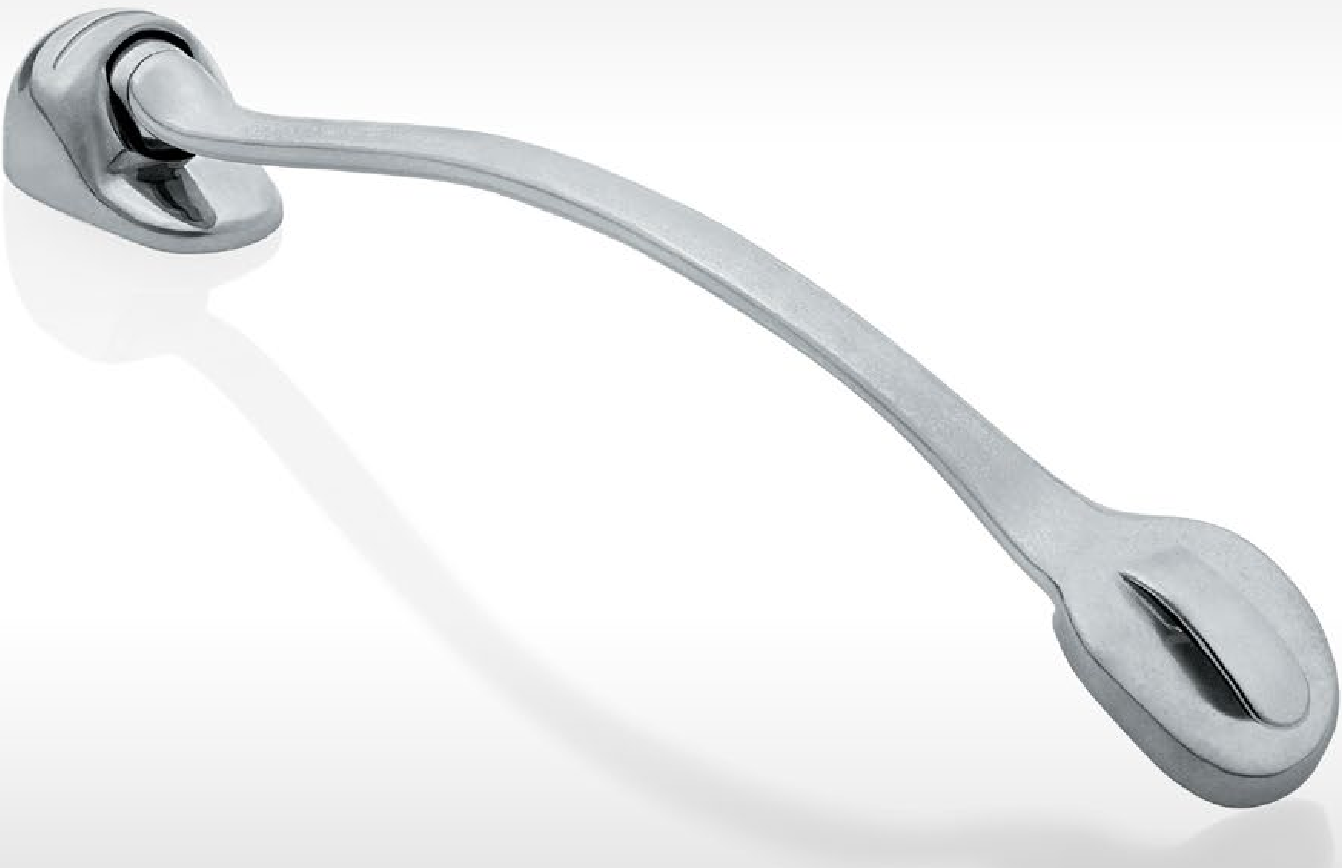
Vol. 8 No. 1 MARCH 2014

FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

POWDER INJECTION MOULDING INTERNATIONAL



in this issue

Company visit: Ortho Organizers

HIP of MIM components

Profile: Centorr Vacuum Industries

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POWDER INJECTION MOULDING INTERNATIONAL

For the metal, ceramic and carbide injection moulding industries

New opportunities in established markets

Welcome to the first issue of *PIM International* for 2014. With the PM World Congress scheduled for May 2014, those in our industry will once again have an unmissable opportunity to get together with parts producers, industry suppliers and leading researchers from around the world to share information and discover new opportunities. We look forward to exhibiting at PM2014 Orlando in May and hope to see you in the exhibit hall.

This issue of *PIM International* features two reports on PIM in the dental sector, one of the most important markets for PIM globally, but particularly in North America. "PIM in the dental sector: From orthodontic components to implants" reviews the growth of this sector and the new opportunities for the future (page 33), whilst our report on a visit to Ortho Organizers provides real insight into the use of MIM at a captive facility (page 38).

We also review the use of Hot Isostatic Pressing (HIP) for the processing of MIM parts to full density. As our report reveals, there are many reasons to consider the HIP process, however there are also several important considerations to bear in mind in order to gain the desired benefits (page 43).

As our industry continues to grow many part producers are feeling real pressure on their production capacity. The potential for investments in new furnaces is therefore generating a lot of interest in the industry from furnace manufacturers. We profile Centorr Vacuum Industries, one of North America's longest established suppliers of high temperature vacuum furnaces, and review their experiences of the MIM industry and recent commercial successes (page 51).

Looking to Asia, the recent and highly successful APMA 2013 conference attracted a truly international audience and we present a report on some MIM related highlights (page 73). We also publish a paper from Zhuzhou, China's hardmetal capital, on the injection moulding of hardmetal cutting tool inserts (page 73).

Nick Williams
Managing Director and Editor

Cover image
A Carriere Distalizer™ appliance manufactured by Ortho Organizers

March 2014

Powder Injection Moulding International





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To submit news for inclusion in *Powder Injection Moulding International* please contact Nick Williams nick@inovar-communications.com

ARC Group expansion and MIM school dates announced

ARC Group Worldwide, Inc., based in Longmont, Colorado, USA, announced in December 2013 that Advanced Forming Technology, Inc. (AFT), a division of the company's Precision Components Group, has begun expanding its operational capabilities at its Colorado location.

Commenting on the expansion, Jedidiah Rust, Director of Operations for AFT Colorado, stated, "AFT continues to experience growth within our major market segments, demonstrating the quality of our products and services, and necessitating the need for additional capacity." AFT currently services five growth industries, including automotive, medical, aerospace, firearms and consumer. Rust further commented, "In order to continue to meet our customers' high demand, AFT has taken the initiative to increase output through infrastructure investment and lean manufacturing initiatives." Expansion completed to-date represents the first of several phases, with the planned expansion expected to be complete by December 2014.

Thomas K Houck, Vice President of US Metal Injection Molding Operations, stated, "This capital expansion and infrastructure upgrade will enable us to meet current demand and grow our business for the future. Our continued focus on operational excellence will provide the company with the strengthened ability to expand volumes, provide additional value added processes to our customers and increase our 3D printing capabilities/services. These initiatives, combined with our expected growth, should position the company for continued success with a diverse, captivated customer base, innovative technology solutions, and increased shareholder value."

Schedule for AFT's "MIM School" seminars announced

AFT's MIM School, started in 2005, is a two day training seminar for new and existing ARC customers, vendors and shareholders wishing to learn more about the MIM process. Since the start of the school, AFT has trained over 250 students on MIM technology. The seminar walks students through the technical aspects of MIM, from the materials technology of metal powders, to the compounding and secondary processes, through the use of both classroom and hands-on training on the manufacturing floor.

New to the school this year is a course on the company's 3D printing capabilities. Through the launch of ARC's new division, 3D Material Technologies (3DMT), ARC has been building up capital equipment of the newest models of metal and plastic 3D printers. 3DMT is housing its 3D printers in Colorado, as well as ARC's other facilities. The new 3D course will allow students the unique opportunity to get hands-on experience with a number of new 3D printing technologies.

The 2014 classes are free to attend for registered applicants only, and will be held on the following dates from February 19-20, May 21-22, August 20-21 and November 12-13. For more information email mimschool@arcmim.com. The company states that spaces are only for qualified attendees and are limited.

www.arcgroupworldwide.com

Arcam acquires MIM titanium powder manufacturer AP&C from Raymor Industries

Sweden's Arcam AB has signed an agreement to acquire the AP&C division from Raymor Industries for a total of $35 million Canadian dollars. The move will see Arcam secure its supply of titanium powder used in the production of components via the company's Additive Manufacturing process.

AP&C is a global manufacturer of high quality metal powders and has been a supplier of titanium powders to Arcam since 2006. Titanium powder is an important part of Arcam's offering to its customers.

"With this acquisition Arcam secures access to the optimum production of high grade metal powders for our customers and we also add technology and expertise in powder metal production for 3D-printing in general and other advanced applications," stated Magnus René, President and CEO of Arcam.

AP&C uses proprietary plasma atomisation technology to produce metal powders, with titanium alloy powder being the largest product. A significant part of AP&C sales is to the 3D-Printing industry with other markets including Metal Injection Moulding (MIM), powders for spray coatings as well as powders for HIPed components. Arcam and the team at AP&C intend to continue to expand the powder business and advance the plasma atomisation technology.

The AP&C division is expected to generate CAD$6.5 million of revenue during 2013 with an EBITDA result of around CAD$1.5 M. The acquired business, which currently employees 29, will become a subsidiary of Arcam and continue operating with the existing management team.

"With this deal we will be a part of the leading company in 3D-printing in metals. Because of our long term close cooperation with Arcam we know that this deal will give us a very good platform for continued growth in the 3D-printing industry. Being part of a larger group will also help in accelerating growth to better service the overall metal powder market", stated Jacques Mallette, President of Raymor Industries and future President of Arcam's powder business.

The total purchase price amounts to CAD$35 million where a cash payment amounting to CAD$20 million will be paid on closing and the remaining part as two instalments to be made in 2015 and 2016 subject to certain targets being met. Closing of the acquisition is subject to customary closing conditions and is expected to take place in the first quarter 2014.

www.arcam.com

New cooling system from Hasco prevents hot spots in injection moulding

Hasco Hasenclever GmbH of Lüdenscheid, Germany, a supplier of metric mould plates, mould components and hot runner systems, has introduced its new CoolCross Z99 cooling system for injection moulds. The system makes possible, for the first time, to have cooling channels crossing each other on the same plane in a flexible manner without any major outlay on production.

CoolCross permits a homogeneous temperature distribution at the core or insert, as well as constant cavity cooling on all four sides for the full duration of the injection moulding cycle, states Hasco. The system also prevents hot spots. It also provides protection against rotation, which is achieved through a locking mechanism, and prevents the unintended closure of the cooling channels. Different independent cooling circuits cross each other on the same plane, making it possible to incorporate small plate thicknesses and inexpensive accessory components in the mould design. By reducing the plate thickness, use can be made of shorter nozzles, guidance and attachment elements.

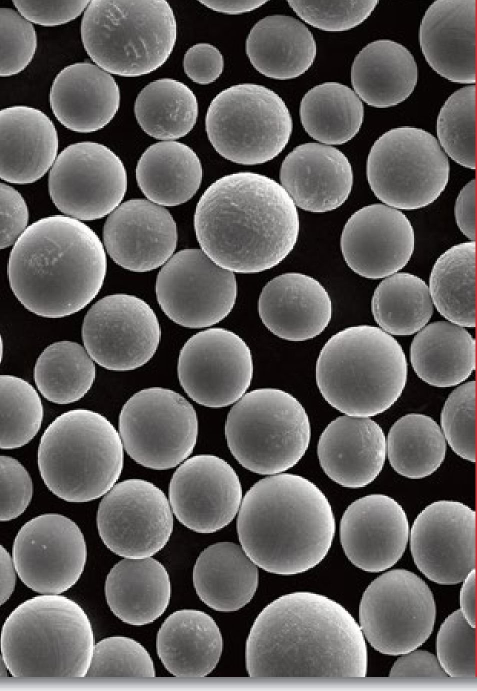
Hasco states that the number of cooling channels can be reduced, since it is no longer necessary to have a cooling channel in an additional plane. If the CoolCross is used in thicker plates, the installation depth can be selected on a variable basis. "By employing the innovative CoolCross Z99, which is available exclusively from Hasco for the cooling circuits in injection moulds, it is possible to achieve time and cost savings for both the mould unit and the accessory components," the company stated.

www.hasco.com ■

Medical/dental ceramics market to grow to $2.3 billion by 2018

NanoMarkets, of Glen Allen, Virginia, USA, has published its report "Worldwide Medical Ceramics Markets: 2013" providing a detailed analysis of emerging medical and dental market opportunities for alumina, zirconia, silicon ceramics, hydroxyapatite, bioglass, piezoceramics, nanoceramics and other materials. The company forecasts worldwide medical ceramics to grow from $1.1 billion in 2013 to $2.3 billion in 2018. Ceramics are widely used in medical and dental applications including hip and knee implants; crowns, bridges and other dental implants; surgical and diagnostic tools, implantable electronic implants and regenerative medicine.

www.nanomarkets.net ■



Your Premier Source for Hydride–Dehydride (HDH) and Plasma Spheroidized (PS) Titanium Powders.

AMETEK Specialty Metal Products – Reading Alloys is a leader in producing specialty alloys and fine powders. The company uses a variety of manufacturing processes to produce titanium powders that meet ISO 9001 / AS 9100 requirements.

The company utilizes a new plasma manufacturing method which combines an established powder manufacturing process with the Plasma Spheroidization (PS) process to offer customers an alternative high volume, spherical titanium powder production process.

HDH Powders: Greater Control of PSD

- Available in Ti Sponge, CP Ti, Ti-6AL-4V Standard (Grade 5) & ELI (Grade 23)
- Particle sized from 45 microns (325 mesh) to 500 microns (35 mesh)
- Solid, angular, block or sponge morphology

PS Powders: Free Flowing

- Available in CP Ti, Ti-6AL-4V Standard (Grade 5) & ELI (Grade 23)
- Particle sized from 45 microns (325 mesh) to 500 microns (35 mesh)
- Spherical morphology, tight PSD, no satellites, agglomerates or entrapped argon

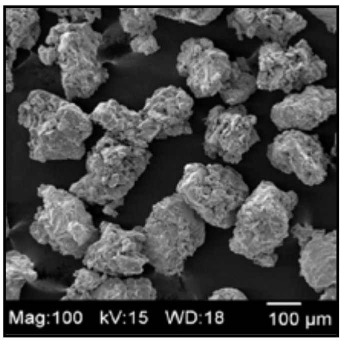
Powder Metallurgy Applications

- Plasma Spray/Coating
- Cold Isostatic Pressing / Sintering
- Hot Isostatic Presssing
- Metal Injection Moulding
- Additive Manufacturing

Markets Served

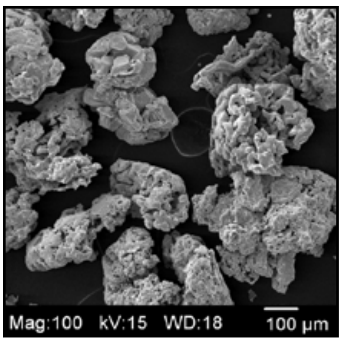
- Medical Device
- Thin Film (Sputtering Target)
- Feedstock
- Electronics
- Net Shape

Morphology: HDH Magnesium Reduced Titanium Sponge Powder



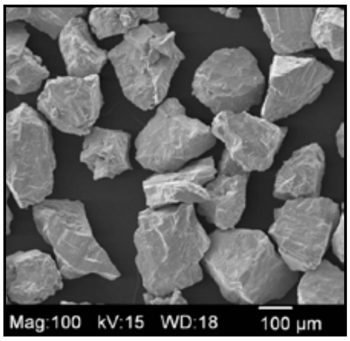
SEM image (x100) of HDH Magnesium Reduced Ti Sponge Powder, 70 mesh (212μm) x 100 mesh (150μm)

Morphology: HDH Sodium Reduced Titanium Sponge Powder



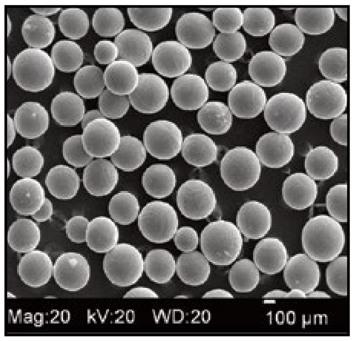
SEM image (x100) of HDH Sodium Reduced Ti Sponge Powder, 70 mesh (212μm) x 100 mesh (150μm)

Morphology: HDH CP Titanium Powder



SEM image (x100) of HDH CP Ti Powder, 70 mesh (212μm) x 100 mesh (150μm)

Morphology: PS CP Titanium Powder (99%+ spherical)



SEM image (x20) of PS Ti powder, 35 mesh (500 microns) x 45 mesh (355 microns)

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Milestone passed in PM scientific publishing

In early January of this year Professor Randall German, Associate Dean for Engineering Research at San Diego State University, California, USA, passed a milestone with his 1000th scientific publication.

The specific article was a collaborative effort with Jose Alvarado-Contreras, Eugene Olevsky and Andrey Maximenko entitled "A continuum approach for modeling gravitational effects on grain settling and shape distortion during liquid phase sintering of tungsten heavy alloys" published in *Acta Materialia* (2014, vol. 65, pp. 176-184). It detailed modelling research in preparation for experiments to be performed on the International Space Station in 2015.

Now, at 1003 published articles, German is also responsible for fourteen books, five recorded short courses, four market studies, nineteen edited books, and twenty-five issued patents.

His newest book "Sintering: From Empirical Observations to Scientific Principles" is just being released by Elsevier Scientific. His first article was in *Metallurgical Transactions* in 1972 and with George St. Pierre dealing with the high temperature thermodynamics of nickel-titanium alloys. He has typically averaged two to three co-authors per paper, involving more than 300 graduate students and postdoctoral researchers working under his supervision.

Submitting News

To submit news to *Powder Injection Moulding International* contact Nick Williams: nick@inovar-communications.com

Amedica signs agreement with Kyocera for silicon nitride medical devices

Amedica Corp, Salt Lake City, Utah, USA, a biomaterials company using silicon nitride ceramics for application in a broad range of medical devices, has announced that it will collaborate with Kyocera Industrial Ceramics Corp. to manufacture medical devices using Amedica's silicon nitride. The silicon nitride components will be produced at Kyocera's Vancouver, Washington, facility and will include Amedica's FDA approved spinal interbody devices.

Silicon nitride devices have been shown to help promote bone growth and have anti-infective properties. They are also semi-radiolucent with clearly visible boundaries enabling an exact view of intra-operative placement and post-operative fusion assessment via common imaging modalities.

Sandvik Osprey appoints new sales manager for the Asian market

Sandvik Osprey, a leading supplier of gas atomised metal powders for the Metal Injection Moulding (MIM) industry, based in Neath, South Wales, UK, has appointed Dr Paul Davies as Powders Sales Manager for the Asian market.

Davies was formerly Sales & Marketing Manager at Osprey from to 2001 to 2008 but has spent the past five years working for Tata Steel Europe managing the development of new advanced materials for the Defence and Security Industries.

Dr Martin Kearns, Powders Group Director at Sandvik Osprey, told *Powder Injection Moulding International*, "Paul brings with him not only a wealth of knowledge and experience in Powder Metallurgy from his previous time with the company but also new business development skills from his time working in the steel industry. We are very excited that he is re-joining the team at Sandvik Osprey and look forward to working with him to grow or activities throughout Asia."

Davies told *Powder Injection Moulding International*, "I am delighted to be coming back to work at Sandvik Osprey. It's been great to see the impressive growth that the company has achieved over the past five years and I look forward to contributing to the future success of the business. Asia is a dynamic market with significant potential both in Metal Injection Moulding as well as other advanced Powder Metallurgy applications."

www.smt.sandvik.com/osprey

2014 APMI fellows announced

John R Engquist, Consultant, JENS Solutions LLC., a recognised expert in the PM industry, and Z Zak Fang, University of Utah Metallurgical Engineering Professor who is a world-renowned expert in the cemented carbides and titanium industries, have been selected to receive the 2014 Fellow Award from APMI International.

The fellowship is APMI's most prestigious award, recognising members for their significant contributions to the goals, purpose and mission of the organisation as well as for a high level of expertise in PM. The recipients will receive elevation to Fellow status at the PM2014 World Congress in Orlando during the Industry Luncheon on May 19, 2014.

www.mpif.org

MIM helps Raytheon improve product performance and lower manufacturing costs

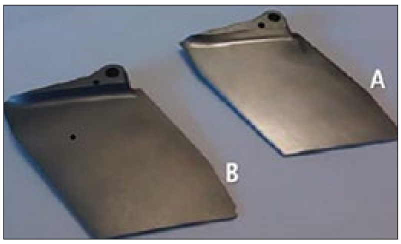

Fig.1 Excalibur control fin. The machined part (A) and the part manufactured by MIM (B) (Courtesy Raytheon Corp)

Raytheon Company is an international aerospace and defence supplier headquartered in Waltham, Massachusetts, USA, with sales exceeding $24.4 billion in 2012. Writing in *Raytheon Technology Today* (2013) Mitchell Gross stated that Metal Injection Moulding (MIM) is employed by the company to produce metal structures both simple, complex and sophisticated, with some of the latter being difficult if not impossible to produce using other manufacturing methods.

One example cited is a control fin for the Excalibur, a guided artillery projectile which provides precision fire at extended ranges for all current and future 155 mm howitzers. The original Excalibur control fin (Fig. 1A) was composed of 17-4PH precipitation hardened stainless steel. Machining was extremely time consuming, requiring tens of passes with a tool bit

Stainless Steel Material	Yield Strength (ksi)	Ultimate Tensile Strength (ksi)	Elongation %	Modulus Elasticity (Gsi)
MIM 17-4PH H1100	138	149	14	37.9
SAE AMS 5604 H1100	115 minimum	140 minimum	5 minimum	28.5
MIM 17-4PH H1025	139	158	9	42.4
SAE AMS 5604 H1025	145 minimum	155 minimum	5 minimum	28.5
MIM 17-4PH H900	164	184	10	32.1
SAE AMS 5604 H900	170 minimum	190 minimum	5 minimum	28.5

Table 1 Properties of MIM 17-4 PH steel in comparison to precipitation hardened 17-4 PH steel (i.e., SAE AMS 5604) for different heat treatment conditions (H1100, H1025, H900)

on both sides of the fin to create its complex shape. Tolerances were said to be difficult to maintain from machined part to machined part.

Using MIM, the airfoil shape is injection moulded in one piece (Fig.1 B). Because the hardened steel die cavity used in MIM does not change from shot to shot over a minimum of a quarter of a million shots, tolerances are easily achieved. After debinding, the fin is sintered at 1260°C, with the sintered part containing less than 2% porosity. The airfoil is then coined to the final shape and attachment points are machined to their final dimensions. Final heat treating is undertaken to attain the strength required.

Table 1 compares the properties of MIM 17-4PH material with 17-4PH precipitation hardened stainless steel, showing that they have comparable performance. The cost of manufacturing the control fin by MIM, however, is said to be only 25% that of the cost using traditional machining methods.

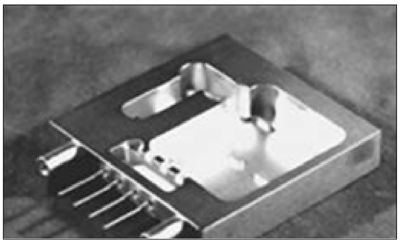

Fig. 2 Metal Injection Moulded RF housing (Courtesy Raytheon Corp)

The second example of a MIM application at Raytheon is a radio frequency (RF) housing, or electronics enclosure, which the company uses in large quantities. MIM is said to be ideally suited to producing the thin walls and unique hole design (Fig. 2).

Electronics packages normally require a thermal management system attached with an adhesive bond. The thermal management system is, therefore, limited in its ability to remove heat from the electronics package because of the thermal properties of the adhesive and the bond thickness.

With MIM, alloy compositions can be adjusted so that the electronics package and the thermal management system can be co-moulded and processed together to achieve higher heat dissipation.

Raytheon states the Kovar ratio of iron and nickel can be changed to lower the coefficient of thermal expansion (CTE) of the alloy so the CTE of the alloy is closer to the CTE of glass. This lower alloy CTE reduces the probability of cracking the glass seal around package leads, a common cause for the loss of package hermeticity and of the failure of electronic components inside the package. These attributes are said to result in higher-value systems that have better performance at lower cost.

www.raytheon.com

Submitting News

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New report on the global market for Metal and Ceramic Injection Moulding

BCC Research of Wellesley, Massachusetts, USA, has released a new 482 page report (January 2014) reviewing the global MIM and CIM components market. In the last BCC Research report issued in 2009 it put the market size for MIM to be $985 million, with expected growth to $1.9 billion by 2014. Now BCC Research projects the global market to grow to nearly $2.9 billion by 2018 from $1.5 billion in 2012 and register a six-year compound annual growth rate of 11.4% from 2012 to 2018. Although the emphasis is mainly on the MIM market, the CIM market is also discussed.

BCC Research stated that the global market for components manufactured by Metal Injection Moulding and Ceramic Injection Moulding has exhibited spectacular growth over the last two decades and the trend is expected to continue. While the market crash in 2008 and 2009 had its share of repercussions in the MIM industry, with automotive sales dropping significantly across the world, larger companies have weathered the storm and have grown stronger and larger in the meantime. Key to this staying power has been diversification across other applications such as medical and dental, aerospace, electronics and communications and luxury products.

Growth in the market for cell phones and other handheld products has been a boon to the MIM industry for the last decade and this has also resulted in the industry shifting its base slowly to emerging markets and Asia, where most of the electronics manufacturing takes place. The report discusses various trends in the geographical growth of the industry.

Another significant change in the industry is the size of the largest companies starting to cross the $50 million sales mark. The first two MIM manufacturers to break this barrier, stated BCC Research, are the ARC Group, because of its recent acquisitions, and Indo-US MIM Tec. BCC Research expects that there could be five to ten players crossing the $50 million mark by 2018, with several crossing the $100 million threshold. The final chapter of the report provides detailed profiles of nearly 200 leading companies.

On the supply side, several companies are expected to be enjoying $50 million in purchases from the MIM industry in the next few years. The supply side chapter covers powder manufacturers, feedstock manufacturers and equipment makers who support the MIM and CIM industry. Application markets are analysed in detail, particularly for the firearms market.

The report, *Global Market for Metal and Ceramic Molding*, is available for $6650.

www.bccresearch.com

Metalysis' titanium powder used to 3D print automotive parts

Metalysis Ltd, Rotherham, UK, has announced that low-cost titanium powders developed by the company have been used to 3D print automotive parts for the first time.

The use of titanium powders in 3D printing has been prohibitively expensive until now because titanium powders currently sell for $200 - $400 per kilogram, states Metalysis. Metalysis has developed a new way of producing low-cost titanium powder, which it believes will heralds a new era in Additive Manufacturing and see greater use of titanium in components across the automotive, aerospace and defence industries.

In a further development, the titanium powder used to manufacture the automotive parts is also a world-first, as Metalysis has created titanium from rutile sand, a naturally occurring titanium ore present in beach sands, in one single step. The use of this inexpensive and plentiful feedstock for titanium manufacture will dramatically reduce the cost of titanium production, allowing its increased use.

The Metalysis process is radically cheaper and environmentally benign compared with existing titanium production methods, states the company. Currently, the manufacture of titanium powder involves taking the metal sponge produced by the Kroll process, which is then processed into ingot billets, melted into bar form and finally atomised into powder, resulting in a costly and labour-intensive four-step process. Metalysis takes rutile and transforms it directly into powdered titanium using electrolysis. The low-cost titanium powder can be used in a variety of new applications whereas previously the metal has been excessively expensive for use in mass production of lower value items.

The Mercury Centre, which sits within the Department of Materials at Sheffield University, used Renishaw's 3D printer to make the parts, demonstrating the feasibility of producing titanium components using additive layer manufacturing. 3D printing brings further cost benefits by reducing waste because the current means of production is subtractive, as components are shaped out of metal billets, which wastes a huge amount of material. Metalysis' low-cost titanium powder enables additive manufacturing with its metal powder, thereby reducing the quantity of material required.

Dion Vaughan, CEO of Metalysis added, "Metalysis' rutile-derived titanium powder is produced at lower cost and is suitable for 3D printing so that manufacturing metal components becomes more economical. The Metalysis process could reduce the price of titanium by as much as 75%, making titanium almost as cheap as specialty steels."

www.metalysis.com

Nanostructured powder helps improve the performance of structural ceramics

Innovnano, a manufacturer of high performance ceramic powders based in Coimbra, Portugal, has produced 3 mol % yttria-stabilised zirconia (3YSZ) for high strength structural ceramics. Manufactured to ensure retention of an intrinsic nanostructure, Innovnano's 3YSZ powder provides the advantageous properties associated with small grain sizes, including high chemical homogeneity and density. Ideal for technical ceramics such as valves and process equipment, nanostructured 3YSZ feedstock translates to ceramic components with exceptional bending strength, fracture resistance and tribological performance, to withstand the strain and friction that is placed on moving parts and structural elements for long-term performance.

Offering enhanced electrical and thermal insulation properties, Innovnano's nanostructured 3YSZ is produced via the patented Emulsion Detonation Synthesis (EDS) manufacturing technology, which ensures an even distribution of yttria throughout the zirconia lattice. Importantly, this high chemical homogeneity overcomes brittleness and supports a constant coefficient of thermal expansion, negating any risk of thermal stress and subsequent material fracture for steadfast reliability. Furthermore, with an expansion coefficient similar to steel, nanostructured 3YSZ is an ideal corrosion-resistant alternative for traditional steel components.

For optimal processability and user-handling, Innovnano's 3YSZ can be supplied in an application-specific form, as a spray-dried granulated powder (with or without binder), suspension or slurry. João Calado, Chief Technology Officer at Innovnano, stated, "It is important for us to respond to and develop practical solutions to the increasingly demanding applications required by the ceramic industry. For this reason, we provide our powders in a range of formats, enabling our customers to explore and take advantage of the enhanced chemical and physical properties offered by a nanostructured material, but with the handling ease of conventional microstructured feedstock."

Compared to microstructured ceramic powders, powders with an inherent nanostructure show increased chemical activity. Importantly this enables ceramics to be produced at lower sintering (processing) temperatures, reducing energy costs and minimising grain growth. In this way, the nanostructure is retained, translating to higher performance ceramic parts and increased value of the end-product.

www.innovnano-materials.com

Powder Injection Moulding at the PM2014 World Congress, Orlando

The global PIM and PM communities will soon come together for the 2014 PM World Congress and Exhibition, taking place at the Walt Disney Dolphin Hotel, Orlando, Florida, from May 18-21. The various programmes scheduled at the event promise to offer a surfeit of development and technology trends on all aspects of PM. There are, however, numerous activities at PM2014 that will be of specific interest to those in the PIM community.

It is not the first time that the PM World Congress and Exhibition has been held in Orlando. Such is the attraction of the destination, with its many world class theme parks and recreation activities, it is little wonder that the Metal Powder Industry Federation (MPIF) and the American Powder Metallurgy Institute (APMI) decided to return to Orlando for the most important PM event of the year. Even for the most hardworking delegates and exhibitors there will opportunities to enjoy some of the excitement of Orlando at social events such as the Opening Night Dinner (Sunday, May 19) during the International Street Festival and the spectacular Epcot Illuminations. The PM2014 Gala Closing Dinner (Wednesday, May 21) promises to be the social highlight of the congress. There will also be a specific 'Metal Injection Moulding Industry Event' (by invitation only) on Tuesday evening, May 20.

MIM highlights at PM2014

There are no fewer than ten sessions devoted to MIM as part of the large offering of PM technology presentations in the PM2014 technical programme. These sessions will cover binder materials, moulding, powder distribution effects in feedstock, magnetic materials, titanium, high temperature alloys, and advanced materials and processing. In addition, there are three sessions on microPIM and Ceramic Injection Moulding.

Running alongside the main PM2014 Technical Programme will be a number of Special Interest Programmes which, whilst focusing on mainstream PM materials, will also be relevant to the PIM community. These include fatigue properties of PM steels, NDT/failure analysis in PM, machinability of PM components, and technology for growth.

There will be a special luncheon during PM2014 (Tuesday, May 20) at which the MPIF Annual Awards for PM Design Excellence will be presented. It is anticipated that a significant number of MIM parts will feature as winning components. The special session devoted to regional reviews of the PM industries in North America, Europe and Asia (Wednesday morning, May 21) will focus on industry business conditions, technology trends, and markets for PM and particulate materials, including Powder Injection Moulding

Inaugural Additive Manufacturing Conference at PM2014

Of interest to the MIM community from the perspective of producing prototypes will be the inaugural conference on 'Additive Manufacturing with Powder Metallurgy – AM/PM' (May 19-20) which will run alongside the PM2014 World Congress.

Additive Manufacturing (AM) has made great strides in recent years for the fast production of complex three dimensional solid objects by, in most cases, adding successive laser sintered layers of metal or alloy powder material. The thirteen technical sessions at the Additive Manufacturing conference will review recent developments and applications for a number of sectors including medical devices and implants, aerospace and defence.

Sessions will also cover metal powders suitable for AM, equipment technology, design and process modelling/simulation, and non-powder AM technologies.

PM2014 Exhibition: Diverse powder technologies on offer under one roof

Bonding all of the diverse powder technologies which will be highlighted during the PM2014 Powder Metallurgy World Congress will be the PM2014 Exhibition. This is open from Monday morning through to Wednesday afternoon. The PM2014 exhibition will be a great opportunity to meet worldwide MIM and PM colleagues in the exhibition hall conveniently located next to the PM2014 Congress at the Walt Disney World Dolphin Hotel.

Some 115 exhibitors are taking over 150 booths at this year's World PM Congress from every part of the PM supply industry, including producers of metal powders, lubricants, MIM feedstock, tooling, compacting presses, injection moulding machines, additive manufacturing equipment, sintering furnaces, furnace belts, powder handling and blending equipment, quality-control and automation equipment, particle-size and powder-characterization equipment and much, much more. The team from *Powder Injection Moulding International* and *Powder Metallurgy Review* will be exhibiting on booth 222.

A major attraction in the PM2014 Exhibition will be the display of all the entries submitted to the Annual PM Design Excellence Awards competition. Winning parts will be identified following the Awards Luncheon on Tuesday, May 20.

A buffet lunch will be held in the PM2014 exhibition area to promote the location of the following PM World Congress & Exhibition which will be held in Hamburg, Germany, in 2016.

www.mpif.org

New approach to produce high performance MIM heat sinks from copper matrix nanocomposites

Metal Injection Moulding (MIM) is already a proven and well accepted manufacturing process for heat sink devices used in thermal management systems for modern microprocessors, with sintered MIM copper heat sinks often outperforming commercially pure cast copper alloys. However, with the continuing trend towards lower cost and smaller sized high performance electronic devices generating more heat, new thermal management solutions are needed to provide cost effective ways of dissipating heat.

Responding to these challenges researchers in the Mechanical Engineering Department at the University of Technology Petronas (UTP), Tronoh, Malaysia, have developed a new generation heat sink material made from copper powder reinforced with multiwall carbon nanotubes (MWNTs) using MIM technology.

Ali Samer Muhsan and colleagues have reported on the outcome of their work to develop the new copper matrix/MWNTs in the open access *International Journal of Manufacturing Engineering* (Vol. 2013, 2013, Article ID 386141, 9 pages). Key to ensuring a homogeneous dispersion of MWNTs in the copper powder matrix was using a new multilevel mixing approach.

The authors stated that the MWNTs were first purified and functionalised using concentrated acids. Functionalisation was then applied to enhance the dispersion of purified MWNTs and to create sidewalls groups that have the potential to bind MWNTs to the Cu matrix. MWNTs were subsequently washed with distilled water, dried and mixed with paraffin wax (PW) diluted in heptane solvent along with magnetic stirring. This was followed by a sonic agitation process to ensure uniform dispersion of MWNTs in PW solution.

The dried MWNTs/PW mixture was then mixed with the other components of the binder system (polyethylene; stearic

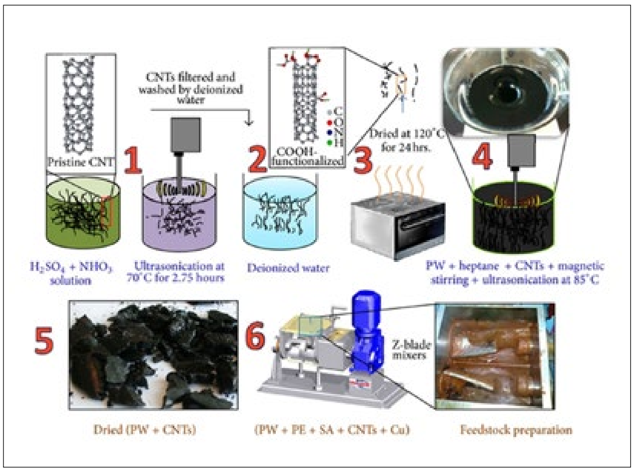


Fig. 1 Nanoscale dispersion process of MWNTs in Paraffin Wax (PW)

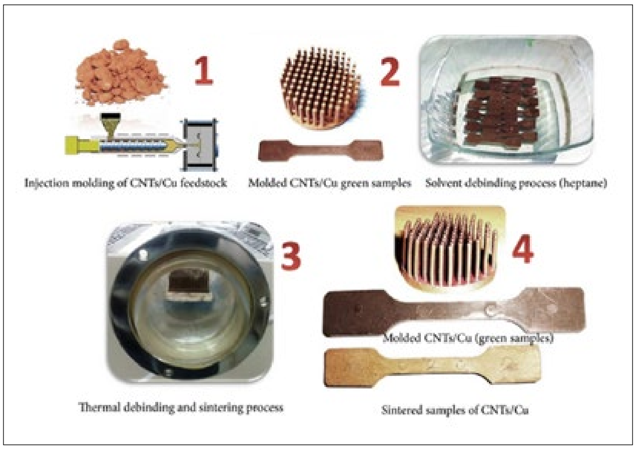


Fig. 2 MIM of Cu-MWNTs nanocomposites

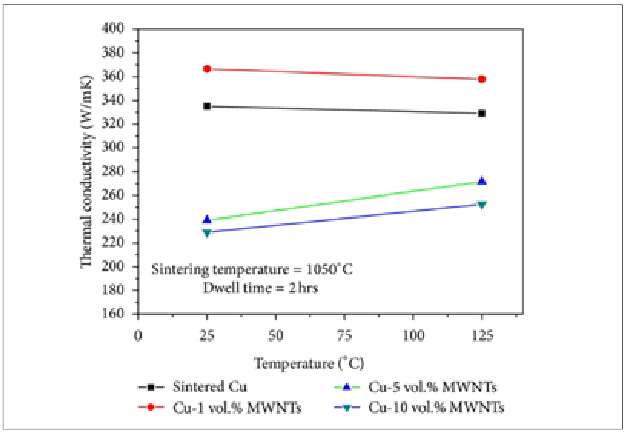


Fig. 3 Thermal conductivities of MWNTs/Cu composites as a function of MWNTs volume fraction with two different measuring temperatures (25 and 125°C)

acid) in a Z-blade mixer at 160°C. This step provided a high-viscous liquid medium with high shear forces that allowed the MWNTs clusters to be properly mixed resulting in uniform dispersion in the molten binder system. The copper powder was added gradually during the mixing process to form Cu-MWNTs-binder feedstock. (Fig. 1)

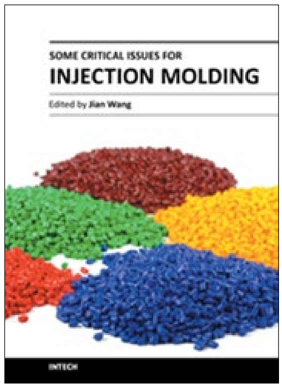
Following injection moulding the binder is removed from the Cu-MWNTs matrix by solvent and thermal debinding followed by sintering in argon at 950, 1000 and 1050°C.

Microstructural analysis showed that the dispersion of MWNTs was significantly improved using the new multilevel mixing technique, and that there was little mechanical damage to MWNTs structure. There was good interfacial bonding at the Cu/MWNTs interfaces. It was also found that the high-shear forces from the binder system components during feedstock preparation helped to disperse and exfoliate the MWNTs clusters into the Cu matrix homogeneously.

The thermal conductivities of Cu-MWNTs nanocomposites as a function of MWNTs volume fraction with two different measuring temperatures (25 and 125°C) is shown in Fig. 3. The highest value was recorded at Cu-1 vol.% MWNTs (366.516 W/mK) sintered at 1050°C for 120 minutes. This value corresponds to an increase of ~32 W/mK (11.25%) over that of the sintered copper (Cu matrix).

www.utp.edu.my

Open access book covers 'Some critical issues for injection moulding'



InTech Europe and InTech China have made available, as an open-access book, a practical 270 page guide to injection moulding. The book, first published in hardback form in 2012, is edited by Dr Jian Wang and contains individual chapters written by leading academics who provide some clear presentations of the injection moulding process and equipment. The book has the aim of directing people in plastics and MIM/CIM manufacturing to solve problems and avoid costly errors.

The first two chapters provide an introduction to PVT properties of polymers for injection moulding, and optimisation of the injection moulding process. The following five chapters are all devoted to Powder Injection Moulding. The PIM chapters include a comprehensive introduction and review of PIM technology by Joamín González-Gutiérrez and colleagues covering feedstock preparation, binder formulation, metal and ceramic powders used including submicron powders, injection moulding machines, debinding and sintering.

Other chapters involving PIM include: 'Wick Debinding - An Effective Way of Solving Problems in the Debinding Process of Powder Injection Molding' by Lovro Gorjan; 'Micro Metal Powder Injection Molding' by Kazuaki Nishiyabu; 'Ceramic Injection Molding' by Zdravko Stanimirovi and Ivanka Stanimirovi; 'Optimization and Simulation for Ceramic Injection Mould of ZrO_2 Fibre Ferrule' by Bin Lin, *et al*.

The remaining chapters review developments in microcellular foam injection moulding; insert moulding process employing vapour chamber; thermoplastic matrix reinforced with natural fibres – study of interfacial behaviour; and finally the properties of injection moulded high density polyethylene nanocomposites filled with exfoliated graphene nanoplatelets.

'Some Critical Issues for Injection Molding' , Dr Jian Wang (Ed.), ISBN: 978-953-51-0297-7.

www.intechopen.com/books

Submitting News

To submit news to *Powder Injection Moulding International* contact Nick Williams: nick@inovar-communications.com

Feedstock specialist launches new unit for the automatic solvent debinding of MIM and CIM parts

PIM binder and feedstock specialist eMBe Products & Service GmbH, based in Thierhaupten, Germany, has launched a new unit for the automatic solvent debinding of MIM and CIM parts. The debinding unit, branded Dee-Solver, is the result of a partnership with Germany's Techtor GmbH. The companies state that this custom manufactured equipment is suitable for all PIM producers who are developing and producing parts using a solvent debinding process.

The Dee-Solver system will be offered in a number of different sizes. The launch model, with a 50 litre capacity, works fully automatically with an SPS controller and up to ten processes can be programmed and stored on a touchscreen by the user. The unit contains a programmed solvent recovering system, a binder separator and a vacuum dryer with condenser. A nitrogen flush prevents dangerous solvent-air mixing.

The system, which is designed and manufactured in Germany, uses solvent-resistant long-life components and meets the most stringent safety and environmental regulations. The same system can be used for water-debinding as well. In this process a recovery of the binder is not needed, as the removed biodegradable binder can be put in the waste-water.

Michael Bayer, Managing Director of eMBe Products & Service GmbH, told PIM International "The advantage that we bring with this product is that it has been developed and manufactured by people who have a deep knowledge of PIM binder and feedstock compositions. Many small companies who are interested in PIM are scared when they receive quotations for capital equipment. PIM is a very expensive technology and many products will never be launched because of this. Here we see an opportunity to offer attractive, simply made products with low purchase and running costs. We invite interested parties to come and test the system in our facility."

Commenting on the trend towards solvent debinding, Bayer stated "Solvent debinding, in particular ethanol-debinding, is a method where you do not need additional chemistry such as nitric acid, which is strictly regulated by export rules world-wide, and expensive afterburners to deal with formaldehyde loaded gas. This solvent is readily available worldwide and having it in a DeeSolver system as a debinding medium is risk-free. The procedure is as simple as starting a dishwasher." For more information contact Michael Bayer, email servicepoint@embe-products.com.

www.embe-products.com

Exterior view of the new Dee-Solver solvent debinding unit from eMBe Products & Service GmbH

Interior view of the new Dee-Solver solvent debinding unit from eMBe Products & Service GmbH

New company created for the 3D printing of functional ceramic components

A selection of printed ceramic components including gears, filter elements and a watch case

Dutch Ceramic Injection Moulding (CIM) specialist Formatec Ceramics, based in Goirle, the Netherlands, consulting company Innotech Europe and Energy Research Centre Netherlands (ECN) announced in December that their research collaborations have resulted in the foundation of a new company, ADMATEC Europe BV. Since spring 2013 the parties worked together to develop a commercially viable ceramic printing process for functional ceramic components. Based on the rapid technical progress and the solid results, it was stated that founding a new company was the obvious next step.

ADMATEC Europe BV controls the complete printing process in-house, a process named ADMAFLEX. The ingredients used in the printing material were also developed in-house and allow for full control of the process. Similarly, the development of the printing equipment was undertaken within the group. During the past months the process has been qualified resulting in two validated materials, one for alumina and one for zirconia. The technology's roots are based on a photosensitive resin that is homogeneously mixed with ceramic powder. The material is then, layer-by-layer, activated through a light source. Following debinding and sintering the part reaches its final properties.

In the coming months ADMATEC will focus on realising a diverse range of commercial orders. A wide range of customers have discovered, through Formatec Ceramics, some unique uses for this new technology. Customers are not only based in the high-tech Brainport region near Eindhoven, but also internationally. It was stated that innovations will continue and will most likely focus on increasing product sizes whilst retaining a high level of precision. The ADMAFLEX process will also be tested for printing metals.

Michiel de Bruijcker, Managing Director of ADMATEC, told *PIM International*, "The technical developments have been very rapid over the past year. These results position ADMATEC on a unique spot in the rapidly growing market for printing functional components. With a great amount of trust we are looking towards the future, further enhancing the technology and further market developments."

www.admatec.nl ■

Thin-film heating technology cuts energy consumption during moulding

The Fraunhofer Institute for Mechanics of Materials IWM, Freiburg, Germany, has developed thin-film heating technology which claims to reduce energy consumption in injection moulding by as much as 90% compared with conventional practices. This is achieved by heating only the part of the tooling that comes into contact with the plastic melt, or in the case of PIM, the plasticised feedstock.

Variothermic methods are typically used to heat the entire tool during the injection moulding process. In the case of plastic parts with high end surfaces, the entire forming tool is heated to around 110°C. Cooling the tool sufficiently (to around 20-30°C) so that the moulded part can be removed without damage adds time to the cycle. By minimising the amount of tooling surface that must be heated and cooled during each cycle, significant energy savings can be realised.

Fraunhofer researchers teamed up with Kunststoff Zentrum (Leipzig, Germany), which provides consulting services to injection moulding professionals, to develop a process that would reduce energy consumption by 90% while improving moulded part quality. "The thin film heater, in combination with a thin film thermocouple, is able to very accurately regulate the interfacial temperature, [thus, increasing] the reproduction quality of small structures on the mould surface," said Fraunhofer's Alexander Fromm. "It can also lead to a decrease in cycle time."

The process developed involves only heating the thin film surface of the tool that actually comes into contact with the plastic melt. The thin film coating on the wall of the forming tool is produced using a vacuum-based coating technique known as sputtering. The sputtered material is a specially designed conductive hard material which is deposited in layers only a few micrometers thick. This extremely thin coating is used to heat the forming tool surface to the desired temperature.

Electrical insulation is provided by a ceramic layer that shields the conductive heating layer from the steel tool underneath. Fromm stated that the challenge of sputtering lies not only in producing a perfect insulating layer so as to avoid any short circuits, but also in integrating a sensor into the thin-film heating layer to measure the temperature of the tool wall and used to regulate the manufacturing process. To achieve this, the IWM researchers set about integrating incredibly fine thermocouples, made from nickel or nickel-chrome alloy and each just a few hundred nanometers thick, into the insulation layer. Due to their extremely low mass, thermocouples react incredibly fast to the temperature changes and make it possible to directly measure the temperature of the tool wall.

www.iwm.fraunhofer.de

Metal plate with internal honeycomb structure made by Metal Injection Moulding

A new Metal Injection Moulding (MIM) process for producing double-sided metal plates having an internal honeycomb structure has been developed at the Korea Institute of Industrial Technology (KITECH) in Yeonsu-gu, Incheon. Kwangho Shin and his colleagues reported on their research on the new MIM process in the open access journal *Materials* (2013, No. 6, 5878-5892) and stated that the rectangular metal plates with the internal complex structure are expected to be used under severe compressive load conditions with applications including insulating panels, pressure vessel chassis and floor panels.

In the process developed at KITECH one mould was used to produce the sacrificial polymeric insert which

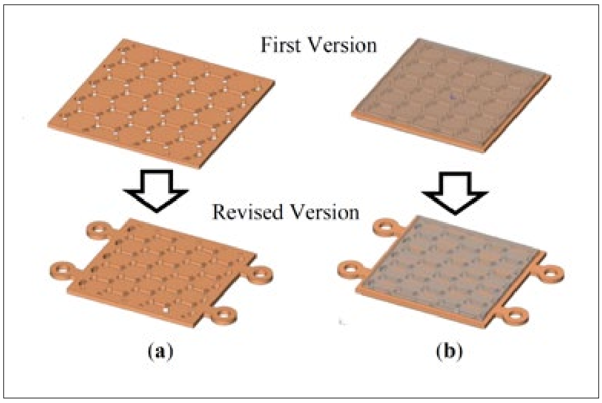


Fig. 1 Modification for the metal plate with an internal structure (honeycomb): (a) sacrificial polymer insert; and (b) sacrificial insert with metal plate

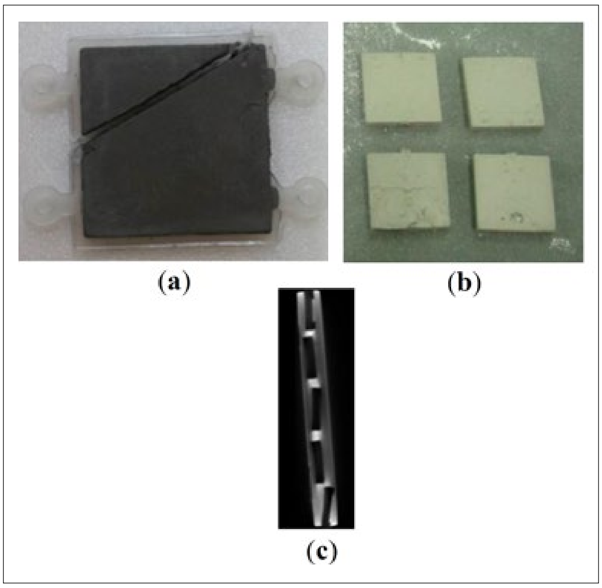


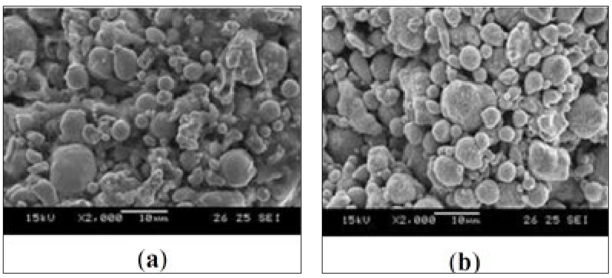
Fig. 2 Green parts and sintered parts: (a) green part; (b) sintered part; and (c) section view of sintered part

was then placed into the mould for injection moulding the double sided metal plate from 17-4PH stainless steel feedstock. The sacrificial insert is removed during debinding and sintering to leave the honeycomb structure between the metal plates. Each cell, or channel, in the sacrificial insert was interconnected so as to be removed without air being trapped during debinding and sintering. These interconnections would act as channels for fluid applications. Because of the potential deformation of the internal polymer insert during injection of the MIM feedstock, the researchers undertook CAE analysis to predict the complex flow behaviour of feedstocks for the production of a stable part.

The authors stated that the initial design of the finished part was based on a 30 mm square plate having a thickness of 2.4 mm, including the polymer sacrificial insert of 0.8 mm designed as a honeycomb, with each metal side also being 0.8 mm thick. It was established that for successful processing by Metal Injection Moulding the size of the square plate should be reduced to 22 mm. Four holes were added to the insert to prevent movement within the cavity during injection moulding of the 17-4 PH feedstock, which had a powder loading of 59% in a wax based binder system containing 2% stearic acid (Fig. 1). Side gates were used to fill the metal feedstock for both outer plates. The position of the gate was decided and made between the shorter sides of the fixing points. The insert was found to have suitable rigidity to endure the MIM process.

Fig. 2 shows the green part and the sintered part, and Fig. 3 shows SEM images for the green part, brown part and sintered part. The shrinkage of the target part during debinding and sintering was about 16.3% in thickness and 15.5% in width direction. The final sintered thickness was approximately 2.0 mm. The researchers used an X-ray NDT technique to check the internal structure, and it was found that the internal honeycomb structures were well-fabricated.

www.kitech.re.kr



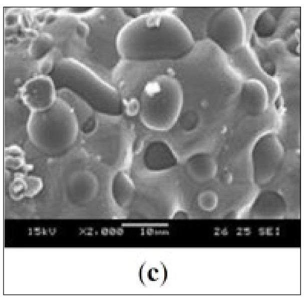


Fig. 3 SEM images for the part at each phase. (a) green part; (b) brown part; and (c) sintered part

Thermoplastic micropart moulding market grows

According to a recent market report published by Transparency Market Research, the global polymer and thermoplastic micro moulding market was valued at $308.0 million in 2012 and is expected to grow to $763 million by 2019, representing CAGR of 14.2% over the period. Growing demand for micro moulded products from various end use industries such as medical, automotive and telecommunications is expected to drive the market over the forecast period. The rapid development of micro fluidics and micro optics technologies, mainly in the US and Europe, is also expected to augment the market. In addition, the growing number of MIS (Minimally Invasive Surgeries), which extensively use micro moulding, is expected to boost the market for thermoplastic micro moulding over the forecast period.

Medical and healthcare emerged as the leading market for micro moulding and accounted for 35% of the total thermoplastic micro moulded products in 2012. This is expected to be the fastest growing market for micro moulded thermoplastics over the forecast period. MicroPIM could benefit from the growth and growing awareness of micro moulding in the plastics sector.

www.transparencymarketresearch.com

Powder Injection Moulding in the dental sector: From orthodontic components to implants

The dental sector has for many decades been an important market for the PIM industry, driven initially by the production of small orthodontic brackets. These brackets, often weighing as little as 0.02 g, today account for around 16% of global MIM sales by value. As Prof Randall German, San Diego State University, USA, explains, significant opportunities remain not only in the orthodontics brackets business, but also in the development of metal and ceramic dental implants and dental hand tools.

The dental field always seems to be an add-on in industry reports on Metal Injection Moulding (MIM). It is hardly included in a 2013 report from the Metal Injection Molding Association (MIMA), an association of North America's Metal Powder Industries Federation (MPIF), and likewise is poorly documented in the studies from other tracking organisations. Part of the issue is that most dental MIM is performed by captive operations that choose to remain invisible. Dentistry is a large field with global billing estimated at $120 billion per year; about half of that is in the USA based on approximately 195,000 dentists, some of which are focused on research or teaching. Japan has about half that number of dentists and Europe slightly less than twice that number.

One of the big winners for MIM is in orthodontia, in the form of orthodontic brackets for straightening teeth [1]. This article gives emphasis to orthodontic brackets, but it also ponders what is next for MIM in dentistry to identify some growth opportunities.

Conceptual framework for dental MIM applications

Dental orthodontic brackets, commonly known as braces, remain an enduring application for MIM. Early demonstrations arose as MIM devices moved into the marketplace guided by Johnson & Johnson, American Orthodontics, Rocky Mountain Orthodontics, and Minnesota Mining and Manufacturing or 3M. Traction arose because MIM offered a lower cost yet higher quality in contrast to investment casting. Nearly thirty years later, the actors have changed, but MIM dental brackets remain in widespread production.

As shown in Fig. 1, orthodontic brackets are small devices (the

Fig. 1 Top and bottom pictures of a typical MIM dental bracket, each weighs about 0.04 g

Metric	Value
US population	317 million
US dentist population	195,000
US orthodontist population	9,500
US percent of global orthodontia sales	4 %
Annual growth rate for orthodontia billings	4%
Average annual billing per US orthodontist	$950,000
Number of 12 year olds per year	4 million
Percentage of youth treated by orthodontist	42%
Percentage of orthodontic patients starting as adults	15%
Average number of new starts per orthodontist per year	220
Typical treatment time	18 months
Typical cost of orthodontic treatment	$4,320
Typical chair time per treatment	13 h
Typical orthodontist time spent with patient	5 h
Typical orthodontist billing per hour	$800
MIM orthodontic bracket market in the US	$200 million
Number of orthodontic brackets per year in the US	34 million
Powder used in the US for orthodontic brackets per year	1.5 tonne

Table 1 Background statistics on orthodontics in the USA as relevant to the MIM business

Fig. 2 A picture of the final dental ultrasonic scaler tip after moulding, sintering, heat treatment, and titanium nitride coating for wear resistance (Courtesy CetaTech)

Fig. 3 Endodontic abrasive tip with up to 1500 bumps of 20 to 50 μm formed by MIM using sintered and heat treated stainless steel with titanium nitride coating (Courtesy CetaTech)

pictured brackets have 0.04 g mass each) consisting of various combinations of bumps, slots, grooves, holes, posts, logos, and textures, commonly with 100 or more geometric specifications. Stainless steel dental orthodontic brackets perfectly match the MIM design window; small mass at 0.02 to 0.04 g, small sizes in the 2 to 5 mm range, over 75 features, production quantity over 200,000 per year, and in some cases reaching millions per year. These brackets therefore have a high production value roughly equal to $50,000 per kg, so the powder cost has lower impact on the manufacturing cost. There is ample room for further products in dental MIM.

A statistical view of US dentistry

We start by reviewing some statistical profiles on dentistry in the USA. The USA population is approaching 320 million. This is just 5% of the more than 7 billion global population. But the USA is disproportionate in dental care expenditures. About 60 to 70% of the population receives regular dental care provided by a pool of about 195,000 dentists.

As evident in Table 1, orthodontics is a significant MIM evaluation, at least in economic value if not in terms of powder tonnage. Curiously, dental orthodontic brackets account for about 16% of the MIM global product sales based on far less than 0.1% of the powder sales, and most of this is in the USA.

In the USA, about 20 firms constitute the MIM production base for orthodontic brackets. Depending on materials, design, and geography the brackets sell for $2 to $70 each, with an average price near $5 each. The lower price of $2 each is for large volume buyers, such as distributors in international markets for older designs. Because brackets are produced both in-house by captive facilities and by custom fabricators, often the MIM trade price is as low as $0.70, so the $2 sale price is realistic for large volume distributor purchasers [2]. In various reports, the direct manufacturing cost of a typical bracket is estimated at $0.35 each, and with overhead the MIM industry trade price reaches about twice this or $0.70 each. Of course, when sold to the patient the price is significantly higher and some products are reportedly priced at $35 or more.

The production of dental brackets

Dental bracket production was an early success for MIM and drove much interest in the production of fine alloy powders versus mixtures of iron-nickel-chromium based on carbonyl iron. Experiments using mixed powders to form stainless steel by diffusional alloying were successful, but corrosion resistance required long sintering hold times that were not productive. Thus, early adoption of spherical stainless steel prealloyed powder was a clear choice for this field.

In visiting some of the early adopters during 1992, the typical situation was four to twelve moulders, four to twelve cavities, and one or two batch sintering furnaces. Most of the dental firms purchased a license from the original half dozen technology leaders, but generally elected to purchase feedstock since their needs were too modest to justify a mixing facility.

Unlike other parts of the MIM industry, the dental firms did not complain about the cost of powder. They were converting gas atomised stainless steel at $60/kg into $50,000 per kg product. Or as one industry insider said at an early PIM conference in Boulder, Colorado, "... they are selling a bucket of product for a million dollars."

The growth of other PIM dental applications

Dental orthodontic brackets were an early adopter of MIM, but curiously once MIM penetrated that application the spread to other dental products was slow. The current range of PIM products in dentistry includes the orthodontic bracket, ultrasonic scaler tip, endodontic abrasive tips, zirconia implant posts, titanium implant posts, and various hand tools [1-9]. These items have been pictured frequently and are the subject of several reports since they significantly challenged the PIM technology. Some examples include the following:

- Ultrasonic cleaner tips fabricated from 17-4 PH (AISI 630) stainless steel. After sintering they are coated with titanium nitride, mass about 1 g, pictured in Fig. 2 [3,4].
- Microminiature endodontic abrasives fabricated in a similar manner, with hundreds and even thousands of 20 to 50 µm abrasion teeth moulded on the tips as pictured in Fig. 3 [5].
- Zirconia tooth implant posts formed using stabilised zirconia with diameters in the 1 to 2 mm range and surface texture for attachment to bone as pictured in Fig. 4 [6].
- Titanium implant tooth anchors generated with two material injection moulding to form tissue attachment core with strength and elastic modulus matched to bone, as pictured in Fig. 5 [7,8].
- Distiliser, a longer span combination of pad, moveable ball, curved rod, and hook fabricated from zirconia, stainless steel, or nitinol. The distiliser helps to span large distances for proper jaw alignment.

The largest segments rely on 17-4 PH (AISI 630) and 316L stainless steels, with some minor role

Alloy	Yield Strength MPa	Tensile Strength MPa	Fracture Elongation %
316L	170	515	50
17-4 PH	1050	1185	6
CP Ti	660	760	13
Ti-6Al-6Nb	420	500	20

Table 2 Typical mechanical properties for MIM dental alloys

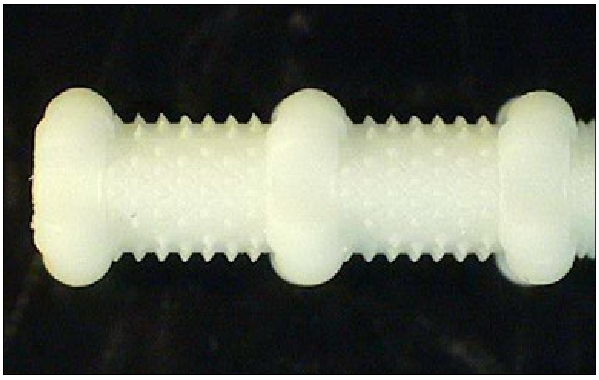


Fig. 4 Dental implant from zirconia focused on the moulded bumps to ensure affixation to bone (Courtesy CetaTech)

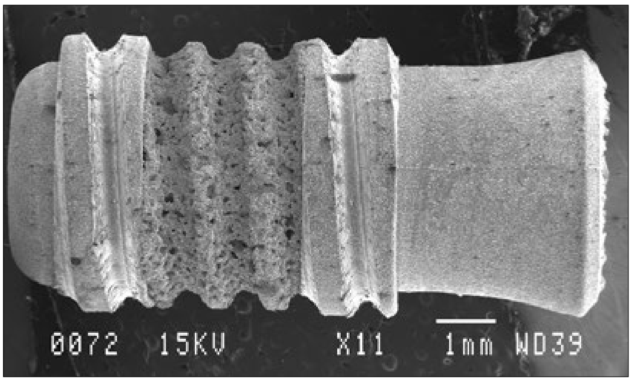


Fig. 5 Titanium dental implant with a porous central region for tissue ingrowth (Courtesy National Research Council of Canada)

Fig. 6 This picture contrasts the translucent dental bracket sintered from alumina with the traditional stainless steel sintered brackets, showing the distinct aesthetic difference

Fig. 7 Small MIM gears used in an electric toothbrush (Courtesy FloMet)

from Co-Cr-Mo or other nickel free compositions, and small levels of titanium and titanium alloys, including NiTi (Nitinol). For these applications the typical MIM mechanical properties are cited in Table 2 [1,2,10].

Emerging opportunities in dental MIM

Orthodontic brackets

Dental orthodontic brackets were an early success for MIM, but the custom industry was small forcing many of the user firms to integrate production into their operations. In the most recent survey of the MIM industry, a total of 39 firms claimed to be in production for dental products. Of this, over half were captive firms. Besides the USA, they were located in Canada, China, Germany, India, Italy, Mexico, and New Zealand. In at least one case, the MIM facilities were set up by dentists seeking to fabricate their own bracket designs. The only significant challenge remaining seems to be the fabrication of translucent or transparent brackets, such as those shown in Fig. 6. Most of the trials are with alumina or magnesia-alumina spinel sintered in hydrogen to near optical transparency to form invisible brackets with good strength and compatibility [11].

Dental implants

Dental implants continue to be the subject of much research, largely because the current machined posts are priced in the \$350 to \$450 range. Two target materials are widely discussed – titanium and zirconia. The titanium might be treated with hydroxyapatite and have a porous section with 200 μm pores for bone ingrowth formed using space holder ideas. On the other hand, the zirconia is affixed to the bone using threads, posts, bumps, or tapers.

In the USA there are about 500,000 new implants installed per year. Since the implant is not mating with other components, the tolerances are generally at ± 0.5%, but the size is from 8 to 15 mm length and 1 to 5 mm diameter.

Dental hand tools

Hand tools and even consumer devices are a continuing opportunity for MIM. Early electric toothbrushes used small MIM gears to generate the rotating motion and reportedly at peak production reached 85,000 gears per day. Fig. 7 is a picture of this early product. As dental health education progresses, there are some opportunities in other consumer hand tools, for example flossing devices. Although most of the device will be plastic, the critical stress bearing floss support tips will be from metal.

Dental drills are another potential for MIM. They are used in the dental office and in the dental laboratory where restorations are created. The 10 mm diameter by 16 mm long turbine wheels (about 1.5 mm wall thickness) are driven by air turbines and reach high speeds with low mass, so would require aluminium. Similar to the automotive turbocharger, the geometry is ideal for MIM. Further the ability to hollow out the geometry is a competitive advantage for MIM since it will lower mass to make the turbine more responsive and minimise the potential for soft tissue damage. The opportunity is in the million units per year range, ideal for MIM.

In other opportunities for MIM, note the distalizer consisting of ball, rod, pad, and hook has entered production and is a high value addition to the MIM portfolio. Additionally, small MIM components are part of new sleep apnea positioner's designs. An example of a titanium MIM component in a sleep apnea device is shown in Fig. 8.

Fig. 8 Part of a sleep apnea device made by MIM from Ti Grade 4 feedstock (Courtesy OBE GmbH & Co KG)

Conclusions

MIM for dental applications was one of the first major successes, but generally has remained hidden. Several significant opportunities are under discussion, but still stainless steel orthodontic brackets remain a mainstay in the USA. The estimated $200 million in MIM dental brackets annual sales in the USA is equal in value (but not tonnage) to the often discussed applications in firearm, medical, industrial, and other fields combined.

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Ortho Organizers: A global leader in the development and manufacture of MIM orthodontic products

Ortho Organizers, Inc., based in Carlsbad, California, USA, provides a wide range of orthodontic products to the worldwide dental market. A part of the orthodontic portfolio of Henry Schein, Inc., Ortho Organizers' in-house MIM operation has since 1996 enabled the company to remain at the forefront of the manufacture of innovative and complex orthodontic products. Prof Randall German, San Diego State University, recently toured its MIM facility with a group of PIM Design students and reports on the development and use of MIM at the company.

As part of the PIM Design course taught as a senior mechanical engineering elective at San Diego State University, a group of 16 students visited Ortho Organizers in Carlsbad, California in December 2013. The location is on a hilltop near the Pacific Ocean (Fig. 1), with palm trees making up a lovely industrial setting, as evident in the photograph of some students outside the facility (Fig. 2).

Ortho Organizers was founded in 1976 by Lindsay Brehm, a former employee of "A Company" (Johnson and Johnson). Lindsay is the son of Waldemar Brehm, a famous orthodontist and teacher of orthodontics. The first products were organisation racks and storage cases for orthodontists. They acquired Micro Metal Products in 1981 and San Marcos Medical Plastics in 2001. The company subsequently grew and changed hands, and in 2006 moved to its current 65,000 square foot facility in Carlsbad (north San Diego County). In 2008 it was purchased by Henry Schein, Inc., a Fortune 500 Company. Ortho Organizers (commonly known as O2) became part of the Dental Specialties Group where it operates under ISO 13485 and FDA CFR 820 standards.

Fig. 1 The Ortho Organizers headquarters in Carlsbad, California (Courtesy Ortho Organizers)

Ortho Organizers today

In the past few years Ortho Organizers has grown rapidly, both by the absorption of other companies and by the leverage from the Henry Schein sales force. The current management team has deep experience in the dental-medical arena, starting with the President Russ Bonafede (since 2009) who has 24 years in orthodontics, implant dentistry, and related surgical fields. Ted Dreifuss is

Vice President of Sales and Marketing with 30 years related marketing and management experience. Mark Payne is Director of Engineering with 21 years of experience in orthodontics. He is responsible for new products and new production processes.

Celine Cendras is Director of Marketing with a focus on launching new products and managing the distribution channels for Ortho Organizers and the recently acquired ClassOne and Masel brands. She has 18 years related experience. Robin Marks is Director of International Sales that now account for over 50% of Ortho Organizers sales via 200 international distributors.

As with all firms working in the dental-medical field, Ortho Organizers has a keen focus on regulatory affairs and Foster Boop serves as Director of Regulatory Affairs and Quality Assurance with more than 20 years' experience. Sue Fix is Director of Operations and has nine years prior experience in dental device manufacturing and supply chain management, with another ten years managing procurement and purchasing. Chief Financial Officer for the Dental Specialties Group is Alison Weber.

Chhattar Kucheria is Senior Research Scientist and Edward Soblewski is Production Manager, and these two individuals kindly guided the visit by the San Diego State University PIM Design class. Not included in the visit is a separate facility that forms elastomeric products, archwires, and waxes. The company also has other products such as gold caps, ceramic brackets, and sapphire brackets.

One impressive aspect of the company is the commitment to education. They host up to 250 short courses per year, taught by industry leaders as a service to the profession. In addition, in-house training is important in developing the workforce. Recent courses have given emphasis on sleep apnea treatment using orthodontics.

Ortho Organizers is well regarded in the industry and has been noted frequently for leadership via awards.

Fig. 2 Students from the SDSU PIM Design course outside the Ortho Organizers facility in Carlsbad, California

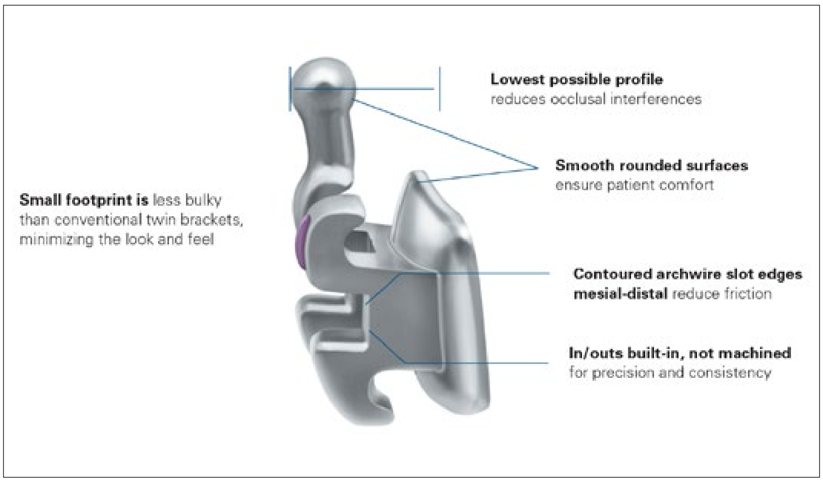

Fig. 3 The Maestro Low-Profile Bracket System from Ortho Organizers features rounded contours for patient comfort and highly complex structures that machined brackets cannot duplicate (Courtesy Ortho Organizers)

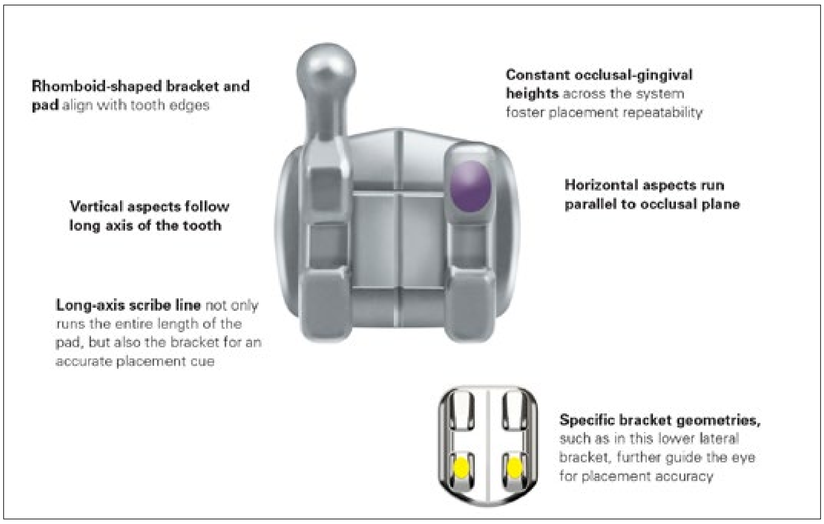

Fig. 4 Further details of the design and function of the Maestro Low-Profile Bracket System from Ortho Organizers including features that enable the positioning of brackets in their ideal location (Courtesy Ortho Organizers)

Fig. 5. Moulding area in the Ortho Organizers MIM facility in Carlsbad, California (Courtesy Ortho Organizers)

Most recently in October 2013 they were featured with the cover article in *Orthotown*, and in the past two years their brochures and packaging designs have been given notice by industry trade groups.

The application of MIM at Ortho Organizers

Ortho Organizers initiated MIM production with a license from Injectamax (formerly located in nearby Escondido, California, now part of the ARC Wireless Group). Essentially they still practice that technology, but with many adaptions, such as relying on premixed feedstock and adjusting the solvent debinding technology. Two alloys constitute the primary production, 17-4 PH (AISI 630) stainless steel and a trademarked Nickel-Lite® cobalt-chromium alloy.

A typical MIM bracket geometry is shown in Figs. 3-4. Surprisingly, there are more than 1500 distinct SKU (Stock Keeping Unit) numbers in the MIM inventory. Consequently, Ortho Organizers love the flexibility that comes with a captive MIM facility. In a captive facility the production runs are shorter, and products shifts are frequent, so inventory is replenished with short lead times.

With this trend, of course, a natural forward projection is an eventual shift to Additive Manufacturing to fabricate one bracket at a time, just in time, but that is off in the future in what Ortho Organizers calls digital dentistry. Additive Manufacturing for orthodontics exists now in plastics, so projecting adoption of the process for metals is realistic. For now, Ortho Organizers sees no disadvantages to a captive MIM operation, especially in light off the fast response possible.

The only mentioned negative aspect of MIM is the cost of moulds. Considering that each tool requires a few weeks tool room effort, and there are a thousand designs, a comment was made that replacing the tooling would now require considerable effort.

From a technological view, the company has 17 issued patents and licenses another six patents while operating with well over 50 trademarks, including Nickel-Lite® for the cobalt-chromium MIM brackets. New designs arise from internal efforts or via inventors, usually well regarded orthodontists. A few years ago they purchased another MIM company, ClassOne Orthodontics, and now produce and sell the Carriere self-ligating brackets and distalizers as part of their premier product line.

MIM tooling expertise

In terms of production, the company forms its own tooling, with EDM electrodes used to form inserts in-house. Considerable engineering expertise exists in terms of gate size, wall size, draft angles, runner design,

Fig. 6 An Andrews2™ bracket manufactured by Ortho Organizers. The inner surface features an 80-Gauge micro-etched bondable mesh pad for improved adhesion (Courtesy Ortho Organizers)

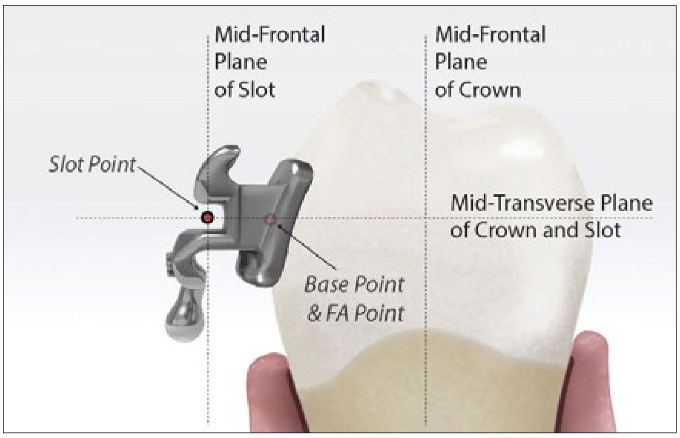

Fig. 7 An illustration showing the siting of a MIM orthodontic bracket, in this instance a lateral perspective of an Andrews2™ bracket manufactured by Ortho Organizers (Courtesy Ortho Organizers)

and tool surface finish. Tooling is single cavity for the larger distalizer parts, but reaches two, four or even eight cavities for the orthodontic brackets. One consequence is that moulding is automated with robots, requiring only a couple employees for the nearly dozen moulding machines. Fig. 5 shows a picture of the moulding area consisting of Boy moulders with robots. Tools are repaired in house.

Mould filling simulations are provided in some cases by outside vendors, but this is not a typical activity for Ortho Organizers. On occasion there is some difficulty with balancing mould filling, but otherwise most of the tool design builds on years of experience. Tools maintenance varies with the geometry, but tool life reportedly reaches to about 300,000 shots prior to return to the tool room.

Debinding and sintering

The moulded components are staged on trays and spaced to avoid contact. Debinding is rather rapid since the components are small. The first step is a solvent debind followed by backbone removal during heating to the sintering temperature. Three furnaces are available in the furnace room, but most production is performed in an Elnik batch furnace in hydrogen. The heating cycle has a sequence of holds to ensure proper breakdown of the backbone polymer prior to soaking at the peak temperature. One furnace run per day is typical.

An additional bell jar hydrogen furnace can be employed for sintering, but most of its use is for brazing. A third vacuum furnace is employed also for brazing. Some of the components are laser welded instead of brazed. After sintering the components move to finishing and inspection, involving bench labour and working through magnification glasses.

Production volumes and markets

Production ranges from 1.4 to 2 million brackets per month. Likewise in recent years the employment has ranged from 200 to 300 people. About 5% of the employees are technical, mostly young engineers involved in automation, process development, and tooling developments.

Fig. 8 The Carriere Distalizer™ appliance manufactured by Ortho Organizers. This larger MIM orthodontic product, consisting of ball, rod, pad, and hook, has entered production and is a high value addition to the MIM portfolio. Lengths range from 16 to 27 mm (Courtesy Ortho Organizers)

The sale of orthodontic brackets produced by MIM is a competitive business. In the USA there are 9,500 orthodontists and the specialisation is distinguished by further years of training after dental school. In addition, about 7,000 general practitioner dentists do some orthodontia cases. Thus, Ortho Organizers is looking at over 16,000 potential customers in the USA. Curiously, orthodontic treatments are generally outside dental insurance coverage, so the company has low sensitivity to changes occurring in the insurance industry - orthodontia is an elective treatment.

Statistics shared during the visit are that a typical orthodontist in the USA has 200 to 300 new cases per year, of which about 15% are adults. The roughly 7,000 general practitioners that practice limited orthodontia average about 20 new starts per year. Each new case is a sales opportunity for MIM. Although plastic appliances are an alternative to the MIM brackets, generally the plastics with lower elastic modulus fail to produce the desired permanent repositioning of the teeth in the typical 24 month treatment time used in orthodontics.

The company reports about 20 competitors. Ortho Organizers is positioned in the top third of the industry based on sales and is growing rapidly. Their growth is a combination of the leverage from Henry Schein, acquisitions, and organic growth; reportedly giving a 200% increase in the past three years. In the USA they promote via a combination of 20 traveling field sales agents that directly call on dentists, ten inside sales agents, ten customer service representatives, and ten selling teams acting for the Dental Specialties Group of parent Henry Schein. They also export to more than 90 countries and as mentioned export accounts for over 50% of sales.

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An introduction to the Hot Isostatic Pressing (HIP) of MIM components

Whilst Metal Injection Moulding can achieve sintered part densities that are significantly higher than conventional Powder Metallurgy products, there are a number of advantages to achieving full density in a MIM part. As Dr Stephen J Mashl explains, using HIP to achieve full density MIM products can improve mechanical properties, assure a higher quality of surface finish for polished and electroplated applications and offer greater peace of mind for medical device manufacturers. There are, however, a number of considerations to bear in mind when planning the HIPing of MIM parts.

During the 1970s and 1980s, when the Metal Injection Moulding (MIM) process was seeing tremendous growth, some parts makers would state that MIM components could sinter to full density, thus implying that the finished MIM product was pore-free. Given the very high as-sintered density levels of typical MIM materials and the significantly lower density values of conventional pressed and sintered ferrous PM parts, this optimistic view can be understood.

In truth however, most MIM components have between 1 and 3% residual porosity after sintering. In most applications, this small amount of residual porosity is acceptable and a MIM part performs very well in the as-sintered condition. In other instances, this small amount of residual porosity is a problem and its presence limits specific properties or performance to a level less than optimal.

Reasons to HIP MIM products

Hot Isostatic Pressing, or HIP, is a common post-sintering process by which residual porosity in MIM parts can be eliminated. This article examines the HIP of MIM materials, presents the reasons for considering the addition of HIP to a process scheme and reviews the potential problems and benefits that might be encountered in the combined process.

To improve mechanical properties

To an engineer, the improvement of mechanical properties is always desirable. The effect of residual porosity on the mechanical properties of PM materials is well understood with studies dating back more than 60 years. One landmark study by Alexander Squire in 1947 examined the effect of varying amounts of residual porosity in iron PM materials [1]. Fig. 1 shows Squire's data illustrating the effect of residual porosity upon tensile strength, ductility measured as percent elongation, and impact resistance. In these plots residual porosity is quantified as relative density where:

$$\textit{Relative Density} = 1 - \textit{Pore Fraction}$$

Thus with 2 volume percent residual porosity or a pore fraction of 0.02, the relative density would be 0.98. A pore-free material has a relative density of 1.0.

In the three graphs that comprise Fig. 1, the slope of the line representing the property data as it approaches the pore-free state, where the relative density equals 1.0, indicates the relative sensitivity of that property to small amounts of residual porosity. The steeper the slope of the line as it approaches a relative density of 1.0, the greater the effect of porosity on the property examined. In Fig. 1, Squire's graphs have been modified to illustrate the impact of

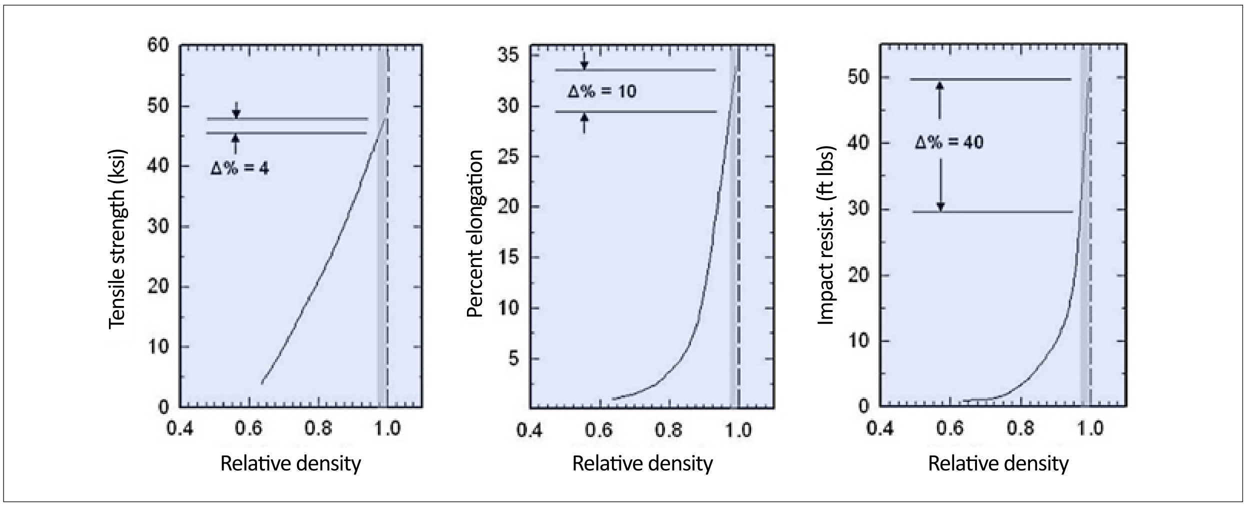

Fig. 1 Three graphs showing the effect of residual porosity, reported as relative density, on mechanical properties. A relative density of 1.0 represents the pore-free state and is indicated by the vertical broken line. The gray, shaded region immediately to the left represents the range from 98% dense to the fully dense state. The difference in properties at these two density levels varies from small (4% change in ultimate tensile strength), to moderate (a 10% change in tensile ductility), to large (a 40% variation in fracture toughness). The data is from Alexander Squire's 1947 paper "Density Relationships in Iron-Powder Compacts" [1].

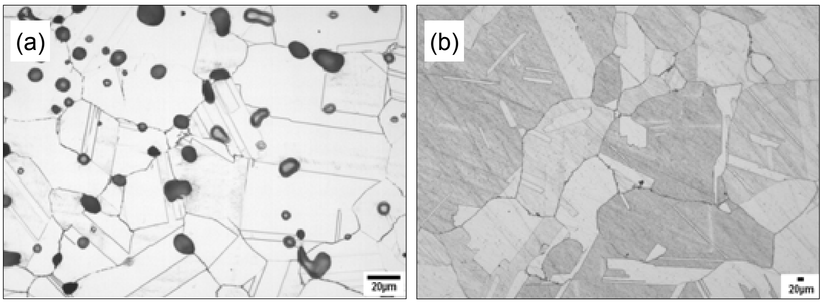

Fig. 2 Optical micrographs showing, (a), the microstructure of a cobalt-chrome medical implant alloy (F2886) sintered and solution annealed to a density of 7.80 g/cm³ (95% of pore-free density) and (b), The same material after sinter, HIP, and solution anneal to a density of 8.25 g/cm³. The complete elimination of residual porosity in the sintered and solution annealed alloy following HIP is apparent. Comparison of the scale markers indicates a nominal five-fold increase in grain size with HIP. After Sago, et al. [2]

2% porosity on strength, ductility and impact resistance and also show the relative improvement that one might expect were that porosity removed with HIP. A gray band covers the density regime from 0.98 to 1.0, i.e. a 2% porosity range. It is very common for MIM ferrous alloys to contain 2% porosity and this comparison illustrates the typical change in density that can be achieved when as-sintered MIM parts are HIPed to full density (relative density = 1.0). By examining the point at which the line representing Squire's data contacts the left-most edge of the gray band and comparing that to the point where the line contacts the broken line representing a relative density of 1.0, the relative effect of HIP can be evaluated on these three specific mechanical properties. For ultimate tensile strength, the improvement with HIP can be estimated at 4%. A 10% increase in ductility is predicted and a large, 40% improvement in impact resistance is indicated. High cycle fatigue life is another property that, like impact toughness, is very sensitive to small amounts of residual porosity.

In practice, when a MIM part is HIPed, the improvement in tensile and yield strength often falls within the noise of repeated measurements and thus the effect is seen as negligible or non-existent. The improvement in ductility can be more significant and, when a parts maker faces a challenging ductility specification, the use of HIP may bump tensile ductility to a level sufficient to ensure customer satisfaction. The truly significant improvements in mechanical properties with HIP are seen in the areas of impact resistance/fracture toughness and high cycle fatigue. Elimination of the last few percent of porosity in a MIM component can lead to dramatic improvements in these areas.

The images shown in Fig. 2 and the data in Tables I and II illustrate the behaviour predicted by the Squire data. Fig. 2a shows the unetched microstructure of a MIM cobalt-chrome medical implant product. The as-sintered material contains a fairly high 5 volume percent porosity. HIPing this material yields the result shown in Fig. 5b, a pore-free material, albeit one in which the HIP process appears to have induced a 5 fold increase in grain size.

The tensile properties that accompany the images in Fig. 2 are presented in Table I. Comparing as-sintered to HIPed tensile properties one observes no change

	MIM F2886 H_2/N_2	MIM F2886 H_2/N_2
	Sintered, Solution Annealed	Sintered, HIPed, Solution Annealed
UTS (MPa)	897 min	897 min
YS (MPa)	552 min	552 min
Elongation (%)	15	20
Density (g/cm³)	7.81	8.20
Density (% Pore-Free)	95	100
Hardness (HRC)	16	15

Table I Tensile properties of MIM Co-28 w/o Cr-6 w/o Mo. After Sago, et al. [2]

	1260°C, 1 h. sinter	1285°C, 1h. sinter
	Impact energy, (J)	Impact energy, (J)
As-sintered	5.4	6.8
1121°C HIP	24.4	21.7
1163°C HIP	17.6	20.3

Table 2 Charpy V-notch impact test results for MIM 17-4PH stainless steel produced from blended powder. After LaGoy, Bulger [3]*

**The powder employed in making the samples represented here was a blend of carbonyl iron + master alloy (75% by volume) combined with water atomised, pre-alloyed powder (25% by volume). HIP was performed with a 4 hour hold at 103 MPa , at the temperature indicated.*

in ultimate tensile or yield strength but a 33% increase in ductility.

Impact toughness data presented by LaGoy and Bulger [3] is shown in Table 2. Again matching the behaviour predicted by Squire, the gains in impact toughness are large. In this segment of their work, where two HIP temperatures and two sintering temperatures were used, it is interesting to note that the highest impact toughness value is reported for the lowest sinter temperature and lowest HIP temperature. While no error data was provided to allow evaluation of the significance of the reported differences, it is possible that grain growth, occurring during sinter, HIP or both processes resulted in the lower sinter and HIP temperatures being most effective.

It should be noted that residual pores are only one of several types of microstructural defects that are encountered in MIM products. Inclusions, in the form of oxides, sulfides, silicates, etc., have a detrimental effect on mechanical properties similar to porosity. A high population of these non-pore defects can limit or eliminate any improvement that might be achieved with HIP. It is also important to realise that changes taking place during sinter, HIP, or heat treat can offset each other. For example, it is possible to HIP a component and remove all porosity, potentially enhancing ductility, only to have grain growth and its detrimental effect on ductility counter the positive effect of pore removal.

For appearance sake: highly polished surfaces

Improvement in mechanical properties is not the only reason to consider the use of HIP to remove residual porosity from MIM parts. Highly polished surfaces may be required for functional reasons such as in the polishing of the mould dies employed in plastic forming. As illustrated in Fig. 3, in these applications sub-surface porosity that becomes exposed during the polishing operation is considered a defect [4-6].

Polishing need not be done for functional reasons, a high gloss finish may be desired for aesthetic reasons alone. Metal Injection Moulding is increasingly used in the manufacture of jewellery and highly decorative items such as the eyeglass frame shown in Fig. 4. When a surface is polished to a mirror-like finish for appearance, fine porosity exposed during the polishing becomes readily visible to the naked eye. The presence of even a small amount of porosity can send a near-finished part to the scrap bin.

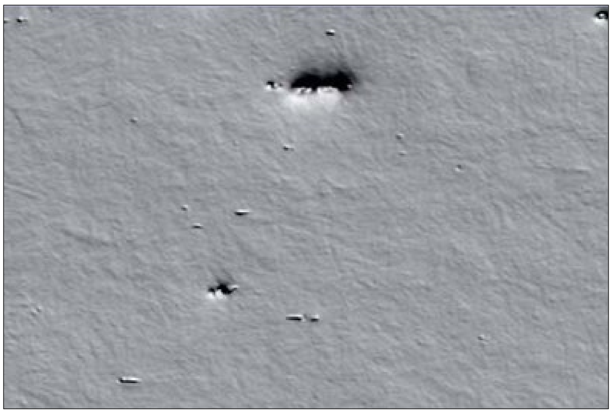

Fig. 3 Pores, such as those shown on the right, that become exposed during a polishing operation meant to deliver a mirror-like high gloss surface are considered defects. The use of HIP to eliminate near-surface porosity prior to polishing operations has been shown to dramatically improve yields (courtesy Fraunhofer IPT) [4]

Fig. 4 When MIM surfaces are highly polished for aesthetic reasons, such as the decorative eyeglass frame shown here, exposure of subsurface porosity during polishing can send a near-finished part to the scrap bin. After Williams [7] (Photo courtesy OBE GmbH & Co. KG)

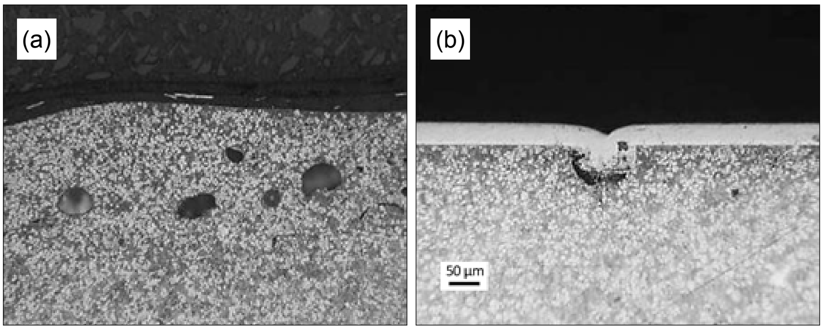

Fig. 5 Two micrographs showing near-surface porosity in a die casting (a) that, following polishing and plating (b) results in a highly visible imperfection in the plated surface. While these images are from sectioned die castings, identical behaviour can occur in MIM components. After Annetts [8]

Fig. 6 MIM produced medical components are often HIPed to eliminate sub-surface porosity that would become exposed in final machining operations. This now surface connected porosity is seen as a harbour for bacteria and a potential source of infection (image MPIF)

Electroplating and electropolishing might be undertaken for either functional or aesthetic reasons, depending upon the application. Regardless, this is another situation where near-surface porosity, such as that shown in Fig. 5a becomes exposed during surface preparation and results in a defect such as that shown on the cross section of a plated die casting in Fig. 5b. Again, highly polished highly reflective surfaces tend to make small surface defects more visible.

To prevent infection: medical applications

The medical industry is a sizable market for the MIM industry and the HIP of MIM components to be used in surgical applications appears to be growing with increasing production of parts such as the a high-compression jaw used in laparoscopic surgery shown in Fig. 6.

In medical applications, fine sub-surface porosity exposed during machining and finishing operations is regarded as a harbour for bacteria [9]. In today's litigious society, elimination of the risk of infection in the operating room can be a driving force large enough to justify the additional cost of HIP, even when the elimination of porosity is not required to meet mechanical property specifications.

Grain growth in MIM alloys – an obstacle to densification

If HIP is to be incorporated into a processing scheme, it may be necessary to review upstream procedures if optimum mechanical property improvement is to be achieved. The relationship between pore fraction and mechanical properties is well known and it is this relationship that drives the manufacturers of MIM components to pursue increasingly higher sintering temperatures in order to decrease pore fractions in the as-sintered part. While this approach may help in achieving higher density levels, it can be counterproductive if HIP will be used to attain pore-free density. Grain growth and its effect on diffusional densification are the issue.

The latter stages of densification

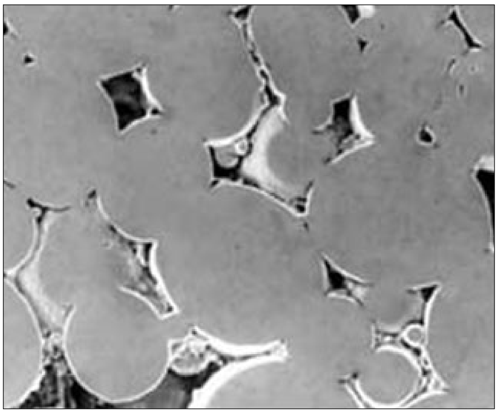

Fig. 7 An image of partially consolidated spherical powder. During the consolidation process the mated particle-particle surfaces become grain boundaries, regions of atomic misalignment that serve as a high-speed route for atoms and vacancies to move, thus facilitating further densification

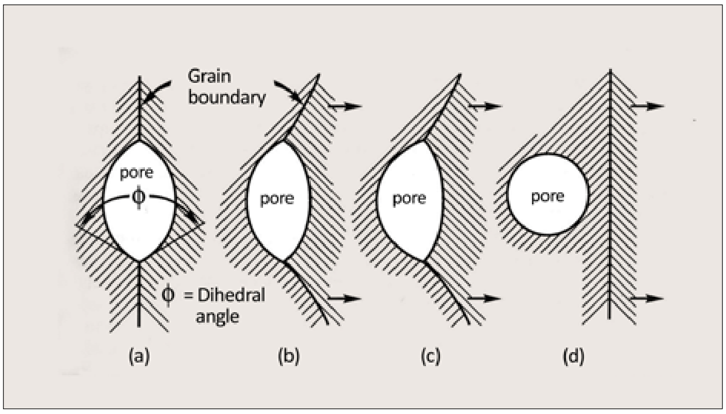

Fig. 8 A schematic of pore isolation resulting from grain growth. Image (a) shows the initial state with grains to the left and right of the pore and the grain boundary extending vertically away from the pore. Conditions are such that the grain to the left is moving to the right. Images (b) and (c) show the grain boundary moving rightward but being pinned in place by the now deformed pore. In (d), the grain boundary has separated from the pore and it will continue to move to the right, now unhindered by the pore. Densification of the pore will now slow considerably [10]

during HIP rely on diffusion, specifically the transport of atoms to the pore surface and the movement of atomic vacancies to the part surface. During the initial stages of HIP when powder particles are deformed plastically, the mated surfaces of collapsed particle surfaces fuse and become grain boundaries, connecting the pores to each other and to the part surface. This concept can be visualised in Fig. 7.

Along these newly formed grain boundaries, where the atomic lattice of one grain is mismatched with the lattice of an adjacent grain, the relative openness of the atomic structure serves as a superhighway for atom-vacancy movement, enhancing the rate at which a material will densify under HIP conditions.

MIM products are made using metal powders that are much finer than those used in other HIP PM operations. Grain growth is driven by nature's desire to decrease a systems total energy by decreasing the amount of high energy surface, in this case, grain boundary surface. Because MIM materials are made from powders that are much finer than other PM products, MIM materials have a higher driving force for grain growth. While this same driving force is what drives sintering and allows MIM components to achieve the very high as-sintered densities characteristic of the process, in this case it works against the parts maker.

'the move to incorporate HIP must be taken with consideration for all of the factors that affect densification during HIP and the resultant mechanical properties'

Fig. 8 shows, schematically, the process of a grain becoming isolated by a pore while the microstructure of the MIM 420 stainless steel component shown in Fig. 9 is "over-sintered" if HIP is to be considered. The material shown in Fig. 9 has experienced grain growth to the point where many pores are now isolated within grains. With such a structure, it is unlikely that a conventional stainless steel HIP cycle will fully eliminate the pores from this material.

The parts maker can take steps to avoid grain growth and promote full densification during HIP. By using the sinter temperature that is on the low end of the spectrum for a given alloy it is possible to reach the point where pores within the sintered material have just become isolated but have not separated from grain boundaries. Likewise in HIP, keeping temperature at the low end of the feasible range, maximising pressure, and minimising dwell time at temperature will decrease the driving force for grain growth and thus facilitate maximum densification.

Summary

There are many reasons why a company might consider using HIP to eliminate porosity from a MIM product. Improvement in mechanical properties, especially impact toughness and fatigue are one; visual appeal and medical safety are others. Regardless of the reasons for adding HIP to a MIM production operation, the move to incorporate HIP must be

The HIP process

The Hot Isostatic Pressing (HIP) process can be described as a high temperature pressure treatment. When processing MIM materials the goal is to remove porosity from the metal product. To accomplish this, the parts are heated to a point where the flow stress of the alloy is significantly decreased and diffusion becomes active. The applied, hydrostatic pressure then drives densification, initially via the plastic flow of the metal and in the later stage of the process by diffusional creep mechanisms. Industrial HIP units can typically achieve temperatures up to 1300°C and pressures of 210 MPa, while specialised units have reached 2200°C and 700 MPa. Typically an inert gas is used as a pressure transfer medium with argon being most common. A basic HIP system is shown schematically in Fig. 1.

As applied to the processing of sintered MIM components, the HIP process is simple in concept.

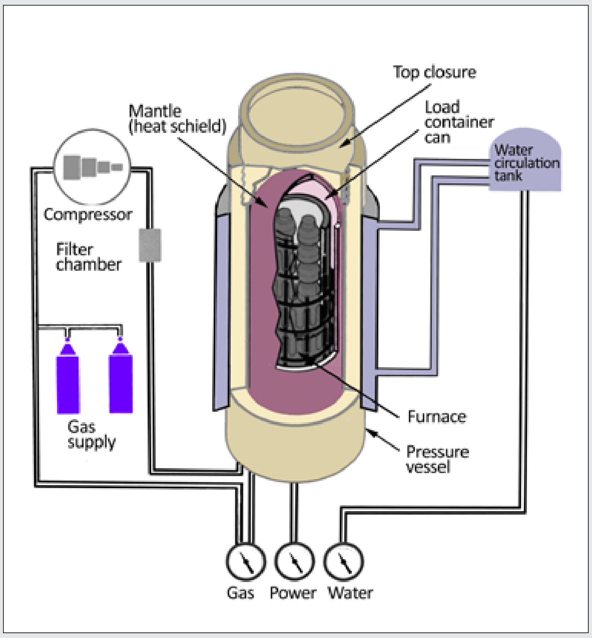

Fig. 1 A schematic of a hot isostatic pressing (HIP) system. The HIP unit, shown in the centre of the image can be described as a pressure vessel that contains a resistance heated furnace. The parts to be processed are placed within the furnace, the pressure vessel is closed and the furnace/pressure vessel is evacuated and then back-filled with an inert gas. The gas supply and compressors provide the high pressure gas which serves as the pressure transfer medium. Electrical power heats the furnace, and water is used to cool the pressure vessel. In a typical HIP cycle, temperature and pressure are ramped up to the target values simultaneously, held at their desired values, then the furnace is cooled and pressure is released. After Mashl, et al. [13]

Prior to HIP, the sintered MIM components are assumed to have been sintered to a point where all porosity is isolated from the surface. It is also assumed that nothing resides within the isolated pores that might hinder densification, e.g., large molecular weight, low diffusivity gasses such as argon. These assumptions are not always true. Not all porosity can be removed via hot isostatic pressing. As shown in Fig. 2, pores or pore-networks that connect to the surface of the part will not be eliminated with HIP. In this situation the high pressure

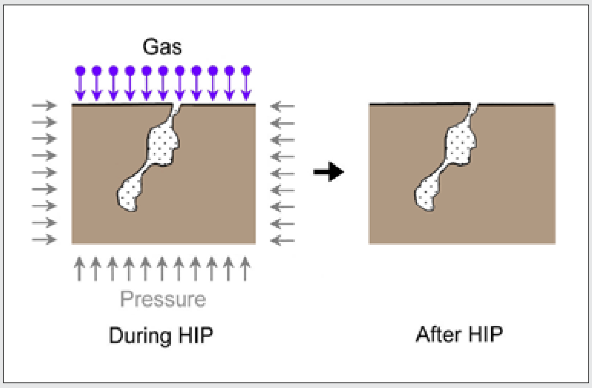

Fig. 2 A schematic showing the HIP of material containing a surface-connected pore. In this situation high pressure gas enters the pore and the gas pressure within the pore matches the external applied pressure. No densification occurs. Image after Mashl [14], a modification (with permission) of images from Atkinson, Davies [15]

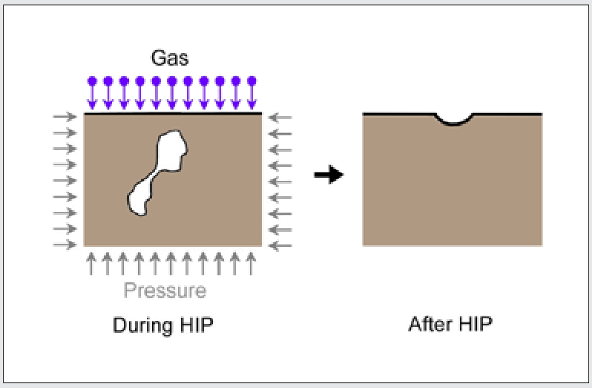

Fig. 3 When a pore is sub-surface and does cannot contain any low diffusivity gasses HIP can completely eliminate that pore. Change in shape and dimensions do occur. There will be an overall volumetric shrinkage equal to the volume fraction of porosity present prior to HIP. Localized deformation during HIP can result in the formation of dimples in the surface of the part, an indication that metal has flowed into a near-surface pore. Image after Mashl [14], a modification (with permission) of images from Atkinson, Davies [15]

Fig. 4 HIP Dimples on the surface of investment cast platinum alloy jewelry in the rough, as-cast state. The dimples indicate that metal has flowed into a near-surface pore. After Fryé, et al. [16]

gas simply penetrates the pore and the forces acting on the exterior of the part are matched by equal and opposing forces acting within the pore. If gas is trapped within a pore at the point at which the pore becomes isolated during sintering and that gas cannot diffuse away from the pore during HIP then a phenomenon known as TIP (Thermally Induced Porosity) is often observed. In this situation, the trapped gas becomes pressurised as the external hydrostatic force acts on the exterior of the part. The parts cool under pressure and thus following HIP the original pores are now much smaller but they contain a pressurised gas. Any subsequent exposure to elevated temperature at ambient pressure, such as in a post-HIP heat treatment will see the metal softened during heat treat and the pores expand back to their near original volume.

With the exception of surface connected and gas filled porosity, HIP is very effective in completely eliminating porosity. A common question is "Will my parts become distorted during HIP?" The quick answer is "Yes". When pore volume is eliminated, there will be an overall volumetric shrinkage that occurs, equal to the pre-HIP pore volume. In MIM products, where the post-sinter porosity is general small and uniformly distributed within the part there will not be any localised distortion, rather a uniform shrinkage of the piece. As shown in Figs. 3 and 4, in cases where near-surface porosity is present, indentations may be observed on the surface of the part after HIP. This is the result of the local flow of metal into the subsurface pore volume.

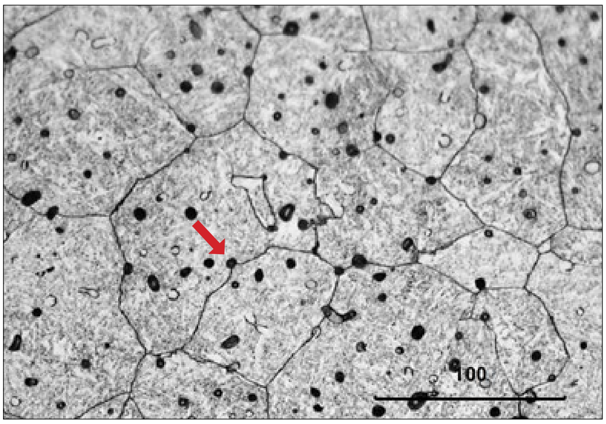

Fig. 9 The microstructure of a MIM 420 stainless steel component in the as-sintered condition. While pores are still seen to decorate the current grain boundaries, many pores have become isolated within the grains after high temperature sintering. The pore indicated by the red arrow still pins a grain boundary but it is about to become isolated as that boundary breaks free and moves to the lower left of the image. It is likely that this part would not consolidate to a pore-free density level during a conventional HIP cycle. Image courtesy of Sandvik Osprey (Tingskog) [11]

taken with consideration of all of the factors that affect densification during HIP and the resultant mechanical properties. A casual approach, simply using established de-bind and sinter practice and adding the most inexpensive HIP option that appears suitable for the alloy being processed may not deliver the full potential of the combined sinter plus HIP process. A high concentration of non-metallic inclusions or the inability of the HIP cycle to eliminate porosity that has become isolated within grains, a result of grain growth during sintering and/or HIP can conspire to thwart the intentions of the parts maker. On the other hand, when HIP is combined with conventional MIM processing and the entire process route is examined and designed to work toward the achievement of a pore-free product, exceptional properties and performance is possible.

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Centorr Vacuum Industries: Celebrating 60 years of innovation in vacuum furnace production

Centorr Vacuum Industries is one of North America's largest manufacturers of standardised and custom high temperature vacuum furnaces and the company has supplied furnaces to the MIM industry for nearly 30 years. During this period the company's product range has continued to evolve as MIM has transformed into a dynamic global industry. Nick Williams interviewed Centorr's Bill Nareski and Scott Robinson and reports on the company's history, its long association with the MIM industry and the unique perspective that the company can offer on technical and commercial developments.

This year Centorr Vacuum Industries (CVI), based in Nashua, New Hampshire, USA, celebrates sixty years of vacuum furnace production. Over this period the company has installed over 6500 furnaces worldwide. The MIM industry is an important market for the company and over the last year more MIM furnaces were commissioned than any other product line in the company's diverse offering, accounting for 25% of its total equipment sales.

Centorr Vacuum Industries was actually formed in 1989 following the merger of two separate furnace companies, Centorr Furnaces, founded in 1962 in Suncook, New Hampshire, a maker of small laboratory and R&D furnaces, and Vacuum Industries, a manufacturer of large production vacuum furnaces founded in 1954 in Somerville, Massachusetts.

Bill Nareski, CEO of Centorr Vacuum Industries, told *PIM International*, "It was the perfect fit between the two companies. It gave the business an extremely broad product line from the smallest laboratory furnace to large production-sized industrial equipment. Our experience in laboratory furnaces and working with corporate laboratories, as well as many major universities with a materials science programme, allowed us to get in on the ground floor of new breaking technology and, when customers are looking to take a new advanced material from the laboratory to full scale commercial production, we can be there with the large scale equipment which we tailor as needed to run the customer's process."

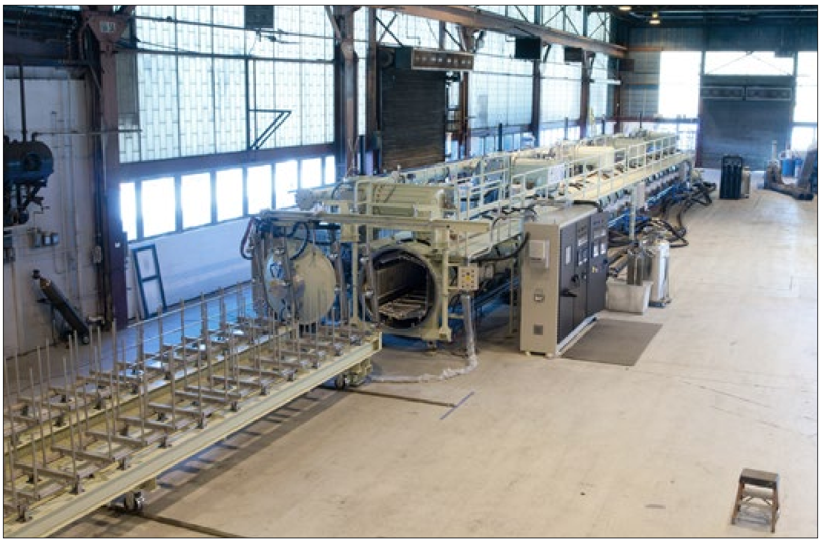

Fig. 1 Centorr Vacuum Industries manufactures some of the world's largest vacuum furnaces, including several 30 m long furnaces for the nuclear industry

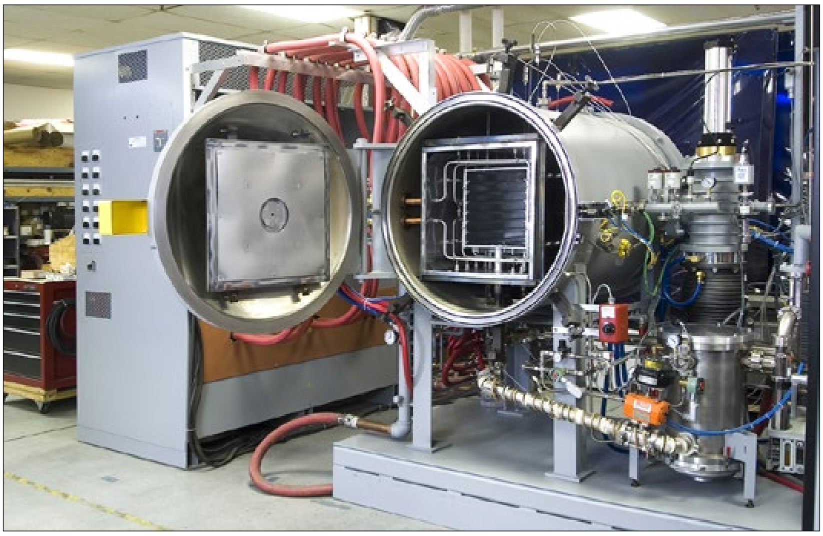

Fig. 2 A Centorr MIM-Vac furnace under construction

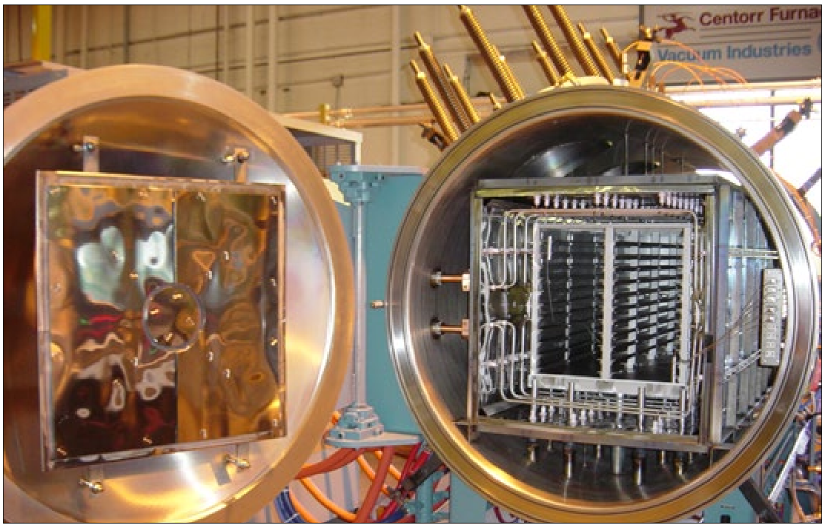

Fig. 3 A Centorr MIM Gas Plenum Retort constructed of advanced Lanthanated Molybdenum (ML) and TZM compositions for improved strength and durability

CVI employs approximately sixty people at its Nashua facility, with around twenty additional sales and service agents representing the company in Europe, Asia, Australia and South America. The Nashua facility has a 2,790 m^2 manufacturing and laboratory area plus an additional 1,860 m^2 of office space. A large 3 x 3 x 3 m pit in the main facility allows for development of tall equipment, essential to support the company's optical fibre drawing furnace product line which supports the photonics and telecommunications industry, as well as the construction of metal wire annealing furnaces.

Over the past ten years, CVI's MIM furnace business has grown to between 15 and 25% of the company's total annual sales. "While this may seem low compared to other vacuum furnace companies, who specialise in only one or two product lines or serve a single main industry and live or die on the strength of that business, in CVI's case - with sales of between \$25 and \$30 million annually - our MIM sales at 25% match or exceed the annual MIM output of most of our competitors. Our MIM numbers are just dwarfed by our higher sales figures for the over 65 different styles of furnaces that we offer for hundreds of applications in the metals and ceramics industry." The company exports approximately 40 - 50% of sales, with most of these furnaces being to Asia, Europe and Canada.

'Over the past ten years, CVI's MIM furnace business has grown to between 15 and 25% of the company's total annual sales'

In January 2002 CVI completed a new building at its Nashua headquarters which includes a tower facility with 5 ton hook and crane with 1200 amp service and 350 KVA chilled glycol system. This enables CVI to design and build large Vacuum Hot Presses, Chemical Vapour Deposition and Chemical Vapour Infiltration furnaces as well as other vertical furnace designs for custom applications in the nuclear industry.

"This investment in facilities and testing is necessary in order to support ongoing interest in advanced ceramics for the semi-conductor, automotive and armour markets," stated Nareski. "To-date the new building has been used for the construction of two large scale vacuum/controlled atmosphere hot presses for advanced ceramics for use in China and the United States, a tall vertical furnace for a proprietary process, a SiC CVD furnace and a host of other furnaces."

For large furnace projects that do not fit in the company's existing building, CVI has access to another site in Nashua with up to 3,715 m^2 of manufacturing space. Since 2008 this facility has been used to construct a number of large 30 m long furnaces, some of the largest vacuum furnaces in the world, for processing nuclear tubing for zirconium fuel rods and Inconel power water reactor tubing.

MIM furnace development at Centorr

Centorr was an early entrant into the market for Metal Injection Moulding furnaces. "We broke into the field by being the first company to offer MIM process and cycle support in our Applied Technology Center. Our early work for some of the MIM industry's pioneers turned into our first orders from companies such as Remington Arms, Megamet, Porite and Moldmasters," stated Scott Robinson, Market Manager for the PIM and PM sectors.

CVI's first dedicated design for the growing MIM industry was the Injectvac™ furnace, introduced in the 1980s, which included a graphite hot zone and a revolutionary gas plenum retort design with hole pattern designed to ensure proper flow dynamics of the inert and process gases over the surface of all the parts. The Injectavac™ furnace processed MIM parts from wax-polymer binder removal through to sintering in one continuous cycle and included a dual pumping system (high-vacuum and low-vacuum) which allowed it to be used to remove both the first stage wax binders as well as the second stage polymers, before going up to sintering temperatures.

The first stage wax binders were removed using a diffusion pump (sub-atmospheric evaporation) at dewax pressures less than 10^{-3} torr. The second stage polymers, on the other hand, would clog the diffusion pump but thermally break down at temperatures over 350°C into compounds with relatively high vapour pressures. The high vapour pressures allow the use of CVI's proprietary Sweepgas™ BRS™ (Binder Removal System) to carry the vapours away from the parts in the direction of the pumps. Inert gas is bled into the furnace chamber and the retort plenum, where it entrains polymers vapourised from the work pieces and carries them out towards the BRS pump. "The unit was very successful at the time," commented Robinson, "and CVI enjoyed the largest installed base of graphite MIM furnaces worldwide with over 20 units installed in the USA and Europe and an additional 22 units in China, Korea, and Japan sold through CVI's licensee."

Fig. 3 A MIM-Vac furnace installed at Dynacast's US MIM operation

Fig. 4 A MIM-Vac furnaces at Dynacast's Singapore operation

As processes and technologies evolved the MIM industry moved away from first stage thermal debinding in batch furnaces and relied more on hot water or solvent debind in the case of traditional wax/polymer binder systems, or catalytic debinding for polyacetal feedstocks. This processing of first stage binder offline meant that MIM vacuum furnaces no longer needed to handle the lengthy wax debind portion of the cycle. This allowed furnace manufacturers to focus on second stage debinding followed by sintering, where such expensive capital equipment could be better utilised.

As newer, higher quality MIM feedstock materials entered the market more stringent demands on physical properties such as carbon control became important and new designs utilising a refractory metal hot zone were needed to process these materials in a cleaner environment.

Leaning on its experience in high temperature refractory metal furnace designs for high temperature sintering at temperatures from 1600°C to 2600°C, and its experience in debinding of over 200 different types of organic binders, CVI introduced its new line of Metal Hot Zone MIM units, named the MIM-Vac, in 1999. "Today's MIM-Vac™ furnaces

Fig. 5 Refractory metal hot zones in Centorr's small continuous belt furnace designed specifically for the micro-MIM market

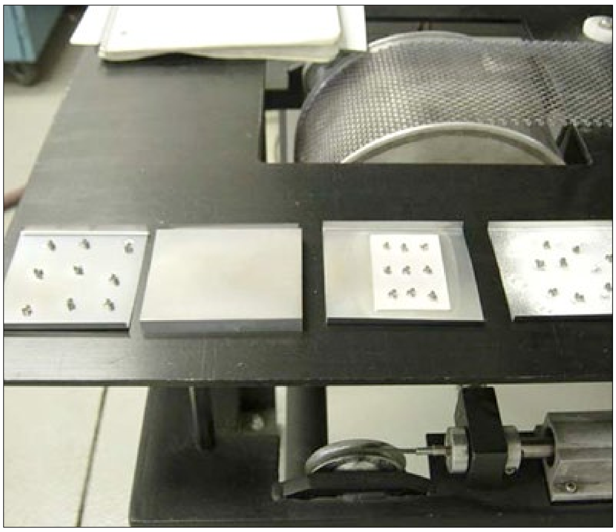

Fig. 6 Trays of small hinges on varied ceramic setter materials including sapphire, ceramic paper and alumina powder

are the result of almost 30 years of experience in PIM technology and over 40 years debinding and sintering experience in metals and ceramic parts. The core design is based on our Workhorse® Metal Hot Zone furnaces which have been sold worldwide for the sintering of PM parts, with over 350 units in the field, and a variation of the binder removal technology from Sintervac® units that were developed for the tungsten carbide industry with over 600 units in use worldwide," Robinson stated.

"The MIM-Vac™ furnace is designed primarily for second stage binder removal and sintering, and has a number of design improvements specific for use with MIM feedstock. Tight partial pressure control and even gas flow, in conjunction with effective event-based programming and sound retort design, allows the entire load to view the same series of conditions as a function of time. This results in consistent microstructures and repeatable carbon control. The judicious use of advanced molybdenum alloys in the hot zone and retort, such as Lanthanated Molybdenum and TZM, offers excellent creep resistance, higher recrystallisation temperatures and longer service life. Our revolutionary new gas-plenum retort has rows of perforations allowing even gas flow across all the work trays and now includes removable plenum panels inset in a strong structural molybdenum framework that allows fast and easy removal and replacement of individual panels, minimising repair costs and downtime."

'The judicious use of advanced molybdenum alloys in the hot zone and retort, such as Lanthanated Molybdenum and TZM, offers excellent creep resistance, higher recrystallisation temperatures and longer service life'

The company states that the use of a heavy duty molybdenum skeleton framework with large cross-section structural members results in less warping and creep over the life of the retort, as evidenced by current units in the field which, CVI claims, are providing two to three times the life expectancy of competitive designs.

A unique perspective on MIM industry growth

Equipment suppliers are well placed to understand industry growth trends and Centorr Vacuum Industries is no exception. Nareski told *PIM International*, "We are encouraged by the recent period of high growth in MIM over the past three years, spurred in part in the US by the rapid increase in the firearms business. It is also refreshing to see firms new to MIM, such as Dynacast International, embrace this technology as a way to increase their product offering and provide their customer base with new materials and improved physical properties."

As reported in the December 2013 issue of *PIM International (Vol. 7 No. 4, p 16)*, CVI received orders for four MIM-Vac M furnaces for Dynacast's new MIM operation in 2013. "In our opinion, Dynacast was a savvy purchaser who had tremendous production experience as an international investment casting firm operating 22 manufacturing facilities in 16 countries. In February 2013 they announced they would be adding MIM to their service offering and the addition of MIM as a manufacturing process meant that the company could expand its ability to produce small, complex components using a far wider variety of metals," stated Nareski.

"Their new twist on the MIM process involved the use of their own revolutionary die-casting machines, modified to run conventional MIM feedstock. We believe it was their desire to have equally high performing furnace equipment to go along with their new process. After a few in-depth meetings where we discussed the features and benefits of our design we agreed that, while cost was an important factor in any purchase decision, Dynacast should take all factors into consideration including overall cost of ownership and life expectancy of the expensive hot zone components."

CVI also commented that it is reassured by ongoing growth and follow up orders from well established firms in the MIM industry such as Parmatech Corporation, which is continuing the growth and expansion plan that it started in 2010. There is also optimism about the growth of MIM in Asia.

Trends in materials

Commenting on trends in MIM feedstock usage Robinson stated, "The range of MIM feedstock on the market truly offers MIM producers a wide choice of materials. We continue to be surprised at the differences that various feedstock systems have on the day-to-day operation of MIM furnaces and their impact on maintenance schedules. We have some customers who are cleaning traps every one or two cycles, whilst others just check the traps once a quarter!"

In terms of the evolution of MIM material grades, CVI sees progress in MIM titanium. "As the technology for stainless steels and low-alloy steels and their applications becomes saturated, it is only natural that the industry will look to new materials for this innovative processing technique. Titanium MIM presents its own barriers to commercialisation but it is impressive to see the progress that has been made in powder and feedstock formulations."

Opportunities in micro-MIM

Centorr is following the technical work done in the micro-MIM field very closely as it believes that this will continue to grow into one of the largest and most profitable segments of MIM. The company has recently enhanced its MIM furnace portfolio with the further development of a small continuous belt furnace designed specifically for the micro-MIM market. Based on CVI's standard continuous belt furnace design the furnace is constructed in a similar way to a vacuum furnace, with water-cooled chamber and refractory metal hot zone. It can achieve a hydrogen gas dew point of -60°C in the main chamber after as little as 45 minutes of purging with inert gas. Unlike conventional belt or pusher furnaces, the hot zone can be brought up to maximum temperature in as little as 60 minutes and the unit can be turned off at night and is cold within an hour.

"Our belt furnace has been used successfully to process small MIM parts, such as orthodontics, in our Applied Technology Center using a 100% hydrogen environment and has achieved product densities of up to 98% theoretical density in 17-4PH and 316L parts with cycle times under 120 minutes door-to-door. Future work in this field will centre around further optimising cycle times," stated Robinson. "We believe that this new small inline continuous MIM belt furnace design could be a real game changer in terms of reducing manufacturing costs and provide a greener footprint for MIM parts makers."

Efficiency gains and process improvement

Centorr has worked with a number of key customers to improve the debinding efficiency of their process and explore ways to tighten up the hot zone and retort to ensure a higher degree of carbon removal. "In one case the collaboration resulted in a reduction in the carbon content in stainless steel parts by over 50% and reduced the effect of carburisation of the hot zone components, increasing hot zone and retort lifetimes," stated Robinson. "Other work in this area has primarily centred on improving trapping efficiencies and looking for ways to reduce the downtime caused by maintenance tasks, all in an effort to shorten MIM cycles and the turnaround time between runs. To do this we have been working closely with vacuum dry pump manufacturers to find ways to improve the robust operation and service life of their equipment."

CVI's Applied Technology Center undertakes all in-house research

Fig. 7 The MIM-Vac 'T/P' Trap (Trap Over Pot) for easy wax/polymer removal features a double O-ring groove seal for H_2 safety

Fig. 8 A basket of dual mechanical filtration media is claimed to be more efficient that condensation-based trapping approaches

Fig. 9 MIM-Vac furnaces are conveniently mounted on a skid which allows for full unobstructed access to the chamber and surrounding equipment

Fig. 10 A MIM-Vac control panel

and is regarded as a major asset to the business. The Applied Technology Center can simulate processes from high-vacuum to positive pressure inert gas and operation in 100% hydrogen gas, with capabilities from 1000°C to 2800°C. This allows customers to test run their materials in different gas atmospheres, vacuum levels and hot zone materials when making the final adjustments to a new process.

"Process evaluation is an important aspect of customer service, enabling prospective users of high-temperature equipment to test ideas and perfect operating procedures so that equipment can be properly specified to perform planned operations. Testing in our Applied Technology Center's continuous furnace has allowed several MIM manufacturers to produce high-quality materials in a quarter of the time required in a conventional batch furnace design due to the reduced furnace cross-section and lower thermal mass. Because of the tightness of the vacuum-tested chamber and refractory metal hot zone inside and use of a proprietary patented molybdenum mesh belt design, these belt furnaces can operate in 100% hydrogen atmospheres at dewpoints as low as -60°C, offering cleanliness unlike traditional continuous furnace designs. The variable and programmable belt speed allows for fast ramp rates and the small cross-section uniform temperature hot zone at < +/- 3°C means that parts can be processed in a quarter of the time when compared to batch furnace cycles."

Furnace maintenance

CVI strongly emphasises the importance of good training and maintenance procedures in order for MIM part producers to achieve the highest levels of efficiency, productivity and reliability from a MIM vacuum batch furnace. "For any company installing vacuum batch units for the first time there will definitely be a learning curve as they get to know the furnace and develop a plan for its maintenance. These units are not heat treatment furnaces where you fill up the vacuum pumps with oil once a quarter and "let 'em run". These furnaces process several kilograms of nasty plastic binder residue each and every cycle and they do it in such a way as to keep the internals of the furnace clean enough to process low-carbon grades of expensive materials. Traps need to be emptied and cleaned, manifolding needs to be checked at major preventative maintenance campaigns and dry pumps need to be cleaned periodically, either manually or with routine solvent flushes."

"Centorr has learned these facts since our development work in the 1980s and we have found several ways to reduce the time required for these maintenance tasks and make them easier to accomplish. We all know that you could write the best SOPs for your team to follow, but the more difficult something is to maintain, the less likely it will get done right, or done at all! Our MIM furnaces have easily accessible large water-cooled traps with a removable basket filled with two different types of filtration media, including a simple binder pot on the bottom that can be removed and cleaned after each run. While one trap is good, two are even better, and we include a second trap on the exit-end of the dry pumping system to catch any residual binder that makes it through the manifolding and dry pump. With this design smelly binder off-gas and small pieces of uncombusted residual binder that make it past the flame tower exhaust, don't rain down all over your nice clean furnace room," commented Robinson.

CVI's Engineering and Aftermarket Field Service Group plays an important role in maintaining a reputation for high quality capital equipment. Nareski stated, "Our customers know that we will be around for the long term providing spare parts, hot zones and field service, as well as furnace maintenance programmes, upgrades and retrofits, rebuilds and controls. In a recent informal survey, we found that nearly 70% of the equipment we have built since 1980 was still up and running and we routinely provide spare parts for some equipment built during the 1970s. It is this attention to quality that allows our capital equipment to perform under all types of conditions with repeatable and reliable service."

'The need for this type of equipment is only going to grow in the future and we're going to be prepared to meet that need'

Selecting the right MIM furnace

CVI states that, when planning a MIM operation, of utmost importance is being sure of the actual material types that a part producer will be processing. Low-alloy steels are commonly processed in simple large sintering furnaces outfitted with small traps and no retorts, using vacuum or low partial pressures of inert gas, because the debinding step is less critical due to the high carbon content in the remaining parts.

Higher grades of stainless steel such as 17-4PH and 316L require more precise operating conditions with higher partial pressures (from 100-300 torr) of typically Hydrogen gas and require more consistent gas flow across the surface of the parts; hence expensive refractory metal gas plenum retort designs, to ensure low carbon content in the finished parts. This can be below 0.03% in the case of some 316L applications. Likewise, titanium MIM grades also require the cleanliness that only a vacuum batch MIM furnace can provide in order to process end product with acceptably low oxygen levels.

"When the end-user considers the overall capacity required, they need to factor in their product mix. Large volumes of only one or two different parts can point towards using continuous furnaces, while a mix of several different product grades or part sizes usually requires the flexibility that only vacuum batch MIM units can offer with their ability to vary cycle times and temperatures for each individual need. While small lower-cost box furnaces of 1 to 2 ft^3 are prevalent in the industry and used by firms as they gear up their production line, once volumes increase it can make sense to consider larger vacuum/hydrogen MIM furnace designs that routinely process 6 or 9 ft^3 of material at a time and offer active fan-cooling features that can drastically shorten overall cycle times producing higher output," stated Robinson.

"When it comes to understanding total cost of ownership, a MIM firm needs to understand their costs for power and gas usage and weigh this against their production schedule. While vacuum batch MIM furnaces typically have larger power supplies than continuous furnaces, they are only operational during heating and not on 24/7 as with continuous pusher designs. Hydrogen gas usage for an average MIM cycle in a 9 ft^3 furnace could be as low as 25,000 litres for some alloys where reduced hydrogen flow is used to prevent decarburisation of the parts, and up to 80,000 litres per cycle for other grades like 17-4PH. Vacuum batch units additionally do not require 24 hour operation as the units can be scheduled so that loading and unloading can occur at defined times."

Outlook

"CVI has been at the forefront of high-temperature vacuum/controlled atmosphere furnace manufacturing for decades and we have no intention of slowing down. My long-term goal for the company is to build on the position of strength we already have and bring Centorr Vacuum Industries to a point where we are considered universally the primary resource for ultra-high-temperature equipment in all the significant applications," stated Nareski. "The need for this type of equipment is only going to grow in the future and we're going to be prepared to meet that need."

"Our depth of experience in engineering and manufacturing has resulted in several new advancements and technologies for the processing of MIM parts. We have excellent financial stability and a history of offering customers a long-term partnership so they can be assured of a ready supply of spare parts and frequent upgrades and improvements to the design in order to keep their equipment operating on the cutting edge."

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APMA 2013 conference: Advances in MIM applications and materials presented in Xiamen

APMA 2013, the 2nd International Conference on Powder Metallurgy in Asia, took place in Xiamen, China, November 3-6, 2013. This, the second conference to be endorsed by the Asian Powder Metallurgy Association (APMA), has helped to firmly establish this conference series as important international events to match Europe's EuroPM and North America's PowderMet conferences. Lin Dongguo and Seong Jin Park, from Pohang University of Science and Technology (POSTECH), Korea, report on a number of presentations relating to MIM applications and materials.

APMA 2013 was the second international conference on Powder Metallurgy in Asia to be endorsed by the Asian Powder Metallurgy Association (APMA) following the first conference, APMA 2011, held in Jeju, Korea, from October 30 to November 2, 2011.

The 2013 event was successfully organised by the China Strategic Alliance for Technological Innovation in Powder Metallurgy Industry (SATI-PM) in conjunction with the Taiwan Powder Metallurgy Association (TPMA). Co-sponsors of the event were the Japan Powder Metallurgy Association (JPMA), Japan Society of Powder & Powder Metallurgy (JSPM), Korean Powder Metallurgy Institute (KPMI), Korean Powder Metallurgy Association (KPMA), Powder Metallurgy Association in India (PMAI), Malaysian Powder Metallurgy and Particulate Materials Association (MPM2A) and Powder Metallurgy Australia (PMA).

The conference's technical programme featured around 180 papers that were presented in six sessions in both oral and poster formats. Main topics included PM iron base materials, refractory metals and hardmetals, PM functional materials, powder forming, post-sintering treatment and PM non-ferrous materials. In addition to a large number of papers from China, reflecting the rapid progress and development of China's PM industry, papers were submitted by participants from all world regions including the USA, Europe, Japan, Korea, India, Australia, Malaysia and Taiwan.

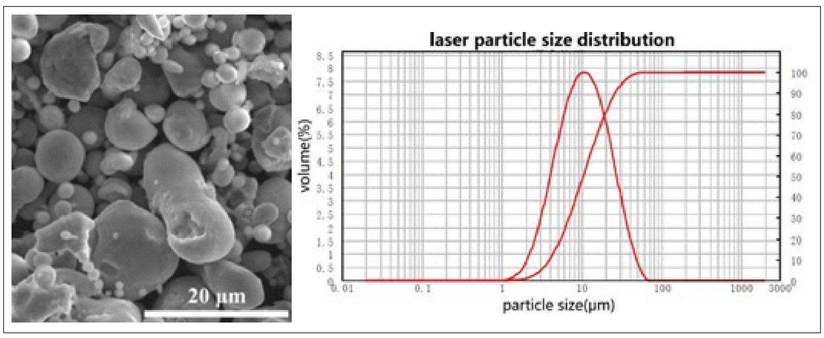

Fig. 1 SEM image and particle size distribution of HK30 powder [1]

Advances in MIM technology

The process and properties for MIM heat resistant stainless steel

Heat resistant austenitic stainless steel is widely used for components working at high temperature, such as engine and turbocharger parts. Investment casting (IC) has been a conventional process for manufacturing these components in complex shapes. However the parts always needed some secondary operations

Element	C	Si	Mn	P	S	Ni	Cr	Mo	Nb
Content	0.25-0.35	0.75-1.75	≤1.5	≤0.04	≤0.04	19-22	23-27	≤0.5	1.2-1.5

Table 1 Chemical composition of HK30 powder, wt.% [1]

because of unsatisfactory dimensional tolerances and poor surface finish. The use of MIM as a net-shape approach to producing these small and intricate parts in large quantities has been able to solve the problem in an effective way.

A research group consisting of W Wang, M Ouyang, D Ye, J Song, F Pen and Y Yu from Xiamen Honglu Tungsten Molybdenum Industry Co. Ltd. along with Z Zhuang from China National R&D Center for Tungsten Technology, reported the research results of MIM processing of HK30, one of the heat resistant austenitic stainless steel powders. They also showed images of actual production components such as the nozzle ring for variable geometry turbochargers (VGT) fabricated by the MIM process.

Based on a wax-polymer binder system, HK30 MIM feedstock was prepared. The powder morphology and particles size distribution are shown in Fig. 1, and Table 1 shows the chemical composition of this powder.

The MIM feedstock was prepared with a powder loading value of 60 vol. %, and the feedstock was injection moulded into tensile test specimens with the appropriate conditions. Two-stage debinding was carried out for the debinding process; solvent debinding was done in heptane for 8 h, and thermal debinding was done in N_2 flow gas in a batch furnace. Finally, sintering was carried out at 1300°C for 120 min in partial atmosphere (N_2 or Ar).

Table 2 shows the effect of sintering atmosphere on final properties of the sintered parts. In conclusion, the N_2-sintered parts have higher density, hardness, tensile strength and yield strength, but the elongation is much lower than the Ar-sintered ones. The authors claimed that the differences were mainly caused by the differences in microstructure formation during the sintering process because of the different atmospheres.

	N_2-sintered	Ar-sintered
Sintering Temp. (°C)	1300	1300
Density (g/cm^3)	7.65	7.50
Micro hardness (HV5/25)	247	162
Rockwell harness (HRB)	90	78
Ultimate strength R_m (MPa)	730	560
Yield strength $Rp_{0.2}$ (MPa)	332	194
Elongation A (%)	12.5	33.4

Table 2 Properties of sintered parts at different sintering atmosphere [1]

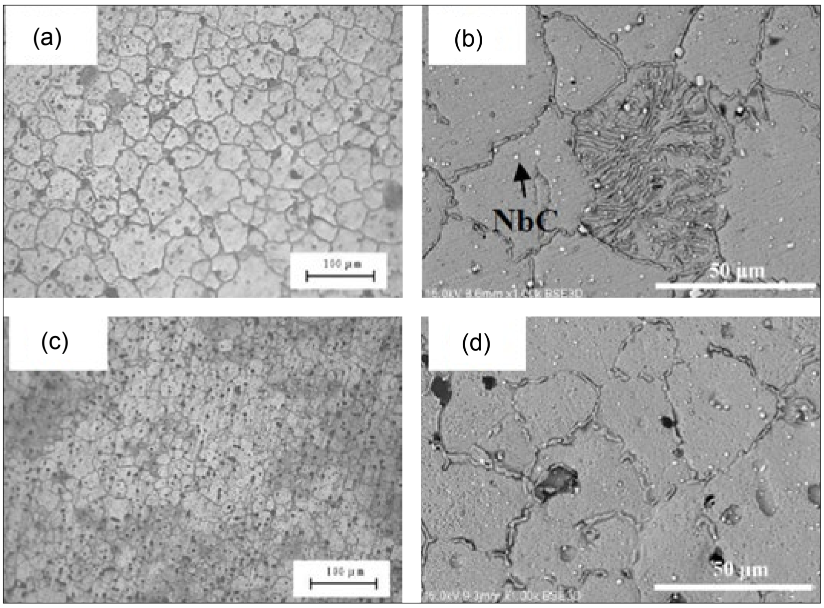

Fig. 2 Microstructures of HK30 parts sintered in different atmospheres: (a) LOM image, N_2 sintered; (b) SEM image, N_2 sintered; (c) LOM image, Ar sintered; (d) SEM image, Ar sintered [1]

Fig. 2 shows the microstructures of HK30 parts sintered in different atmospheres. For N_2-sintered parts, the grain size was in the range of 60-70 μm and it was detected that Cr and Fe rich phases existed along the grain boundary. At the same time NbC particles dispersed uniformly in the austenite matrix. These precipitates were thought to be the reason for the increases in mechanical properties. However, in Ar-sintered parts smaller grain sizes of around 30-40 μm were found and it was considered that this increased the elongation of the sintered parts.

The authors concluded that the MIM process for HK30 heat resistant stainless steel was now well advanced and the MIM process highlighted had been successfully applied for the fabrication of the guide vanes of the nozzle ring for VGT applications as shown in Fig. 3.

Performance improvement of MIM Fe-50%Ni soft magnetic material

Fe-50%Ni is one of the most well-known soft magnetic materials and MIM has been proved as a predominant method for the production of soft magnetic parts as an alternative

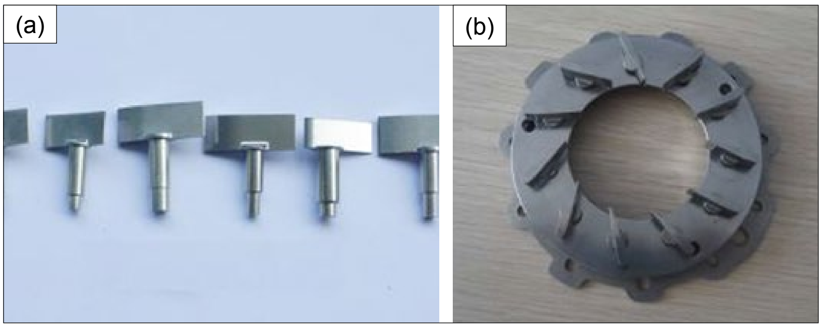

Fig. 3 HK30 products fabricated by MIM, (a) HK30 guide vanes; (b) an assembled VGT nozzle ring [2]

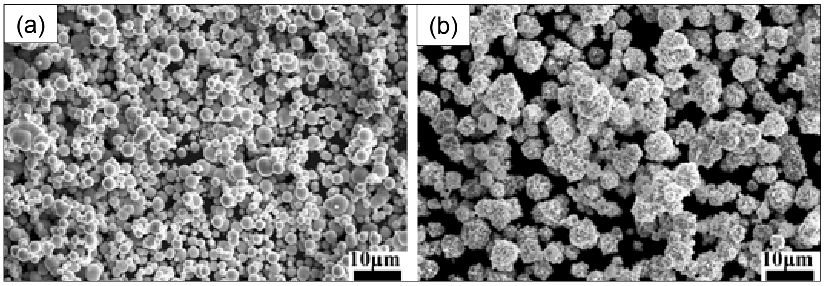

Fig. 4 SEM images for powders: (a) carbonyl iron; (b) carbonyl nickel

Sintering temperature (°C)	Sintering time (h)	Saturation induction B_s (T)	Coercive force H_c (A/m)	Maximum permeability μ_m	Relative density (%)
1300	2	1.315	15.60	12386	93.8
1320	2	1.368	14.36	13222	94.3
1340	2	1.386	13.09	15673	94.6
1360	2	1.397	11.06	21897	94.6
1360	6	1.468	8.25	36058	97.0
1360	10	1.484	6.84	43541	97.2
1360	12	1.484	6.82	43542	97.2
1360	14	1.484	6.79	43581	97.2

Table 3 Effects of sintering conditions on density and magnetic properties [2]

Treatment	Relative density (%)	B_s (T)	H_c (A/m)	μm
Sinter	97.2	1.484	6.84	43541
HIP	100	1.564	10.90	32039

Table 4 Density and magnetic properties before and after HIP [2]

Time (h)	B_s (T)	H_c (A/m)	μm	Relative density (%)
2	1.568	2.98	78159	100
4	1.570	2.82	81869	100
6	1.572	2.66	86446	100
8	1.578	2.56	88714	100
10	1.579	2.52	88754	100

Table 5 Effects of heat treatment on magnetic properties [2]

to traditional casting and machining. A paper by Jidong Ma, Mingli Qin, Li Zhang, Lusha Tian and Xiaofeng Zhang, University of Science and Technology Beijing, China, aimed at studying the MIM process for fabricating high magnetic property Fe-50%Ni alloy, where the values of maximum permeability and saturation induction were 88745 and 1.58 T respectively, far higher values than typically reported for MIM Fe-50%Ni parts.

Carbonyl iron and carbonyl nickel powders with particle sizes of 4.33 μm and 4.45 μm respectively were used as starting materials. The powder morphologies are shown in Fig. 4, where iron powder was regular spherical but nickel had branched chain.

The feedstock was made by mixing the two kinds of powders with weight ratio of 1:1 and mixed with a wax-based binder system. After injection moulding and debinding, sintering + Hot Isostatic pressing (HIP) + heat treatment was carried out. Ring-shaped samples were used to measure the magnetic properties.

Table 3 illustrates the effects of sintering temperature and sintering time on the density and magnetic properties of sintered Fe-50%Ni alloy. The density, saturation induction and maximum permeability increased with increase of temperature from 1300-1360°C, but the coercive force decreased. It was the same situation for an increase in sintering time. After sintering for 10 h, there were modest value changes for density and magnetic properties.

Fig. 5 shows the microstructures of specimens fabricated in different process conditions. Some results can be obtained from the microstructures in Fig. 5 a-d as follows: i) the amount of porosity decreased with the increase in sintering temperature from 1320-1360°C; ii) grain growth developed from about 30 μm to 100 μm with temperature increase from 1320°C to 1360°C and a similar effect was seen with an elongation of sintering time; iii) grain boundaries became straight when sintering time was 10 h at 1360°C and there were minor changes in microstructure with

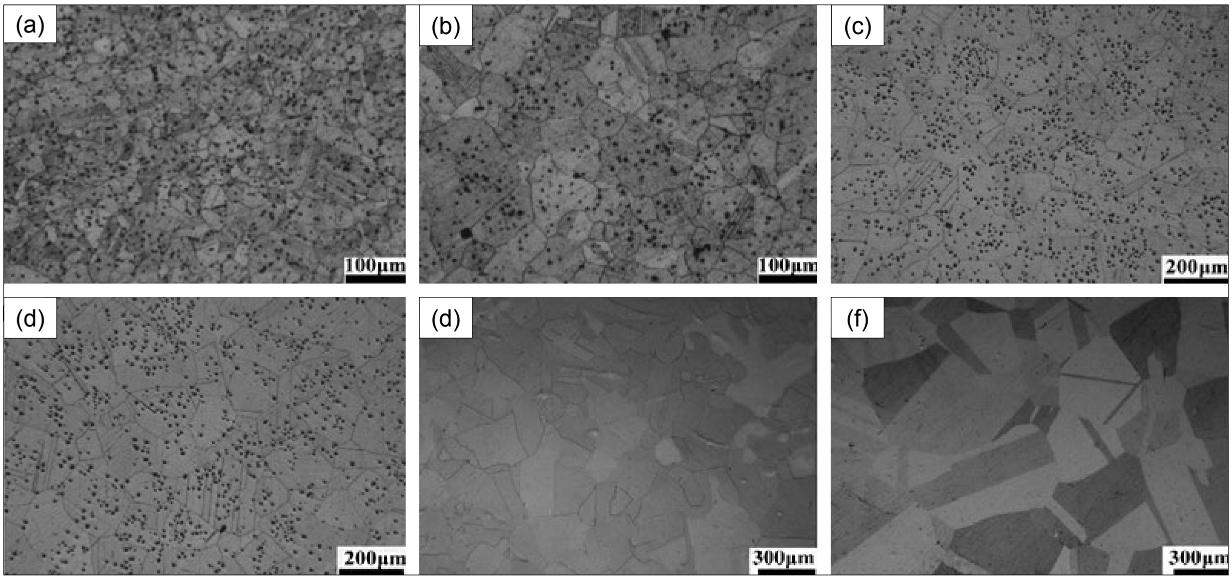

Fig. 5 Microstructures of different samples: (a) sintered at 1320°C, 2 h; (b) sintered at 1360°C, 2 h; (c) sintered at 1360°C, 10 h; (d) sintered at 1360°C, 12 h; (e) sintered at 1360°C, 10 h, HIP; (a) sintered at 1360°C, 10 h, HIP and 6 h heat treatment [2]

a further increase in sintering time.

The authors claimed that for soft magnetic materials the saturation induction depends on the material and its density, but is not affected by grain size. Compared to saturation induction, the maximum permeability and coercive force are microstructure sensitive parameters influenced by density, porosity and grain size. When the grains grow to a certain size, the driving force and resistance reach a balance. Further sintering cannot induce the grain growth and the density does not increase any further.

Fig. 5e shows the microstructure after a HIP process. There are no pores and the sample is fully densified. Table 4 shows the different properties of samples before and after HIP. The authors indicated that the existence of internal stresses after HIP caused a decrease of maximum permeability.

Additionally, high temperature heat treatment was carried out with a view to eliminating or reducing stresses and improving magnetic properties. The effects of heat treatment on magnetic properties are shown in Table 5. The maximum permeability increased largely with longer heat treatment times and changed little when a certain value was reached. Fig. 5f shows the microstructure of a sinter + HIP + heat treatment sample. Rapid grain growth occurred with HIP and heat treatment because of the elimination of porosity and the high temperature involved.

Finally, the authors concluded that a high performance Fe-50%Ni soft magnetic alloy was successfully fabricated by MIM and the maximum permeability value reached 112 mH/m, a significantly advanced result compared to previously reported values for MIM Fe-50%Ni of 22.36-42.50 mH/m) after sinter + HIP + heat treatment.

Developments in MIM related R&D

A significant amount of research work relating to conventional Powder Metallurgy was reported during the four day conference and research relating to refractory metals and hard materials accounted for a significant proportion of this. China has the largest refractory and hard materials industry in the world, and Xiamen, where APMA 2013 took place, is a major centre for the production of tungsten and its alloys in China. In this second part of our APMA 2013 conference report, two papers relating to powder production technologies for tungsten and its alloy powders, and two papers relating to application developments, are reviewed.

Near spherical tungsten powder prepared from tungsten oxide

An impressive tungsten powder fabricating method was reported in a paper by Wang Congcong *et al.* from University of Science and Technology, China on behalf of their co-authors, Gai Guosheng and Yang Yufen from Tsinghua University, China. Tungsten oxide powder was treated by the Particle Composite System (PCS) to obtain the nearly spherical tungsten powder through reduction technology.

PCS is a device that treats the powder in a dry mechanised way by high speed gas impact, which can modify the materials processed into spherical tungsten powder by compression and frictional force between the particles. This manufacturing process provides some significant advantages, such as a simple process, efficient production, short production term, easy parameter control, low cost etc.

The raw material used in this study was tungsten oxide powder with an average particle size of 12.61 μm. The raw powders were treated in a modified PCS device with a rotational speed at 4000 r/min for five different processing times (5, 10, 30, 45 and 60 min). The modified powders were reduced in a hydrogen atmosphere at 780°C for 120 min to obtain nearly spherical tungsten powders.

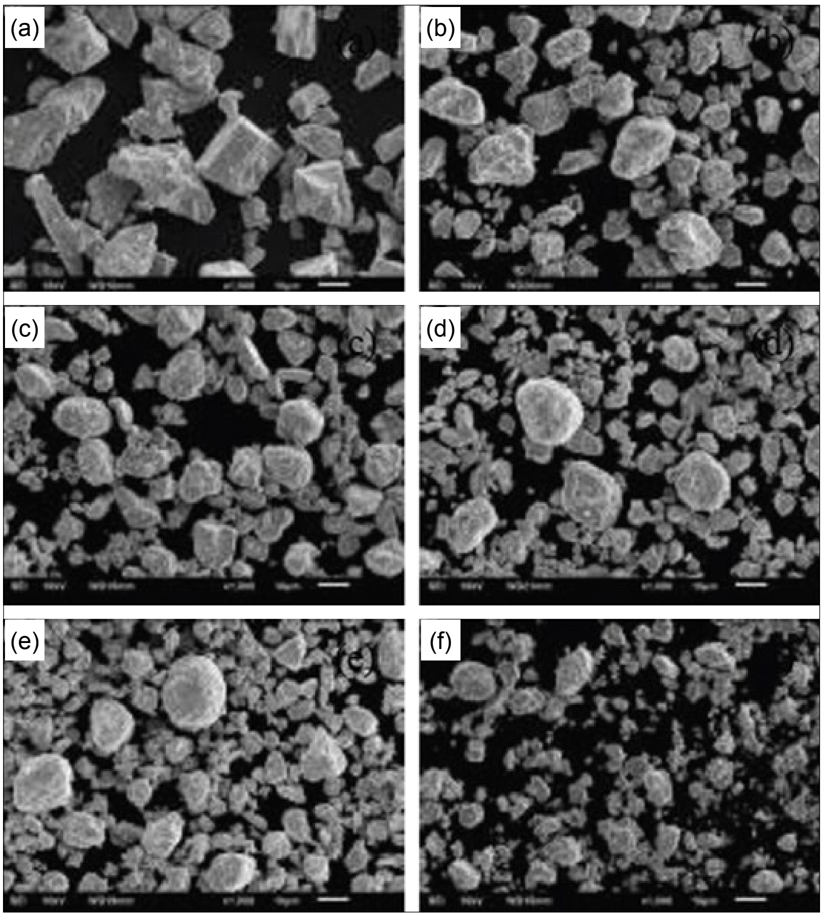

Fig. 6 SEM images of tungsten oxide powders under different PCS processing times: (a) raw material; (b) 5 min; (c) 15 min; (d) 30 min; (e) 45 min; (f) 60 min [3]

Time (min)	0	5	15	30	45	60
D_{50} (μm)	12.61	4.92	4.76	4.24	4.15	2.65

Table 6 Mean particle size under different processing time

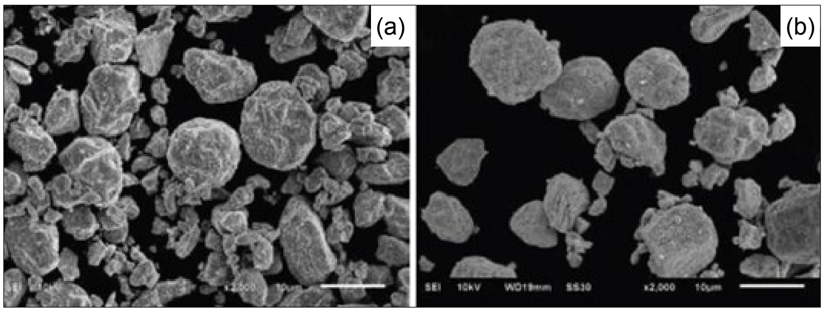

Fig. 7 SEM images of powders before and after reduction: (a) before reduction; (b) after reduction [3]

Sample	Element	Energy (keV)	Mass (%)	Atom (%)
Before reduction	O K	0.525	20.07	74.26
	W M	1.774	79.93	25.74
After reduction	O K	0.525	0.47	5.21
	W M	1.774	99.35	94.79

Table 7 EDS analysis of powder before and after reduction [3]

The SEM morphologies of the tungsten oxide powders under different processing times with PCS are shown in Fig. 6. At first, the raw tungsten oxide powder showed blocky granular shape and sharp corners, but with the increase of PCS processing time, the powders became subglobose as a result of worn-off powder edges and corners. As mentioned in this study, after 15 min treatment, the edges and corners of large particles had almost been removed and reached the best treatment effect. When the processing time went up to 60 min, the particle size decreased obviously because of a crush phenomenon, which indicated too much energy and ground off edges. The relationship between particle size and processing time is shown in Table 6.

The 15 min treated tungsten oxide powder with best treatment effects was reduced in hydrogen atmosphere at 780°C for 120 min. The results are shown in Fig. 6c and Table 6. The reduction of tungsten oxide can typically be divided into four steps as follows:

1) $WO_3 + 0.1H_2 = WO_{2.9} + 0.1H_2$
2) $WO_{2.9} + 0.1H_2 = WO_{2.7} + 0.18H_2$
3) $WO_{2.72} + 0.72H_2 = WO_2 + 0.72H_2$
4) $WO_2 + H_2 = W + 2H_2O$

In Fig. 7, the surface of the powder was polyporous and flawed after reduction because the cracks made by generated H_2O evaporated from the particles as can be seen in the chemical equations mentioned above. This also led to a loose structure and some large particles broke down into small ones. Finally polyporous nearly spherical tungsten powder was obtained with high porosity due to the water vapour release effect during reduction. The EDS analysis results (Table 7) showed that tungsten oxide powder was almost totally reduced into pure tungsten powder.

Finally, the author concluded that the method using PCS followed by the reduction process can be used to produce nearly spherical or spherical tungsten powder particles in mass production, which simplifies the production process and also has many advantages, such as low cost, easily controlled parameters and so on.

Synthesis of superfine W-Ni-Fe powders by a chemical method

The W-Ni-Fe alloy is an important material for both scientific and military applications due to its excellent wear resistance, good ductility and high strength. Many methods have been investigated to improve the mechanical properties of tungsten heavy alloys through the refinement of microstructures. A paper about the preparation of superfine W-Ni-Fe composite powders using a soft chemical method, the Glycine-Nitrate Process (GNP), was given by Yan Liu and Jigui Cheng *et al.* from Hefei University of Technology, China.

The raw materials used for synthesis were APT, $Fe(NO_3)_3 \cdot 9H_2O$ and $Ni(NO_3)_2 \cdot 6H_2O$. They were dissolved in distilled water to form a homogeneous solution with the Ni and Fe content of 3.5% and 1.5%, respectively. Black precursor powders were obtained by heating up the glycine ($C_2H_5NO_2$) added solution. It was then calcined at 500°C to remove residual organics and reduced in hydrogen atmosphere at 800°C to convert into W-Ni-Fe alloy powder. The SEM morphologies for precursor powders and the reduced powder are shown in Fig. 8. The precursor powder had an average particle size of around 2.4 µm with nearly spherical shape. However, after reducing the powders became smaller, with an average particle size of about 150 nm, and the powders become agglomerated because of their high surface energy.

The synthesised W-Ni-Fe powders were sintered at temperatures ranging from 1350-1500°C to study their sintering behaviour. Table 8 shows sintered densities at different sintering temperatures. The nano-sized particles and fine tungsten, nickel and iron ingredients in W-Ni-Fe played an important role in improving sintering densification. A relative density of about 92.3% was achieved when sintered at 1400°C, a temperature below the melting point of γ-(Fe, Ni) phase, which was compatible to that obtained from thermomechanical method. At 1500°C, a higher temperature than the melting point of γ-(Fe, Ni) phase, liquid-phase sintering occurred and a maximum density of 98.6% was achieved.

Microstructures of the W-Ni-Fe sintered compacts are shown in Fig. 9. From Fig. 9 a-c the grains shown are irregular in shape and the average grain sizes increased from 2.51 µm (1350°C) to 3.99 µm (1450°C). However, when sintering at 1500°C, the tungsten phase was nearly spherical in shape and grain growth was clear.

The authors concluded that a superfine 95W-3.5Ni-1.5Fe composite powder, spherical particles with an average size around 150 nm, was successfully produced by the gluyine-nitrate process. After sintering at 1480°C, a high relative density (above 96%) and 2 µm grain sized W-Ni-Fe was obtained and with liquid-phase sintering at 1500°C the sintered density increased to 98.6% and the microscopic hardness values was about 480 MPa.

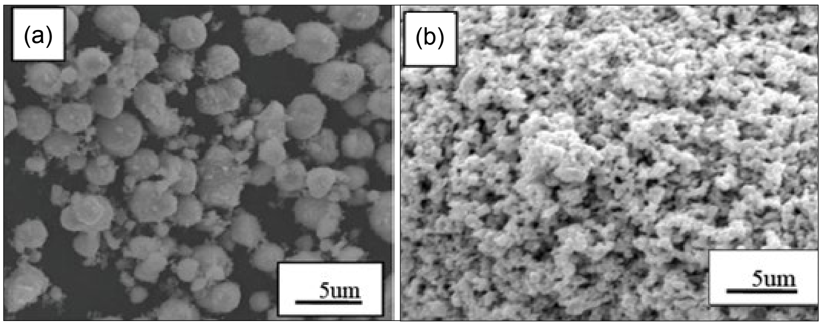

Fig. 8 SEM images of two powder: (a) calcined powders; (b) reduced powder [4]

Density	Sintering temperature (°C)			
	1350	1400	1450	1500
Sintered density g/cm³)	17.32	17.62	17.93	18.33
Relative density (%)	92.3	93.9	97.5	98.6

Table 8 Densities after sintering at different temperatures [4]

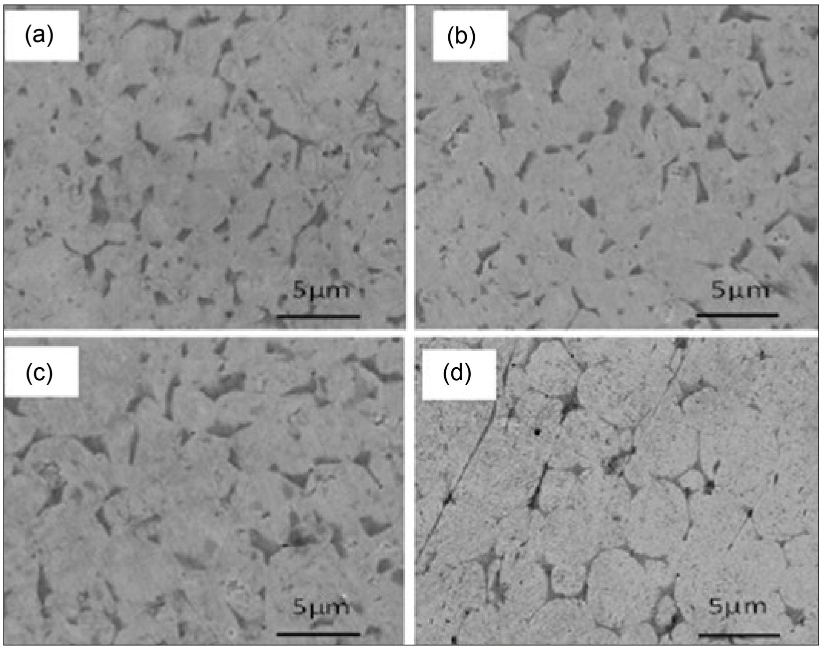

Fig. 9 Microstructures of W-Ni-Fe samples sintered at different temperatures: (a) 1350°C; (b) 1400°C; (c) 1450°C; (d) 1500°C [4]

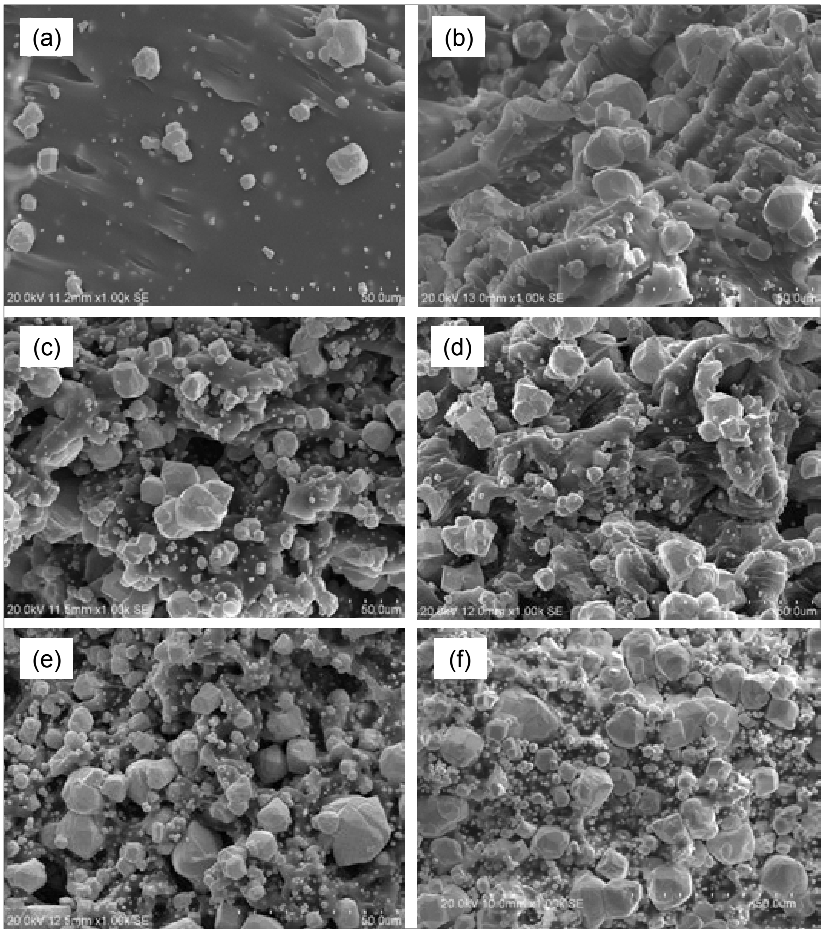

Fig. 10 SEM images of fracture surfaces of tungsten/TPE composites: (a) 10 vol. %; (b) 20 vol. %; (c) 30 vol. %; (d) 40 vol. %; (e) 50 vol. %; (f) 60 vol. % [5]

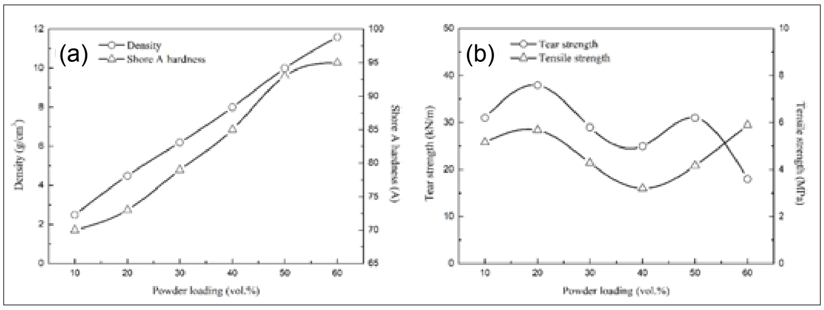

Fig. 11 Densities and mechanical properties at different powder loading: (a) density and shore A hardness; (b) tear strength and tensile strength [5]

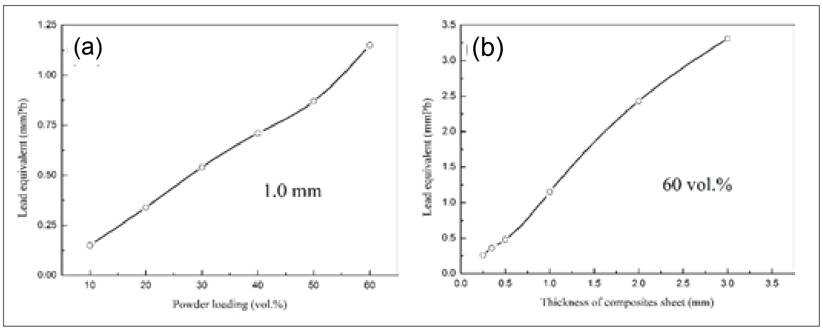

Fig. 12 Lead equivalent values of different tungsten/TPE composites: (a) different powder loading (b) different thickness [5]

High-density tungsten/polymer composites used in lead-free X-ray radiation shielding

Tungsten and its alloys have become some of the most essential industrial materials in numerous applications because of their outstanding performance, such as high density, hardness, conductivity and so on. A report on new application of tungsten was given by Guozhang Zhao *et al.* from Xiamen Honglu Tungsten Molybdenum Industry Co., Ltd, China, with co-authors Zhigang Zhuang (Xiamen Tungsten Co., Ltd, China) and Qingdong Zhong (Shanghai University, China). Their paper aimed at the study of high-density tungsten/polymer composites used in lead-free X-ray radiation shielding.

Because of the outstanding shielding performance against both X-rays and γ-rays, tungsten and its alloys have been utilised in many radiation protection systems as a lead-free material. In this work, a tungsten/polymer composite with high density, perfect radiation shielding performance, good flexibility, excellent workability and perfect environmental suitability, based on thermoplastic elastomer (TPE) and micro-sized tungsten powders, was successfully produced with powder loading of 60 vol. %. The composite was calendered into 2 mm thick sheets for properties measurement.

Fig. 10 shows the microstructures of fracture surfaces of tungsten/TPE composites with different tungsten powder loadings. A widely particle size distributed tungsten powder was used after physical and chemical modification in order to obtain high flowability and high bulk density. At powder loading of 60 vol. %, the tungsten particles were tightly stacked, smaller ones were packed in spaces between larger ones and spaces in close-packed particles were filled with TPE. The author claimed that the blurred boundaries between tungsten particles and TPE matrix showed an efficient polymer-filler interfacial network had been constructed.

The densities and mechanical properties of tungsten/TPE composites are summarised in Fig. 11. The density reached 11.6 g/cm^3 at powder loading of 60 vol. %, a higher value than that of pure lead (11.3 g/cm^3), which was helpful in improving its radiation shielding ability. Also, the tear strength and tensile strength reached 18 kN/m and 5.9 MPa, respectively.

Lead equivalent value is the thickness of lead that gives the same reduction in radiation. In this work, lead equivalent values of different tungsten/TPE composites were measured under conditions of 120 kV for incident voltage, 2.5 mm Al for filtration and 6.0% for uncertainty. The results are shown as Fig. 12. A lead equivalent value of 2.43 mm Pb was obtained with 2 mm in thickness at 60 vol. % powder loading.

The author said that three effects, Compton Effect, Photoelectric Effect and Electro Pair Effect, are the main interaction patterns between X-rays and substance. In this work, since the incident voltage is 120 kV, the former two effects played the leading role in absorption of X-ray energy. Thus, the X-ray tube voltages of devices in nuclear medicine and security inspection are generally below 120 kV, this tungsten/TPE composites sheets can satisfy radiation shielding requirements at different X-ray exposure conditions and working environments. Finally, they have concluded that a lead-free high-density tungsten/polymer composite was produced for the field of X-ray and γ-ray radiation shielding. The composite was demonstrated to have the necessary performance such as high density, perfect radiation shielding performance, good flexibility, excellent workability and environmental suitability.

Ultra high purity tungsten and its applications.

Another paper was reported by Yang Yu *et al.*, also from Xiamen Honglu Tungsten Molybdenum Industry Co., Ltd, focused on production and application of ultra-high purity tungsten powder.

Ultra high purity (>99.9999 wt.%) tungsten and its high purity (>99.999 wt.%) alloys are the vital materials for special lighting and semiconductor industries. The 5N W (>99.999 wt.%) and 6N W (>99.9999 wt.%) electrodes are usually used in ultra-high-performance (UHP) gas discharge lamps, and high purity W-Ti and W-Si are used for sputtering targets in semiconductor chips.

In-house developed 6N W powder has been introduced in this study. Based on the powder, W rods and wires were made for special lighting applications, and 5N W-Ti and 4N W-Si sputtering targets were also developed successfully. Three different processes were used to produce each product as following:

- 5N W electrodes:
  6N W powder -> CIP -> Sintering -> Forging -> Machining & Polishing
- 6N W rods (wire):
  6N W powder -> CIP -> Sintering -> Swaging & Drawing -> Straightening (Electrolytic cleaning)
- High purity W alloys:
  6N W powder & Ti (Si) powder -> CIP -> Hot pressing -> Machining & Polishing

The contents of impurity elements in 6N W powder are shown in Table 9, and the mean particle size was about 2.4 μm with a nearly spherical shape. Using this powder, W electrodes (diameter: 12-40 mm) and W wires (diameter: 0.02-12 mm) were produced with a W purity value of 99.99989 wt.% and 99.999922 wt.%, respectively, and their microstructures are shown in Fig. 13. Higher

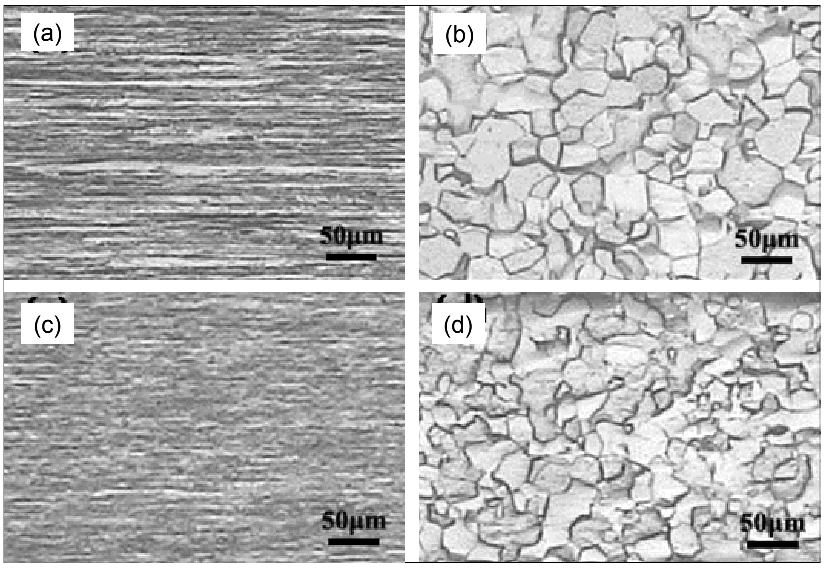

Fig. 13 Microstructures of W rod and W wire: (a) axial section of W rod; (b) cross section of W rod; (c) axial section of W wire; (d) cross section of W wire [6]

Element	Content (ppm wt)	Element	Content (ppm wt)	Element	Content (ppm wt)
Si	0.04	K	<0.01	U	<0.0001
P	0.07	Na	<0.01	Th	<0.0001
Ca	0.04	Ti	0.02	C	32
Cr	0.07	Mo	0.05	N	42.9
Fe	0.08	Ba	0.31	O	450
Co	0.008	As	0.01	S	10
Ni	<0.005	Cu	0.01	H	63.9
Total purity of W: 99.999931 wt.%					

Table 9 Impurities analysis of 6N W powder [6]

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purity was couple with lower diameter product, because the fine wires were fabricated by self-resistance sintering at higher temperature around 2700-2800°C, which was favourable to improving the purity of W. Also, 5N W-10Ti alloy and 4N W-30Si alloy targets were produced successfully with W purity value of 99.999 wt.% and 99.99 wt.% respectively.

The authors have concluded that using the in-house developed technology a type of 6N W powder had been mass produced, and using this powder full dense 5N and 6N W rods and wires had been produced successfully. In parallel, related W alloy products, 5N W-10Ti and 4N W-30Si sputtering targets, had been developed for micro electronic industries successfully.

There is no doubt that this type of material will be used in many other industrial areas where there is a need for very high purity levels in the final product. It is believed that such high purity powders can be used in the MIM industry for the production of a number of new high purity tungsten and tungsten alloy parts.

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Advances in PIM process simulation speed up the development of micro parts

Simulation technology for the Powder Injection Moulding (PIM) process has advanced rapidly in recent years, with software now tailored specifically to modelling the injection moulding of PIM feedstock. In this report Jochen Heneka and Tobias Müller, Karlsruhe Institute of Technology (KIT), Germany, and Dr Marco Thornagel and Dr Laura Florez, SIGMA Engineering GmbH, Germany, present an overview of the most important developments for the simulation of PIM applications along with a case study illustrating how effectively simulation can support mould design and quality control in micro-PIM applications.

The simulation tools available to the PIM industry have evolved substantially over the last five years. During Arburg's 50th Anniversary PIM Conference, held in Lossburg, Germany, June 2013, Ingo Cremer (EPMA President 2007–2013) gave an overview of the leading new technologies in PIM. In his presentation he highlighted simulation as one of the key technological trends for PIM and stated that simulation software "is now mature" and "a must for part development."

Simulation is not new to the PIM industry. It is widely recognised that the injection moulding stage is critical for green part quality and that errors induced here cannot be corrected later on. Therefore, from the very beginning moulders have relied on conventional injection moulding simulation tools – designed to model the behaviour of thermoplastic materials – to gain some insight into feedstock behaviour while being moulded. Large deviations were found, however, and simulation was regarded sceptically as a tool able to deliver, at best, only a general idea of flow behaviour.

That deviations were found are understandable: thermoplastics are quite different in nature from PIM feedstock and its rheological and thermal behaviour requires different modelling. In the last few years

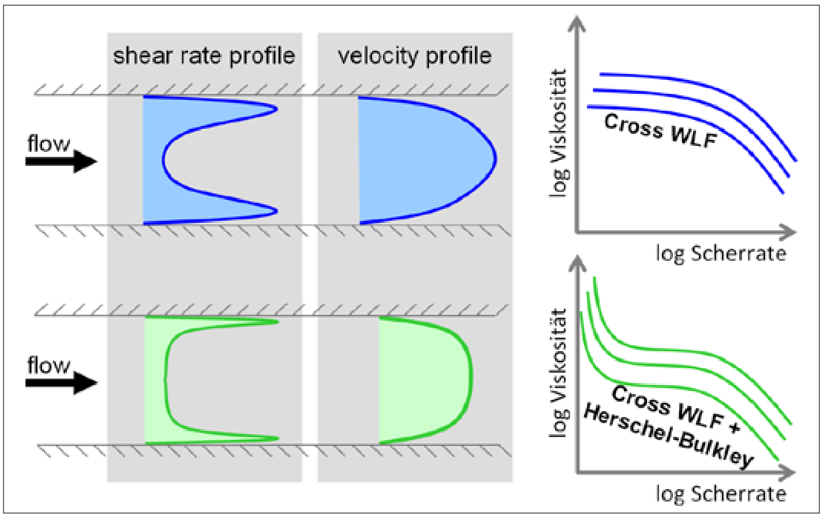

Fig. 1 PIM feedstock shows an increase in viscosity at low shear rates, responsible for several flow effects. A suitable rheological model must be used in the simulation to capture this particular behaviour [1]

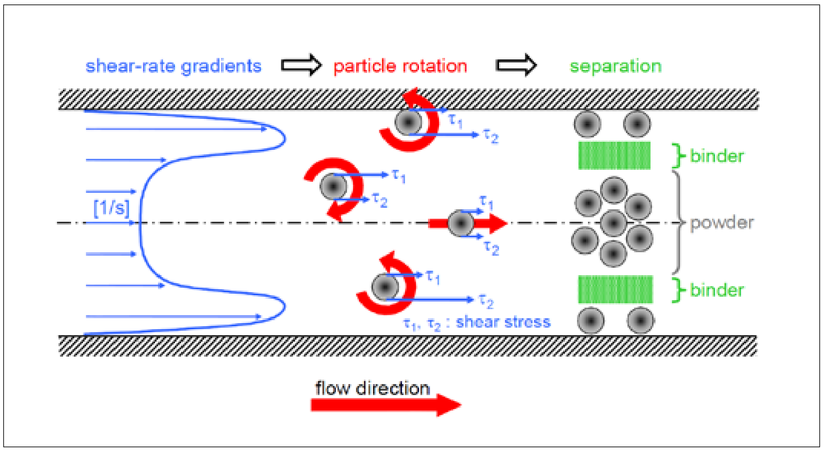

Fig. 2 An increase in the shear rate can produce particle separation from the binder system. Simulation can identify the high shear rate regions susceptible to particle segregation [2]

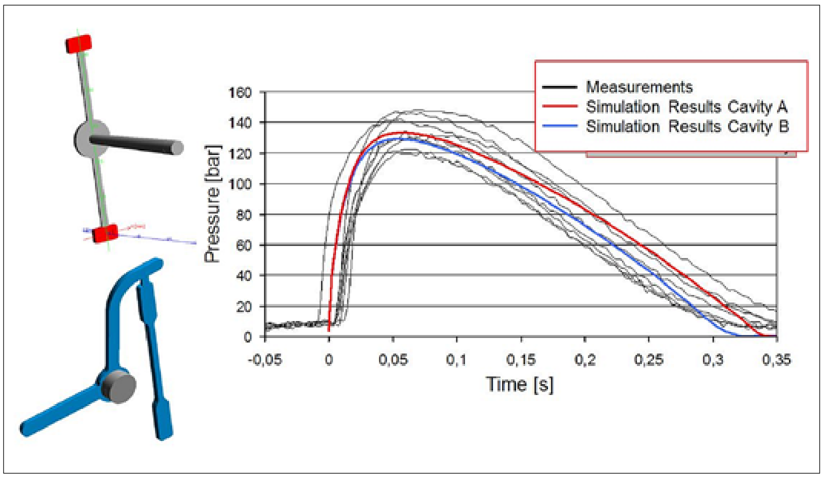

Fig. 3 SIGMASOFT® includes material models able to capture the moulding behaviour of microparts. The pressure curves for the two-cavity mould (left on the top) proved to correlate good with real measurements [4]

important developments have been made to integrate suitable material models able to capture the particular behaviour of PIM feedstock.

The SIGMASOFT® software provided by SIGMA Engineering GmbH based in Aachen, Germany, has proved that simulation is now far closer to solving the challenges faced by the PIM industry than ever before. Models integrated to predict the rheological behaviour at low shear rates, and the ability to predict the effect of the high thermal conductivity of PIM feedstock in the thermal behaviour of the mould, now deliver a very accurate correlation between simulation and reality, capturing even complex flow kinetic effects such as jetting or particle segregation.

As a result, the time-to-market is shortened, the mould design – even the design of the tempering media or layout – is optimised and, foremost, the risk of mould rework or iteration on the steel mould is reduced and in some cases even avoided altogether.

A review of current applications of simulation in Powder Injection Moulding

SIGMASOFT® software has been used widely in PIM companies, for material selection and part design through to mould evaluation and process setup. When developing a new application, simulations are run to compare the behaviour of different materials, to evaluate factors such as the pressure requirements, weld line appearance, air entrapments, required cycle time and the balance of multi-cavity moulds. Also regions of extreme shearing, where particle segregation is likely to appear, can be identified at an early stage.

Modelling viscosity changes at low shear rates

A model recently integrated in SIGMASOFT® which captures the increase in viscosity at low shear rates has brought enormous benefits to the flow front predictability [1]. This particular viscosity increase is not only highly relevant for the packing phase, but also for the behaviour of the feedstock close to the mould walls where the shear rate is low, as seen in Fig. 1.

In comparison to thermoplastic materials, which show a plateau at low shear rates, the filling pattern is changed, as well as the position of weld lines and effects such as jetting are more likely to appear. The internal stress distribution is also different. All these effects call for the use of software able to capture PIM flow behaviour, instead of conventional software for thermoplastic moulding simulation.

Understanding the thermal interaction between mould and feedstock

Another important commercial use of SIGMASOFT® software has been in the selection of a tempering system. Because the thermal conductivity and the heat capacity of PIM feedstock can be one order of magnitude higher than those of thermoplastics, it is also necessary to use thermal models that are able to simulate the complex thermal interaction between mould and feedstock. A highly developed thermal solver originally developed for the simulation of foundry processes is able to calculate, at every time-step in the moulding cycle and at every location of the mould, the precise temperature, product of the complex heat balance between the hot feedstock releasing large amounts of energy into the mould, and the moulding system. It has for example been possible to determine in which cases air, water or even a copper insert should be used to remove heat from a particular hot-spot in a moulding application, or to identify which material alloys to select for the mould to improve cycle time.

Advanced modelling of powder-binder separation

SIGMASOFT® is investing significant development resources to predict the effect of powder-binder separation [2]. When flowing through narrow cross sections in the mould, feedstock may undergo high shear rates, which force particle segregation from the binder, as can be seen in Fig. 2. This undesired effect can lead to pores or differences in local density of the sintered part, which in extreme cases may lead to stress concentration and part failure. By using tracer particles the defects created by segregation can already be identified.

In 2013 a major breakthrough was brought to the market with a new technology, called SIGMASOFT® Virtual Molding. Besides predicting the flow and solidification behaviour of feedstock materials, it is now possible to reproduce the complete injection moulding process, with all its details over several cycles. The effect of several moulding parameters, such as the waiting time between cycles, the insert pre-heating temperature or the effect of after-moulding thermal treatments can be considered. This new technology, released at the K Show 2013, brings the accuracy of simulation to a complete new level and delivers a vast insight into the root causes of several effects found in the injection moulding process.

As a result of these developments it is possible to move ever closer to the needs of PIM industry. The integrated models are now able to reproduce with unprecedented accuracy the flow and thermal behaviour of feedstock so that mistakes can be avoided early in the design stage and an optimum solution minimising cost and time effort can be found.

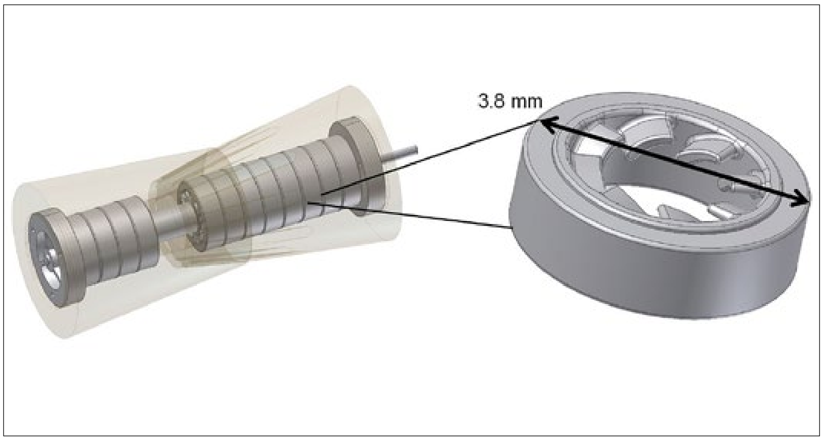

Fig. 4 Left: overall demonstration micro turbine used in the SFB 499. Right: turbine guide wheel [3]

Case study: Simulation delivers first-shot success in micro PIM

Despite being a highly profitable market with limited competition, micro moulding still remains somehow a "taboo" area for PIM companies. There are several reasons for this. On the one hand the handling, positioning and assembly of micro parts, as well as quality inspection, are challenging. On the other hand, micro-dimensions bring a completely new behaviour into the process. The tiny dimensions change the basic physical behaviour of the feedstock, and phenomena which do not have any relevance in conventional PIM suddenly have a major importance. Effects such as surface tension, increased surface-to-volume ratio or heat transfer in micro dimensions require a steep learning curve.

'When flowing through narrow cross sections in the mould, feedstock may undergo high shear rates, which force particle segregation'

The challenges of mould design for micro PIM

Moulds used for micro PIM need to fulfil special requirements in terms of quality and precision. A vario-thermal process control is required, and the cavities must be evacuated to provide the best preconditions to get a complete mould filling. Otherwise defects such as diesel-effect marks, or sink marks, can appear. The runner system and the inserts are crucial; insert manufacturing is expensive and time consuming, because the requested tolerances are often on the limits of conventional machining methods, such as HSC milling or micro-EDM, and sometimes special manufacturing methods such as X-ray or UV LIGA processes are required. These cost factors require careful mould design and make later iteration costly and risky.

Simulating injection moulding in micro dimensions

In recent years significant development efforts have been made, in parallel to our PIM activities, to improve the prediction of injection moulding simulation in micro dimensions. Micro dimensions create specific challenges for simulation

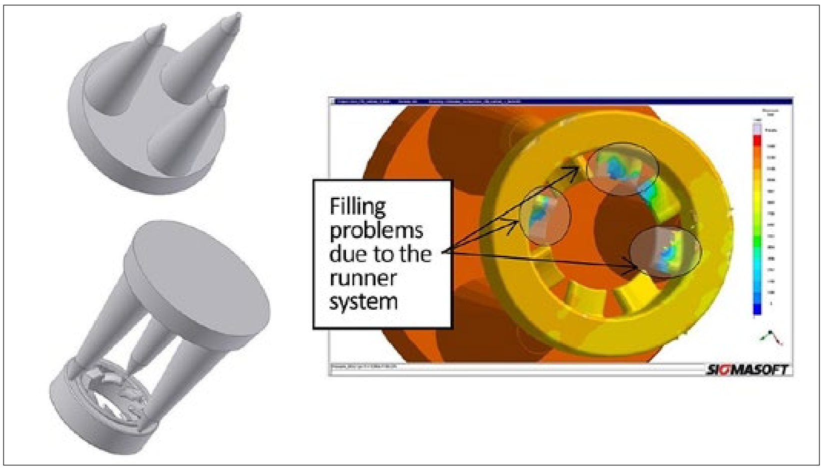

Fig. 5 Left: first design of the turbine guide wheel with three runner channels. Right: pressure result after 1.5 s filling time. Incomplete filling is seen [3]

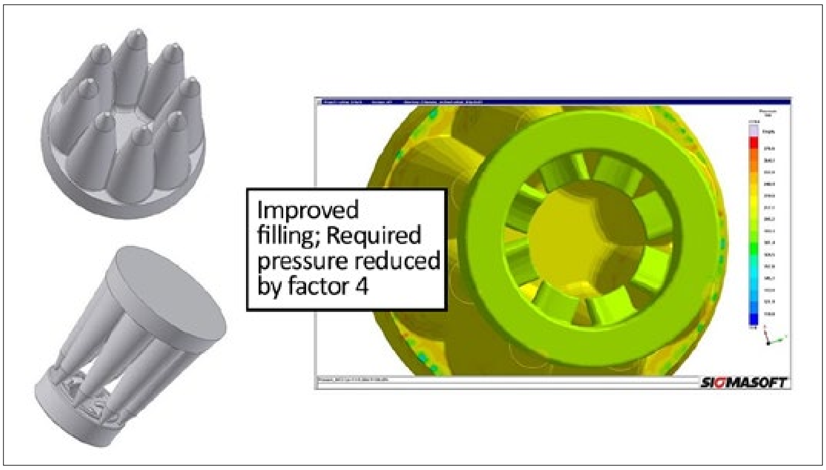

Fig. 6 Left: Improved runner design with eight channels. Right: pressure result after 1.5 s filling time. Complete filling is observed [3]

as the tiny geometrical dimensions can influence the numerical algorithms. A specific R&D project was undertaken to integrate and validate material models suitable for the modelling of micro-parts [4]. In Fig. 3, the two micro applications used to validate the research results are presented. The upper left image is an instrumented two cavity mould and the lower left image is a micro tensile test specimen mould. On the right side of Fig. 3 both measured and simulated pressure curves are presented for the two cavity mould. The shape of the pressure curves, the maximum level and the signal-lost point are well predicted by the simulation. Thus, it was proved that a predictive simulation tool is available for micro moulding applications. In combination with the PIM specific functionality, MIM and CIM micro-applications can therefore also now be simulated with using SIGMASOFT® software.

'Micro dimensions create specific challenges for simulation as the tiny geometrical dimensions can influence the numerical algorithms'

The development of a ZrO_2 micro turbine guide wheel

At the Karlsruhe Institute of Technology (KIT), Germany, a research project (SFB 499) was conducted dealing with the process chain for the development of highly stressed micro parts made out of ceramic and metallic alloys [3]. Within the project, a micro turbine guide wheel was moulded out of ZrO_2, as seen in Fig. 4. As a requirement, the free-form surfaces of the blades had to be free of defects to fulfil their function.

In this case simulation was used to validate the concept proposed for part moulding. In a first step, the feedstock, containing 50.5% ZrO_2 and a combination of polyethylene, paraffin wax and dispersant as binder, was characterised. The viscosity was measured using a high pressure capillary rheometer and with the same equipment the thermodynamic properties were studied, producing a PvT curve (pressure vs. specific volume and temperature). Beyond this, the thermal conductivity and specific heat capacity were measured.

The first tested concept considered three distribution channels, systematically arranged as seen in Fig. 5. The moulding parameters foreseen in the simulation were an injection time of 1.5 s, a holding pressure time of 1.6 s and cooling time of 15 s. The simulation showed an incomplete filling, high injection pressure and the presence of weld lines in functional areas over the turbine blades.

After these defects were observed, a new design was proposed: the number of runner channels was raised to eight and all edges were rounded to improve the flow conditions of the melted material, as can be seen in Fig. 6. Each runner channel was connected to the turbine guide wheel in the middle of each turbine blade to avoid weld lines in functional areas of the final part. The production parameters used in the simulation were the same as in the first simulation.

The results of this second iteration showed a significantly improved picture. The required moulding pressure was considerably lower, and weld lines were placed in non-functional areas of the part (between the turbine blades). In summary, the design of the optimised runner system promised good pre-conditions for the later replication of the green parts, and was used for the fabrication of the mould in steel.

The moulded ZrO_2 micro turbine guide wheel green parts showed a well shaped outer contour without any visible defects (Fig. 7). The mould with the optimised mould inserts and runner system provided a stable replication process suitable for mass production without additional iteration needed on the steel mould. The sintered parts had a theoretical density of over 99%, without the presence of shrink holes, burrs or chip-offs and the linear shrinkage was around 21%. This first-shot success was based on, amongst other factors, the use of injection moulding simulation at the mould design stage and on the rigorous characterisation of the feedstock data.

Motivated by the success achieved, a new micro PIM project was proposed at the KIT. A design concept was created and optimised, based on the described simulation approach. After the feasibility of the production process was demonstrated through simulation, the virtual results were used to apply for the necessary funding. The budget was awarded, the mould built and the next first-shot success achieved.

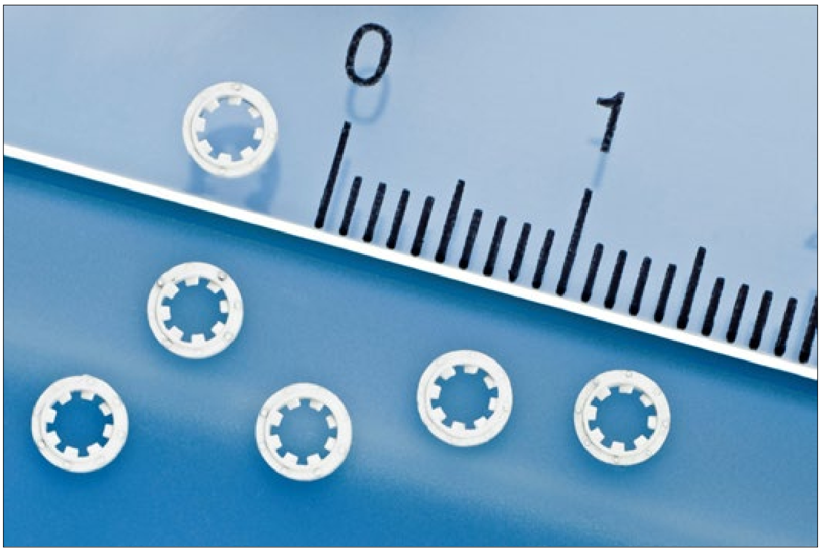

Fig. 7 Three-plate mould built following simulation recommendations and green parts produced [3]

Summary

PIM has become a key production technology for the manufacturing of metal and ceramic micro parts. The moulding stage determines part quality, as defects induced here cannot be healed later on. Furthermore, in micro PIM the costly mould manufacturing methods do not make iteration on steel mould viable. These arguments make simulation an important and profitable tool to be applied early in the design stage.

SIGMASOFT®'s software, developed specifically to serve the interests of PIM industry, involves several features and material models which dramatically improve reliability and accuracy of the flow and thermal behaviour predicted. Even in micro-moulding applications, simulation has proved to be a useful tool up to the point of delivering first-shot success.

Simulation has to be understood as a valuable tool and has to be well-integrated into the part and mould design process. Only then will the expected success potential for micro PIM actually be achieved.

Acknowledgements

The micro-moulding project was carried out with the support of the Karlsruhe Nano Micro Facility (KNMF, www.kit.edu/knmf), a Helmholtz Research Infrastructure at Karlsruhe Institute of Technology (KIT, www.kit.edu). The funding of this work within Collaborative Research Center SFB 499 by Deutsche Forschungsgemeinschaft (DFG) is gratefully acknowledged. Thanks also to the colleagues in the SFB 499, especially Markus Welz and Klaus Plewa for their work.

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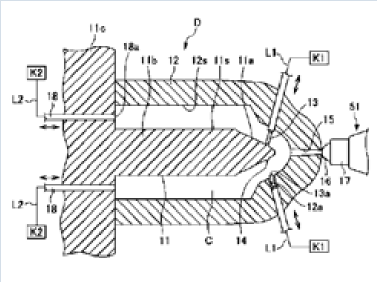
Global PIM Patents

The following abstracts of PIM-related patents have been derived from the European Patent Organisation databases of patents from throughout the world.

JP2011099406 (A)
METHOD OF MANUFACTURING FUEL INJECTOR AND NOZZLE SINTERED BODY
Publication date: 2011-05-19
Inventor(s): Yamagishi Hiroaki, Honda Motor Co Ltd, Japan

The problem to be solved with this patent is to provide a method of manufacturing a fuel injector capable of promptly and reliably forming fine fuel injection ports at the front end of a metallic nozzle without the need for expensive facilities.

The method of manufacturing the fuel injector includes a pin inserting step. Protruding sliding pins, having diameters, numbers, positions and angles corresponding to the fine fuel injection ports to be formed at the front end of the nozzle are inserted from an injection moulding die



into a cavity corresponding to the shape of the nozzle to be formed. After mould closure an injection moulding step of pressing and filling binder-added powder metal into the cavity is performed.

The sliding pins are moved back to the injection moulding die. The nozzle moulding is removed from the injection moulding die and then sintered to manufacture a nozzle sintered body with slender tubular voids formed by the sliding pins.

KR20110125549 (A)
HEAT RESISTANT STEEL ARTICLES AND METHOD FOR PREPARING THE SAME
Publication date: 2011-11-21
Inventor(s): Song Jun Ho et al., PIM Korea Co Ltd, Korea

A Metal Injecting Moulding (MIM) method of manufacturing a heat-resistant steel part in order to enhance erosion resistance, hardness, wear resistance, and durability under a high temperature is discussed in this patent. The heat-resistant steel part is formed from Cr-Ni-Nb heat-resistant steel and has a surface hardness of over 180VHN.

DE102010028432 (A1)
MANUFACTURING A SPATIAL IMPLANT STRUCTURE FROM PURE TITANIUM
Publication date: 2011-11-03
Inventor(s): Grundei Hans Dr Ing, S & G Implants GmbH, Germany

The patent describes a method of manufacturing a spatial implant structure made of pure titanium or titanium alloy with an open-mesh, three-dimensional network structure having mesh sizes of 1.5-6 mm partially covering the surface. The process comprises of producing a green compact via metal Powder Injection Moulding, transforming the green compact into a brown body by debinding, holding the brown body on a solid implant base body made of pure titanium or titanium alloys, and producing the finished implant from the solid implant base body by sintering. The surface of the implant is sprayed with titanium plasma, where the layer thickness of the titanium coating is 1-10 mu m.

DE102009046003 (A1)
METHOD FOR PRODUCING A COMPONENT WITH A CERAMIC FIRST AREA AND A METALLIC SECOND AREA
Publication date: 2011-04-28
Inventor(s): Dirscherl Florian, Robert Bosch GmbH, Germany

This patent describes the method for producing a component with a ceramic first area and a metallic second area, where a ceramic material is present in the first area and a metallic material is present in the second area. The process comprises of applying the ceramic material in a first tool area of a tool by means of a first injection moulding step. The metallic material is applied in a second tool area of the tool by means of a second injection moulding step.

Laser radiation and microwave radiation is used to heat the tool to 1200-1500°C. The tool is heated in such a way that the ceramic material is connected with the metallic materials.

The structure and performance of WC-10%Co Cemented Carbide inserts made by Powder Injection Moulding

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[3] 3 Central South University, Changsha, Hunan ,410083, China

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Powder Injection Moulding (PIM) is a very important Powder Metallurgy (PM) forming method that can meet the precision requirements for the manufacture of complex shaped cutting tool inserts. PIM processing imparts less deformation and is suitable for the manufacture of complex shaped parts. This paper investigates the structure and performance of WC-10%Co cemented carbide cutting tool inserts made by PIM. The performance of PIM products is affected by both the binder and the debinding process during manufacturing. By adding a special polymer binder, which comprised PW, oil, DOP, HDPE, EVA etc., mixed together with powder, inserts were manufactured by injection moulding. The solvent and thermal debinding processes were analysed. By adapting and optimising the debinding and sintering processes, high quality inserts were produced. Cutting test results indicate that the lifetime of the inserts after Physical Vapour Deposition (PVD) coating is better for those made by PIM than those made by conventional die compaction.

With the development of high performance cutting technology, cemented carbide cutting tool inserts are becoming ever more complex as tolerance requirements increase [1, 2]. Die compaction technology cannot meet the requirements of complex inserts because of the non-uniformity of the density. Powder Injection Moulding (PIM), which is derived from plastic injection moulding, is a net-shape forming process that combines high part complexity with high production quantities. The products prepared by PIM are expected to have a more homogeneous microstructure since hydraulic pressure is applied during injection moulding and the mould is filled uniformly, which avoids the density gradients experienced in conventional press/sinter processing [3]. It is important to control the solid load ratio of the binder because of the high WC powder density. During the production of inserts by PIM, it is important to reduce product defects, improve accuracy and control carbon content [4, 5]. Additionally, cemented carbide is sensitive to the binder and the debinding process. By investigating the ultra-fine (sub-micron) WC–10%Co insert manufacturing process, this paper summarises some critical results by investigating the application of PIM for the production of ultra-fine sub- micron cemented carbides.

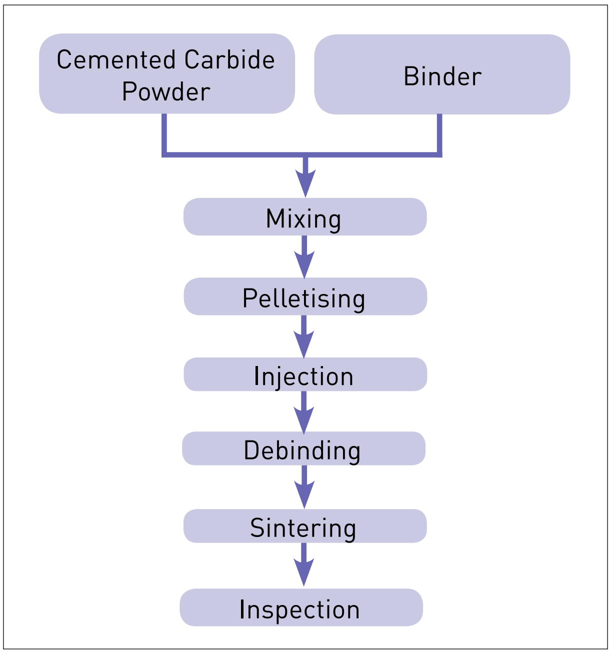

Fig. 1 The PIM process

Component	Chemical structure	Melting temperature T_m(°C)	density ρ(g/cm^3)	Content %
2 paraffin	C_nH_{2n+2}	54~80		50
EVA	— $[CH\text{-}CH_2]\text{-}[CH\text{-}CH_2]_y]_n$ — | $COCH_3$	90	0.96	10
HDPE	— $[CH\text{-}CH]_n$ —	140	0.98	12
SA	$CH_3[CH_2]_{16}COOH$	66	0.96	2~3
DOP	$C_6H_4[COOH_2[CH_2]_6CH_3]_2$	18	0.97	2~3
OA	—	0~5	0.917	1~2

Table 1 Binder components

Experiment

Materials

The WC–10%Co powder mixture used in the experiment was supplied by Zhuzhou Cemented Carbide Works. The characteristics of the WC powder are 0.79 µm Fisher particle size and that of the Co powder is 1.23 µm. 0.5% TaC (1.2 µm) was also added.

The binder components comprised PW (Paraffin Wax), oil, DOP (Dioctyl-Phthalate), HDPE (High-Density Polyethylene), EVA (Ethylene Vinyl Acetate) etc., and this binder is then mixed together with the powder. The contents and properties of each binder component are listed in Table 1.

Experimental procedure

The binder ingredients were melted in a special device at 150°C for 1 h. Then, the WC–10%Co powder mixture was blended with the binder in a SHR-10A mixer at 140°C for 2 h. The inserts were then injection moulded in a HTF58x2 injection moulding machine at 140°C at an injection pressure of 55 MPa. After this, a solvent debinding step was performed, followed by a thermal debinding step. Sintering took place at 1410°C for 1 h in a COD633 furnace. Fig. 1 shows the PIM manufacturing process.

Property measurements

An SEM JSM-5600LV was used to observe the cross-sectional microstructures of the green PIM insert, the insert after solvent debinding and the sintered insert. The mechanical properties of the specimens were determined using an Instron material tester and a HV hardness tester. The magnetic performance was measured by ZS-1 Co-magnetic and coercimeter 93-IE. The thermal performance of EVA and HDPE was analysed by DSC. Cutting tests were made by machining and the machined material was 0.45% carbon steel.

Results and discussion

Manufacture of the PIM inserts

Because of the high density of the cemented carbide powder and the selection of a finer WC particle size for the WC-10%Co material used in the experiment, the solid powder loading rate cannot be set at a high value [6][7]. In order to achieve ideal rheological performance and shape retention, the EVA and DOP components in the binder were increased and HDPE was decreased. The solid load ratio selected was 55%. By adopting a suitable mixing process, the inserts ZTBD02002-MG were manufactured using the WC-10%Co feedstock. The quality uniformity of the finished PIM insert is related to the uniformity at the time of injection. During the injection moulding process, the quality uniformity can be checked by the deviation of the weight of the inserts [8]. Table 2 shows the insert weight data.

The weight deviation is between 1.754 g and 1.783 g, with the percentage of the weight deviation being 0.3%. The tolerance is 0.004 g, which can meet the accuracy requirement of 0.1%

Fig. 2 Insert ZTBD02002-MG

1.782	1.780	1.781	1.782	1.779	1.779	1.780	1.780	1.782	1.783
1.782	1.783	1.783	1.779	1.782	1.779	1.781	1.783	1.780	1.780
1.783	1.779	1.780	1.780	1.780	1.781	1.780	1.780	1.782	1.783
1.783	1.783	1.782	1.782	1.782	1.779	1.780	1.781	1.780	1.781
1.783	1.780	1.780	1.782	1.780	1.782	1.781	1.780	1.781	1.782
1.781	1.782	1.782	1.779	1.782	1.780	1.781	1.781	1.779	1.779
1.783	1.780	1.780	1.782	1.780	1.782	1.781	1.780	1.781	1.782
1.781	1.782	1.782	1.779	1.782	1.780	1.781	1.781	1.779	1.779
1.782	1.783	1.783	1.779	1.782	1.779	1.781	1.783	1.780	1.780
1.783	1.779	1.780	1.780	1.780	1.781	1.780	1.780	1.782	1.783
1.783	1.783	1.782	1.782	1.782	1.779	1.780	1.781	1.780	1.781
1.782	1.780	1.781	1.782	1.779	1.779	1.780	1.780	1.782	1.783

Table 2 Insert weight data (g)

tolerance on dimension. The injection moulding machine and mould is of course a factor in achieving stable insert weight; however, the flowability and shape retention of the mixing powder is critical. Fig. 2 shows images of the insert ZTBD02002-MG. From this picture, it can be seen that there are no cracks or other defects.

Analysis of the debinding process

Solvent debinding process

After the green inserts were produced, solvent debinding, which is critical for the quality of the inserts, took place. During solvent debinding, the soluble component of the binder is dissolved in the solvent solution. The binder component paraffin dissolved when the solvent was heated to around 80°C. Running this process too quickly or with higher temperatures will cause defects. Porosity will form from the surface to the middle during solvent debinding and the pores that are created become connected. Fig. 3 shows the structure of the inserts during different solvent debinding stages. Normally, the dissolved component should be desorbed almost entirely during solvent debinding. Correct selection of solvent debinding temperature and time will contribute to quality uniformity.

As can be seen from Fig. 3, by adopting the experimental process, there are no defects in the solvent debound insert and the porosity distribution is uniform and this will contribute to good thermal debinding.

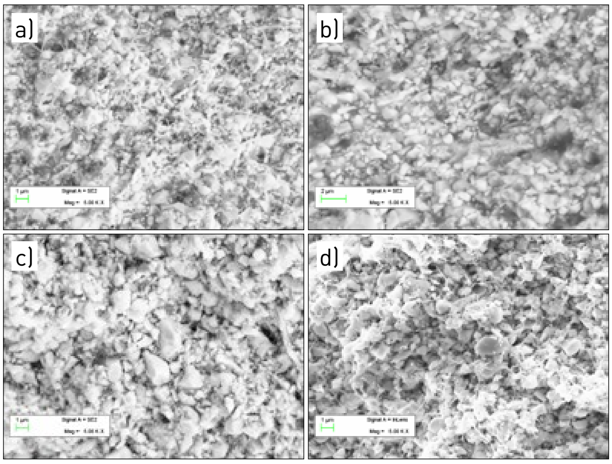

Fig. 3 SEM morphology of different debinding times (a: before debinding, b: 50 min debinding, c: 100 min debinding, d: 240 min debinding)

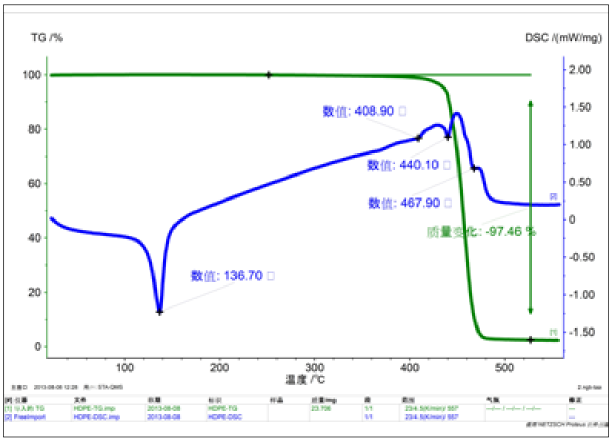

Fig. 4 HDPE TG-DSC curve

Thermal decomposition of undissolved binder material

After the low molecular weight component of the binder system has been removed, the remaining binder in the cemented carbide will decompose prior to sintering. Heating up rate and time are related to the heat characteristic of the polymer binder. By DSC analysis, we can obtain the data of the polymer heat up. Figs. 4 and 5 show two polymer TG-DSC curves. The curves show that HDPE and EVA will melt at 136°C and 92°C separately. HDPE will start to decompose at 408.9°C, strongly decompose in the temperature range of 440-468°C and decomposition will end at 467.9°C. EVA will start to decompose at 300°C, strongly decompose in the temperature range of 416-460°C and decomposition will end at 480°C.

Based on the thermal characteristics of EVA and HDPE, three debinding and sintering cycles were designed as follows:

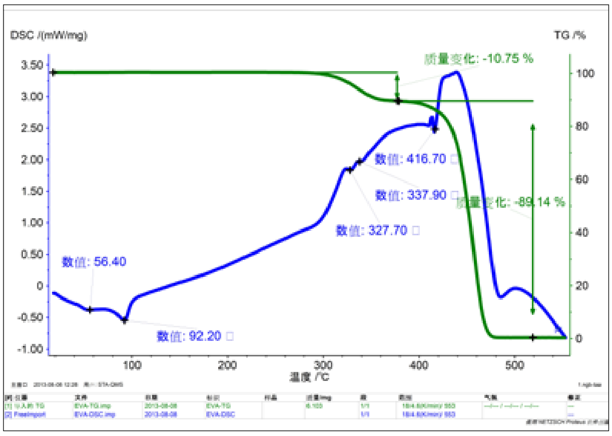

Fig. 5 EVA TG-DSC curve

Cycle 1: From room temperature the furnace is heated to 350°C at a speed of 2°C/min followed by a holding time of 60 min, then heated to 450°C at a speed of 3°C/min followed by a further holding time of 60 min. Finally the furnace is heated to 1410°C at a speed of 10°C/min for a sintering period of 60 min in an argon atmosphere of 40 bar pressure. Up to the 450°C hold, a H_2 flow rate of 50 slm and a pressure of 1020-1030 mbar are used.

Cycle 2: From room temperature the furnace is heated to 320°C followed by a holding time of 120 min, then to 380°C followed by a holding time of 60 min, and then to 450°C at a speed of 2°C/min followed by a holding time of 60 min. Finally the furnace is heated to 1410°C at a speed of 10°C/min, for a sintering period of 60 min in an argon atmosphere of 40 bar pressure. Up to the 450°C hold, a H_2 flow rate of 50 slm and a pressure of 1020-1030 mbar are used.

Cycle 3: From room temperature the furnace is heated to 380°C at a speed of 2°C/min followed by a holding time of 180 min, then heated to 450°C at a speed of 2°C/min followed by a holding time of 120 min. It is then heated to 1410°C at a speed of 10°C/min for a sintering period of 60 min in an argon atmosphere of 40 bar pressure. Up to the 450°C hold, a H_2 flow rate of 50 slm and a pressure of 1020-1030 mbar are used.

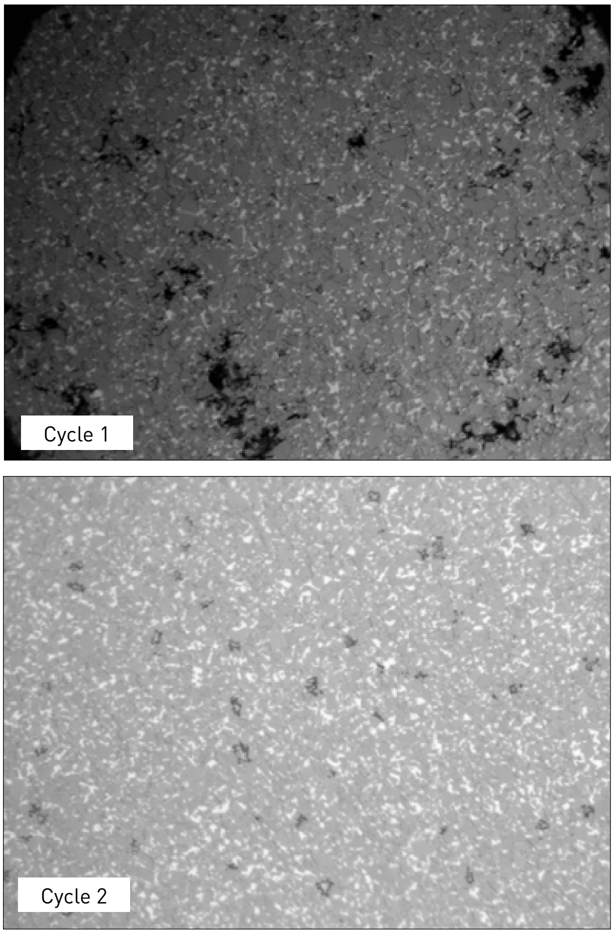

Fig. 6 Metallographic images of the WC-10% inserts produced using cycles 1 and 2

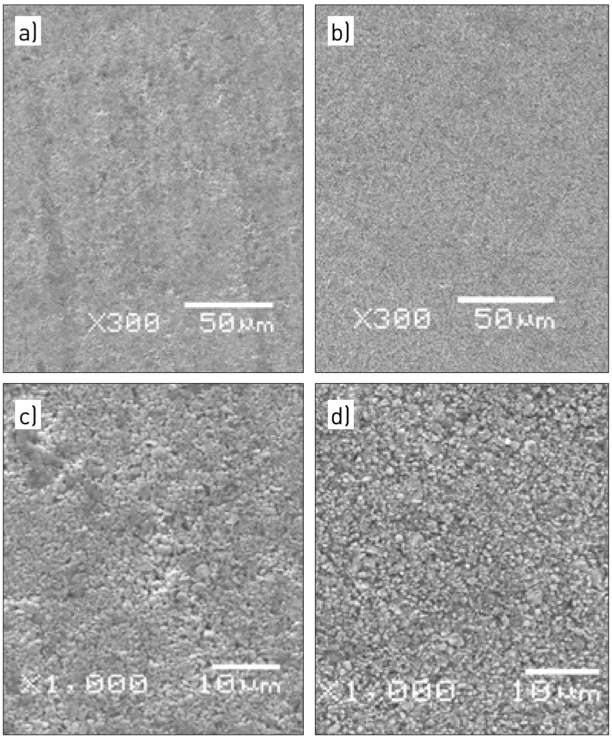

Fig. 7 The surface morphology of die compacted and PIM inserts (a and c are die compacted sintering inserts; b, d are PIM inserts)

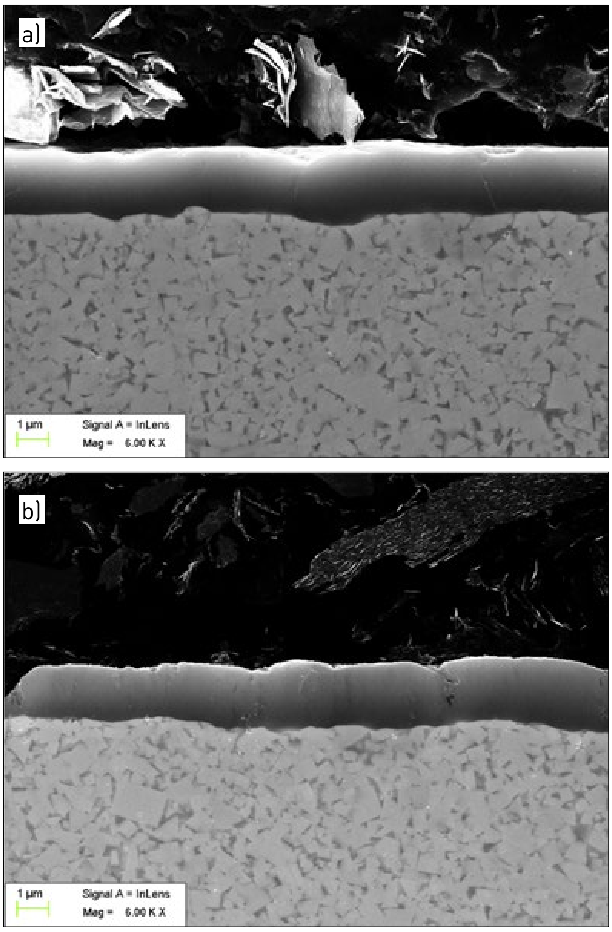

Fig. 8 SEM morphology photo of AlTiN coated inserts (a: PIM, b: Die compacted)

Method	D (g/cm^3)	Com (%)	Hc (KA/m)	Hv	TRS (N/mm)	Metallography
Die compacted	14.44	8.9	22.8	1670	2240	A02B00C00E00
PIM cycle 1	14.42	10	21.3	1630	1980	A02B00C06E00
PIM cycle 2	14.45	9.0	22.6	1660	2320	A02B00C00E00
PIM cycle 3	14.45	8.9	23.1	1670	2280	A02B00C00E00

Table 3 Physical and mechanical performances of WC-10%Co debinding and sintering process 1, 2 and 3 plus a conventional die compaction process

Through these three different debinding and sintering cycles, it was shown that there is a carbon phase in the inserts produced using the first cycle. The structure is normal in the second cycle as shown in Fig. 6. The physical and mechanical performance is showed in Table 3.

From Fig. 6 it is shown that the low debinding temperature and short debinding time will cause debinding deficiencies and there is carburisation in the cemented carbide. On the other hand, in cycle 2, with appropriate debinding temperature and sufficient time, the binder can decompose adequately. Although the holding time of cycle 3 is longer than that of cycle 2, the final performance varies little as shown in Table 3. This indicates that the debinding time in cycle 2 is long enough.

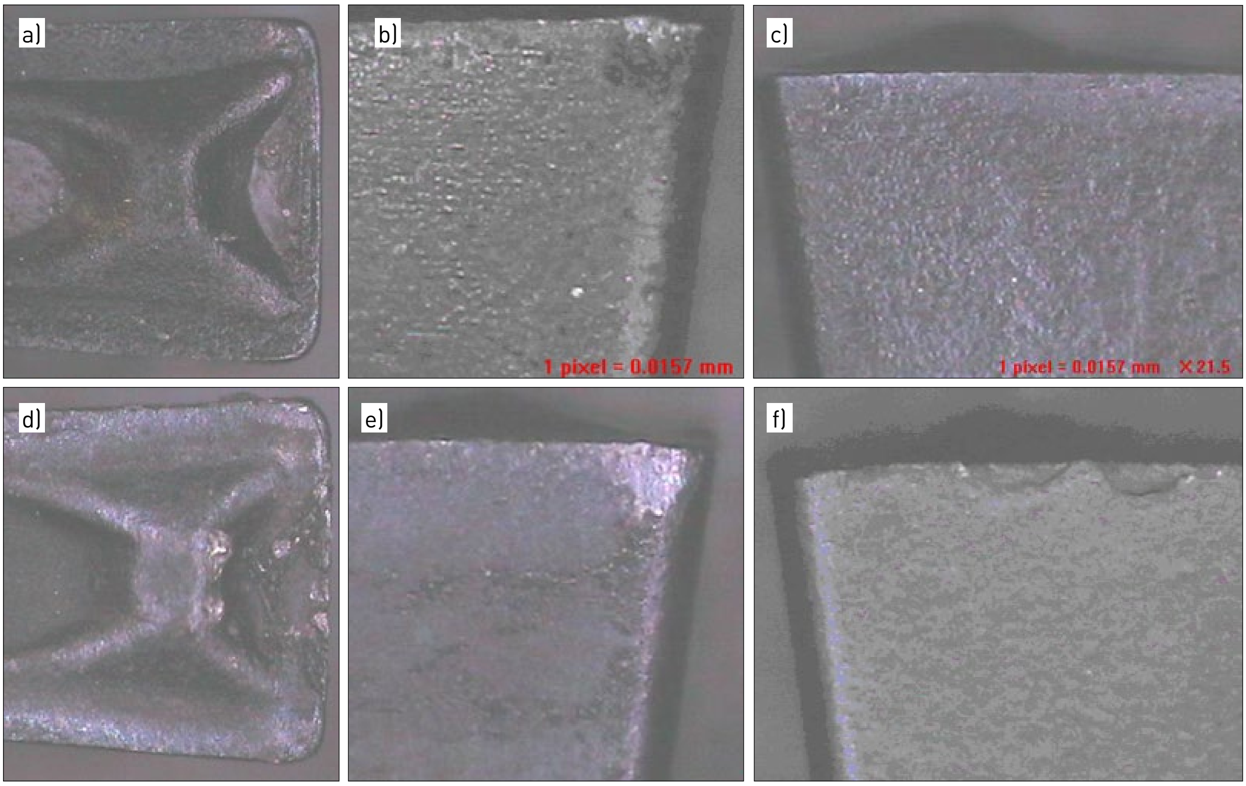

Fig. 9 Wear morphology of inserts after 30 grooves (a, b and c: PIM inserts; d,e and f die compacted inserts)

Considering the forming method, a surface quality comparison was made. The surface structure of the PIM inserts is more uniform and flat. The roughness values of the PIM insert and die compacted inserts are Ra0.346 and Ra0.345 respectively. Fig. 7 shows the surface structure.

From the images in Fig. 7, we can see that the surface of the PIM insert is more uniform than that of the die compacted insert. After confirming the necessary PIM production process, the inserts ZTBD02002-MG were made by both PIM and die compaction. The dimensions of the insert ZTBD02002-MG were measured. The 3-dimensional shrinkage rates of the die compacted inserts are 18.37% (length), 18.03% (width) and 17.36% (height). The 3-dimensional shrinkage rates of the PIM inserts are 18.55% (length), 18.53% (width) and 18.85% (height). The shrinkage rate deviation of the PIM products is 0.3% while that of the die moulding inserts is 1%. The PIM inserts ZTBD02002-MG are therefore more accurate than the die compacted inserts.

Inserts cutting test

Groove cutting tests were undertaken with both the PIM and die compacted inserts. The inserts were coated with AlTiN. SEM images of the coated inserts are shown in Fig 8. These images show that the inner structures of the PIM inserts and the die compacted inserts are very similar to each other. Because the blank surface of the PIM inserts is more uniform, the joint between the substrate and AlTiN coating is better for the PIM inserts.

The machined material is a 0.45% carbon steel. The cutting parameters are: cutting speed 120 m/min, the depth of cut 3 mm and the feed 0.1 mm/r. After 30 grooving tests, Fig. 9 shows that the flank wear of PIM YBG202/ZTBD02002-MG is lower than that of the die compacted inserts, the flank wear of PIM inserts is 0.15 μm, while the flank wear of the die compacted inserts is 0.26 μm. Fig. 10 shows that the flank wear value of PIM inserts after machining 100 grooves is equal to that of the die moulded inserts after machining 60 grooves.

The cutting lifetime of PIM inserts is therefore longer than that of die compacted inserts. One of the reasons for this is that the uniform density of the PIM inserts results in higher accuracy of the complex inserts and less deformation. Secondly, compared with die compaction, there is less friction in the case of the PIM process, so this improves the cutting edge quality compared to the die compacted inserts. Fig. 10 shows that there are micro-breaks in the cutting edge of the die compacted inserts compared with the PIM inserts. Thirdly, by adapting the binder and debinding process, the mechanical performance of the ultrafine 10%Co-WC cemented carbide inserts is the same as that of the die compacted inserts.

Conclusion

By adopting a binder comprising PW, oil, DOP, HDPE, EVA etc. and mixing together with the ultrafine powder WC-10%Co, ZTBD02002-MG inserts were made via Powder Injection Moulding. Because of the high specific surface of the fine WC powder, the solid load of the binder is approximately 55%. Although this is a relatively low solid loading, the weight of the green inserts is very stable and there is good shape retention.

After sintering, there is more uniform shrinkage rate in the three orthogonal directions compared with the die compacted inserts.

Using the TG-DSC method, the thermal characteristics of HDPE and EVA were investigated and appropriate debinding and sintering processes were designed. Compared with the die

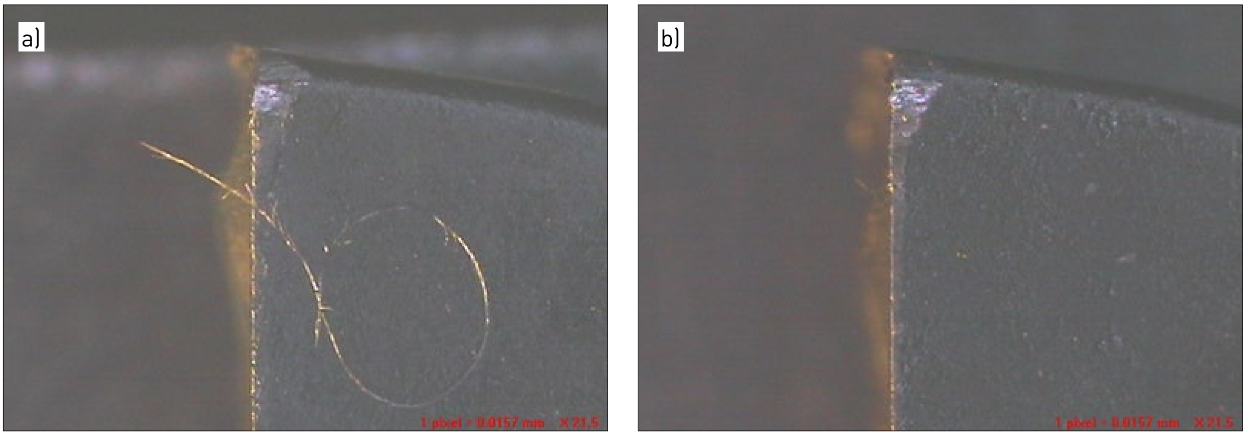

Fig. 10 a) wear morphology of PIM inserts after 100 grooves b) wear morphology of PM die compacted inserts after 60 grooves

compacted inserts, there is similar mechanical performance, morphology structure and surface roughness. The surface structure of the PIM inserts is more uniform than that of the die compacted equivalents. For complex shape inserts, there is higher accuracy when using the PIM process, because there is a more uniform density distribution.

An AlTiN coating was applied to the WC-10%Co/ZTBD02002-MG inserts and cutting tests were undertaken. The results show that the lifetime of the PIM inserts is longer than that of die compacted inserts.

Acknowledgements

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