**Final CRADA Report for
CRADA No. NFE-06-00414, Amendment 1
with
Toyota Motor Engineering & Manufacturing of North America, Inc. (“TEMA”)**

**Use of High Magnetic Field to Control Microstructural Evolution
in Metallic and Magnetic Materials**

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Abstract

The Amendment 1, referred to as Phase 2, to the original CRADA NFE-06-00414 added tasks 3 through 7 to the original statement of work that had two main tasks that were successfully accomplished in Phase 1 of this project. In this Phase 2 CRADA extension, extensive research and development activities were conducted using high magnetic field processing effects for the purpose of manipulating microstructure in the SAE 5160 steel to refine grain size isothermally and to develop nanocrystalline spacing pearlite during continuous cooling, and to enhance the formability/forgability of the non-ferrous precipitation hardening magnesium alloy AZ90 by applying a high magnetic field during deformation processing to investigate potential magnetoplasticity in this material. Significant experimental issues (especially non-isothermal conditions evolving upon insertion of an isothermal sample in the high magnetic field) were encountered in the isothermal phase transformation reversal experiments (Task 4) that later were determined to be due to various condensed matter physics phenomenon such as the magnetocaloric (MCE) effect that occurs in the vicinity of a materials Curie temperature. Similarly the experimental deformation rig had components for monitoring deformation/strain (Task 3) that were susceptible to the high magnetic field of the ORNL Thermomagnetic Processing facility 9-T superconducting magnet that caused electronic components to fail or record erroneous (very noisy) signals. Limited experiments on developing nanocrystalline spacing pearlite were not sufficient to elucidate the impact of high magnetic field processing on the final pearlite spacing since significant statistical evaluation of many pearlite colonies would need to be done to be conclusive. Since extensive effort was devoted to resolving issues for Tasks 3 and 7, only results for these focused activities are included in this final CRADA report along with those for Task 7 (described in the Objectives Section of this report).

Objectives

The proposed objectives for the Phase 2 (Amendment 1) project included the following research and development objectives:

1. Investigate application of HMF processing to a non-ferrous alloy, AZ90, and a ferrous alloy, S45, for the purpose of enhancing/improving the formability/forging response of these material systems. Experiments will be performed using an electromechanical tensile load versus displacement system incorporated into the 9-T magnet. Enhanced formability or improved forging response will be reflected in changes in the maximum (UTS) load, P_{Max} , and uniform elongation, e_u , that will be recorded during these experiments. Results will include an evaluation and comparison of the resulting microstructures and load versus displacement data from the HMF experiments with those obtained in similar experiments without a magnetic field being present. Ambient and elevated temperature (one per alloy family) experiments will be conducted to simulate cold- and warm-working forming conditions. In addition, a second approach will be investigated whereby deformation under ambient (no applied) magnetic fields will be followed by HMF processing. These tests will be performed at room temperature or a slightly elevated temperature.

2. Perform HMF experiments with the goal of validating magnetic field effects reported in the literature as well as ORNL thermal-magnetic processing concepts for grain refinement, increased solute solubility, increased precipitation kinetics, and nano-pearlite formation. The Participant shall provide at least 2 ferrous alloys (to be down selected from S45, SCM20, SVdH20, and SCM20Nb) for the HMF processing experiments as defined under Section V. The Contractor and the Participant shall perform numerous thermo-magneto experiments using the 9-Tesla magnet and custom designed heating/cooling system. Specimen characterizations will include microstructural characterization and correlation with mechanical properties determined by the Participant. High-speed quenching dilatometry experiments will be conducted on an as-needed basis to characterize phase transformation temperatures as required for the specified steels (see task descriptions under Section V) for the purpose of defining the process control parameters for the HMF experiments.

The breakdown of the planned Phase 2 experimental test matrix for Tasks 3 through 7 follows (Tasks 1 and 2 were defined in Phase 1). Of these, actual experiments were conducted only on various aspects of Task 3, 4, and 7 while the other areas did not get accomplished due to significant experimental and/or equipment difficulties with these 3 tasks that utilized the entire project budget. Although original experiments were planned (see Task description below) to be run on the Japanese steel alloy S45 (Japanese equivalent to SAE 1045 steel), the SAE 5160 alloy was deemed a more ideal candidate to investigate the various phenomenon for Tasks 4 and 7.

PHASE 2

TASK 3 Investigation of the potential for formability/forging enhancement through magnetic field processing on the Participant's non-ferrous alloy designated AZ90 and ferrous alloy S45.

Task 3.1 Modify and incorporate an existing ORNL portable 10 KN (2 KIP) electromechanical tensile testing apparatus into the bore of the 9-T superconducting magnet with the ability to do ambient and elevated temperature deformation (tensile load versus displacement) experiments on Participant supplied tensile specimens.

Task 3.2 The Parties shall perform HMF load versus displacement experiments on both the AZ90 and S45 alloys at the Contractor's facilities at both ambient and one elevated temperature (different for the AZ90 and S45 alloys). The elevated temperature experiments will be conducted using two separate induction heating frequency ranges (~10 KHz and ~170 KHz) to determine if superimposed non-contact acoustic/ultrasonic signals (via the ORNL EMAT technology) accentuate the magnetic field effect to overcome activation energy barriers.

Task 3.3 The Parties shall perform characterization of microstructure of these HMF processed alloys.

TASK 4 Grain refinement through isothermal magnetic field cycling on the Participant's ferrous alloys designated S45 and SCM20.

Task 4.1 The Parties shall conduct high-speed quenching dilatometry experiments to define the AC₁, AC₃, AR₁, and AR₃ temperatures in the SCM20 ferrous alloy for the purpose of identifying the isothermal hold temperature for the magnetic cycling experiments This was accomplished already for the S45 alloy in Phase 1.

Task 4.2 Modify existing specimen thermal insert apparatus to cycle in and out of the high magnetic field region of the 9-T superconducting magnet while maintaining isothermal temperature conditions around tensile and Charpy specimen blanks.

Task 4.3 The Parties shall perform isothermal HMF cycling processing experiments on both the S45 and SCM20 ferrous alloys at the Contractor's facilities for 2 separate cycle times (periods).

Task 4.4 The Parties shall perform characterization of microstructure and mechanical properties of these HMF cyclically processed ferrous alloys.

TASK 5 Investigate high magnetic field processing as a means to increase solubility in the Participant's SVdH20 and SCM20Nb ferrous alloys.

Task 5.1 The Parties research staff shall provide experimentation on HMF processing of the SVdH20 and SCM20Nb ferrous alloys. Increased solubility heat treatments will be conducted for two separate temperatures, T_{sol} , above the A1 temperatures of these alloys such that: 1) $T_{sol} > T_{Curie\ of\ \alpha-Fe}$ and 2) $T_{sol} < T_{Curie\ of\ \alpha-Fe}$.

Task 5.2 The Parties shall perform characterization of microstructure and mechanical properties of these HMF processed SVdH20 and SCM20Nb ferrous alloy specimens. This effort will require the use of the ORNL super-microprobe or similar analytical instrument on a limited number of specimens (1 per alloy).

TASK 6 Investigate high magnetic field processing as a means to increase precipitation in the Participant's SVdH20 and SCM20Nb ferrous alloys.

Task 6.1 The Parties research staff shall provide experimentation on HMF processing of the SVdH20 and SCM20Nb ferrous alloys. Increased precipitation heat treatments will be conducted for two separate precipitation temperatures, T_{prec} , above the A1 temperatures of these alloys such that: 1) $T_{prec} > T_{Curie\ of\ \alpha-Fe}$ and $T_{prec} < T_{Curie\ of\ \alpha-Fe}$.

Task 6.2 The Parties shall perform characterization of microstructure and mechanical properties of these HMF processed SVdH20 and SCM20Nb alloy specimens. This effort will require the use of an ORNL FE-SEM or similar characterization instrument to document increased precipitation (on one specimen per alloy) in addition to the mechanical property tests.

TASK 7 Investigate development of nano-pearlite in the Participant's S45 ferrous alloy through high magnetic field processing.

Task 7.1 The Parties research staff shall provide experimentation on HMF processing of the S45 ferrous alloy.

Task 7.2 The Parties shall perform characterization of microstructure and mechanical properties of these HMF processed S45 alloy specimens.

Technical Discussion

The discussion section of this final report is represented principally as a compilation of summary slides/tables that are commented and therefore are self-explanatory and self-consistent. Relevant comments will be interspersed appropriately to identify specific aspects not obvious in the figures. The salient conclusions are summarized in the final "Conclusion" section of this report.

The following phase diagram figure for an SAE 5160 ferrous alloy, predicted using the ThermoCalc thermodynamic software program, shows the potential impact of a 9-T magnetic field on shifting the phase boundaries and transformation temperatures in this alloy. This alloy was chosen since the no-field phase diagram shows this 0.6 wt.% C chemistry is right at the eutectoid chemistry and therefore is a more ideal candidate (than S45) to study pure austenite (gamma) transforming isothermally to alpha (ferrite) and carbide (and back again when the field is removed and the same cycled multiple times) . Similar effects occur for the S45 alloy steel (Japanese alloy equivalent to SAE 1045) and so a sample of that alloy would be anticipated to go through a phase change isothermally when held in an appropriate two-phase field regime and cycled in and out of a high magnetic field which would induce this predicted phase boundary shift. In this way, the cellular decomposition (initiates at grain boundaries and progresses into the grain interiors) of the gamma phase to ferrite and carbide would result in successively finer grain size after repeated excursions in and out of a high magnetic field. Although not shown in this phase diagram (conventional phase diagrams only show first-order phase changes that involve crystallographic changes), hypoeutectoid steels have a Curie temperature (second order phase transition [with no accompanying crystallographic change] from ferromagnetic to paramagnetic upon heating) that causes sample temperature to change due to the magnetocaloric effect, MCE, (causing an entropy and therefore temperature change – see several schematics below that show this condensed matter physics phenomenon) that was not anticipated during the planning and execution stages of these experiments and took extensive evaluation to identify as the cause of non-isothermal conditions during these experiments.

5160 Steel Alloy

The following figure shows the linear robotic device with mounted induction heating system in relation to the 9-T superconducting magnet system that was used for the isothermal phase transformation reversal (IPTR) experiments of Task 4. The schematic shows the induction heating system initially outside the magnet system and then later after insertion into the superconducting magnet.

IPTR System Schematic

Multiple issues had to be investigated to understand the temperature variations experienced during our intended isothermal phase transformation reversal experiments as detailed here.

Five effects that have to be considered when attempting to understand the isothermal cycling experiments, most recently at 740 C

- 1. MCE (changes most rapidly in field gradient region)
 - Exothermic (temperature increase) on insertion into 9T
 - Endothermic (temperature decrease) on extraction from the magnet
- 2. Effective magnetic permeability (changes when $B < 1$ T)
 - Effective permeability > 1.0 for “no- field” condition (if $T < T_c$)
 - Whereas effective permeability $= 1.0$ for $B = 9T$
 - Surface resistance decreases on insertion → requires increased induction
 - Surface resistance increases on extraction → requires reduced induction
- 3. Bulk resistivity vs. Magnetic Field (changes most rapidly in field gradient region)
 - Resistivity increase on insertion into 9T (requires reduced induction current)
 - Resistivity decrease on extraction (requires increase in induction current)
- 4. Phase change Energy (may be delayed following insertion/extraction)
 - Exothermic on insertion (phase boundary shifts up past setpoint temperature)
 - [Transition from γ to $(\alpha + \text{Fe}_3\text{C})$]
 - Endothermic on extraction (phase boundary down up past setpoint temperature)
 - [Transition from $(\alpha + \text{Fe}_3\text{C})$ to γ]
- 5. Resistivity differs with phase (may be delayed following insertion/extraction)
 - Resistivity decrease on insertion into 9T (requires increased induction current)??
 - [Assumes that $(\alpha + \text{Fe}_3\text{C})$ has lower resistivity than γ -phase]
 - Resistivity increase on extraction (requires less induction current)??
 - [Assumes that γ -phase has higher resistivity than $(\alpha + \text{Fe}_3\text{C})$]
- *In the case of #5, if only one end of the sample transforms on insertion, it will have reduced surface resistance and therefore reduced heating (note that the surface current density remains constant, independent of resistivity variations).*

Magneto-caloric effect & Magnetic refrigeration

Adiabatic
 ΔT_{ad}

Isothermal
 ΔS_m

Cooling effect

The Magnetocaloric Effect is Maximized in the Vicinity of the Curie Temperature

FIG. 2. — Magnetocaloric effect of Fe, Co and Ni plotted vs temperature T_0 without field.

Electromagnetic Modeling of the Heating Uniformity using FDTD modeling software

Thermocouple data from recent samples revealing temperature non-uniformity along the length of the samples

Data files from March 4th through May 1st, 2009

- Mar 4 TEMA_5160_760C_NF_5M_Fast HeQ_1
- Mar 4 TEMA_5160_760C_NF5m_10m9T_125psiHeQ_1cycle
- Mar 5 TEMA_5160_750C_5m_NF_125psi HeQ_T1
- Mar 5 TEMA_5160_740C_5m_NF_125psi HeQ_T1
- Mar 5 TEMA_5160_740C_NF5m_10m9T_125psiHeQ_1cycle
- Mar 5 TEMA_5160_740C_NF5m_10m9T_125psiHeQ_1cycle-2
- Mar 16 TEMA_5160_740C_NF5m_10m9T_125psiHeQ_1cycle_A
- Mar 16 TEMA_5160_740_5m_NF_5m
- Mar 16 TEMA_5160_730_5m_NF_5m
- Mar 17 TEMA_5160_735C_NF5m_10m9T_125psiHeQ_1cycle
- Mar 17 TEMA_5160_735_5m_NF_125HeQ
- Mar 18 TEMA_5160_740C_NF5m_10m9T_125psiHeQ_1cycle_midTC
- Mar 18 TEMA_5160NSP_780_NF5m_20HeQ_1
- Mar 23 TEMA_5160NSP_800_NF5m_10m9T_Ar ambient_2 reused sample
- Mar 24 TEMA_5160NSP_800_NF5m_10m9T_Ar ambient_3 reused sample
- Mar 24 TEMA_5160NSP_800_NF5m_10m9T_Ar ambient_4 reused sample
- Mar 31 TEMA_5160NSP_800_NF5m_10m9T_Ar&4%Hambient new 1
- Mar 31 TEMA_5160NSP_800_NF5m_10m9T_Ar&4%Hambient new run2 Cu IR reflector
- Apr 3 TEMA_5160NSP_800_NF5m_10m9T_Ar&4%Hambient Ti gettering
- Apr 3 TEMA_5160NSP_800_NF5m_10m9T_Ar&4%Hambient Ti get CuRefl
- Apr 9 TEMA_5160NSP_740_NF5m_10m9T_Ar&4%Hambient_Ti_Get
- Apr 9 TEMA_5160NSP_740_NF5m_10m9T_Ar&4%Hambient_Ti_Get_CuRefl
- Apr 17 TEMA_5160NSP_740_NF9m_9m9T_NF9m_9m9T_NF9m_4%H2_TiGet_CuRings
- Apr 24 TEMA_5160NSP_740_NF9m_9m9T_NF9m_9m9T_NF9m_Argon_CuTube
- Apr 29 TEMA_5160NSP_740_NF9m_9m9T_NF9m_9m9T_NF9m_Argon_Zircn_CuTube
- May 1 TEMA_5160NSP_740_NF9m_9m9T_Cycling_TandB Manual_Zircn_CuTube

$\frac{d^2}{dt^2} \rho_{\text{He}} Q_{\text{He}}$

Temperature History of the Center and Both Ends of 5160 Sample, Ambient Argon Cooldown
 [The sample was heated in the fully extracted home position, then moved to the normal 270 mm position at 930 sec. 23-March-2009]

Temperature of 5160 as a Function of Time. New Sample - 1st run 31-Mar-09
 Using Argon/4% Hydrogen purge gas, Slow cool in Argon/H₂

Temperature of 5160 as a Function of Time New Sample - 2nd Run 31-Mar-09
With Argon/4% Hydrogen purge gas, & Copper IR reflector, Slow cool in Argon/H2

Temperature of 5160 Sample as a Function of Time, New Sample, 1st run 3-April-2009
With Argon/ 4% H2 purge gas, Titanium getter tube spaced 0.5" in front of sample, slow cool

Temperature of 5160 as a Function of Time New sample - 2nd run 3-April-09
 With Argon/ 4% H2 purge gas, Titanium getter tube spaced 0.5" in front of sample,
 Using 4" long slotted copper tube IR reflector & H-field straightener, slow cool

Temperature of 5160 as a Function of Time New Sample - 1st run 9-April-09
 TEMA_5160NSP_740_NF5m_10m9T_Ar&4%Hambient_TiGet
 Argon 4% H2 purge and slow cool, Titanium getter tube spaced 0.75" from proximal end

Temperature of 5160 as a Function of Time New sample - 2nd run 9-April-09
 TEMA_5160NSP_740_NF5m_10m9T_Ar&4%Hambient_TiGet_CuRefl
 Argon 4% H2 purge and slow cool, Titanium getter tube spaced 0.75" from proximal end

Temperature of 5160 as a Function of Time with Cyclic Magnetic Field 17-April-09
 TEMA5160NSP_740_NF9m_9m9T_NF9m_9m9T_NF9m_4%H2_TiGet_CuRings

Temperature of 5160 as a Function of Time with Cyclic Magnetic Field 24-April-09
TEMA5160NSP_740_NF9m_9m9T_NF9m_9m9T_NF9m_Argon_CuTube

Temperature of 5160 as a Function of Time with Cyclic Magnetic Field 29-April-09
TEMA5160NSP_740_NF9m_9m9T_NF9m_9m9T_NF9m_Argon_Zircn_CuTube

Temperature of 5160 as a Function of Time with Cyclic Magnetic Field 1-May-09
TEMA5160NSP_Manually_Cycling_Temperature_&_B_Argon_Zirconia_CuTube

Summary of Toyota Tensile Testing

Fast Quenching with Original 5" long Induction Coil

All samples heated up at "no-field" and held at temperature for 5 min. Samples that were then inserted into the magnet were held there for 10 min.

Date	T (C)	Field (T)	Proximal	Center	Distal
3/4/09	760	0.016	64.5	63.1	64.1
3/4/09	760	9.0	64.6	64.4	62.4
3/4/09	750	0.016	64.2	64.3	63.8
3/5/09	740	0.016	63.7	55.5, 57.0	61.8
3/5/09	740	9.0	63.9	62.8	10.6-48.3
3/5/09	740	9.0	Helium off –	slow quench	

Fast Quenching using 6" coil for more uniform heating

In addition, the sample was recessed 25 mm inside of the sample tube to improve the end-to-end thermal symmetry, and to reduce air ingress from the distal end.

Date	T (C)	Field (T)	HRC–proximal	HRC–Center	HRC–distal
3/16/09	740	9.0	52.1, 59.8	60.4, 62.6	62.5

3/16/09	740	0.016	66.5	60.2	60.1
3/16/09	730	0.016	39.4, 39.9	30.4, 30.5	34.7, 36.7
3/17/09	735*	9.0	27.5	48.7-51.5	45.7-49.1
3/17/09	735*	0.016	52.8-56.1	42.0-46.3	51.0-56.6
3/18/09	740*	9.0	64.0, 64.3	58.5-64.6	63.7, 64.0
3/18/09	780*	0.016	Slow quench at	10 - 40 C/s	cooling rate

* Starting 3/17/09, two thermocouple were used, located at the center and the proximal end of the sample

Examine temperature non-uniformity in paramagnetic state at 800 C

Add a third thermocouple so that can monitor the temperature at both ends of the same, as well as at the center. To simplify the situation, start by looking at temperature uniformity at 800 C, where the sample is paramagnetic both inside and outside of the magnet

Date	T (C)	Result
3/23/09	800	<ul style="list-style-type: none"> > Center was 15-20 C hotter than the distal end > Center was 20-36 C hotter than the proximal end > Temperature differences increasing with time > Magnetic field had no affect on the thermal profile
3/24/09	800	<ul style="list-style-type: none"> > Proximal end 20 C cooler than center > Distal end was nearly 20 C hotter than center > Magnetic field had no affect on the thermal profile > Discovered sample was not centered in the induction coil - repeat > Temperature differences increasing with time
3/24/09	800	<ul style="list-style-type: none"> > Proximal end 22-28 C cooler than center > Distal end was nearly 5-10 C cooler than center – no field > Distal end was slightly hotter than center after insertion > Distal end was 15-20 C cooler than center after extraction > Temperature differences constantly changing with time

Reduce time variation of radiation loss by controlling oxidation

Attempt to limit the oxidation (and hence the time variation of the radiative power loss) by switching to a 4% hydrogen/96% Argon purge gas, and adding a Titanium getter tube that is heated by the proximal end of the induction coil.

Date	T (C)	Result
3/31/09	800	<ul style="list-style-type: none"> > Proximal end 10 C cooler than center > Distal end was nearly 3-4 C cooler than center > Magnetic field had no affect on the thermal profile
3/31/09	800	<ul style="list-style-type: none"> > Reused same sample following slow Argon cooldown > Added slotted copper tube to improve H-field uniformity > Proximal end 15-17 C cooler than center > Distal end was 4-6 C hotter than center

		> Magnetic field had no affect on the thermal profile
4/3/09*	800	> Both ends now hotter than the center > Proximal end 8-10 C hotter than center > Distal end was 1-4 C hotter than center > Magnetic field had no affect on the thermal profile
4/3/09*	800	> Reused same sample following a slow Argon cooldown > Added slotted copper tube to improve H-field uniformity > Proximal end 4-6 C hotter than center > Distal end was 2-4 C cooler than center > Magnetic field had no affect on the thermal profile
4/9/09*	740	> Both ends hotter than the center > Proximal end 12-15 C hotter than center > Distal end was 5-8 C hotter than center > Magnetic field had a modest affect on the thermal profile
4/9/09*	740	> Reused same sample following a slow Argon cooldown > Added slotted copper tube to improve H-field uniformity > Both ends at same temperature, 6-8 C hotter than the center > Magnetic field had a major affect on the thermal profile > At 9 T field, both ends are significantly cooler than center > Proximal end > 20 C cooler, distal end about 7 C cooler
4/17/09**	740	> Place thin copper rings at both ends to reduce the “end” effect > Cycle field multiple time to determine if behavior is reproducible > Proximal end is 5 C hotter than center in steady-state @ 9T > Proximal end is 3-15 C cooler than center in s-s @ no-field > Distal end is 15-20 C cooler than center in s-s @ 9T > Distal end is nearly same as center in s-s @ no-field > Time behavior is extremely complicated but very reproducible

* Added Titanium getter tube starting 4/3/09

** Add thin copper rings at both ends of the sample

Thermally insulate the samples

By wrapping the samples in high temperature thermal insulation (silica fiber, zirconia fiber, alumina fiber), the sample can be thermally isolated from the surrounding environment. This reduces the heating requirement by a factor >2, which reduced the relative importance of the uniformity of the heating profile, as the thermal conductivity of the sample has greater influence, on a relative basis. The thermal insulation also eliminates the radiative heat loss, so that progressive oxidation is no longer an issue. In addition, the copper helix used to center the sample in the quench stream is no long needed and can be removed, which eliminate a source of helical asymmetry. The thermally insulated samples result in reproducible behavior over multiple field cycles, but the “R-value” of the insulation was less than expected, at most a factor of two reduction in the heat loss rate.

Date	T (C)	Result
4/24/09	740	> Controlling distal end by mistake > Thermal insulation greatly simplified temperature behavior

		> Silica insulation only reduced heat loss slightly, < 1.5x > Entire sample has uniform temperature at the “no-field” position > In the magnet (9T), the center is 15-20 C hotter than both ends > Thermal behavior is basically reproducible for 3 cycle
4/29/09	740	> Zirconia insulation reduced heat loss by a factor of 2x > Entire sample has uniform temperature at the “no-field” position > In 9T field, proximal end is 20 C cooler than center > In 9T field, distal end is 2-4 C hotter than center
5/1/09	Manual control (730 at end)	> > > >

Summary of Experimental Progression

>> Heating uniformity was substantially improved by:

Lengthening the induction coil from 5” to 6”

Positioning a slotted copper tube inside the coil to enforce the H-field uniformity

Placing thin copper rings at the very ends of the sample to suppress the “end” effect

Result: End-to-end temperature difference reduced, but temperatures drift with time, presumably resulting from variations in the radiative heat loss, due to surface oxidation

>> Variations in the radiative heat loss were mitigated by taking steps to reduced oxidation of the sample (which impacts the optical emissivity) by:

Using a 4% hydrogen/96% Argon mixture for the purge gas

Placing a hot Titanium getter tube just upstream from the proximal end of the sample

Result: Temperature differences and thermal drift were reduced, but not eliminated.

However, now find that magnetic field cycling at 740 C upsets thermal control

>> Thermally isolate the sample to:

Reduce the importance of heating uniformity

Eliminate radiative heat loss

Also eliminates the colder copper helix which may produce a helical asymmetry

Result: Behavior observed with field cycling is reproducible over many cycle. Insulation “R-value” is still too low, only a factor of two in heat loss rate

DEFORMATION PROCESSING EXPERIMENTS

Deformation Insert Extracted as Far as Possible (42" motion)

Deformation Rig Fully Inserted Into 9 Tesla Magnet

The above schematic shows the deformation assembly rig with induction heating capability built to conduct the magnetoplasticity experiments (enhanced deformation/forging processing study) in the ORNL 9-T superconducting magnet system. The figure below defines how the tensile sample was held in the deformation rig during the experiments.

Deformation Processing Experiments: Influence of **9T** Magnetic Field on Tensile Response of AZ31 Mg Alloy Tested at Room Temperature

To conduct the deformation processing experiments, a prototype tensile test rig with induction heating capability was designed and built (utilizing this project's funding) for insertion into the ORNL 9-T superconducting magnet system.

Before failure of the deformation equipment electronics due to exposure to the very high magnetic fields, this initial data suggested that deformation processing at ambient temperatures has the potential to increase the amount of total deformation achieved in a precipitation hardening magnesium alloy. Although not statistically significant (only one run), this data suggests the need to pursue follow-on activities in this very promising area.

A comparable experiment run at elevated temperature (150C) shown in the next figure did not show comparable results but the sample heating was determined not to be uniform and so these data are highly suspect as not being representative or reproducible. Non-isothermal conditions existed for both the no-field and field (9-T) experiments conducted at 150C. A more robust heating system that will guarantee sample temperature uniformity during the entire deformation processing step needs to be developed in a follow-on endeavor.

Deformation Processing Experiments Influence of **9T** Magnetic Field on Tensile Response of **AZ31 Mg Alloy** Tested at **150C**

Comments on Deformation Processing Experiments

- **Temperature Uniformity was an issue**
 - Initiated experiments varying sample location in induction coil
 - Shift of ~4mm caused apparent failure location to shift from one end of gage length to the other
 - Encoder connector broke that terminated experiments
 - Even room temperature tests could not be run then
- **Encoder Assembly had to be replaced**
 - More robust electrical connectors
 - New encoder component just received this week

NANOCRYSTALLINE SPACING PEARLITE EXPERIMENTS

Materials and Method:

- lengths of 38mm x 12.7mm 5160H plate were purchased from a metals distributor, see chemistry in Table 1
- 90mm x 8mm diameter cylindrical samples were machined from the rectangular plate by an outside lab and provided to ORNL
- Thermocouples were attached at three locations along the length of the samples (Figure 2)
- The 'Center' thermocouple location was used to control the induction furnace response
- ORNL utilized their HMF system (Figure 1) to austenitize and cool the samples with or without a 9-T magnetic field
- This system has three major components; robotic insertion arm, induction furnace, and superconducting, horizontal bore 9-T magnet
- samples were austenitized, quenched just below the eutectoid temperature, held to allow transformation to ferrite/pearlite, and finally quenched to RT (the heat treat schedule for each of the samples is given in Tables 3, Pg. 5)
- Thermocouple traces showing the actual temperature at each location is provided in Figures 3-8, Pgs. 6-8
- ORNL provided 6 samples (3 each processed with and without 9-T field) to TTC-MED for evaluation, see Table 2
- Due to concerns for uniform heating along the length of the samples, TTC evaluated only microstructure and hardness at the three thermocouple locations to confirm the presence of a nanopearlite phase
- Samples were sectioned and cold-mounted in 2-part epoxy with the cross-sections shown in Figure 2
- Vicker's hardness (HV10) was measured at each location
- based on the hardness data, two samples (1 each with and without the 9-T field were investigated for microstructure)
- Samples were etched with 2% Nital and optical microscopy used to capture microstructure features
- Additional inspection by conventional tungsten filament SEM was also performed in an effort confirm the presence a nanopearlite phase

Table 1 - Chemistry of sample material based on certification.

	C	Si	Mn	P	S	Ni	Cu	Cr	Mo	V	Sn	Cb
Samples	0.62	0.23	0.95	0.011	0.02	0.098	0.23	0.94	0.015	0.008	0.012	0.032
AISI5160	0.55 - 0.65	0.15 - 0.35	0.65 - 1.10	0.035 max	0.04 max	0.25 max	0.35 max	0.6 - 1.00	0.06 max	-	-	-

Table 2 - TTC sample ID number and corresponding description.

TTC Sample No.	Description (ORNL Sample ID)
10-022	TEMA5160_830C_NF10m_HeQ25psi_650C_NF8m_s1
10-023	TEMA5160_830C_NF10m_HeQ25psi_650C_NF8m_s2
10-024	TEMA5160_830C_NF10m_HeQ25psi_650C_NF8m_s3
10-025	TEMA5160_830C_9T10m_HeQ25psi_650C_9T8m_s3
10-026	TEMA5160_830C_9T10m_HeQ25psi_650C_9T8m_s4
10-027	TEMA5160_830C_9T10m_HeQ25psi_650C_9T8m_s5

Materials and Method:

Movable Induction Insert Extracted 44" from Magnet Center

17-Mar-09 v1B

Movable Induction Assembly Fully Inserted Into 9 Tesla Magnet

Figure 1 - Schematic of magnet and robotic insertion arm used in HMF experiments.

Figure 2 - Sample geometry showing thermocouple locations and evaluation planes.

Materials and Method:

Table 3 - Heat treat details for each sample.

10-022

8-Dec-09

Description	Start Temp	End Temp	Time	Magnetic Field
Heat-up	RT	700	8 min	NF
Heat-up	700	750	1 min	NF
Heat-up	750	760	1 min	NF
Heat-up	760	830	2 min	NF
Hold	830	830	10 min	NF
Quench	830	650	4 sec	NF
Hold	650	650	8 min	NF
Quench	650	<40	~1min 20sec	NF

Notes: • Heated indirectly using Ti susceptor

10-025

7-Dec-09

Description	Start Temp	End Temp	Time	Magnetic Field
Heat-up	RT	700	8 min	9T
Heat-up	700	760	1 min	9T
Heat-up	760	770	1 min	9T
Heat-up	770	830	2 min	9T
Hold	830	830	10 min	9T
Quench	830	650	4 sec	9T
Hold	650	650	8 min	9T
Quench	650	<40	~75 sec	9T

Notes: • Heated indirectly using Ti susceptor

10-023

9-Dec-09

Description	Start Temp	End Temp	Time	Magnetic Field
Heat-up	RT	700	8 min	NF
Heat-up	700	750	1 min	NF
Heat-up	750	760	50 sec	NF
Heat-up	760	830	2 min 10 sec	NF
Hold	830	830	10 min	NF
Quench	830	650	4 sec	NF
Hold	650	650	8 min	NF
Quench	650	<40	80 sec	NF

Notes: • titanium susceptor
• He quench 830 → 650 was shorter than normal**10-026**

7-Dec-09

Description	Start Temp	End Temp	Time	Magnetic Field
Heat-up	RT	700	8 min	9T
Heat-up	700	760	1 min	9T
Heat-up	760	770	1 min	9T
Heat-up	770	830	1 min	9T
Hold	830	830	10 min	9T
Quench	830	650	4 sec	9T
Hold	650	650	10 min	9T
Quench	650	<40	75 sec	9T

Notes: • Heated indirectly using Ti susceptor

10-024

9-Dec-09

Description	Start Temp	End Temp	Time	Magnetic Field
Heat-up	RT	700	8 min	NF
Heat-up	700	750	1 min	NF
Heat-up	750	760	50 sec	NF
Heat-up	760	830	2 min 10 sec	NF
Hold	830	830	10 min	NF
Quench	830	650	4 sec	NF
Hold	650	650	8 min	NF
Quench	650	<40	80 sec	NF

Notes: • titanium susceptor for indirect heating
• did not use Cu wire cage to center sample in susceptor for this sample**10-027**

7-Dec-09

Description	Start Temp	End Temp	Time	Magnetic Field
Heat-up	RT	700	8 min	9T
Heat-up	700	760	1 min	9T
Heat-up	760	775	1 min	9T
Heat-up	775	830	2 min	9T
Hold	830	830	10 min	9T
Quench	830	650	4 sec	9T
Hold	650	650	8 min	9T
Quench	650	<40	~75 sec	9T

Notes: • Heated indirectly using Ti susceptor

Materials and Method:

Materials and Method:

Figure 5 - Thermocouple trace for sample 10-024.

Figure 6 - Thermocouple trace for sample 10-025.

Materials and Method:

Figure 7 - Thermocouple trace for sample 10-026.

Figure 8 - Thermocouple trace for sample 10-027.

Hardness (HV10)

Sample #	Location	1	2	3	Avg
10-022	Proximal	313.1	319.9	317.8	317
	Center	298.3	298.5	297.1	298
	Distal	302	299.6	305.9	303
10-023	Proximal	330.4	335.2	334.5	333
	Center	300.5	302	297.3	300
	Distal	297.3	299.6	296.2	298
10-024	Proximal	339.8	339	342.8	341
	Center	307.8	301.8	304.3	305
	Distal	299.7	307.7	305.9	304
10-025	Proximal	347.8	348.8	348.9	349
	Center	307.3	302.4	300.7	303
	Distal	297.4	296.6	294.3	296
10-026	Proximal	352.4	347.7	353.6	351
	Center	302.4	305.9	304.8	304
	Distal	295.9	296.7	286.3	293
10-027	Proximal	355.5	360.2	360.7	359
	Center	307.1	304.1	306.9	306
	Distal	301.8	297.4	303.8	301

Optical Microscopy Sample #10-022 (Conventional Process)

Optical Microscopy Sample #10-027 (HMF Process)

Results:

Scanning Electron Microscopy

Sample #10-022 (Conventional Process)

Proximal

Center

Distal

Results:

Scanning Electron Microscopy

Sample #10-027 (HMF Process)

Proximal

Center

Distal

Results:

Scanning Electron Microscopy

The optical and scanning microscopy results are rather limited and therefore not conclusive on the impact of high magnetic field processing on developing nanospacing pearlite in this SAE 5160 alloy steel. Significantly more experiments for statistical evaluation would be needed to determine pearlite spacing distributions to document this type of impact.

Subject Inventions

Intellectual Property Opportunities

- **Records-of-Invention need to be formalized as U.S. Patent (& potentially foreign) Applications:**
 - 1300002089 *“Enhanced Performance Precipitation Hardening Alloys”*, G. M. Ludtka, G. Mackiewicz-Ludtka, J. B. Wilgen, R. A. Kisner, and J. Clark (Toyota - TEMA)
 - Experimental data (Mg alloy work on Phase 1) is available to support claims.
 - 1300002090 *“Method and Apparatus for Improved Deformation Processing and Material Performance Enhancement”*, G. M. Ludtka, G. Mackiewicz-Ludtka, J. B. Wilgen, R. A. Kisner, and J. Clark (Toyota - TEMA).
 - Phase 2 Deformation Rig experiments are focused towards demonstration of these concepts.

Commercialization Possibilities

The results of this preliminary investigation indicated that the thermomagnetic processing technology has major potential commercialization applications from process energy efficiency improvement perspectives as well as making major improvements in component performance metrics (properties) for a broad range of applications. Future collaborations (next section) with Eaton and potential equipment suppliers (AjaxTOCCO [induction heating equipment] and American Magnetics Inc [superconducting magnet technology]) will pursue those possibilities.

Plans for Future Collaborations

The Toyota Motor Engineering & Manufacturing of North America, Inc. (“TEMA”) is open to finding appropriate DOE solicitations to submit a proposal for obtaining additional funding resources to continue their evaluation of ORNL’s thermomagnetic processing technology. In addition, they would like to pursue potential focused endeavor opportunities in the future RAMP-UP program that may be funded through EERE in the future. Potential technical areas (not inclusive) include the following in this Future Vision slide used during the last CRADA project meeting held at ORNL in 2009.

Future Vision: ARPA-E Opportunity -Potential Topics

- **Magnetocaloric Effect Refrigeration Development for Automotive Air Conditioning**
- **EMAT of Magnesium, Aluminum, or Cast Iron Alloys**
 - Potential wrought property performance in as-cast materials
- **Improved Performance of Paramagnetic Precipitation Hardening Alloys for Lightweight Automotive Applications**

Conclusions

In this Phase 2 CRADA extension, extensive research and development activities were conducted using high magnetic field processing effects for the purpose of manipulating microstructure in the SAE 5160 steel to refine grain size isothermally and to develop nanocrystalline spacing pearlite during continuous cooling, and to enhance the formability/forgability of the non-ferrous precipitation hardening magnesium alloy AZ90 by applying a high magnetic field during deformation processing to investigate potential magnetoplasticity in this material. Significant experimental issues (especially non-isothermal conditions evolving upon insertion of an isothermal sample in the high magnetic field) were encountered in the isothermal phase transformation reversal experiments (Task 4) that later were determined to be due to various condensed matter physics phenomenon such as the magnetocaloric (MCE) effect that occurs in the vicinity of a materials Curie temperature. Similarly the experimental deformation rig had components for monitoring deformation/strain (Task 3) that were susceptible to the high magnetic field of the ORNL Thermomagnetic Processing facility 9-T superconducting magnet that caused electronic components to fail or record erroneous (very noisy) signals. Limited experiments on developing nanocrystalline spacing pearlite were not sufficient to elucidate the impact of high magnetic field processing on the final pearlite spacing since significant statistical evaluation of many pearlite colonies would need to be done to be conclusive. Since extensive effort was devoted to resolving issues for Tasks 3 and 7, only results for these focused activities are included in this final CRADA report along with those for Task 7.