DOE Annual Merit Review: June 7-11, 2010

1. Formability of Continuous Cast Mg Sheet

2. Friction Stir and Ultrasonic Solid State Joining of Magnesium to Steel

Presenter: Mark T. Smith, (509) 375-4478, mark.smith@pnl.gov

Pacific Northwest National Laboratory

Project ID # LM011_Smith_2010

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Formability of Continuous Cast Mg Sheet

Friction Effects & Post-Formed Property Characterization

Principal Investigator: Aashish Rohatgi, (509) 372-6047, aashish.rohatgi@pnl.gov

Presenter: Mark T. Smith, (509) 375-4478, <u>mark.smith@pnl.gov</u> Pacific Northwest National Laboratory

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Overview

Timeline

- Start: Mid-year 2008
- Finish: Mid-year 2010
- 90% complete

Budget

- Total project funding
 DOE \$600K
- FY08 Funding \$180K
- ► FY09 Funding \$350K
- FY10 Funding \$70K

Barriers

- High Cost of Mg Sheet (AZ31B)
 - Continuous-cast vs. Wrought sheet
- Processing and Performance Issues
 - Formability and post-formed properties
 - Non-graphitic/boron nitride lubricant

Partners

- USAMP team AMD602
 - GM (Paul Krajewski)
 - Ford (Peter Friedman)
 - Chrysler (Jugraj Singh)
 - Troy Tooling Tech. (Dennis Cedar)
- Commercial Mg sheet vendors
- Canada Center for Mineral & Energy Technology (CANMET) (Kevin Boyle)
- University of Virginia (Sean Agnew)

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Objectives

Reduce processing costs for automotive sheet products by replacing conventional wrought processed Mg sheet with continuous-cast sheet.

Evaluate formability of continuous-cast Mg sheet

- Identify non-graphitic/boron nitride lubricant for Mg forming at T ~350°C
- Determine relation between Mg sheet surface roughness & formability
- Determine bi-axial forming limits via limited dome height (LDH) tests

Evaluate post-formed properties of continuous-cast Mg sheet

- Identify forming conditions for maximum post-formed tensile strength
- Determine correlation: microstructure ↔ formability ↔ postformed performance

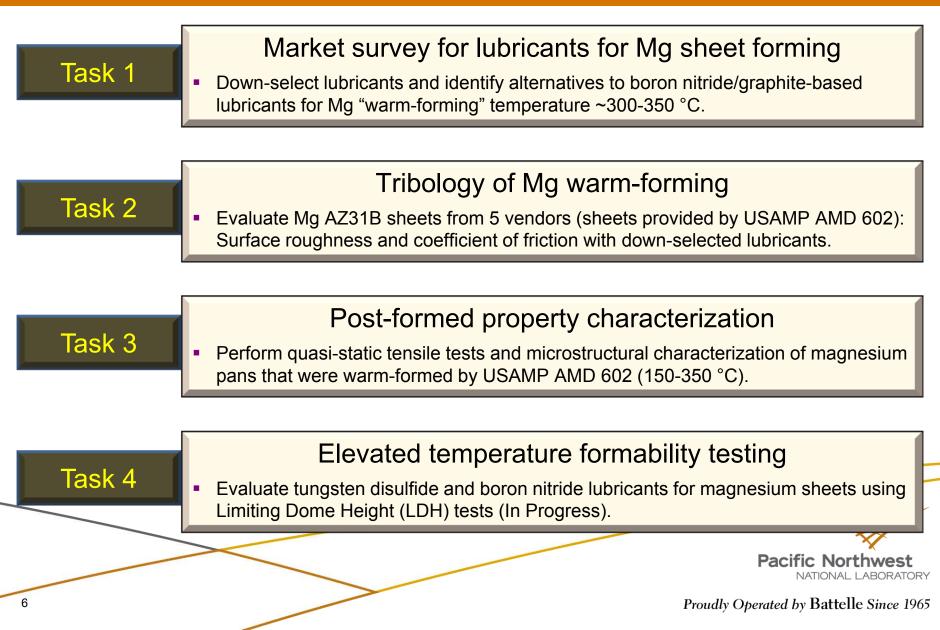
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Milestones

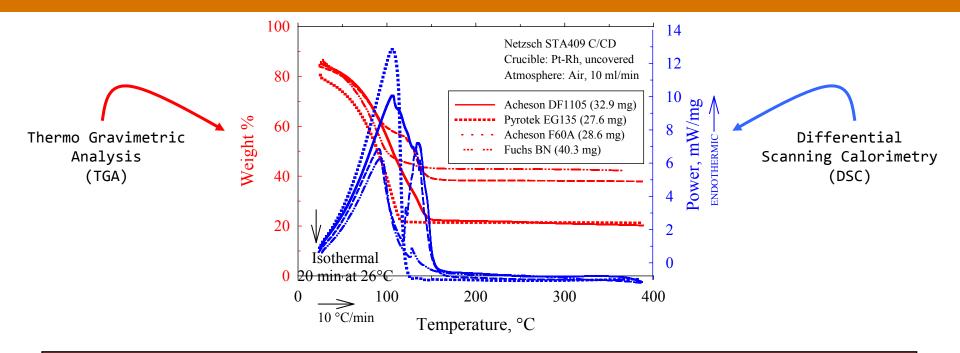
Month/Year	Milestone or Go/No-Go Decision
March 2009	Complete an upgrade of the PNNL bi-axial forming test apparatus to include the capability to test at temperatures upto 350 °C.
Sept. 2009	Complete 1 st round evaluation and down-selection of tooling lubricant type.
Dec. 2009	Of the five commercial vendors whose continuous-cast AZ31B sheets were warm-formed into test pans, determine the post- formed mechanical properties of sheet material that demonstrated successful formability (i.e. crack-free and wrinkle-free test pans) over the widest temperature and pressure range.
March 2010	Of the five commercial vendors whose continuous-cast AZ31B sheets were warm-formed into test pans, determine the post- formed microstructural texture of sheet material that demonstrated successful formability (i.e. crack-free and wrinkle-free test pans) over the widest temperature and pressure range.

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Technical Approach



Technical Accomplishments Thermal Analysis of Lubricants

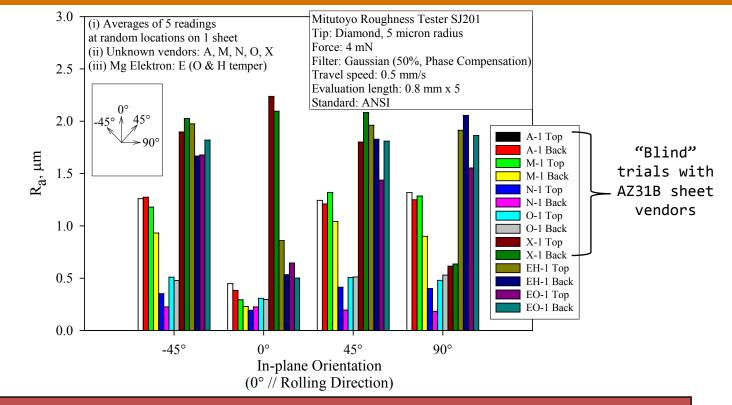


- 1. Thermal stability of boron nitride (used by USAMP-AMD602) and 4 down-selected lubricants was determined.
- 2. Lubrication during Mg warm-forming (175-350°C) likely occurs through *solid residue* due to thermal decomposition/evaporation of the lubricant/carrier upon heating to test temperature.
- 3. Non-isothermal warm-forming may lead to mixed lubrication (solid + liquid) depending upon temperature distribution and rate of thermal decomposition /evaporation of the lubricant.

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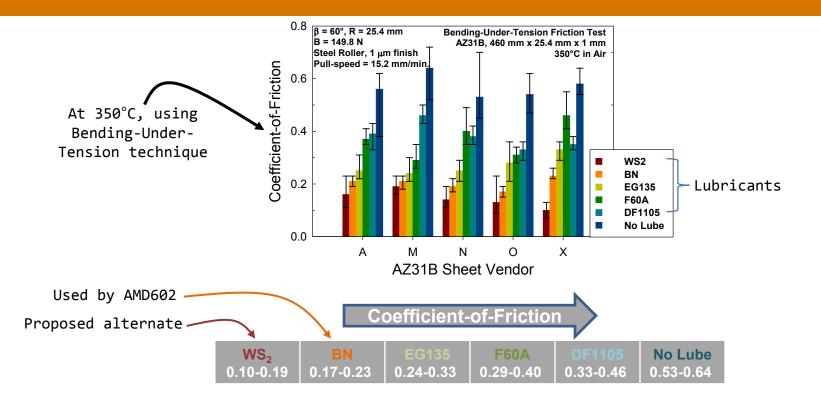
Technical Accomplishments Surface Roughness of Mg AZ31B Sheets



- 1. Wide vendor-to-vendor variation in Mg sheet surface roughness ($R_a 0.2-2 \mu m \& R_y \sim 2-15 \mu m$).
- 2. Mg sheet roughness was related to its warm-formability:
 - "High" roughness ~ "Good" formability (vendors A, X).
 - "Low" roughness ~ "Poor" formability (vendors O, N).
 - Exception: Vendor M → additional factors e.g. microstructure need to be considered too.

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Technical Accomplishments Coefficient-of-Friction of Mg + Lubricants



- 1. COF for Mg-lubricant combinations was provided to OEMs for their mathematical models to simulate Mg warm-forming.
- 2. Tungsten disulfide (WS₂) was identified as a potential alternative (to BN) for Mg warm-forming.
- 3. Thick layer of residue masked the effect of sheet roughness on COF.

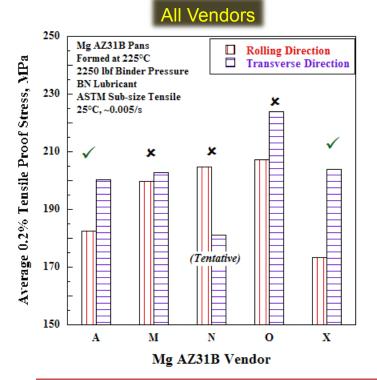
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Technical Accomplishments *Post-Formed* Mechanical Characterization

Vendor A 250 Average 0.2% Tensile Proof Stress, MPa Mg AZ31B Pans Vendor A 190°0° Depth 150 mm 3000 lbf Binder Pressure 70 mm **BN Lubricant ASTM Sub-size Tensile** 225 25°C, ~0.005/s Transverse Direction (90°) 200 175 Rolling Direction (0°) 150 225 275 125 175 325 375 Pan-Forming Temperature, °C

Forming at 175°C-200°C → Maximum strength (suitable for oil lubricants!)

- Forming at 150°C-350°C → ~10% spread in strength
- 3. True strain to failure ~20%

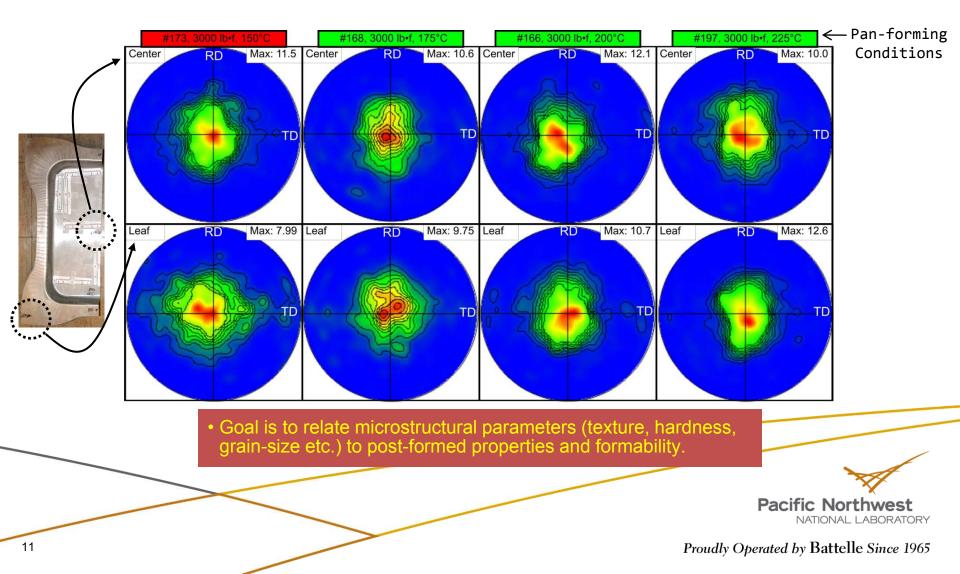


 Best formability ≠ Maximum strength (Vendor A) (Vendor O)
 ~10-15% improvement in strength in "A" might be possible.



Technical Accomplishments *Post-Formed* Microstructural Characterization (90% complete)

Vendor A Mg Pan {0001} Pole Figures via Electron Backscatter Diffraction (EBSD)



Accomplishments and Impact

1. Identified effect of Mg sheet surface roughness on warmforming process window.

Guidance to Mg sheet vendors for desired sheet characteristics

- Identified tungsten disulfide as a potential alternative lubricant to currently used graphite/ boron nitride based lubricants.
- Identified warm-forming conditions for best post-formed tensile strength.
- 4. Identify microstructural effects on Mg warm-formability & post-formed strength.



Cost-reduction and process simplification of Mg warm-forming and down-stream processes

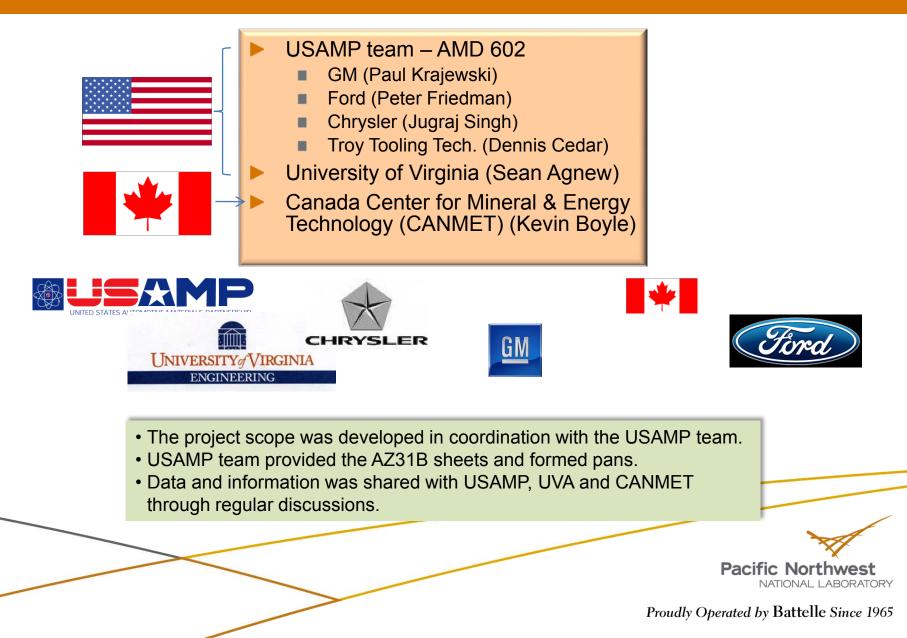
Cost-reduction by avoiding hightemperature (>300 °C) processing and associated lubricant issues



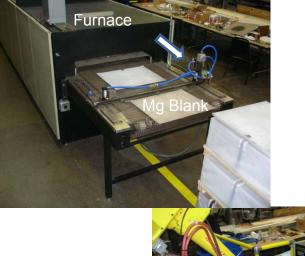
Optimum combination of formability and post-formed mechanical strength

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Collaboration & Coordination with Other Institutions



Current Status Warm-forming Integrated Line (Courtesy AMD 602)



Robotic Arm







Proposed Future Work

Remainder FY2010

- Relate microstructure, post-formed strength and formability
 - EBSD and metallography
- Forming limits under bi-axial forming
 - Limited Dome Height testing

- Communicate findings to the OEMs
- Journal/conference publications

Path Forward

- Non-isothermal warm-forming of Al and Mg
 - Tribology under non-isothermal conditions
 - Model thermal and strain distribution in sheet
 - Enhance durability of forming-die for high-volume manufacturing

Enable high-volume manufacturing of AI and Mg sheet parts for OEMs

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Summary

- 1. Owing to its lower cost, continuous-cast Mg sheet can help overcome the cost-barrier of the conventional wrought Mg sheet and enable US automakers to achieve automotive weight reduction targets.
- 2. Correlation between Mg sheet surface roughness, coefficient-of-friction (with lubricants) and its warm-formability, developed in this project, could help establish Mg sheet property guidelines for commercial vendors.
- 3. Tungsten disulfide was identified as a potential alternate lubricant to currently used graphite/boron nitride-based lubricants.
- Maximum post-formed strength (for vendor A) was obtained by forming at ~175-200 °C. Forming at these (i.e. < 300 °C) temperatures can be done with conventional oil lubricants, eliminating the cost and issues associated with high-temperature lubricants.
- 5. Microstructural analysis (in progress) is expected to identify optimum parameters (initial microstructure and forming temperature) for simultaneous warm-formability and high post-formed tensile strength.

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SUPPLEMENTAL SLIDES

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Lubricant Down-Selection Table

Product Name	Company	Chemistry	Form	Lubricant Removal	Usable Temperature	Cost		
Deltaforge 1105	Acheson	Carbovylatoo	Water based	Water	370 C			
Deltaforge F-60A	Colloids	Carboxylates	water based	vvalei	370 C	~3.5 \$/lb		
Pyroslip 135	Pyrotek	Graphite + Polymer	Water soluble synthetic polymer	Water	>660 C	~\$8/lb		
Tungsten Disulfide	Lower Friction	WS ₂	0.5 μm dry powder	Water	~538 C	~45 \$/lb		
Forge Ease 06 ALWF	Fuchs	BN	Liquid suspension	Water	>1000 °C	Provided by AMD 602		

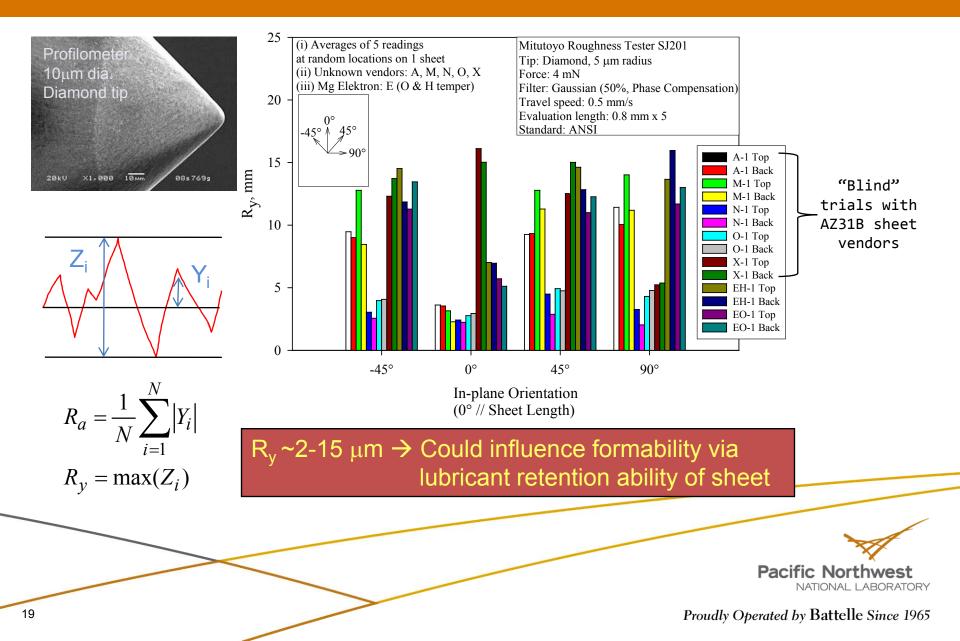
1. Lubricant selection criteria

- a. Thermally stable at 350 °C
- b. Easy removal
- c. Low cost

2. Lubricants evaluated via friction tests (350 °C)

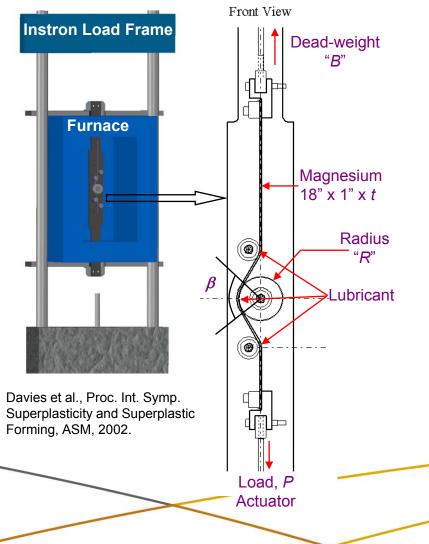
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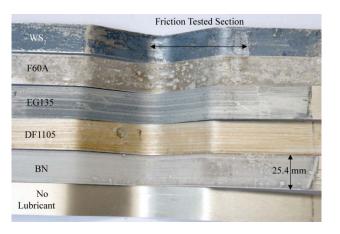
AZ31B Sheet Roughness R_v: Maximum Two-Point Height of Profile



Coefficient-of-Friction (COF) Experiments

Modified Bending-under-Tension

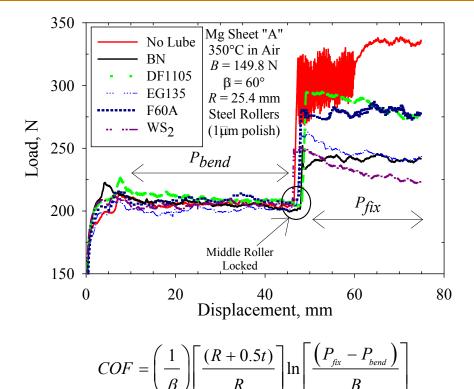






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Supplemental Slide
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Coefficient-of-Friction (COF) Determination

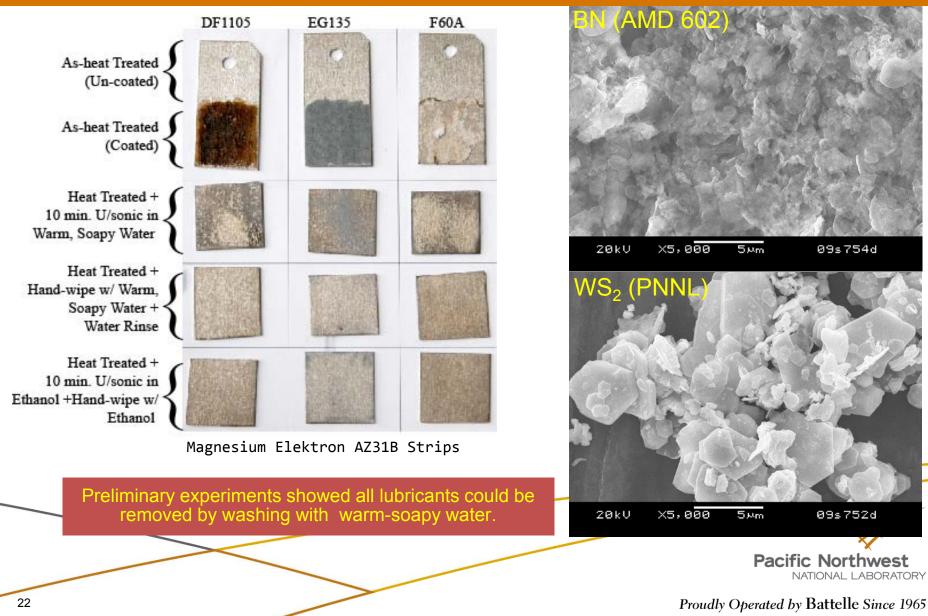


- Bending-Under-Tension technique
- 5 AZ31B Vendor sheets
- 5 lubricants
- 350°C in air

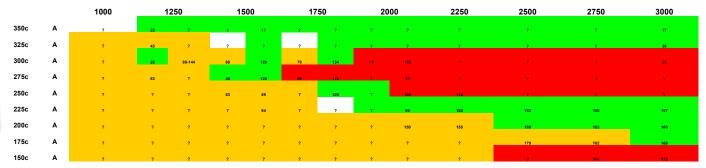
COF of Mg sheet/lubricant combinations under simulated elevated temperature forming conditions was determined.



Lubricant Removal from AZ31B Sheets



AZ31B Forming Trial Results Courtesy AMD 602 Team

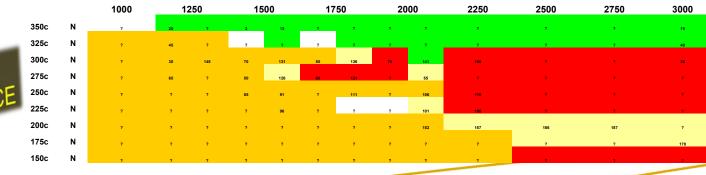


A Summary Matrix



- W/C Wrinkles and Cracks W Wrinkles G Good Cracks
- Green = Good • Red = Bad





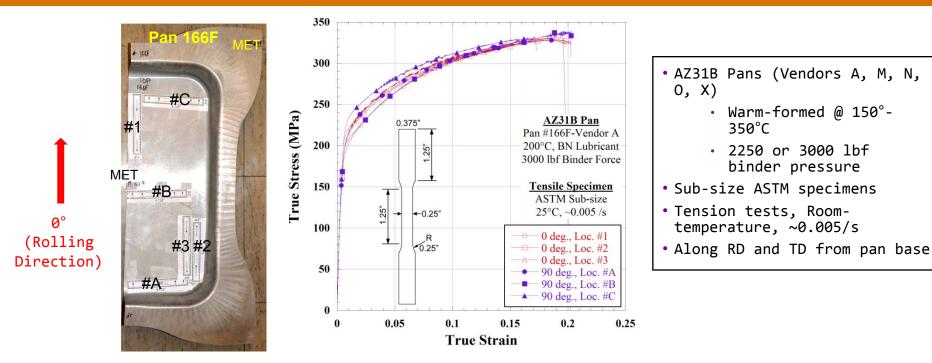
N Summary Matrix

Wrinkles and Cracks Wrinkles

> Good Cracks

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Post-formed Mechanical Testing

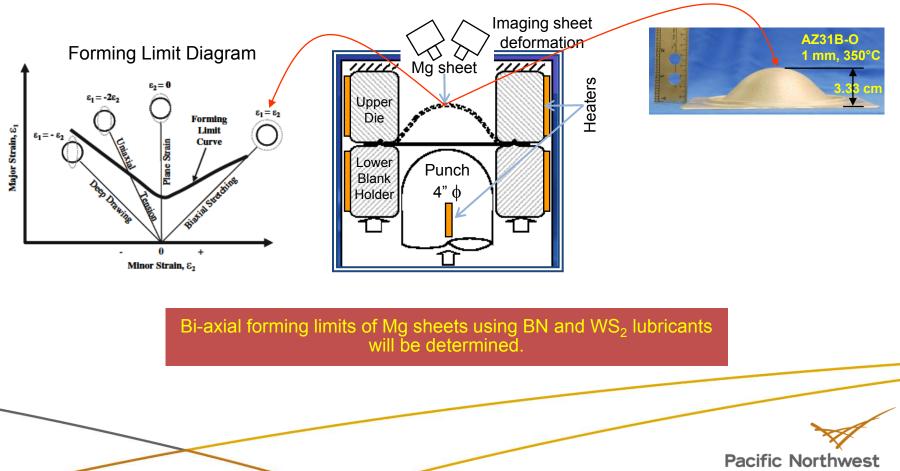


Attention was focused on the best forming material (vendor A).



Limiting Dome Height (LDH) Testing (In Progress)

PNNL Heated LDH Setup with Strain-Imaging Capability



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Questions?

Contact:

- Aashish Rohatgi (509) 372-6047
 <u>aashish.rohatgi@pnl.gov</u>
- Mark Smith (509) 375-4478 mark.smith@pnl.gov



Project No: 55220, Agreement No: 17242

Friction Stir and Ultra Sonic Solid State Joining of Magnesium to Steel

Yuri Hovanski, (509) 375-3940, yuri.hovanski@pnl.gov

Glenn Grant and Saumyadeep Jana Pacific Northwest National Laboratory Michael Santella Oak Ridge National Laboratory

Presenter: Mark T. Smith, (509) 375-4478, mark.smith@pnl.gov

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Project ID: PNL011

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Project Overview

Project Timeline

- Start: Mid-year 2008
- Finish: 2011
- ▶ 60% complete

Budget

- Total project funding
 - DOE \$1.5M
 - 50/50 Split with ORNL/PNNL
- FY08 Funding \$200K
- FY09 Funding \$500K
- FY10 Funding \$500K
- FY11 Funding \$300K

Technology Gaps/Barriers

- Few options exist for joining Mg components to steel structures
- Solid state technologies have yet to be investigated, but have potential for providing low-cost high strength direct bonding between Mg and Steel

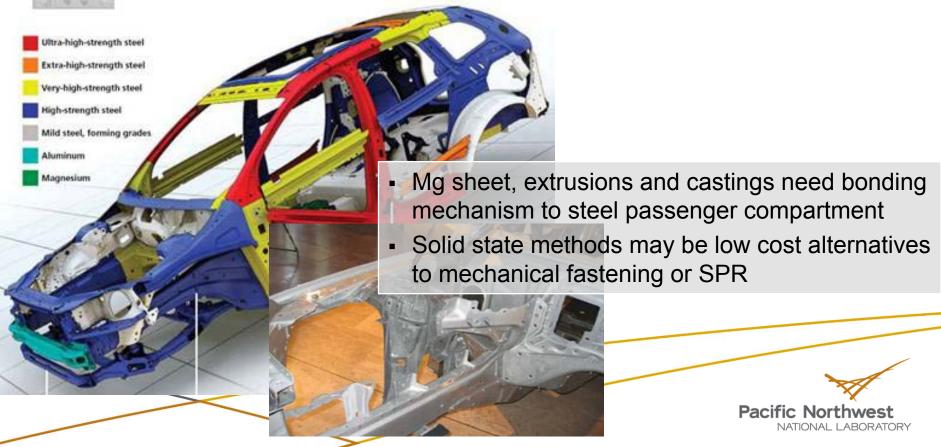
Partners

- USCAR Joining team
 - GM (J. Quinn & B. Carlson)
 - Ford (E. Hetrick)
 - Chrysler (J. Becham)
- Commercial Mg/Steel sheet Producers
- Mg-Front End R & D team (MFERD)
- Oak Ridge National Laboratory

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Relevance: Project Motivation

Development of solid-state technologies for bonding Mg to steel may enable broader application of Mg alloys in automotive structures requiring integration with steel components



Project Milestones

DOE	Month/Year	Milestone or Go/No-Go Decision
<u>Significance</u> Feasibility	Jan. 2009 Initial Decision Gate	Demonstrate Solid State Joining of Mg to Steel Achieve a minimum tensile strength criteria of 40% joint efficiency or 1.0 kN x sheet thickness for spot configurations.
Structural Significance	Sept.2009 Midterm Decision Gate	Demonstrate Baseline Structural Joining Achieve a minimum tensile strength criteria of 60% joint efficiency or 1.5 kN x sheet thickness for spot configurations
Full Structural Capability	Sept. 2010 Final Decision Gate	Complete Comprehensive Structural Development Achieve a minimum tensile strength criteria of 75% joint efficiency of 2.0 kN x sheet thickness for spot configurations
Demonstrated Technology	July 2011 Final Milestone	Demonstrate Corrosion Protection Strategies for Solid State Mg/Steel Joints
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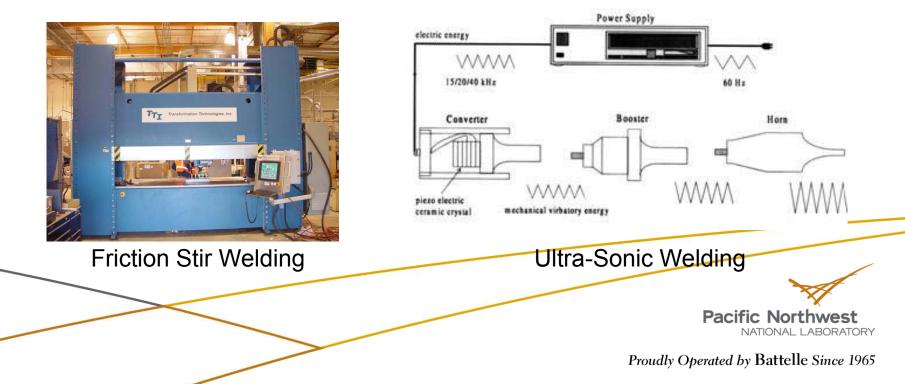
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31

Approach: Solid State Development

Task 1 – Process development

- Systematically evaluate the application of friction stir welding and ultrasonic welding of Mg to Steel (parameter scoping)
 - Develop an improved understanding of the interaction of process parameters with appropriate alloy/product form combinations
 - Develop appropriate tooling appropriate to materials
 - Access process applicability per Initial Gate (completed)



Approach: Solid State Development

- Task 2 Characterization / Structural Strength
 - Evaluate interface of joint produced during Task 1
 - Investigate metallurgical compatibilities
 - Correlate process parameters to mechanical properties
 - Refine parameters and tooling to achieve structural baseline
 - Access results per midterm decision gate for go-no go
 - Completed Sept. 2009 with greater than 60% joint strength achieved
 - Develop process parameters for structural (>75%) joint strengths
- Task 3 Corrosion / Interlayers
 - Access joint corrosion based on MFERD specifications
 - Tests will be ran based on similar criteria for direct comparison
 - Investigate the effect of coatings on corrosion and joint strength
 - E-coats & paints on Mg alloys, Zn variations on steels
 - Introduce interlayers and other isolation strategies

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Current Progress and Scheduled Work

✓ Completed Tasks 1 and 2

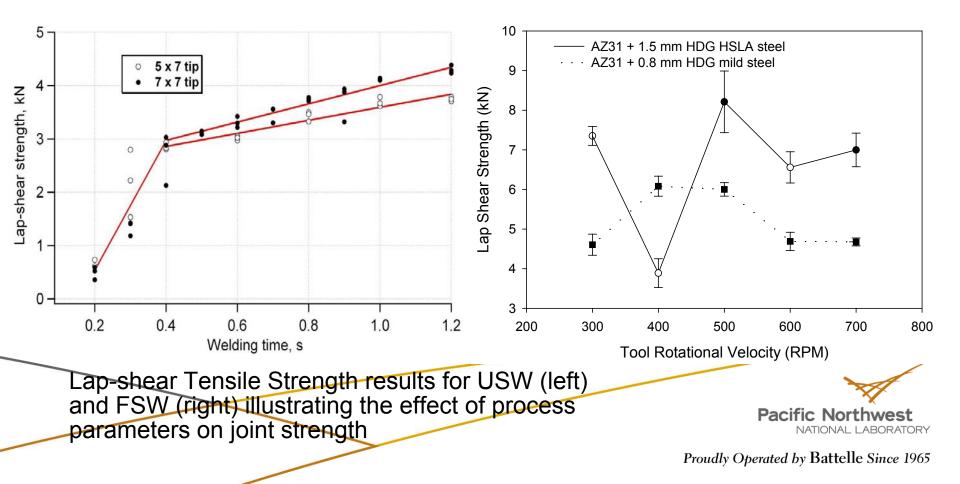
✓ Passed Initial & Secondary Decision Gates (40% & 60% joint strength)

Completed work			Fiscal Year 2008		Fiscal Year 2009			Fiscal Year 2010				Fiscal Year 2011						
Completed Gate		Quarter	Q 1	Q 2	Q 3	Q 4	Q 1	Q 2	Q 3	Q 4	Q 1	Q 2	Q 3	Q 4	Q 1	Q 2	Q 3	Q 4
Future Decision Gate	~	Task IA: Develop FSW																
		1.1 Material Selection																
Future Work		1.2 FSW relationships																
		Decision Gate																
		Task IB: Develop USW																
		1.1 Material Selection																
Completed Work		1.2 USW relationships																
Completed Work –	ĺ	Decision Gate																
		Task 2: Bond Characterization																
		2.1Material Selections																
		2.2 FSW/FSSW Structural																
		2.3 Ultrasonic Structural																
		Decision Gate																
Near Term Gate	7	2.4 FSW Advanced Development																
		2.5 USW Advanced Development																
		2.6 Joint Characterization																
		Decision Gate																/
	خم	Task 3: Corresion													•			
Corrosion and		3.1 Material Selection														X	<	
		3.2 Joint Corrosion							1								est	
Interlayers		3.3 Interlayer Corrosion							1								RAIO	, γ
	L	3.4 Characterization									Prou	dly C						65
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Technical Accomplishments:

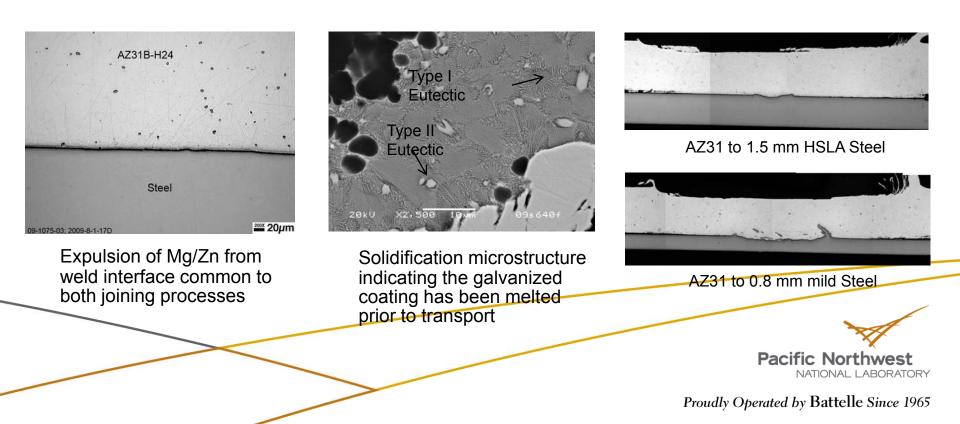
Demonstrated Baseline Structural Joining by achieving a tensile strengths > 60% joint efficiency (FSW) and > 1.5 kN x sheet thickness for spot configurations (USW)

Represents a 20% increase in joint strength from last year



Technical Accomplishments:

- Characterized the joint interface via optical and electron microscopy, EDX/EDS, microhardness, etc.
 - Evaluated the metallurgical mechanisms affecting joining
 - Characterization on Mg/Steel joints not previously understood
 - Understanding the role of Zn in the both processes



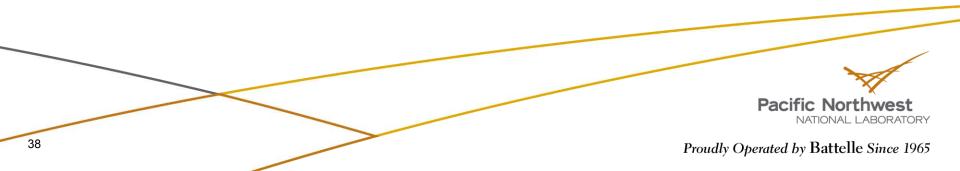
Summary and Upcoming Work

- High strength solid state welds are possible using both ultrasonic and friction stir welding processes
 - Intermediate strength gate was passed with both processes
- Joint interface characterization
 - Strength increased with weld size for both processes
 - Zn coating appeared critical initially, yet Zn is transported from weld interface with both processes
 - Effects of coatings, adhesives, and paint on materials
- Future efforts will continue to address issues related to the fundamentals and manufacturing of each process
 - Zn coating effects on joint strength and corrosion protection
 - Clamping forces, weld offsets and sheet stack-ups
 - Sheet thickness and material compatibility

Structural joints >75% joint strength

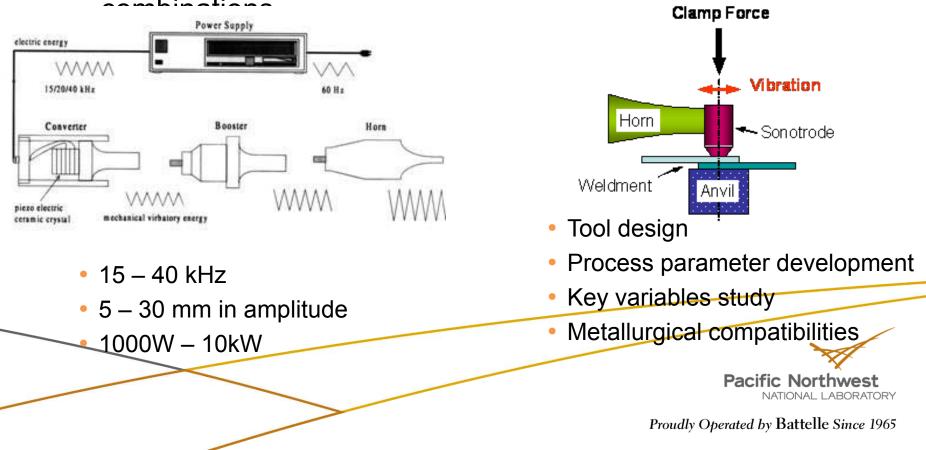
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Supplemental Slides



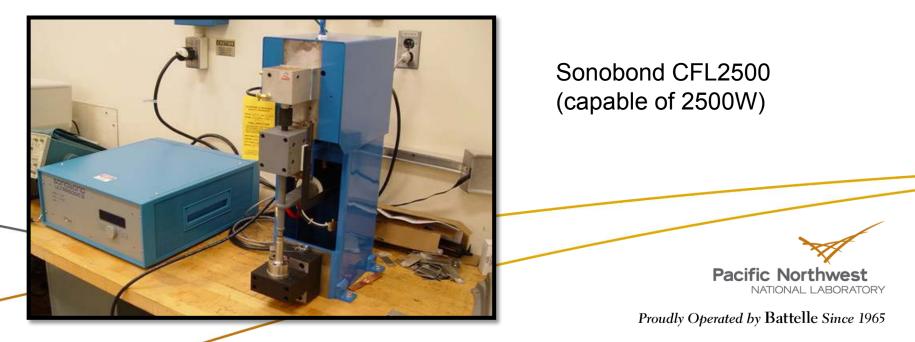
Ultrasonic welding

- Task 1 Process development
 - Systematically evaluate the application of ultrasonic welding of Mg to Steel
 - Develop an improved understanding of the interaction of ultrasonic energy source with appropriate alloy/product form



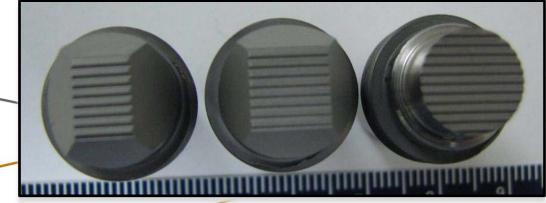
Activities Updates

- Welds were made using:
 - 1500 W, 0.2-1.2 s, 90 psi line pressure
- Mg was AZ31B-H24 (1.5 mm thick), buffed to remove oxide
- Steel was 0.8-mm-thick mild steel HDG
 - Acquired from auto assembly plant
 - Solvent cleaned to remove lubricants and other extraneous matter



USW Sonotrode tips

- 5-mm x 7-mm is cited in published technical literature
- 7-mm x 7-mm has diagonal of 9.8 (within flange width limits)
 - Larger weld area should increase joint strength
- 10-mm-Ø (within flange width limits)
 - Further increase in weld area for same perimeter limit
- USW machine limits maximum applied pressure
 - Welding at constant nominal pressure was possible for 5 x 7 and 7 x 7
 - Pressure limit prevented welding with 10-mm-Ø
 - 5x10-mm-Ø7wars re-machined to 8-mm-Ø



Tool material is hardened T1 tool steel

USW spot welds are in range of RSWs of AZ31

Table 2—Summary of Welding Conditions for Sound Spots in 0.064-In. AZ31A-0 Sheet with the Three-Phase Low Frequency Converter Machine at 8⁴/₇ Cycles

Electrodes $1^{1}/_{4}$ in. diameter, 4-in. R dome tips, wire-brushed sheet

Electrode force, lb	Range of weld strength, lb	Range of spot diameters, in.	Range of welding currents, amp	% Penetration	Limiting conditions
1750	581	0.223	35,200	30 - 45	Cracks and expulsion
2000	719-823	0.253 - 0.289	37,900-39,900	30-55	Cracks and expulsion
2340	833-941	0.283-0.336	43,600-48,500	35 - 60	Cracks and expulsion
2550	896 - 1075	0.305 - 0.379	48,500-56,700	35 - 65	Cracks and expulsion
2650	878-1078	0.313-0.373	48,500-58,600	30 - 65	Cracks and expulsion
2860	901-1166	0.311 - 0.420	50,800-65,200	25 - 65	Cracks and expulsion
3400	1008 - 1283	0.328 - 0.431	53,200-67,900	20 - 80	Cracks and expulsion

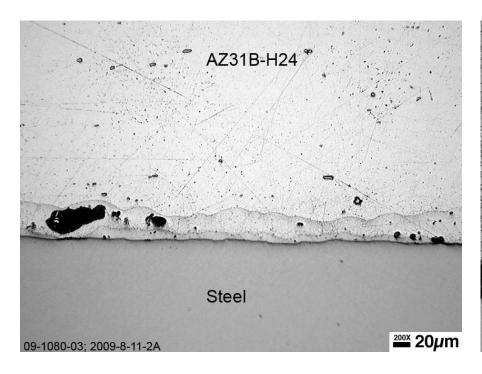
"Spot Welding of Magnesium with Three-Phase Low Frequency Equipment,

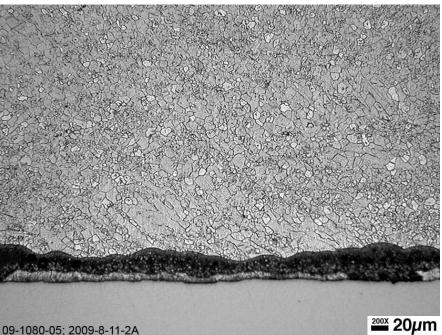
P. Klain, D. L. Knight, and J. P. Thorne, Welding Journal, v32 (1953) 7-18

- RSW lap-shear strength range: 2.6-5.7 kN (depends on spot diameter)
- AZ31A sheet was bright pickled + wire brushed (more extensive cleaning)
- Equivalent diameters for USWs:
 - $5 \times 7 \rightarrow 0.26$ -inch-dia; USW strength = 3.7 kN max.
 - $7 \times 7 \rightarrow 0.31$ -inch-dia; USW strength ≈ 4.0 kN

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Mg-Zn reaction is apparent at shorter weld times (0.4 s)



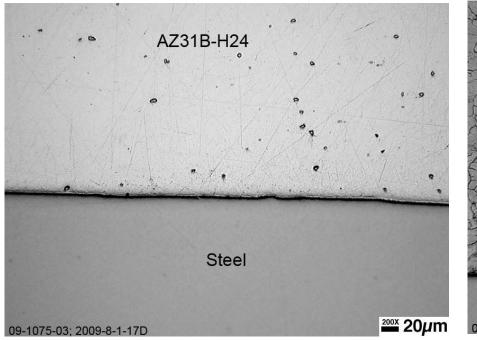


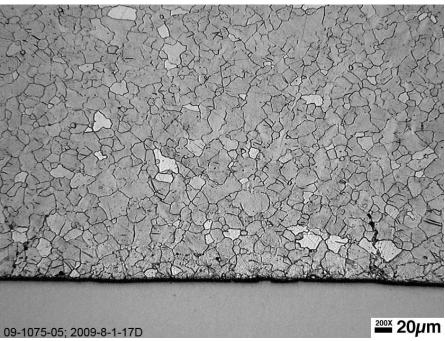
Unetched condition highlights reaction layers of Mg & Zn

Composition of interlayers suggests mass transport of Mg towards the steel Etched condition shows Mg microstructure is nearly unchanged

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Reactions are barely visible at longer weld times (1.0 s)





- Unetched condition shows reaction layers are not clearly visible at weld center
- Mg/Zn interlayer appears to be completely expelled after 1.0 s

Etched condition shows Mg has recrystallized

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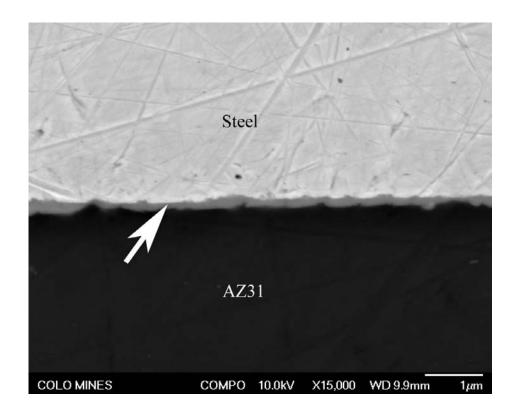
More detailed Examination of Reaction Layer

Mg and Fe are immiscible

 No reaction or mass transport leads to weak bonding

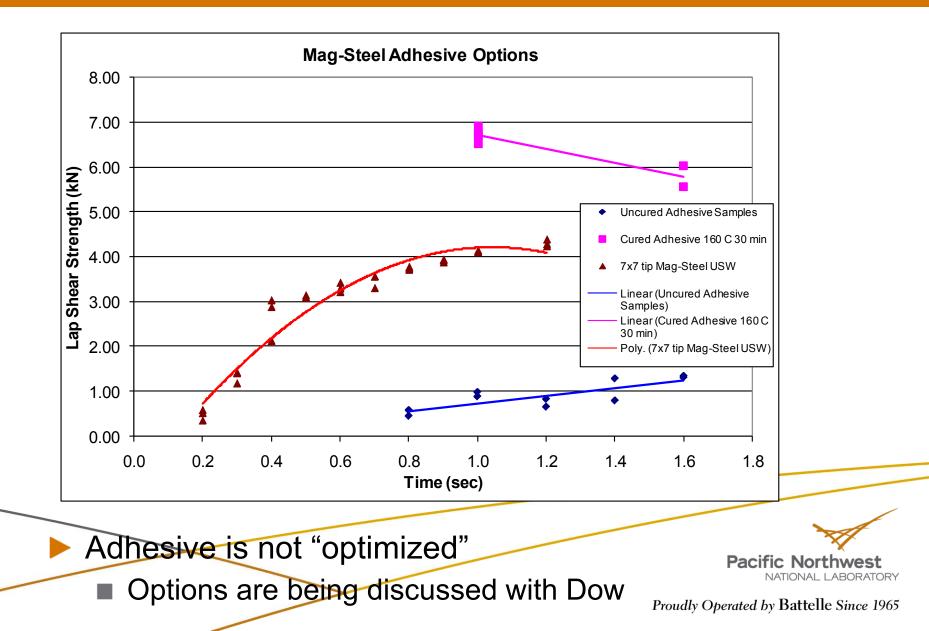
Reaction Layer

- Al-rich reaction layer could be responsible for high weld strength
- Al reacts with both Mg and Fe



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Contribution of Adhesives: Preliminary



Friction Stir Welding of Mg to Steel

Magnesium

Linear Welds in Lap Configuration

- Mg sheet on top, with zinc coated steels serving as backing
- Initial focus on characterization and parameter development

Process Parameter Development

- Feed Rate, Rotational Velocity, Plunge Force
- Tool Design, Material
- Offset

Offset

Steel

- Material Combinations
 - AZ31 to 0.8 mm HDG mild steel
 - AZ31 to 1.5 mm HDG HSLA steel



- High Strength Precision Friction Stir Welding Machine at PNNL
 - Base runout less than 0.0002"
 - Capable of 30,000 lb down force and 7,000 lb lateral loads

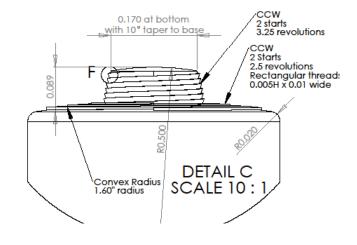
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FSW Tool Designs for Lap Welds in Mg/Steel

- Tool Material:
 - Hardened H-13 Tool Steel
- Flat Scrolled Shoulder Tool
 - Phase I development with 2.0 mm thick AZ31B
 - Poor depth range
- Convex Scrolled Tool
 - Designed for Phase II work with 2.3 – 2.5 mm Mg sheet
 - Provides variable depth
 - Increases heat input with features

206

-Convex Scrolled shoulder with a tapered and threaded pin

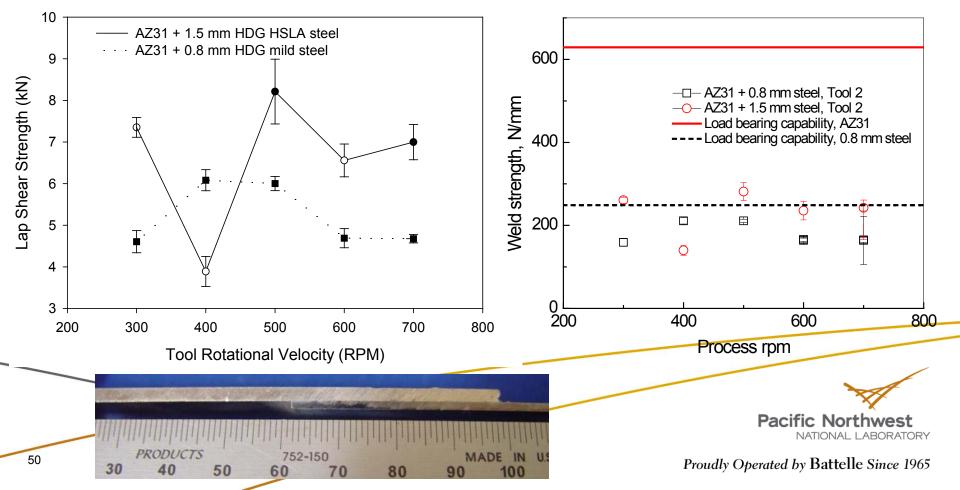


- Flat Scrolled shoulder, threaded pin with flats

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Effect of Process Parameters on Joint Strength

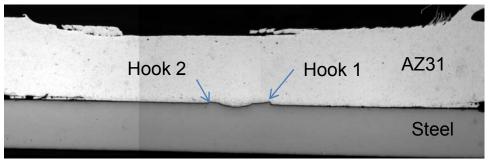
- Material specific operating windows
 - 0.8 mm steel configurations showed peak at 400 RPM
- Peak joint strengths as a function of load bearing capability



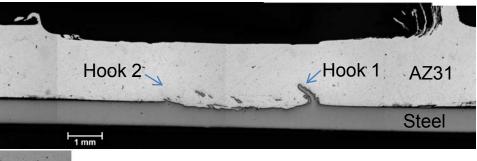
Comparison of Cross-section Macrographs

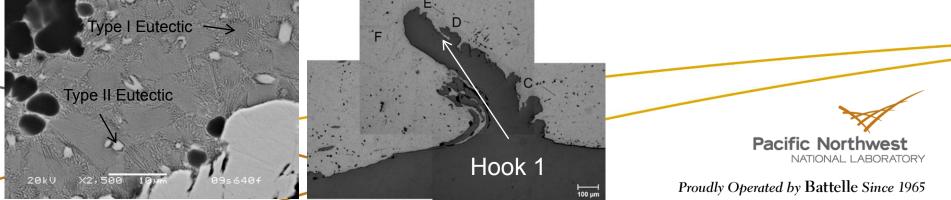
- Hooked region (deformation under tool pin) of the 0.8 mm steel sheet was
 ~5.13 mm, while on the 1.5 mm steel it was ~1.89 mm
- Joint strength was higher with 1.5 mm HSLA, yet joint efficiency was higher with the 0.8 mm mild steel welds
- Along the Mg/Steel interface, existence of a new phase can be clearly seen. This new phase results from Zn melting and subsequent reaction with solid Mg. SEM images confirmed the same. Hexagonal particles are also noticed.
- Mg/Zn alloy is transported from area directly below FSW pin, with no alloy remaining in the pin deformation zone

AZ31 to 1.5 mm HSLA Steel

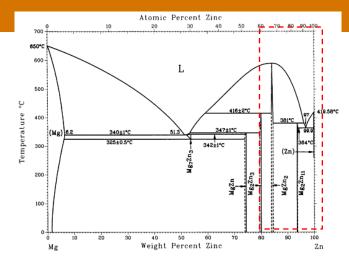


AZ31 to 0.8 mm mild Steel

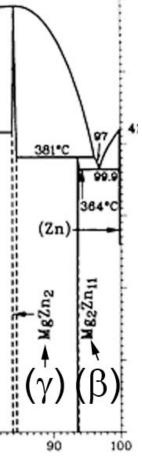


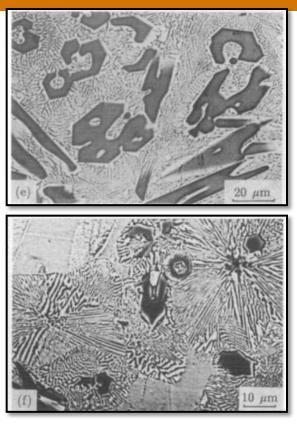


Solidification Microstructures in Zn-Mg system



Zn-Mg system is known to exhibit competitive growth between two eutectic systems, α -Zn- β -Zn₁₁Mg₂ (at 3.05 wt% Mg and 364°C) and α -Zn- γ -Zn₂Mg (at same Mg conc. & lower temp.) Eutectic reaction involving γ does not occur under eqlb. conditions. α - γ eutectic is spiral shaped, whereas α - β eutectic is lamellar. Further, primary γ is hexagonal and primary β is cube shaped. (*Liu and Jones, Acta. metall. mater.* 40, 1992, 229-239.)

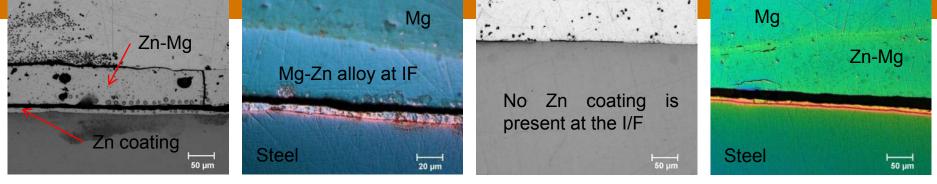


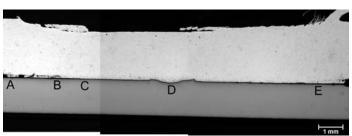


Examples of Zn-Mg solidification microstructure; (e) Zn-5wt%Mg and (f) Zn-3.4wt%Mg (*Liu and Jones, Acta metall. Mater., 40, 1992, 229-239.*)

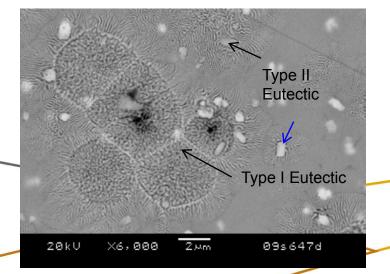
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Interface Microstructure: 1.5 mm Steel Sheet





Loc. A



Loc. B

Loc. C

Loc. E

- During the welding process, the Zn coating on the steel sheet was melted, allowing for expulsion beneath the tool and alloying with the Mg at the edges
- SEM examination revealed solidification microstructure of Mg-Zn alloy
- Two types of eutectic microstructure are noted inside the Zn-Mg alloy. Moreover, particle shapes were also found to differ. Most particles were hexagonal, only few were cube shaped (Blue Arrow).

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