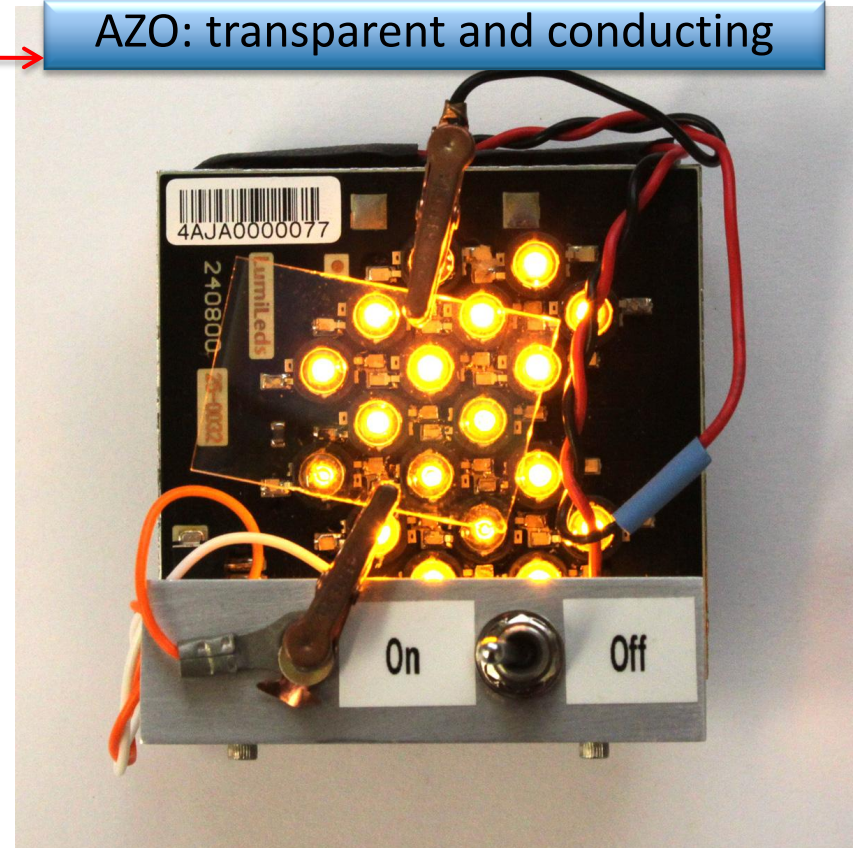


AZO: transparent and conducting



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

*The focus of
this report.*

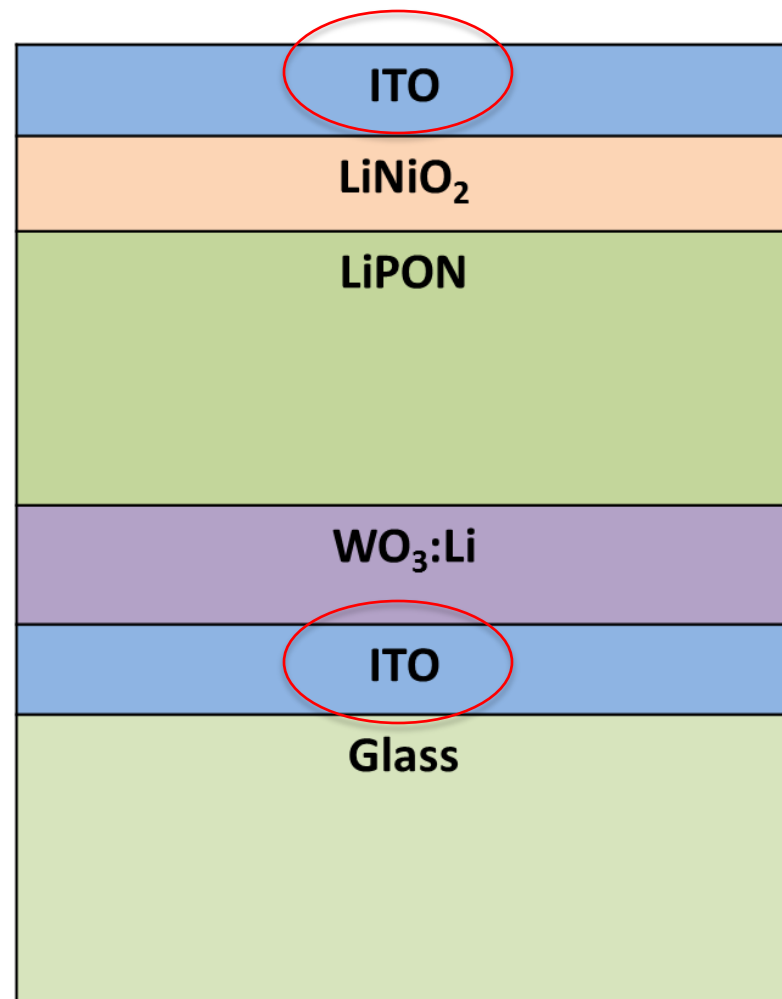
Task 2: Produce films of oxide nanocrystals relevant to dynamic windows by terminated cluster growth,

- *started in FY 13*
- a contribution to possible [disruptive EC window technology](#)

*A slide at the end
of this report.*

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



(*) D. Arasteh et al., Proc. 2006 ACEEE Summer Study on Energy Efficiency in Buildings, Pacific Grove, LBNL-60049

Project Focus:

- The project aims to lower the cost of electrochromic windows with focus on ITO replacement because ITO is not scalable to $\geq 10^8$ m² per year (for comparison: world market of coated glass is 10^9 m²)
- 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

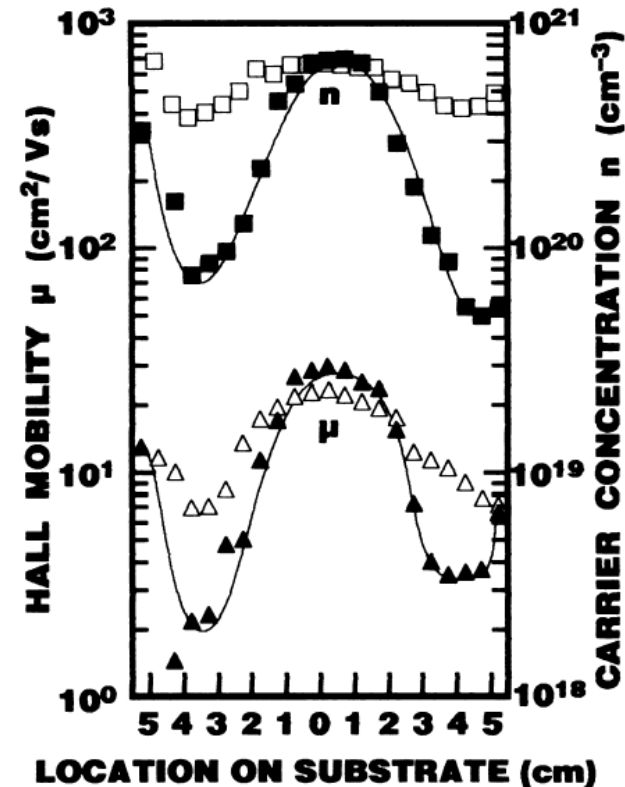
Approach:

- 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

Illustration of ion damage



T. Minami, T. Yamamoto, T. Miyata,
Thin Solid Films 366 (2000) 63.

Approach 1:

Plasma formation and negative ion filtering

Heated glass
substrates

Plasma Lens as Negative Ion Filter

Magnetron in high
power pulsed mode

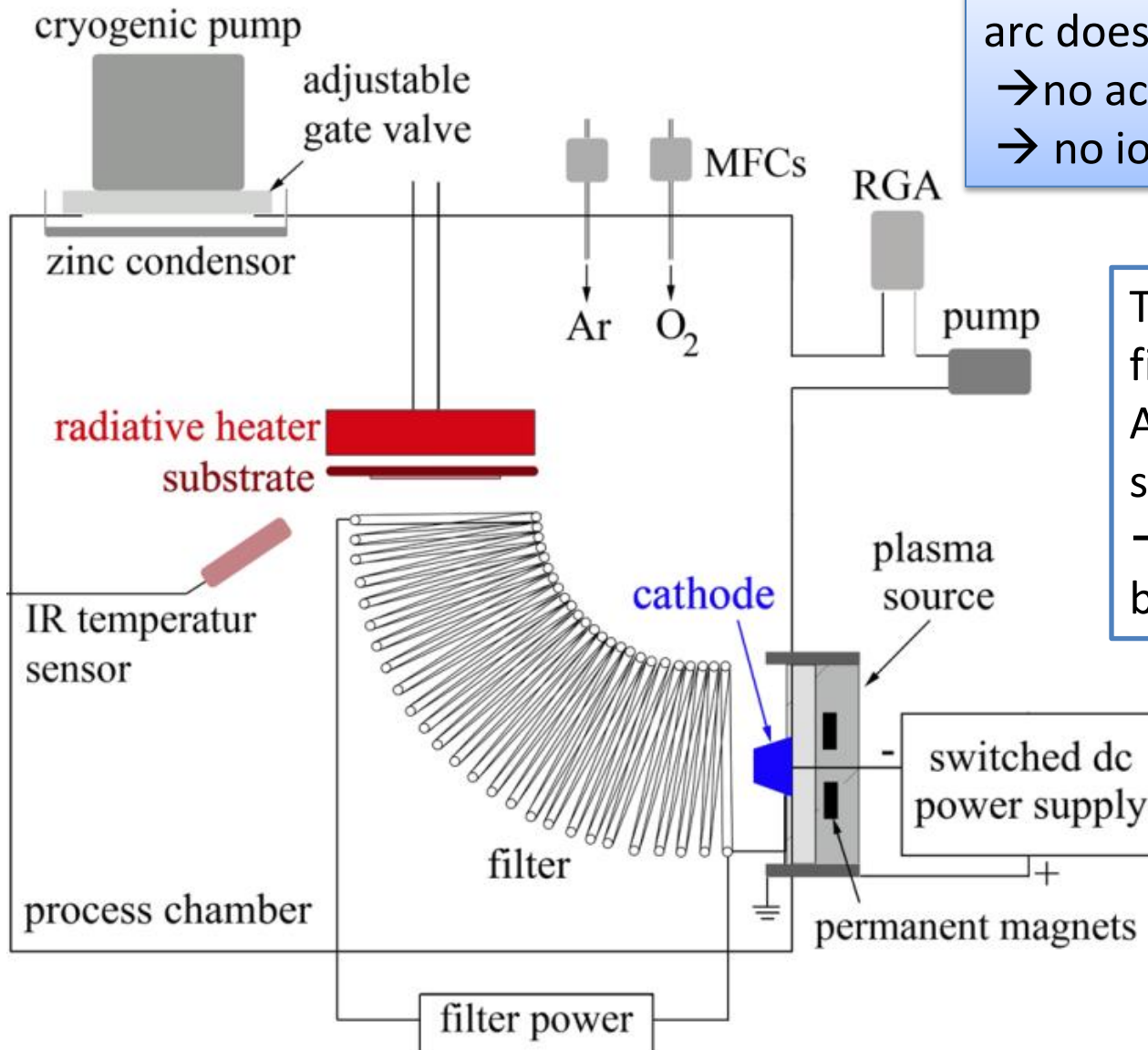
Ar/O₂ gas

Target
/cathode
Zn:4at%Al

A. Anders, patent application US2011031566.

Approach 2:

Low voltage processing using a filtered arc

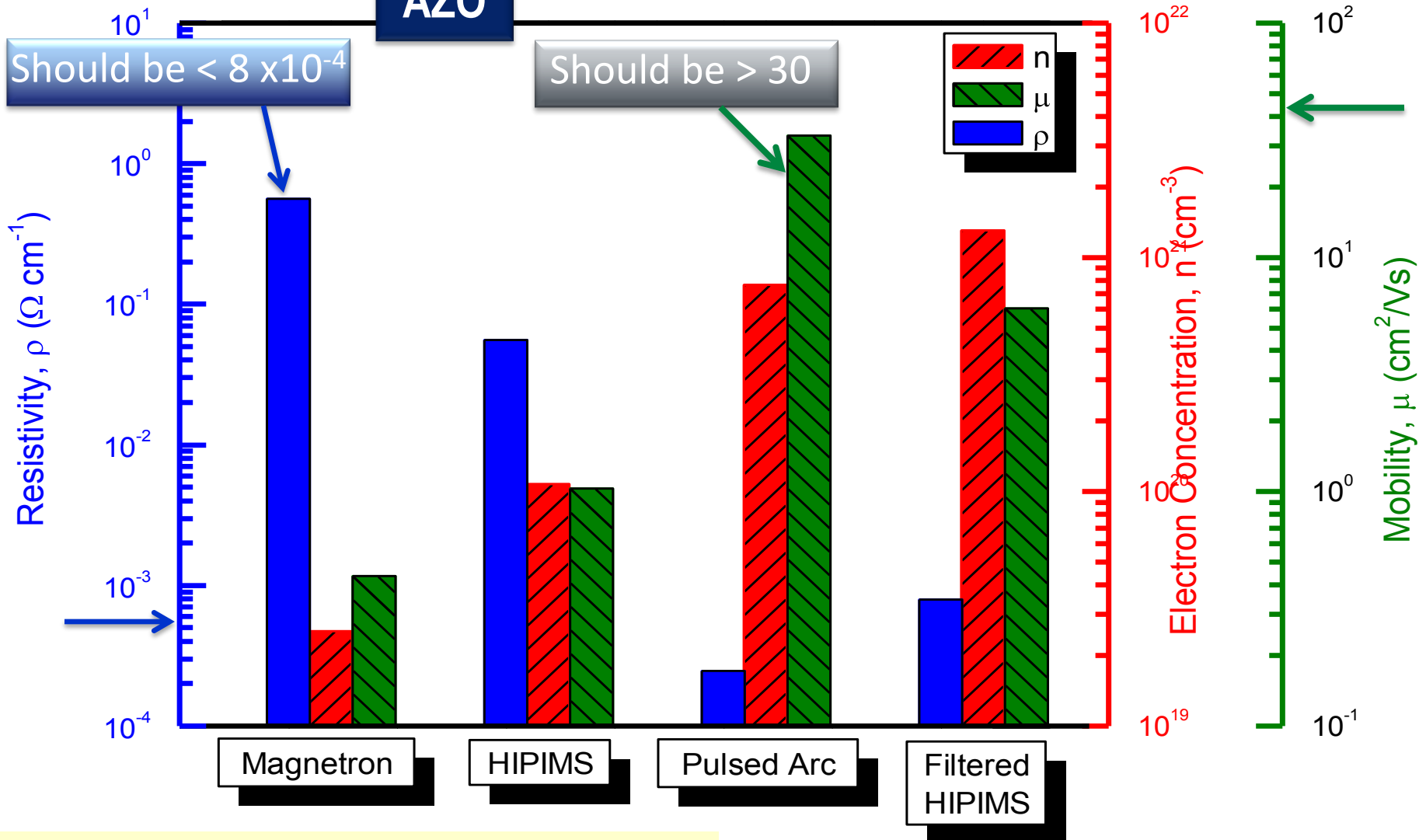


arc does not use high voltage
→ no acceleration of negative ions
→ no ion damage

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

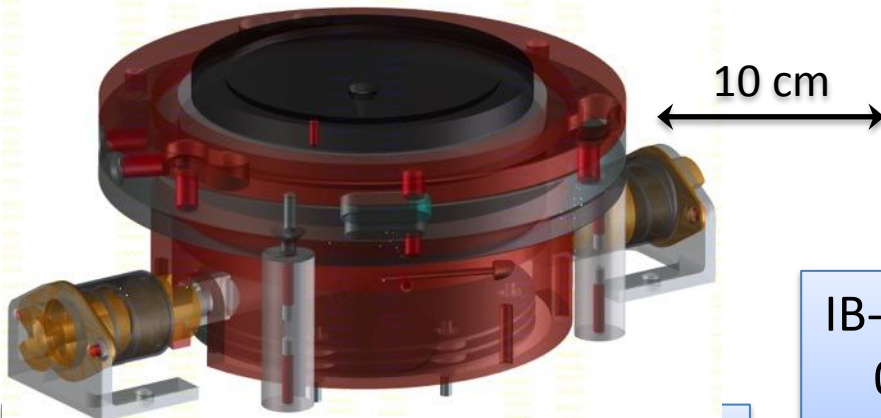
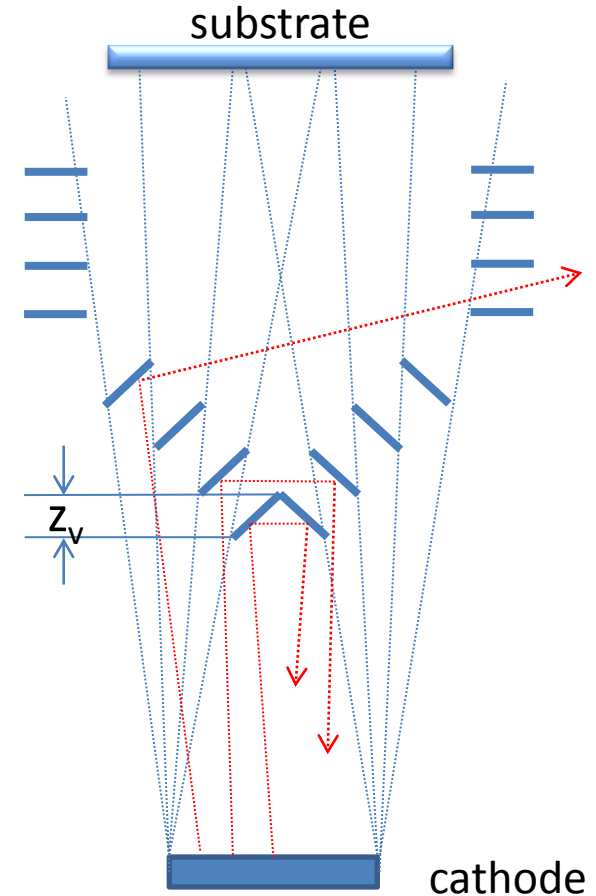
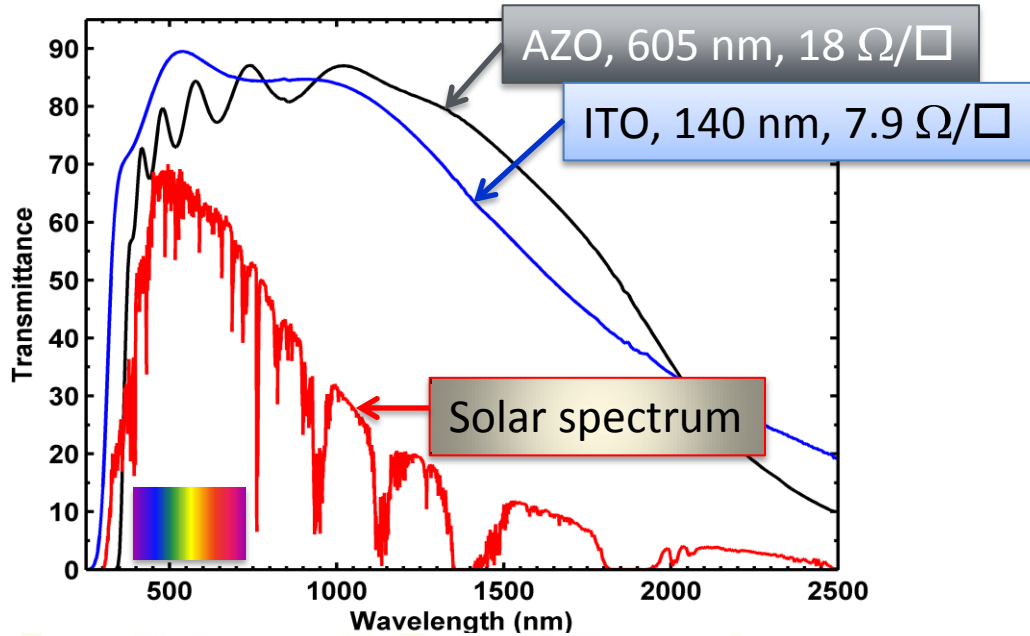
AZO



To be published at MRS Spring Meeting 2013.

Accomplishments and Progress

Development of innovative sources and filters



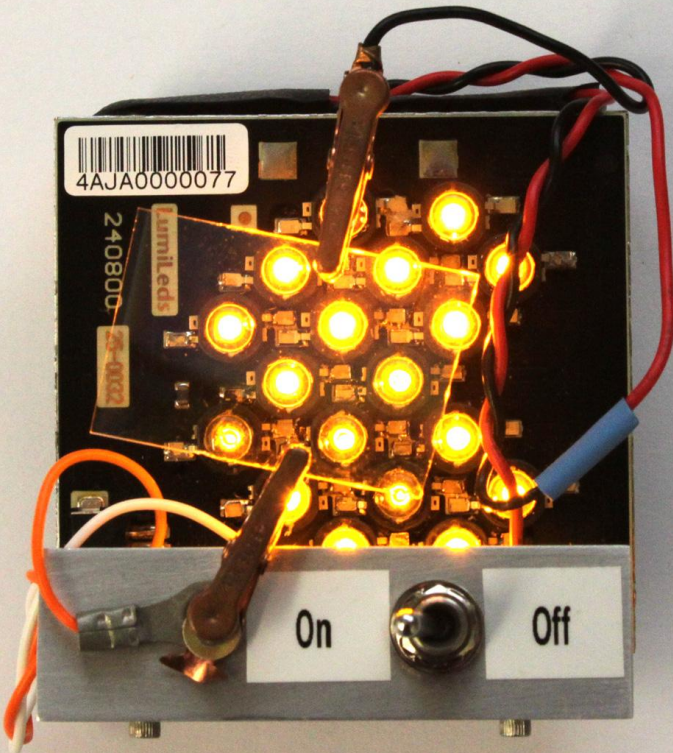
IB-3310: arc source for AZO

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 than the previous record AZO made with a 90-degree filter.

Gadget showing working of AZO deposited on glass. We also accomplished deposition at room temperature (!) relevant e.g. for OLEDs

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

Summary					Legend							
WBS Number or Agreement Number	BT-480010-13				Work completed							
Project Number	18835				Active Task							
Agreement Number	7310				Milestones & Deliverables (Original Plan)							
					Milestones & Deliverables (Actual)							
	FY2012				FY2013				FY2014			
	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)
Task / Event												
Project Name: Low-cost Solutions For Dynamic Window Materials;												
Task 1: ITO replacement												
Q4 Milestone: Produce AZO at high rate > 100 nm/min on cm ² sample				◆								
Q4 Milestone: Milestone: lay out concepts for scaling principles				◆								
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.

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

Budget History for Task 1

FY2010		FY2011		FY2012	
DOE	Cost-share	DOE	Cost-share	DOE	Cost-share
0	0	50	0	200	0

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:

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

