Low-cost Solutions For Dynamic Window Materials

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Task 1: Reduce cost of transparent conducting oxide (TCO) for electrochromic windows,
• started in FY11
• Applicable to existing technology of electrochromic window and other applications

Task 2: Produce films of oxide nanocrystals relevant to dynamic windows by terminated cluster growth,
• started in FY 13
• a contribution to possible disruptive EC window technology

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Purpose & Objectives

Problem Statement:

- Dynamic windows have a potential to better meet the demands of energy savings and comfort than static windows because they can adjust/optimize VIS-IR transmission depending on conditions and needs; savings potential 2-3 Q (*)

- Coatings affect both solar radiation (UV-VIS-near IR) and thermal radiation (far IR)

- Although first dynamic windows are on the market, there is a unfulfilled need to reduce cost in materials and fabrication methods for much broader market penetration

- We want to address the cost issues of the two ITO (indium tin oxide) layers

Purpose & Objectives

Project Focus:
- The project aims to lower the cost of electrochromic windows with focus on ITO replacement because ITO is not scalable to $\geq 10^8 \text{ m}^2$ per year (for comparison: world market of coated glass is $10^9 \text{ m}^2$)
- Assuming today’s materials supply, the cost of Zn is less than 10% of cost of In; the cost of AZO (aluminum-doped zinc oxide) is about 25% of ITO
- Besides a material solution, a high rate, low cost fabrication method is needed
- Material and method must be compatible with existing production equipment (in-line coaters using sputtering)

Impact of Project: Removing one barrier for broad introduction of EC windows
Approach
ITO vs. AZO, fabrication methods

Approach:
• In previous work, AZO was identified as the lowest cost TCO material having good performance when deposited with optimized conditions
• AZO can be deposited by various methods, including sputtering
• Best performing film material has been obtained with filtered cathodic arc plasma deposition

Key Issues:
• For sputtering: negative oxygen ions are accelerated to the substrate, where they cause “ion damage” – degradation of optical and electrical properties
• For filtered cathodic arc deposition: plasma has particulates, and current filters are difficult to scale

Approach 1: Plasma formation and negative ion filtering

Heated glass substrates

Plasma Lens as Negative Ion Filter

Magnetron in high power pulsed mode

Ar/O₂ gas

Target /cathode Zn:4at%Al

A. Anders, patent application US2011031566.
Approach 2: Low voltage processing using a filtered arc

- arc does not use high voltage → no acceleration of negative ions → no ion damage

This known “conventional” filtered arc gives very good AZO films but is difficult to scale in this configuration → the task is to develop a better approach.
Accomplishments and Progress: Comparative study of processes

To be published at MRS Spring Meeting 2013.

Should be < 8 x10^{-4}

Should be > 30

Resistivity, $\rho$ (\(\Omega\) cm\(^{-1}\))

Electron Concentration, n (cm\(^{-3}\))

Mobility, $\mu$ (cm\(^2\)/Vs)

AZO

Magnetron

HIPIMS

Pulsed Arc

Filtered HIPIMS

$10^1$

$10^0$

$10^{-1}$

$10^{-2}$

$10^{-3}$

$10^{-4}$

$10^{19}$

$10^{20}$

$10^{21}$

$10^{22}$
Development of innovative sources and filters

IB-3310: arc source for AZO

AZO, 605 nm, 18 Ω/☐

ITO, 140 nm, 7.9 Ω/☐

Solar spectrum

IB-2013-049 as preliminary step, now IB-2013-071 “Island filter”, LBNL decided to file on 2013-03-26 as US patent application
• In a preliminary experimental verification of the new, scalable arc-filter geometry, we deposited AZO that has comparable or even better properties that the previous record AZO made with a 90-degree filter.

Efforts in rest of FY13 will focus on demonstrating the scalability to 6” linear system, showing at least 6”x6”, possibly 6”x12” sample by the end of FY13.
**Project Plan & Schedule**

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<th>WBS Number or Agreement Number</th>
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<tbody>
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<th>FY2013</th>
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<td><strong>Project Name: Low-cost Solutions For Dynamic Window Materials;</strong></td>
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<td><strong>Task 1: ITO replacement</strong></td>
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<td>Q4 Milestone: Produce AZO at high rate &gt; 100 nm/min on cm^2 sample</td>
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<td>Q4 Milestone: Lay out concepts for scaling principles</td>
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**Current work and future research**

- Q2 Milestone: Evaluate effectiveness of negative ion filter
- Q3 Milestone: Evaluate liquid metal plasma source
- Q4 Milestone: Based on findings, use ion-filtered sputtering or particle-filtered arc deposition to demonstrate high-quality, high-rate AZO on 6"x6" sample
- Q2 Milestone: Demonstrate miniature linear system in preparation of industrial scaling based on go-no go decision for filtered sputtering vs. filtered arc
- Q4 Milestone: Develop scaled system in collaboration with industrial partner; transfer technology

- Preliminary report on negative ion filtering will be given as Invited Talk at MRS Spring Meeting on 2013-04-05; direct O⁻ detection is still outstanding.
- Liquid metal source gave many unexpected problems, we changed approach, which lead to 3 invention disclosures in FY13, incl. a novel, scalable arc filter geometry
- We are on track with overall goals for FY13.
Project Budget

Funding (FY13): $430K for task 1, and $270K for task 2 (incl. c/o from FY12).

Team: André Anders (Sr. Scientist), Cesar Clavero (Research Scientist), Jonathan Slack (Sr. Research Associate), Jonathan Kolbeck (Student Intern); Rueben Mendelsberg (Visiting Scientist). Total effort funding = 2 FTEs for tasks 1 and 2.

- Task 1 – started in FY11
- Task 2 – started in FY13

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<th>Budget History for Task 1</th>
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Partners, Subcontractors, and Collaborators:

- Several companies have shown interest provided reasonably sized prototypes (~ 1 sq. ft) are demonstrated with properties comparable to the ITO benchmark, incl. Sage (St. Gobain), Guardian, Pilkington, Glas-Trösch
- AZO is also of interest to PV manufacturers, and inquiries come from this side of the market.

Technology Transfer, Deployment, Market Impact:

- 8 invention disclosures have been filed since 2010, and 2 of them were selected for filing with the USPTO
- After having demonstrate 6”x6” samples by the end of FY13, we’ll actively seek partners to built a system for in-line coating. Sage communicated interest.

Communications:

- 12 journal papers on TCO and related processes since 2010,
- most notable paper: R. J. Mendelsberg, et al., J. Phys. D: Appl. Phys. 44 (2011) 232003, which was downloaded more than 500 times in the first 90 days, and which was selected by the journal as one of the most impactful publications of the year.
- 4 Invited Talks at International Conferences
Next Steps and Future Plans: Task 1

a) For the negative ion filter approach:
   • Prove the absence of negative ions at the substrate in a direct way
   • Demonstrate linear scaling of the neg. ion filter

b) For the filtered arc approach:
   • The quality of the material was demonstrated to be good, the deposition rate on a small area is high, AZO cost is low compared to ITO → demonstration of a linear source, scalable for in-line coaters, delivering equally good features and properties is needed. A 6-inch source is under construction.

Scaling to even larger size, sputtering or arc, should be done with an industrial partner. Work is toward a go / no-go decision for those two approaches by the end of 2013.
For completeness: Task 2
Objectives & approach, first results

Task 2: Produce films of oxide nanocrystals relevant to dynamic windows by “terminated cluster growth”.

• Nanocrystals enable the switching of the Solar IR portion of the spectrum → an EC window can be designed that switches independently in the solar IR and in the visible (a different project, funded by ARPA-E, Milliron et al.).
• Here, task 2: demonstrate nanocrystal fabrication that can be integrated with existing sputter technology: we use “terminated cluster growth”.
• Nanoparticles (copper, vanadium) have been synthesized and analyzed
• First oxide nanoparticles have been made and deposited as a film