High-Strength Electroformed Nanostructured Aluminum for Lightweight Automotive Applications

Robert Hilty
Xtalic Corporation
2017 DOE Vehicle Technologies Program
Annual Merit Review

Project ID: LM089

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Overview

Timeline

- Project start: Oct 2014
- Project end: Sep 2018
- Percent complete: 60%

Budget

- Total project funding
  - DOE share: $2.50 M
  - Xtalic cost share: $2.56 M
- Funding for FY15
  - DOE share: $643K
  - Xtalic share: $660K
- Funding for FY16
  - DOE share: $679K
  - Xtalic share: $697K
- Funding for FY17
  - DOE share: $644K
  - Xtalic share: $660K

Barriers

- Performance: Achieve substantially better properties. (Minimum specifications: UTS> 600 MPa, ductility>8%).
- Manufacturability: Manufacture advanced materials in production quantities and with the required precision and reproducibility.
- Cost: High potential cost is the greatest single barrier to the market viability of advanced lightweight materials.

Project Partners

- Xtalic Corporation (Lead)
- Fiat Chrysler Automobiles US
- Tri-Arrows Aluminum

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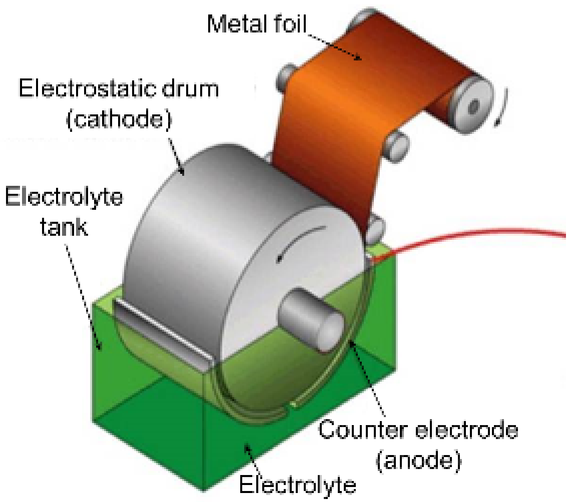
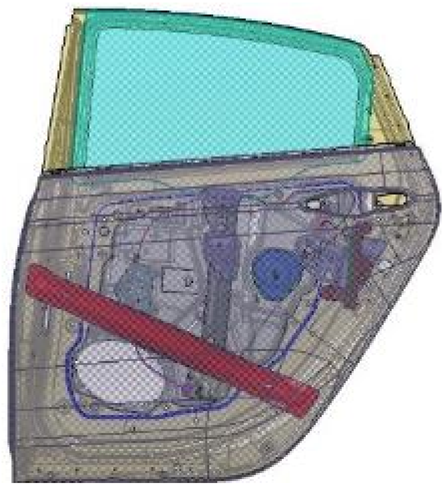
Relevance and Project Objectives

- **Overall objectives of DOE program and the DOE-VT MYPP:**
  - Develop a commercial process to manufacture high-strength nano-Al sheet



Schematic of a continuous system used to electroform metal foils



Example of nano-Al demo part (Rear door side impact beam)

- **Project Objectives** (Budget periods 2 & 3)**:**
  - Develop nano-Al sheet electroforming capacity
  - Optimize process output and consistency
  - Fabricate alloys, optimize and down-select

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Milestones Budget Period 3

Tasks	Program Quarter			
	1 Jan-Mar	2 Apr-Jun	3 Jul-Sep	4 Sep-Dec
Produce alloys, optimize and down-select				
• Alloy Fabrication (M5)				
• Alloy Testing (M6)				
• Root Cause Analysis (M7)				
• Alloy selection (M8)				
Go/No Go: Economic conditions (M9)				

Xtalic utilized a no-cost-extension.
Project is operating one quarter behind fiscal calendar.

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Approach/Strategy

Tasks	Year			
	1	2	3	4
Optimize process output and consistency				
Develop continuous electroforming system				
<u>Go/no-go: Engineering feasibility of design</u>				
Build and validate pilot line				
<u>GNG: Verify system functionality, deliver 1 sample of 6” x 6” sheet</u>				
Fabricate alloys, optimize properties, downselect				
Economic modeling				
<u>Go/no-go: Economic viability of nano-Al sheet production</u>				
Fabricate preferred alloy(s), test against full specs				
Lab demonstration of economic viability				
Management and reporting				

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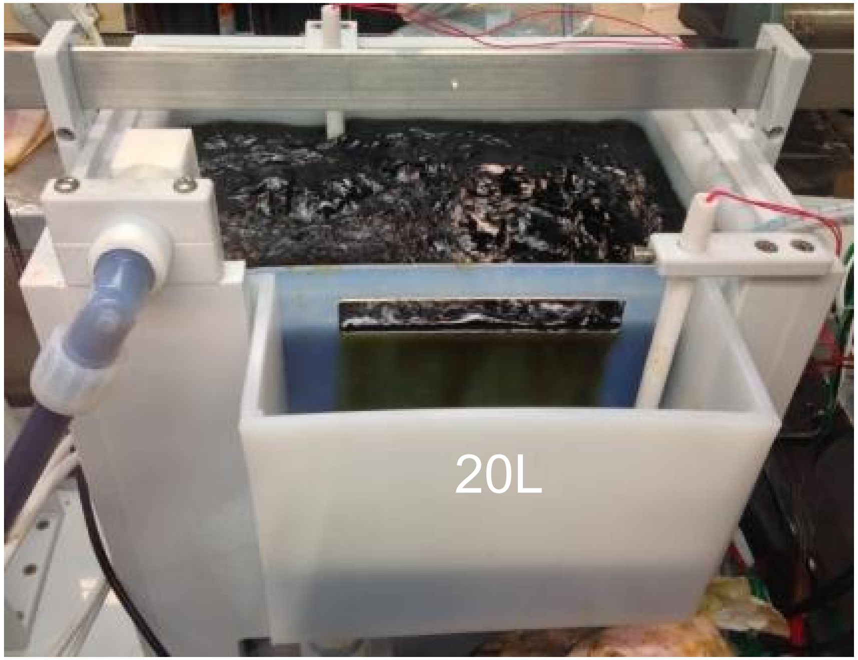
Nano-Al Electrodeposition

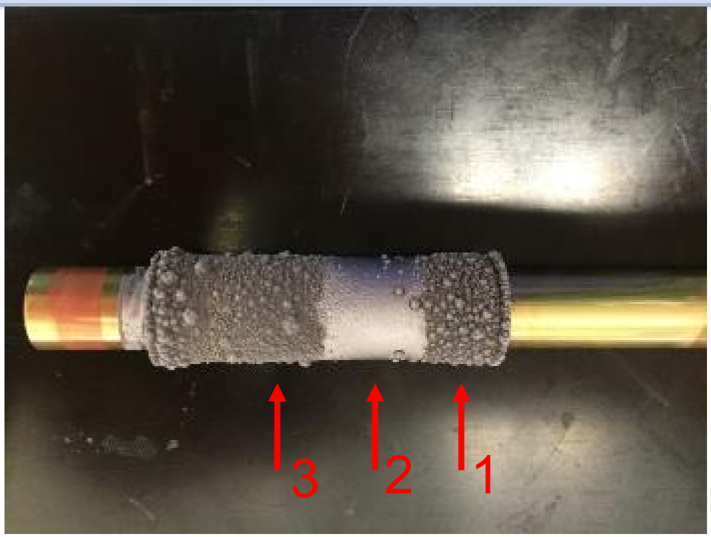
$$4\ Al_2Cl_7^- + 3e^- \longrightarrow Al + 7\ AlCl_4^-$$

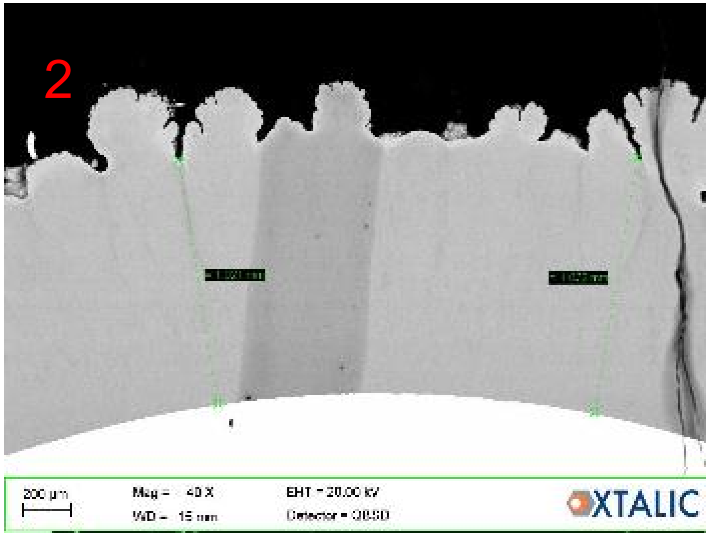


- Al can't be plated from aqueous based electrolytes
- Ionic liquids provide source of Al ions and electrical conductivity
  - Lewis acid EMIM:Cl
  - Less conductive than aqueous
  - More metal content
  - Requires new additive strategy
- 20 liter scale up reactor

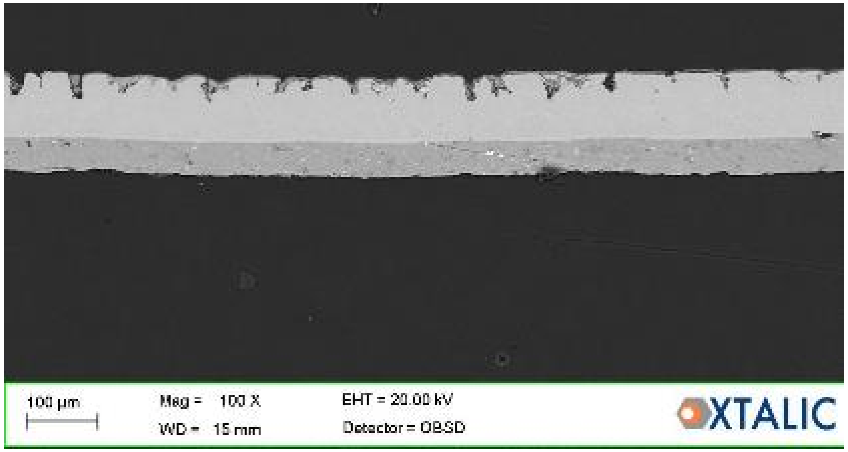
Technical Accomplishments and Progress

Plating Chemistry and Porosity





- Plating additive has been a key development issue
- Additives prevent dendrites
- Product must be smooth



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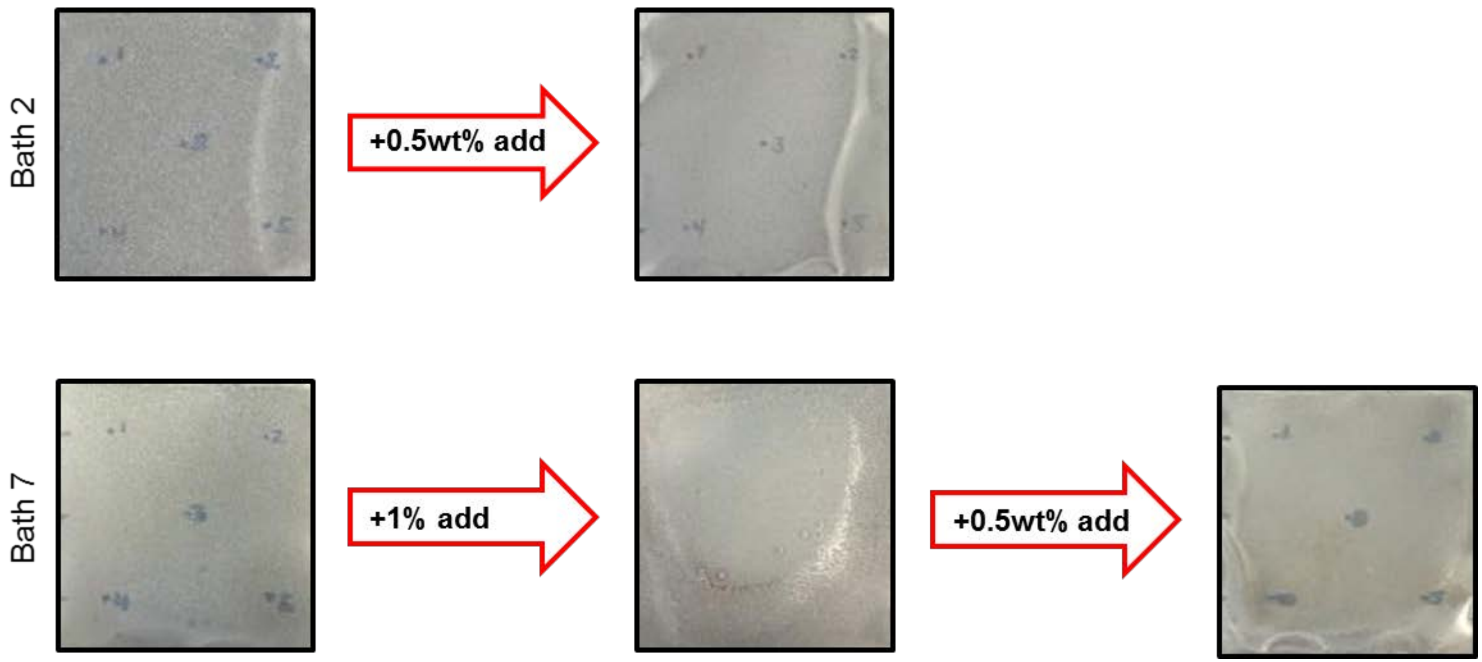
Technical Accomplishments and Progress

Plating Chemistry and Porosity

- Plating baths show a decrease in additive activity over time
  - Additive consumed or lost with bath age



- Additive loss rates computed and tracked

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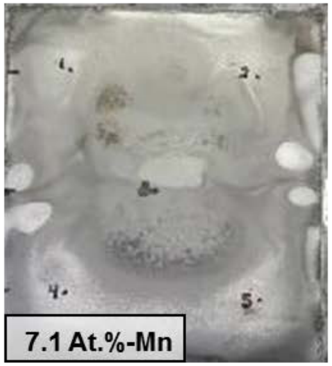
Technical Accomplishments and Progress

Plating Chemistry and Porosity

- Additive optimization improves color
- Mn content linked to additive use and plating temperature



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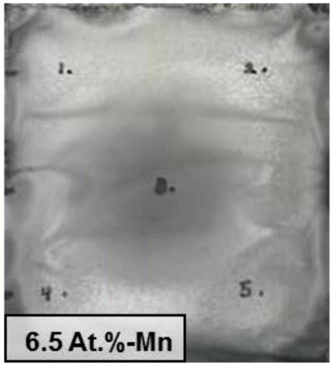

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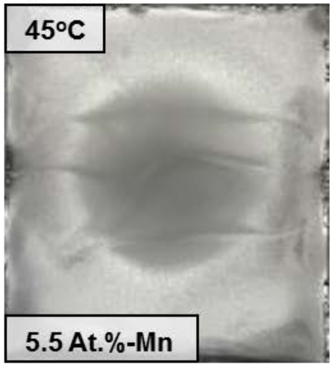
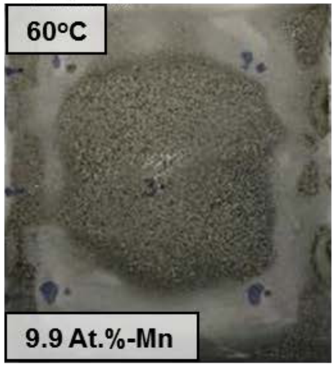
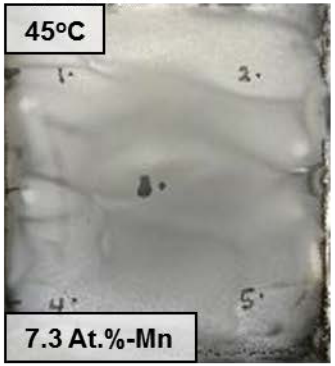


020217
Waveform 2
250-445um



020617
Waveform 2
320-400um



020717
Waveform 3 (rest increase)
215-400um

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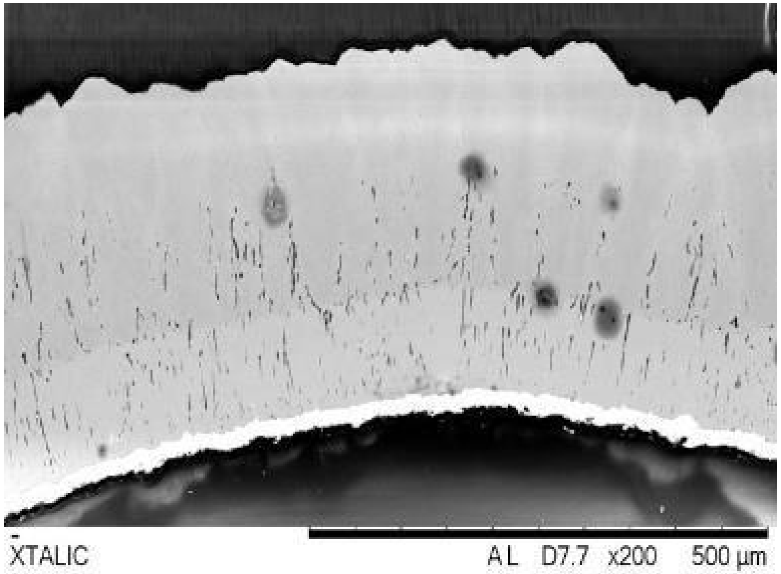
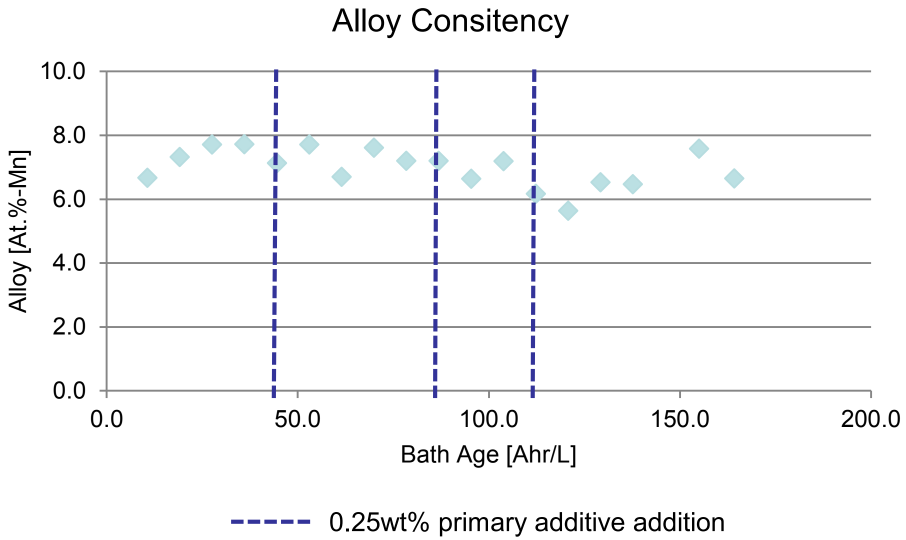
Technical Accomplishments and Progress

Plating Chemistry and Porosity

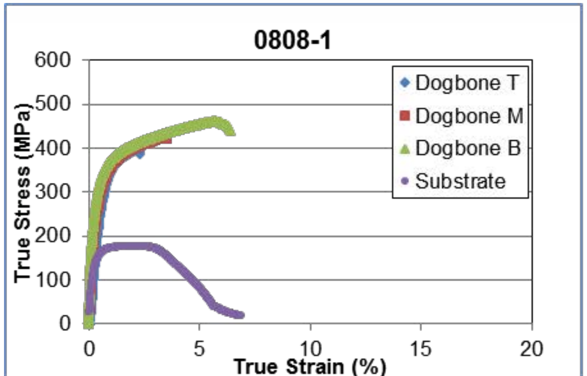
- Additive component can be over-dosed, leading to porosity
  - Treatment process developed to remove
- Thickness:
  - > 250um is now routine
  - 500um common

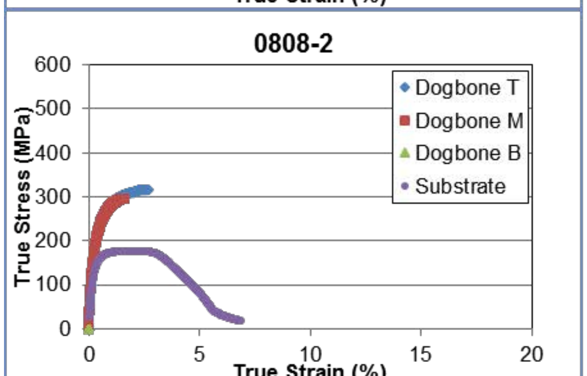


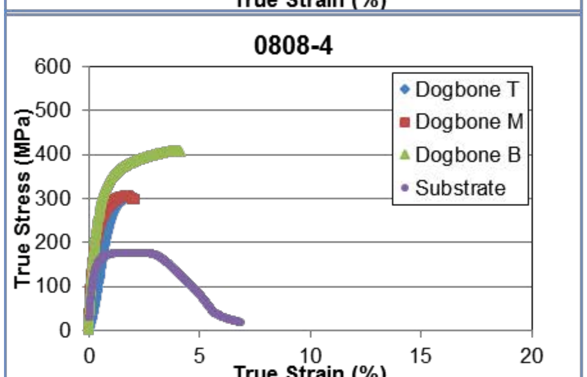


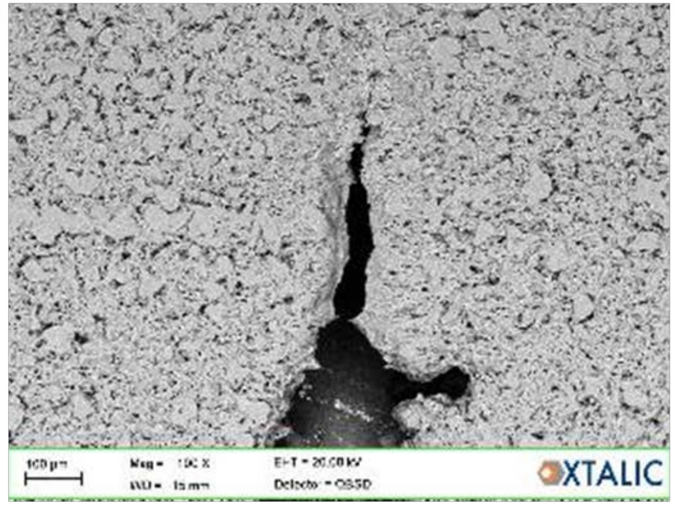
Technical Accomplishments and Progress

Plating Chemistry and Porosity









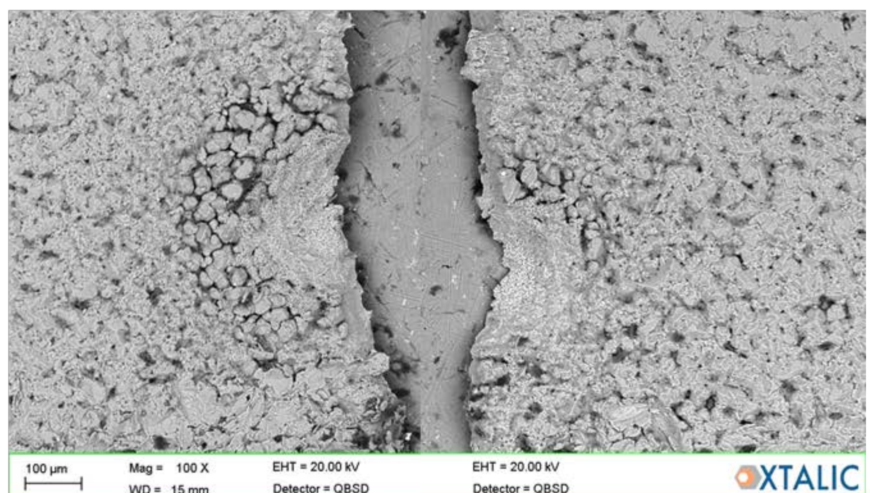
Partial fracture in tensile sample

Evidence of local ductility

Surface roughness and microcracks reduce ductility

Need smoother deposits and better sample prep

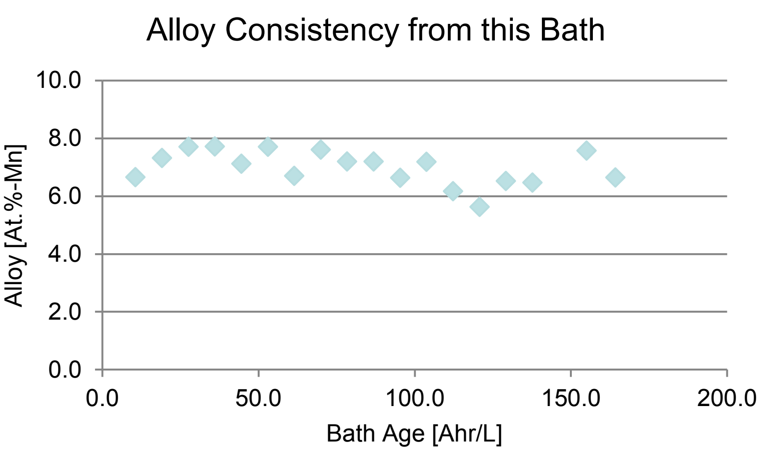
Improved filtering required

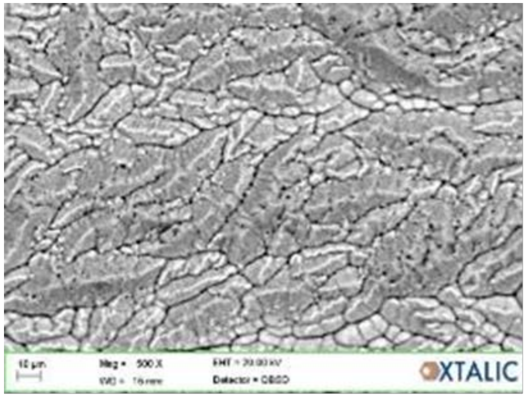


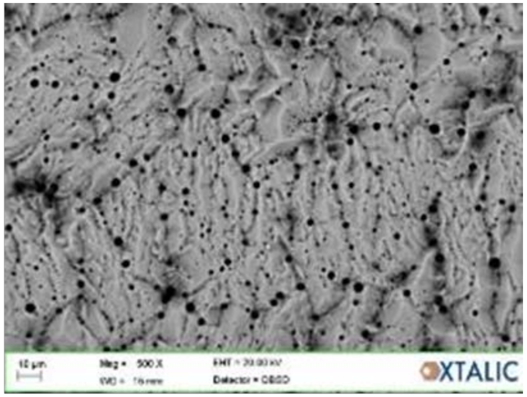
Technical Accomplishments and Progress

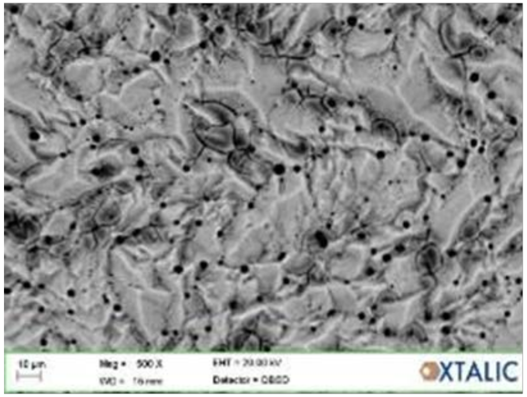
Plating Chemistry and Porosity

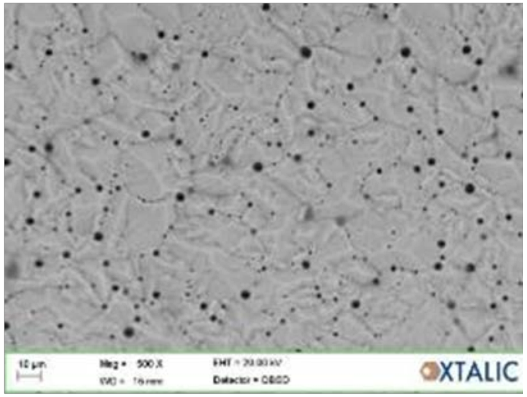
Sample ID	Composition [At.%-Mn]	Thickness [um]
091216	7.6	300
091916	7.0	520
092216	7.0	450
092616	7.1	450




091216 topdown QBSD 500x


091916 topdown QBSD 500x


092216 topdown QBSD 500x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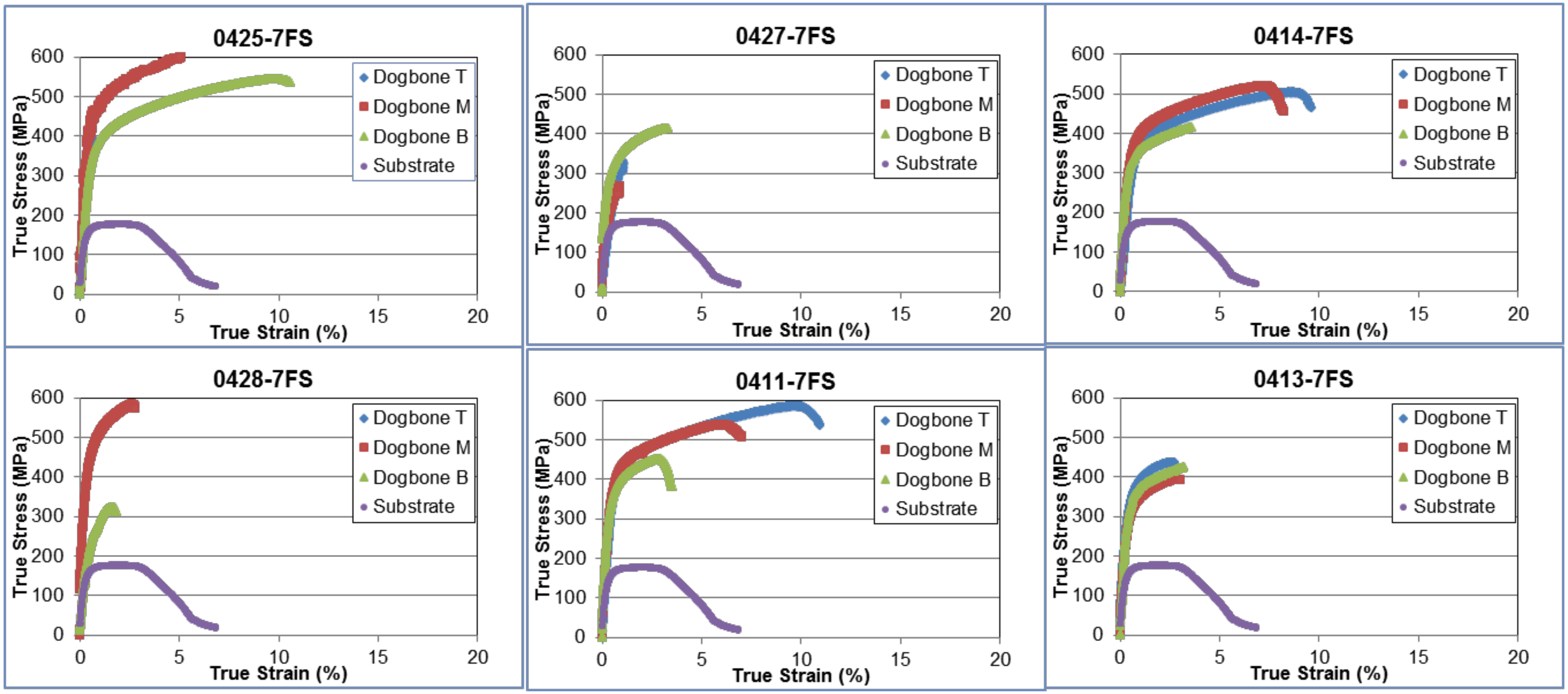
Technical Accomplishments and Progress

Free Standing Nano-Al Mechanical Properties



- Free standing foils removed from substrate
- Variable ductility can lead to inconsistent strength
- Higher Mn content boosts strength (Mn target increase)

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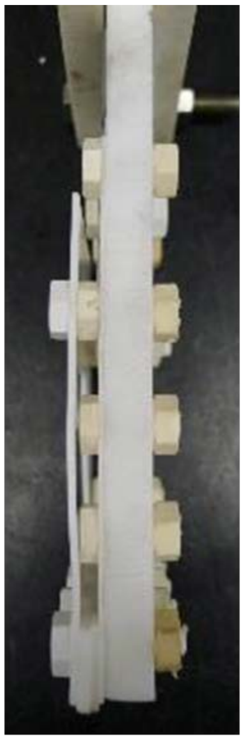
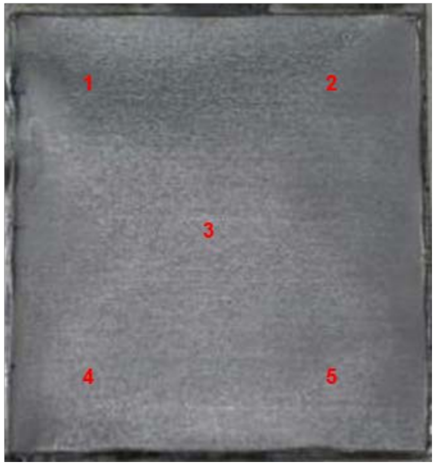
Technical Accomplishments and Progress

Plating Scale-Up: 50x50mm



- Scale process to 50x50mm flat panels
- 20 liter reactor
- PTFE fixturing with controlled gap
- Flat anode, parallel to cathode
- Thickness at target (400um+)
- *Goal: Improve thickness uniformity*

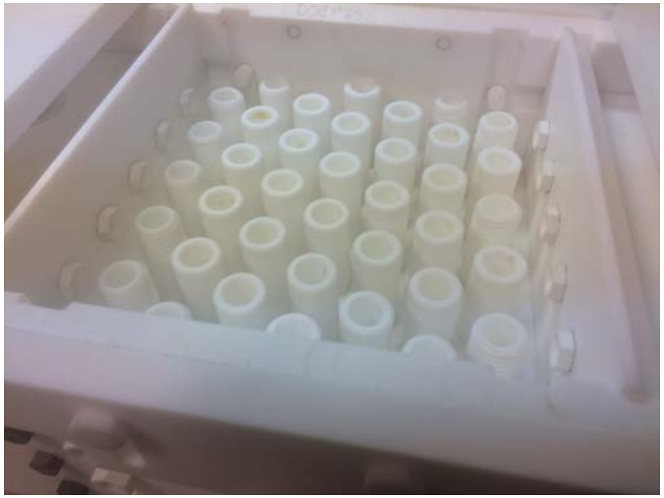


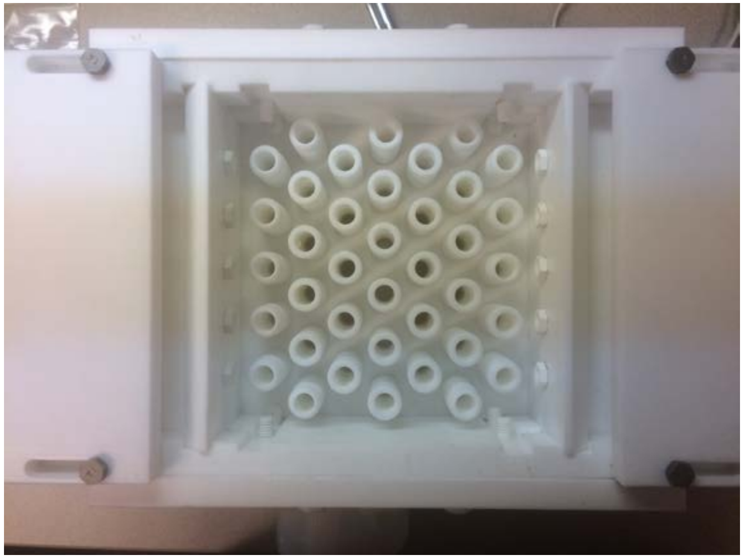
Location	Thickness [um]
1	480
2	470
3	380
4	480
5	460

Technical Accomplishments and Progress

Plating Cell

- Scale-up of plating cell
- 6x6 inch plating cell
  - Large flow field with 38 jets
  - Anode is placed between jets
  - Jets can be adjusted for
    - Anode to cathode spacing
    - Diameter of orifice
  - Jets can also be selectively depopulated to control flow pattern
  - PTFE and Ti construction for chemical compatibility
    - Other polymer materials may be possible





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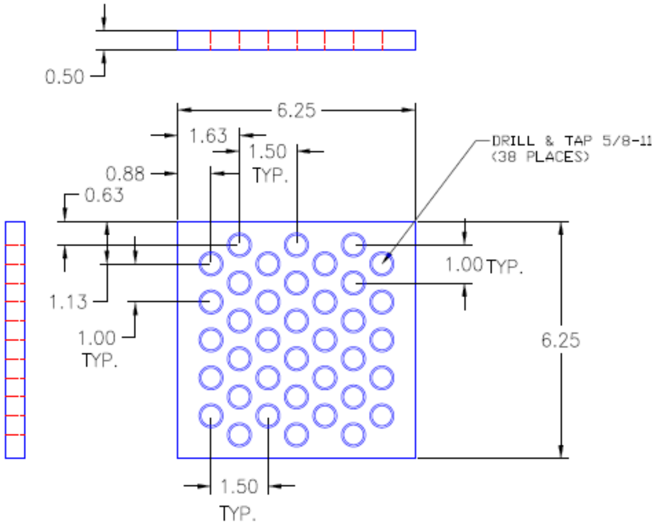
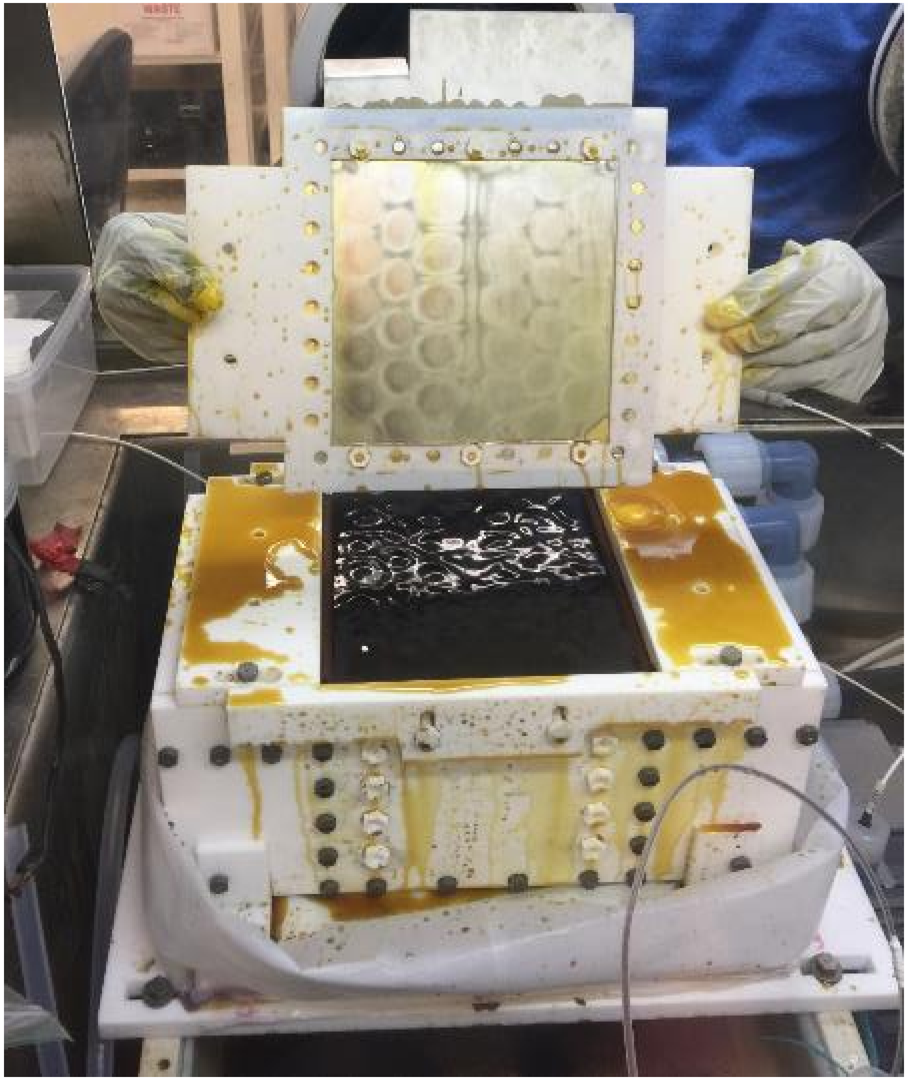
Technical Accomplishments and Progress

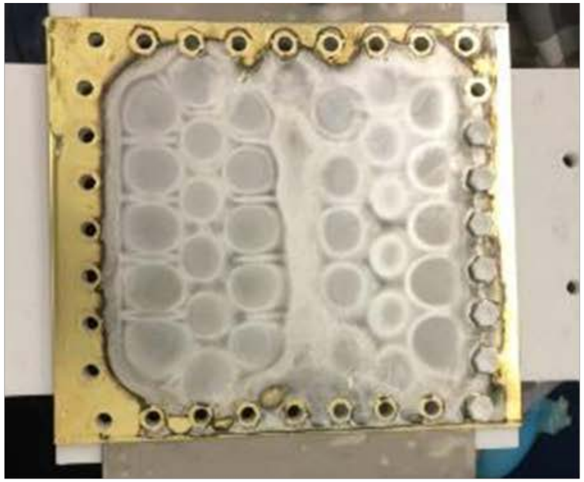
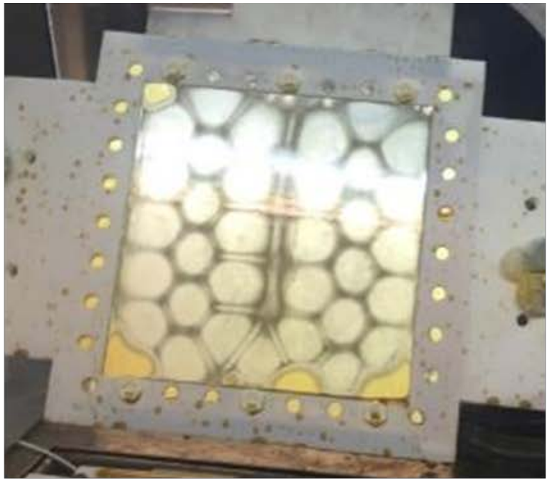
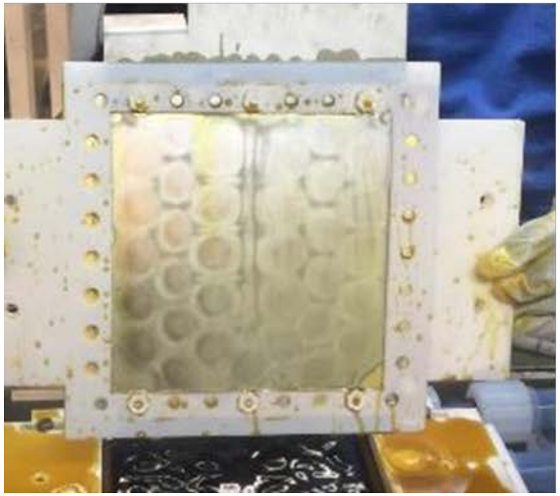
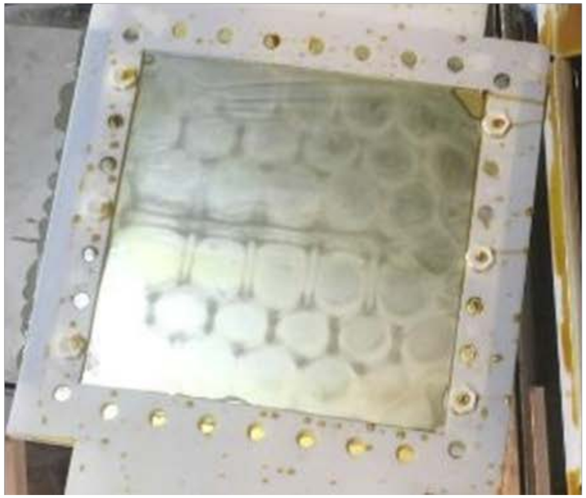
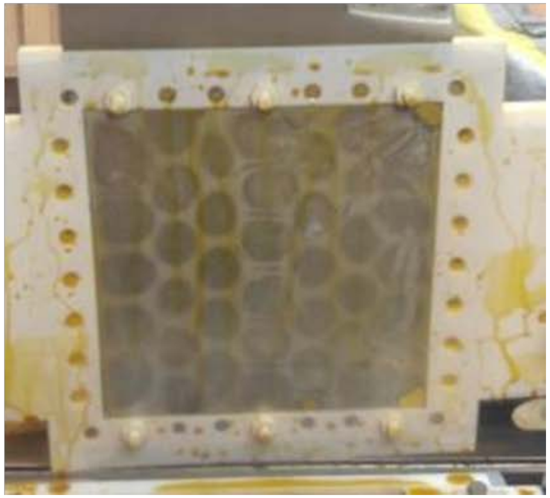
Pilot Line Commissioning





Technical Accomplishments and Progress

Pilot Line Commissioning: Optimize Plating

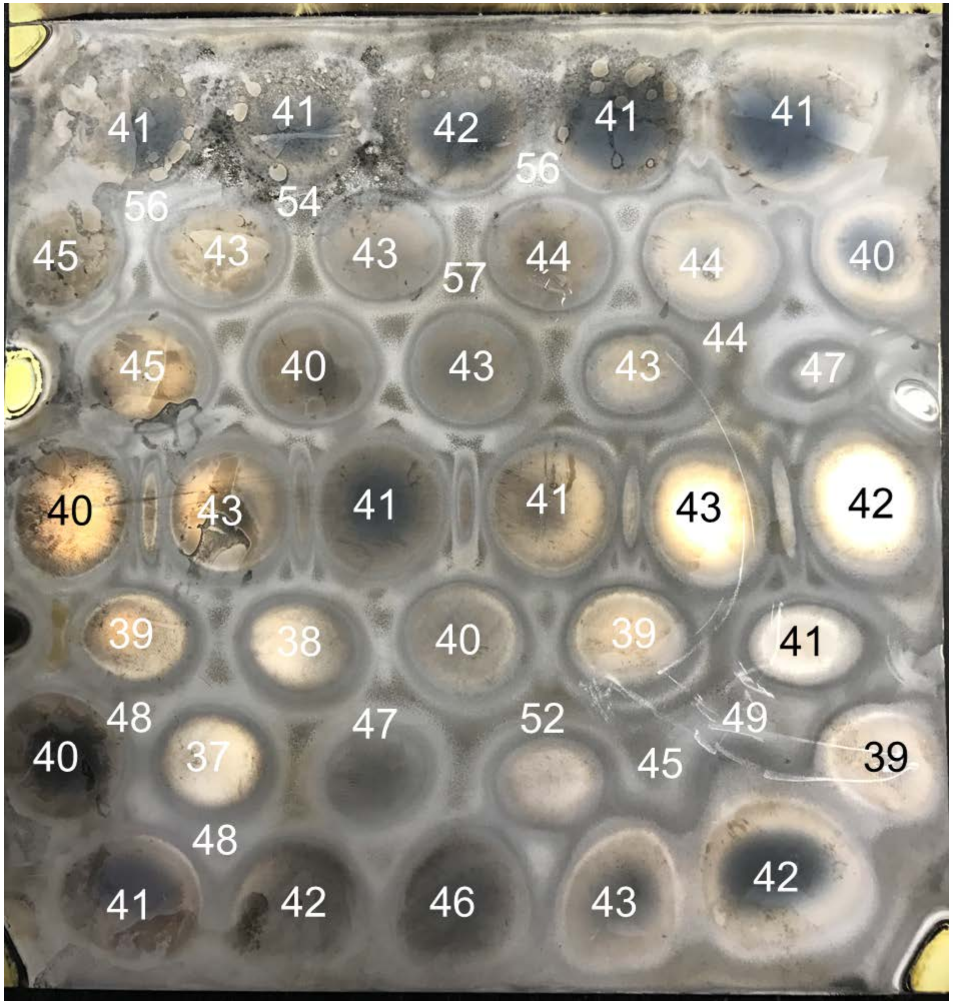







Technical Accomplishments and Progress

Pilot Line Commissioning: Optimize Plating

Characterization of initial plated sheets:

- Brass substrate
- 50um coating, single sided
- Variable roughness
- Verify composition & hardness
- Residual ionic liquid staining
  - Boost rinsing



6x6 inch sheet showing thicknesses in um.

Technical Accomplishments and Progress

Pilot Line Commissioning: Optimize Plating

- Completed plating 6”x6” Aluminum sheet
  - 50-60um per side
    - 1x plating rate
  - Plating bath no longer has co-solvent

- Flow
  - With current nozzle set up, height, flow rate, weir adjustments have seem to have only minor effects on appearance & composition
  - Potential next step:
    - New baseplate with different flow nozzles
    - Boost flow rate to boost mass transport at the cathode

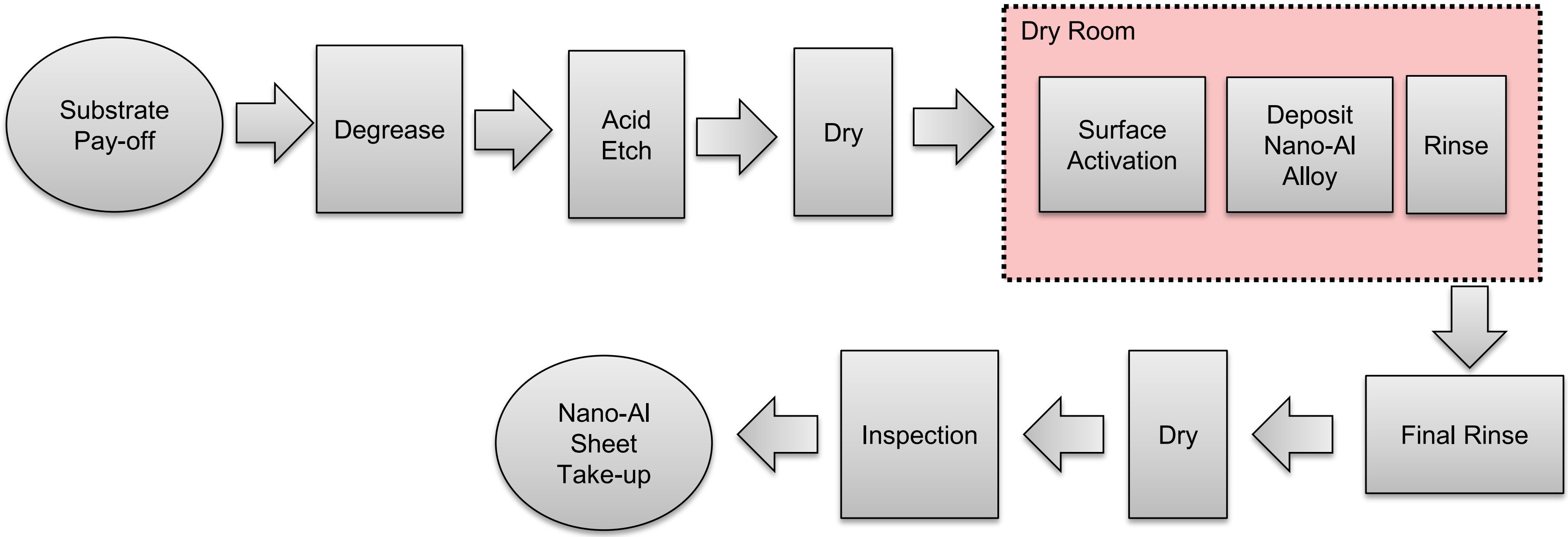
- Equipment
  - Teflon Inlay, and top lid to flow cell are bowing inward
    - Improve strength of design
  - Current cooling coil is insufficient to prevent temperature increase during plating

Technical Accomplishments and Progress

Cost Model

- IBIS Assoc. aided with cost model development
- Focus on sheet thickening step
  - 200μm substrate; 800μm nano-Al
- Continuous, high-volume production

Cost model considers materials, direct labor, equipment, tooling, building, maintenance, electricity, supervisory labor

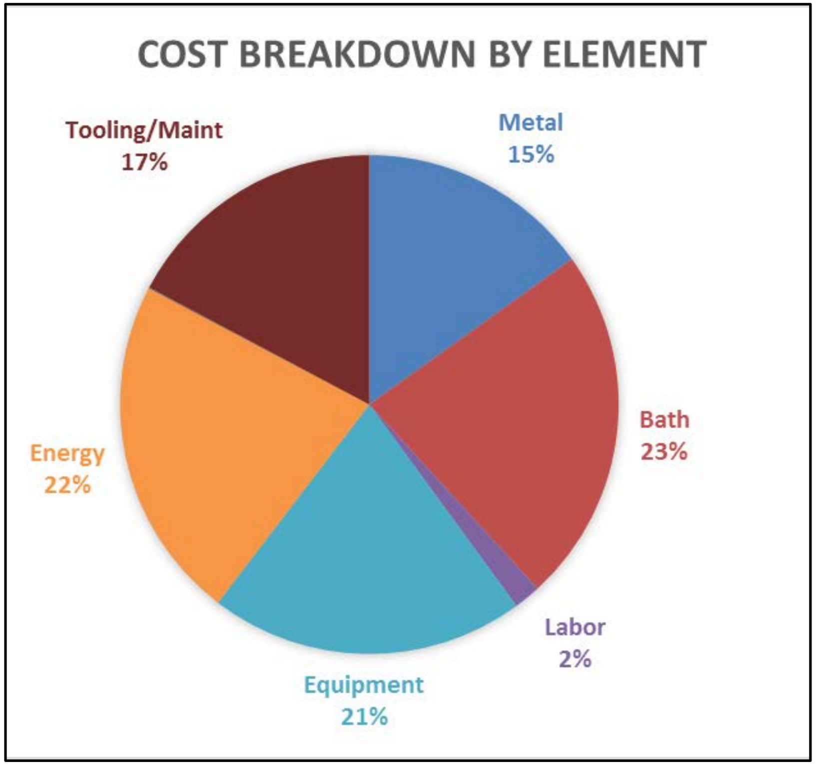


Technical Accomplishments and Progress

Cost Model

Sheet dimensions	1mm x 2m x 250m
Coil weight	1,525 kg
Tank length	19 meters
Annual production	10,000,000 m^2
Annual production	29,000,000 kg
Production rate	4,000 kg/hr
Plating lines	66
Building footprint	23,000 sf *
Sheet cost	$6.52/lb

* Note: football field = 52,800 sf



Sheet cost of $6.52/lb = target of $2/lb saved

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Response to Previous Year Reviewers' Comments

- Clarify the role of sandwich structures in meeting the overall objective, including cost.
  - Our approach is to use a 200um core of AA6061 plated with 400um of nano-Al per side for a total thickness of 1mm. Our cost model indicates that we can achieve the price/performance.
- Are there any corrosion concerns with these alloys?
  - There are always concerns. However, nano-Al alloys have excellent corrosion performance due to their structure (single phase alloys).
- Reviewer states, "the real application is nano-Al on Mg." Can this be achieved?
  - We have begun work on applying nano-Al to Mg sheet. We are a subawardee on another DOE program which started in October 2016.

Partners/ Collaborators

- Xtalic – Project Prime
  - Develop nanostructured alloys with unique strength/weight
  - Build and optimize nano-Al process capability
  - Electroform nano-Al sheets
- Fiat Chrysler Automobiles US – Project Subcontractor
  - Consult on simulation for forming of sheet
  - Corrosion testing
- Tri-Arrows Aluminum – Project Subcontractor
  - Evaluate continuous electroforming as potential sheet manufacturing process

Challenges and Barriers

- Validation of manufacturing process on industrial scale
  - Nano-Al bath chemistry and process are unique
  - Scale up to larger format with acceptable tolerances
- Materials need to meet full specifications
  - Demonstrated target strength requirements of >600 Mpa, 8% elongation
  - Need to meet the broader spec for a given application
- Achieving target cost of $2/lb of weight saved
  - Layered structure improves cost without sacrificing performance
  - Cost model has been built and will be used to identify best opportunities to reduce manufacturing costs

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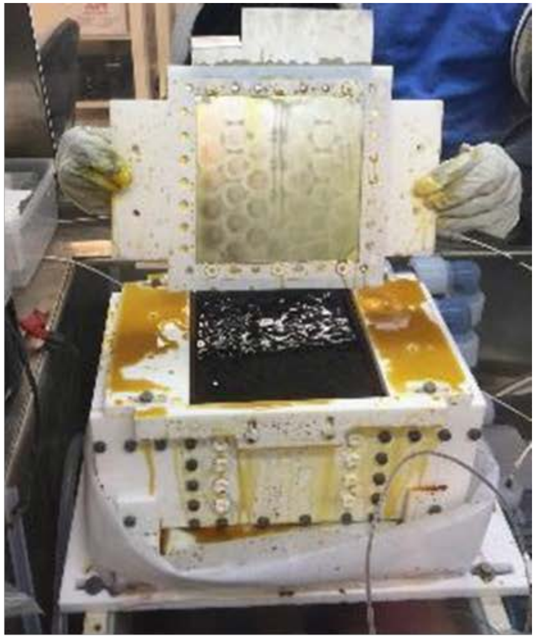
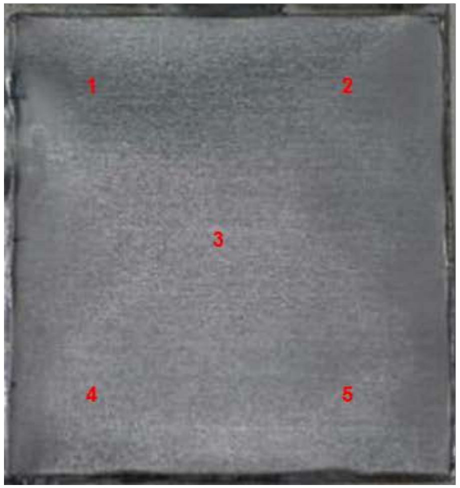
Future Research

- Efforts for Budget Period 4 and completion of project:
  - Fabricate Preferred alloy(s), test against full specs
  - Lab demonstration of economic viability
- This project is scheduled to end in December 2018.
  - No efforts are currently planned or budgeted beyond this.
- Any proposed future work is subject to change based on funding levels.

Summary

- Xtalic's nano-Al alloys can be plated thick and strong
- Nano-Al sheet electroforming system is commissioned
  - 6x6 inch sheets demonstrated
- System optimization required in order to achieve $2/lb saved target





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