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Hoeganaes Corporation - USA

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Cover: Grand Prize–winning parts from MPIF’s 2009 Design Excellence Awards Competition

The International Journal of Powder Metallurgy (ISSN No. 0888-7462) is a professional publication serving the scientific and technological needs and interests of the powder metallurgist and the metal powder producing and consuming industries. Advertising carried in the Journal is selected so as to meet these needs and interests. Unrelated advertising cannot be accepted.

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Notwithstanding the major global economic downturn, and its direct and indirect impacts on the PM industry, PowderMet2009 will go on record as an unqualified success by the 600-plus delegates who experienced a high-quality technical program embracing the PM and particulate materials spectrum. The special interest program on developments in titanium PM drew a “standing room only” audience with the expectation that this technology is poised for further commercialization and market penetration.

In this post-show issue of the Journal, the text of the “State of the PM Industry in North America—2009,” presented by MPIF President Mark Paullin, is included. Also, Peter Johnson reviews the “2009 PM Design Excellence Awards Competition.” The Grand Prize–winning parts are shown on the front cover.

As a further postscript to PowderMet2009, the four Axel Madsen/CPMT Scholar reports again confirm the value of this program in encouraging students to learn more about PM technology and to consider a professional career in the PM industry. In different ways, each of the grantees derived tangible and indirect benefits by attending the conference and exhibition.

International consultant Olle Grinder is the author of the “Consultants’ Corner.” Readers’ questions he addresses focus on the projected growth of hot isostatic pressing (HIP), the mechanical properties of HIPed metals compared with their cast/wrought counterparts, and materials selection for PM tooling in the compaction of soft magnetic materials.

In the “Research & Development” section, Jozwiak et al., examine the exothermic self-propagating high-temperature synthesis (SHS) reaction that occurs during the sintering of elemental mixes of iron and aluminum powder. The study confirms that the SHS reaction temperature and enthalpy are a function of the aluminum content, and that formation of the intermetallics (FeAl3, Fe2Al5, and FeAl) is complex.

Recently, I read a paper published in Metallurgical and Materials Transactions on the processing, microstructure, and properties of iron-based metallic glasses utilizing laser-engineered net shaping. To the author’s knowledge, the study represents the first time that the fabrication of net-shaped bulk metallic glass components has been demonstrated by laser direct deposition. In light of its potential interest to readers of the Journal, the article is reproduced in the “Research & Development” section.

I am usually cautious when opening e-mail communications from unknown sources. However, the title of a recent, somewhat macabre, entity “On the Highway to Heaven,” piqued my curiosity. Invented by Steve Radz, the idea of a Mobile Cremation Urn came to him as an epiphany in a dream! An aspiring funeral director, Radz developed the concept into a unique patented product which his company manufactures: galvanized, chrome- or nickel-plated, and powder-coated urns in a variety of colors—clearly an opportunity here for the metal powder producers! These Final Ride Urns are made to last several lifetimes, braving the elements and the open road, keeping the memory of a loved one alive, close, and cruising. Though developed for the motorcycle, the urns can be mounted on any vehicle, for example, a golf cart. The rider can, of course, also stay close to past four-footed friends with these urns. Happy riding. For more information visit: www.1FinalRide.com.
June 27–30
The Westin Diplomat
Hollywood (Ft. Lauderdale), Florida

For complete program and registration information contact:
METAL POWDER INDUSTRIES FEDERATION ~ APMI INTERNATIONAL
105 College Road East, Princeton, New Jersey 08540 USA
Tel: 609-452-7700 ~ Fax: 609-987-8523 ~ www.mpif.org
Höganäs Powder Sales Sag
Swedish metal powder producer Höganäs AB reports first quarter 2009 sales declined 42 percent to MSEK 916 (about $112 million). Production volumes fell sharply in all regions.

Chinese PM Industry Results
PM parts production in China declined five percent to 102,048 short tons last year, reports the PM Association of China, Beijing. Iron-base parts production declined to 95,706 short tons while copper parts production weakened slightly to 6,342 short tons.

New Resource for Metal Injection Molding Information
The Metal Injection Molding Association (MIMA), one of the six federated trade associations of the Metal Powder Industries Federation (MPIF), has launched a new industry-funded Web site, mimaweb.org, to promote the benefits of metal injection molding (MIM) as a part-manufacturing technology.

Miba Advances amid Softening Sales
Miba AG, Laakirchen, Austria, announced a 2.1 percent increase of fiscal year 2008-09 sales to 374.6 million (about $508 million), despite a sharply declining fourth quarter. Based on the slowdown in the automotive market, the Sinter (PM parts) Group reported a 15 percent sales drop to 135.4 million (about $183 million).

New Powder Production Line Increases Capacity
Tekna Plasma Systems Inc., Sherbrooke, Québec, Canada, has installed a new induction plasma production line for metal and ceramic powders. Chiefly devoted to spherical cast tungsten carbide (SWC) powder and customized powder treatments, the new line increases Tekna’s annual capacity to more than 250 metric tons.

GKN Sales Decline
Automotive giant GKN plc, London, U.K., reports an eight percent drop in first quarter sales to £1,085 million (about $1.6 billion). However, excluding the acquisition of Filton from Airbus, group sales fell by 33 percent.

Kennametal Sells High-Speed Steel Line to Chinese Company
Kennametal Inc., Latrobe, Pa., has agreed to sell its high-speed drills, related product lines, and assets to Top Eastern Drill Co., Ltd. (TDC), a member of the Top-Eastern Group, Dalian, China. The transaction includes four facilities in the U.S., Mexico, and China, as well as 400 employees.

Metaldyne Joins Bankruptcy Parade
Metaldyne, Plymouth, Mich., joins a long line of automotive parts suppliers filing for Chapter 11 bankruptcy, a direct result of faltering North American vehicle sales and production. The filing does not include the company’s non-U.S. operations or Asahi Tec Corp., its parent company.

H.C. Starck Signs Refractory Metal Supply Agreement
H.C. Starck Inc., Newton, Mass., and Comet Network Co., Ltd., Seoul, South Korea, have reached a long-term agreement for the supply of refractory metal for sputtering targets. Starck will supply molybdenum materials from its plant in Euclid, Ohio, and will make additional capacity investment there as necessary to meet Comet’s requirements.

New PM Development Facility
TAT Technologies, Inc., Summit, N.J., will open a Technology Development & Training Center in a recently acquired 5,000 sq. ft. building located in the Stackpole complex in St. Marys, Pa., reports Harb S. Nayar, president. The facility will be used for technology development, new equipment demonstration, pilot-run processing, and hands-on training in sintering, delubing/debinding, annealing, and brazing and other thermal processes.

High-Alumina Bricks for PM Furnaces
Sunrock Ceramics Company, LLC,
Chicago, Ill., offers 99.5 percent alumina bricks and other shapes for PM furnace applications, which have been field tested recently in the U.S. and Europe. The high-density material, designated HPA-99, is ideal for hot-face linings in high-temperature hydrogen-atmosphere sintering furnaces, the company reports.

**SCM Buys Brazing Business**

SCM Metal Products, Inc., Research Triangle Park, N.C., has purchased the brazing product line from Tricon Industries, Downer's Grove, Ill. The asset purchase included all of Tricon’s equipment, customer list, formulations of copper, copper alloy and nickel-based brazing pastes, and intellectual property.

**Automotive PM Parts Maker Reports Sales and Earnings Slump**

Miba AG, Laakirchen, Austria, reports a 27 percent decline in its fiscal first quarter sales to 74.3 million euros (about $103 million). Earnings plunged to 1.2 million euros (about $1.7 million) compared to 13.3 million euros (about $18.5 million) during the same period in 2008.

**Industry Leaders Meet in Las Vegas**

Executives, technical managers and researchers from metal powder producers, PM parts and products makers, equipment builders, and universities are gathered in Las Vegas for the PowderMet2009 International Conference on Powder Metallurgy & Particulate Materials. Mark C. Paullin, MPIF president, and C. James Trombino, MPIF executive director/CEO, welcomed the delegates at the opening general session on June 29.

**Industry Recognition Awards**

C. James Trombino, MPIF executive director/CEO, announced the MPIF Board of Governors’ decision to rename the annual Outstanding Technical Paper Award in honor of the late Howard Sanderow, well-known consultant and powder metallurgist. “The Howard I. Sanderow Outstanding Technical Paper Award recognizes excellence in scientific and technical written communication and now carries, in perpetuity, the legacy of a
truly great technical contributor to our conferences,” Trombino said. The award was presented to Chris Schade and Thomas F. Murphy, Hoeganaes Corporation, and Alan Lawley and Roger Doherty, Drexel University, for their paper presented at the 2008 PM World Congress, “Development of a Dual-Phase Precipitation-Hardening PM Stainless Steel.”

The MPIF Distinguished Service to Powder Metallurgy Award, which recognizes contributions of individuals who have done excellent work in the PM industry for at least 25 years, was presented to the following industry professionals: Gary L. Anderson, John C. Hebeisen, Thomas J. Jesberger, Shiz Kassam, Lou Koehler, Kalathur S. Narasimhan, FAPMI, Charles L. Rose, John A. Shields, Jr., Thomas L. Stockwell, Jr., Ted A. Tomlin, and Robert F. Unkel.

PM Industry Trends
In the annual State of the PM Industry presentation at PowderMet 2009 in Las Vegas, MPIF President Mark C. Paullin reports that North American iron powder shipments declined 19 percent in 2008 to 327,272 short tons. Total metal powder shipments declined 18.4 percent to 415,427 short tons.

Outstanding PM Parts Applications
Winners of the 2009 Powder Metallurgy Design Excellence Awards Competition sponsored by the Metal Powder Industries Federation were announced at PowderMet2009. Receiving grand prizes and awards of distinction, the winning parts are outstanding examples of powder metallurgy’s (PM) precision, performance, complexity, economy, and innovation.

Ametek Acquires Hoeganaes Stainless Steel Powder Business
AMETEK Specialty Metal Powders (SMP), Eighty Four, Pa., has purchased the ANCOR® Specialties stainless steel powder business from Hoeganaes Corporation, Cinnaminson, N.J. The acquisition includes the customer list and an annealing furnace that will be moved to the Eighty Four plant.

New PM Machining Operation Opens
Super Abrasive Machining Innovations LLC (SAMπ) offers machining services for as-sintered and as-heat-treated materials at its new 7,500 sq. ft. facility in Ridgway, Pa., reports Rocco Petrilli, CEO of Prima Business Specialists LLC and SAMπ. The company provides high-volume precision grinding at speeds and costs similar to those generated by single-point machining that will enhance net shaping.
SPOTLIGHT ON ...

SHAWN METCALFE, PMT

Education:
Chemical Engineering Technology Diploma, Mohawk College, 2001

Why did you study powder metallurgy/particulate materials?
My job often involves the examination of the influence of a specific material on an entire process. Thus, it was necessary to be aware of the wide variety of pertinent technologies and manufacturing steps. At the beginning, I found both the Basic PM Short Course and the PMT certification program to provide an excellent foundation.

When did your interest in engineering/science begin?
I have always been interested in science. Even at a young age I was interested in general science and the solar system. I did not realize that this was to be my chosen career path until my last years in high school. Under a government co-op program I worked in a coal-fired hydro generating station. Working in an industrial laboratory with hands-on experience in chemistry persuaded me to continue my education in a related field.

What was your first job in PM? What did you do?
I assembled a small apparatus, which involved spreading powder mixes over a fine- mesh screen and then exposing the powder to a gas stream. This was followed by analyzing the composition and the weight loss to determine their impact on powder mixes during both handling and transportation.

Describe your career path, companies worked for, and responsibilities.
Most of my work experience has been with Vale Inco Limited. I started as a technician for the Hydro-metallurgy Research Group on a one-year co-op term. The work focused on the extraction of nickel, cobalt, and precious metals from the ore. I found this challenging and interesting, as the entire industrial process was reproduced on a laboratory scale. After completing college, I accepted a position at Vale Inco in electromagnetic shielding research. This work focused on technologies for plating nickel onto carbon substrates and then incorporating the product into plastic injection molding. I then transferred to the Battery Research Group which gave me my first experience with metal powders. My interest in this field grew and I transferred to the PM group, and have been with this group for four years. My work focuses on customer support, product application development, and technical support for our operations.

What gives you the most satisfaction in your career?
I get the most satisfaction from the constant learning experience provided by working on materials preparation, and the testing of PM steels, hardmetals, diamond tool binders, and heavy alloys, involving practically every step in PM manufacturing.

List your MPIF/APMI activities.
Vale Inco Limited supports participation in short courses, and attendance and presentation of technical papers at MPIF conferences, as well as the PMT certification program.

What major changes/trend(s) in the PM industry have you seen?
The sharp rise in raw-materials prices a few years ago put pressure on the selection of alloying elements in
high-strength steels. Now the situation has reversed, with the world economy struggling and raw-materials costs at below historical averages. This presents high-strength PM steels with an opportunity to expand into parts that have historically been uncompetitive with alternative metal-forming technologies. I see alloying content in future PM parts being optimized to allow parts manufacturers the ease of processing to higher densities, which can grow PM’s advantage and market share.

Why did you choose to pursue PMT certification?
PM is a broad field and with each project I was limited in knowledge based on my experience alone. PMT certification offered an avenue to expand my knowledge across many scientific fields and to test myself. This has improved my ability to communicate with customers and to make me aware of the advantages and limitations of the PM process, from start to finish.

How have you benefited from PMT certification in your career?
It has encouraged me to become familiar with areas of PM that are outside those of my work responsibilities. This has helped me to better communicate with customers and colleagues.

What are your current interests, hobbies, and activities outside of work?
I enjoy taking on renovation projects. These are frequently not your normal projects, and often expand into weeks of planning and work, and have included building, with friends, an entire house, from the foundation up. When not renovating I generally spend time with my wife and close family.

Would you like to be featured here? Have you been PMT Certified for more than 2 years? Contact Dora Schember (dschember@mpif.org) for more information.
Which powder metallurgy (PM) technology will achieve the highest growth in the next five years? What is the projected growth of selective net-shape hot isostatically pressed (HIPed) PM products over the next five years and what is driving this growth?

Before I answer this important and difficult question, it is appropriate to quote the Swedish author Falstaff Fakir (pseudonym), who 150 years ago stated: "It is difficult to foresee—especially about the future."

I want to limit my forecast to the five dominant powder-shaping and -compaction methods, i.e., uniaxial pressing, metal injection molding (MIM), powder forging (PF), HIPing, and laser/electron beam sintering. My firm belief is that the HIPing of high-alloy PM steels will see the highest growth rate of these five methods in the next five years. The HIP production volume of billets, semi-finished products, and near-net-shape (NNS) parts will continue to expand. There are several concurrent reasons to support my selection:

- Significant improvements and important developments in HIPing equipment, resulting in
  - Larger HIP units with a maximum load >30 mt
  - Faster cycle times—now down to 12 h
  - A 100% increase in the number of cycles/yr. over the last decade
  - A 65% decrease in the relative cost of HIPing over the last two decades
- Extensive investments in large and modern production facilities, e.g., MEGA HIP units by Bodycote Hot Isostatic Pressing AB, Sweden, and Kinzoku Giken, Japan, as well as in production facilities for inert-gas-atomized powders by Erasteel Kloster AB, Sweden.
- The recent introduction of high-performance steels developed specifically for the production (inert-gas atomization + HIPing) of nitrogen-alloyed tool steels and high-temperature/oxidation-resistant steels.
- New applications for near-net-shape parts and increased knowledge on how to design the capsules and run the HIPing process in order to obtain NNS parts with optimal shape and properties.

Thus, the projected increase in production in the coming five years is a direct result of improved equipment and processes, new and better materials, new applications, and decreased production costs, enabling the PM route to be more competitive.

The world production of specialty PM steels by HIPing can be estimated for 2008 and forecast for 2013, Table I.

<table>
<thead>
<tr>
<th>High-Speed Steels (HSS) and Tool Steels*</th>
<th>2008</th>
<th>2013</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Steels**</td>
<td>5,000</td>
<td>10,000</td>
</tr>
</tbody>
</table>

* Billets, semi-finished products (bars, tubes)
** NNS products

For metals, how do the mechanical properties of HIPed PM parts compare with those of cast or wrought products?

The mechanical and functional properties of HIPed PM metal parts are generally superior to those of cast parts, if the latter have the same chemical composition and have been heat treated in the same way as the HIPed PM parts. The cast parts contain defects such as inclusions, small

*PM Technology AB, Global PM Consultants, Drottning Kristinas Vag 48, S-114 28 Stockholm, Sweden; E-mail: grinder@algonet.se and Associate Professor, Royal Institute of Technology, Stockholm, Sweden
cracks, residual porosity, and often segregation. The extent and the negative effects of these imperfections vary from case to case and from metal to metal.

The HIP manufacture of NNS parts and semifinished products exhibits several inherent advantages from a material perspective:

- A fine and homogenous microstructure
- No segregation, which is important in carbide-rich steels such as HSS and tool steels
- Isotropic properties

These microstructural advantages lead to an improvement in both the mechanical and functional properties of HIPed metals compared with those of cast metals of the same shape and composition. An important benefit of the HIPed metals is that, being isotropic, they can be subjected to quality control (QC) by ultrasonic testing.

It is well known that, frequently, the functional properties of PM steels manufactured by HIPing surpass those of conventional steels. HIPed high-alloy duplex stainless steels have, in some cases, exhibited improved corrosion resistance as a direct result of the fine, homogenous microstructure. It has been reported that the risk of hydrogen-induced stress cracking (HISC) in subsea equipment can be eliminated by using a HIPed duplex stainless steel. Another, well-known example is the excellent performance of PM HSS in cutting operations. However, the negative effect of an inferior microstructure in cast metals can be reduced by subsequent hot- and cold-forming operations. This is especially effective in carbide-rich HSS and tool steels.

Of utmost importance is the fact that the HIP consolidation of inert-gas-atomized powders can be used in the development of new alloy systems with unique chemical compositions, microstructures, and functional properties. These alloys cannot be manufactured by conventional casting + forging due to their high alloy content, which results in segregation problems, or because of thermodynamic restrictions. Some examples of new PM base-alloy systems are:

- Nitrogen-alloyed HSS and tool steel grades
- Development of application-specific grades with improved wear and corrosion resistance
- High-temperature corrosion-resistant steels
- Compound metal systems
- Composite and dispersion-hardened alloys

Q

Is there a preferred material for the punch-es in the compaction of soft magnetic parts? The compaction pressure needed to densify to 7.30 g/cm$^3$ is ~800 MPA.

A

I refer to what I wrote in the “Consultants’ Corner” two years ago (Int. J. of Powder Metallurgy, 2007, vol. 43, no. 5, pp. 11-14) in relation to the choice of tool-steel grades in order to maximize tool life and to minimize tooling costs.

A number of parameters have a decisive influence on the choice of tooling material; these include:

- The shape of the part to be produced (will the tooling parts have thin sections, chamfers, or sharp corners?)
- Tolerances
- Number of parts to be produced
- Compaction pressure
- Powder grade, e.g., tendency for the powder to clad on the tool material
- Mechanical and functional properties of the tool steel or cemented carbide such as wear resistance, hardness, toughness, and machinability
- Material costs

Thus, the direct answer to your question is No. When you want to choose a tool steel, it is often logical to begin with an analysis, based on previous experiences, of the potential failure mechanism(s) of the punches while in operation (e.g., the risk of cracking), plastic deformation, abrasive/adhesive wear, or galling.

The next step is to choose a tool steel based on available data on the mechanical properties (hardness and toughness) of the steel. Two important figures are presented in the cited article and these relate impact energy to the hardness of the steel, and abrasive-wear resistance to hardness. These figures provide a guide on how to select your tool steel.

Readers are invited to send in questions for future issues. Submit your questions to: Consultants’ Corner, APMI International, 105 College Road East, Princeton, NJ 08540-6692; Fax (609) 987-8523; E-mail: dschember@mpif.org
Animesh has made important contributions and is widely recognized for his innovative work in materials processing. Having dedicated more than 25 years to the PM industry, he is internationally acknowledged for having helped spread the technology of powder injection molding (PIM) globally through licensing and technology transfer activities. He is also recognized for his contributions in alloy development, especially refractory metals, carbides, hardmetals, intermetallic compounds, and other advanced materials.

Animesh completed his BTech in Metallurgical Engineering and PhD in Engineering (Powder Metallurgy) from Indian Institute of Technology, Kharagpur, in 1982. He is President and CEO of Materials Processing, Inc., a company he co-founded in 1999 that is involved in PIM of hardmaterials, cermets, and advanced ceramics. His journey to North America commenced as Visiting Scientist, Rensselaer Polytechnic Institute, in 1985. He has been much traveled in his career including executive positions with Parmatech Corporation (director of R&D), Powdermet (executive VP), and Advanced Metalworking Practices, LLC (general manager).

A member of APMI for over 23 years, Animesh is a past member of the APMI Board of Directors. He has served as co-chairman for several international conferences including 6 MPIF conferences on tungsten, refractory, and hardmaterials. He has also been a member of numerous technical program committees. Animesh has published over 115 technical papers, and authored/co-authored several books, including Injection Molding of Metals and Ceramics. He is listed as inventor/co-inventor on 8 U.S. patents, 3 of which were placed under Government Secrecy Order due to their importance to national security. He is a member and Fellow of ASM International and a life member of PMAI (India).

The award was presented at PowderMet2009 in Las Vegas, Nevada.
OUTSTANDING POSTER AWARD

SOLID LUBRICANT-INFUSED TIN BRONZE–BEARING MATERIALS PRODUCED BY POWDER METALLURGY

Gregory A. Vetterick, Iver E. Anderson, and D.J. Sordelet
Iowa State University, Ames Laboratory
Ames, Iowa
The *International Journal of Powder Metallurgy* would also like to recognize the Posters of Merit from PowderMet2009:

**Effect of WC Particle Size on Microstructural and Mechanical Properties of WC-Reinforced Al Metal Matrix Composites**
Ahmet U. Söyler, Hasan Gokce, Mustafa L. Ovegoglu and Burak Ozal, Istanbul Technical University

**Effects of Hot Deformation on Aluminum-Silicon Powder Metallurgy Alloys**
Winston G. Mosher, W.F. Caley, G.J. Kipourous and D. Paul Bishop, Dalhousie University and Ian W. Donaldson, GKN Sinter Metals

**Comparison of Fatigue Resistance of Green-Machined Components vs. Components Machined After Sintering**
Alexandre Bois-Brochu and Bernard Tougas, Université Laval
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GRAND PRIZE WINNERS

The five parts selected as the Grand Prize winners are shown in Figure 1.

The Grand Prize in the automotive—engine category goes to Capstan, Inc., Carson, California, and its customer Jacobs Vehicle Systems, a division of Danaher, Bloomfield, Connecticut, for a PM steel manifold, Figure 2, assembled with a solenoid into the valve train of a heavy-duty diesel I-6 truck engine. The part assists with the activation of the “Jake Brake” system inside the engine cylinder head dur-

Figure 1. Grand Prize winners

The awards were presented at PowderMet2009 in Las Vegas, Nevada.

*Contributing Editor, International Journal of Powder Metallurgy, APMI International, 105 College Road East, Princeton, New Jersey 08501-6692, USA; E-mail: pjohnson@mpif.org
ing the exhaust cycle, reducing horsepower and performing a braking action to slow the vehicle. Made to a minimum density of 6.7 g/cm³, the manifold has a minimum yield strength of 345 MPa (50,000 psi) and an ultimate tensile strength of 414 MPa (60,000 psi). Its complex design features include the variation in thickness levels and cylindrical radius. Secondary operations include machining the solenoid bore and two port holes. The PM process provided an estimated 20% cost reduction over the alternative casting process.

ASCO Sintering Co., Commerce, California, received the Grand Prize in the hardware/appliances category for a lockset retractor assembly, Figure 3, made for Best Access Systems–Stanley Security Solutions, Indianapolis, Indiana. The PM steel assembly functions as the heart of the mechanism in a heavy-duty door lockset system. Its “3D puzzle” design of two identical halves not only satisfied various functional force-transfer modes and geometry requirements, but allowed ASCO to partner with equipment suppliers to develop a pick-and-place “green” stage assembly from two consecutive parts that allowed the customer to remove an entire riveting sub-assembly line at an annual cost savings of $250,000. The “single jaw” design easily withstands the required 2,222 N (500 lbf) load and meets an axial pull requirement. Made to a net shape, the assembly has a density of 6.7 g/cm³, 414 MPa (60,000 psi) tensile strength, 862 MPa (125,000 psi) transverse rupture strength, a fatigue limit of 159 MPa (23,000 psi), and 73 HRB hardness.

FMS Corporation, Minneapolis, Minnesota, and its customer Team Industries, Bagley, Minnesota, have earned the lawn & garden/off-highway Grand Prize for an assembly of five complex PM steel parts (two shift forks, two sector gears, and a park pawl) that go into an all-terrain vehicle transmission, Figure 4. The sector gears have AGMA Class 6 splines. Four of the parts are made from PM sinter-hardened steel to a density of 7.2 g/cm³ and have a minimum ultimate tensile strength of 759 MPa (110,000 psi). One sector gear, a net shape, is made from 4300 steel and has a tensile strength of 1,103 MPa (160,000 psi) and a 30 HRC minimum hardness. The other parts require only minimal machining for features such as the integrated pins, undercuts, and dimensional qualification on one of the shift tracks. The customer saved an estimated 60% by choosing PM over machined parts.
FloMet LLC. Deland, Florida, won the hand tools/recreation category Grand Prize for a 316L stainless steel compressed-air nozzle, Figure 5, made for Silvent AB, Borås, Sweden. Fabricated by the metal injection molding (MIM) process, the hollow nozzle consists of top and bottom halves that are molded separately and then joined together into one piece during debinding and sintering. The nozzle’s air-flow capacity is tightly controlled to ensure optimum use of compressed air as well as to comply with U.S. and European Union machine device noise regulations. It can withstand high ambient temperatures and corrosive environments, and meets hygienic requirements of the food processing industry. The complex part has a density of more than 7.6 g/cm³, an ultimate tensile strength of 517 MPa (75,000 psi), yield strength of 172 MPa (25,000 psi), a 50 percent elongation, 67 HRB hardness, and Charpy impact energy rating of 190 J (140 ft.-lbf). After sintering, the seams where the two sections join together are laser welded for a leak-free seal. Significant cost savings result from the elimination of scrap, machining, and secondary operations.

Advanced Materials Technologies Pte Ltd., Singapore, won the Grand Prize in the electrical/electronic components category for a 17-4 PH stainless steel MIM flagstaff nose or EMI nose shield, Figure 6, which serves as an external connector for a high-performance fiber-optic module. The part has a density of 7.5 g/cm³, a tensile strength of 897 MPa (130,000 psi), a yield strength of 731 MPa (106,000 psi), an eight percent elongation, and a 27 HRC as-sintered hardness. The intricate one-piece design would have been almost impossible to produce by any manufacturing process other than MIM. Secondary operations are limited to coining on the two latches and the application of a 0.5 µm gold coating for appearance and corrosion resistance. Specifying MIM gave the customer an estimated 40% cost savings.

AWARDS OF DISTINCTION
Four parts were selected for Awards of Distinction, Figure 7.
**PMG Indiana Corporation.** Columbus, Indiana, won the Award of Distinction in the automotive—engine category for three high-precision PM steel parts—slide, housing, and rotor, Figure 8—which operate in oil pumps in new larger hybrid SUVs. Choosing PM improved the oil pump’s efficiency in addition to reducing energy consumption and lowering vibration. The variable-displacement vane pump delivers oil when needed on demand, eliminating unnecessary oil flow. All three parts are made to a net shape and only require double-disk grinding for maintaining thickness and flatness tolerances. The parts have a density range of 6.5 g/cm³ for the housing, 6.6 g/cm³ for the slide, and 6.8 g/cm³ for the rotor. They are made on an automated compacting–sintering–sizing flow line because of their complexity and fragility.

**Porite Taiwan Co. Ltd.** Taiwan, won the Award of Distinction in the hardware/appliances category for a copper-infiltrated PM steel weight balance, Figure 9, used in a new compact compressor and manufactured for **Taiwan Hitachi Co. Ltd.** Taiwan. Made to a density of more than 7.2 g/cm³, the multi-level part has a tensile strength of 1,800 MPa (261,000 psi), a yield strength of 1,503 MPa (218,000 psi), and a hardness range of 48–52 HRC. Choosing the MIM process offers a very significant savings over machining.

**Parmatech Corporation.** Petaluma, California, captured the Award of Distinction in the hand tools/recreation category for a MIM 420 stainless steel housing block used in a 45-caliber handgun, Figure 10. It contains the firearm’s spring mechanism and provides sliding action with other mechanical parts. The complex MIM part features wings, undercuts, through-holes and blind holes, as well as thin and thick cross sections. Formed to a final density of 7.7 g/cm³, the part has a tensile strength of 1,800 MPa (261,000 psi), a yield strength of 1,503 MPa (218,000 psi), and a hardness range of 48–52 HRC. Choosing the MIM process offers a very significant savings over machining.
For 90 years, ACuPowder has been delivering the finest quality powders and the most conscientious service. Our customers know that serving their needs and solving their problems is our highest priority.

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It was most interesting to attend PowderMet2009 and I would like to thank the Scholarships and Grants Committee of the Center for Powder Metallurgy Technology (CPMT) for a CPMT/Axel Madsen Conference Grant. I would also like to thank my supervisor Professor Carl Blais for his support in the preparation of my poster. The first thing I noted about PowderMet2009 is that it was a great networking event. Representatives from all sectors of the powder metallurgy (PM) industry were at the conference, either to present their results (thus maximizing the sharing of knowledge), or to promote a product. All of the conference attendees had the opportunity to learn about advances in PM, be they scientific or technological. This was impressive since it was my first international conference.

PowderMet2009 was held in Las Vegas, a city in which everything is greater than reality. I arrived on Friday, June 26, and my fellow students and I had time to visit parts of the city. Seeing the shows in front of the hotels, such as the volcano, and standing in the casinos was something special. The city is much more grandiose than is portrayed in the movies.

The technical sessions in which I assisted provided invaluable information that I will use throughout my career. The value of this conference was the variety of PM technologies that were discussed. There were many captivating subjects and, even though I planned my schedule carefully, I was forced to miss several concurrent technical sessions. Of particular interest were the sessions on sinter hardening and titanium. In the former, I learned about optimizing alloy composition to obtain enhanced mechanical properties. The titanium sessions provided a complete overview of the technology of PM titanium and attendant market possibilities.

In conclusion, I would like to reiterate that attending PowderMet2009 was a great opportunity. I had the chance to learn extensively about PM, and to develop new contacts that will be useful during my career.

ANDREW CHAN
Drexel University
Philadelphia, Pennsylvania

I had never been to Las Vegas before and the heat was a new experience; it soaked through my clothes but the dry heat was not particularly uncomfortable. I arrived at The Mirage hotel and, after a long check-in process, I managed to settle into my room. Afterwards I went downstairs to the conference area to register and set up my poster. I returned to my room to change into more casual clothes and went downstairs to the welcoming dinner. The dinner was fantastic with delicious food and plentiful beverages along with a dolphin pool as a backdrop. There was also Sigfried and Roy’s Secret Garden which housed a collection of lions and tigers adjacent to the dinner area. It was

**Axel Madsen/CPMT Conference Grants are awarded to deserving students with a serious interest in PM. The recipients were recognized at the Industry Recognition Luncheon during PowderMet2009.**
great being able to chat with members of the PM industry in a casual setting. I left the dinner area and explored the casino before going to bed. While enjoying breakfast, I looked over the Conference Guide to select the presentations that I wanted to attend. I attended the technical sessions on compaction processes and stainless steel surface modification. After these sessions I attended the Industry Recognition Luncheon and was formally recognized for receiving a CPMT/Axel Madsen Conference Grant. The luncheon was great and I was able to converse with my fellow grant recipients, along with PM industry members and several Drexel University alumni.

Las Vegas is an eccentric city, to say the least. I walked in and out of several casinos to take in the sights. I regret not bringing my camera to take photographs, but what I do not regret is taking a cab to the "In and Out Burger" to enjoy a small celebratory dinner using my casino winnings. I wish I had made it to one of my classmates’ presentation on pharmaceutical powder compaction but was attending another session.

I would like to thank the Scholarships & Grants Committee of the Center for Powder Metallurgy Technology (CPMT) for this award. It was eye opening and informative to see the work on PM being conducted around the world and to see what engineers were really up to. Once again, I am grateful for the grant and the experience it offered me.

Although I have visited Las Vegas, Nevada, many times before, my trip in June 2009 was different from those of the past. As always, I was impressed by the vastness of the strip, the architecture of the hotels, and the throngs of people everywhere, despite the heat—and the economy! But this particular visit created a new sense of excitement in me. I was excited as to what I might learn and whom I might meet at PowderMet2009.

As this was my first professional conference, I was unsure what to expect. The opening night event in The Mirage's Dolphin Habitat was a great kick-off. The atmosphere was inviting, and I quickly learned that the PM industry is a tightly networked group with many members spending the majority of their careers in PM. It was great to see standing friendships amongst the attendees from different companies and sectors of the industry.

During Monday's keynote address, I was interested to learn how closely the PM and automotive industries are tied. Despite the uncertain economic climate, the atmosphere was surprisingly positive. This was further evidenced by the conference attendance, though lower than usual. The keynote address reinforced the many opportunities for PM developments in the future.

From the technical sessions, I began to appreciate the breadth of research occurring in PM. Though many of the sessions interested me, I chose to attend those pertaining to my research. While the presentations were oftentimes quite specific, the speakers were adept at concisely conveying their research results and observations in such a way that both novices, such as me, and experts could benefit. I particularly enjoyed the friendly debate on defining “cooling rate” during the sinter hardening sessions.

Over the course of the conference, I was continually impressed with the PM community. During the technical sessions and the social events, I interacted with people from many countries and from many areas of PM. The common thread that I observed many times was that the PM community is passionate about their work and the opportunities available in the field. The industry is both inviting and exciting to a newcomer like me.

PowderMet2009 was an incredible experience for me. I am now revived for a return to my research and to contribute to the field. I would like to thank the Scholarships and Grants Committee of the Center for Powder Metallurgy Technology (CPMT) for a CPMT/Axel Madsen Conference Grant and my research advisor, Professor Nikhilesh Chawla, for affording me the opportunity to attend the conference.
I landed in Las Vegas a few days before PowderMet2009 to the sound of rain on the tarmac. Water falling from the sky was most certainly not what I had expected toward late June in the Nevada desert. It stuck with me through trips to the Hoover Dam and the Grand Canyon. As the conference approached, however, the sun and heat returned. My trek in the hot sun (I regret the decision not to take a cab) from my original hotel to The Mirage was well rewarded. I was impressed with the cleanliness and upscale appearance of the hotel.

The opening night event in the Dolphin Habitat was not only a great way to beat the heat, but also a useful way to meet many of the faces in the PM world. In what seemed like a very short time, I was introduced to many people, some of whom I have consulted with or referenced while working toward my degree. It was comforting to be welcomed so warmly and to be accepted into such a tight-knit group.

For me, these social events—including the opening night event, luncheons, the main social event, and the industry trade exhibition—were important components of the conference. Each of these functions allowed me to meet people from the companies and universities involved in PM. I was also able to understand what they do, and in some cases discover that they are very good at it, as exemplified at the Industry Recognition Luncheon. The insight gained at the conference will prove invaluable as I near the end of my Master’s work.

The technical portion of the conference was informative in its own right. I attended many presentations on a variety of subjects throughout the conference. I was impressed with the Special Interest Programs. The sessions on PM titanium gave an excellent summary of the current technology and expected future developments. Presenting my first technical paper at PowderMet2009, in addition to my poster, was an honor. Having a kind, helpful audience was encouraging and made it an enjoyable experience.

I am grateful that I was given the opportunity to experience PowderMet2009. Receiving a CPMT/Axel Madsen Conference Grant afforded me the opportunity to attend. Without this grant, my industry-sponsored research would have made travel to the conference difficult in this slow economy. Having the opportunity to be recognized for my poster, networking at the conference, and learning more about the PM industry was exceptional. I feel that the CPMT/Axel Madsen Conference Grant does exactly as intended, namely, “to encourage students to learn more about PM technology and eventually pursue careers in the PM industry.”
STATE OF THE PM INDUSTRY IN NORTH AMERICA—2009

Mark Paullin*

THE BROAD PICTURE

IHS Global Insight, an economic forecasting organization, reports that most manufacturing surveys are beginning to show signs of stabilization, but also a very sharp reduction in inventories, which is driving production lower. It expects that inventory adjustment in the second half of 2009 will become less turbulent. Nevertheless, overall manufacturing production in the U.S. is expected to drop 12%, its worst decline since 1946, followed by a small improvement next year. Many in the PM industry say we have finally hit the bottom but disagree about how long the bottom will last.

However, we should never forget that the PM industry was built during difficult times. Many of the early pioneers and entrepreneurs got their start during the Great Depression. Their spirit of innovation, hard work, and grit overcame mountains of obstacles in the years that followed. That same spirit is still alive today, and will hopefully sustain the industry during these current difficult times.

2008: A YEAR REVIEW

2008 was a painful year. We found ourselves in a declining industrial market and the beginning of an inflationary spiral. The PM business year began on a hopeful note but weakened in the second half, especially in the final quarter. This was due primarily to a sharp decrease in automotive production in November and December.

The significant hikes in commodity prices, oil, and labor costs played havoc with our bottom lines. In addition, production in general declined significantly, with durable goods orders off and vehicle production down 16%.

Metal powder shipments remain the most accurate barometer for our industry’s health. In 2008, iron powder shipments declined 19% to 296,901 mt (327,272 st), with the PM parts share off by almost 22% to 258,513 mt (284,957 st), Figure 1. Copper and copper-based powder shipments declined 13% in 2008 to 15,785 mt (17,400 st), with the PM segment declining 10.5% to 13,473 mt (14,851 st), Figure 2. We estimate that stainless steel powder shipments declined about 20% last year to 7,031 mt (7,750 st). We also estimate that total metal powder shipments in North America declined 18.4% to 376,875 mt (415,427 st), Table I.

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Presented at PowderMet2009 in Las Vegas, Nevada.
In contrast, European PM-grade iron powder shipments fell 11.2% to 177,071 mt (195,184 st) and copper and copper-based shipments dropped 17% to 11,863 mt (13,077 st). In Japan, total iron powder shipments declined about 1% in 2008 to 225,861 mt (248,965 st), and copper powder shipments declined 4% to 8,573 mt (9,450 st).

2009 OUTLOOK

Unfortunately, North American iron and copper shipments continued deteriorating in the first half of 2009, impacted severely by the sharp decline in light-vehicle production as well as the shift away from large cars and SUVs to smaller cars with four-cylinder engines. Through May 2009, iron powder shipments declined 44%.

From a national economy point of view, there was also a significant decline in industrial production as the durable goods market collapsed. The only hope for the powder market is an expected spike in production beginning in September, driven by a positive change in the automotive and home building markets. Several PM industry observers anticipate an improvement in the fourth quarter, and we hope they are right.

We do have some good news in commodity prices, which have declined drastically from the hyper-inflationary numbers experienced during the summer of 2008: remember the pain of $1.06/L ($4.00/gal) gasoline, $150/barrel oil, $882/mt ($800/st) steel scrap, $55/kg ($25/lb) nickel, and $8.8/kg ($4.00/lb) copper? While rising somewhat, steel scrap is currently in the $495/kg to $550/kg ($225/lb. to $250/lb.) range, copper seems to be settling in the $5.1/kg to $5.5/kg ($2.30/lb. to $2.50/lb.) range and nickel is well below $22/kg ($10/lb.). This softening of commodity prices has certainly helped both our cash flows and our bottom lines.

North American vehicle production plunged 45 percent in May, the third lowest month in the past 18 according to Automotive News, with U.S. production accounting for a 51.6% decline. Through June, the annualized production rate is 7.2 million vehicles. Early estimates put the total year’s production at 8 million vehicles, a 36% drop from the 12.6 million produced in 2008 and a 46% drop from the 15 million vehicles manufactured in 2007.

A correlation between North American light-vehicle production and iron powder shipments since 2002 puts things into stark perspective, Table II.

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<td>Iron &amp; Steel</td>
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*(1 st = 0.9072 mt)

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<th>TABLE II. NORTH AMERICAN VEHICLE PRODUCTION</th>
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<td>Vehicle Production (millions)</td>
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*(1 st = 0.9072 mt) **year-to-year change in iron powder production
The big questions are, “When will the domestic automotive market rebound, and will there still be parts suppliers in business when it does?” We can say with confidence that those suppliers left standing will be leaner, stronger, better financed, and better prepared to compete in the global marketplace. Those suppliers will be able to serve the growing industrial and automotive markets in North America.

CSM Worldwide, a well-known international automotive forecaster and consulting organization, forecasts global light vehicle production to exceed 78 million vehicles by 2015, a 50% increase from the 52 million vehicles slated to be produced in 2009, Table III. The forecast for growth in North America is still robust. Of the seven geographic regions, North America has the highest projected increase of vehicle production and is number two in percentage increase.

Taking this figure, we can project the average estimated PM usage to range anywhere from 6.8 kg to 10.5 kg (15 lb. to 23 lb.) globally, which translates into a potential global PM automotive market of somewhere between 535,248 mt and 816,480 mt (590,000 st and 900,000 st). The average PM content in North American vehicles is expected to remain at about 18.6 kg (41 lb.) per vehicle. North American PM companies must position themselves globally to capture a major share of this market.

The PM industry still has much to offer car manufacturers in conventional powertrains, in diesel engines, and in hybrids. We can offer innovative engineering/materials solutions, cost savings, and a technology that is, for the most part, environmentally safe. For example, the Metal Powder Industries Federation (MPIF) has just released the PM Automotive Parts Catalog study that has already identified more than 300 PM automotive applications representing more than 750 total parts. These are conservative numbers: some estimates put the number at 1,000 or more parts, taking into account applications not yet identified.

MPIF will also shortly release the 2009 edition of Standard 35, Materials Standards for PM Structural Parts. The newly updated version contains new materials and mechanical property data and new engineering information on hardenability and corrosion resistance. This new information will be helpful to automotive design engineers.

New applications exist in advanced transmissions such as dual-clutch designs and continuously variable transmissions (CVTs), as well as in five- and six-speed transmissions. Europe is well on its way to unseat the dominance of manual transmissions, which could open up new PM opportunities in automatics. The trend toward increasing power in smaller engines with turbochargers offers potential applications in the turbo drive system and electric power generators which use high rpm transmissions.

Diesel engines represent another potential growth market for PM parts, especially for powder-forged connecting rods and high-alloy PM materials. Clean diesels offer 30% better fuel economy and 25% fewer CO₂ emissions.

The PM industry must also join the hybrid bandwagon or be left behind. The Nikkei Business Daily recently reported that the global hybrid vehicle market will grow to 11.28 million vehicles by 2020. Potential applications in hybrids include electric traction drives and electric motor gears.

Finally, structural changes in the North American automotive market, with the new GM and Chrysler organizations perhaps, could force a new wave in the consideration of cost savings and innovative technologies intrinsic to PM. We can only hope so.

DIVERSIFY OR DIE

While the automotive market will still be important for the PM industry, diversification into new markets is vital for our future. Renewable “green” energy presents opportunities for conventional PM, metal injection molding (MIM), and nanotechnology. Wind turbines use electric motors and transmissions that need metal parts and bearings. For example, bearings in wind turbines are noted for their high maintenance requirements: is it possible to make high-performance full-density
PM bearings? Solar power is still another growing market for commercial and consumer applications.

MIM remains a bright spot that serves diverse market segments such as medical, dental, and electronics applications. One observer suggests that quick-change tooling could open up new automotive applications by helping MIM parts makers compete more successfully with investment casting.

To further promote MIM’s advantages to the design community, the Metal Injection Molding Association (MIMA) this year launched a dedicated Web site devoted to the benefits of using the MIM process. Visit www.mimaweb.org.

Still in their infancy, rapid prototyping and rapid manufacturing are exciting technologies ready to hit the commercial production scene. There are seven companies worldwide that make machines that use metal powders. It is estimated that 100 such machines are in use, with about 50% of them at academic institutions. However, a company in Europe is producing 10,000 hip replacement cups annually via rapid manufacturing.

Nanotechnology is another promising area. According to a recent report in The Economist, MIT researchers have developed a nickel–tungsten alloy as a substitute for chrome plating. Adding tungsten to nanocrystalline nickel produces a hard material capable of being plated but more environmentally friendly than chrome. Tests are underway with the alloy on truck bumpers.

**SUSTAINABILITY—NOTHING NEW TO PM**

Green is hot. Products and manufacturing processes that promote green products and environmentally sensitive material, that are non-polluting, and that conserve energy and natural resources are economically sound. PM is a sustainable, net-shape manufacturing process that has long been recognized as a green technology, for minimizing energy consumption and for recyclability. Metal powders are produced from recycled materials and most PM parts can be recycled. But we have not done a good job in promoting PM’s sustainability story.

This is about to change. MPIF is coordinating a new effort to promote PM’s sustainability. It is featured in a new promotional campaign planned for release later this year or early next year. The campaign will include a new “PM Green Technology” logo to be used in advertisements, on member company material, boxes, letterheads, etc. There will be more details to come on this campaign.

**INVESTING IN NEW TECHNOLOGY**

It is gratifying to note that many PM companies are still investing in new technology. And when the marketplace returns to normalcy, those investments will reap rewards.

Metal powder suppliers are working on new materials to increase densities to 7.5 g/cm³ with single pressing (SP) and single sintering (SS). They are also aiming R&D at providing lower-cost alternative materials to replace diffusion-bonded powders and high-nickel grades. Specialty-powder makers are developing cleaner prealloyed iron-, nickel-, and cobalt-base powders for high-performance parts. We must move beyond plain iron powder to meet the demand for high-temperature materials that will open new markets. Copper-powder makers are also diversifying into extra-fine spherical powders for electronic applications. Green bullets, antimicrobial products, and anti-fouling paints are niche markets for copper powders that offer additional promise.

While the PM industry faces many challenges, it will still be an important materials technology and manufacturing process. As the current recession subsides, the market will return and grow. The U.S. automotive and industrial markets will still need innovative suppliers that offer precision products and cost savings. PM companies that remain will exit the recession much leaner and stronger and more capable of serving customers in the new economy.
INTRODUCTION

In the past decade, a series of new bulk metallic glasses (MGs) with a multicomponent chemistry and high glass-forming ability (GFA) have been developed, including Zr-, Mg-, La-, Pd-, Ti-, and Fe-based alloy systems,(1–4) using various solidification techniques; this has engendered interest in the synthesis and application of bulk MGs. In turn, the properties of these bulk MGs, the strength and anticorrosion properties of which are typically superior to those of their crystalline counterparts, have stimulated researchers to explore techniques to fabricate net-shaped bulk MG components. While some progress has been documented,(5–8) significant breakthroughs have been hindered by the less-than-optimum GFA and high viscosity that are typically associated with MGs.

Compared with most other MGs, such as Zr- and Pd-based MGs, the advantages of Fe-based MGs include a much lower material cost and excellent mechanical and physical properties.(8) The major obstacle to forming Fe-based MGs has traditionally been their limited GFA, although some progress has been documented in the literature.(8–11) The high GFA value of these Fe-based MGs has been rationalized on the basis of three empirical rules proposed by Inoue et al.:[12,13] (1) multicomponent alloy systems consisting of more than three constituent elements, (2) significantly different atomic size ratios, and (3) negative heats of mixing among the constituent elements. It was also reported that the addition of Mo increases the GFA of Fe-based alloys.(11,14) The addition of Mo and W to Fe-based alloys can also

In this article, the laser-engineered net shaping (LENS) process is implemented to fabricate net-shaped Fe-based Fe-B-Cr-C-Mn-Mo-W-Zr metallic glass (MG) components. The glass-forming ability (GFA), glass transition, crystallization behavior, and mechanical properties of the glassy alloy are analyzed to provide fundamental insights into the underlying physical mechanisms. The microstructures of various LENS-processed component geometries are characterized via scanning electron microscopy (SEM), X-ray diffraction (XRD), differential scanning calorimetry (DSC), and transmission electron microscopy (TEM). The results reveal that the as-processed microstructure consists of nanocrystalline α-Fe particles embedded in an amorphous matrix. An amorphous microstructure is observed in deposited layers that are located near the substrate. From a microstructure standpoint, the fraction of crystalline phases increases with the increasing number of deposited layers, effectively resulting in the formation of a functionally graded microstructure with in-situ-precipitated particles in an MG matrix. The microhardness of LENS-processed Fe-based MG components has a high value of 9.52 GPa.

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enhance their corrosion resistance.\(^{[14–16]}\) Moreover, adding Zr significantly increases the glass transition temperature \(T_g\) and adding Mn decreases the liquidus temperature and Curie point. At the same time, increasing the amount of B can also enhance GFA.\(^{[1]}\) In this study, gas-atomized \(\text{Fe}_{58}\text{Cr}_{15}\text{Mn}_{2}\text{B}_{16}\text{C}_{4}\text{Mo}_{2}\text{Si}_{1}\text{W}_{1}\text{Zr}_{1}\) (at. pct) powder was used for the laser processing of MG components. The other elements (e.g., Cr, B, and Si) enhance thermal stability and provide increased additional solid solution strengthening.

Gas atomization (GA) is a practical and effective approach for producing MG powder. However, the gas-atomized MG powder cannot be used directly as a structural component. Accordingly, gas-atomized powder is generally processed through a series of conventional powder metallurgy steps such as degassing, cold isostatic pressing or hot isostatic pressing, and extrusion to form dense bulk materials, which are then machined into final components. In this article, we explore the potential of using the LENS process to form fully dense, three-dimensional (3-D) net-shaped Fe-based MG components through laser deposition. Inspection of the technical literature reveals that the results obtained for the laser glazing\(^{[17–18]}\) and laser cladding of MGs\(^{[19–22]}\) have been reported, although these are concerned with surface treatments and coatings. To our knowledge, this work represents the first time that the fabrication of net-shaped bulk MG components has been investigated by laser direct-deposition techniques.

LENS (laser-engineered net shaping) provides a novel pathway for producing net-shaped bulk MGs. The LENS process is a laser-assisted, direct metal manufacturing process originally developed at Sandia National Laboratories (Albuquerque, NM).\(^{[23]}\) The LENS process incorporates features from stereolithography and laser cladding, using a computer-aided design file to control the forming process; a 3-D part can be generated point by point, line by line, and layer by layer via additive processing. The LENS process can produce relatively high, localized cooling rates at each deposition point due to the very small size of the melt pool and the conduction of thermal energy into the substrate.\(^{[24,25]}\) Therefore, it should be possible to manufacture net-shaped MG components using LENS, because the material is deposited as sequential and cumulative layers. In addition, it should be possible to optimize the cooling conditions for MG processing by controlling the processing parameters such as the laser power and travel speed.

The LENS process provides several advantages from an engineering standpoint. The LENS process can be used to generate materials that contain multiple length scales, thereby facilitating the optimization of physical properties. In the case of MGs, it should be possible to fabricate net-shaped components with a high material yield and attractive physical properties. In this article, Fe-based Fe-Cr-Mo-W-C-Mn-Si-Zr-B MG components were deposited via LENS processing. Their microstructure was characterized via scanning electron microscopy (SEM), X-ray diffraction (XRD), differential scanning calorimetry (DSC), and transmission electron microscopy (TEM). The mechanisms thought to be responsible for the observed microstructure, GFA, and mechanical properties are discussed.

**EXPERIMENTAL PROCEDURES**

Gas-atomized Fe-based \(\text{Fe}_{58}\text{Cr}_{15}\text{Mn}_{2}\text{B}_{16}\text{C}_{4}\text{Mo}_{2}\text{Si}_{1}\text{W}_{1}\text{Zr}_{1}\) (at. pct) powder with a size range of 10 to 110 µm was selected as the starting material. A hot rolled 304 stainless steel plate with a thickness of 6.35 mm was used as the substrate. The LENS 750* system used in this study was manufactured by Optomec, Inc. (Albuquerque, NM) and consists of a continuous-wave mode Nd:YAG laser operating up to 650 W, a four-nozzle coaxial powder feed system, a controlled-environment glove box, and a motion control system. The nominal laser beam diameter is 6.3 mm and has a <0.5-mm diameter circular beam waist at the focal zone. The energy density used in the present article was in the range \(2 \times 10^4\) to \(1 \times 10^5\) W/cm². Three types of sample geometries were fabricated for our study: shells (A, B, and C), solid cubes (D, E, F, and G), and coatings (H, I, J, and K), as shown in Figure 1, with different process parameters shown in Table I. In the case of the coating and cubic samples, the area of each layer corresponded to 10 × 10 mm. The hatch space is 0.38 mm and the layer thickness, \(\Delta z\), is 0.25 mm. The coating samples were fabricated with different numbers of deposited layers (1 for H, 2 for I, 4 for J, and 8 for K), to investigate the microstructure evolution during laser deposition. Successive layers were deposited with the hatch lines of two

\*LENS 750 is a registered trademark of Sandia National Laboratories, Albuquerque, NM.
adjacent layers at an angle of 90 deg. The entire process was carried out in an Ar environment, to avoid oxidation during deposition. The oxygen level in the glove box was maintained below 10 ppm during deposition.

The as-deposited Fe-based components were sectioned along the center axis and perpendicular to the laser travel trace on the top layer; they were then mounted, ground, and, finally, finely polished using the conventional techniques for metallographic characterization studies. Scanning electron microscopy coupled with energy-dispersive X-ray (EDX) spectrum analysis, XRD with Cu $K\alpha$ radiation, DSC, and TEM were used for the microstructure characterization and phase analysis of the deposited samples. The XRD scans were performed on the free surfaces of the laser-deposited coatings. Microhardness measurements were conducted using a Vickers indenter with Buehler MicroMet 2004 apparatus (Buehler Ltd., Lake Bluff, IL) under a 100-g load on metallographically mounted cross sections of the laser-deposited samples.

**RESULTS AND ANALYSIS**

**Gas-Atomized Powder**

The gas-atomized Fe-based Fe-Cr-Mo-W-C-Mn-Si-Zr-B alloy powder exhibited a lognormal size distribution. Observations of the powder with SEM secondary electrons revealed that most of the powder is spherical in shape, which is typical for a gas-atomized powder,(26) with variations in the surface morphology, as shown in Figure 2(a). The surface of the smaller powder was generally smoother than that of the larger powder, the associated roughness being attributed to solidification shrinkage.(26,27) Moreover, the surface features were noted to be indicative of the microstructure: smooth in the case of the amorphous powder and rough in the case of the crystalline powder.

The cross-sectional microstructures of the different powder sizes were studied using SEM backscattered electrons (BSEs) and revealed the typical microstructure shown in Figure 2(b). The powder <20 µm revealed a featureless microstructure, indicating that no crystallization had occurred during GA. The featureless powder was considered to be amorphous. The extent of crystallization was noted to increase with the powder size, consistent with published results.(28–30) It is important to note that, although a starting powder with a wide particle size range (from 10 to 110 µm) and different initial microstructures (from amorphous to crystalline) was used in this work, all the particles will be completely melted and resolidified during LENS deposition. Therefore, the influence of the initial microstructure of the starting powder on the microstructure and properties of the laser-deposited layers was deemed to fall outside of the

| TABLE I. Process Parameters Used for Laser Deposition via LENS |
|-----------------|----------------|-----------------|-----------------|-----------------|
| Sample Number   | Laser Power, P(W) | Travel Speed, v(mm/s) | Power Exposure, P/v(J/mm) | Powder Feed Rate (g/min) |
| Shell A         | 180             | 12.7             | 14.17           | 10              |
| Shell B         | 280             | 8.47             | 33.06           | 6               |
| Shell C         | 280             | 12.7             | 22.05           | 10              |
| Cubic bulk D    | 180             | 12.7             | 14.17           | 10              |
| Cubic bulk E    | 180             | 8.47             | 21.25           | 10              |
| Cubic bulk F    | 296             | 8.47             | 34.83           | 10              |
| Cubic bulk G    | 180             | 4.23             | 42.55           | 10              |
| Coating S, T, U, and V | 180 | 12.7             | 14.17           | 10              |
scope of the present study. The primary considerations used to select the starting powder were morphology (i.e., spherical), size (<150 µm), GFA, and laser energy absorption. For the irradiation with Nd:YAG laser (wavelength 1.06 µm), the absorptivity of Al (0.06 to 0.2) and Cu (0.04 to 0.3) alloys is much lower than that of Fe alloys (0.25 to 0.35).\(^{31}\)

**Microstructure of LENS-Deposited Samples**

The material initially deposited on the cold substrate during LENS experiences a high quenching effect; it is in this region that an amorphous microstructure is likely to be observed. The magnitude of the cooling rate during the first-layer deposition can be estimated to be $10^3$ to $10^4$ K/s on the basis of numerical simulations.\(^{32,33}\) Figure 3 shows a micrograph, imaged with SEM BSEs, of an initially deposited layer obtained with a laser output power of 280 W and a travel speed of 12.7 mm/s. The features of the melt pool shape and the overlap between subsequent deposited lines are clearly visible. The microstructure is featureless, which suggests that the first layer is amorphous. This indicates that the cooling rate experienced by the deposited materials was high enough to form an amorphous microstructure, consistent with that reported for these Fe-based MGs.\(^{19,34}\) The top surface contained some partly unmelted particles that retained the original partially amorphous structure of the powder, as shown in Figure 3(a). These particles will be completely melted and resolidified during the subsequent layer deposition, because the surface of the previously deposited layer is always partially remelted by the laser beam during deposition. Only the surface of the final deposited layer con-
tains partly unmelted particles. The presence of light-colored phases in the second layer suggests that melting and resolidification occurred in this region presumably under a low cooling rate, e.g., approximately $10^2$ to $10^3$ K/s.\(^{(33,35)}\)

In order to investigate any possible reactions of the deposited layer with the substrate, an identical powder was deposited on an amorphous substrate using the same parameters as were used for deposition on the 304 SS substrate. Similar to the case of a crystalline substrate, laser deposition on the amorphous substrate made of Fe-based Fe-Cr-Mo-W-C-Mn-Si-Zr-B thick amorphous coatings (400 µm) on a 304 SS plate via high-velocity oxygen fuel thermal spraying\(^{(36)}\) also resulted in overlapping, amorphous melt zones. The absence of microstructural features in these zones indicates that the region is initially melted by the laser beam and then resolidified as glass, regardless of the nature of the underlying substrate. The absence of significant dilution or reaction products at the interface of the deposited Fe glass and the 304 SS substrate is attributed to the high solidification rates associated with LENS processing, which suppresses the diffusion-controlled phase transformations of crystallization such that solute trapping will occur and large segregation no longer operates.

As the thickness of the deposited material increases, the microstructure coarsens and precipitation becomes increasingly evident, particularly at the boundary of the melt pool region, which, for purposes of this discussion, may be described as a heat-affected zone (HAZ). The thickness and morphology of the HAZ strongly depends on the LENS processing parameters. This trend is consistent with the decrease in the cooling rate that accompanies an increase in the number of deposited layers.

In the case of the LENS-deposited samples with cubic and shell geometries, the macroscale variables that influence deposition are a buildup of height and melt depth into the previous layer. To ascertain the results of these samples, metallographic cross sections of the deposited materials were made. The height buildup was measured from the substrate surface to the upper surface of the deposited material. Similarly, the melt depth was taken to be the depth of the region in which dissolution is evident. The measured melt depth tends to increase from a minimum value of 0.05 mm to a maximum value of 0.25 mm with increasing laser power and decreasing laser travel speed. When the laser travel speed is low, there is time for powder accumulation, and slightly more penetration occurs. In terms of geometrical considerations, the laser power and travel speed also played a significant role in the accumulation and remelting processes. The experimental results suggest that there was a strong correlation between the height buildup of the materials and a ratio defined hereafter as the laser power exposure (laser power/laser travel speed). Figure 4 shows the variation in the height buildup with the laser power exposure. The overall thickness of the bulk cubic and shell samples increases with increasing laser power exposure. The relationship between the height buildup $H$ and the processing parameter $P/v$ was derived using curve-fitting techniques; it is given as

\[
H = 2.33 + 0.35(P/v) - 0.004 \frac{P}{v^2}
\]  

for bulk geometries \[1\]

\[
H = 3.48 + 0.14(P/v) - 0.001 \frac{P}{v^2}
\]  

for shell geometries \[2\]

where $P$ is the laser output power and $v$ is the laser travel speed. The thickness of the bulk geometry is larger than that of the shells and can be attributed to the fact that there is overlap between the deposition lines during the fabrication of the former, whereas there is none during the deposition of the latter.

The typical microstructure of laser-deposited
bulk cubic geometry is shown in Figure 5. This sample was produced with a laser power of 280 W and a travel speed of 12.7 mm/s. It is evident that the microstructure from the top layers is much coarser than that present in the bottom layers. The results from this sample confirm that the temperature of the deposited materials increases with thickness during laser deposition, and that the cooling rate decreases with increasing distance from the substrate. In addition, the heat generated by the subsequently deposited layers can promote crystallization of the preceding layers. Approximately two layers revealed a featureless microstructure, which was considered to be amorphous.

Figure 6 shows the microstructure evolution in the laser-deposited Fe-based MG shell component processed with a laser output power of 280 W and a travel speed of 12.7 mm/s. As one moves from the bottom to the top layers, the microstructure varies from amorphous to an MG matrix composite, effectively forming a functionally graded microstructure. It is interesting to note that, if one compares the microstructure of the shell sample to that of the corresponding location in the cubic sample, it is shown that the former is coarser than the latter (Figure 5). This difference is attributed to the higher thermal conductivity of the bulk metal (cubic sample) as compared to the heat dissipated via convection into the environment (shell sample). In the case of a given set of process parameters, the microstructural evolution during LENS is primarily influenced by the conduction of thermal energy through the deposited material into the substrate, which effectively behaves as a heat sink. Convective and radiative losses into the environment appear to have only a limited effect on the microstructural evolution. An additional factor that contributes to the observed difference in microstructure between the shell and cubic samples is the fact that the time interval between two sequential layers is shorter for
According to the experimental and numerical simulation results, the temperature at the end of each deposition cycle increases with a decreasing interval time, which corresponds to a decrease in the cooling rate during deposition. In addition, the accumulation of thermal energy that is likely to develop during the deposition of multiple layers is also likely to promote crystallization of the microstructure.

The energy-dispersive analysis line-scanning results (Figure 7) show that the precipitated white phase contained a higher concentration of Cr, W, and Mo relative to the matrix. During the initial stages of phase precipitation, the boundary between the precipitated phases and the matrix is not well defined as the component atoms begin to segregate and cluster. The phase morphology of the precipitated phases becomes as well defined as in the upper regions of the samples, because extensive precipitation has been facilitated by a low cooling rate.

Figure 8 shows the XRD patterns obtained from the top surface of laser-deposited Fe-based MG coatings with different numbers of deposited layers. The XRD curve of the deposited first layer presents a diffuse broad halo peak at around $2\theta = 44.4$ deg, illustrating the amorphous nature of the specimen. The broad diffraction peak becomes less obvious with increasing numbers of deposited layers. The crystalline peaks appeared superimposed on a broad diffraction peak for all the coating samples, which is similar to the pattern obtained from the gas-atomized powder. Even though the SEM BSE images of the first-layer sample revealed a featureless microstructure within a molten pool region, some crystalline peaks still appeared on the corresponding XRD curve. In fact, these peaks were attributed to the precipitated crystalline in the HAZ and partially melted powder, which retained the original partially amorphous structure. Three broad peaks at
around 44.4, 64.5, and 81.7 deg, corresponding to the reflections of the nanocrystalline α-Fe phase, can be observed together with a halo peak, which confirms the presence of precipitated nanocrystalline α-Fe embedded in an amorphous matrix. The other phases presented are likely to be γ-Fe, tetragonal Fe₂B, cubic Cr₂₃C₆, and other unidentified phases.

The microstructure of laser-deposited MG samples was also observed using TEM. Although the initial deposited layers appear featureless during observation with SEM, as shown in Figures 3, 5, and 6, some nanocrystallites of α-Fe with a size of 5 to 20 nm, embedded in an amorphous matrix, were observed via TEM. This is illustrated in Figure 9(a), in which the bright-field image and its corresponding diffraction pattern reveal the presence of α-Fe diffraction rings. The increased deposition thickness resulted in additional precipitation, as illustrated in Figure 9(b).

The TEM results also confirmed the observation of an increased degree of crystallinity with the increased deposition thickness. This phenomenon is illustrated in Figure 9(b), which shows a bright-field image and its corresponding diffraction pattern from an upper layer.

The size of the precipitated crystals that are evident in this figure is approximately 100 nm. In all of these cases, a primary crystallization reaction occurs and starts with the formation of nanocrystalline α-Fe grains. Further growth of these particles and the appearance of new grains ensued as additional layers were deposited.

Figure 10 shows the DSC trace curve of the first layer of the laser-deposited Fe-based MG coating, as determined with a heating rate of 20°C/min. With increasing temperature, the curve shows an amorphous transition, followed by the appearance of a supercooled liquid region and crystallization. The glass transition temperature $T_g$ corresponds to the temperature at which the curvature of the endothermic reaction on the DSC curve is maximum, while the crystallization onset temperature $T_x$ is defined as the temperature at which the tangential lines between the supercooled liquid and the exothermic peak intersect each other. The temperature interval of the supercooled liquid region $\Delta T_x$, defined by the difference
between the glass transition temperature and the onset temperature of crystallization, $\Delta T_x = T_x - T_g$, is as large as 110°C. In addition, the $T_g/T_m$ value is 0.37. The large supercooled liquid region implies a high thermal stability of the supercooled liquid against crystallization. Generally, the GFA increases with increasing $\Delta T_x$ and $T_g/T_m$.(3, 37)

**Mechanical Properties**

The microhardness of the LENS-deposited Fe-based Fe-Cr-Mo-W-C-Mn-Si-Zr-B MG component was measured using a 100-g load with a Vickers microhardness tester; the results are summarized in Figure 11. The results show the variation in microhardness at the center of the cubic sample as a function of distance from the substrate surface in the height buildup direction. The variations in microhardness of the cubic MG sample in the transverse direction, as well as in the center of a shell sample, as a function of the distance from the substrate surface were also measured. No significant variations in microhardness were observed in the cases of the transverse measurements for the cubic sample and the center measurements for the shell sample, despite obvious differences in the degree of devitrification. The variation in microhardness values obtained from the bottom region is significantly smaller than that of the upper region, as a result of the predominantly amorphous structure present near the bottom region.

The variations in microhardness summarized in
Figure 11 can be attributed to the extent of precipitation throughout the deposited material. It is also interesting to note that the microhardness of the deposited materials was slightly higher for the higher laser travel speed, which corresponds to a decrease in the interaction time of the laser materials and the associated high cooling rate during deposition. The microhardness of laser-deposited Fe-based MG materials is approximately 900 HV (9.5 GPa), with a corresponding tensile strength (~1/3 hardness) of approximately 3.1 GPa, which is significantly higher than the values for conventional steels. The variation in the average microhardness of the LENS-deposited bulk MG samples with laser exposure is also shown in the inset of Figure 11. It was found that the average microhardness decreases almost linearly with increasing laser exposure (laser power/laser travel speed), which corresponds to a decreasing cooling rate during deposition. The relationship between the average microhardness $MH$ and the laser exposure $P/v$ was derived by curve fitting, and is given as

$$MH = 846.7 - 0.23 \left( \frac{P}{v} \right)^3$$  \[3\]

where $P$ is the laser output power and $v$ is the laser travel speed.

**DISCUSSION**

The microstructure evolution of laser-deposited Fe-based Fe-Cr-Mo-W-C-Mn-Si-Zr-B MG materials during LENS processing is complex, because it not only depends on the thermal history but it is also a function of the alloy system. Due to the layer-additive nature of the LENS process, the thermal behavior associated with the LENS process involves numerous reheating cycles. Thus, the assessment of the microstructure evolution necessitates an understanding of the response of the alloy to these cycles. As shown in previous numerical and experimental studies,\(^{25,32,33}\) the thermal behavior associated with the LENS process involves a series of wave-shaped thermal cycles. Each peak represents a laser heating event as the laser beam passes over a layer and effectively reheats layers that were deposited previously. The thermal excursions dampen out when the laser energy source moves away during the deposition of subsequent layers. After reaching an initial peak temperature, the heat is quickly conducted away for the first layer, which experiences rapid heating and cooling, non-equilibrium conditions. Because of rapid heat loss through the substrate during initial deposition, this initial thermal transient results in a high cooling rate ($10^3$ to $10^4$ K/s\(^{33,35}\)) during solidification and can produce a glassy microstructure in the case of glass-forming alloys, as shown in Figures 3 and 9(a). However, each subsequent pass reheats the previously deposited layers such that after several layers are deposited, the initial deposited layers continue to experience thermal excursions. When the reheating temperature is higher than the crystallization onset temperature $T_x$, new phases will precipitate, implying MG devitrification. However, the time it takes a deposited upper layer to reheat a lower layer to a temperature above $T_x$ is very short (<0.05 s) for each deposition sequence, and the thermal energy is rapidly dissipated into the lower layers. Our microstructural analysis shows that a heating time as short as this was not sufficient to fully crystallize the lower layers.

The accumulation of thermal energy during component fabrication is likely to promote devitrification. The accumulation of thermal energy at the end of each cycle causes the temperature to be somewhat higher than that at the end of the previous cycle. Accordingly, the temperature increases monotonically with the increasing deposited material thickness, as confirmed via numerical and experimental results.\(^{24,33}\) It then follows that the cooling rate decreases with the deposition thickness, consistent with the observation that the microstructure coarsens from the bottom to the top layers, as illustrated in Figures 5, 6, and 9(b). Only when the deposited sample attains a sufficient thickness will the temperature of a lower layer exceed $T_x$ and complete crystallization occur. From our modeling results,\(^{33}\) the stable temperature for a 25.4-mm distance from the substrate can reach 400°C, when the overall deposited material is approximately 50.8 mm. This complex thermal cycling will influence not only the microstructure but also residual stresses as the material is tempered or aged.

There are two key parameters of the LENS process, laser output power and travel speed, that directly determine the local thermal conditions (temperature and cooling rate) and therefore con-
trol the resulting microstructure of deposited MGs. These two parameters can be combined into the energy intensity of a laser beam, which can be determined by dividing the power of the beam by the spot area:

\[ I = \frac{P}{vd} \]  

where \( P \) is laser output power, \( v \) is laser travel speed, and \( d \) is the laser beam spot size in diameter on the substrate. For a given laser power, increasing the laser spot size and scanning speed will decrease the intensity, while a high laser power will increase the intensity. This thermal energy input melts the injected powder and the surface layer of the substrate; it also heats the underlying material. Both the depth of the melt zone and the thickness of the HAZ increase with increasing heat input. A fast laser-scanning speed leads to a short interaction time between the laser beam and the material, which can also promote high quenching and lead to the higher strength of the deposited Fe-based MGs, as shown in Figure 11. The process parameters of laser output power, travel speed, and initial temperature of the substrate have significant influences on the thermal history of the deposited materials; the cooling process can be controlled by changing these variables. The present results suggest that a combination of a laser power of 150 W and a travel speed of 12.7 mm/s with a powder feed rate of 10 g/min appears to be close to ideal for forming a melt pool necessary for powder incorporation without causing much crystallization in the initial deposition.

The behavior of MGs synthesized via LENS laser deposition depends on both the process parameters and the alloy composition. The Fe-based Fe-Cr-Mo-W-C-Mn-Si-Zr-B alloy used in the present article has a limited GFA, as is evidenced by the microstructure of both the gas-atomized and LENS-deposited samples (Figures 2 and 3 and 4 through 8). The basis of the present alloy is the Fe-Mn-Cr-B system,\(^{[2,9-11]}\) which satisfies the three empirical rules.\(^{[12,13]}\) The addition of Mo, W, Zr, C, and Si can cause an increase in the atomic size difference and the generation of new atomic pairs with various negative heats of mixing. For a multicomponent MG consisting of constituent elements, the criterion of the ratio of the atomic size difference \( \lambda_n \) has been used to evaluate the optimum solute concentration for high a GFA: \(^{[39]}\)

\[ \lambda_n = \frac{n-1}{\sum B=1 \left( \frac{r_B}{r_A} \right)^3 - 1} C_B \]  

where \( r_A \) and \( r_B \) are the solvent and solute atom radius, respectively, and \( C_B \) is the solute concentration (at. pct) of element B. It is found that the values of \( \lambda_n \) of bulk MG formers with a great GFA in Zr-, Pd-, Mg-, Nd- and Fe-based systems are approximately constant of 0.18. The \( \lambda_n \) is 0.21 for the composition corresponding to the Fe\(_{58}\)Cr\(_{15}\)Mn\(_{2}\)B\(_{16}\)C\(_4\)Mo\(_2\)Si\(_1\)W\(_1\)Zr\(_1\) (at. pct) alloy. The large atomic size difference in the multicomponent system can result in the highly dense and randomly packed structure of MG alloys. The nucleation and growth of the crystalline phase may be suppressed in the supercooled liquid by inhibiting the long-distance diffusion and increasing the melt viscosity. It is also very difficult for the multiple elements in the alloy to simultaneously satisfy the composition and structural requirements of the crystalline compounds. Minor alloying additions are frequently added to MGs, to improve GFA and effectively "tune" the mechanical and physical properties of bulk MGs.\(^{[40]}\) The addition of metalloid elements such as B, C, and Si has a significant effect on the GFA, thermal stability, and properties of Fe-based MG-forming alloys. Proper additions of small atomic-sized elements such as B, C, and Si can tighten the alloy structure and stabilize the alloy against crystallization. Minor additions of Zr enable this alloy to behave as a liquidlike structure at low temperatures and to remain amorphous as it solidifies, due to the strong affinity Zr has for O. However, the as-received alloy used in the present investigation shows a limited GFA. Recently, Fe-based alloys with a high GFA have been developed (e.g., Fe-Cr-Mo-Y-C-B) and should be of interest for LENS studies, because results show that a fully amorphous structure in the gas-atomized powder size range 40 to 150 \( \mu \)m were obtained.\(^{[41]}\)

The observed Fe-based Fe-Cr-Mo-W-C-Mn-Si-Zr-B amorphous phase in the laser-deposited samples can be attributed to the rapid solidification that occurs during rapid cooling as the thermal energy is transferred into the substrate. In this case, the nucleation and growth of crystallites are hindered by the local rapid drop in temperature. Beyond the
initial layers, however, the temperature of the deposited material constantly increases as additional layers are added, which results in a decrease in the cooling rate and, hence devitrification, as shown in Figures 5 and 6. According to the experimental and modeling results,\(^\text{(25,33)}\) the temperature could increase to a value of approximately 150°C at the end of the second-layer deposition, and the cooling rate would decrease to approximately \(10^{-2}\) K/s for the third-layer deposition. This indicates that a constant cooling rate of at least \(10^{-2}\) K/s is required for depositing this type of Fe-based MGs via the LENS process, based on the absence of crystallization.

The inherent variation in the cooling rate during LENS processing can lead to the formation of graded materials with an in-situ-formed MG matrix composite, as shown in Figures 5 and 6. It is interesting to note that this type of microstructure has been reported to be beneficial for strength and fracture toughness.\(^\text{(7,42)}\) In these microstructures, fracture may be suppressed by limiting strain localization by the precipitated reinforcing phase. The precipitated phases can also help distribute both shear bands and microcracks, limit shear band extension, suppress shear band opening, and avoid crack development.

These preliminary research results on LENS-deposited MG components indicate their flexibility in processing novel materials, even those in which the thermal and solidification conditions must meet certain conditions. The cracks that were evident in the deposited MGs, as shown in shell sample C in Figure 1, are attributable to the lack of ductility of MGs and to residual stress development, which is an intrinsic outcome of incremental deposition processes such as LENS.\(^\text{(43)}\)

**SUMMARY**

The microstructure of the gas-atomized Fe-based alloy Fe-Cr-Mo-W-C-Mn-Si-Zr-B powder used in the present work was not fully amorphous. During the initial stages of deposition with LENS processing, significant rapid quenching occurs and two Fe-based predominantly amorphous layers are created. With an increasing deposit thickness, new crystal phases precipitated and the microstructure coarsens due to the imposition of reheating cycles in combination with a decreasing cooling rate. The number of \(\alpha\)-Fe particles and other precipitated phases increases with the increasing number of deposited layers, forming graded composites with in-situ-precipitated particles in an MG matrix. The microhardness of the deposited Fe-based Fe-Cr-Mo-W-C-Mn-Si-Zr-B MGs is approximately 900 HV (9.52 GPa), a value that is attractive for engineering applications.

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**REFERENCES**

EXOTHERMIC REACTIONS DURING THE SINTERING OF ELEMENTAL IRON-AND-ALUMINUM POWDER MIXES
Stanisław Jóźwiak*, Krzysztof Karczewski**, and Zbigniew Bojar***

INTRODUCTION

The heating of iron-and-aluminum powder mixes is accompanied by heat generation, due to the exothermic nature of the material. Initiation of an exothermic reaction in the compacted iron-and-aluminum powder mix is termed self-propagating high-temperature synthesis (SHS), or combustion synthesis.1–5 In combustion-synthesis reactions, the mix of reactant powders is compacted and subsequently ignited, either locally or by heating the entire compact to the ignition temperature of the exothermic reaction.

Compared with conventional sintering, the primary advantages of SHS are:

• A short exothermic reaction time
• The generation of a high reaction temperature that can volatilize low boiling-point impurities and result in enhanced purity
• A high thermal gradient and rapid cooling that can give rise to new nonequilibrium or metastable phases
• The opportunity for simultaneous synthesis and densification

The main disadvantage of SHS is the relatively high level of porosity in the final product, resulting in densities that can exceed 50% of the pore-free level.1–4,6 To reduce or eliminate porosity, the use of elemental iron-and-aluminum powder mixes with prealloyed intermetallic powders has been proposed.1 Others have described the application of pressure during combustion or sintering, which adds to the complexity of the overall process.2–4,7–10

Joslin et al.3,4 analyzed the fabrication of iron–aluminum alloys and determined that several factors affect the progress and timing of the attendant synthesis reactions. Powder-particle size, reaction atmosphere, heating rate, and composition all play a role. Ultimately, these
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factors affect heat generation and accumulation, the processes that govern the outcome of the reaction.

In our previous work, a cylindrical compact was sintered under a load of 50 kN. The load measurement was carried out under compression in an Instron Model #8802. Figure 1 shows that there is a significant change in the loading curve during the SHS reaction. This is indicative of the generation of high reaction energy and is believed to be the source of porosity in the sintered compact. The work presented here was undertaken to elucidate details of the reactions in the iron-aluminum binary system.

EXPERIMENTAL PROCEDURE

Elemental powders of iron (99.8 w/o) with an average particle size of 200 µm and aluminum (99.6 w/o) with an average particle size of 70 µm were combined to give the following compositions: Fe-40 a/o Al (24.4 w/o Al), Fe-45 a/o Al (28.3 w/o Al) and Fe-50 a/o Al (32.6 w/o Al). The powders were mixed utilizing ball milling and were subsequently cold compacted uniaxially.

Compacts were fabricated at a pressure of 1,000 MPa and cut into standard cylindrical DSC specimens (2 mm dia. and 70 mg weight) by spark machining. Thermal analysis was performed utilizing a DSC/thermogravimetric (TGA) instrument with a heating rate of 10°C/min from room temperature to 750°C in flowing argon (16 ml/min). The heating rate of 10°C/min was the same as that used during sintering in our previous work. DSC was used to determine the starting temperature of the SHS reaction and the change in heat evolution during the SHS reaction.

Microstructural evaluation and chemical analyses in sintered areas were carried out utilizing a Philips XL30 (LaB6) scanning electron microscope (SEM) integrated with X-ray energy dispersive microanalysis (EDAX). X-ray diffraction (XRD) phase analysis was performed on a 3003TT Seifert diffractometer with access to a PDF-2 database. Cu Kα, radiation (λ = 0.15405 nm), at an accelerating voltage of 40 kV and a current of 40 mA, was used in this study. The scan range was from 2θ = 20 to 120° with a scan time of 3 s and a step size of 0.02°.

Microindentation hardness measurements were carried out at room temperature using a Shimadzu Vickers tester with a 100 g load and 5 s dwell time.

RESULTS AND DISCUSSION

DSC traces for the Fe-40 a/o Al, Fe-45 a/o Al, and Fe-50 a/o Al powder mixes are shown in Figure 2. The corresponding DSC reaction parameters are cited in Table I.

Figure 3 shows changes in the microindentation hardness of the iron-and-aluminum constituent powders after heating the compact at a rate of 10°C/min. Cold work, as a result of deformation of the compact under 1,000 MPa pressure, caused an increase in the microindentation hardness of the aluminum powder from 26HV0.1 to
EXOTHERMIC REACTIONS DURING THE SINTERING OF ELEMENTAL IRON-AND-ALUMINUM POWDER MIXES

52HV0.1 and of the iron powder from 52HV0.1 to 123HV0.1. Supplying thermal energy to the compact resulted in a decrease in the microindentation hardness of the aluminum at 250°C and of the iron at 400°C, due to recovery and recrystallization.

Microindentation hardness increases after heating at temperatures >500°C for the aluminum powder and 550°C for the iron powder; however, it does not reach the same hardness as that of deformed iron and aluminum powders. Figure 4 illustrates the microstructure of a compact after heating to 600°C. No changes are visible in the compact compared with the microstructure of the pressed samples. Etch pits in the iron powder particles are the result of polygonization.

Figure 5 is a representative microstructure of a compact subjected to interrupted sintering at 610°C. At some contact points of the iron and aluminum particles, precipitates of Fe₅Al₂ are visible. Based on the binary Fe-Al equilibrium diagram, and in conformity with Fick’s laws of diffusion, the formation of FeAl₃ should precede the formation of Fe₅Al₂, however, FeAl₃ was not observed.

### Table I. DSC Reaction Parameters*

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<th>Aluminum Content (a/o)</th>
<th>Measurement Number</th>
<th>Onset of Peak #1 (°C)</th>
<th>Maximum of Peak #1 (°C)</th>
<th>Onset of Peak #2 (°C)</th>
<th>Maximum of Peak #2 (°C)</th>
<th>Offset (°C)</th>
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*Refer to Figure 2

---

**Figure 3.** Microindentation hardness of Fe-50 a/o Al powder mix after heating at a rate of 10°C/min.

**Figure 4.** Representative microstructure of Fe-50 a/o Al compact after heating to 600°C temperature.
The strain hardening of iron and aluminum after heating above 400°C suggests that the increase in microindentation hardness can be attributed to the precipitation of intermetallics with a high aluminum content and which have a larger lattice parameter than does iron and aluminum. The formation of FeAl$_3$ precipitates and the transformation in Fe$_2$Al$_5$ is reflected in the heat generation observed in the DSC curves (Figures 2 and 3).

The exothermic processes involving the formation of FeAl$_3$ and Fe$_2$Al$_5$ precede the SHS reaction (Figures 2 and 3) and influence the changes in the thermodynamic parameters cited in Table I during sintering of the compact. The release of internal energy resulting from the formation of FeAl$_3$ and Fe$_2$Al$_5$ is considered to be a fundamental factor in influencing the starting temperature of the SHS reaction. A higher aluminum content in the starting powder mix decreases the temperature of formation of the two intermetallics (Figure 6), and decreases the SHS start temperature (Figure 7). The increase in the formation enthalpy of FeAl$_3$ and Fe$_2$Al$_5$ (Figure 8) is reflected in an increase in the total energy of the compact; this allows the SHS reaction to initiate with lower external energy, i.e., at the lower heating temperature of the compact (Figure 7). According to the Gibbs-Duhem equation, the increase in aluminum content is responsible for the increase in energy released during the SHS reaction. Figure 9. This assumes that the enthalpy generated during the SHS reaction of the intermetallics remains approximately constant.

The thermodynamic parameters show monotonic changes during diffusion preceding the SHS reaction, and are dependent on the aluminum content (Figures 6, 7, 8, and 9).

The results of the XRD phase analysis are shown in Figure 10. These show that iron, FeAl$_3$, Fe$_2$Al$_5$, and FeAl are present after the SHS reaction. A quantitative determination of the proportions of each constituent was not made.

**SUMMARY AND CONCLUSIONS**

A DSC investigation of iron-and-aluminum powder mixes reveals that the reaction enthalpy undergoes monotonic changes over the aluminum range evaluated. The maximum enthalpy is attained in the stoichiometric composition FeAl. The analysis confirms the complexity of the Fe-Al intermetallic formation, which influences the pri-
mary SHS reaction parameters. Precipitation at the contact points of the iron and aluminum powder particles, observed prior to the SHS reaction, is an example of this complexity.

REFERENCES

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- 1 m = 3.28 ft. (foot)
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- 1 mm = 0.0394 in. (inch)
- 1 µm = 39.4 µin (microinch)
- 1 nm = 10 Å

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- 1 cm² = 0.155 in.² (square inch)
- 1 m² = 1.550 in.² (square inch)
- 1 cm³ = 0.061 in.³ (cubic inch)
- 1 m³ = 35 ft.³ (cubic foot)
- 1 L = 1,000 cm³ (cubic centimeter)
- 1 L = 0.264 gal. (gallons)

#### Amount of Substance Conversion:
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- 1 g/cm³ = 0.0361 lb. / in.³ (pound per cubic inch)
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- to convert K to °F (fahrenheit), multiply by 1.8 then subtract 459.4°F
- to convert °C to °F (fahrenheit), multiply by 1.8 then add 32°F

#### Heating and Cooling Rate Conversions:
- 1 K/s = 1°C/s = 1.8°F/s
- 1 K/min = 1.8°F/min

#### Mass Conversions:
- 1 g = 0.035 oz. (ounce)
- 1 kg = 2.2 lb. (pound)
- 1 Mg = 1.1 ton (ton = 2,000 pounds)

#### Force Conversions:
- 1 N = 10¹² dyne
- 1 N = 0.225 lbf (pound force)

#### Pressure, Stress and Strength Conversions:
- 1 Pa = 0.0075 torr (millimeter of mercury)
- 1 Pa = 10 dyne/cm² (dyne per centimeter square)
- 1 kPa = 0.145 psi (pounds per square inch)
- 1 MPa = 9.87 bar (atmosphere)
- 1 MPa = 145 psi (pounds per square inch)
- 1 MPa = 0.145 kpsi (thousand pounds per square inch)
- 1 Gpa = 145 kpsi (thousand pounds per square inch)

#### Energy Conversions:
- 1 J = 9.48·10⁻⁴ btu (British thermal unit)
- 1 J = 0.737 ft. · lb. (foot pound force)
- 1 J = 0.239 cal (calorie)
- 1 J = 10⁷ erg
- 1 J = 2.81·10⁻⁷ kw · h (kilowatt hour)
- 1 J = 6.24·10¹⁸ eV (electron volt)
- 1 J = 4.83 hp · h (horsepower hour)
- 1 J = 1 W · s (watt second)
- 1 J = 1 V · C (volt coulomb)
- 1 kJ = 0.239 kcal (kilocalorie)

#### Power Conversions:
- 1 W = 0.737 ft. · lb. / s (foot pound per second)
- 1 W = 1.34·10⁻³ hp (horsepower)

#### Thermal Conversions:
- 1 J/(kg · K) = 2.39·10⁻⁴ btu/(lb. · °F) (British thermal unit per pound per degree fahrenheit)
- 1 J/(kg · K) = 2.39·10⁻⁴ cal/(g · °C) (calorie per gram per degree celsius)
- 1 W/(m · K) = 0.578 btu/(ft. · h · °F) (British thermal unit per foot per hour per degree fahrenheit)
- 1 W/(m · K) = 2.39·10⁻³ cal/(cm · s · °C) (calorie per centimeter per second per degree celsius)

#### Viscosity Conversions:
- 1 Pa · s = 1 kg/(m · s)
- 1 Pa · s = 10 P (poise)
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