ZCO-64: Inhibition Layer Formation and Galvanneal Growth on Advanced High Strength Steels

Hongjoo Rhee[1,2], Bin Li[1,2], and Paul Wang[1,2]

[1]Center for Advanced Vehicular Systems (CAVS)
[2]Department of Mechanical Engineering
Mississippi State University, Mississippi State, MS 39762

Sponsored by International Zinc Association (IZA)

October 20, 2011

Galvanized Autobody Partnership (GAP) Program Review Meeting
Point Clear, AL

MISSISSIPPI STATE UNIVERSITY

JAMES WORTH BAGLEY COLLEGE OF ENGINEERING MISSISSIPPI STATE UNIVERSITY

CAVS

Outline

- What CAVS/MSU is
- Building block portfolio
- CAVS steel research program
- ZCO-64 program update

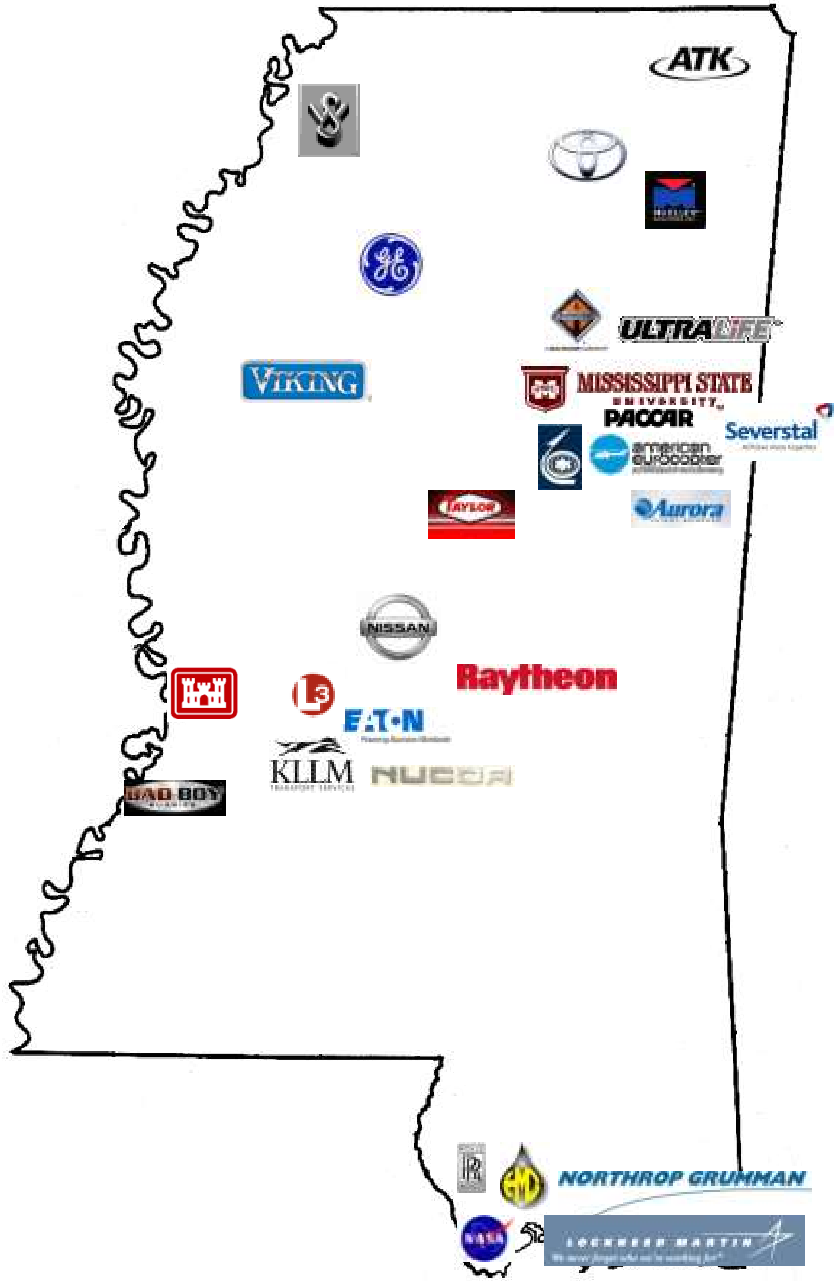
Center for Advanced Vehicular Systems (CAVS)

State Focus, Regional Asset

Vehicle Cluster

Center for
Advanced Vehicular Systems

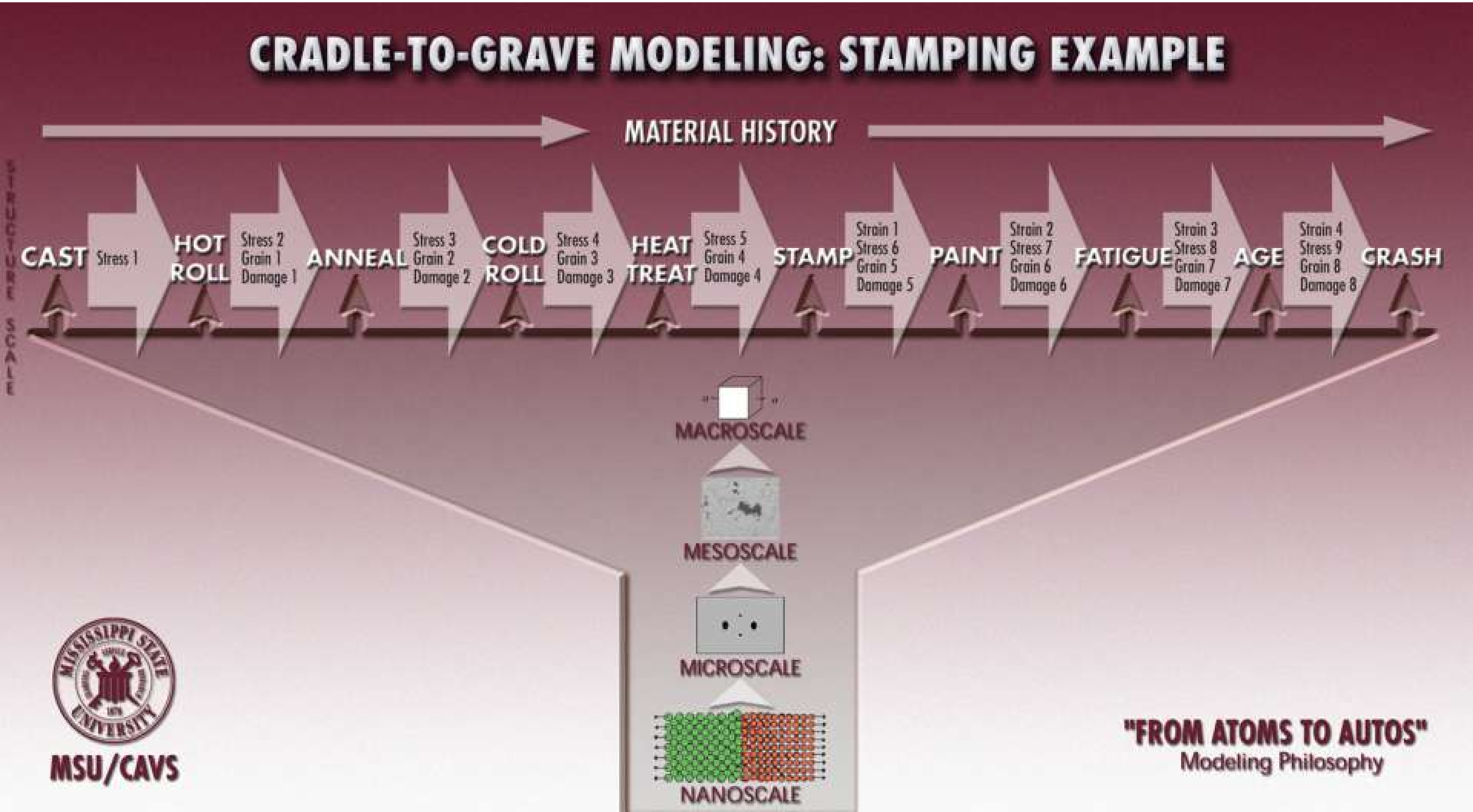
Examples of Mississippi manufacturing sector



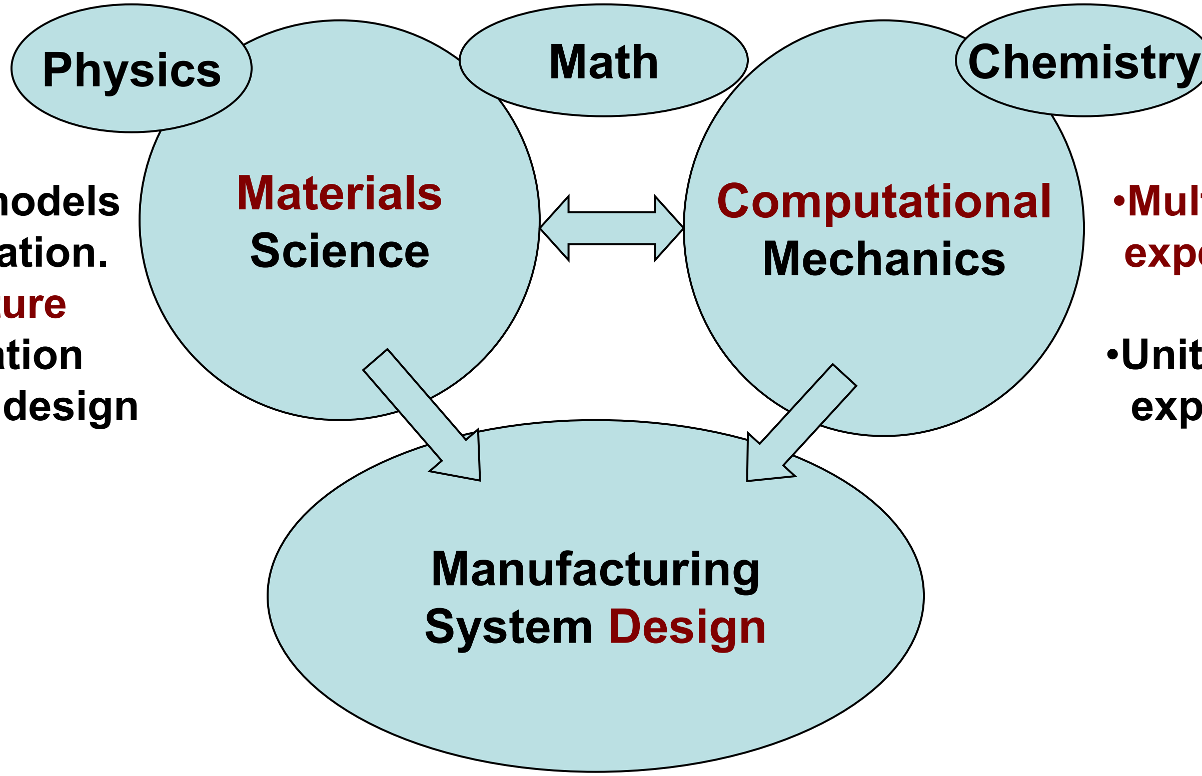
Building Block Portfolio Examples

Computational Manufacturing and Design

Mission: We couple multidisciplinary research of solid mechanics, materials, physics, and applied mathematics in three synergistic areas: theoretical modeling, experimentation, and large scale parallel computational simulation to optimize design and manufacturing processes.



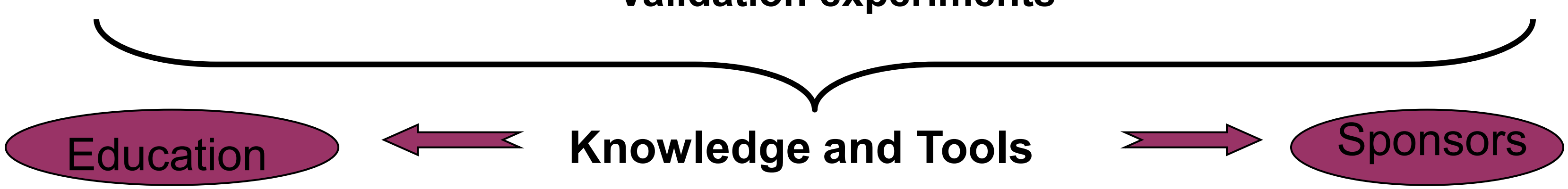
Expertise Required - Interdependency



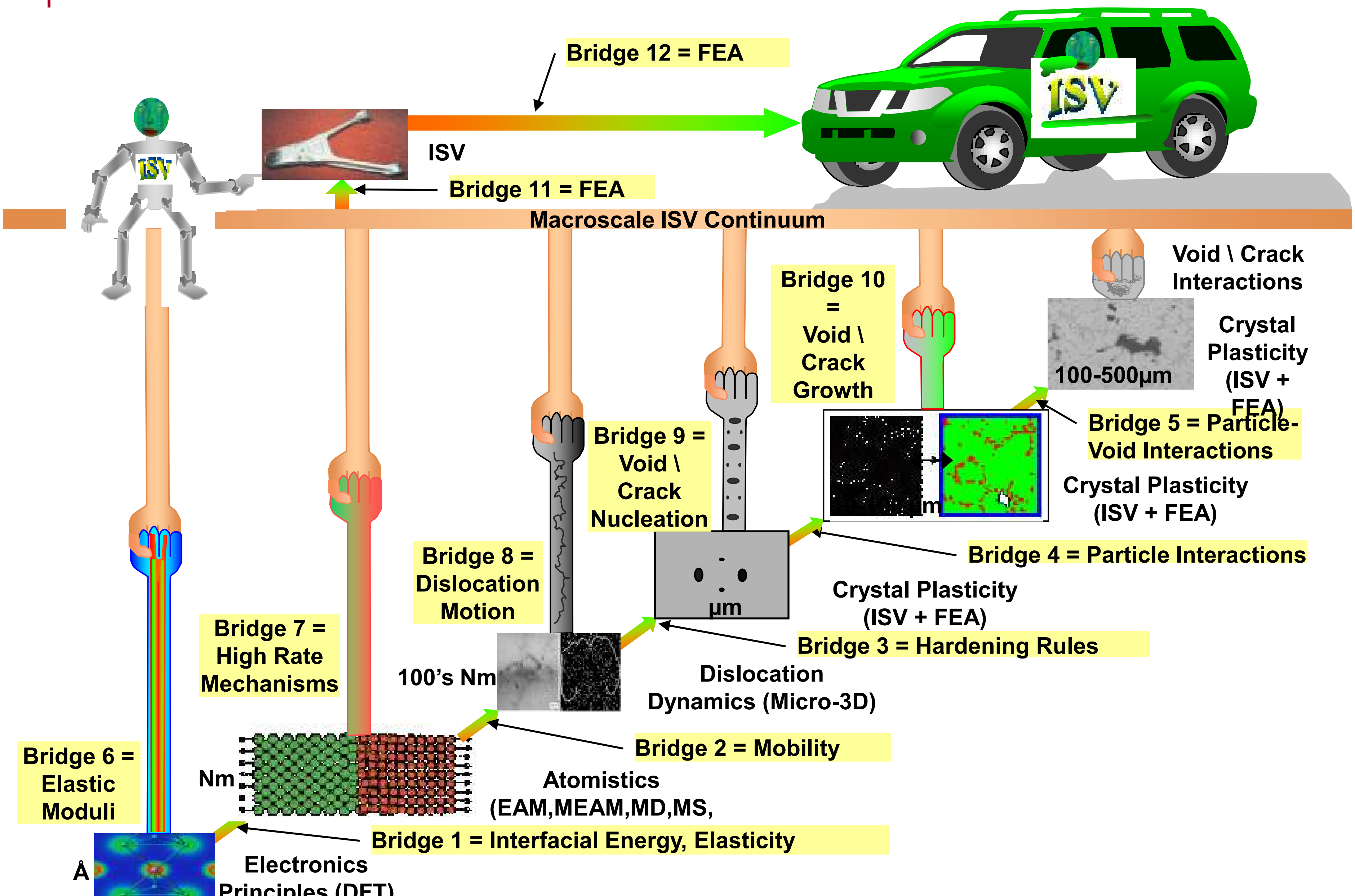
- Mechanism models & experimentation.
- Microstructure Characterization
- Alloy/material design

- Multi-scale models, experimentation, & integration.
- Unit process model, experimentation,& design

- Large engineering system design & integration.
- Component characterization & analysis
- Optimization/uncertainty
- Validation experiments

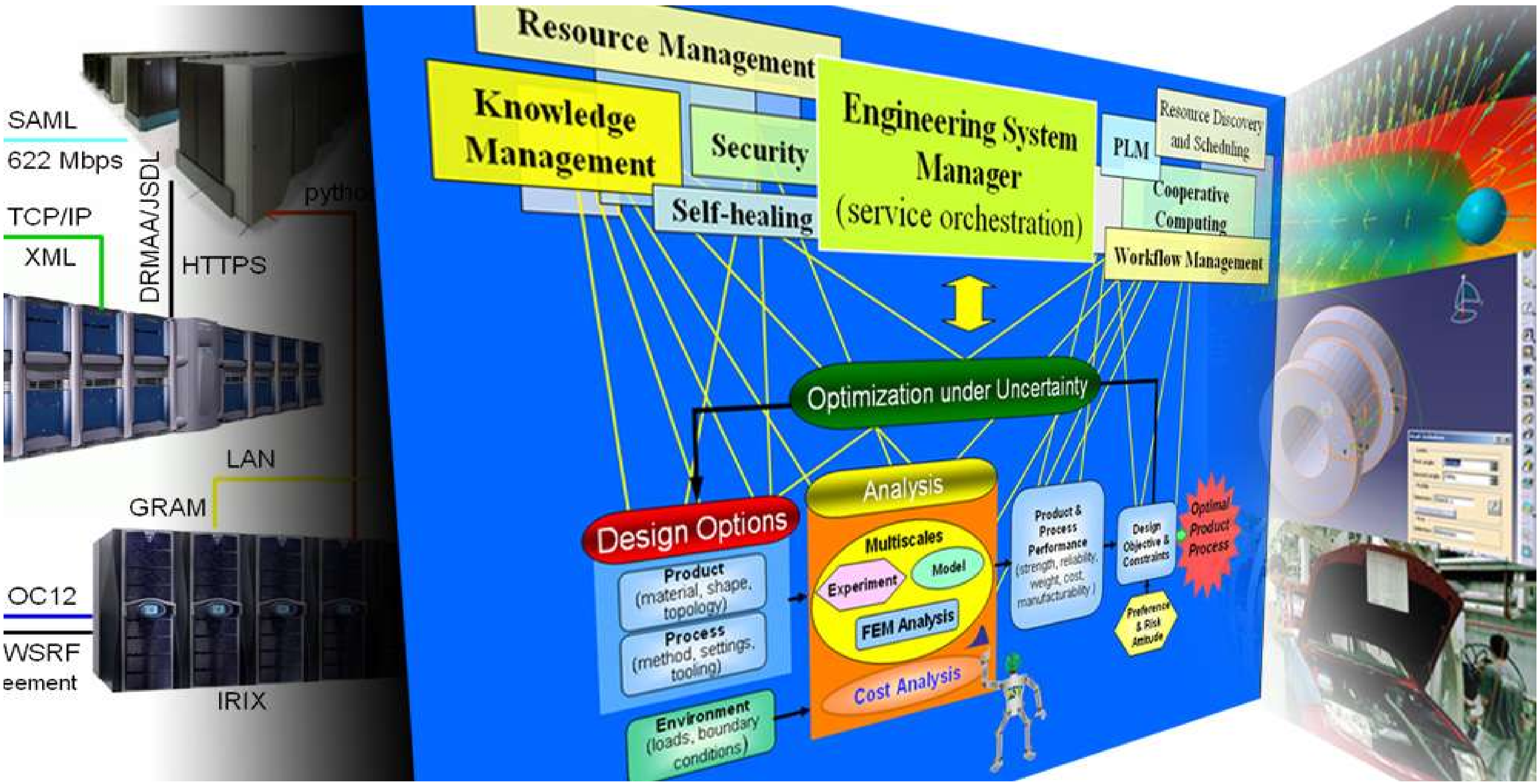


Multiscale Modeling



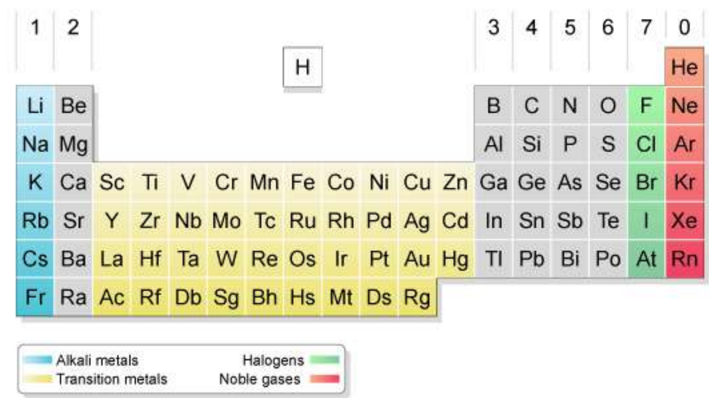
CyberInfrastructure

IT technologies (hidden from the engineer)	Conceptual design process (user-friendly interfaces)	Engineering tools (CAD, CAE, etc.)



CAVS - MEAM Interatomic Potential Development

- Aluminum
  - Al-Mg, Al-Si, Al-Cu, Al-Fe, etc.
- Magnesium
  - Mg-Al, etc.
- Steel
  - Fe-V, Fe-C, etc.



- Nuclear Applications
- Polymer/composites

New research areas

Still needs some work…

- Needs to be efficient!!!
- Optimization techniques – what methodology works best?
- Ease of transferability to new potentials
- Addition of new response variables (e.g., stacking fault energy)

Multi-scale Material Approach

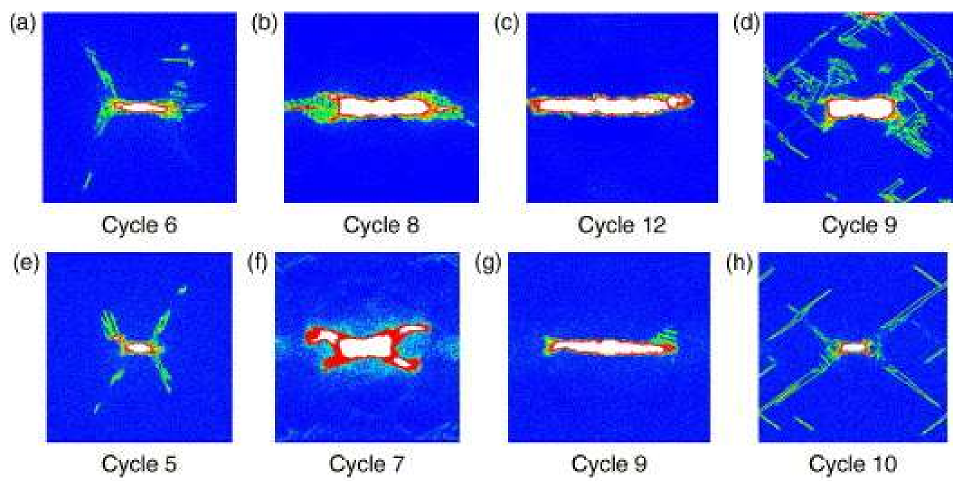
Dislocation mobilities for Al multiscale models

Single crystal dislocation nucleation

Grain boundary dislocation nucleation

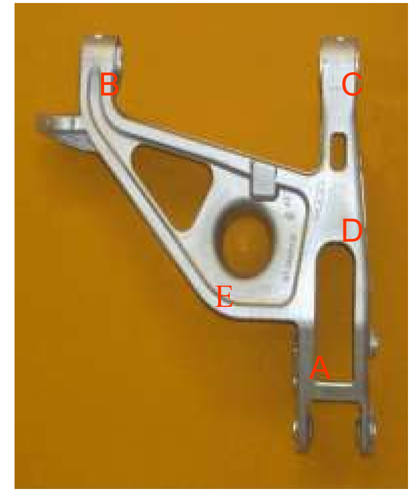
Void growth and coalescence

Fatigue and Damage
Potirniche, Horstemeyer, et al. IJF (2005)

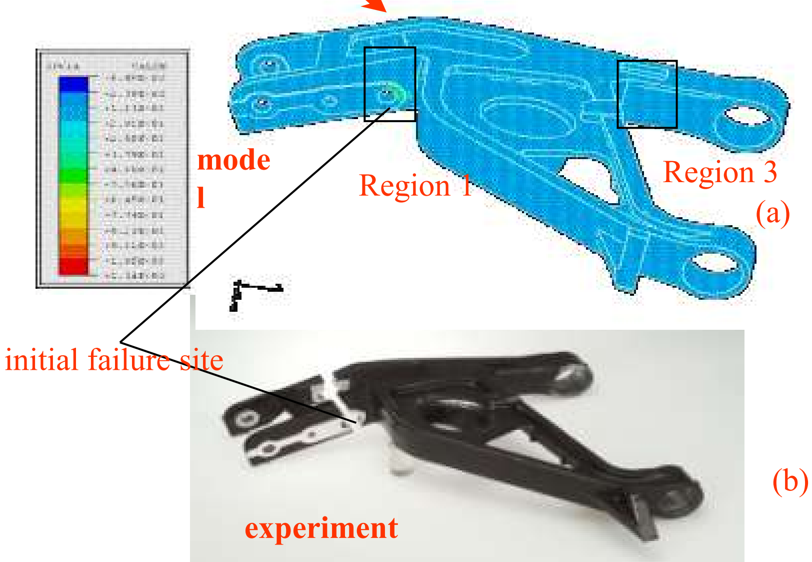


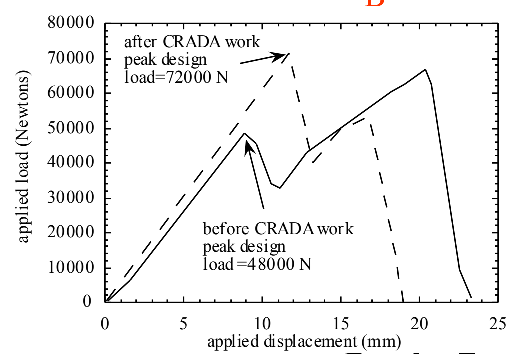
GM CADILLAC CONTROL ARM LIGHTWEIGHT DESIGN

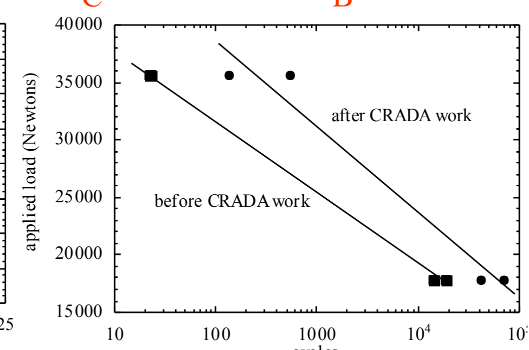
Objective: *To employ multiscale material modeling to reduce the weight of components*



Stress (from highest to lowest)	Inclusion (from most severe to less severe)	Damage (from most severe to less severe)
D	B	A
A	E	D
C	A	E
E	D	C
B	C	B







Result: *To optimize a redesign such that*

25% weight saved

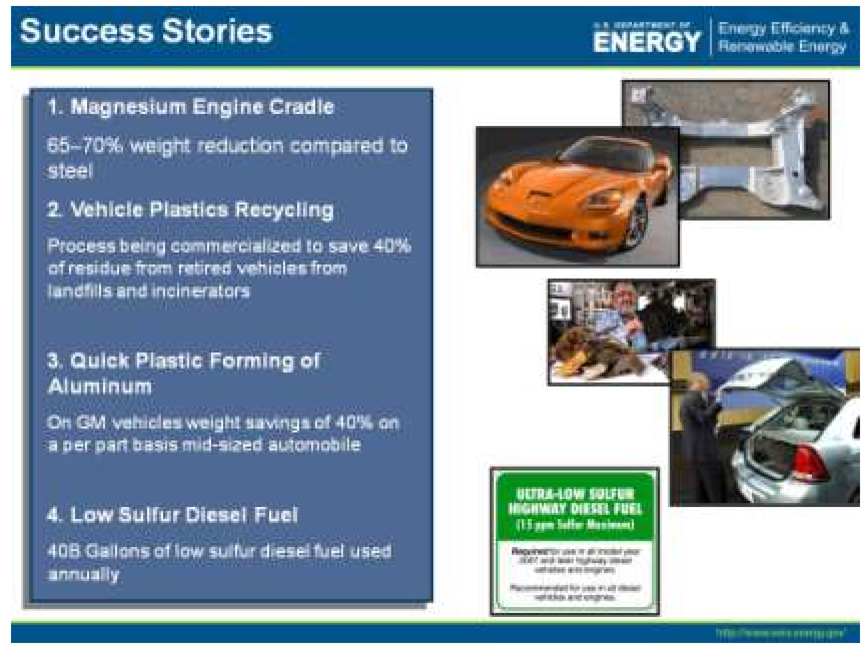
50% increase in load-bearing capacity

100% increase in fatigue life

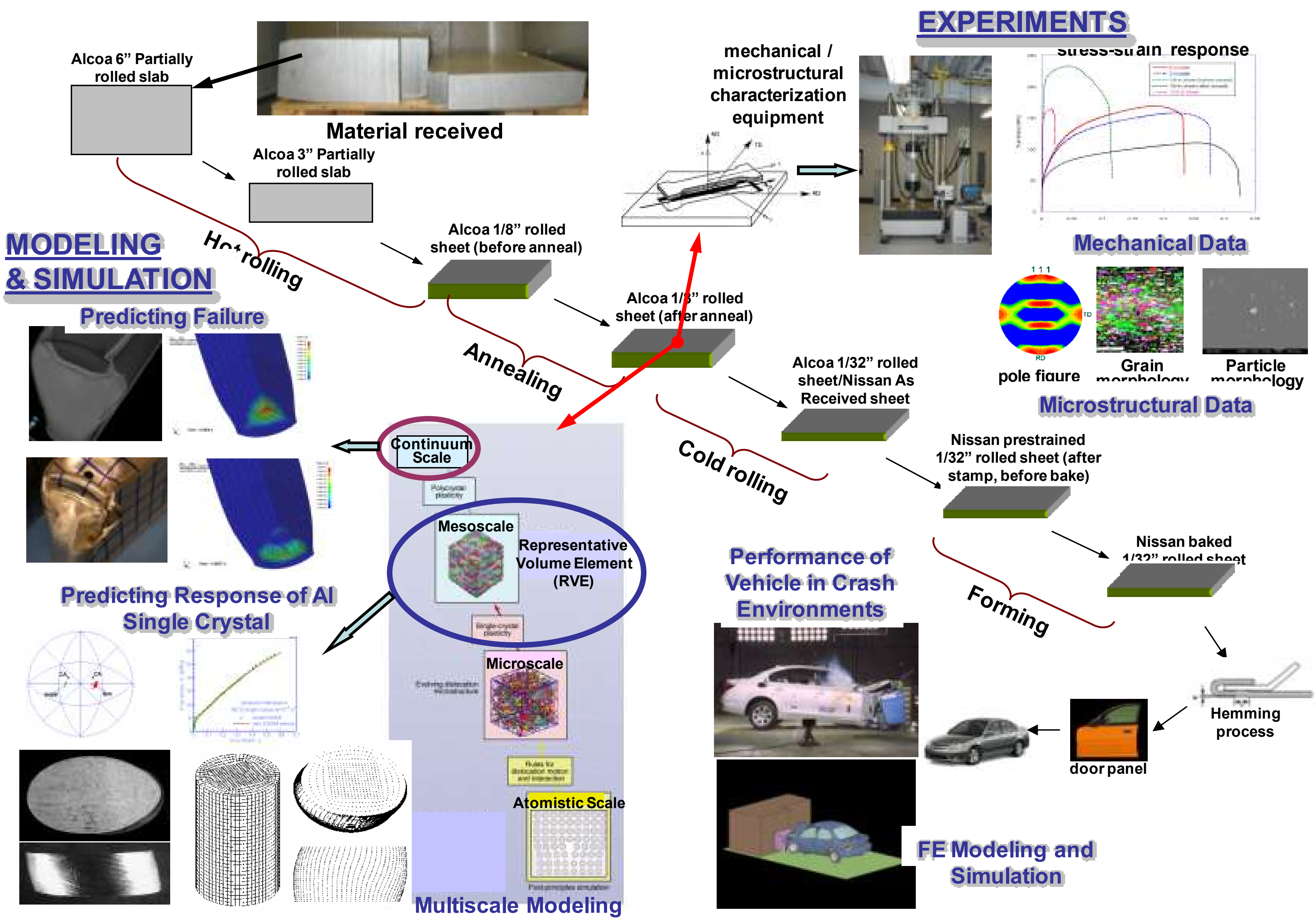
$2 less per part

Innovative Use of Lightweight Materials

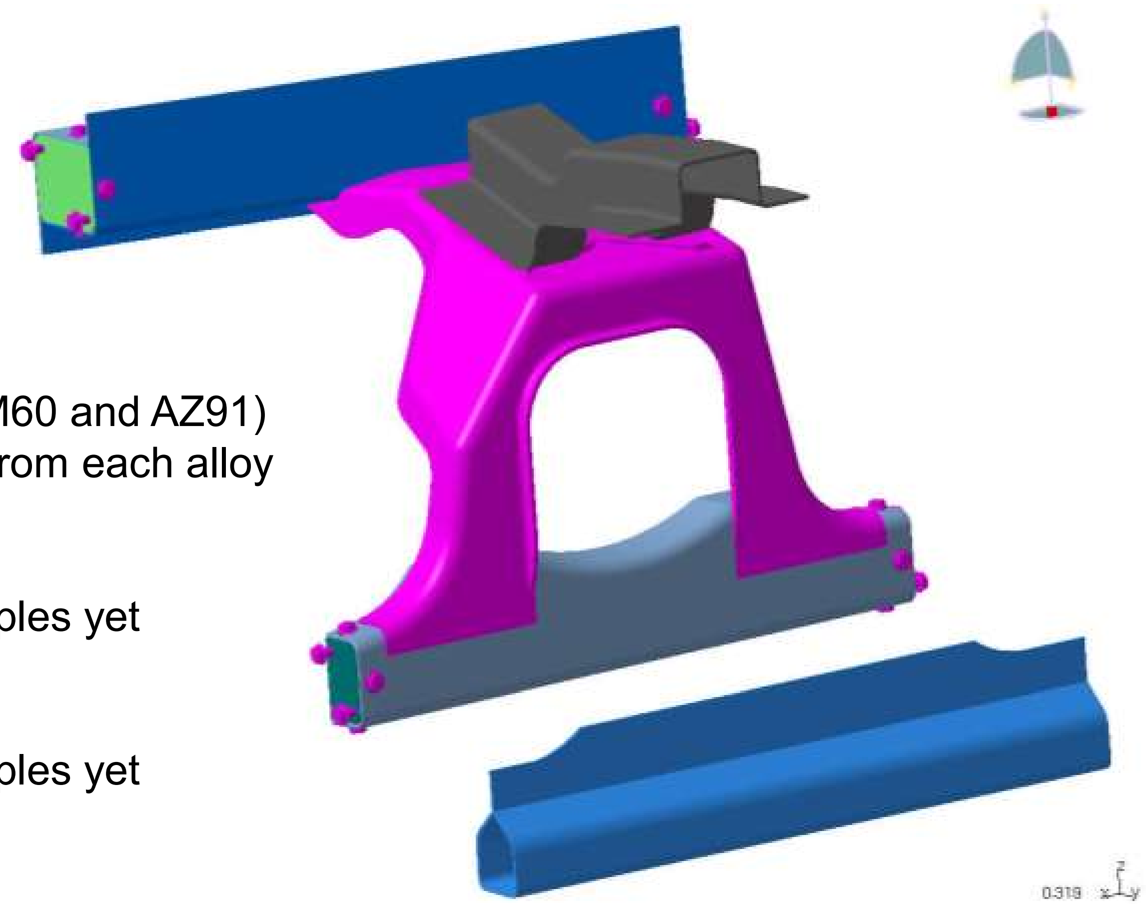
CAVS is a leader in the use of lightweight materials for automotive applications.



Multiscale Material Testing/Modeling/Simulation



USAMP-Auto Demo structure



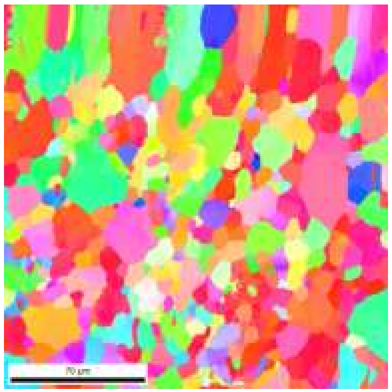
- ***Casting*: shock tower**
  - Alloys (casting team: AM60 and AZ91)
  - Received two samples from each alloy
- ***Extrusion*: Rail**
  - Alloy: AM30
  - Received no demo samples yet
- ***Stamping*: sheet**
  - Alloy: AZ31
  - Received no demo samples yet

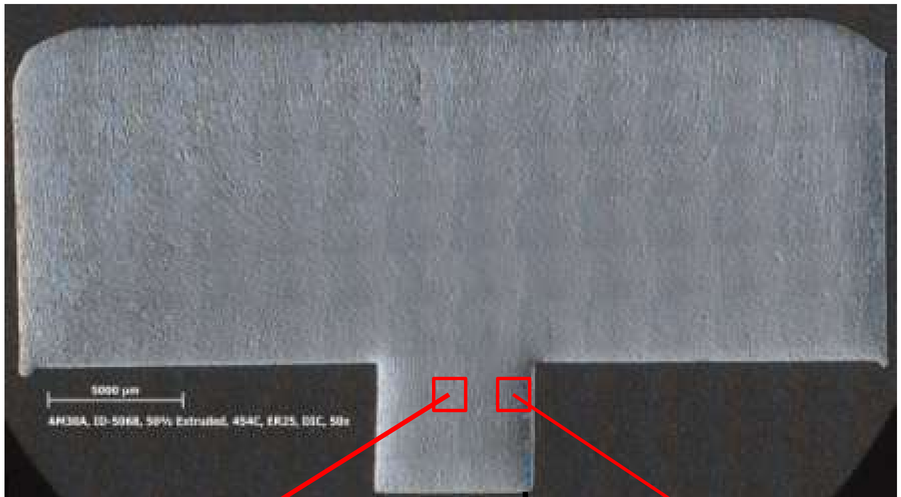
Work with USAMP casting, sheet and extrusion teams to implement local property using zone mapping methods

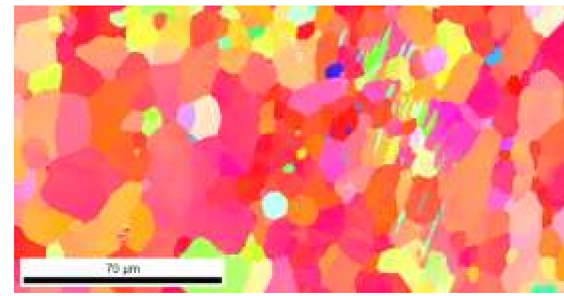
Mg Extrusion Results

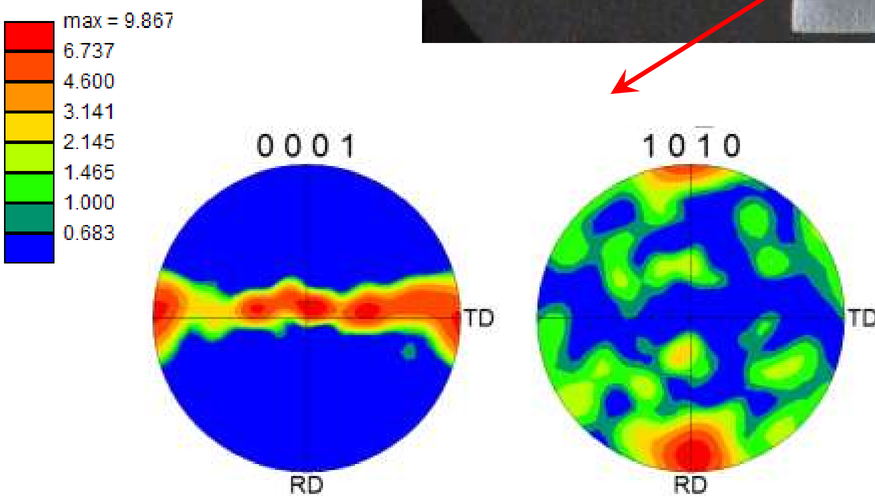
Flat-Die - AM30

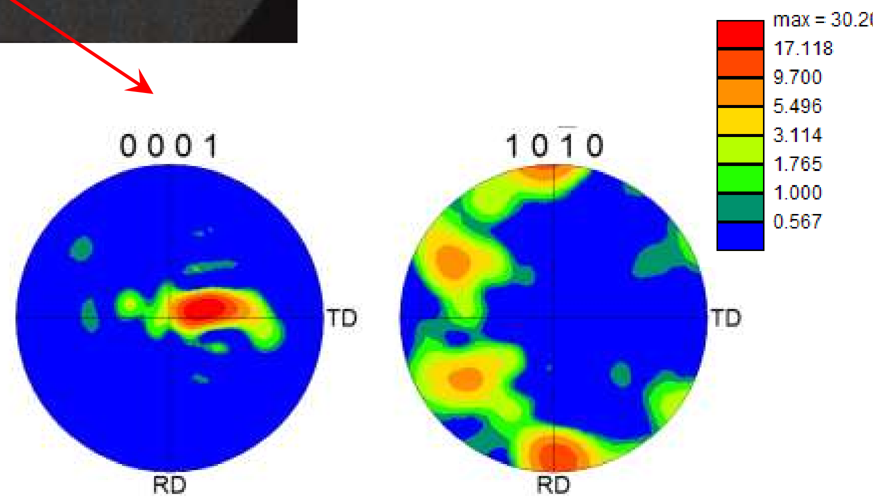
5 mm/min, 454°C, ER=25, 50% extruded



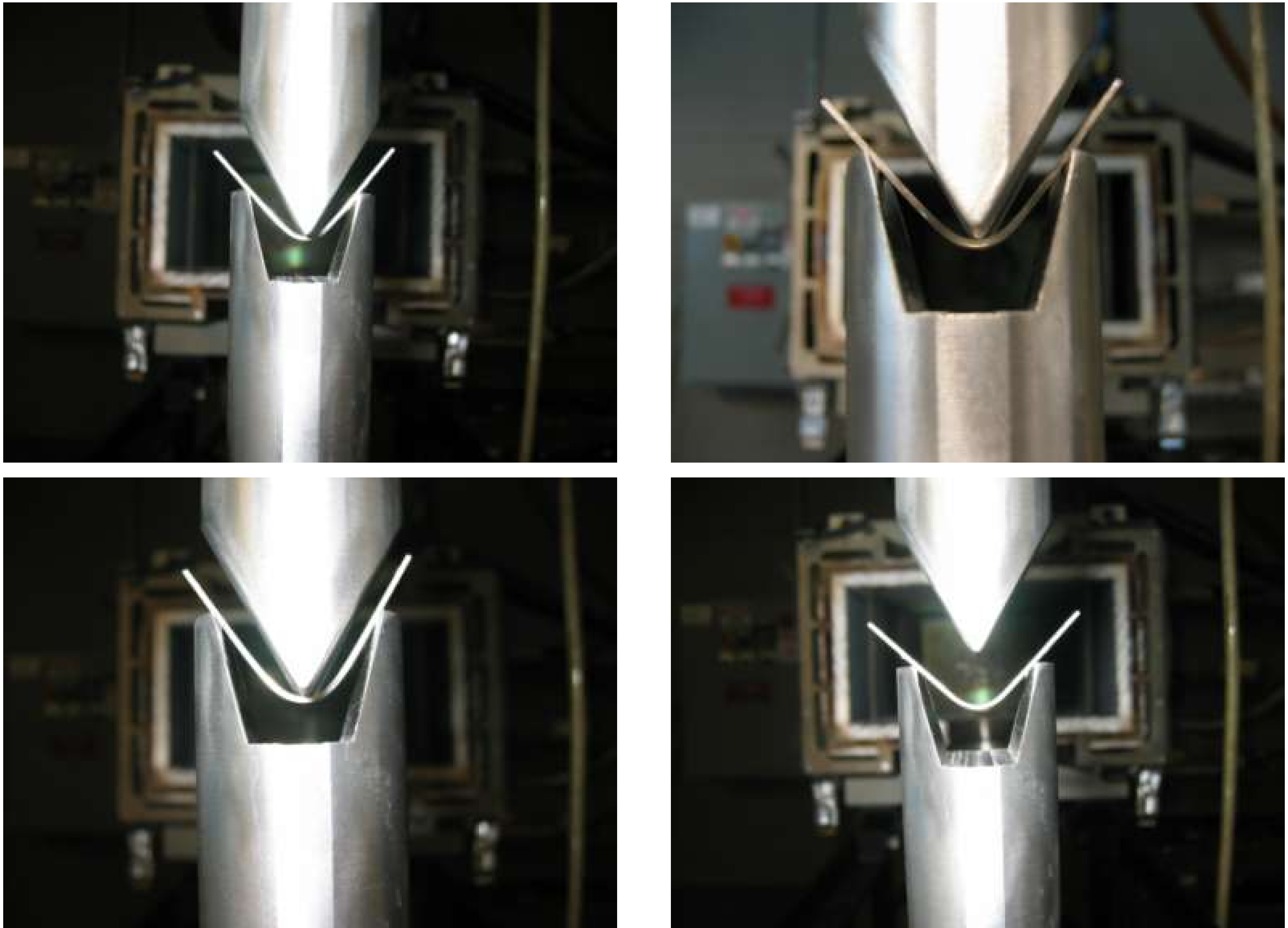








Mg AZ31 Sheet Bending - INSTRON



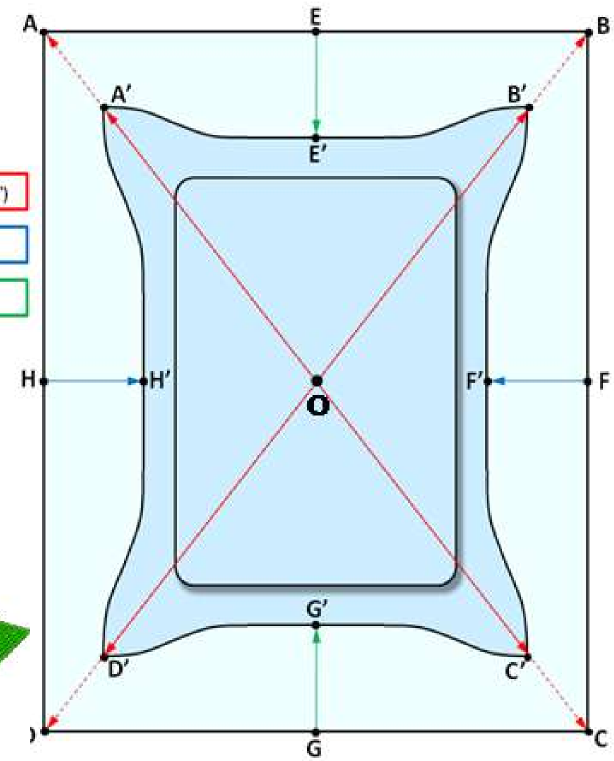
Pan Forming - Dimensions and Thickness Profiles
Material AZ31-O

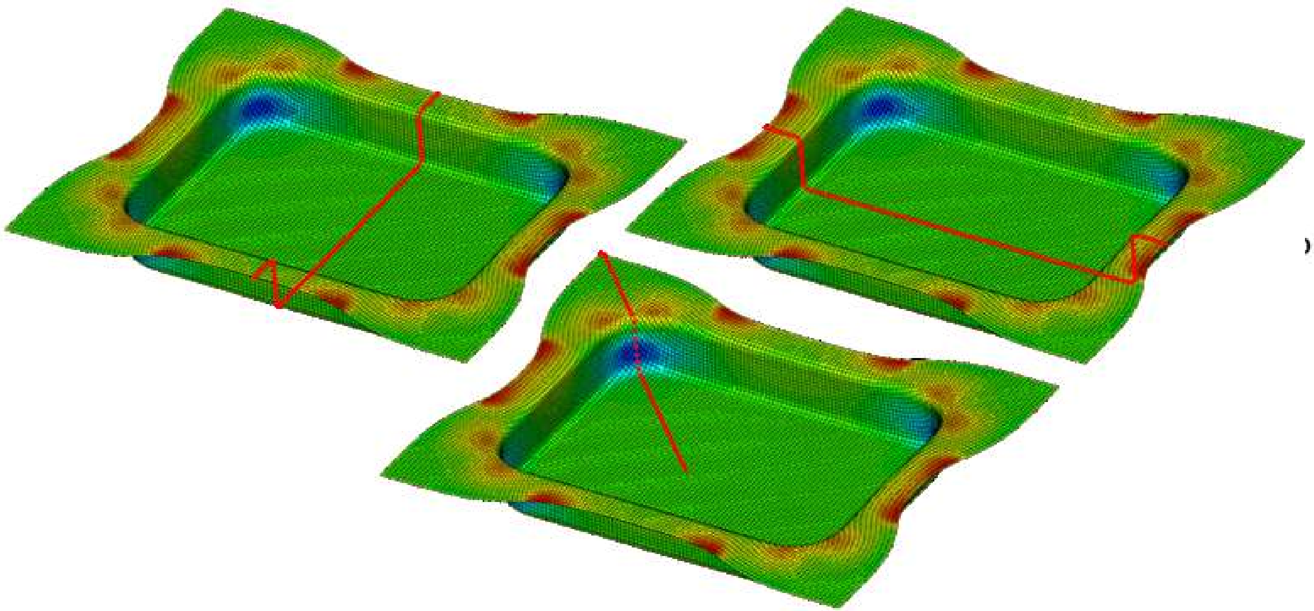
Draw Dimensions d_1, d_2 and d_3 used for comparing numerical and experimental pan forming results

d_1 = (AC-A'C')+(BD-B'D')

d_2 = HH'+FF'

d_3 = = EE'+GG'





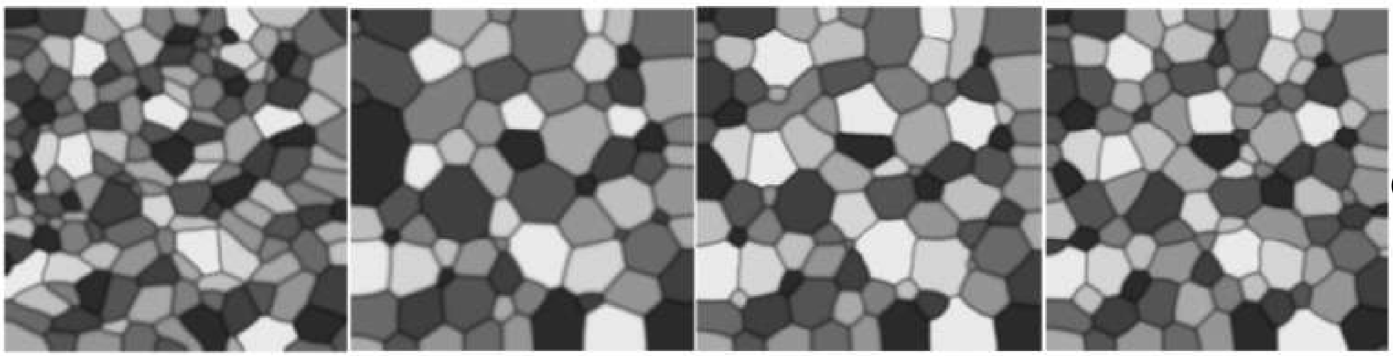
Different nodal paths for outputting thickness profiles.

Grain Growth without and with Particles

Example: Cu-Ni-Si: high strength, electric and thermal conductivity because of re-aging δ - Ni2Si precipitates

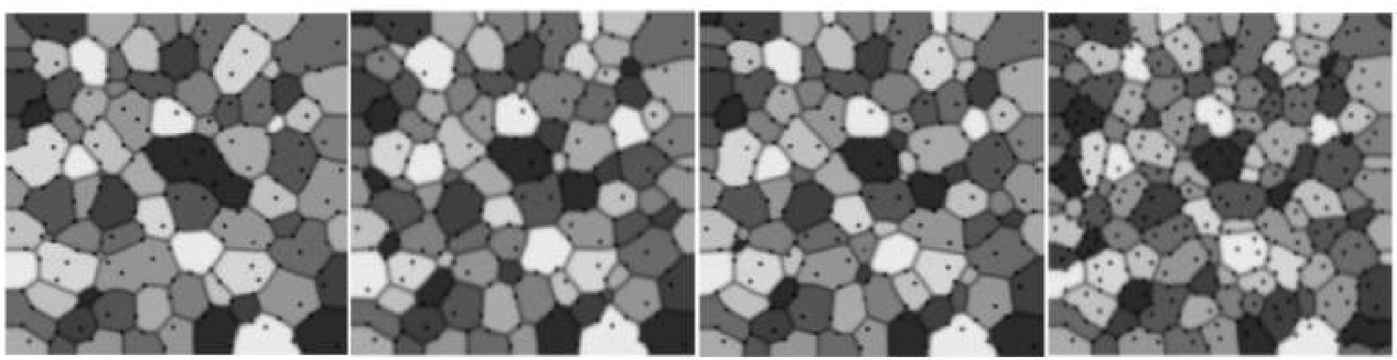
Grain growth during re-aging process in the Cu-Ni-Si alloy

$\bar{R}_{g\ initial} \cong 7.5$ Domain: 500×500 Non-dimensionalized length scale



Initial grains | Isotropic | RS | MRS

$(after\, 10^4\ scaled\ time\ steps)$



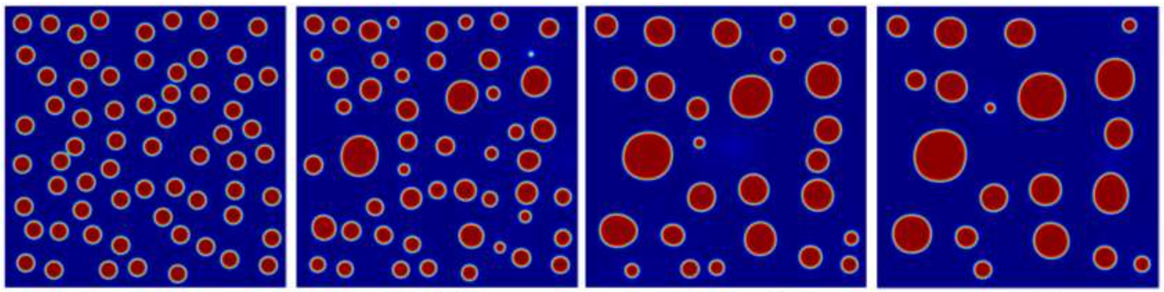
Isotropic | RS | MRS | MRS

$r_p = 2,\quad V_f = 0.015$ | $V_f = 0.015$ | $V_f = 0.015$ | $V_f = 0.03$

Phase Transformation - Two Dimensional Analysis

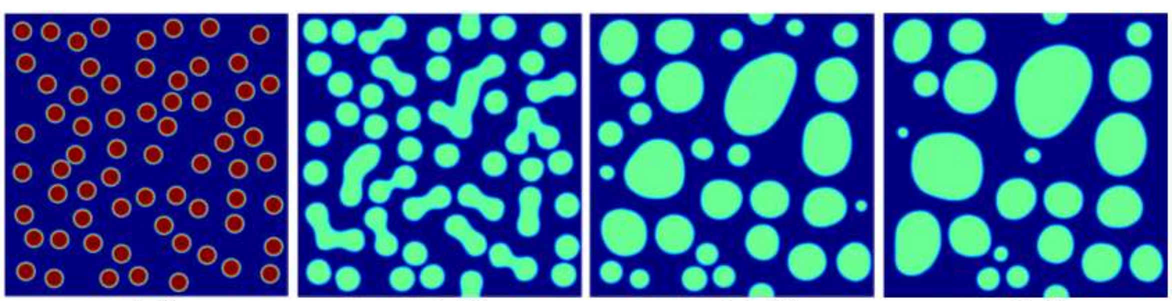
The coarsening power law:

$$(r_t)^n - (r_0)^n = K t$$

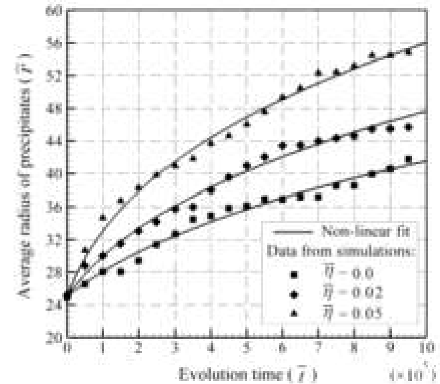


$\bar{t} = 0$ $\bar{t} = 10^5$ $\bar{t} = 4\times10^5$ $\bar{t} = 10^6$

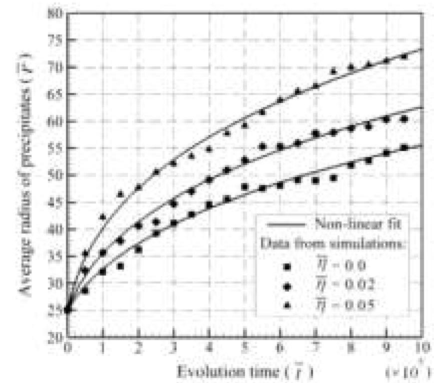
	$V_p^f \cong 0.2$		$V_p^f \cong 0.4$	
η	n	K	n	K
0	3.58	0.52	3.49	1.25
0.02	3.57	0.88	3.53	2.12
0.05	3.60	1.86	3.52	3.59



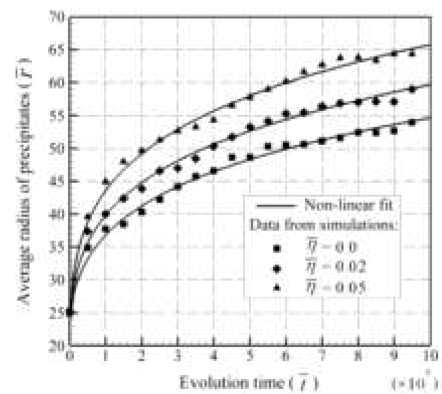
$\bar{t} = 0$ $\bar{t} = 10^5$ $\bar{t} = 5\times10^5$ $\bar{t} = 10^6$



$V_p^f \cong 0.2$

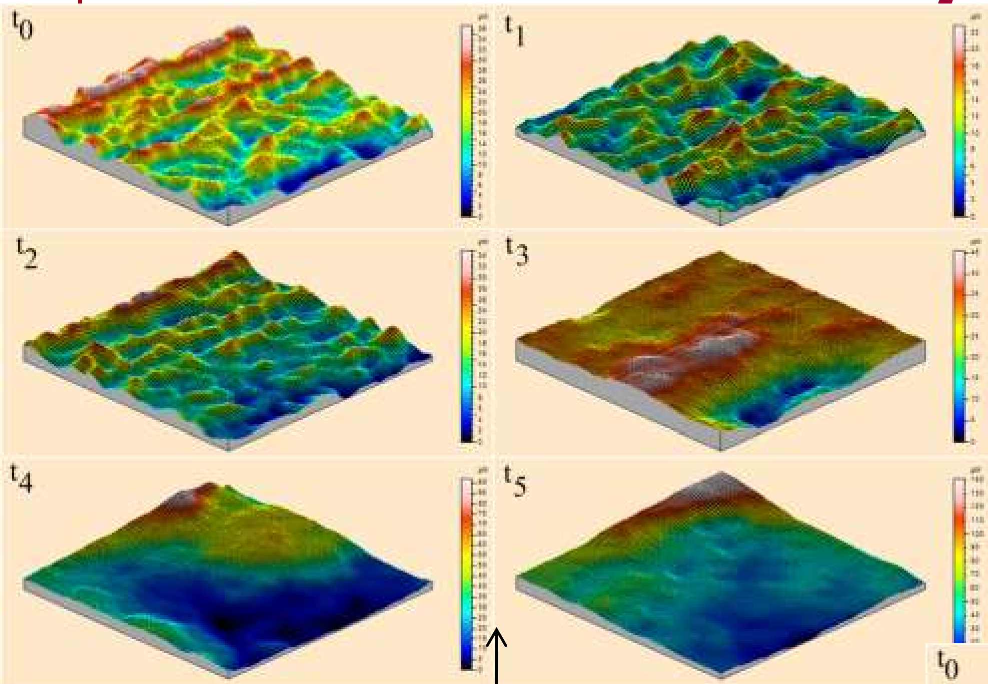


$V_p^f \cong 0.4$



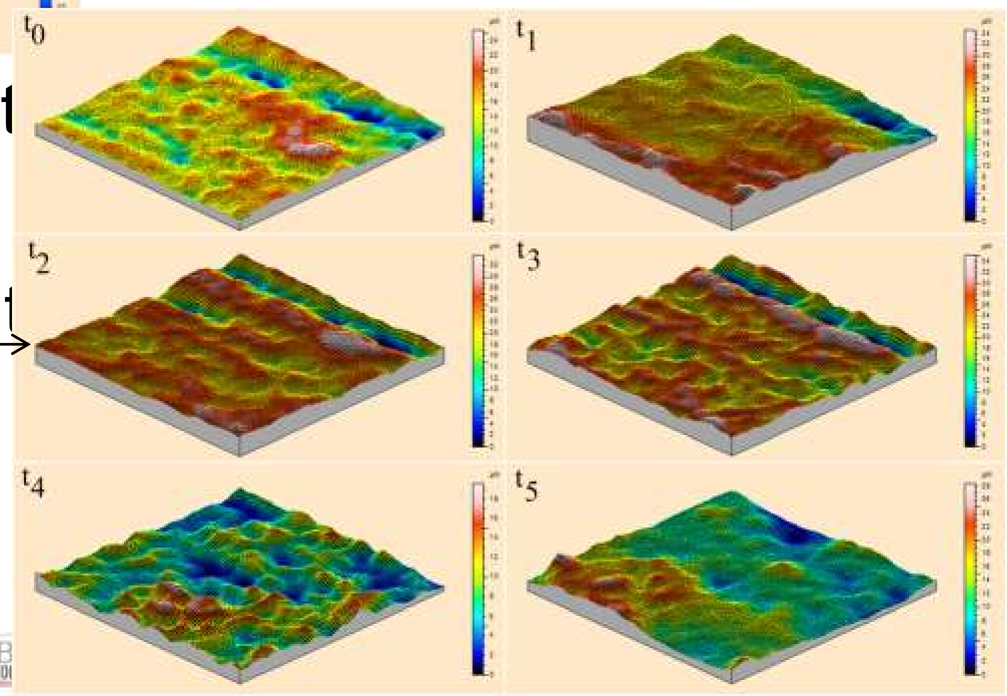
$V_p^f \cong 0.2$

Surface Profilometry – AZ31



- Small difference in number of pits present
- Pits grew larger on the salt spray surface by t_5
- Higher volume
- Pits covered more area on immersion surface by t_5
- Higher surface area

- Salt Spray Environment
- Immersion Environment



Materials Research for Nuclear Energy

1. NEAMS provides MS State with a great opportunity for highly visible R&D that aligns with our strength of computational science and engineering
2. NEAMS allows a fairly long term research program
3. NEAMS allows for MS State collaboration with national labs and other prestigious academic institutions

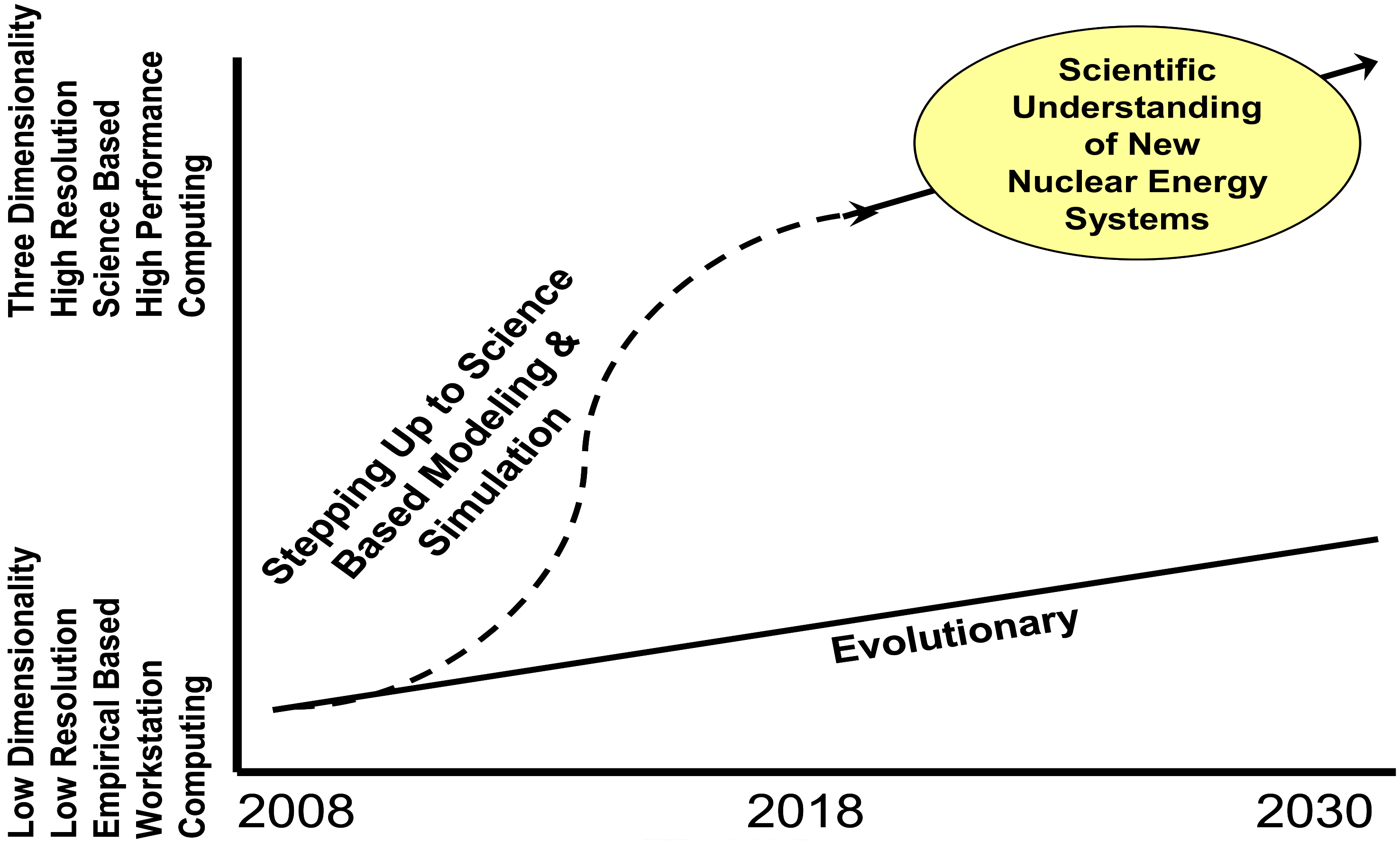
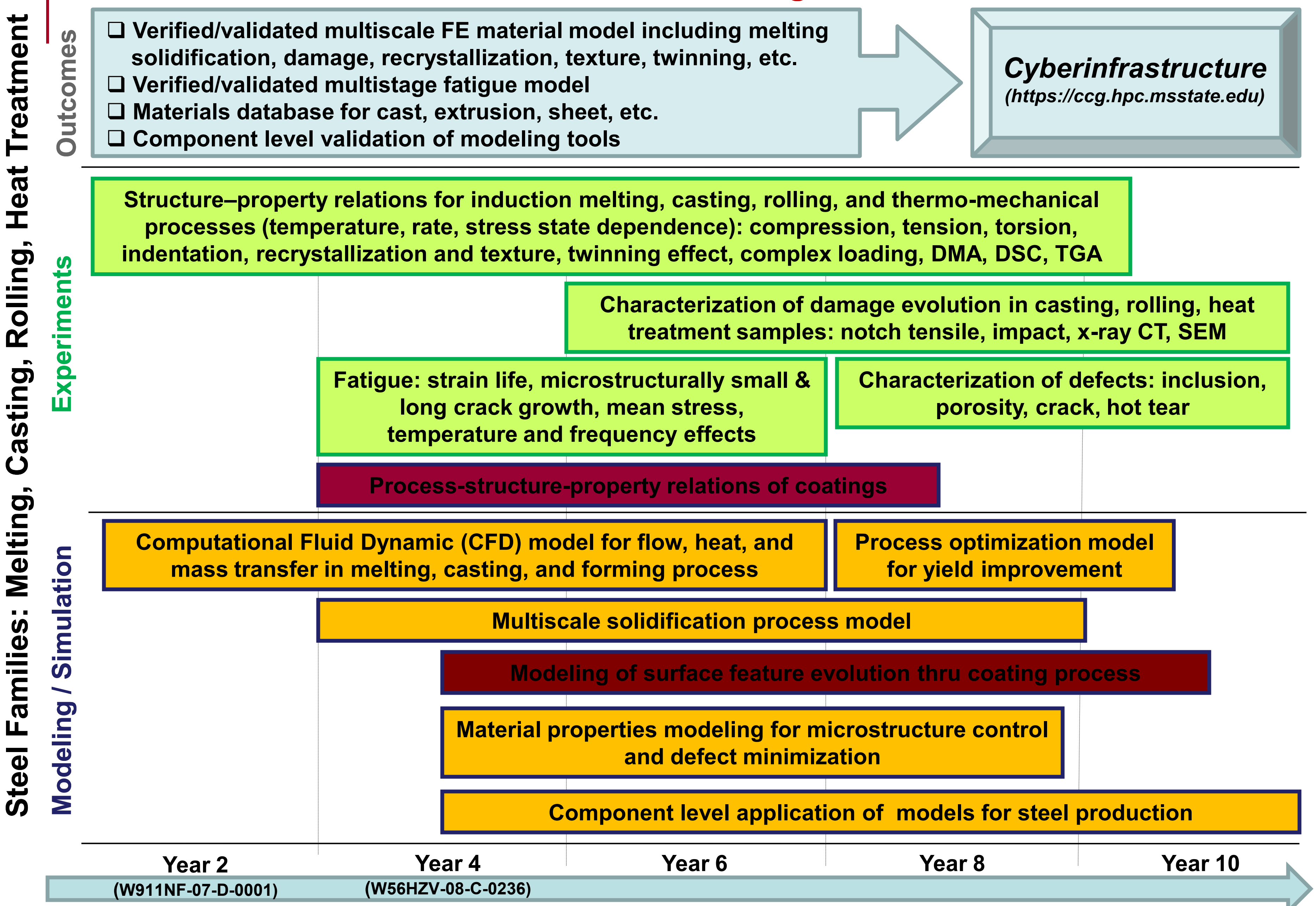


Figure - Moving to a New Level of Capability

CAVS Steel Research Portfolio

- Lab-scale experimentation to address process-structure-property relationships
- Computational tools to simulate heterogeneous boundary conditions in processing and performance cases

10 Year Plan for Multiscale Modeling of Steel Research



CAVS Steel Research Organization

Research Group Leader
Dr. Hongjoo Rhee

Advisory Board
Dr. Paul Wang

Dr. Roger King

Dr. Mark Horstemeyer

Dr. Ron Radzilowski
(& Severstal Representatives)

Dr. Frank Goodwin
(International Zinc Association)

Alloy Design Group
Dr. Haitham El Kadiri

Dr. Quancang Ma (Post Doc.)

Students

Casting & Solidification Group
Dr. Sergio Felicelli

Dr. Liang Wang

Students

Lab Testing & Characterization Group
Dr. A. Oppedal (Research Leader)

Mr. S. Horstemeyer (Lab Manager)

Mr. R. Malley (Technician)

Research Associates

Students

Thermomechanical Processing Group
Dr. Esteban Marin

Dr. Mohsen Asle Zaeem

Mrs. Clemence Bouvard

Students

Sheet Formability & Performance Group
Dr. Mark Horstemeyer

Dr. Marcus Lugo (Post Doc.)

Students

Sheet Forming & Coating Group
Dr. Bin Li

Dr. Mohsen Asle Zaeem

Dr. Youssef Hammi

Students

CAVS Current Experimental Capabilities

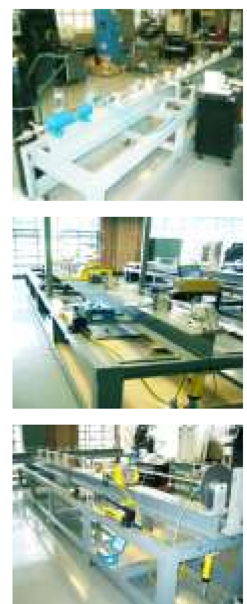
- Materials Characterization Facilities
  x-ray CT scanner, high performance FEG-SEM, EVO–SEM, optical microscope, surface analyzer, particle size analyzer, spectrometer, ……
- Mechanical Properties Testing Facilities
  nano- & micro-indenter, high strain-rate Hopkinson bar testers (compression, tension, and torsion), Instron machines (50 kN and 100 kN load capacity), bi-axial Instron machine, MTS machines (uni- & bi-axial modes), hardness testers, ……
- High Temperature Characterization Facilities
  TGA, DSC, DMA, dilatometer, microwave sintering furnace, muffle furnaces, powder injection molder, extruder, powder compaction machines, ……
- Specimen Preparation Facilities
  saws/cutters, polishers, mounting press, ultrasonic cleaner, etching capabilities (hood, etchant, storage, etc.), machine shop, ...


FEG-SEM


X-ray CT System


Nanoindenter


Talysurf


Hopkinson Bar Testers


Bi-axial Instron Machine


High Temp. Vacuum Furnace


High Temp. Infrared Chamber for Elevated Temp. Mechanical Testing

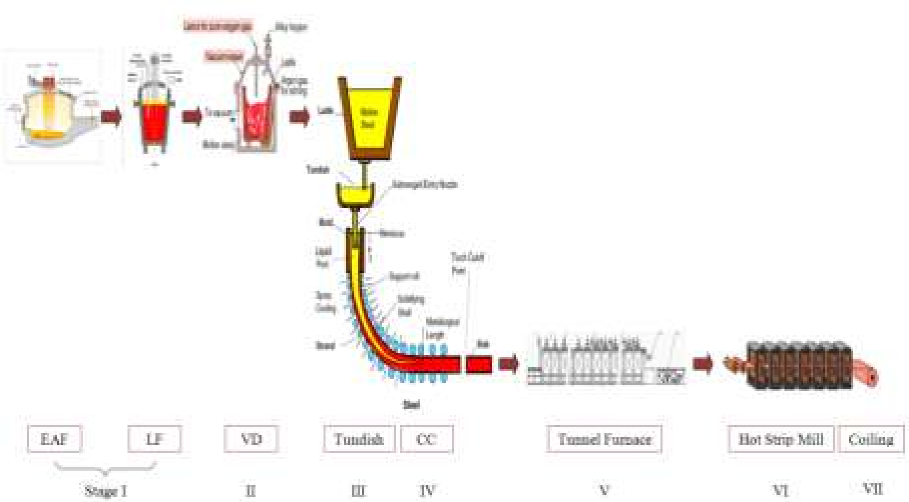
High Performance Computing

- High-Performance computing systems
  - Talon: 3072 processors, 6 TB RAM
  - Raptor: 2048 processors, 4 TB RAM
  - Matador: 512 processors, 512 GB RAM
  - Maverick: 384 processors, 480 GB RAM
- Storage
  - 250 TB of high-speed disk storage
  - 2 PB of near-line storage
- Based on the June 2010TOP500 list, Talon is equivalent to the 331st fastest computer in the world and the 18th fastest computer in US academia
- Based on the June 2010 Green500 list, Talon is equivalent to the 9th most power efficient system in the world (power efficiency is calculated as millions of floating point operations per second per Watt of energy consumed)

Manufacturing Process Optimization

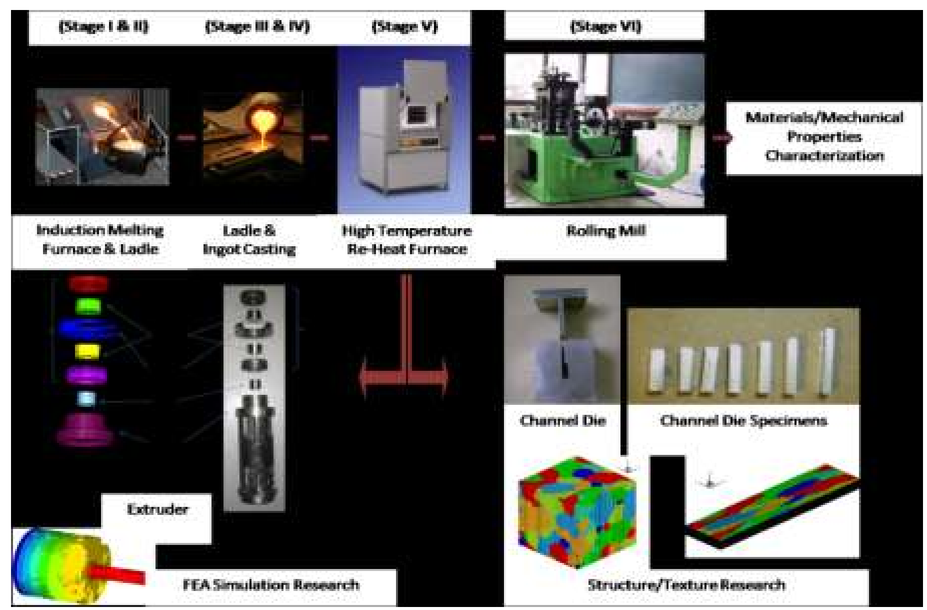
Steel Manufacturing Processes (Cradle to Grave Concept)

- Identify different stages of steel manufacturing plant processes.
- Among various commercially available mass-product steel manufacturing processes, one of the typical examples of melting, ladle refinement, micro-alloying, continuous casting, and hot rolling for thin slab direct rolling process is schematically illustrated below:

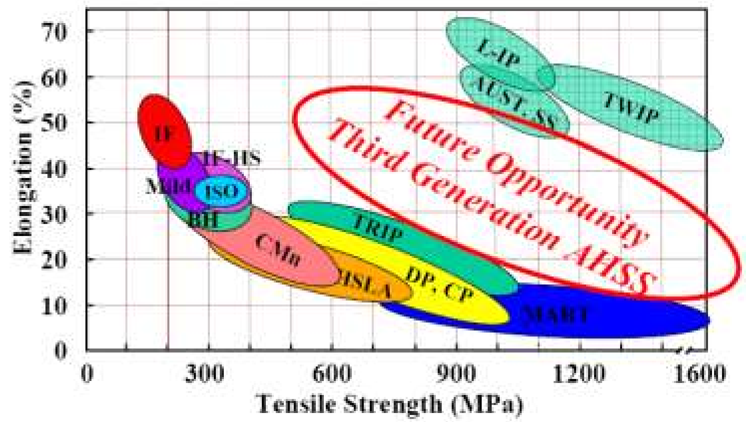


Sub-Scale Manufacturing Cells

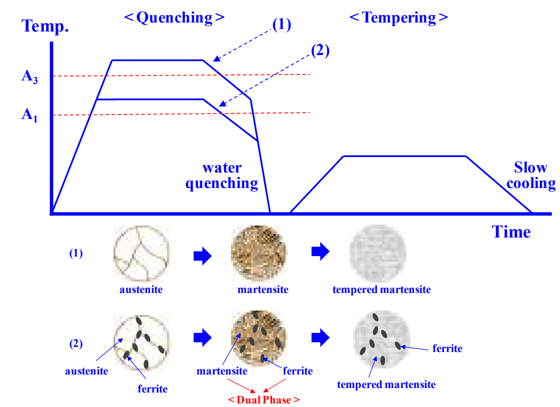
- Construct lab-scale manufacturing cells for aforementioned stages.
- The lab-scale facilities can provide various experimental simulations related to pilot- and mass-product steel manufacturing processes as well as model validation.
- Description of such small-scale cells and applicable experimental simulations regarding previous manufacturing stages are stated as follows:



Advanced High Strength Steels (AHSS) Research

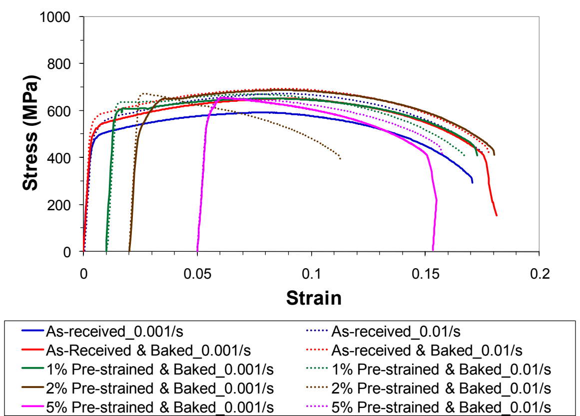


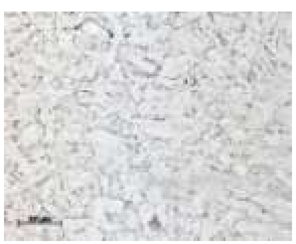
AHSS Overview



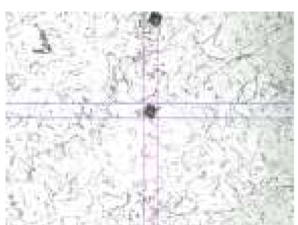
C	Si	Mn	P	S	Cr	Mo	Al
0.123	0.103	1.870	0.0130	0.0046	0.008	0.061	0.049

Chemical Composition



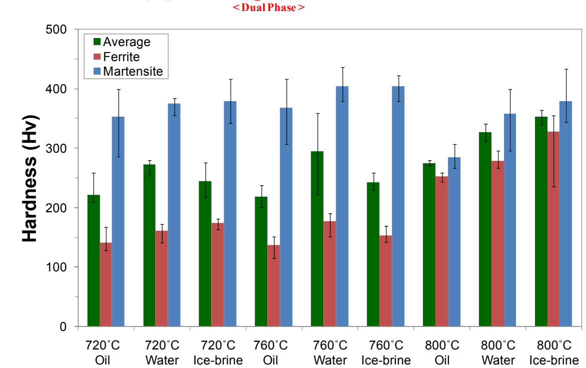


Microstructure



Hardness

Thermomechanical treatment and Mechanical Tests



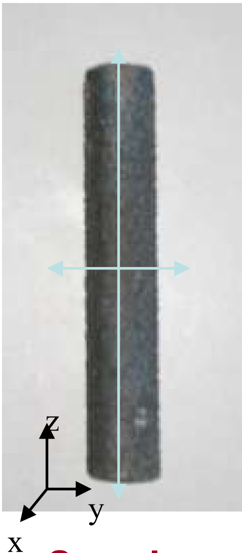
Heat treatment and Mechanical Tests

Mississippi State University

BAGLEY COLLEGE OF ENGINEERING

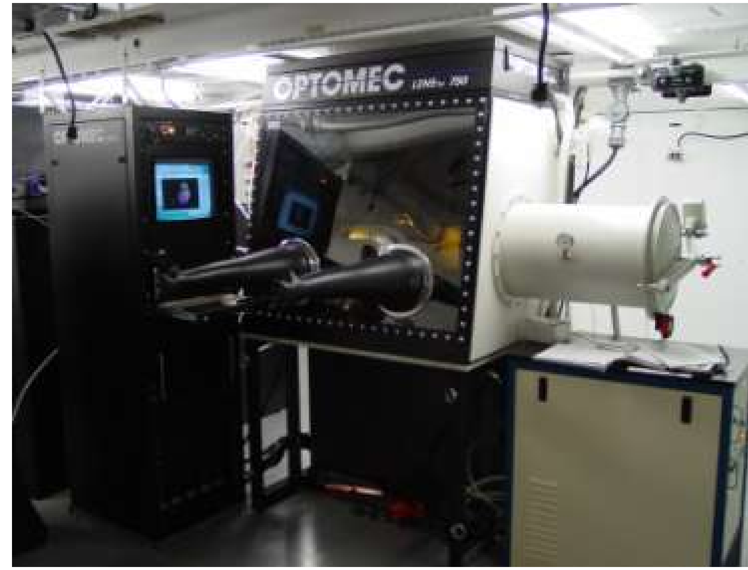
CAVS

Laser Engineered Net Shaping (LENS)

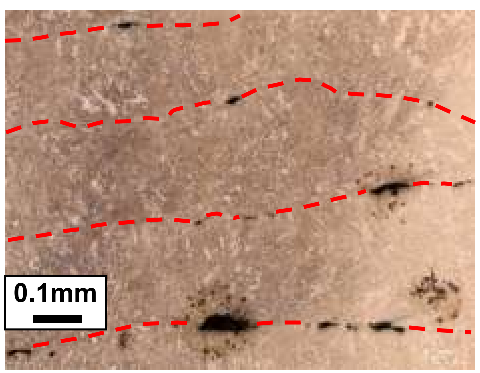


Sample

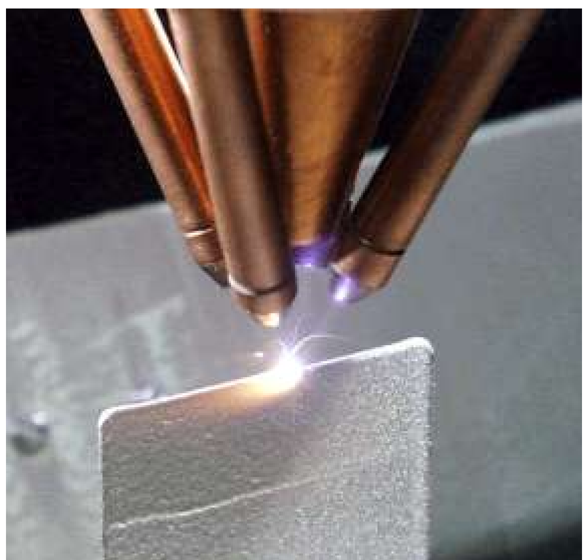
Purpose:
Develop and Integrate thermal mechanical LENS process models with multi-stage fatigue models to produce high quality LENS products for specific usage.

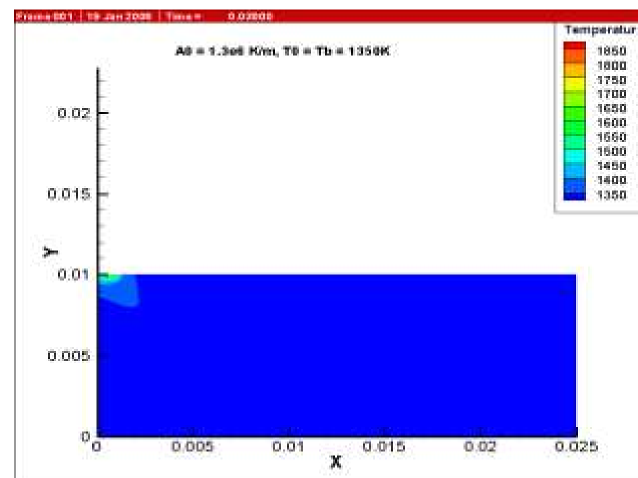


Optomec Lens Machine



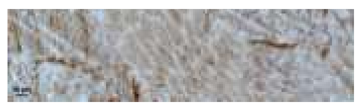
Microstructure in the Sample



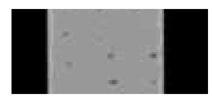


LENS Research Roadmap

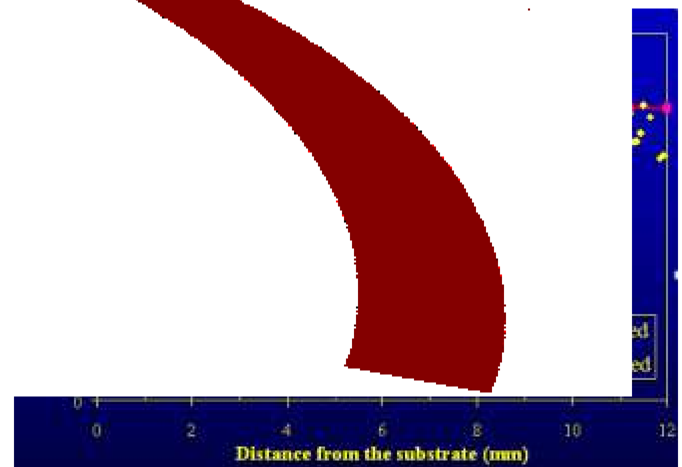
Fatigue life

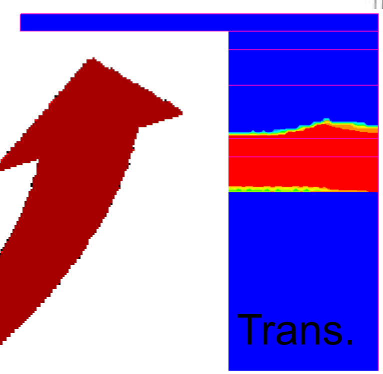


Microstructure



Porosity

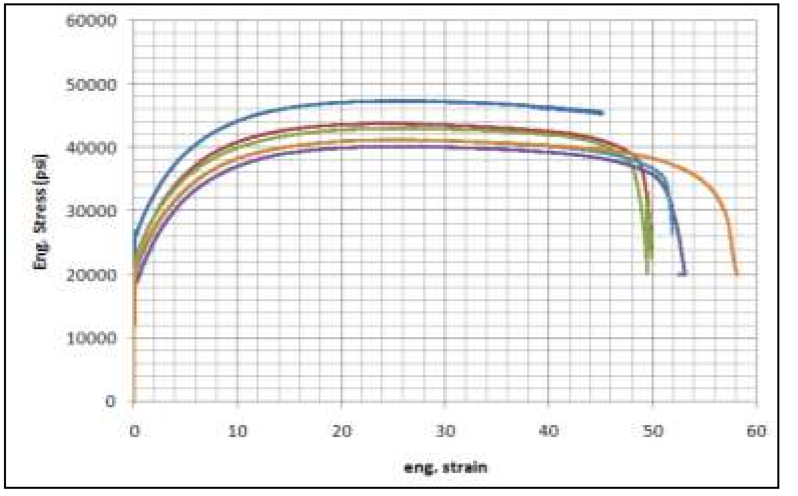




Experiment

Materials Characterization for Steel Manufacturer

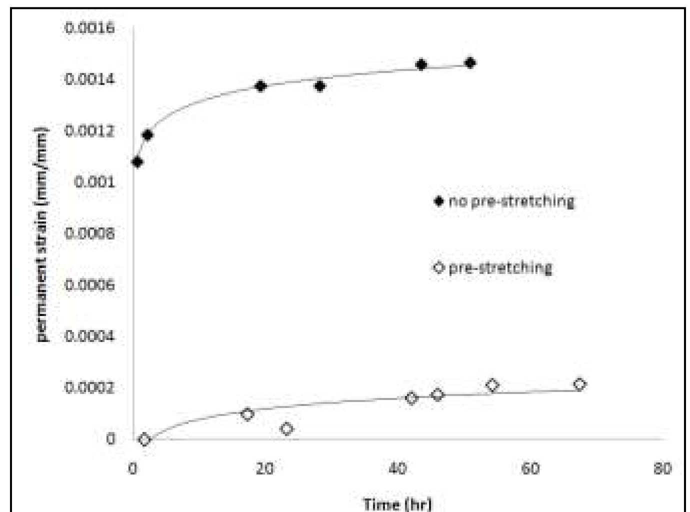
- Provided multiple services for local steel manufacturer, Severstal, located in Columbus, MS.
- Conducted tensile characterization of rolled steel sheets for purpose of validation of mechanical behavior and process parameters.
- Performed annealing process on rolled steel for experiments related to manufacturing process design.
- Currently conducting experiments to understand the cause of corrosion on hot mill presses.



Automotive Manufacturing

- ❑ Assisted Nissan of North America, Canton, MS manufacturing plant in several projects related to manufacturing:
  - ❑ Mechanical characterization and testing of materials/components
  - ❑ Failure analysis
  - ❑ Finite element simulation and modeling
  - ❑ Moving from reactive to proactive support

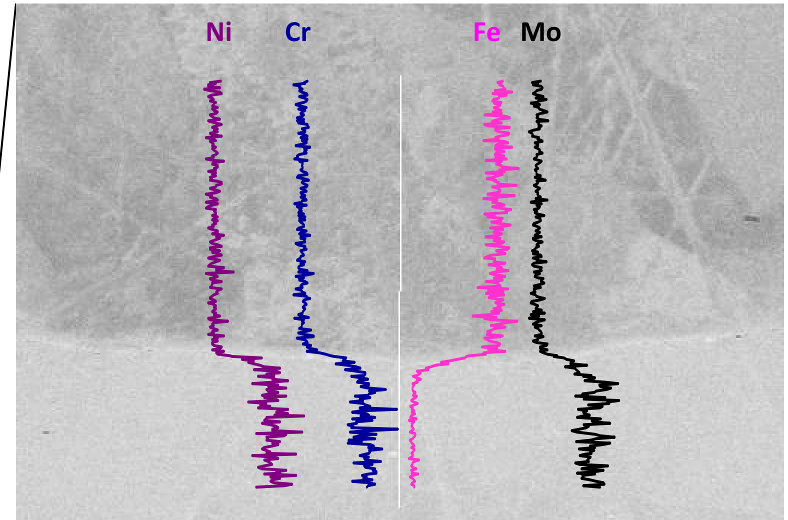
Failure analysis



Experimental creep test and modeling

Analysis of Steel Clad Piping

- Analyzed stainless steel clad piping for German company, Wilh. Schulz GMBH, manufacturing plant under construction in Tunica, MS.
- Chemical analysis via scanning electron microscope was employed to characterize the interlayer of SS clad piping for the purpose of manufacturing process design.
- Compression delamination tests were performed to analyze the interlayer performance of the cladding for improvements to manufacturing.

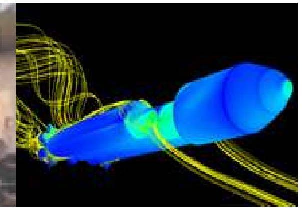


Chemical analysis of steel cladding

Some of CAVS's Capabilities…

NSF Engineering Research Center *for Computational Field Simulation* 1990-2001

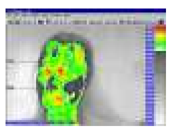
Outreach to Manufacturers



Center for Advanced Vehicular Systems

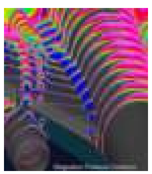
Vehicle Propulsion Systems

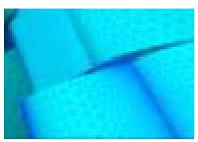
Digital Human and Cognitive Modeling



- Semiconductor Devices
- Motion Capture & Virtual Reality (VR)
- Industrial Ergonomics
- Mechanical Testing
- Materials Characterization
- 6-DOF Motion Base Driving Simulator
- Blast wave simulations

Computational Fluid Dynamics





Fine-Scale Capability

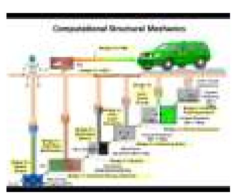
3D Xray Tomography

High Resolution In-situ FEG-SEM

Partnering with Industry

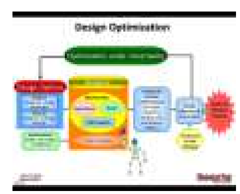
MEP.ms Manufacturing Extension Partnership of Mississippi

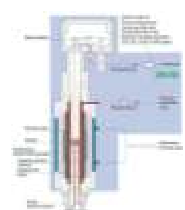
Computational Structural Mechanics



Metamodeling-based Optimization

Design Optimization





Thermal Analysis System

Driving Simulators

Site-Specific Analytical TEM Study on the Inhibition Layer Formation and Galvanneal Growth of Advanced High Strength Steels

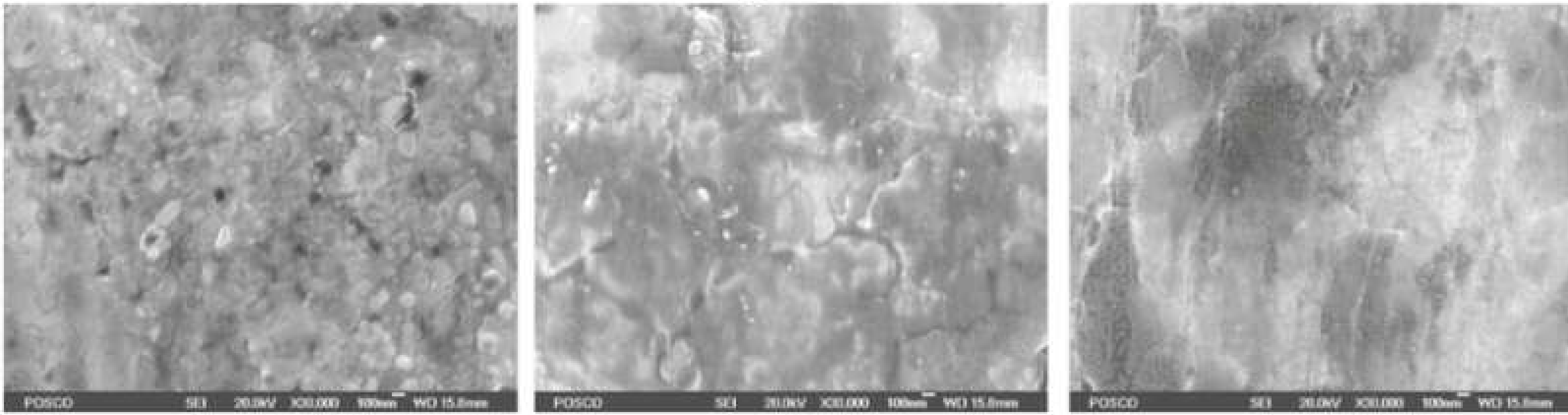
Problem Statement

- Advanced high strength steels (AHSSs), e.g. DP, CP, TRIP and TWIP steels, have increasing applications in automotive industry. Galvanizing and galvanneal of AHSSs are affected by the addition of alloying elements, e.g. Mn and Si. During annealing prior to galvanizing, diffusion of these alloying elements may lead to selective oxidation that affects the formation of the inhibition layer.
- The variability of chemical compositions near surface may affect the kinetics of the formation of the inhibition layer, for instance, the uptake of aluminum in the zinc bath, and may also change the chemistry of the inhibition layer.

Dew point of annealing affects surface oxidation prior to galvanizing

1180TRIP

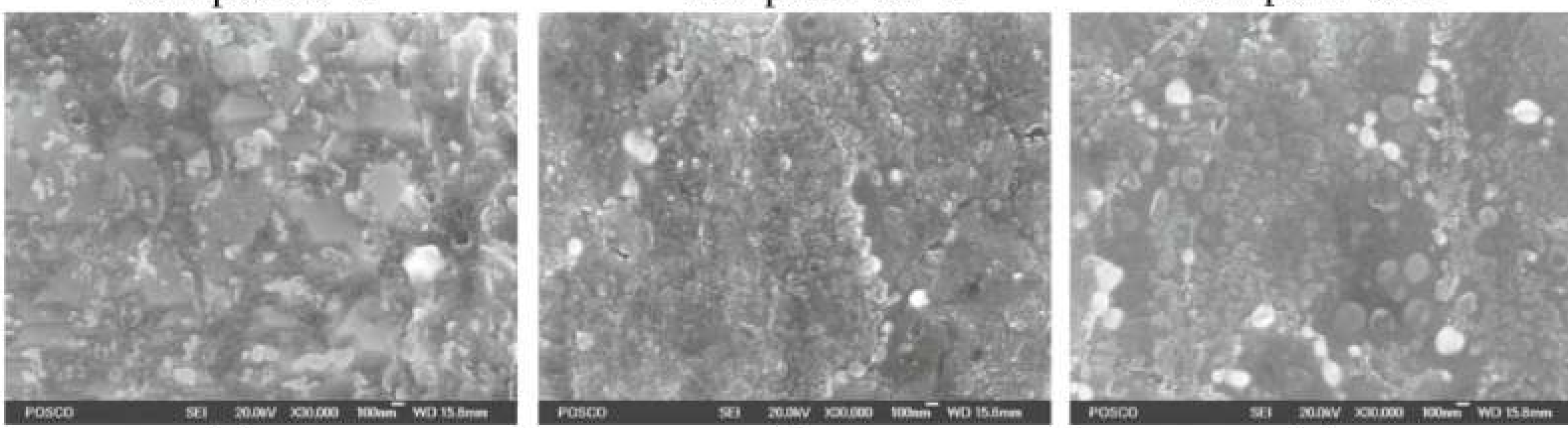
dew point 0°C | dew point -35°C | dew point -65°C



(a) (b) (c)

1180CP

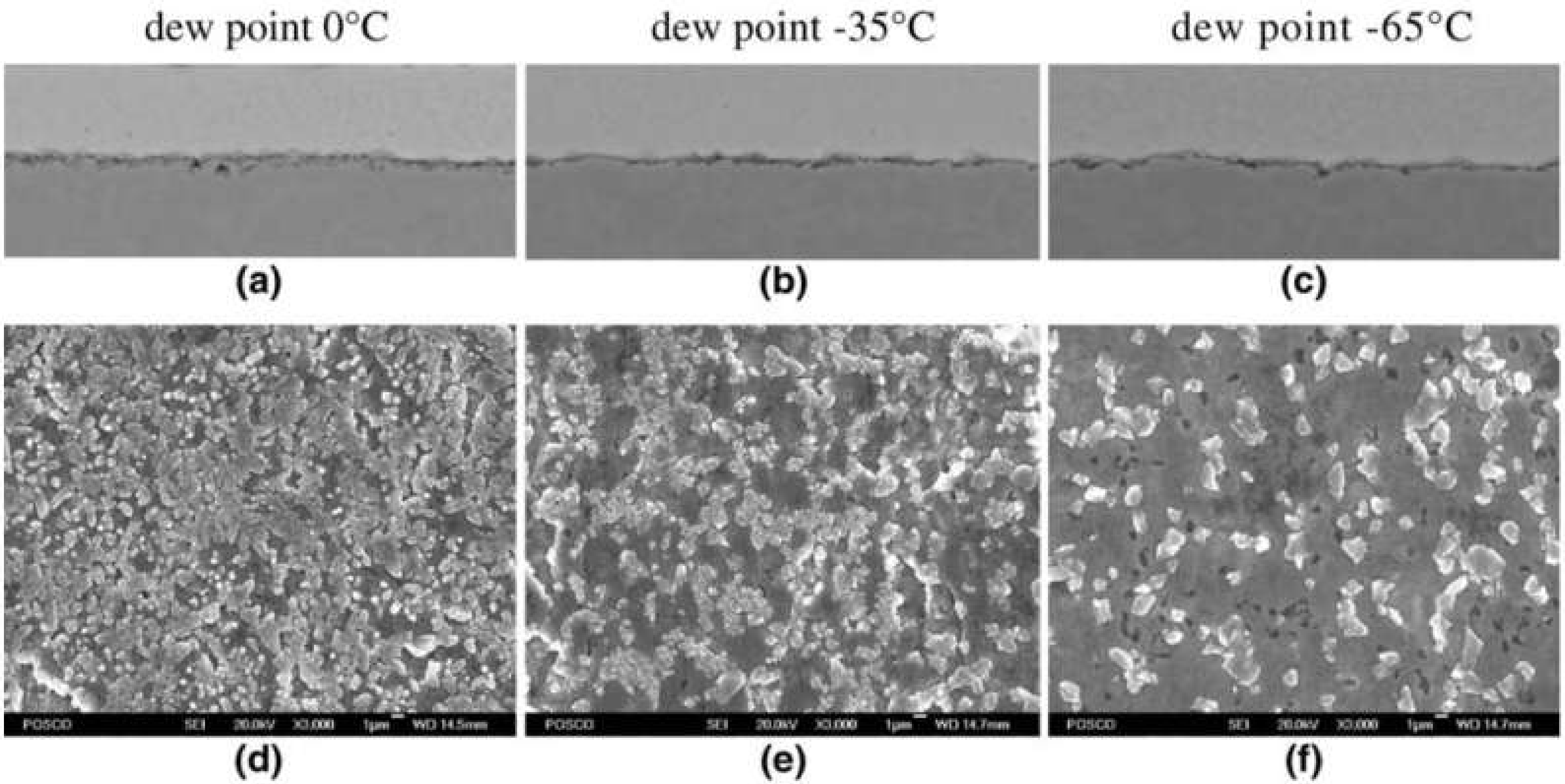
dew point 0° C | dew point -35° C | dew point -65°C



(a) (b) (c)

(M.S. Kim et al, Met Trans A 2009)

Dew point of annealing affects formation of inhibition layer



Discontinuous inhibition layer on 1180TRIP at various dew points. (M.S. Kim et al, Met Trans A 2009)

Types of oxide at various dew points on TRIP and CP

	0°C	-35°C	-65°C
1180TRIP	Mn_2SiO_4 + $MnSiO_3$	SiO_2 appears	Increasing SiO_2
1180CP	Significant MnO	Mn_2SiO_4 + MnO	Mn_2SiO_4

(M.S. Kim et al, Met Trans A 2009)

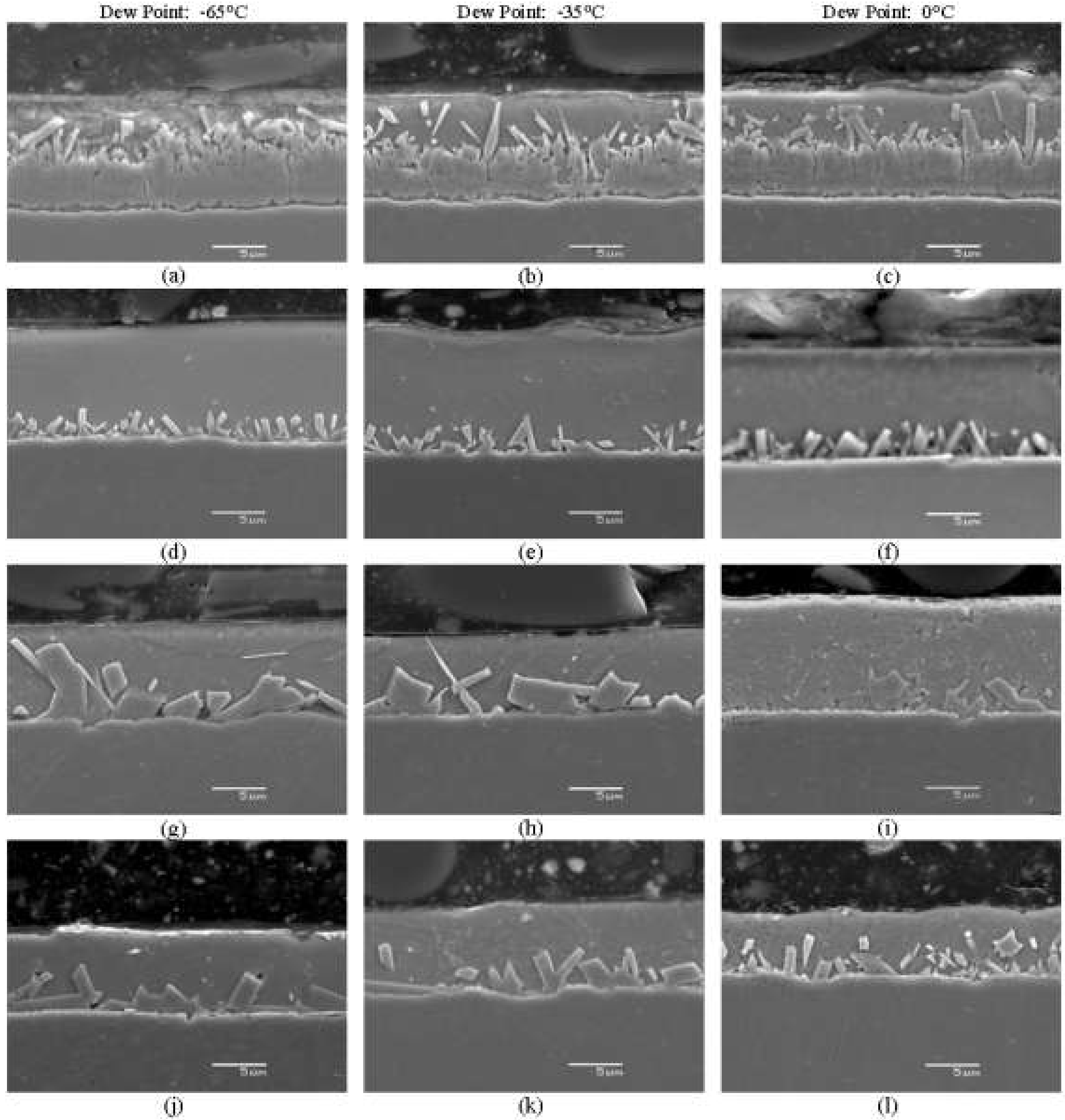
Dew point and compositional effects on formation of inhibition layer

EDDQ
(As hot-dipped)

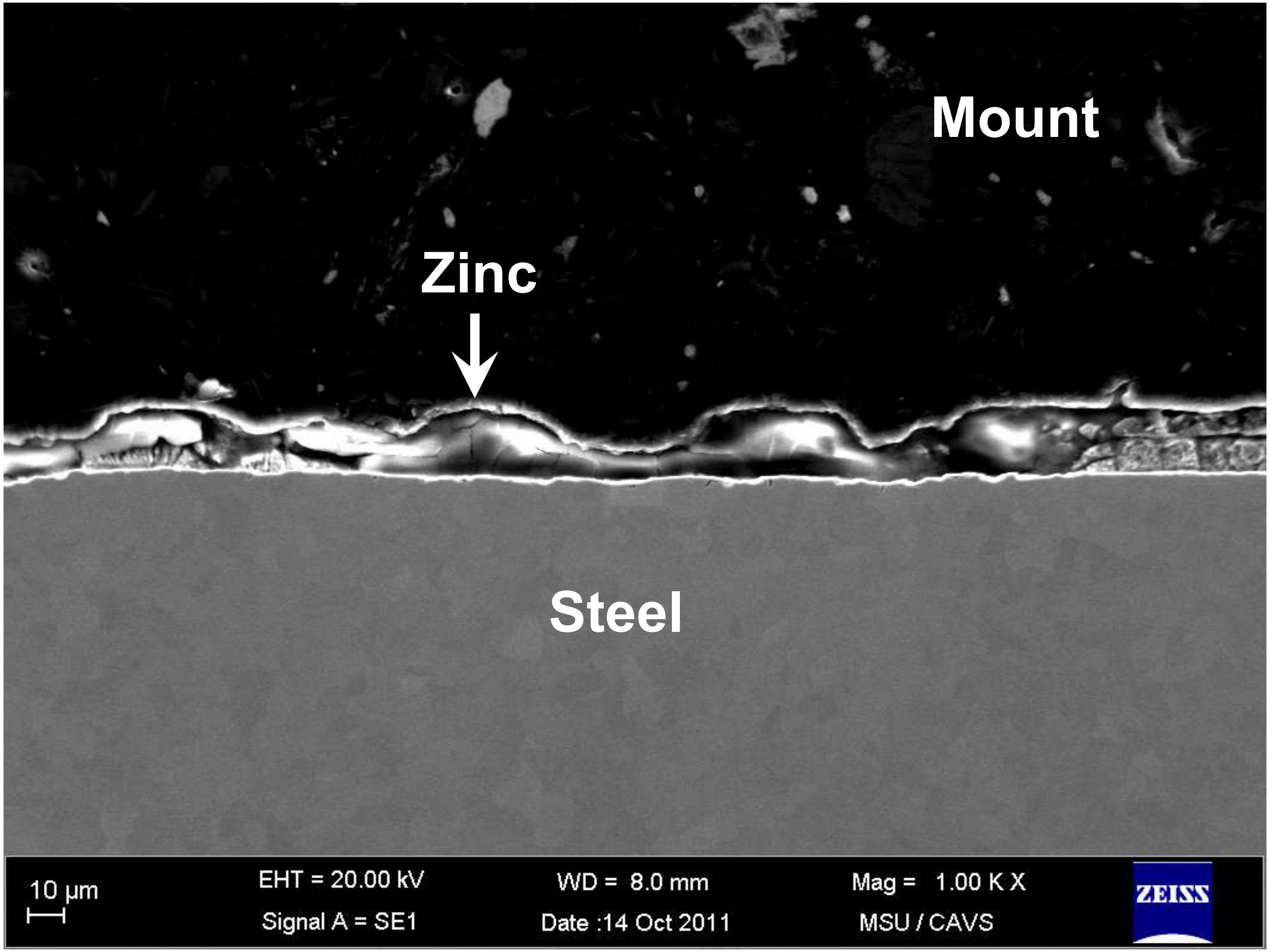
1180CP
(As hot-dipped)

590TRIP
(As hot-dipped)

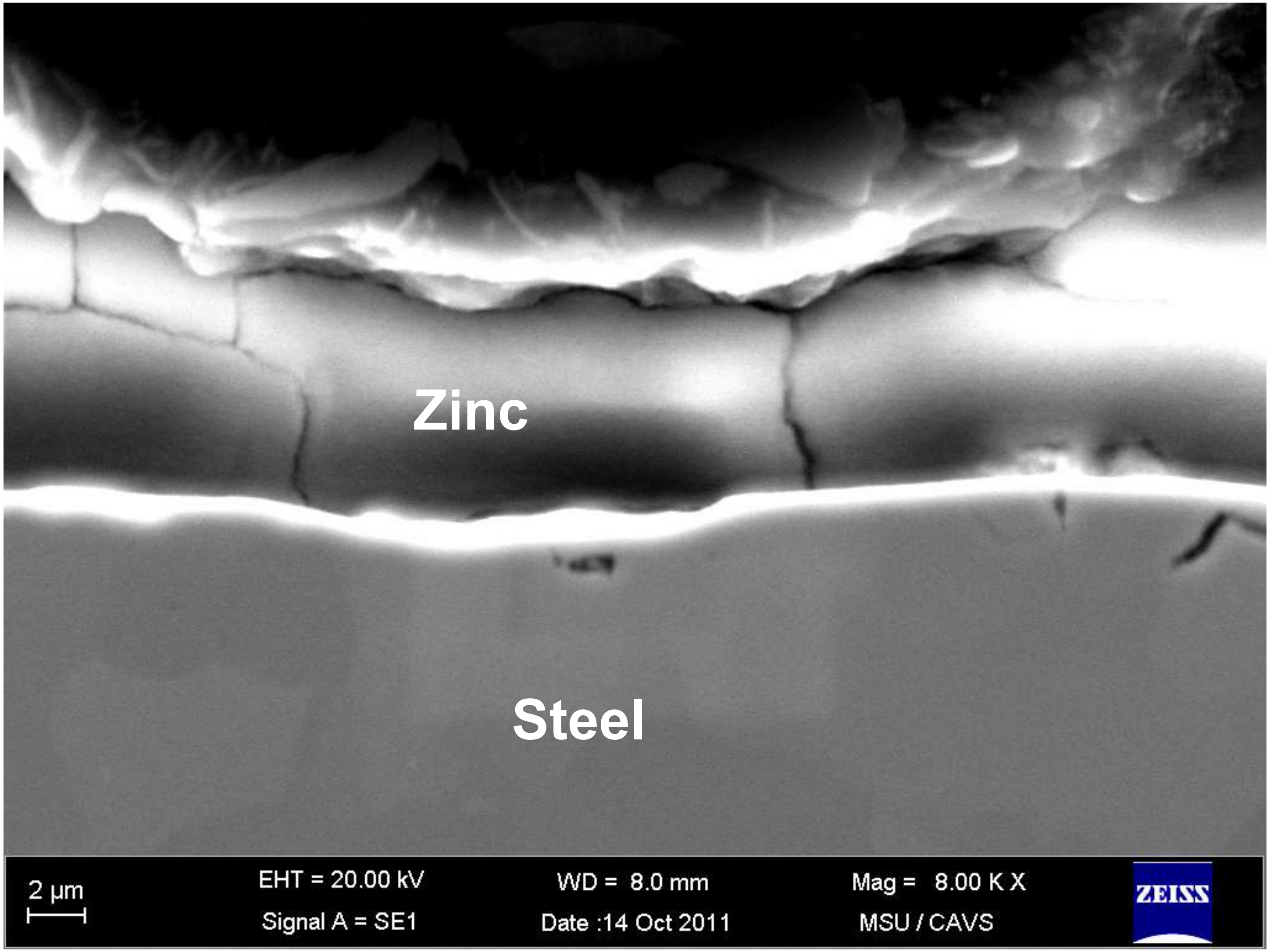
1180TRIP
(As hot-dipped)



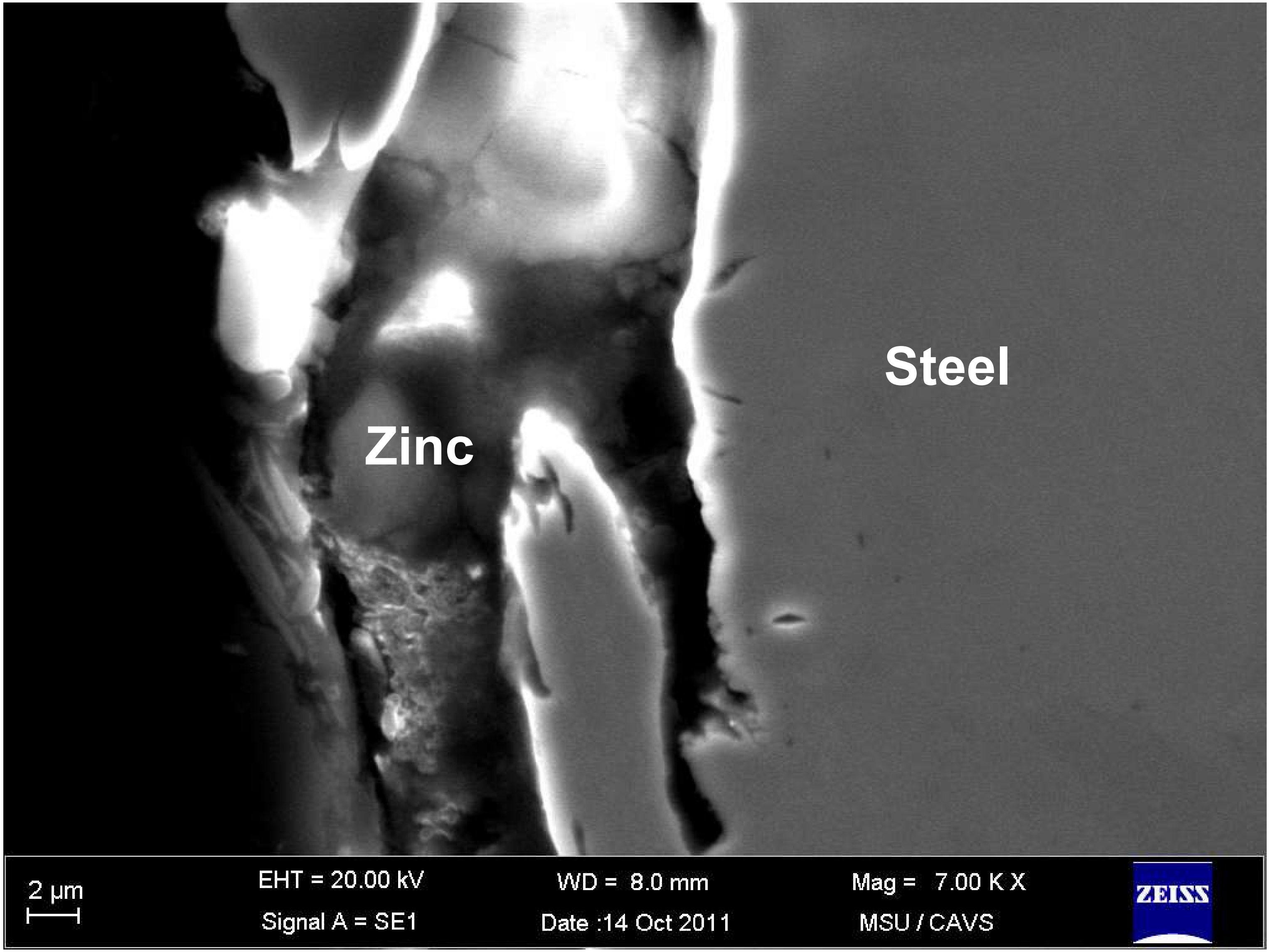
(M.S. Kim et al, The Asia-Pacific Galvanizing Conference 2009)



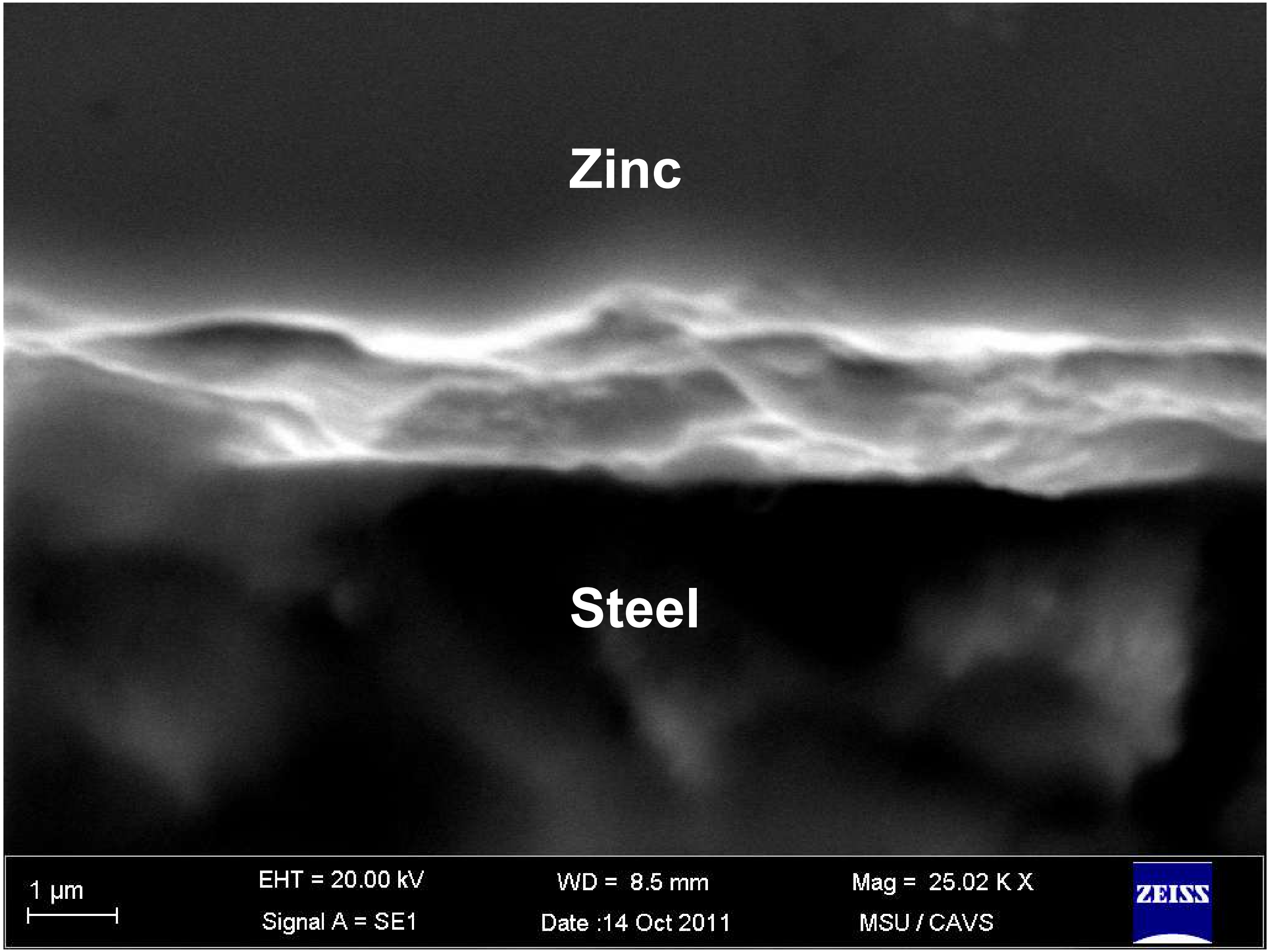
GI sample from Severstal



GI sample from Severstal

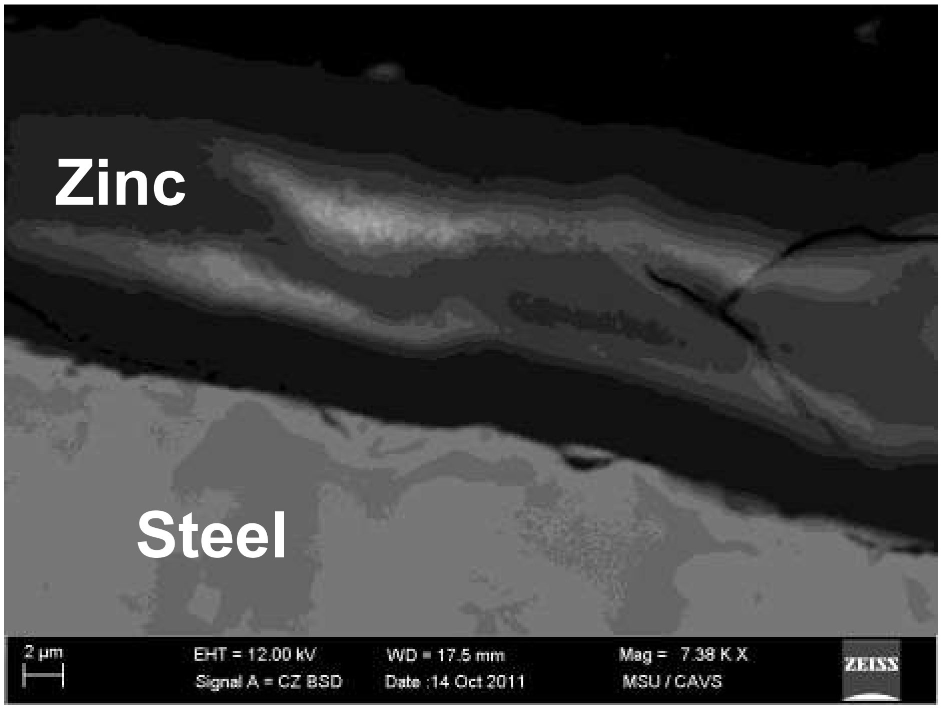


GI sample from Severstal

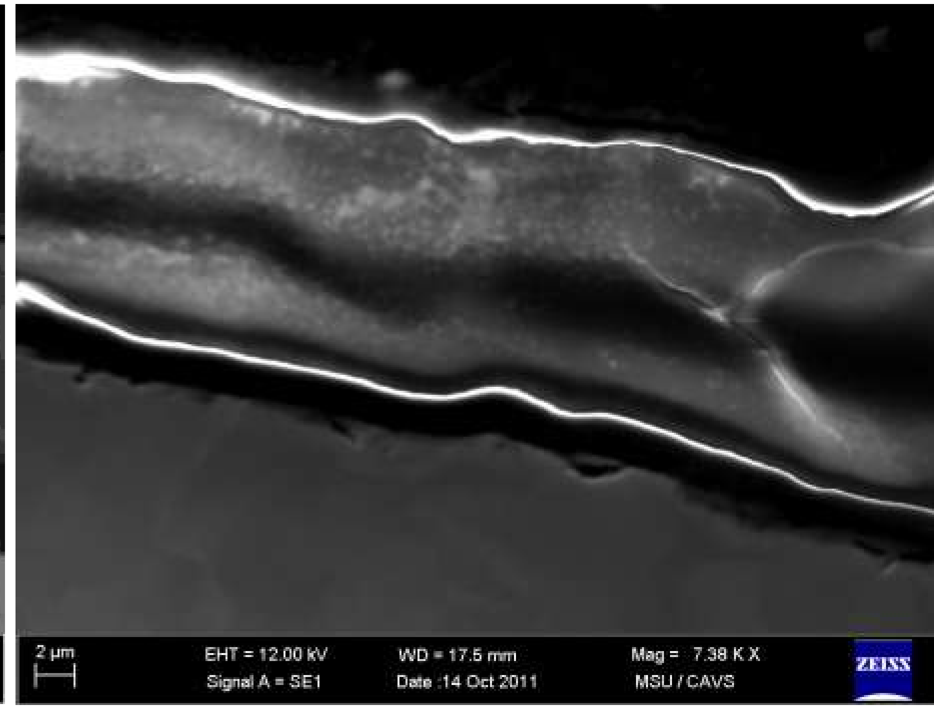


GI sample from Severstal

Backscattered Electron Image



Secondary Electron Image



GI sample from Severstal

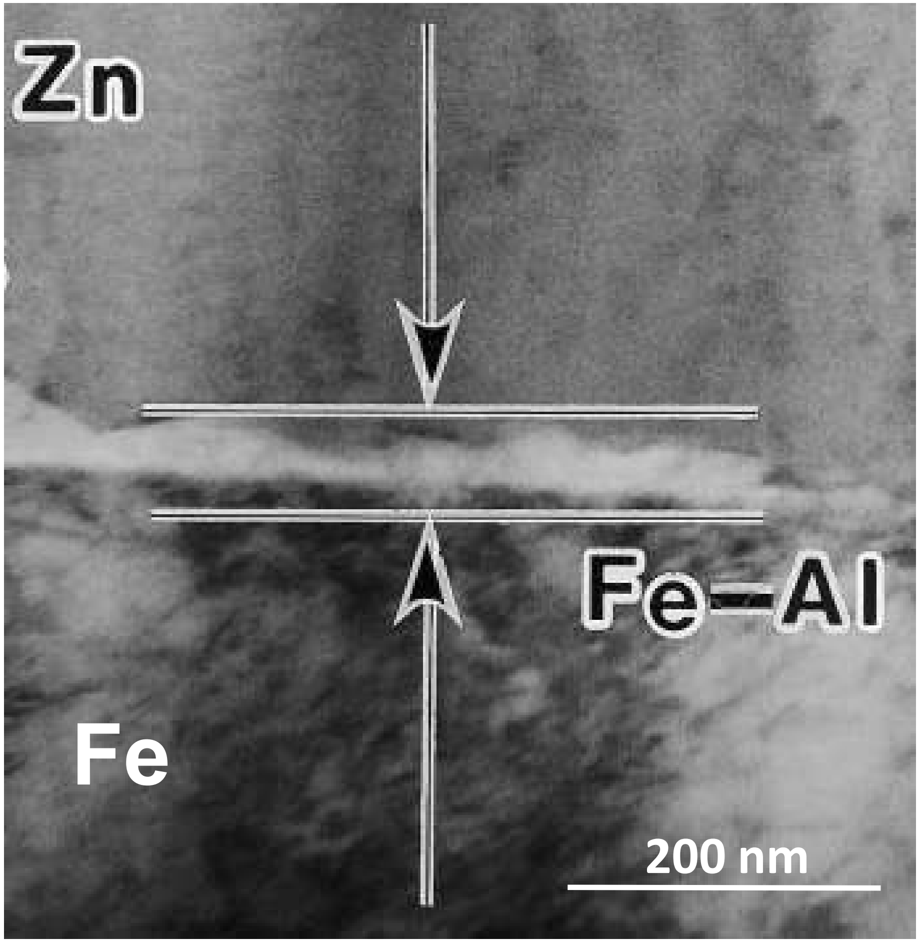
Unknowns and Uncertainties

- Controlling mechanisms of formation of the inhibition layer and the intermetallic phases;
- Reactivities at the interfaces;
- Redistribution of alloying elements such as Mn and its effect on the formation of the inhibition layer;
- Scanning electron microscopy (SEM) analysis may be inconclusive due to the selective polishing and etching during sample preparation.

Objectives

- Study formation kinetics of the inhibition layer under various conditions, i.e., dew points and local chemistry in AHSSs of different grades. Galvanneal growth will be studied as well. Solute mapping will be conducted in the atomic scale near the interface;
- Apply site-specific technique (Focused Ion Beam – FIB) to the preparation of samples that contain Fe-Zn interface and are electron transparent for microstructural and chemical composition analysis;
- Apply advanced TEM and STEM to the study of how surface and subsurface characteristics (microstructural and chemical) of AHSSs influence formation of the inhibition layer.

Formation of Inhibition Layer



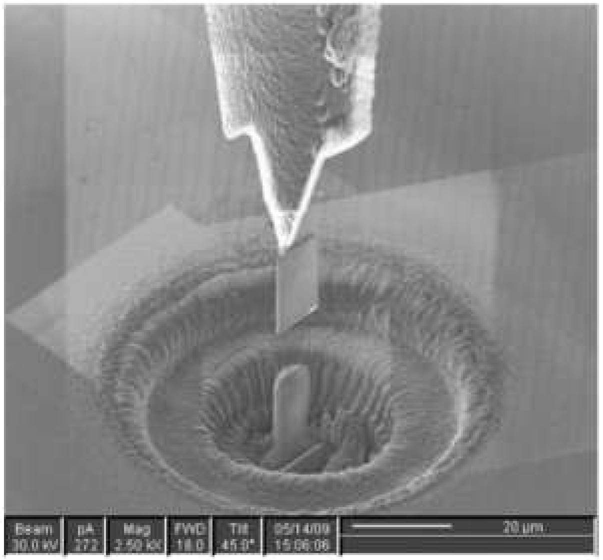
(Kato et al., Acta Mater. 2000)

Because aluminum has a greater affinity for iron than Zn, immediately after the steel enters the coating bath, a layer of stable intermetallic compound forms at the interface. This layer is known as the inhibition layer (Fe_2Al_5) which is extremely thin and retards the Zn-Fe reaction that forms a thick, brittle intermetallic layer.

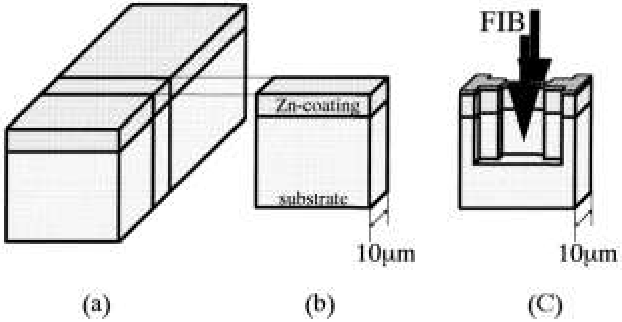
The formation kinetics of the inhibition layer is affected by alloying elements of steels that incur selective oxidation on the surface of the substrate.

Methodology

- **Site-specific technique – Focused Ion Beam, which allows to pinpoint and prepare TEM specimens from specific areas of interest in materials. The specimen thickness can be on the order of a few tens of nanometers.**
- **Transmission electron microscopy (TEM), high resolution TEM (HRTEM), and Z-contrast scanning TEM (STEM) studies on the microstructure and chemistry near the interface.**

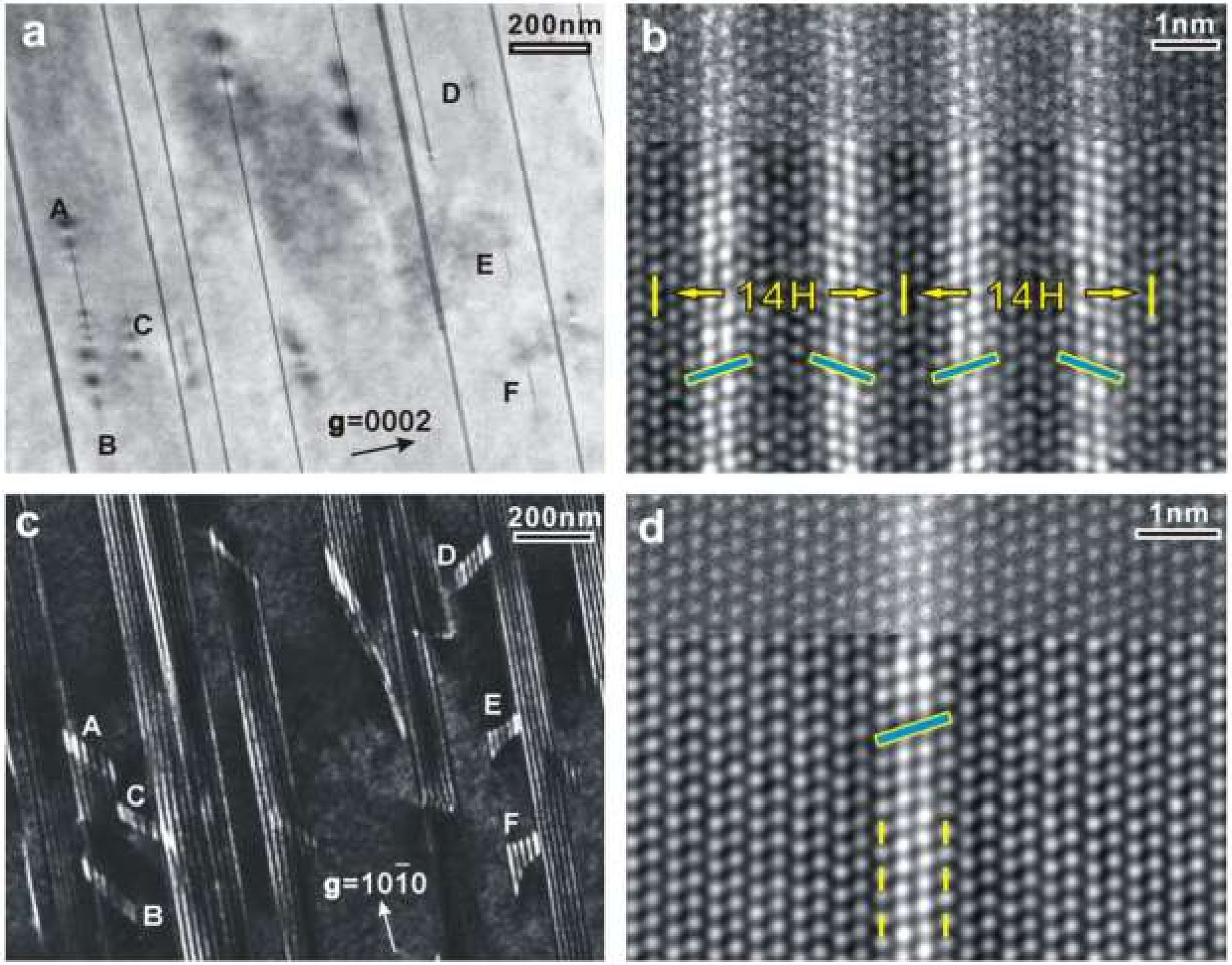


(B. Li et al., Scripta Mater. 2010)



(Kato et al., Acta Mater. 2000)

HAADF-STEM Imaging



(Nie et al. Acta Mater 58, 2010)

Experiment Design

- Three steel grades: IF, DP, TRIP;
- Three dew points: 0°C, -35°C, -65°C;
- Coating: GI (<0.2wt%Al) for study of kinetics of inhibition layer formation;

  GA (<0.13wt%Al) without galvanneal for study of kinetics of inhibition layer formation; and with galvanneal for study of overlay growth.

Collaborative Researches

- **CAVS will actively collaborate with steelmakers and seek technical assistance from the steelmakers for samples and lab scale experimental design.**
- **CAVS will team up with Oak Ridge National Laboratory (ORNL) / Johns Hopkins University (JHU) and use their cutting-edge imaging facilities to conduct the site-specific studies on formation of inhibition layer during hot-dip and growth of overlay during galvanneal.**

Research Plans

- A Ph.D. student will be involved;
- Obtain galvanized and galvannealed AHSS samples from steelmakers;
- Metallography and SEM analysis;
- TEM sample preparation by FIB;
- Perform TEM, HRTEM, and STEM analysis;
- Reports and journal publications.

Thank You!!!