

Materials Characterization Capabilities at the High Temperature Materials Laboratory: Focus on Lightweight Materials

DOE 2011 Vehicle Technologies Annual Merit Review and Peer Evaluation Meeting

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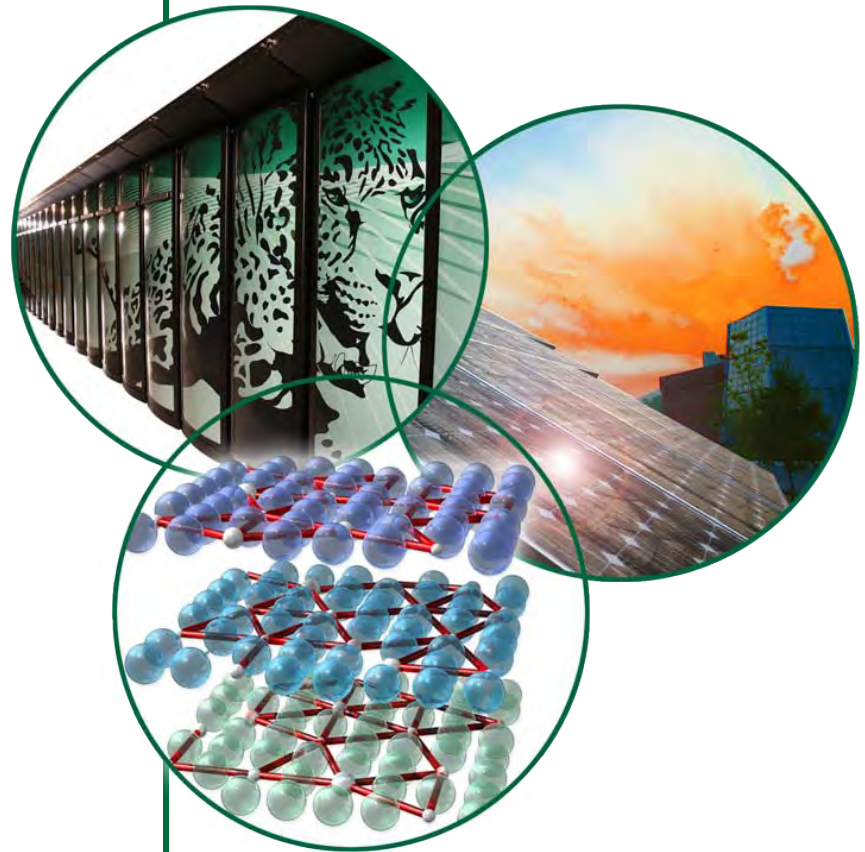
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Project ID: LM039



The HTML User Program: Background

- The High Temperature Materials Laboratory is a National User Facility that supports the missions of DOE, EERE and the Vehicle Technologies Program in particular, by working with industry, universities, and other national laboratories to develop energy-efficient technologies that will enable the U.S. to use less petroleum. The HTML is organized into six user centers, which are clusters of highly skilled staff and sophisticated, often one-of-a-kind instruments for materials characterization.
- Access to the HTML User Program is provided through the HTML User Program proposal process. Research proposals are reviewed by a committee and approved based on scientific merit, relevance of the proposed research to the mission of DOE's Vehicle Technologies Program, feasibility, and non-competition with the private sector. Projects have a well-defined scope, and research is completed within 24 months and often involves multiple user visits to the HTML.
- Both nonproprietary and proprietary research is conducted within the HTML User Program. There are generally no charges for nonproprietary research projects, and users conducting nonproprietary research must agree to submit research results for publication in the open, refereed literature. A nonproprietary project is complete when research ends, accompanied by the required publication in the open literature and/or presentation at a professional conference. For proprietary research, the user owns the research data, and all costs at the HTML are paid by the user based on DOE guidelines for ORNL cost recovery.

The HTML User Program – FY2010 Activity

During FY2010, the HTML User Program collaborated with 18 companies, 25 universities, and 6 national laboratories on 68 user projects addressing critical technical barriers to achieving the goals of DOE's Vehicle Technologies Program. There were 96 researchers, 63% of them first-time users, who visited the HTML for a total of 716 research days.

The HTML User Program FY2010 budget was \$5,312,400 and allocated as follows:

- Capital equipment: \$881,959
- Operations: \$4,430,441

Users cost-share their HTML user projects through:

- 1) direct involvement with HTML staff members during the development of the user project;
- 2) funding their time and travel to the HTML to perform research;
- 3) cost of materials provided by the user or the research performed prior to the user project;
- 4) collaboration with HTML staff members to analyze the data and publish the results.

The HTML also supports the education and preparation of the next generation of scientists and engineers. During FY2010, students and professors from 25 universities participated in the HTML User Program. Five of those students earned their Ph.D. degree and one earned her M.S. degree based in part on research they conducted through the HTML User Program.

Relevance to the VT Program

- The Vehicle Technologies Program funds the operation of the HTML User Program to maintain world-class expertise and instrumentation capabilities for materials characterization to work with industry, universities and national laboratories toward the goals of the Vehicle Technologies Program. The HTML User Program capabilities at the Oak Ridge National Laboratory support the activities of the Vehicle Technologies Program's subprograms in Lightweight Materials, Propulsion Materials, Energy Storage, Solid State Energy Conversion, Combustion & Emissions Controls, Power Electronics & Electric Motors, and Non-Petroleum Fuels.
- During FY2010, the HTML User Program managed **17** characterization projects in **Lightweight Materials**, and this poster presentation highlights **one** of them. The **Magnesium Elektron North America** user project in this presentation is focused on the feasibility of developing industrially viable processes for producing shear rolled sheet. In particular, this project addresses technical barriers related to manufacturability and also adds to knowledge of the performance characteristics of advanced magnesium alloys.

Magnesium Elektron NA

User Project: Milestones



- One of the FY2011 milestones for the HTML User Program is to complete three user projects dealing with the characterization of lightweight and lightweighting materials by September 30, 2011.

Project ID	Organization	Project	Status
2010-009	The Timken Company	Surface/Sub-surface dislocation density analysis of flow forming using transmission electron microscopy	due Jan 2012
2010-018	Atrix Components Inc.	Characterizing heavy vehicle compressor components: Mg and Al castings and Al-MMC cylinder liner interface	Completed ✓
2010-027	Virginia Commonwealth University	Characterization of lightweight materials for automotive applications	Completed ✓
2010-028	University of Alabama-Birmingham	Effect of chemistry on the transformation characteristics of metastable austenite in intercritically austempered ductile iron for automotive applications	Completed ✓

This presentation reviews one of 17 HTML User Program projects active in FY2010 that focused on the characterization of lightweight materials.

Project ID	Organization	Project	Due Date
2010-040	Magnesium Elektron	Magnesium Forming Limit Diagrams	July 2012

Magnesium Elektron NA User Project: "Magnesium Forming Limit Diagrams"



Timeline

- Start date: July 2010
- End date: July 2012
- % complete: 33%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as noted on slide #3.

Barriers

- Cost and lack of alternative forming processes
- Lack of knowledge due to lack of data on crystallographic texture and formability
- Manufacturability issue of poor formability at low temperatures

Collaborators

- **Users:** Magnesium Elektron NA
David Randman
- **HTML Staff:** Thomas Watkins,
Edgar Lara-Curzio
- Materials Processing Group, ORNL
- Industrial Technologies Program, EERE

Project relevance to VT Goals

- Successful large-scale application of the knowledge gained through this characterization project will directly address the cost of magnesium alloys and enable the U.S. automotive industry to achieve improved energy efficiency through wide-spread use of these alloys.
- Specifically, this project supports efforts to reach the Vehicle Technologies Key Goal of achieving a cost-effective 50% weight reduction in passenger vehicle body and chassis systems by the year 2015.

Magnesium Elektron NA User Project: "Magnesium Forming Limit Diagrams"



Collaborations with other organizations

Through a project funded by DOE's Industrial Technologies Program, ORNL and Magnesium Elektron are collaborating on implementing industrial-scale shear rolling. The characterization research conducted through the HTML User Program supports a research-scale mill developed by ORNL's Materials Processing research group. Data gathered during the HTML characterization studies will be included in the operations plan for a new Magnesium Elektron mill to be scaled up in the Summer of 2011.



David Randman



Thomas Watkins, Ed Kenik, Don Erdman,
Burl Cavin, Edgar Lara-Curzio



Murali Muralidharan, Tom Muth, Bill Peter



CPS Project Number: 20740
CPS Agreement Number: 20913

Collaborations with other organizations: ITP Shear rolling scale-up

- Project Tasks
 - Task 1: Design and installation of laboratory scale shear mill
 - Task 2: Alloy selection
 - HTML User Program Support
 - Task 3: Process development for shear rolling
 - HTML User Program Support
 - Task 4: Demonstrate shear rolling in industrial environment
 - Task 5: Fabrication of components
- This project complements the work in the ITP project as well as addressing VT goals.

Magnesium Elektron NA

User Project: "Magnesium Forming Limit Diagrams"



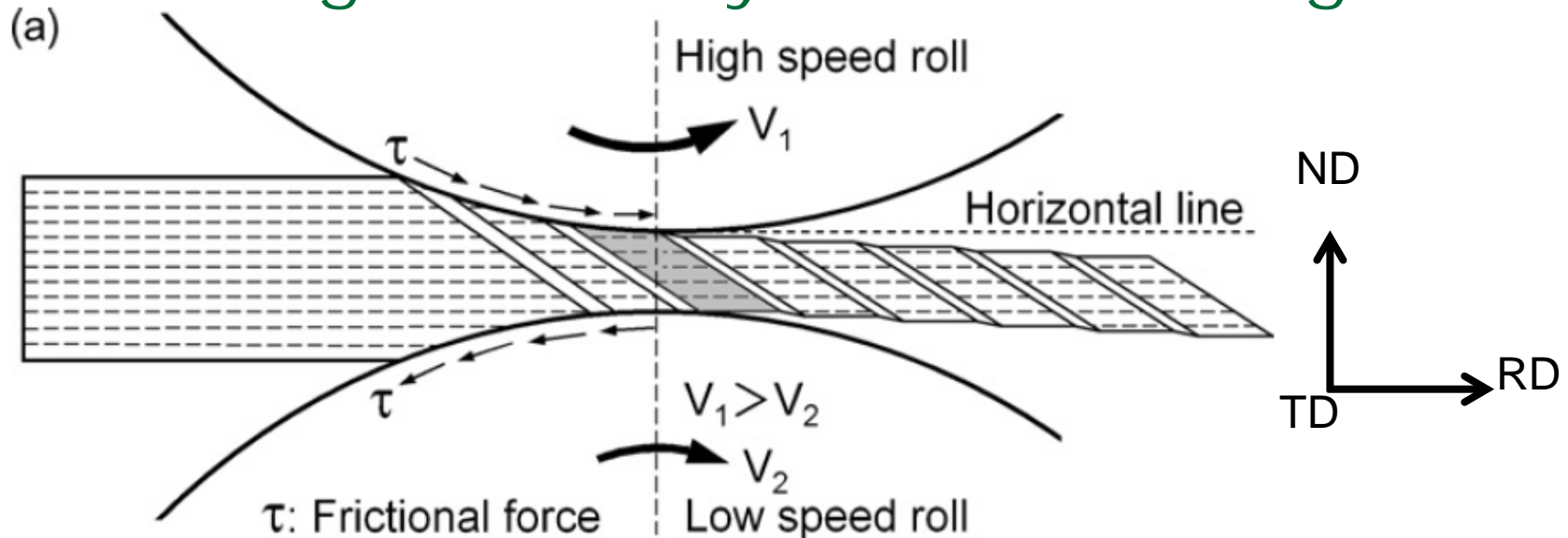
Background

- Adequate design data (material property databases), test methods, analytical tools (i.e., models), and durability data remain inadequate for widespread applications of magnesium alloys in vehicular structures.
- Methods for cost-competitive, high-volume production of automotive components from advanced lightweight materials do not exist.
- Lack of formability at low temperatures or the need for high forming temperatures is one of the factors that contributes to the high cost of magnesium alloys.
- Predominant basal texture in magnesium alloys is an important factor limiting the formability of magnesium alloys.
- Asymmetric rolling has been successful in modifying the basal texture.

Magnesium Elektron NA

User Project: "Magnesium Forming Limit Diagrams"

Background: Asymmetric rolling



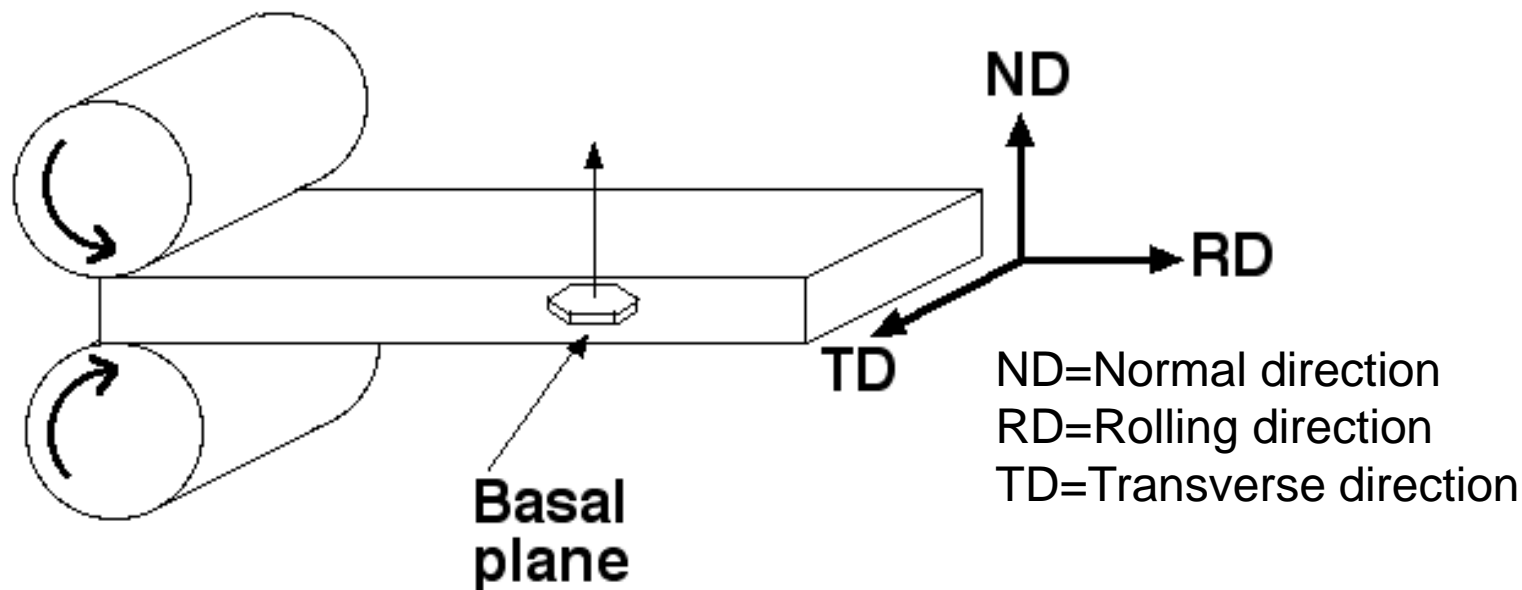
Huang et al. *J. Alloys and Compounds* 2008 **457** 408-412

- Different speed rolls
- Introduces shear deformation into the material
- Can refine grain size and alter through thickness texture
- Sheets are found to have better formability

Magnesium Elektron NA

User Project: "Magnesium Forming Limit Diagrams"

Background: Conventional symmetric rolling causes slip which orients basal plane normals parallel to sheet normal direction



Asymmetric rolling can tilt the basal planes out of the RD-TD plane. This allows slip out of the RD-TD plane which increases formability or manufacturability.

1 Approach: Use advanced characterization tools

- Characterize the macroscopic crystallographic texture of magnesium alloys after asymmetric rolling using X-ray diffraction (XRD).
- Characterize the microstructure and microscopic crystallographic texture of magnesium alloys using electron back-scattered diffraction (EBSD).
- Create forming limit diagrams for the selected alloys and rolling conditions.
- The unique combination of advanced equipment and expertise in one location positions the HTML User Program to help Magnesium Elektron identify the optimum shear rolling processing parameters for the magnesium alloys being investigated.

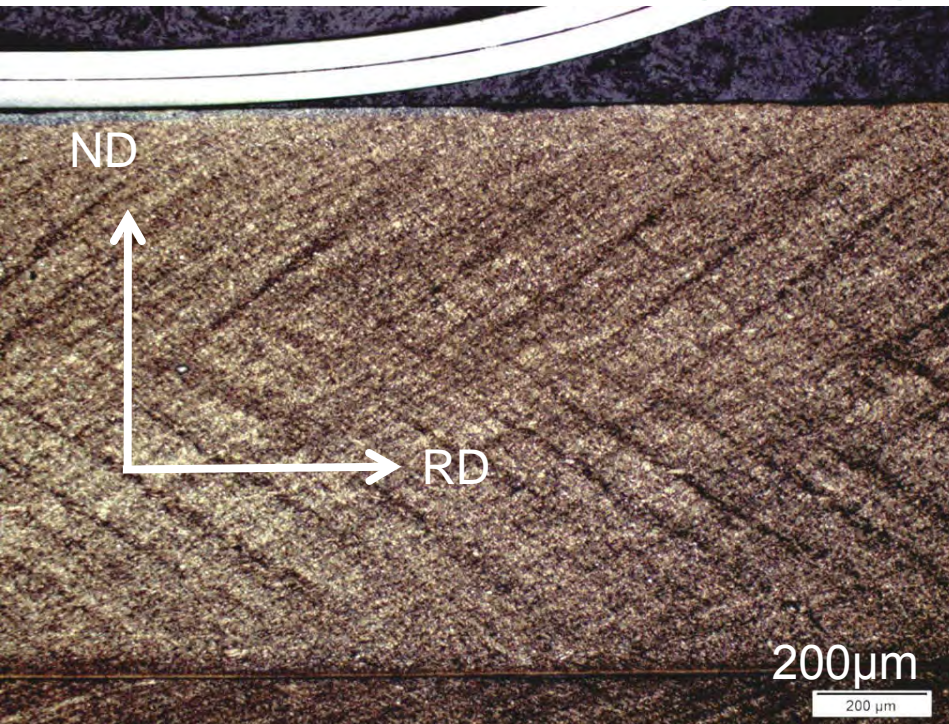
2 Approach: Current samples are pre-heated and then asymmetrically rolled with unheated rollers

- Conditions
 - Alloy
 - AZ31B (3 Al/ 1 Zn/ 0.2 Mn/ bal Mg; wt%)
 - ZEK100 (1 Zn/ 0.1 RE/ 0.1 Zr/ bal Mg; wt%)
 - Preheat temperature
 - % reduction amount
- New \$1.0M mill with heated rollers will be installed in the summer of 2011
 - New condition: Roller temperature

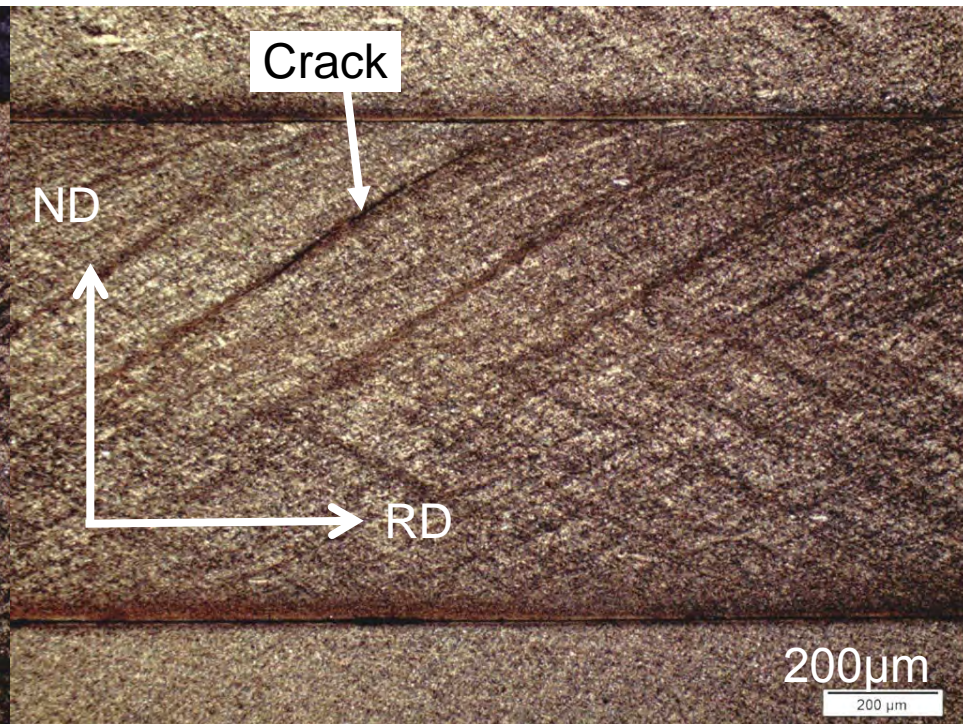
Magnesium Elektron NA User Project: "Magnesium Forming Limit Diagrams"



Accomplishments: Structural analysis revealed that multiple pass rolling of sheet at 180°C resulted in slip and cracking along shear bands.

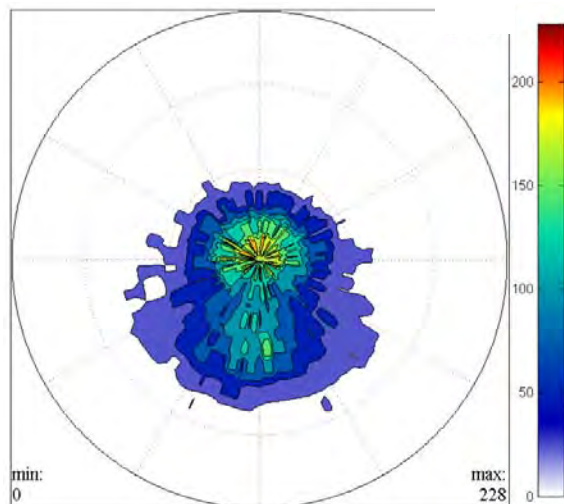


12% reduction

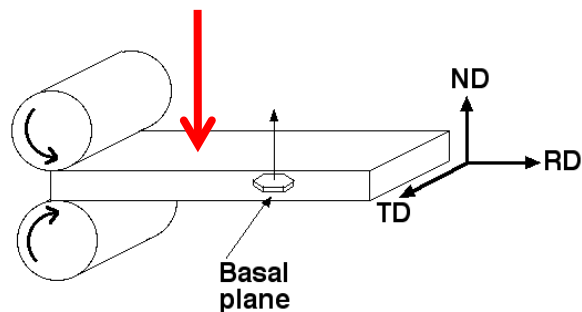


22% reduction

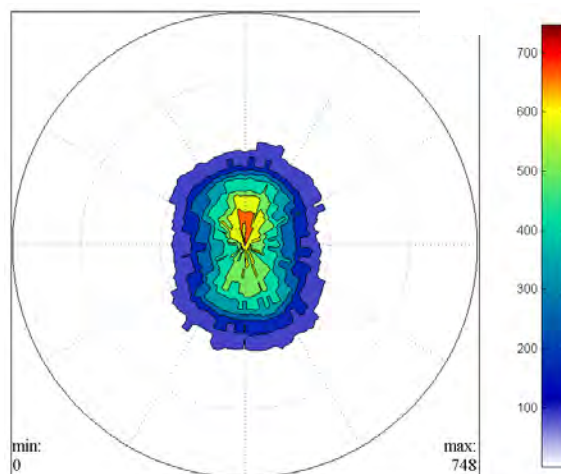
Accomplishments: Slip changes the crystallographic texture



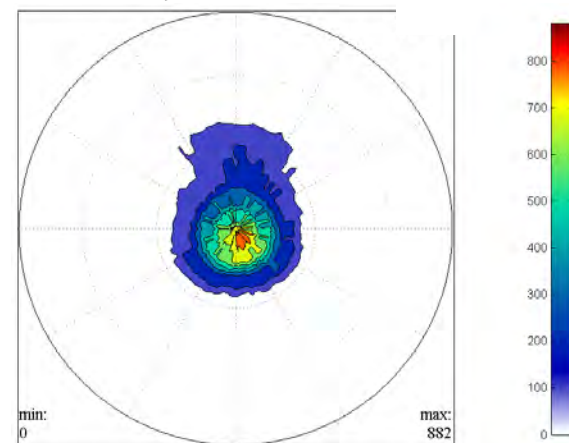
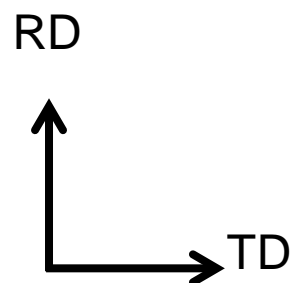
Fast Roll



Broad distribution of {0002} basal poles observed through the thickness in specimen with 22% reduction at 180°C

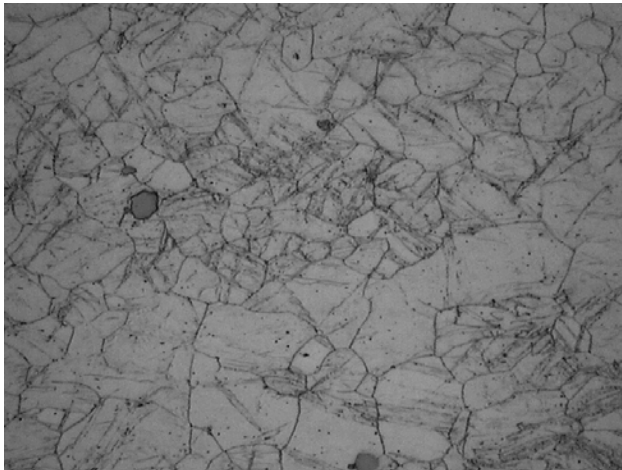


Center



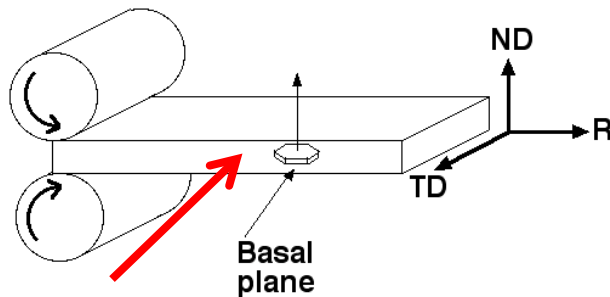
Slow Roll

Accomplishments:
Specimens rolled at preheat
of 225°C show evidence of
recrystallization



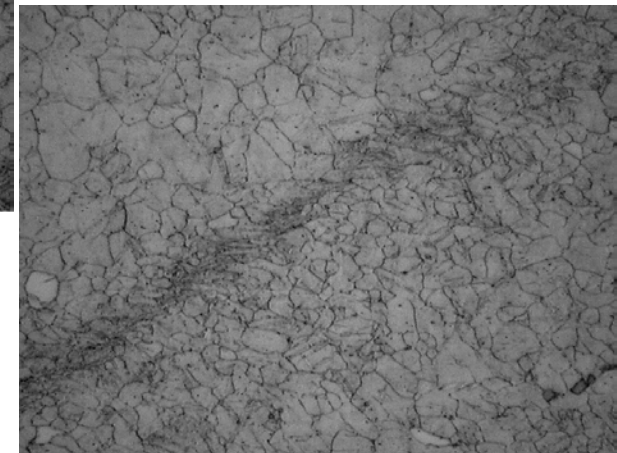
1000X 5μm
100 Ethanol/10H₂O/5 Acetic/5 Picric

14% at 135°C



1000X 5μm
100 Ethanol/10H₂O/5 Acetic/5 Picric

18% at 180°C



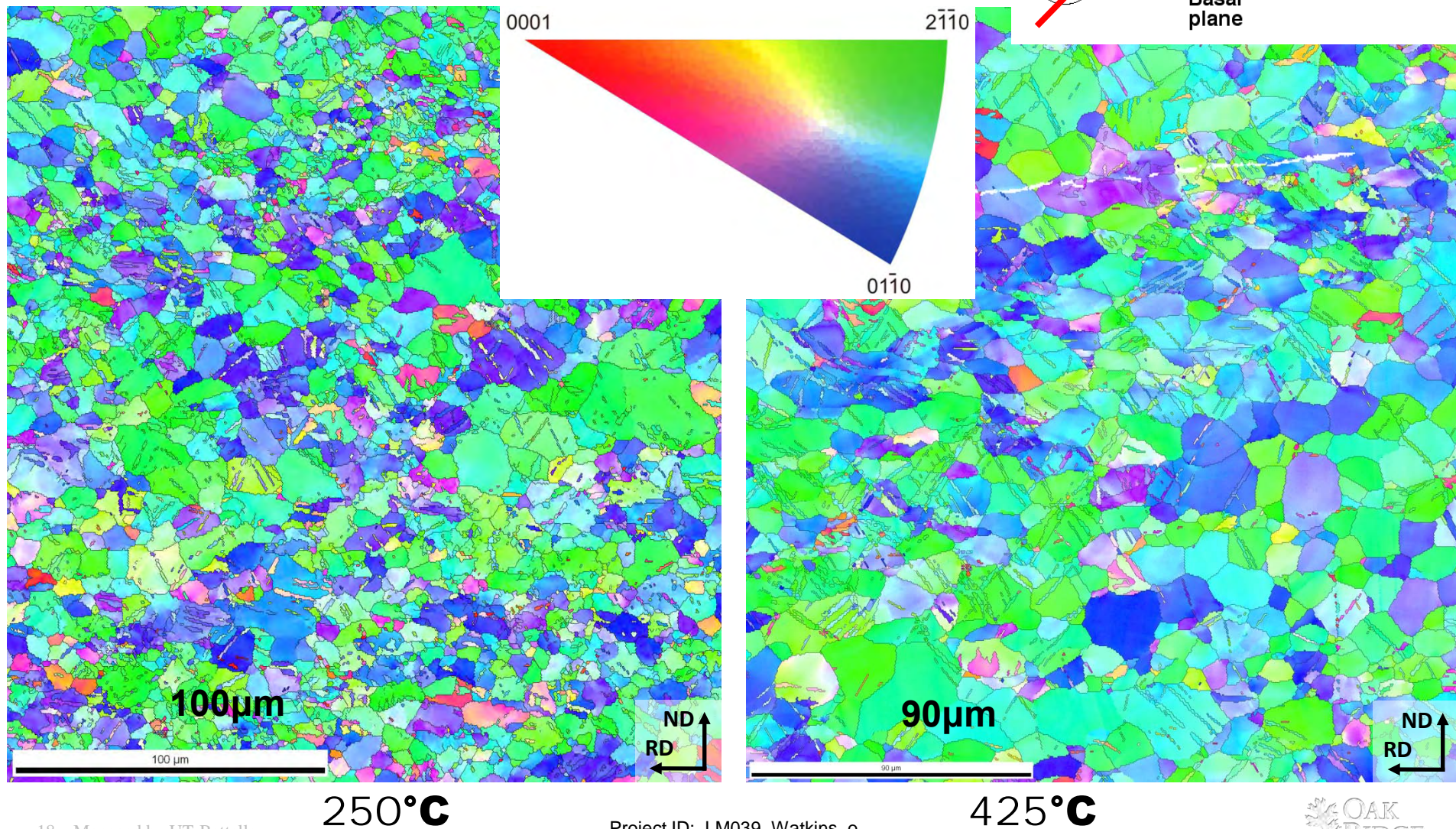
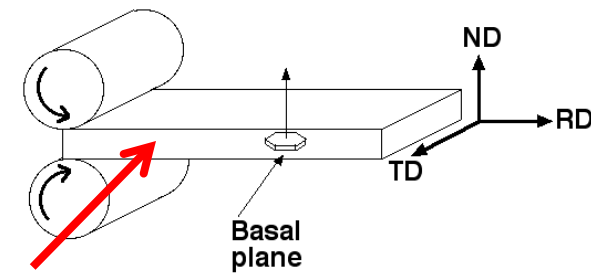
10-1579-11 2P19 AZ31B 35% @ 250°C 1000X 5μm
Arrow Left to Right Band 100 Ethanol/10H₂O/5 Acetic/5 Picric

35% at 225°C

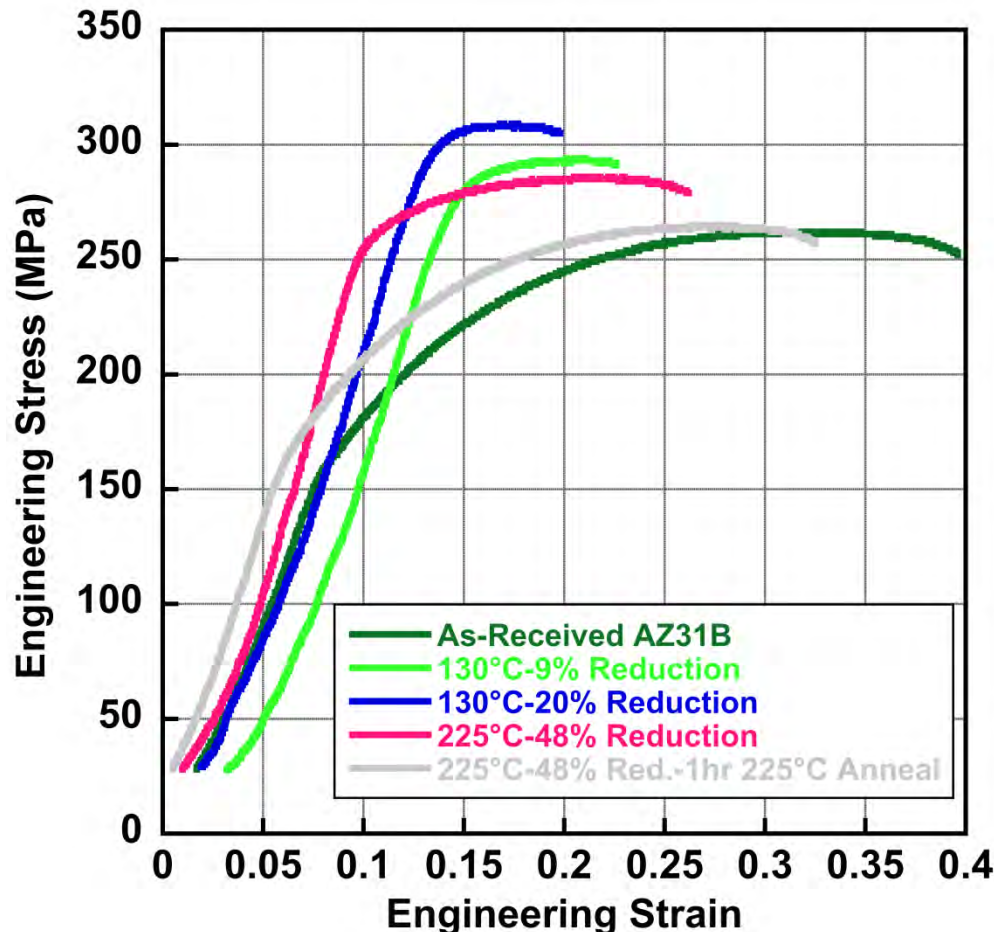
Recrystallization can produce a more random texture
that results in better manufacturability and reduces cost.

Magnesium Elektron NA User Project

Accomplishments: Individual grain orientations from EBSD consistent with XRD



Accomplishments: Uniaxial tensile data are not a good predictor of forming behavior.



- In a uniaxial tensile test, Poisson's contraction is allowed in the two perpendicular directions.
- Forming is biaxial in nature and Poisson's contraction only occurs in the through thickness direction.

Work is in progress to establish an experimental set-up to perform biaxial tests.

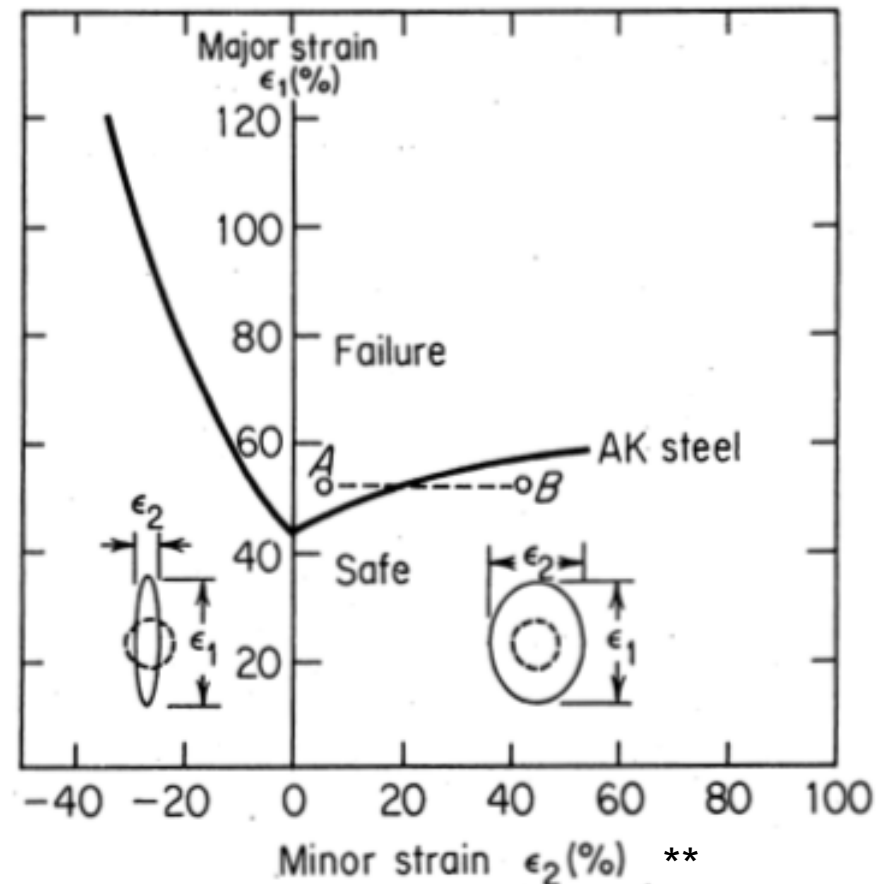
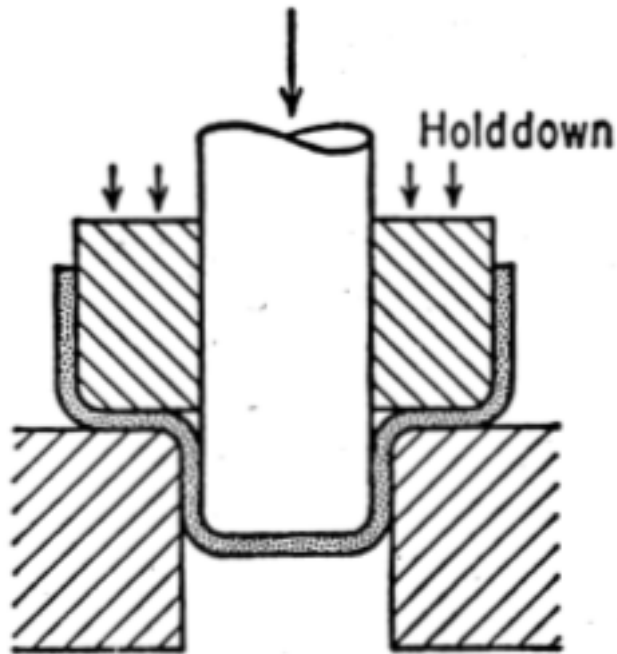
Magnesium Elektron NA User Project Summary



- Magnesium Elektron participated in the HTML User Program to characterize alloys AZ31B and ZEK100 processed by shear rolling under different preheat temperatures and reduction ratios, using a combination of characterization techniques (XRD, EBSD, SEM).
- Asymmetric rolling resulted in a weaker basal texture under all temperature conditions and rolling schedules
 - Basal={0002} poles were observed to be tilted along the RD but toward the trailing edge.
 - Rolling at lower temperatures resulted in cracking.
 - Rolling at low temperatures results in shear band and recrystallization; at higher temperatures, grain growth.
- Magnesium Elektron was able to take advantage of a wide array of materials characterization capabilities available to industry users through the HTML User Program, which maintains world-class expertise and unique instrumentation gathered in one convenient location. The results from this investigation are a fundamental component of the process to produce new materials or materials with improved properties.

Future Work

- Continue to characterize rolled samples with XRD and EBSD
- Complete fabrication of heated biaxial forming dome press for forming limit diagram (FLD); generate FLDs
- Technology transfer



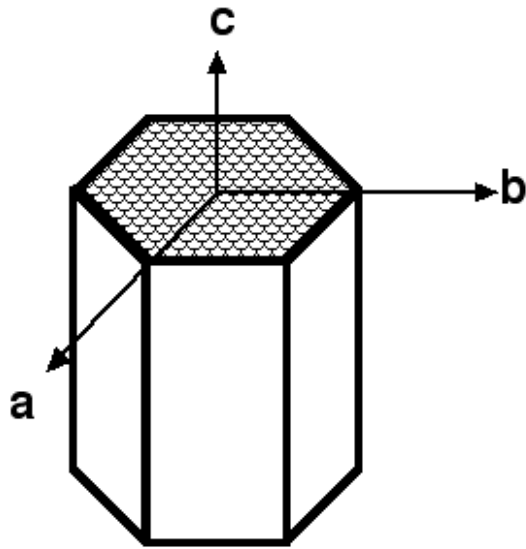
Dieter, *Mechanical Metallurgy*, 3rd edition, 1986, pp. 670, 674.

Magnesium Elektron NA User Project: “Magnesium Forming Limit Diagrams”

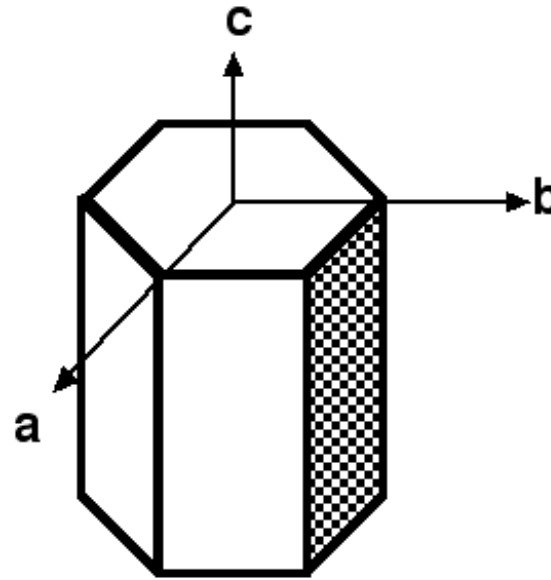


Technical Back-Up

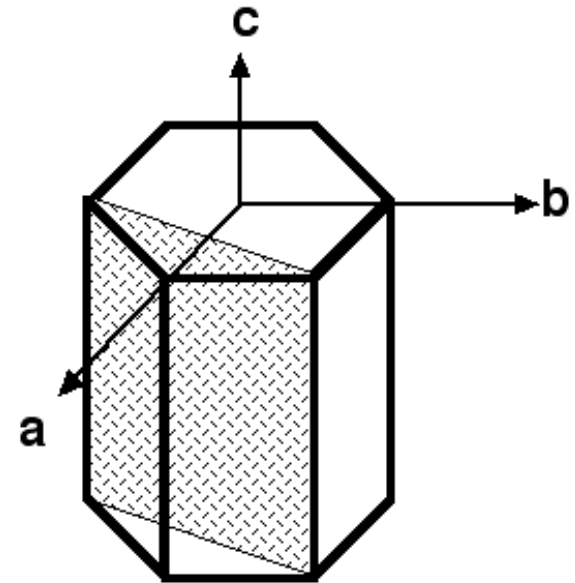
Hexagonal planes key



(0001)
Basal
Planes



(0.1.-1.0)
Prismatic
Planes



(2.-1.-1.0)