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Novel Nanostructured Magnesium Composites for Lightweight Structural Application

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SBIR Phase I Final Technical Report

By

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Prepared by

MATSYS, Inc.
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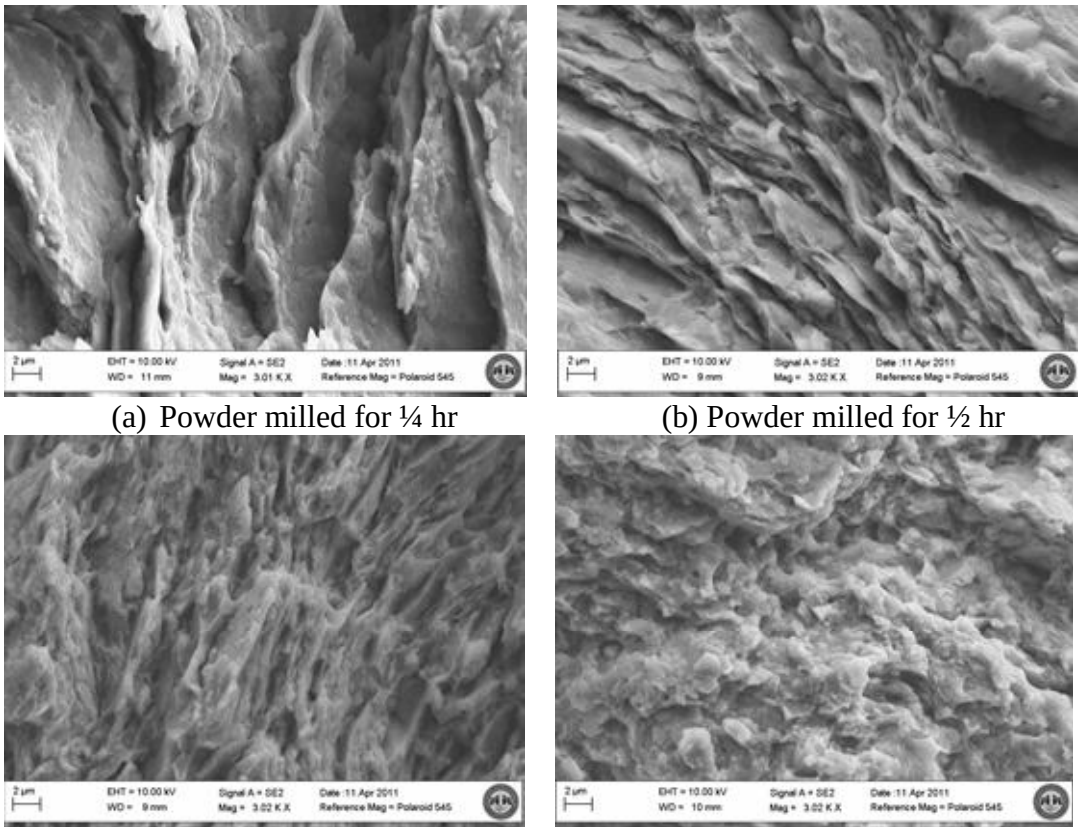
NOVEL NANOSTRUCTURED MAGNESIUM COMPOSITES FOR LIGHTWEIGHT STRUCTURAL APPLICATIONS

EXECUTIVE SUMMARY

There has always been a great interest in low density, high strength materials. Mg-based composites have always been good candidates because of their low density and good workability, properties that are attractive for diverse industrial applications. The challenge has been to maximize the strength of these composites. Mg and Mg-based composites can be strengthened by dispersing hard particles, such as B_4C, or fibers, such as CNTs. The extraordinary properties of CNTs, including high tensile strength and high elastic modulus, make them ideal candidates as reinforcements to increase both stiffness and strength, while contributing to weight savings. One major obstacle to the effective use of CNTs as reinforcement in composites is their agglomeration and poor dispersion within the metal matrix. This problem is amplified as the proportion of reinforcement is increased. In addition, we have observed that as the CNT content is increased the temperature required for full densification is increased and the potential for interfacial reaction between the CNTs and metal matrix is increased.

MATSYS initiated the development of novel compositions and processing techniques to produce lightweight, high-strength, nanostructured, Mg-based composites for structural and light armor applications. We used MA of a mixture of Mg or Mg alloy and reinforcement to synthesize a nanostructured composite powder with the reinforcement uniformly dispersed within the nanocrystalline metal matrix. These powders are then consolidated to full density by instrumented-HIP to produce nanostructured lightweight composites. The effects of reinforcement content as well as the type of the reinforcement on processing as well as on the strength and ductility of the composite is not well understood. We aim to (1) quantify the relationship between the type and amount of reinforcement, including CNTs and B_4C, and the resulting composite strength and ductility; and (2) tailor the process to balance the requirements for strength and ductility to meet the applications requirements.

To date we demonstrated that the tensile strength of nanostructured Mg has increased by 58% over the corresponding coarse-grained material. The temperature required to achieve full densification has been reduced by 30^oC as a result of the microstructure refinement. This reduction in temperature helps to preserve the microstructure of the starting powder in the bulk material. We demonstrated the use of MA and instrumented-HIP to achieve the desired nanostructure in a fully dense material. MATSYS will demonstrate the versatility of the approach by fabrication, from different composite powders, fully dense, low density and high strength nanostructured composites that will enhance the performance of lightweight, structural materials and armor applications.



(a) Powder milled for ¼ hr (b) Powder milled for ½ hr

(c) Powder milled for 1 hr (d) Powder milled for 2 hrs

Figure 1. Bulk nanostructured Pure Mg was produced by high-energy milling of coarse Mg powder followed by instrumented-HIP. A refinement in the microstructure can be seen as a result of milling time. An increase in tensile strength was observed as a result of increased milling time up to 1 hr.

1. PROJECT OBJECTIVES

The overall objective of the project is to demonstrate a powder-based processing technology for fabrication of fully dense, bulk nanostructured Mg-based composites with average grain size less than 100 nm, tensile strengths greater than 600 MPa (87 ksi) and tensile failure strain greater than 5% at room temperature. Our concept combines high energy milling/mechanical alloying (MA) to prepare nanostructured composite powders with our unique expertise in instrumented-Hot Isostatic Pressing (HIP) to fabricate fully dense, bulk nanostructured Mg-based composites.

MATSYS used MA to prepare Mg-based alloy powder with nano-grained structure and strengthen them by dispersing CNTs or B_4C. We used instrumented-HIP, as the primary consolidation process, to minimize the exposure of the powder to high temperature, preserve the microstructure of the starting powder and achieve high strength, bulk nanostructured composites. This consolidation step will be followed by secondary processing using either extrusion or rolling to break any oxide layer and tailor the mechanical properties to meet the strength and ductility requirements.

The specific objectives can be enumerated as follows:

1. Select a high strength Mg alloy;
2. Design the MA process for Mg alloy to obtain nanostructured powders with average grain size less than 50 nm;
3. Design the consolidation process for densification of nanostructured powders into fully dense, bulk nanostructured material with average grain size less than 100 nm;
4. Design the MA process for Mg alloy reinforced with CNTs or B_4C to obtain nanostructured composite powders with average grain size less than 50 nm;
5. Design the consolidation process for densification of nanostructured composite powders into fully dense, bulk nanostructured composites with average grain size less than 100 nm;
6. Characterize the mechanical and thermal properties of the nanostructured materials under quasi-static and dynamic conditions (0.001/s-10,000/s); and
7. Scale-up the process to produce tiles for ballistic testing by US Army.

1.1 Phase I Technical Objectives

The specific objectives of Phase I were as follows:

1. Design MA process for Mg-based nanostructured composite powders;
2. Design consolidation process for densification of composite powders into fully dense, nanostructured composite; and
3. Characterize the mechanical properties of the fully dense composite.

The basic question we were trying to answer to demonstrate the feasibility of the proposed concept is: ***can we prepare Mg-based nanostructured composite powders and***

consolidate them at low temperature into a fully dense compact with the requisite strength and microstructure? A successful answer to this question will enable the introduction of a new class of low density, high strength, and nanostructured Mg-based composites for a wide range of structural and light armor applications.

2. TECHNICAL APRROACH

We used mechanical alloying (MA) to strengthen pure Mg and Mg-based alloys by dispersing fibers, such as carbon nanotubes (CNTs), or hard particles, such as boron carbide and graphene, and achieving a nano-grained composite powder. The composite powder was then consolidated to full density using instrumented-HIP to minimize the exposure of the powder to high temperature, preserve the microstructure of the starting powder and achieve a nanostructured composite. During the project, we demonstrated the use of MA and instrumented-HIP to achieve the desired nanostructure in a fully dense composite and quantify the dependence of the mechanical properties on the amount and type of reinforcement.

The objective is to make composites with tensile strengths greater than 600 MPa (87 ksi) and a tensile failure strain greater than 5% at room temperature. The versatility of the approach will be demonstrated by fabrication, from different composite powders, fully dense, low density and high strength nanostructured composites that will enhance the performance of lightweight structural materials. These materials can also be used for light armor applications. Upon successful demonstration, this powder-based process can be easily applied to different powders, and scaled for cost-effective, high-volume production of fully dense, nanostructured Mg-based composites.

The Phase I effort includes three main tasks:

Task I - MA Process Design for Mg-based Composite Powders

The objective of this task is to manipulate the microstructural architecture of the fully dense material to maximize its mechanical properties.

Task II - Composite Powder Consolidation

The objective of this task is to identify the temperature-pressure-time regime to reach full densification while preserving the microstructure of the starting powder and preventing any interfacial reaction between the reinforcement and the metal matrix.

Task III - Material Characterization

In this task, measurements will be made to examine the microstructure of the fully dense composite and to characterize the mechanical properties of the material systems under investigation.

2. DETAILED TECHNICAL DISCUSSION

2.1 Phase I Results Overview

During Phase I we investigated the processing and mechanical properties for four different Mg-based materials:

1. Pure Mg, as-received (coarse grain) and milled;
2. Two commercially available Mg alloys, WE43 and E21, as-received (coarse grain) and milled;
3. Pure Mg reinforced with CNTs using MA; and
4. A high strength Mg alloy, Mg-2Y-1Zn, prepared by MA.

We initiated the demonstration of our concept by successful development of the processing technology for pure Mg and characterization of the gains in mechanical properties for bulk nanostructured Mg material as compared to bulk coarse-grained material. The as-HIPed coarse-grained Mg had tensile strength and ductility that matched published data. We used high energy milling to produce Mg powder with nano-grained structure. Using instrumented-HIP we were able to reduce the maximum temperature required for full densification by 30^{o}C. This reduction in temperature helped to preserve the nanostructure of the starting powder and achieve a fully dense bulk nanostructured material. The as-HIPed nanostructured material has a tensile strength 58% higher than the coarse-grained material.

Subsequently, we initiated the development of the processing technology for Mg alloys. Two commercially available Mg alloys, WE43 and E21, were selected. We developed the HIP processing schedules for these two alloys to achieve full densification. The as-HIPed coarse-grained material had tensile strength and ductility that matched published data. We used high energy milling to refine the microstructure of the two alloys. We then used instrumented-HIP to consolidate the powder into fully dense, bulk nanostructured material. We did not see the improvements in mechanical properties expected from the refinement in the microstructure, and we suspect that this is the result of impurities within the starting alloy powder.

We then used MA to reinforce ground Mg powder with 1 wt% CNTs. We successfully prepared nanostructured Mg-CNTs composite powder, which was then consolidated into bulk nanostructured composite using instrumented-HIP. The as-HIPed material has a compressive strength of 220 MPa (32 ksi) and a tensile strength of 114 MPa (16.5 ksi). This primary consolidation process was followed by extrusion to break the oxide layer. The extruded material has an average grain size of 61 +/- 3 nm. Its compressive strength is 258 MPa (37.4 ksi) and its tensile strength is 171 MPa (24.8 ksi). Significant improvements were seen in the tensile properties. Mg-based alloys with higher loadings of CNTs need to be investigated to achieve the required mechanical properties.

Based on these results, we selected to work with Mg-2Y-1Zn alloy, a high strength Mg-based alloy originally developed by Inoue and his co-workers [1-3]. We started with a

blend of elemental constituents and used MA to prepare nanostructured powder. We then consolidated the nanostructured powder into fully dense, bulk nanostructured material using instrumented-HIP. For the as-HIPed material, we measured a hardness of 2 GPa and a compressive strength of 550 MPa (79.8 ksi), but the tensile strength was 142 MPa (20.6 ksi) and the material was extremely brittle. We then used extrusion to break the oxide layer and maximize the mechanical properties. The extruded material has an average grain size of 40 nm. The compressive strength increased by 15% to 634 MPa (91.9 ksi) while the tensile strength doubled to 285 MPa (41.3 ksi). In addition, the tensile failure occurred outside the gage area (within the shoulder of the dogbone specimen). This type of failure is the result of introducing defects/imperfections during specimen preparation as a result of the small diameter of the extruded specimen as well as the material being brittle. We have not yet optimized the extrusion process. We also need to investigate heat treatment to tailor the properties of the bulk material and especially the ductility.

The properties obtained with the Mg-2Y-1Zn alloy are extremely promising. The alloy was prepared with ground Mg powder. These properties can be further improved by using high purity starting material, optimization of the alloy composition, optimization of the MA parameters, optimization of the processing parameters during primary consolidation (HIP) and secondary processing (extrusion), and subsequent heat treatment. The addition of reinforcement, such as CNTs or B_4C, should further increase the strength of the material. Based on these initial results, we expect that the proposed refinements will put the target tensile strength of 600 MPa (87 ksi) and tensile failure strain greater than 5% at room temperature well within reach. We have demonstrated the capability to produce bulk, fully dense material with an average grain size significantly less than the target value of 100 nm.

2.2 Phase I Results Discussion

2.2.1 Baseline Material - Coarse-grained Mg and Mg alloys

Powder Selection

We started the project with the characterization of the consolidation and mechanical properties of the as-received, coarse-grained powders to establish the baseline properties of the matrix material. We will then be able to quantify the enhancement in the mechanical properties first as a result of the refinement in the microstructure and second as a result of the addition of an extrinsic reinforcement, such as CNTs or B_4C.

We selected pure Mg powder and two Mg alloys, WE43 and E21. All powders were obtained from Magnesium Elektron. We fabricated HIP canisters with 1½-in OD, filled them with the different powders, degassed them at a maximum temperature of 325°C, and sealed them under vacuum. At least two HIP canisters were made from each powder.

Powder Consolidation

The HIP canisters were consolidated in our instrumented-HIP. Using our High Temperature Eddy Current Sensor (HiTECS) we are able to monitor the change in diameter of the compact during densification and identify the temperature, pressure and time at which full densification has occurred.

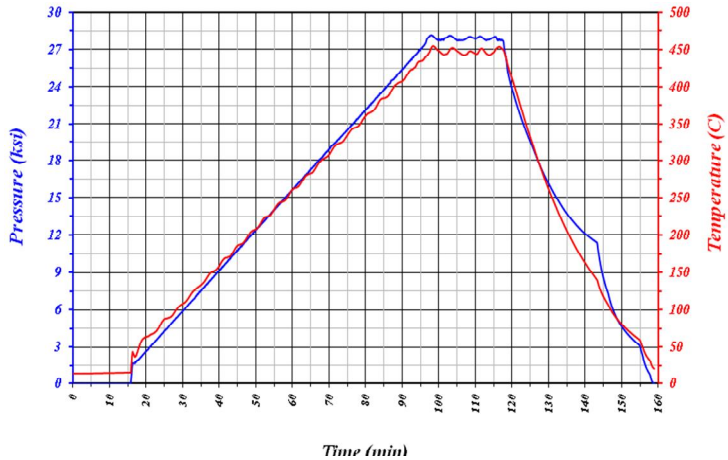
The results from instrumented-HIP experiments for the as-received powders are illustrated in Figure 2 for pure Mg powder and in Figure 3 for WE43 powder. For pure Mg, the HIP schedules are illustrated in Figure 1(a) and the diameter measurements are illustrated in Figure 2(b) and (c). The temperature and pressure were ramped simultaneously to 450^{o}C and 28 ksi (193 MPa), respectively. The densification increases significantly when the temperature reaches 75^{o}C, and all densification occurs by the time the temperature reaches 325^{o}C. The curve for the diameter measurement remains flat above 325^{o}C, an indication of no additional densification as the temperature is further increased to 450^{o}C. The HIP was then cooled to room temperature. Additional compacts were HIPed at 325^{o}C and a measurement of the density of the material after HIP indicated that the material is fully dense, as indicated by the sensor measurement.

The same approach was used for WE43 and the densification response was similar except that the densification rate for WE43 was higher than that for pure Mg. Again full densification occurs by the time the temperature reaches 325^{o}C and the curve for the diameter measurement remains flat above this temperature, an indication of no additional densification as the temperature is further increased to 450^{o}C. The HIP was then cooled to room temperature.

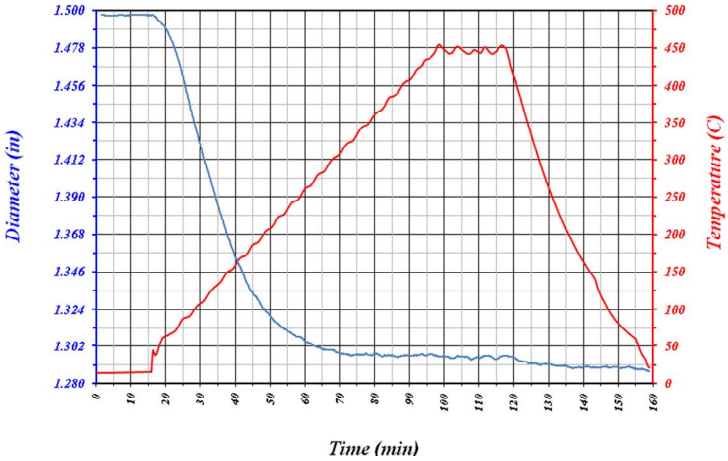
Mechanical Characterization

Tensile and compression specimens were fabricated from the fully dense compacts. The tensile specimen geometry is illustrated in Figure 4. We used EDM to cut multiple cylindrical rods from each compact. Dogbones were then machined from these rods using a CNC milling machine. This dogbone geometry was selected because it minimizes the residual stresses during machining and reduces the effects of any defects on the material response. Cylindrical specimens with a length to diameter ratio of 1.5 were made for compression tests. Three tensile and three compressive tests were performed for each material. Typical stress-strain curves from the tensile tests are illustrated in Figures 5 and 6, while typical stress-strain curves from the compression tests are illustrated in Figure 7.

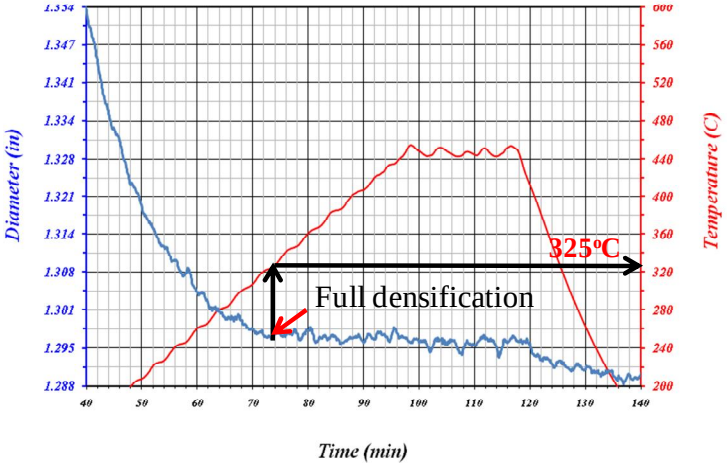
The as-HIPed pure Mg had tensile strength and ductility that matched published data. The tensile strengths for WE43 and E21 are very similar, with WE43 showing significantly higher ductility than E21. The compressive strengths are very similar.



(a) HIP schedules

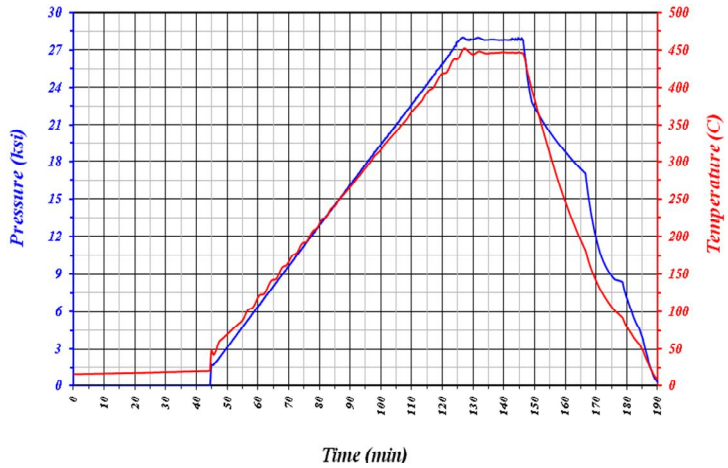


(b) Specimen diameter measurements

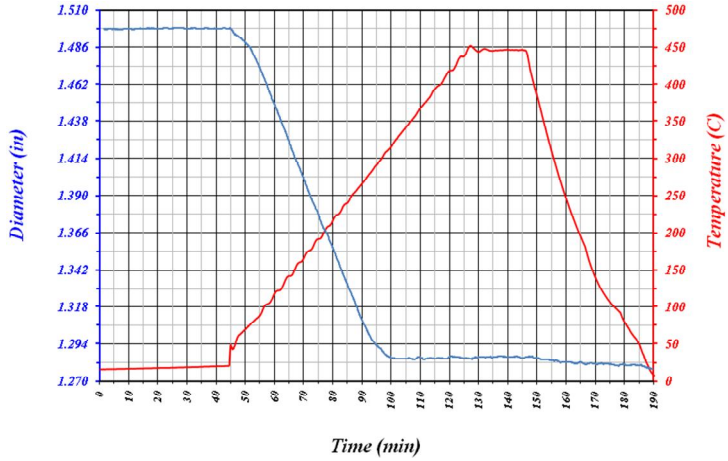


(c) Close-up of diameter measurements near full densification

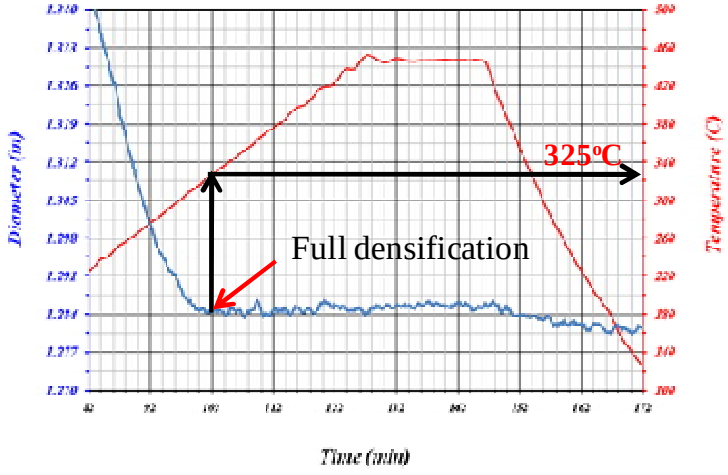
Figure 2. Instrumented-HIP data for pure Mg, as-received powder. The actual measurements of HIP temperature and pressure are illustrated in (a), and the diameter measurements are illustrated in (b) and (c). An examination of the diameter measurements illustrated in (c) indicates that full density is achieved at 325°C.



(a) HIP schedules



(b) Specimen diameter measurements



(c) Close-up of diameter measurements near full densification

Figure 3. Instrumented-HIP data for WE43, as-received coarse powder. The actual measurements of HIP temperature and pressure are illustrated in (a), and the diameter measurements are illustrated in (b) and (c). An examination of the diameter measurements illustrated in (c) indicates that full density is achieved at 325°C.

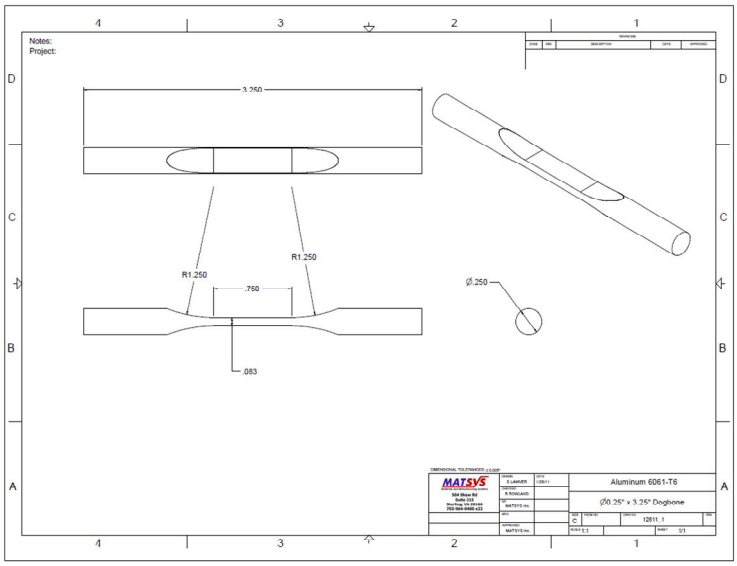


Figure 4. Tensile specimen geometry used for mechanical characterization.

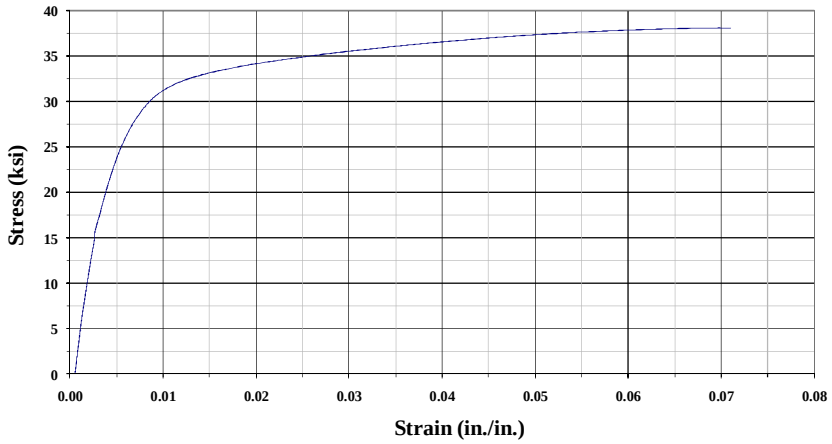


Figure 5. Tensile data for WE43, as-received coarse powder.

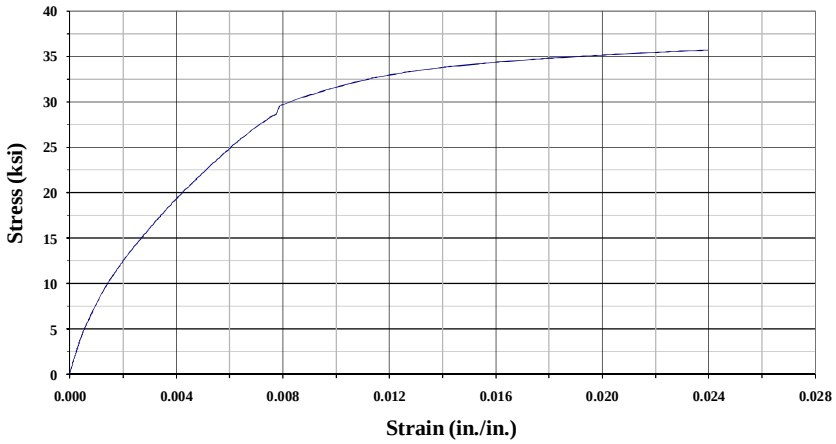
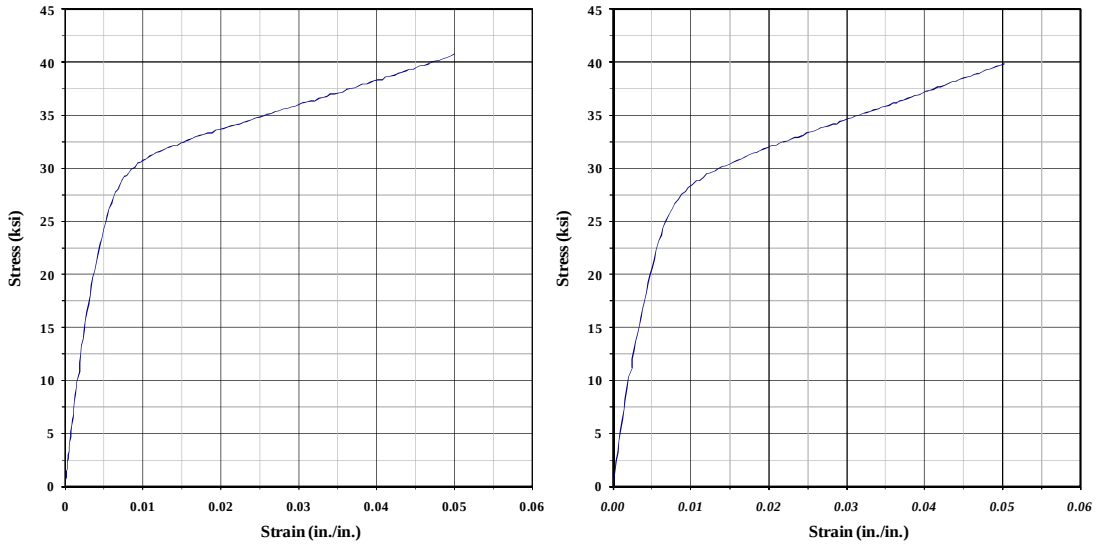


Figure 6. Tensile data for E21, as-received coarse powder.



(a) WE43 (b) E21

Figure 7. Compression data for Mg alloys, as-received, coarse powder.

2.2.2 Milled Material - Nanostructured Mg and Mg alloys

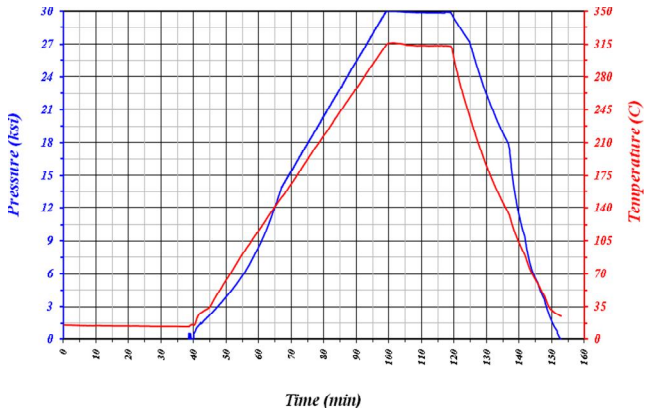
High Energy Milling

We used high energy milling with pure Mg, WE43 and E21 powders to refine the microstructure of these powders. The pure Mg was milled for ¼, ½, 1 and 2 hrs, respectively, to evaluate the effects of milling time on the microstructure and mechanical properties of the fully dense material. WE43 and E21 were milled for 1 hr. All these powders were packed in Al canisters with 1½-in OD, degassed at temperature and sealed.

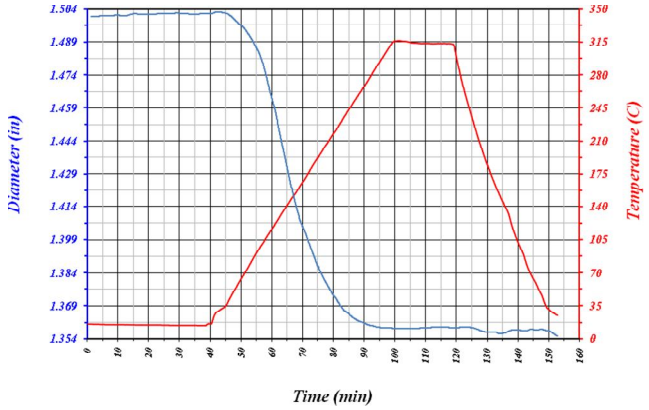
Powder Consolidation

The HIP canisters were consolidated in our instrumented-HIP. Typical results for WE43 powder milled for 1 hr are illustrated in Figure 8. The HIP schedules are illustrated in Figure 7(a) and the diameter measurements are illustrated in Figure 8(b) and (c). The temperature and pressure were ramped simultaneously to 315^{o}C and 30 ksi (207 MPa), respectively. The densification increases significantly when the temperature reaches 75^{o}C and all densification occurs by the time the temperature reaches 295^{o}C. The curve for the diameter measurement remains flat above 295^{o}C, an indication of no additional densification as the temperature is further increased to 315^{o}C. A measurement of the density of the material indicated that the material is fully dense.

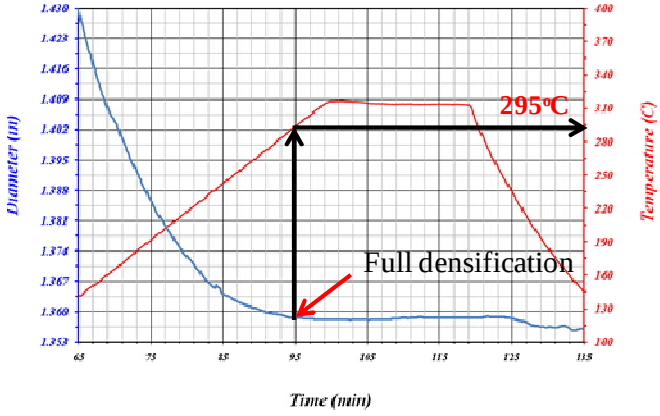
Full densification of nanostructured powders occurs at a lower temperature than the corresponding coarse-grained powder. The reduction in temperature is typically about 30^{o}C. This reduction in temperature helps to minimize grain growth and retain the nanostructure of the milled powder in the bulk material. All compacts were consolidated using the same approach and showed similar densification behavior. The compacts were then subjected to detailed metallographic and mechanical characterization.



(a) HIP schedules



(b) Specimen diameter measurements



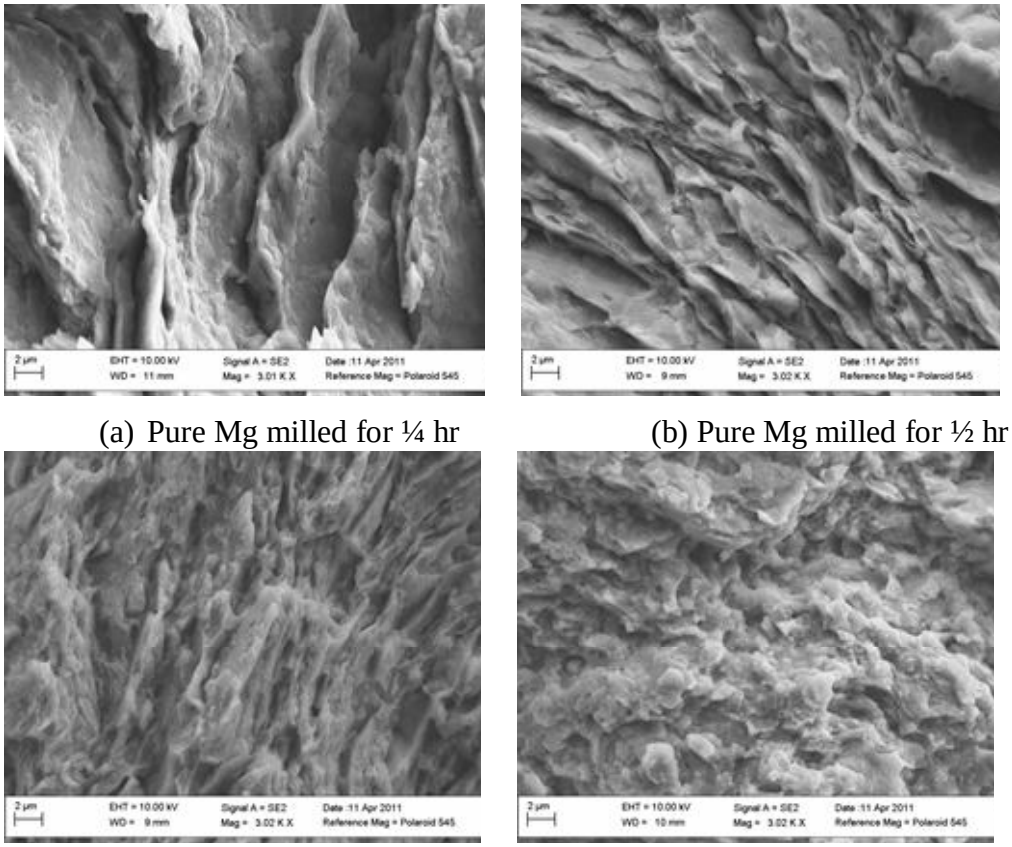
(c) Close-up of diameter measurements near full densification

Figure 8. Instrumented-HIP data for WE43 powder milled for 1hr. The actual measurements of HIP temperature and pressure are illustrated in (a), and the diameter measurements are illustrated in (b) and (c). An examination of the diameter measurements illustrated in (c) indicates that full density is achieved at 295°C.

Material Characterization

A metallographic examination of the pure Mg compacts showed that all the compacts were fully dense. Imaging of the fully dense material showed that the bulk material from pure Mg retained the nanostructure imparted during milling, as illustrated in Figure 9. The milling time affected the microstructure of the bulk material and its mechanical properties, with the highest tensile strength measured for the 1-hr milling time.

The mechanical characterization showed that the tensile strength of pure Mg increased with milling time up to 1 hr. The 1-hr milled powder has a tensile strength 58% higher than the corresponding coarse-grained material.

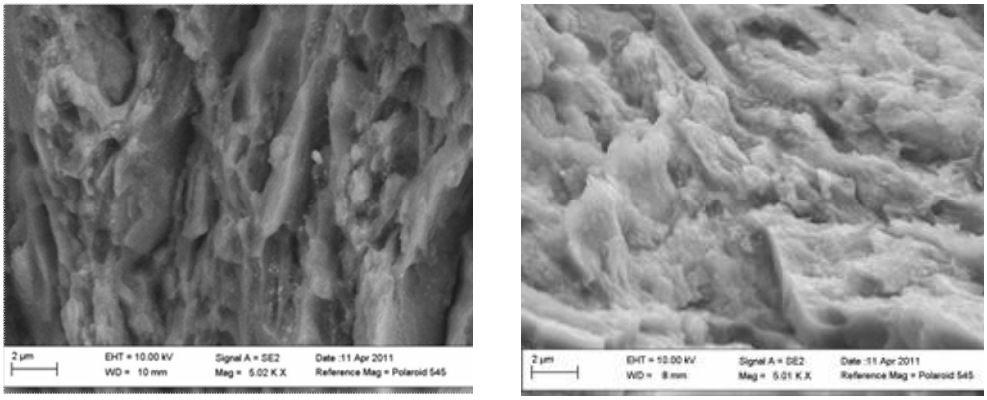


(a) Pure Mg milled for ¼ hr (b) Pure Mg milled for ½ hr

(c) Pure Mg milled for 1 hr (d) Pure Mg milled for 2 hrs

Figure 9. Bulk nanostructured Pure Mg was produced by high-energy milling of coarse Mg powder followed by instrumented-HIP. A refinement in the microstructure can be seen as a result of milling time. An increase in tensile strength was measured as a result of increased milling time up to 1 hr.

A metallographic examination of E21 and WE43 alloys showed that all the compacts were fully dense, and the fully dense material retained the nanostructure imparted during milling, as illustrated in Figure 10. However, we did not see the improvements in mechanical properties expected from the refinement in the microstructure, and we suspect that this is the result of impurities within the starting alloy powder.



(a) E21 powder milled for 1 hr (b) WE43 powder milled for 1 hr

Figure 10. Bulk nanostructured Mg alloy material was produced by high-energy milling of coarse powder followed by instrumented-HIP. While the bulk material retained the nanostructure of the milled powder, we did not see the improvements in mechanical properties and many impurities were observed.

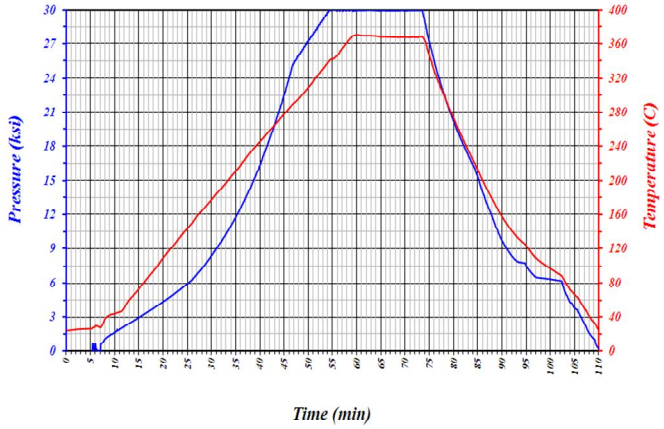
2.2.3 Pure Mg with CNTs

Mechanical Alloying

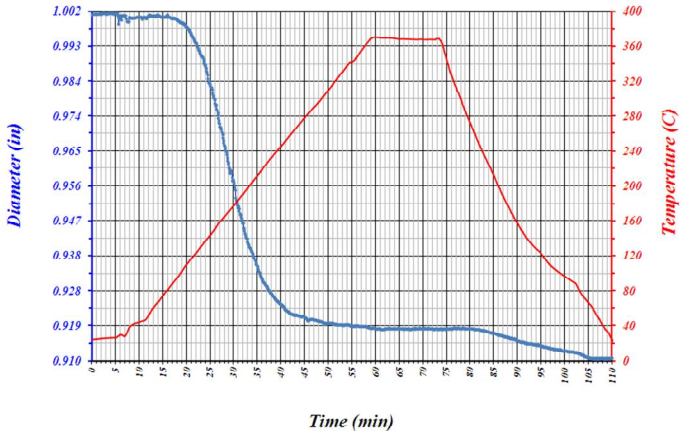
We used MA with a blend of ground Mg and 1wt% CNTs to prepare nanostructured composite powder. The powder blend was milled for 1 hr. The composite powders were packed in Al canisters with 1½-in OD, degassed at 350°C and sealed.

Powder Consolidation

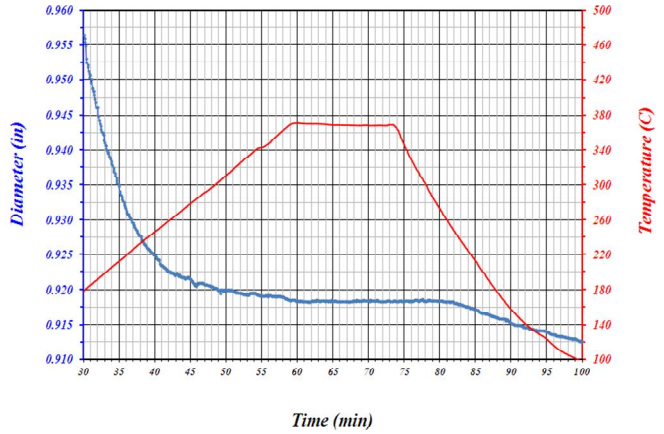
The HIP canisters were consolidated in our instrumented-HIP. Typical results are illustrated in Figure 11. The HIP schedules are illustrated in Figure 10(a) and the diameter measurements are illustrated in Figure 11(b) and (c). The temperature and pressure were ramped simultaneously to 375°C and 30 ksi (207 MPa), respectively. Full densification occurs as the temperature approaches 375°C. The curve for the diameter measurement remains flat during the hold at 375°C. One of the HIPed compacts was then extruded at 400°C and an extrusion ratio of 10.



(a) HIP schedules



(b) Specimen diameter measurements



(c) Close-up of diameter measurements near full densification

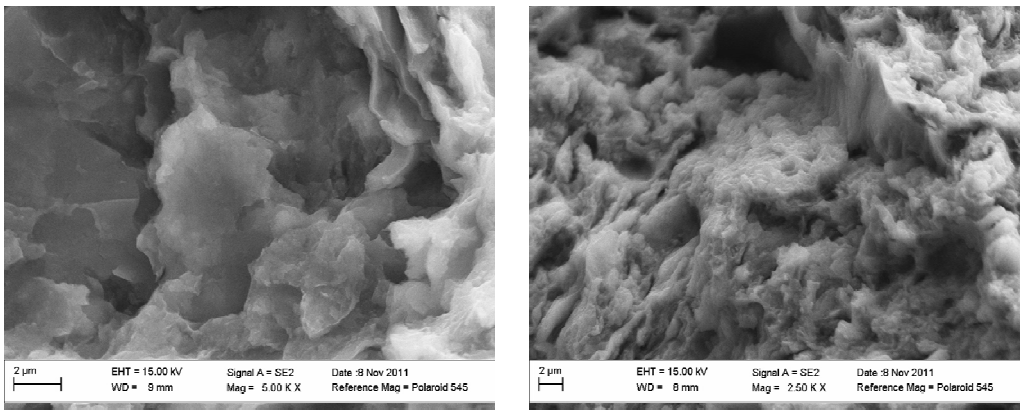
Figure 11. Instrumented-HIP data for pure Mg with 1 wt% CNTs milled for 1hr.

The actual measurements of HIP temperature and pressure are illustrated in (a), and the diameter measurements are illustrated in (b) and (c). An examination of the diameter measurements illustrated in (c) indicates that full density is achieved at 375^{o}C.

Material Characterization

A metallographic examination of the Mg-CNTs compacts showed that all the compacts were fully dense. Imaging of material showed that the bulk material retained the nanostructure imparted during milling, as illustrated in Figure 12. The extruded material has an average grain size of 61 +/- 3 nm.

The as-HIPed material has a compressive strength of 220 MPa (32 ksi) and a tensile strength of 114 MPa (16.5 ksi). This primary consolidation process was followed by extrusion to break the oxide layer. The compressive strength of the extruded material is 258 MPa (37.4 ksi) and its tensile strength was 171 MPa (24.8 ksi), a significant improvement in tensile strength. However, the tensile failure occurred outside the gage area (within the shoulder of the dogbone specimen). This type of failure is the result of introducing defects during specimen preparation as well as the material being brittle. The HIPed compact had to be encased in an Al sleeve to match the size of the available extrusion die. After extrusion, the Al sleeve had to be machined off the extruded material and the final extruded bar had a small diameter. As a result, machining of the dogbone specimens became difficult and that can result in defects within the specimen that can lead to a premature, brittle failure of the material. Larger HIP compacts are planned to eliminate any uncertainty related to the size of the tensile specimens.



(a) As-HIPed (b) HIPed then extruded

Figure 12. Bulk nanostructured pure Mg + 1 wt% CNTs material produced by instrumented-HIP and extrusion. The bulk material retained the nanostructure of the milled powder, and a 50% increase in tensile strength was observed.

2.2.4 High Strength Mg Alloy - Mg-2y-1Zn

A breakthrough has been made within the past four months to guide the future direction of the project. We selected a high strength Mg alloy developed by Inoue and his co-workers [1-3]. Their work on a rapidly solidified nanocrystalline Mg-2Y-1Zn alloy showed very promising results. The tensile strength and elongation, which were dependent on consolidation temperature and heat treatment, were in the range of 480 to 610 MPa and 5 to 16%, respectively.

We have been able to prepare Mg-2Y-1Zn alloy powder by MA, perform instrumented-HIP to achieve full density, and then perform extrusion as a secondary processing to improve the mechanical properties. We measured the hardness for the as-HIPed material at 2 GPa and the compressive strength at 550 MPa (79.8 ksi), but the tensile strength was 142 MPa (20.6 ksi) and the material was very brittle. Following secondary processing by extrusion, the bulk nanostructured material had an average grain size of 40 nm, a compressive strength of 634 MPa (91.9 ksi) and a tensile strength of 285 MPa (41.3 ksi). No attempt has yet been made at optimization of the extrusion parameters and no heat treatment has been performed.

We consider these results to be a breakthrough because they strongly suggest that this Mg alloy by itself or with CNTs or B_4C reinforcement will have properties that exceed the requirements for a tensile strength greater than 600 MPa (87 ksi) and a tensile failure strain greater than 5% at room temperature. These significant results were achieved using the following approach.

Mechanical Alloying

We used mechanical alloying to process a blend of the elemental constituents of the Mg-2Y-1Zn alloy. Elemental powders of Mg (99.8% pure, -200 mesh), Y (99.9% pure, -325 mesh), and Zn (99.8% pure, -325 mesh) were initially blended and subsequently mechanically alloyed in a high energy attritor for 12 hours. The composition was as follows:

	Atomic Percent	Weight Percent	Volume Percent
Mg	97	90.7	96.5
Y	2	6.8	2.8
Zn	1	2.5	0.7

Stearic acid was used as a process control agent to minimize cold welding of the powder. It is important to note that the high energy attritor is hermetically sealed and all processing was conducted under argon atmosphere.

Analysis of the mechanically alloyed powder included electron microscopy (SEM), X-ray diffraction, X-ray fluorescence, and Vickers microhardness. SEM provided a general view of how the powder morphology changed as a function of milling time, while XRD offered insight into how the material was alloying. XRF verified that iron contamination from milling is minimal and should not significantly affect the alloy properties. Microhardness measurements were conducted on green pressed pellets under a 10g load and 20 second dwell time. The results are summarized in Figure 13.

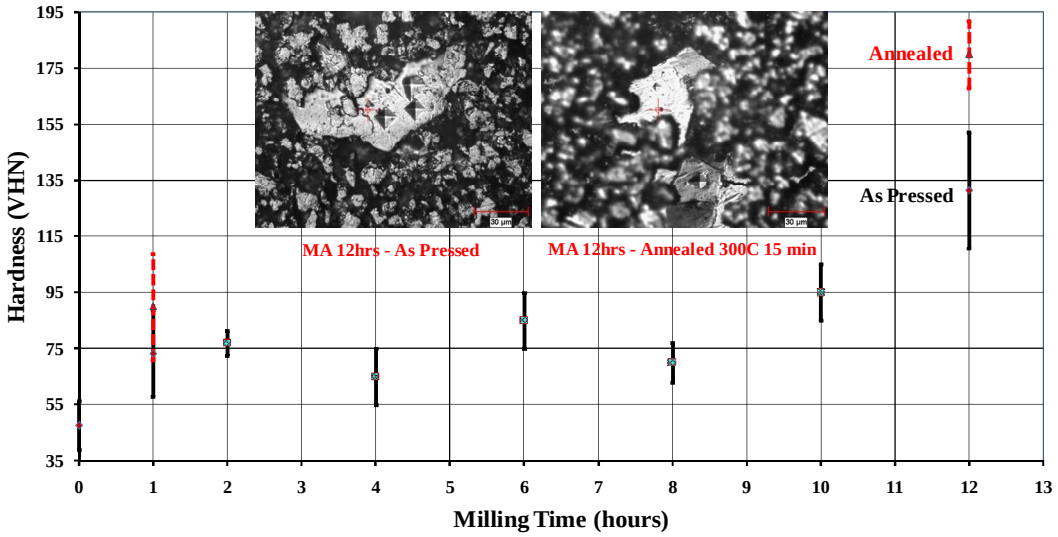
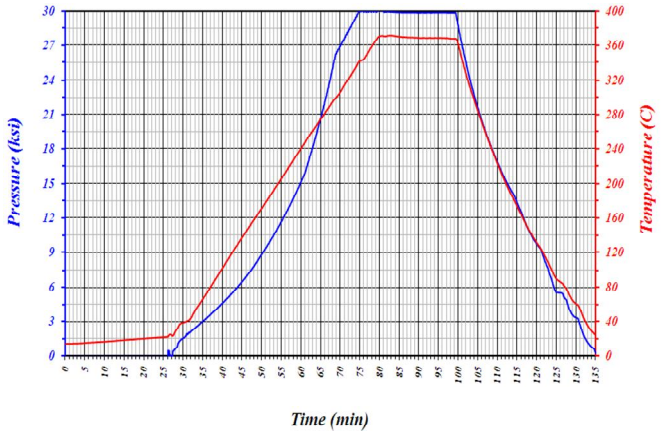


Figure 13. Effect of milling on the microhardness of the powder. An average hardness of 1.3 GPa was measured for the 12-hour, as-alloyed powder, and increased to 1.8 GPa upon annealing at 300°C.

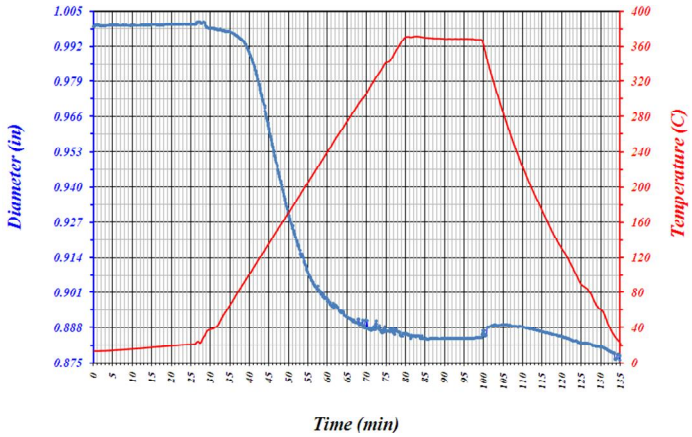
The average hardness was about the same for milling times up to 8 hours, with a significant increase as the milling time was increased to 12 hours. This is significant because milling times are typically 4 to 8 hours and we needed to increase the milling time to 12 hours to improve the properties. An average hardness of 1.3 GPa was obtained for the 12-hour, as-alloyed powder. Upon annealing at 300°C, the average hardness increased to 1.8 GPa. It is interesting to note that the hardness of the milled powder increased as a result of annealing.

Powder Consolidation

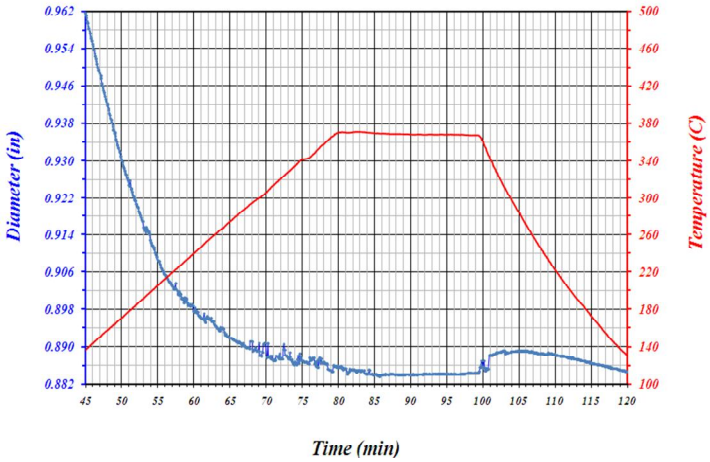
HIP canisters were filled with the powder milled for 12 hours, degassed and sealed. To investigate the effects of degassing temperature (to remove the stearic acid) on the mechanical properties of the material, HIP canisters were degassed at 250, 300 and 350°C. The HIP canisters were then consolidated in our instrumented-HIP. Typical results are illustrated in Figure 14. The temperature and pressure were ramped simultaneously to 375°C and 30 ksi (207 MPa), respectively. The densification increases significantly when the temperature reaches 75°C. The densification slows as the temperature approaches 375°C. The curve for the diameter measurement remains flat during the hold at 375°C. An examination of the material showed that it is fully dense.



(a) HIP schedules



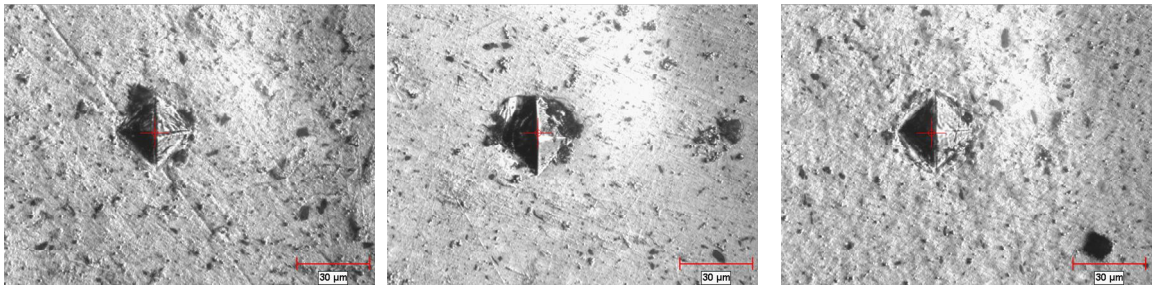
(b) Specimen diameter measurements



(c) Close-up of diameter measurements near full densification

Figure 14. Instrumented-HIP data for Mg-2Y-1Zn milled for 12 hours. The actual measurements of HIP temperature and pressure are illustrated in (a), and the diameter measurements are illustrated in (b) and (c). An examination of the diameter measurements illustrated in (c) indicates that full density is achieved at 375^{o}C.

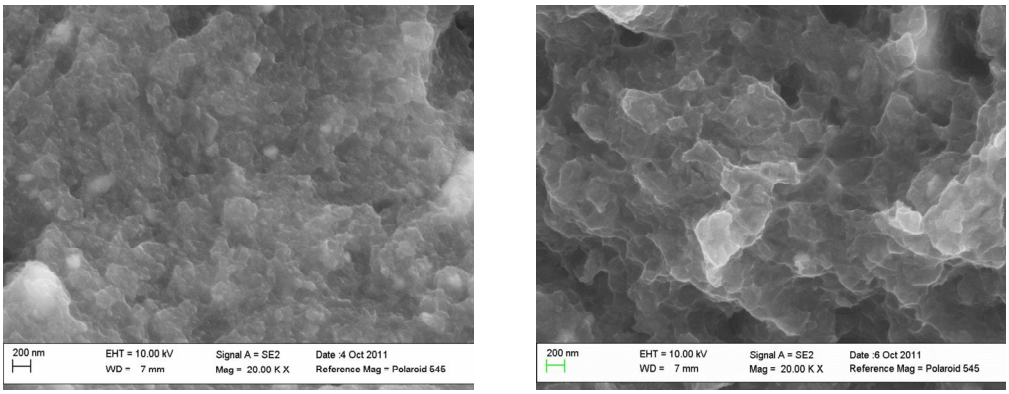
The compacts degassed at three different temperatures were consolidated using the same HIP schedules and showed very similar densification behavior. The compacts were then subjected to detailed metallographic and mechanical characterization. The highest average hardness is 2 GPa and was observed for the HIPed material degassed at 350°C, as illustrated in Figure 15. As such, the higher degassing temperature is required.



MG21 - 211VHN (a) 250°C | MG22 - 220VHN (b) 300°C | MG23 - 225VHN (c) 350°C

Figure 15. Hardness measurements for HIPed specimens as a function of degassing temperature. A small increase in hardness is observed as the degassing temperature is increased. The degassing temperature needs to be at 350°C to remove the stearic acid. The average hardness for the HIPed specimens is higher than reported by Inoue and his co-workers [1-3].

A metallographic examination of the compacts showed that all the compacts were fully dense. Imaging of the material showed that the bulk material retained the nanostructure imparted during milling, as illustrated in Figure 16. The extruded material has an average grain size of 40 nm.



(a) As-HIPed (b) Extruded at 350°C

Figure 16. Microstructure of the Mg-2Y-1Zn alloy as-HIPed and extruded at 350°C. The nanostructure of the starting powder has been retained in the bulk material after HIP and after extrusion.

Mechanical testing of the as-HIPed material showed a compressive strength between 490 MPa (71 ksi) for material degassed at 250°C and 550 MPa (79.8 ksi) for material degassed at 350°C. However, the tensile strength was low and the material was very brittle. As such, secondary processing is required to improve the mechanical properties.

Following extrusion, the material had a compressive strength of 634 MPa (91.9 ksi) and a tensile strength of 285 MPa (41.3 ksi).

Again, the tensile failure occurred outside the gage area (within the shoulder of the dogbone specimen). This type of failure is the result of introducing defects/imperfections during specimen preparation as a result of the small diameter of the extruded specimen as well as the material being brittle. The HIPed compact had to be encased in an Al sleeve to match the size of the available extrusion die. After extrusion, the Al sleeve had to be machined off the extruded material and the final extruded bar had a small diameter. As a result, machining of the dogbone specimens became difficult and that can result in defects within the specimen that can lead to a premature, brittle failure of the material. Larger HIP compacts are planned to eliminate any uncertainty related to the size of the tensile specimens. We also need to examine the effects of heat treatment on the tensile strength and ductility of the material.

In summary, we made significant progress within the past four months and achieved a breakthrough that clearly highlights the next steps required for the successful development of Mg-based composites. We consider these initial properties to be extremely encouraging, especially that no attempt has yet been made at optimization of the alloy material composition, the reinforcement, and the processing parameters for HIP and extrusion. In addition, no heat treatment has been attempted. As such, the requirements for a nanostructured composite with an average grain size less than 100 nm, a tensile strength greater than 600 MPa and a tensile failure strain greater than 5% at room temperature are well within reach.

3. REFERENCES

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2. Matsuura, M., Sakuria, M., Amiya, K., and Inoue, A., "Local structures around Zn and Y in the melt-quenched $Mg_{97}Zn_1Y_2$ ribbon," *Journal of Alloys and Compounds* 353 (2003) 240-245.
3. Abe, E., Kawamura, Y., Hayashi, K., and Inoue, A., "Long-period ordered structure in a high-strength nanocrystalline Mg-1 at% Zn-2 at% Y alloy studied by atomic-resolution Z-contrast STEM," *Acta Materialia* 50 (2002) 3845-3857.