

## ***Project 39: Solute and Precipitate Effects on Magnesium Recrystallization***

***Spring Meeting  
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# Project 39: Solute and Precipitate Effects on Magnesium Recrystallization



- Student: Gillian Storey (Mines)
- Advisor(s): Dr. Kester Clarke (Mines), Dr. Amy Clarke (Mines)

- Problem: Current recrystallization studies focus on texture modification and grain size reduction mechanisms that are not industrially viable
- Objective: Study the effects of varying precipitate and solute content on recrystallization kinetics. Determine effects of kinetics on hot working parameters and material properties. (Proposed alloy: ZK60)
- Benefit: Common alloys may be studied using standard processing parameters modified for industrial benefit.

## Project Duration

MS: August 2019-August 2021

## Recent Progress

- Literature review
- Initial experimental matrix formulation
- Initial study of ZK60 baseline material

## Metrics

Description	% Complete	Status
1. Literature review.	90%	●
2. Determine classical Avrami parameters and Zener pinning parameters in static recrystallization	0%	●
3. Adaptation to dynamic recrystallization and hot working	0%	●
4. Characterization of recrystallization mechanisms that are enhanced (or retarded)	0%	●
5. Investigate mechanical and microstructural properties effected by recrystallization kinetics	0%	●

# Outline



- Project Overview / Industrial Relevance
- Literature Review Highlights
- Sample Preparation
- Cold Rolling Trials
- Proposed Material and Heat Treatments
- Future Work

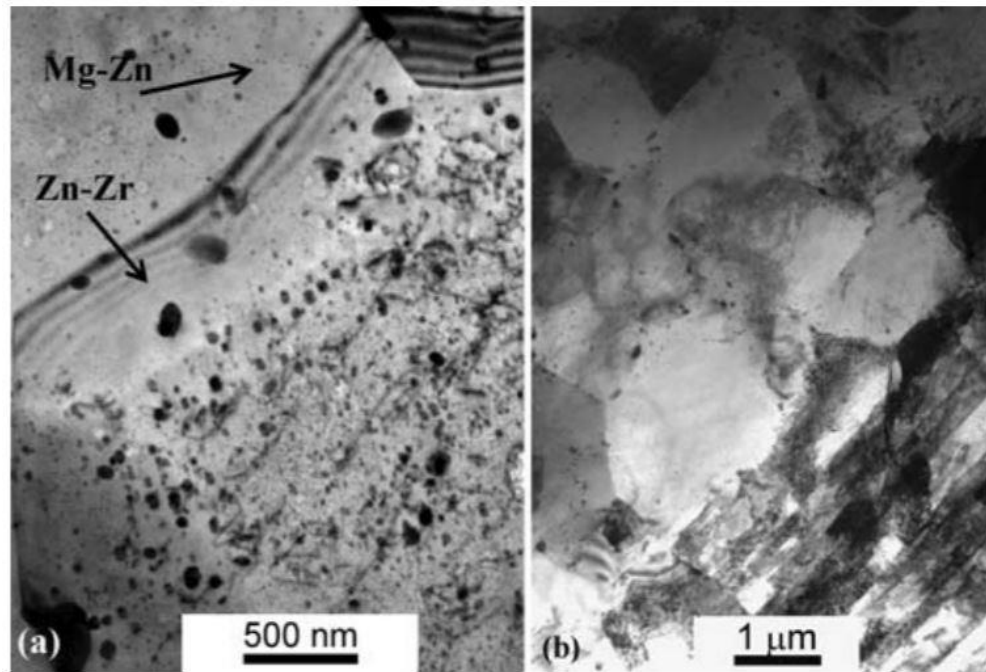
# Project Overview



- Comparative study on effects of varying precipitate and solute content on recrystallization kinetics in Mg alloys
- ZK60: nominally Mg-5.8 Zn-0.65 Zr (wt%) [1]
  - Commercial alloy with insoluble Zn-Zr particles and Mg-Zn precipitates that influence grain size and recrystallization [2]
- Determine classical Avrami and Zener pinning parameters for static recrystallization. Then, adapt to dynamic recrystallization and hot working
- Determine effects of microstructural development kinetics on hot working parameters and material properties
  - Microstructural characterization and texture evaluation through electron backscatter diffraction (EBSD)
  - Static recrystallization studies using conventional furnaces
  - Uniaxial compression tests for dynamic recrystallization using a Gleeble 3500 thermomechanical simulator

# Industrial Relevance

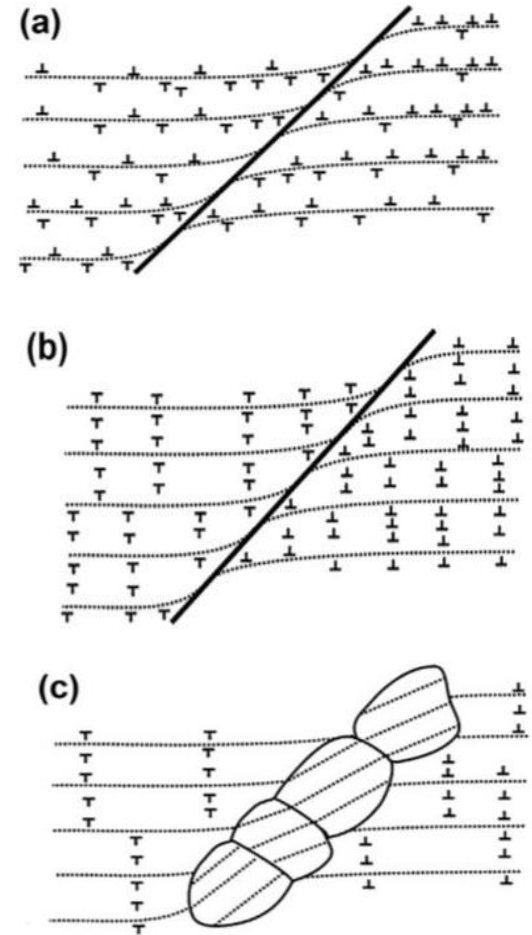
- Understanding the initiation of dynamic recrystallization for constant strain rate hot deformation processes further defines industry processing parameters for ZK60



[3] Shahzad, M., Janecek, M., Wagner, L.,  
International Journal of Materials Research, 2009

# Previous Work

- ZK60: commercial alloy with insoluble Zr particles that influence grain size and recrystallization [2]
  - Age (precipitation) hardening [4]
- Replace Zr with rare earth elements, such as Ce or La. Improves high temperature strength and creep resistance [5]
- Recrystallization driving force ( $T$ ,  $\epsilon$ )



[5]

# Extrusion of Mg Alloys

- ASTM B107/B107M-13 [6]
  - Guideline for Mg-alloy extrusion of bars, rods, profiles, tubes, and wires
  - Bars and wires: Minimum tensile strength and yield strength for ZK60 are 296 MPa (43.0 ksi) and 213 MPa (31.0 ksi)
  - Additional product dimensional tolerances
  - Experimental material conforms to this standard



This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.



Designation: B107/B107M - 13

## Standard Specification for Magnesium-Alloy Extruded Bars, Rods, Profiles, Tubes, and Wire<sup>1</sup>

<sup>1</sup> This standard is issued under the fixed designation B107/B107M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscript (e) indicates an editorial change since the last revision or reapproval.

<sup>2</sup> This standard has been approved for use by agencies of the U.S. Department of Defense.

### 1. Scope<sup>2</sup>

1.1 This specification covers magnesium-alloy extruded bars, rods, profiles, tubes, and wire of the composition given in Table 1.

1.2 The values stated in either inch-pound or SI units are to be regarded separately as standards. The SI units are shown in brackets or in separate tables or columns. The values stated in each system are not exact equivalents; therefore, each system must be used independent of the other. Combining values from the two systems may result in nonconformance with the specification.

1.3 Unless the order specifies the “M” specification designation, the material shall be furnished to the inch-pound units.

1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

### 2. Referenced Documents

2.1 The following documents of the issue in effect on date of order acceptance form a part of this specification to the extent referenced herein.

#### 2.2 ASTM Standards:<sup>2</sup>

B117 Practice for Operating Salt Spray (Fog) Apparatus  
B206 Practice for Temper Designations of Magnesium Alloys, Cast and Wrought  
B557 Test Methods for Tension Testing Wrought and Cast

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee B07 on Light Metals and Alloys and is the direct responsibility of Subcommittee B07.04 on Magnesium-Alloy Cast and Wrought Products.  
Current edition approved Nov. 1, 2013. Published December 2013. Originally approved in 1996. Last previous edition approved in 2012 as B107/B107M - 12. DOI: 10.1520/B0107\_B0107M-13.

For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

### Aluminum- and Magnesium-Alloy Products

B557M Test Methods for Tension Testing Wrought and Cast Aluminum- and Magnesium-Alloy Products (Metric)

B660 Practices for Packaging/Packing of Aluminum and Magnesium Products

B951 Practice for Codification of Unalloyed Magnesium and Magnesium-Alloys, Cast and Wrought

B954 Test Method for Analysis of Magnesium and Magnesium Alloys by Atomic Emission Spectrometry

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition

E527 Practice for Numbering Metals and Alloys in the Unified Numbering System (UNS)

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *extruded bar, n*—a solid extrusion, long in relation to its cross-sectional dimensions, having a symmetrical cross section that is square or rectangular with sharp or rounded corners or edges, or is a regular hexagon or octagon, and whose width or greatest distance between parallel faces is over 0.375 in. [10 mm].

3.1.2 *extruded profile, n*—a hollow or solid extrusion, long in relation to its cross-sectional dimensions, whose cross section is other than that of wire, rod, bar, or tube.

3.1.3 *extruded rod, n*—a solid rod extrusion, long in relation to its diameter, whose diameter is over 0.375 in. [10 mm].

3.1.4 *extruded tube, n*—a hollow extrusion, long in relation to its cross-sectional dimensions, which is symmetrical and is round, square, rectangular, hexagonal, octagonal, or elliptical with sharp or rounded corners, and has a uniform wall thickness except as affected by corner radii.

3.1.5 *producer, n*—the primary manufacturer of a material.

3.1.6 *supplier, n*—includes only the category of jobbers and distributors as distinct from producer.

3.1.7 *wire, n*—a solid section long in relation to its cross-sectional dimensions, having a cross section that is round,

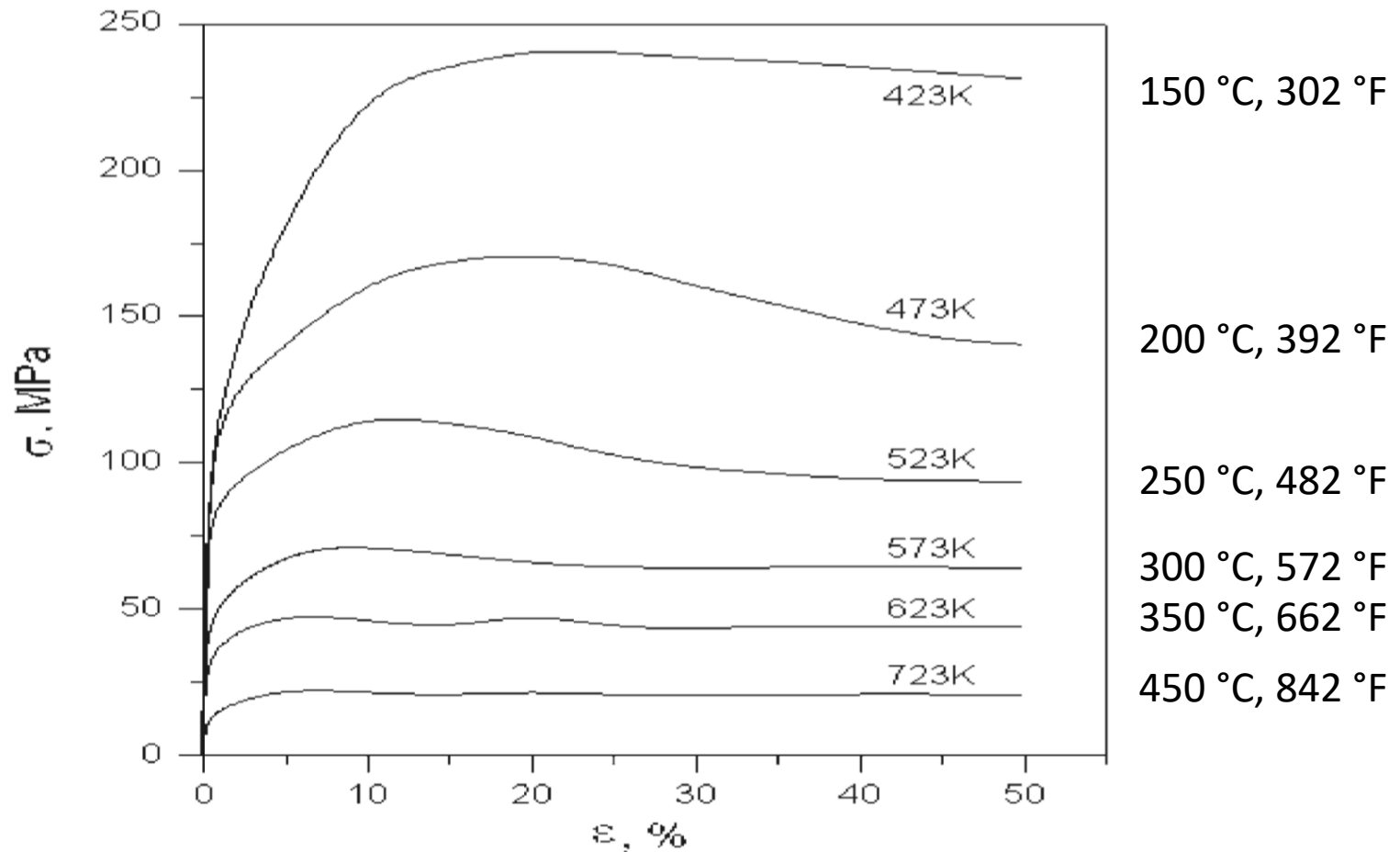
<sup>2</sup>A Summary of Changes section appears at the end of this standard.

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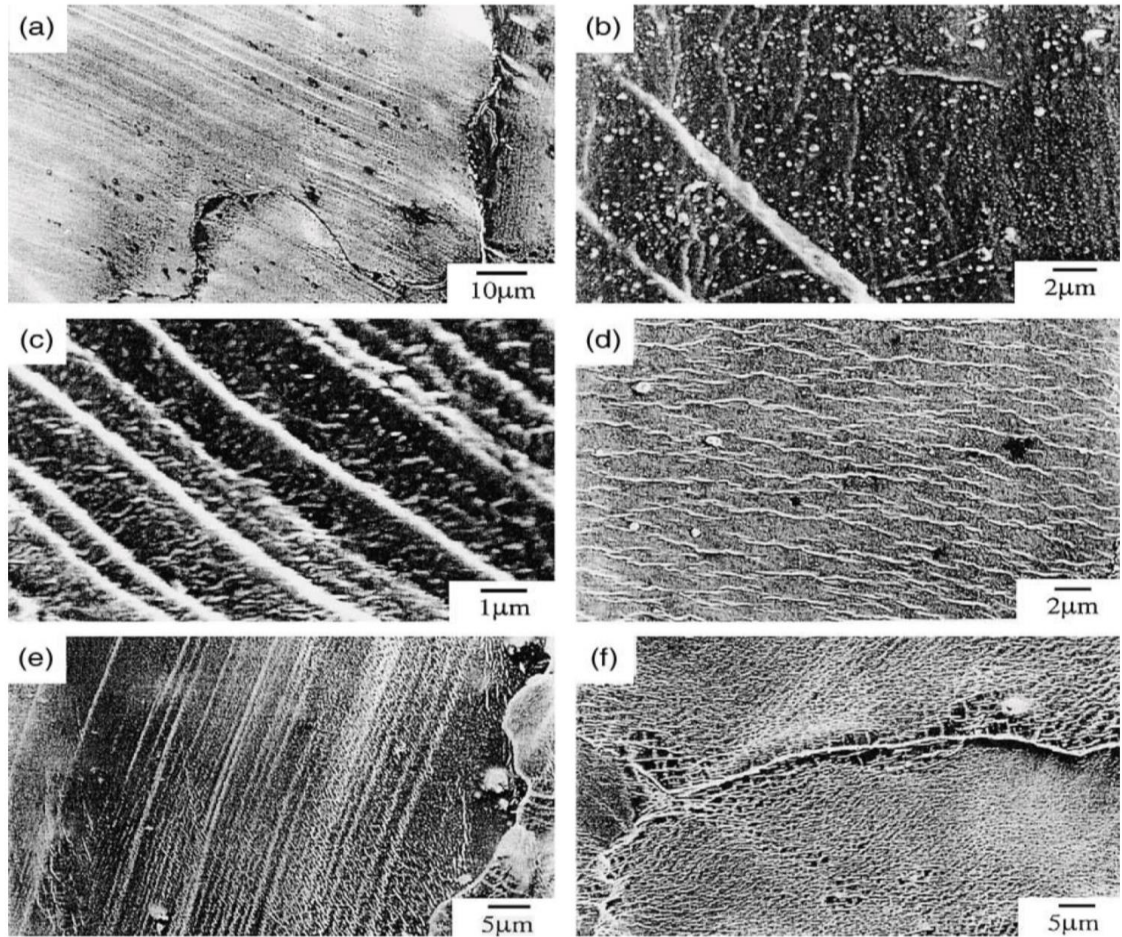
# Flow Curves for ZK60



[7] A. Galiyev, R. Kaibyshev, G. Gottstein, Acta Materialia, 2001

# Deformation Micrographs

- (a) T: 150 °C, 302 °F  
basal slip
- (b) T: 150 °C, 302 °F  
short slip lines of  $\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$
- (c) T: 250 °C, 482 °F  
basal, non-basal
- (d) T: 250 °C, 482 °F  
short wavy lines of cross-slip
- (e, f) T: 350 °C, 662 °F  
extensive multiple slip  
( $\epsilon = 12\%$  and  $\dot{\epsilon} = 2.8 \times 10^{-3} s^{-1}$ )



[7] A. Galiyev, R. Kaibyshev, G. Gottstein, Acta Materialia, 2001

# Recent Progress

# Sample Preparation

- Initial sectioning
  - High-speed abrasive wheel, grinding with 600 grit grinding paper
- Cold epoxy mount
  - Max curing temperature of 40°C for 24 hours
  - With and without conductive filler
- Polishing
  - Grinding to 1200 grit. Polished sequentially to 6 $\mu$ m, 3 $\mu$ m, and 1  $\mu$ m diamond
  - Cleaning with water, soapy cotton ball, ethanol, and hot air
  - Optical microscopy to ensure adequate polishing
  - Unintentional etching at 3 $\mu$ m step, possibly due to pH of diamond suspension.
    - Future polishing steps will include colloidal silica or electropolishing
- Etching
  - 4.2g picric acid, 10mL acetic acid, 70mL ethanol, 10mL distilled water
  - 3-5 seconds

# Cold Rolling Trial Plan

- Experimental material will be pre-strained by cold rolling up to 80% reduction [7]
  - Assume cold rolling increases hardness uniformly due to increase in stored strain energy
- Proposed geometry: flat plates. Thickness=2, 3, 4 mm to test rolling parameters
  - One study cold rolled ZK60 ingots cut to 100 x 50 x 3 mm<sup>3</sup> to a total reduction of 63.3% [8]



Rolling mill in Hill Hall at the Colorado School of Mines

# Proposed Material

- Nominal ZK60 composition (Mg-5.8 Zn-0.65 Zr, wt%) with substitution of various percentages of Ce for Zr
  - The nominal ZK60 composition is stated in ASTM B91-17 [1]
- Experimental matrix based on following compositions

	Complete Solid Solution	~1 % Pinning Phases	~ 3% Pinning Phases
Low solute	-	Mg-1.5Zn-0.4Ce	-
Med solute	-	Mg-3.2Zn-0.4Ce	-
High solute	Mg-4Zn	Mg-5Zn-0.1Ce	Mg-7.2Zn-0.33Ce

# Proposed Heat Treatments

- Performed after all cold rolling trials
- Microstructural analysis will be completed after each time-temperature combination in order to calculate percent recrystallization
- Create static recrystallization model

-	6 hours	8 hours	12 hours	24 hours	48 hours
300 °C/573K	HT+Q	HT+Q	HT+Q	HT+Q	HT+Q
350 °C/623K	HT+Q	HT+Q	HT+Q	HT+Q	HT+Q
400 °C/673K	HT+Q	HT+Q	HT+Q	HT+Q	HT+Q

HT=heat treat

Q=quench

# Heat Treatment Justification

- Rectangular plates of commercial ZK60 ingot cut to 100 x 50 x 3 mm<sup>3</sup>, homogenized at 340°C for 6 h and rolled at room temperature. Recrystallized (unknown percentage) after 10 h at 390°C [8]
- In a DRX study [7], uniaxial compression tests were carried out between 150 and 450°C for time intervals of 10 to 30 hours at strain rates between 10<sup>-5</sup> and 10<sup>-1</sup> s<sup>-1</sup>

-	6 hours	8 hours	12 hours	24 hours	48 hours
300 °C/573K	HT+Q	HT+Q	HT+Q	HT+Q	HT+Q
350 °C/623K	HT+Q	HT+Q	HT+Q	HT+Q	HT+Q
400 °C/673K	HT+Q	HT+Q	HT+Q	HT+Q	HT+Q

HT=heat treat

Q=quench

# Future Work



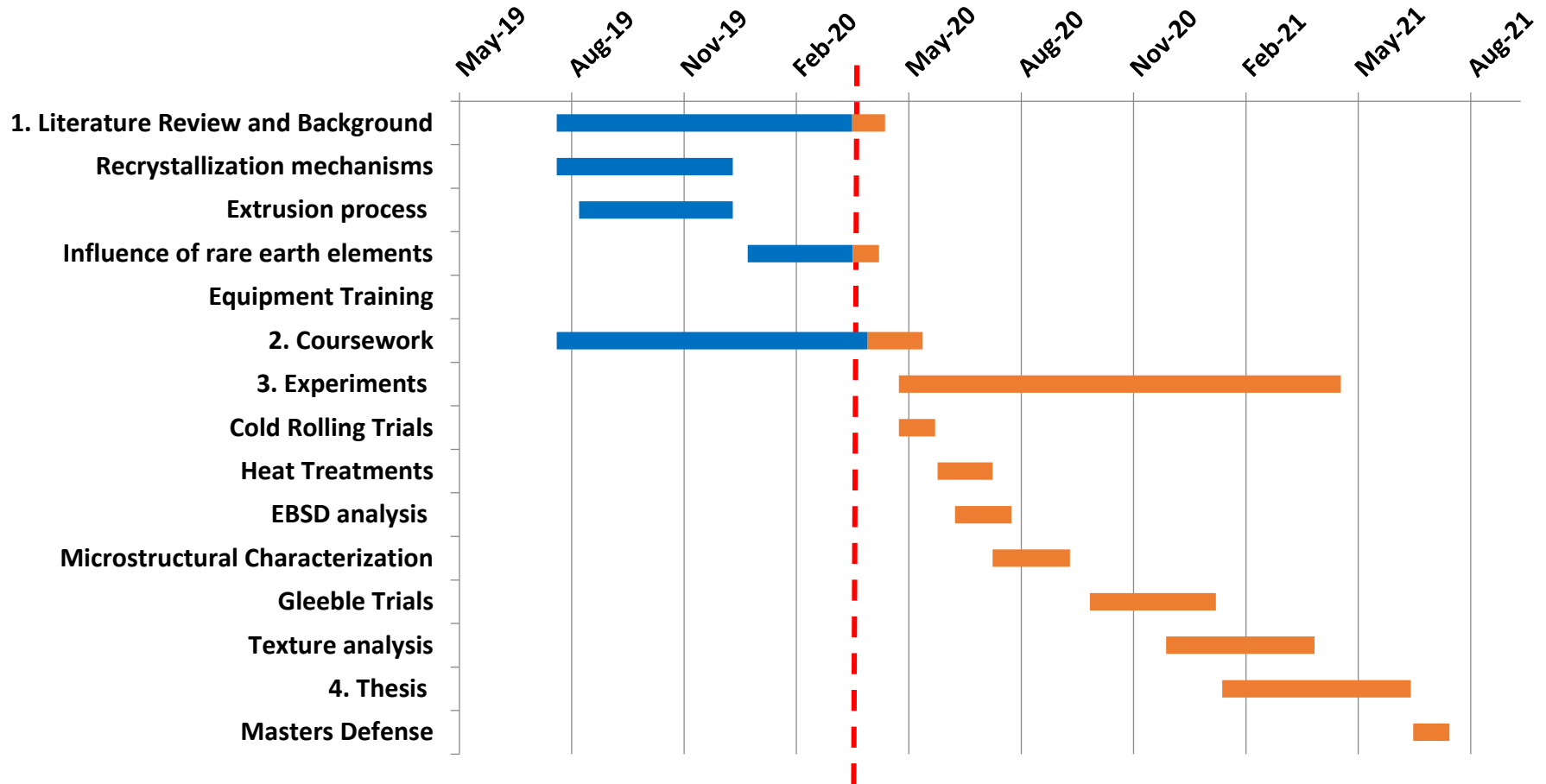
- Continued literature review on effects of alloying ZK60 with rare earth elements
- Cold rolling trials of experimental material
- Heat treatment (300, 350, and 400°C) and times (6 - 48 hours) to determine a static recrystallization kinetics model
- EBSD imaging of ZK60 heat treated specimens to determine percent recrystallization and overall microstructural evolution
- Uniaxial compression tests for dynamic recrystallization using a Gleeble 3500 thermomechanical simulator

# Challenges and Questions



- Lowest Deformation Polish?
  - Unintentional etching of samples during diamond abrasive steps
    - Reached out to the techs at LECO to discuss pH of diamond abrasive. Looking at ways to better control pressure during polishing
    - If perfected, can further polish with colloidal silica, or electropolish
- Future: Best method to measure recrystallization in ESBD?
  - GOS (grain orientation spread), using a field of measurement of 1000-2000 grains to have adequate statistical information

# Progress



# Works Cited



- [1] ASTM Standard: B91-17, Standard Specification for Magnesium-Alloy Forgings, ASTM International.
- [2] T. Bhattacharjee, T. Sasaki, B. Suh, Role of Zr in the Microstructure Evolution in Mg-Zn-Zr Based Wrought Alloys, Magnesium Technology, (2015) 1-16.
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- [7] A.Galiyev, R. Kaibyshev, G. Gottstein, Correlation of Plastic Deformation and Dynamic Recrystallization in Magnesium Alloy ZK60, Acta Materialia. 49 (2001) 1199-1207.
- [8] Jin, W., Fan, J., Zhang, H., Microstructure, mechanical properties and static recrystallization behavior of the rolled ZK60 magnesium alloy sheets processed by electropulsing treatment, Journal of Alloys and Compounds, Elsevier, 2015.

*Thank you!*

*Any comments, questions, or feedback?*

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