPG-GBDC will provide PNNL with preliminary 6” metallized nanoshell array samples for integration of buckling layers and optical measurements.

Project Plan and Schedule (continued, 3 of 3)

Q4 FY15Milestone: Integrate buckling and subwavelength films at the 6" scale and optically characterize said coatings. Performance targets of average NIR transmission delta of 10-15% (over 750-900 nm), with an average visible transmission of at least 50%, and a temperature switching in the range of 30-90C.									◆
Q4 FY15 Milestone: Best available metallized 6" samples will be provided to PNNL for integration and optical characterization.									◆