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FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

# POWDER INJECTION MOULDING INTERNATIONAL

## in this issue



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**Publisher & editorial offices**
Inovar Communications Ltd
2 The Rural Enterprise Centre
Battlefield Enterprise Park
Shrewsbury SY1 3FE, United Kingdom
Tel: +44 (0)1743 454990
Fax: +44 (0)1743 469909
Email: info@inovar-communications.com
Web: www.inovar-communications.com

**Managing Director and Editor**
Nick Williams
Tel: +44 (0)1743 454991
Fax: +44 (0)1743 469909
Email: nick@inovar-communications.com

**Publishing Director**
Paul Whittaker
Tel: +44 (0)1743 454992
Fax: +44 (0)1743 469909
Email: paul@inovar-communications.com

**Consulting Editors**
Professor Randall M. German
*Associate Dean of Engineering, Professor of Mechanical Engineering, San Diego State University, USA*

Dr Yoshiyuki Kato
*Kato Professional Engineer Office, Yokahama, Japan*

Dr Professor Frank Petzoldt
*Deputy Director, Fraunhofer IFAM, Bremen, Germany*

Bernard Williams
*Consultant, Shrewsbury, UK*

**Advertising**
Jon Craxford, Advertising Manager
Tel: +44 (0) 207 1939 749
Fax: +44 (0) 1242 291 482
E-mail: jon@inovar-communications.com

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POWDER INJECTION MOULDING INTERNATIONAL

For the metal, ceramic and carbide injection moulding industries


# Investments in capacity support positive industry outlook

Welcome to the September issue of *PIM International*, our biggest issue to-date. We are extremely grateful to all who have contributed their time and expertise in preparing the material for this issue, as well as the record number of truly global advertisers.

This issue sees the publication of the first part of our two part report on the current status of MicroPIM. This technology is forecast to enjoy significant growth in the coming years as the market for microsize components grows. MicroPIM isn't the only microforming process available, but for high performance applications that require the superior properties of metal and ceramic materials, it is the only viable processing route (page 27).

We also report on Indo-MIM, a producer based in Bangalore that today has the world's largest reported capacity MIM operation. The speed of the company's growth and its willingness to continually reinvest in production capacity is a clear reflection of the current confidence surrounding MIM technology (page 39).

Underlying concerns about powder supply have been one of the few black clouds on MIM's horizon. These fears are now being addressed. Sandvik Osprey's welcome announcement in this issue that a new large capacity atomiser will be installed by Q2 2012 (page 67) will bring relief to many MIM producers. Interest in MIM's powder supply concerns has, however, been attracting the attention of others outside of the industry. In this issue a major iron powder producer for press and sinter components publishes a technical paper outlining an alternative approach to enable the use of coarser low alloyed materials for MIM production (page 78).

We also publish a paper that presents Bulk Metallic Glass (BMG) technology, asking the question whether this process should be regarded as a competing or complementary technology to MIM. With reported interest in the process from major MIM users such as Apple and Swatch, a greater understanding of this process, its limitations as well as its advantages, may be of value to many MIM producers (page 83).

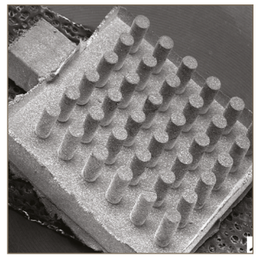
Nick Williams
Managing Director and Editor

**Cover image**
*Furnace at Indo-US MIM Tec (P) Ltd.*

September 2011

Powder Injection Moulding International





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# Technical papers



# Regular features

# Industry News

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

## International conference to focus on the powder processing of titanium

Five materials societies including The Minerals, Metals and Materials Society (TMS), Japan Association of Powder and Powder Metallurgy (JSPM), Titanium Industry Development Association (TiDA), New Zealand, Chinese Society of Metals (CSM), and Materials Australia (MA) are co-sponsoring an international conference on "Powder Processing, Consolidation and Metallurgy of Titanium".

The event will take place from 5-7 December 2011, in Brisbane, Australia and promises to be one of the most important conferences on PM titanium in recent years. The objectives of the conference are to provide an international forum to review recent progress in both the fundamental sciences and the applications of PM Titanium, as well as to discuss future directions.

The scope of the conference includes Ti powder production and processing; Ti powder consolidation by various processes; PM Ti alloy development; Ti injection moulding; additive manufacturing from Ti powder by, for example, cold spray, electron beam or laser melting; Phase transformation phenomena in Ti alloys during heating, isothermal holding and cooling; and PM Ti applications.

The deadline for abstract submissions is 30 September 2011. For more information visit the event website.

www.materialsaustralia.com.au

## SAE publishes new standard for MIM superalloy parts

SAE International published AMS5917 Standard on 'Metal Injected Molded Nickel-based Alloy 718 Parts, Hot Isostatically Pressed, Solution Hardened and Aged' on August 16, 2011.

The new standard was developed by the AmsF Corrosion and Heat Resistant Alloys Committee of SAE as part of the funded programme supported by the U.S. Government's Metals Affordability initiative. This programme helped to characterise the MIM superalloy material and develop IN718 MIM products. SAE states that aero engine producers, General Electric and Honeywell, are intended users of the new standard.

www.sae.org/standards



## US MIM producers upgrading facilities

Two North American MIM producers, Advanced Powder Products (APP) and Smith Metal Products (SMP), are investing in new equipment and manufacturing facilities to meet increasing demand for MIM parts. Advanced Powder Products, based in Pittsburgh, PA., has secured loans totalling $400,000 to purchase a new sintering furnace that will double its sintering capacity, and has recently upgraded its Smartscope inspection capabilities as well as reorganising its moulding floor to allow for further expansion. APP has also installed new epoxy floors to improve the cleanliness in sintering and compounding areas of the plant. The company employs 20 full time and 15 part time employees.

Smith Metal Products, a division of Plastics Products Co., is expected to break ground by the end of this summer on a $3.5 million 60,000 ft$^2$ MIM plant in Center City, New Richmond, WI. SMP was said to be running out of room to expand at its 29,000 ft$^2$ plant in downtown Lindstrom.

SMP General Manager Lori Bjork, daughter of Plastics Products president/owner Marlene Messin, stated that she hoped to have SMP's 25 workers moved to the Center City plant by next summer – in time for a newly purchased 100ft long sintering furnace to arrive from Germany.

www.advancedpowderproducts.com
www.mimparts.com

## Fraunhofer IFAM Dresden signs agreement to help develop New Zealand's PM titanium industry

Professor Bernd Kieback, Director of the Dresden Branch of the Fraunhofer Institute for Manufacturing Technology and Advanced Materials (IFAM), and also director of the Dresden Technical University's Institute of Materials Science, has signed a collaboration agreement with the New Zealand Titanium Industry Development Association (TiDA) during a recent visit to the Bay of Plenty Polytechnic. The agreement will help the TiDA exploit applications for the fine titanium alloy powder now being produced commercially by Titanox Development's new plant in Mount Mauganui.

According to a report in the Bay of Plenty Times, Professor Kieback stated that the TiDA had made a good start in the exploitation of the new Ti alloy powder by forming a strong partnership between academia and engineers. He said during a PM symposium organised at the Bay of Plenty Polytechnic that whilst PM Ti alloy products could be used in industrial, aerospace, automotive and medical sectors, he would advise the TiDA to target the medical market first.

Kieback was impressed by the quality of the Titanox titanium alloy powder. "What is fascinating is that the powder has a small particle size and it has the right properties to go into the new technologies such as medical appliances," he commented. Kieback cited artificial hips, knee joints, bone screws and plates, heart valves, pacemakers, orthodontic brackets and surgical devices as examples of potential PM titanium products.

Fine titanium alloy powder is currently being produced by Titanox Development at a rate of 20 kg/day, or 5 tonnes/year.

www.tu-dresden.de
www.tida.co.nz

## MIM 2012 Call for Papers

The program committee for MIM2012, the International Conference on Injection Molding of Metals, Ceramics and Carbides has issued a Call for Presentations. The focus of the technical program is "Designing MIM Parts and Materials for Performance and Value." Sponsored by the Metal Injection Molding Association, a trade association of the Metal Powder Industries Federation, the conference will be held in San Diego, March 19–21, 2012, at the Sheraton San Diego Hotel & Marina.

Technical program co-chairmen Bruce Dionne, General Manager, Megamet Solid Metals Inc., and Toby Tingskog, Regional Sales Manager-NAFTA, Sandvik Osprey Ltd., request abstracts of 100–150 words, covering any aspect of metal injection moulding including processing, materials, and applications. The program committee requests that abstracts be submitted by September 30, 2011.

www.mimaweb.org

## Phonak notches up success with CIM hearing aid housing

The hearing instrument industry has put significant effort into developing housings with stylish designs using advanced materials to produce ergonomic shapes and functions, as was recently publicised in *PIM International* (March 2011, page 9). Now the Swiss hearing instrument producer Phonak AG, based in Stäfa near Zürich, Switzerland, has reported the successful use of ceramic injection moulding to manufacture a new device which not only has aesthetic appeal, but which also offers extreme hardness and mechanical strength, excellent biocompatibility and thermal shock resistance.

Designated Audéo S SMART High-Tech Ceramic, the polished CIM zirconia housings ensure a scratch-free shine which lasts for years. They also offer more comfort to the user in that the housing quickly reaches and maintains body temperature thereby preventing perspiration behind the ear. The acoustic performance is said to be equal to that of conventional hearing aids with polymer housings.

"With our new high-tech ceramic housing, Phonak once again introduces a significant innovation in the industry," said Valentin Chapero, CEO of Phonak. Stefan Launet, Director of Research and Technology at the company added, "Modern hearing instruments are little high-tech wonders as they integrate the latest chip technology enabling a performance comparable to a PC. The design of this new high-tech ceramic housing now truly reflects the high value of our technology".

The production of the deep-black zirconia housing by powder injection moulding was a real challenge for Phonak. The company uses zirconia powder supplied by Kyocera Corp. for the CIM feedstock which is injection moulded at high pressure. The relation of powder volume to binder content, injection moulding pressure, heat distribution in the mould, and several other parameters play a crucial role in achieving the defined dimensions and mechanical properties of the product. After sintering, during which the component undergoes a shrinkage of 20-30%, the zirconia housings are ground and polished to achieve the smooth, glossy surface. The Audéo S SMART High-Tech Ceramic housings are finally given a special invisible hydrophobic (water-repellent) and wear resistant coating that was developed specifically to make the housings resistant against moisture.

www.phonak.com


*A Phonak hearing aid with ceramic injection moulded housing*

# Quick-cycle HIPs strengthen MIM/PIM parts at lower cost

Today, tens of thousands of structural parts are produced by powder injection moulding (PIM) technology. Many of these are small components for electronic, medical, telecommunications and computing devices. Because of their small load-bearing areas, they require particularly high levels of tensile strength and hardness.

PIM compacts are typically sintered, but often with mixed results. Density and corrosion resistance are improved, but grain growth during the process reduces strength and hardness, making them unsuitable for high-stress applications.

Hot Isostatic Pressing (HIPping) has long been used to eliminate porosity in castings and to form high-strength powder metal parts. In the past decade, post-sinter HIPping has been shown to minimise grain growth and significantly improve the density, tensile strength, hardness and corrosion resistance of PIM parts designed for severe service. The only drawback to standard HIPs has been their long cycle times - 8 to 12 hours depending on batch size. Most of this time has been for natural, unassisted cooling.

Avure Technologies Inc., which specialises in the design, manufacturing, installation, and global support of high pressure presses for densification of advanced materials and critical industrial parts, is now offering a unique solution to this concern. Many of Avure's HIP models can be equipped with its Uniform Rapid Cooling (URC) furnace, which cools the load in a fraction of the time needed by conventional HIP furnaces. This feature reduces total cycle time by as much as 70%, allowing up to three complete cycles per shift, cutting per-unit process HIPping costs by up to 70% as well.

"We presented this high-throughput process at a recent MIM conference," said Dave Peltier, Avure's Director of Sales for the Americas. "The response was very positive, and I believe that most whom I talked to, who felt sintering alone was sufficient, now are strongly considering test cycles to determine the value of post-sinter HIPping. In fact, one gentleman remarked 'This is a game-changer'".

*Fig. 2 Many of Avure's HIP models can be equipped with its Uniform Rapid Cooling (URC) furnace*

Avure is headquartered in Franklin, TN, USA and has significant operations in Vasteras, Sweden and Columbus, Ohio, USA.

Further details and technical data on Avure's Uniform Rapid Cooling are available on the company's website.

www.avure.com/pim

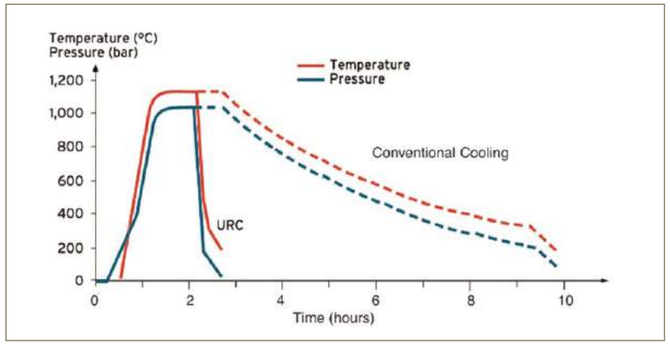


*Fig. 1 Cycle times for small to medium sized HIP's*

## IWC Schaffhausen's ceramic injection moulded watch cases embody high tech Swiss engineering

IWC Schaffhausen has been manufacturing upmarket chronographs at its Swiss headquarters for more than 140 years. In 1986 IWC unveiled for the first time a Da Vinci watch in a zirconia ceramic case. This was produced in only very small numbers because of the enormous difficulty in machining the hard material.

Some 25 years later the ceramic watch case blanks can now be formed more efficiently and with far greater accuracy by powder injection moulding the mixture using zirconium oxide powder. This has resulted in the new Da Vinci Chronograph Ceramic watch case, which combines the extremely scratch-resistant, non-magnetic and acid-proof zirconia, with grade 5 polished titanium.

In another first for IWC, the movement mounting and the seats for the operating parts are machined directly into the ceramic casing ring. The chronograph push-buttons are fitted with newly developed, wear-resistant pushpins, likewise made of ceramic.

In 2011 IWC also introduced ceramic injection moulding to its professional Pilot range of watches. The 46 mm diameter cases of the Pilot Watch Double Chronograph Edition TOP GUN, which retails at over €12,000, are made of black zirconia with crown, push buttons and back cover of matt grey titanium.

The watch, states IWX, is a distillation of all IWC's expertise in the manufacture of Pilot watches with altimeter-style display. The movement is protected against magnetism by a soft-iron inner case.

www.iwc.com

**Interested in ceramic injection moulding for luxury applications?** Find out more in the article "Ceramic injection moulded zirconia products enjoy success in high-value luxury applications", available to download from www.pim-international.com/pdfstore

*IWC Schaffhausen watches featuring ceramic injection moulded cases*

## New President and CEO at Ti powder producer Raymor

Rolland Veilleux, Chairman of the Board of Raymor Industries Inc. has announced the nomination of Jacques Mallette as President and Chief Executive Officer. Mallette also becomes a Director of the Corporation.

Mallette has thirty years of experience in general management and finance for International firms and has been President and CEO of Quebecor World Inc. and of Cascades Paperboard International Inc. Mallette graduated from HEC Montréal and is a chartered accountant.

Raymor Industries is comprised of two divisions, AP&C and Raymor Nanotech. AP&C plasma atomises a wide range of high melting point materials such as titanium and titanium alloys, niobium and cobalt chrome. Raymor Nanotech manufactures highly graphitised Single-Wall Carbon Nanotubes.

www.raymor.com

## Randall M. German Honorary Symposium on Sintering and Powder-Based Materials at TMS 2012

An Honorary Symposium will be held at the 2012 TMS Annual Meeting (March 11-15, 2012, Orlando, Florida) in recognition of Professor Randall M. German, Associate Dean at the College of Engineering, San Diego State University, USA. The symposium will encompass the following four areas, which are not restricted to any particular class of materials:

- Sintering Theory and Practice: includes theoretical and experimental investigations in solid state sintering, current/field-assisted sintering, combustion synthesis, microwave sintering, hot pressing and hot isostatic pressing.
- Liquid Phase Sintering: including theoretical and experimental studies in permanent and transient liquid phase sintering, in addition to transient reactive liquid phase sintering.
- Powder Injection Moulding: encompassing modelling and experimental aspects.
- Powder Metallurgy and Particulate Materials Processing: encompassing all other aspects of powders including, but not limited to, powder production, powder characterisation, powder processing and novel powder consolidation processes.

**German elected to Fellow status in the American Ceramic Society**

It has also been announced that Randall German has been elected to Fellow Status with the American Ceramic Society and will be officially inducted this October in Columbus, Ohio. German is Associate Dean of Engineering at San Diego State University, California, where his research and teaching deal with the net-shape fabrication of engineering materials via sintering techniques.

www.sdsu.edu





## The UK's £10 million Mercury Centre to focus on near net shape powder-based manufacturing

A £10 million development centre which aims to accelerate the deployment of a range of innovative near net shape powder-based manufacturing processes has been created in the UK by the University of Sheffield's Department of Materials Science and Engineering, following dialogue with regional advanced manufacturing companies to understand their future needs.

The Mercury Centre is part financed by the European Union, attracting over £5 million of investment from the European Regional Development Fund, as part of the support for the region's economic development through the Yorkshire and Humber ERDF Programme 2007-13. This European investment is enabling UK industry to secure a globally leading position by accessing faster time-to-market technologies across a range of sectors.

The Mercury Centre has acquired a range of equipment which will deliver cutting edge capabilities in a range of advanced manufacturing technologies such as 3D printing, functional coatings and surface treatment. These include MIM, additive layer manufacturing, functional 2.5D printing of electronics and biomaterials with Aerosol Jet Deposition, novel material processing via Spark Plasma Sintering for rapid development of new ceramics, and electron beam processing for joining, surface treatment and building parts. These are supported by a range of state-of-the-art advanced materials characterisation techniques, and product/process design & simulation capabilities.

Like all new processing technologies, however, industrial deployment generally requires substantial development activity to identify the composition and processing conditions required for a new product or new application, and this is where The Mercury Centre comes in.

Dr Iain Todd, the Centre Director stated, "We are helping companies to adopt these technologies by offering them access to our research facilities and the opportunity to explore the business benefits. We can provide a phased approach, beginning with an initial investigation of business needs and exploratory tests, through to long term-product or process optimisation. We can also offer a "no risk" proof of concept engagement to obtain preliminary data which can then be used in the development of subsequent funding applications".

The University states that there are a range of options for working with The Mercury Centre, ranging from short term contracted consultancy, through to knowledge transfer partnerships (KTPs) and PhD studentships, or even large scale multi-partner projects. Contact the Centre Manager, Dr. Martin I Highett, on +44 (0)114 2225981 for further information.

www.mercurycentre.org

## Submitting News

To submit news to *PIM International* please contact Nick Williams: nick@inovar-communications.com

# MIM components feature prominently in 2011 MPIF awards

Design innovation, superior engineering properties, high end-market visibility and sustainability distinguish the winners of the 2011 Design Excellence awards, the annual powder metallurgy (PM) design competition sponsored by the Metal Powder Industries Federation.

Once again several MIM components secured awards, demonstrating its unique value propositions over competing forming processes such as investment casting, die casting, deep drawing, machining, stamping and fine blanking. The awards were presented during the PowderMet2011 International Conference on Powder Metallurgy & Particulate Materials.

## Grand Prize Awards

**Stainless steel hunting arrowhead**
Parmatech Corporation, Petaluma, California, USA, earned the grand prize in the hand tools/recreation category for a large-game 420 stainless steel hunting arrowhead, the 300Xtreem broadhead (Fig. 1), made for Optek Precision Tooling Ltd. Fabricated by the metal injection moulding process, the thin blade features a critical straightness necessary for accuracy and the external thread for attachment to the arrow shaft is moulded in the design. The part is formed to a final density of 7.7 g/cm$^3$ and has a 48–52 HRC hardness range. Chosing the metal injection moulding route saved 50% on the cost of machining the broadhead from stainless steel bar. The customer sharpens the arrow head edge, the only secondary operation performed.

**Audio housing cup and lid**
FloMet LLC, Deland, Florida, USA, earned the grand prize in the medical/dental category for a housing cup and lid used (Fig. 2) in an audio device with magnetic shielding capabilities. This application is the first of its kind in the high-power audio device sector.

The anti-magnetic MIM material, with a high nickel content, provides electromagnetic interference, or EMI shielding, preventing interference from other electronic signal sources. While the cup has four thin walls for proper assembly, the new lid design must fit securely into the cup opening; this prevents moisture and/or foreign matter from entering the housing, as well as maintaining the EMI shielding capability. Manufactured to 8.30 g/cm$^3$ density, the parts have an ultimate tensile strength of 84,000 psi, yield strength of 32,000 psi, and a 40% elongation. Alternative manufacturing processes such as deep drawing, casting, and machining would have required multiple components. Combining these multiple components through the MIM process provided significant cost savings, in addition to eliminating up to 40% scrap loss.

## Awards of Distinction

**Rifle rear sight**
A rear sight (Fig. 3) used on sporting and military rifles such as the AR-15, M4, and M16 models, received the award of distinction in the aerospace/military market category. Made by Megamet Solid Metals, Inc., Earth City, Missouri, USA, for its customer, Yankee Hill Machine Co., Inc, the nickel steel MIM part features very close tolerances and a complex geometry requiring an elaborate tool design.

The sight allows the shooter to target objects at ranges up to 200 yards by using the larger aperture, and to target objects at longer ranges by flipping the sight down and using the smaller aperture. The part is made to a density of 7.5 g/cm$^3$ and has an as-sintered ultimate tensile strength of 55,000 psi. Choosing the MIM process over investment casting provides a 40% cost saving. Secondary operations are limited to tapping and nitride finishing.

**Multi-purpose tool hammer**
Indo-US MIM Tec (P) Ltd., Bangalore, India, won an award of distinction in the hand tools/recreation market category for a 17-4PH stainless steel hammer (Fig. 4) used in a Leatherman Tool Group multi-purpose military utility tool (MUT) designed for military and law enforcement personnel, and civilians.

Made by the MIM process, the intricate complex part performs five of the MUT's 27 functions. It is formed to a density of 7.5 g/cm$^3$ and has an ultimate tensile strength of 175,000 psi, yield strength of 158,000 psi, heat-treated 35–40 HRC hardness, and a minimum six percent elongation. Secondary operations include threading two tapped holes, age hardening and glass-bead blasting, and an optional blackening treatment performed by Leatherman.

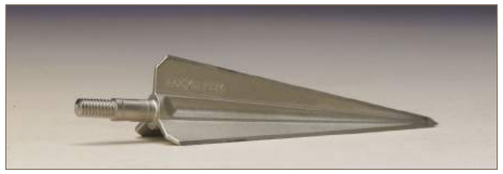

*Fig. 1 Stainless steel hunting arrowhead (Courtesy MPIF)*

*Fig. 2 Audio housing cup and lid (Courtesy MPIF)*

*Fig. 3 Rifle rear sight (Courtesy MPIF)*

*Fig. 4 Multi-purpose tool hammer (Courtesy MPIF)*

*Fig. 5 Distal channel retainer (Courtesy MPIF)*

**Distal channel retainer**
A 17-PH stainless steel distal channel retainer (Fig. 5) formed via the MIM process by Kinetics Climax Inc., Wilsonville, Oregon, USA, received the award of distinction in the medical/dental market category. The complex, multi-level part is the main distal-side component of an articulation joint in an articulating mechanical stapler/cutter used in endoscopic surgery. Formed to a density of 7.7 g/cm$^3$, the part has a typical tensile strength of 175,000 psi, yield strength of 158,000 psi, and a six percent elongation.

The MIM process provided a cost saving in the range of 25 to 30% compared to other design options. Kinetics performs precision sizing and reaming.

www.mpif.org

## OM Group completes Vacuumschmelze acquisition and reports increased sales

The OM Group, Inc. based in Cleveland, Ohio, has completed its previously announced acquisition of Vacuumschmelze GmbH & Co. KG (VAC) of Hanau, Germany, a global market leader in advanced materials and specialty magnetics, for approximately €700 million, including $50 million in common stock. The company announced its intent to acquire VAC on July 5, 2011.

Founded in 1923, VAC is widely regarded as one of the leading producers of advanced materials and technologies, including permanent magnets for electronic equipment markets, alternative energy, automotive, electric vehicles, electrical installation, and energy conversion.

"We are pleased to add the talented people and innovative technology of VAC to our growing enterprise," said Joseph Scaminace, chairman and chief executive officer of OM Group. "This is a financially strong, market-leading business with attractive margins and strong cash flow from operations. Among other things, VAC will accelerate our efforts to move closer to end users in a wider range of stable and fast-growing end markets, particularly alternative energy."

VAC has production facilities in Germany, Slovakia, Finland, China and Malaysia, and employs 4,500 people globally - including 160 scientists and engineers. In the 12 month period ending June 30, 2011, VAC had sales of €423 million and an operating profit of €63 million (calculated under IFRS). OMG purchased VAC from One Equity Partners, the private investment arm of JPMorgan Chase & Co.

The OM Group also reported a 9% increase in sales to $329.5 million in the 2nd quarter of 2011. "We continued to enjoy strong demand in the second quarter across various end markets, particularly within Advanced Materials and Battery Technologies," said Scaminace.

www.omgi.com

## Solar Atmospheres raises the bar with a new 20 bar furnace

Heat treatment specialist Solar Atmospheres of Western, PA, USA, will soon welcome a custom-built horizontal 20 bar vacuum furnace to its Hermitage facility. Designed and built by sister company Solar Manufacturing, this new furnace measures 40" wide x 50" deep x 36" high and is the fastest cooling furnace in the Solar fleet and one of only a few of its kind in the USA.

The excessive pressure and high speed gas velocities of the 20 bar furnace simulate the benefits of oil quenching, but using inert gas as an alternative to oil minimises distortion and provides a much cleaner and greener process. Solar can also better serve customers requiring vacuum carburising by increasing core hardnesses of large cross-sections. This a particular benefit to those in the gear industry.

President Bob Hill stated, "By adding these unique capabilities of the 20 bar to our resources, we can now effectively process a wider range of materials and assist more customers than we could with our 10 bar furnaces."

www.solaratm.com





## Wittmann Battenfeld offers PowerVision quality inspection system

With its new PowerVision system, Austrian injection moulding machine manufacturer Wittmann Battenfeld states that its customers can benefit from a new sophisticated quality inspection system, which can be integrated into the production chain without any specific expert knowledge. The PowerVision system incorporates vision technology from Cognex Corporation, USA, that is integrated into the Wittmann Battenfeld UNILOG B6 control system.

Wittmann Battenfeld states that the required high quality standards for injection moulded products has led to a steadily increasing need for robust quality inspection systems. Due to the demand for 100% quality inspection, with simultaneous reduction in costs, optical quality monitoring with digital cameras has become the accepted standard over the last few years. The visualisation of the measurement function in the UNILOG B6 control enables machine operators to monitor the measurement results, and this integrated concept has been implemented for the first time in the company's MicroPower system.

Measurements are performed by means of the In-Sight Micro ISM 1403-11, an intelligent camera from Cognex. Once its program has been uploaded, it is able to carry out the required measurements automatically. The PowerVision user interface has been specially created for the MicroPower with In-Sight Explorer software from Cognex. Special attention has been paid to making its operation as simple and straightforward as possible. The current version of PowerVision allows customers to carry out presence checks as well as quality inspections on up to 8 cavities per B mould half on the MicroPower's rotary table.

The visualisation is based on a connection between the user interface VisionView from Cognex and Wittmann Battenfeld's Windows XP based UNILOG B6 control system.

The camera is accessed by a program of the vertical Scara robot W8VS2 from Wittmann. The robot transmits the trigger signal for capturing of the image together with the respective cavity number. As the MicroPower has been equipped with a rotary table at the ejector side for short cycles, the respective B mould half for the part to be measured is also transmitted to the camera.

The relevant feedback is then transmitted to the robot, starting with a "picture taken" signal. At this point, the robot can begin to move in order to minimise cycle times. Following evaluation of the picture, the good/bad part selection is communicated to the robot. For the future it is planned to make certain types of defects definable, so that statistic records can be documented and stored.

Many lighting systems in common use require an external transformer to control the intensity of the light required for taking the image. Here, the correct light intensity is set manually with the help of a potentiometer. By contrast, the Cognex In-Sight Micro Ring Light is connected directly and controlled by the camera.

Deploying the latest LED technology, previous high energy lighting systems have been replaced with this power-saving flash device. Unintentional changes in light intensity can also be avoided.

www.wittmann-group.com

# Potential application of waste rubber as a binder for the MIM process

One of the keys to the successful production of MIM parts is the selection of the binders. The binder system provides the metal powders with the fluidity necessary for moulding. It also strongly influences the maximum solid fraction of the mixture that can be moulded, the green strength of the moulded part and the properties of the final products after the debinding process.

Various binder systems have been developed for use in MIM production. However, there are problems associated with the use of some binder systems. Among these problems is the release of hazardous vapours during the mixing, moulding and/or the debinding processes, which can cause irritation to the eyes and respiratory system and damage the environment.

Furthermore, some binder systems offer low green strength or require long debinding process times, as well as being expensive to prepare. To overcome at least some of the problems associated with the other systems, an eco-friendly biopolymer composite binder based on natural sources and waste materials had been successfully developed at the Structural Materials Programme of SIRIM Berhad, Malaysia.

This consists of biopolymer constituents from natural sources, particularly palm stearin and waste rubber as a back bone polymer.

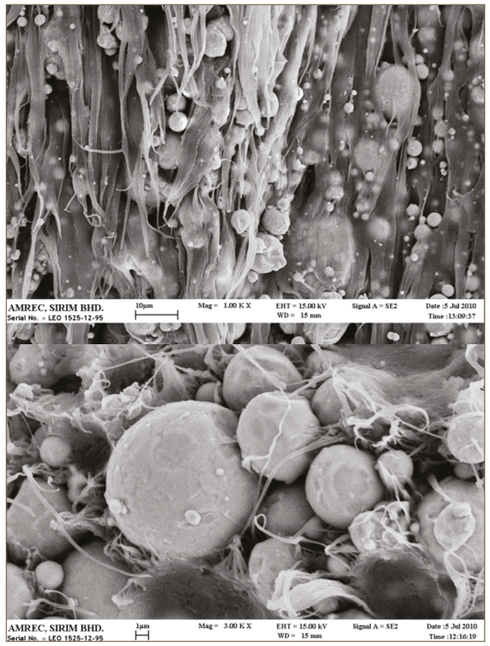


*Fig. 1 SEM of injection moulded sample using waste rubber binder, top) surface and below) fracture*

| Moulding parameters | Value |
|---|---|
| Injection temperature | 200- 210°C |
| Injection pressure | 300 bar |
| Cycle time | 10-15 sec |
| Mould temperature | Room temperature |

*Table 1 Injection moulding parameters*

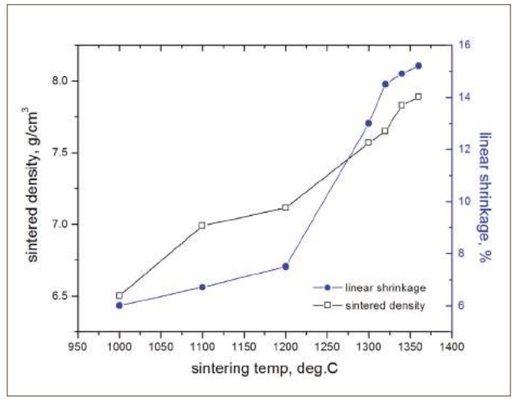


*Fig. 2 Effect of sintering temperature on sintered density and linear shrinkage*

In Malaysia, about 500,000 tones of raw rubber are consumed annually in the making of various rubber products, generating an export revenue about RM 9.0 billion. Latex products, especially rubber gloves, dominate export earnings, contributing RM 7.0 billion. Waste rubber products have to be recycled for environmental purposes or value addition. Therefore, this invention used the waste rubber product from gloves as a binder in the metal injection moulding process.

The waste rubber powder was obtained from rejected gloves or waste gloves by mechanical grinding using crushers to get a polydispersed rubber powder. The waste rubber powder was sieved to obtain a particle size in the range of 100 to 400μm.

The waste rubber binder composition is not only environmentally friendly and non-hazardous, but also enables moulding of feedstock at low moulding temperatures and pressures. This results in less wear on tooling and allows for the use of less expensive tooling materials, such as aluminium, especially for the relatively low volume production of green parts. The waste rubber binder composition is also economical because it utilises waste rubber gloves which are readily available in Malaysia, can be recyclable, and are very low cost.

The new system has been successfully used with metal powders. With properly controlled hydrolysis, these natural sources also provide a spectrum of rheological processing conditions that are suitable for the processing of metal powders. It is hoped that this new binder system can be developed to replace conventional binder systems which typically comprise three to four components.

For more information contact Dr. Mohd Afian Omar.

Email: afian@sirim.my

## Global MIM & CIM market to reach $3.7 Billion by 2017, according to report

A new PIM market report has estimated that, together, the world MIM and CIM markets are forecast to reach $ 3.7 billion by the year 2017. The publisher of the report, Global Industry Analysts, Inc. (GIA), states that, "In post recession period, refocus on increased productivity and capacity requirements in key end-use markets will push demand for MIM and CIM parts as manufacturers begin channelling investments for global competitiveness."

In an announcement relating to the report, GIA states, "Falling cost of ordering and stocking MIM parts, and pricing pressures have brought down profit margins for market participants. Information technology, mechanical, and medical/healthcare industry, which traditionally has been the hot bed for metal fabricators, has significantly cooled down as a result of the recession, thus hurting players in this space. The slumping automotive industry, with specific reference to the United States, has impacted opportunities in this application market in North America. Pressure from the recession, which severely impacted most industrial sectors has forced growth to slow down in the medical/healthcare industry, which was once widely opined to be recession resilient."

"Despite challenges in store, the growing trend towards miniaturisation, and design engineers quest for developing newer components with greater mechanical strength, [there are] opportunities galore for technologies such as MIM and CIM. In the upcoming years, growth in the metal injection molding industry will stem largely from developing markets such as, Taiwan, Malaysia, Indonesia, Mexico and India, among others. The rapid pace of industrialisation represents the prime factor turbo-charging growth in these markets."

The report also claims that "in terms of end-use, Automotive represents the largest contributor to global market revenues of metal injection molding". The report is available to purchase for $4800.

www.strategyr.com

***Looking for the complete picture of global MIM and CIM?***
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## Interest in Euro PM2011 exceeds expectations

With the annual Euro PM Conference and Exhibition just around the corner, the European Powder Metallurgy Association (EPMA) reports that the exhibition has sold out, making it one of the largest dedicated PM exhibitions this year, with over 100 companies from around the world participating. The event takes place in Barcelona, Spain, from 9-12 October.

"The level of interest from exhibiting companies at this year's event is a good indicator that confidence is starting to come back into the Powder Metallurgy industry supply sector," stated Andrew Almond, Euro PM Exhibition Manager.

www.epma.com/pm2011





## Japan's MIM market rebounds in 2010

The Japan Powder Metallurgy Association (JPMA) has published its MIM market data for 2010. Total MIM sales were ¥11.9 billion, showing a strong 9.3% recovery on 2009. The JPMA warned, however, that the recovery of Japan's MIM markets in 2011 is uncertain following the aftermath of the March 2011 earthquake. Data was collected from questionnaires returned by 23 Japanese MIM producers, including both JPMA member and non-member companies. The market breakdown (Fig. 2) shows that industrial machine parts accounted for 21.5% of sales, whilst the percentage of parts destined for the automotive industry fell slightly from 17.5% in 2009 to 15.6% in 2010. "The automotive parts market is not recovering in the way it was hoped," stated the JPMA. By material, stainless steels accounted for 64.1% of sales, and together, stainless steels, Fe-Ni materials and magnetic materials accounted for around 90% of sales. It is predicted that sales of magnetic parts will soon overtake those made from Fe-Ni.

www.jpma.gr.jp

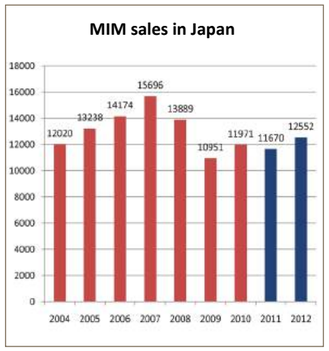



*Fig. 1 MIM sales in Japan, 2004-2010, with estimates for 2011, 2012 (Courtesy JPMA)*

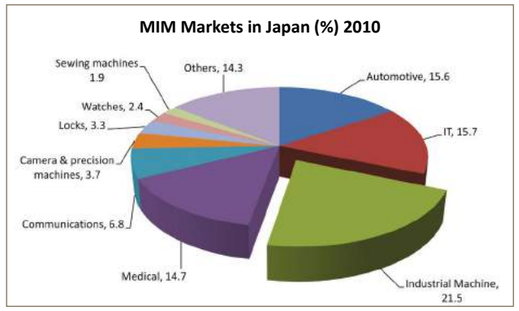



*Fig. 2 MIM markets in Japan, 2010 financial year (Courtesy JPMA)*

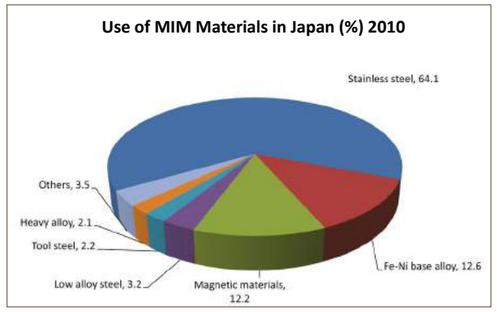
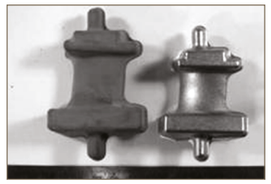



*Fig. 3 MIM materials usage in Japan, 2010 (Courtesy JPMA)*

## Rolls Royce investigates MIM superalloy stator vanes



*The superalloy stator vanes before and after sintering*

UK aero engine producer Rolls Royce plc has been looking at the use of metal injection moulding as a simpler and cheaper manufacturing route to the current 7-stage forging process used to produce nickel-based Inco 718 superalloy stator vanes. The stator vanes are used in the compressors of Rolls Royce aero engines and are made from the Nickel-Iron-Chromium precipitation hardenable superalloy because of the material's good creep properties at high temperatures.

In a recent presentation given at the joint meeting of the Scottish Association for Metals and the Scottish Plastics & Rubber Association held in Glasgow, Scotland, Andrew Russell from Rolls Royce's laboratories in Inchinnan, Renfrewshire, Scotland, described the MIM process used to produce the superalloy stator vanes.

He stated that the alloy powder, with a particle size of 5–40µm, is mixed with a binder based on polymethylmethacrylate (PMMA) and a water soluble polymer, and is fed into a conventional plastic injection moulding machine to form moulded preforms. The preforms are then subjected to a debinding process using solvent extraction with water, followed by a thermal process to depolymerise the PMMA, the decomposition products being vacuum extracted. This, stated Russell, is a critical stage because of the associated shrinkage and the temperature profile and component support both have to be carefully controlled. Tolerances of +/- 0.5% can be achieved. The final step is a single forging stage followed by finishing processes.

The advantages of MIM over the conventional forging sequence are said to be reductions in component times, raw material waste, and energy consumption.

www.rolls-royce.com/civil

## APMA 2011, International Conference on PM in Asia

The Korean Powder Metallurgy Institute (KPMI) in cooperation with the Korean Powder Metallurgy Association (KPMA) is organising "APMA2011" under endorsement of the Asian Powder Metallurgy Association (APMA). Branded as the "APMA 2011 1st International Conference on Powder Metallurgy in Asia", the event will take place in Jeju, Korea , from October 30 to November 2, 2011.

APMA 2011, which also includes a parallel exhibition, will address the latest advances and issues in the science and technology of powder metallurgy and the related PM markets of Asian PM industries. The conference is chaired by Jai Sung Lee (Hanyang University) and Chang Whan Bae (Chang Sung Corporation). For more information contact the Korean Powder Metallurgy Institute.

www.apma2011.org





## GKN plc: Powder Metallurgy sales up 15%, group sales reach £2.9 billion

GKN plc has announced Group sales of £2.9 billion for the first six months of 2011, ended 30 June. The results reflect the continued strong growth in Driveline, Powder Metallurgy and Land Systems and a good performance in Aerospace. The company's Bad Langensalza metal injection moulding facility is one of the largest in Europe.

Group sales were up 11% (£287 million) to £2,988 million, with a trading profit of £247 million, up £45 million, before a £23 million one-off charge relating to the temporary plant closure at the Hoeganaes plant in Gallatin, USA. Powder Metallurgy sales were up 15%, with 9.0% trading margin.

Sir Kevin Smith, Chief Executive of GKN plc, commented, "GKN has continued to make strong progress both in terms of financial performance and in building the future of our global market-leading businesses. The first half trading environment has seen strong market outperformance for GKN's Driveline, Powder Metallurgy and Land Systems businesses. The aerospace market has remained subdued although civil aerospace is now moving into a strong growth phase with volume increases on existing platforms and new aircraft moving into production."

GKN states that the outlook for its major markets is positive although some uncertainty remains, particularly around macro-economic conditions. In automotive, external forecasts suggest that global light vehicle production should reach almost 78 million vehicles in 2011, an increase of 4%, with the strongest growth in India, continuing improvement in North America and Europe and slower growth in China. Vehicle production in Japan is expected to recover strongly in the second half.

www.gkn.com

## Hagen to celebrate 30th Powder Metallurgy Symposium

The Ausschuss für Pulvermetallurgie will be organising the 30th PM Symposium and Exhibition at the Town Hall in Hagen on November 24-25, 2011. Marking this special anniversary event will be a programme of technical presentations (in German) covering sintering and sintering atmospheres, hard materials (including WC-Co, Ferro-Titanit and non-oxide ceramics), heat treatment and sinter hardening of PM parts, modelling of molybdenum sintering, analysis of debinding and sintering of MIM parts, developments in HIP systems and spray forming, and porous materials.

The Ausschuss Skaupy Prize for 2011 will be presented to Dr Gerhard Gille of H.C. Starck GmbH, Goslar, Germany. There will also be an accompanying exhibition, with some 50 exhibitors having already reserved space. For further information contact: Fachverband Pulvermetallurgie, Hagen.

www.pulvermetallurgie.com

# A review of the current status of MicroPIM: Materials, processing, microspecific considerations and applications

In this, the first of a two part report for *PIM International*, Dr Volker Piotter (Karlsruhe Institute of Technology, Germany) reviews the current status of MicroPIM techology. This first instalment covers materials, production equipment, mould design and processing as well as looking at current and potential markets and applications. Examples of commercial and R&D stage microparts are also presented.

Microsystems technology offers a number of opportunities to manufacture entirely new products or to improve existing ones. Still reaching considerable growth rates, the products of microsystems engineering are conquering new markets in a variety of industries such as information technology, life sciences, vehicle and energy technology, mechanical engineering, physical and chemical process technology, to name but a few. Such products are primarily based on production methods to generate microcomponents from silicon or plastics [1, 2, 3].

Many applications, however, in the fields of small precision engineering such as telecommunications, chemistry, and biology require highly loadable microcomponents made from metals or ceramics. The potential of these materials is well known from precision technology applications, where forces, torques, wear, and high temperatures act or small thermal expansions, high corrosion resistance, or biocompatibility are required. To make metals and ceramics usable in microsystems, however, adequate fabrication methods have to be developed further [1, 4, 5, 6]. One such a production method is powder injection moulding.

Powder injection moulding, just like plastic injection moulding, is one of the most efficient methods for manufacturing medium and large quantities of complex precision components. It was, therefore, obvious to enhance PIM for fabrication of micro-sized devices. The large number of publications on this subject can be regarded as conclusive evidence [7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21].

This review will outline the status of the powder injection moulding of microcomponents from metals and


*Fig. 1 Three examples of micro injection moulding machines with plunger units: New on the market is the MicroPower 15 machine from Wittmann Battenfeld (left); Advanced 2C-Microsystem 50 specially equipped with tool evacuation, variothermal temperisation, injection compression option etc., at Karlsruhe Institute of Technology (middle); a further example with quite small pistons (diameter 3mm) is the FormicaPlast from DESMA (right), shown here the 2-component-version*

ceramics, from prototype fabrication to examples in series production. The special microspecific features of materials, tools and process steps will also be highlighted. This review will additionally focus on the various methods to fabricate microstructured tools, as well as the properties of microcomponents that can currently be achieved by powder injection moulding.

To demonstrate that a number of industries will benefit from this economically efficient production technology, a number of examples ranging from laboratory samples to products already on the market will be presented. The second part of this article, which will be published in the December issue of *Powder Injection Moulding International* (Vol. 5. No.4), will cover topics such as special variants of the MicroPIM process and the latest developments in international R&D.

## Feedstocks for MicroPIM

**Powders**

The right choice of powder type is invariably a crucial matter in MicroPIM. For example, it is a rule of thumb that the powder particle sizes should amount to no more than 1/10th, or better still 1/20th, of the smallest dimensions of the cavity. The $d_{50}$ values of typical steels, however, are in the range of 1.5µm to 4.5µm but fractions up to 10µm or above are also used [22, 23, 24, 25]. Powders of still finer granulates, or of other metals, can be obtained by air separation, for example. Further particle size reduction is critical due to the pyrophoric behaviour of metal materials, however, trials with particle sizes around 100nm are also reported [26].

Concerning metal powders, MicroPIM is performed using mainly the conventional PIM steels 17-4PH (1.4542) and 316L (1.4404), however non-ferrous metals such as copper, titanium, tungsten+tungsten alloy powders have to be mentioned [27, 28, 29]. Promising new fields of application are opened up by utilising functional materials or functionalised surfaces [30, 31].

| Process | Geometrical degrees of freedom | Typical aspect ratios | Minimum dimensions [µm] | Dimensional accuracies [± µm] | | Roughness $R_a$ [nm] | Typical insert materials |
|---|---|---|---|---|---|---|---|
| | | | | horizontal | vertical | | |
| UV (SU-8) lithography + electroforming | 2.5D | 1 - 4 (20[a]) | > 2 | 2 | 1-5 | | Ni, Ni alloys |
| X-ray lithography (PMMA resist) + electroforming (LIGA) | 2.5D | 10 - 100 | 0.2 | 0,1 – 1[b] | > 5 | 10 - 50 | Ni, Ni alloys |
| Electron beam lithography + electroforming | 2.5D | 2[c] 4[d] | 2 (grooves)[c] 0.05[d] | n.a. 10nm[d] | n.a. 4nm[d] | n.a. n.a. | Ni, Ni alloys |
| Silicon etching + (electroforming[e]) | 2.5D | 0.1 - 50 | 1 - 5 (typically) 30nm[f] | 0.02g | 0.033[g] | 10 | Si (Ni, Ni alloys)[e] |
| Laser micro caving | 3D | 1 – 10 | 10 | 5-20 | 3-10 | > 200 | mostly steel |
| Laser-LIGA | 3D | 1-10 | 0.200 – 0.4 | 1 | 0.5 | > 50nm | Ni, Ni alloys |
| Micro-machining (milling, EDM etc.) | 3D | 1 - 10 (50[h]) | salient features: < 10 sunken features: 15 | 2 | 3-10 | 300 | steel, brass, aluminium |
| Micro ECM Micro ECF | 3D | < 40 ≤ 10 | 25 0.2[i] - 10 | 2 | | < 200 | almost all electrically conductive materials certain types of steel |
| Micro EDM | 3D | 10 - 100 | 50 | 1-3 | | ≥ 400 | almost all electrically conductive materials |
| Laser sintering | 3D+ [k] | 10 | 50 | 1-10 | < 10 | > 500 | steel (H13, 316L, CoCr4), Inconell, TiV4 |

*a = geometry-dependent, b = concerning linewidths from 1µm to 10µm, c = if intermediate mask is used, d = if shim is used, e = optional process step, f = feasibility limit, g = tolerances vary according to etching depth and would be 10-50 times larger if etching depth considerably exceeds 10µm, h = depending on geometry, i = under restricted conditions only, k = 3D plus hollow and cut-back features*

*Table 1 Microstructuring techniques used for mould insert production*





With regards to ceramic powders, zirconium oxide and aluminium oxide are primarily used for micro replication, for example as dies for wire bond nozzles and optical fibre ferrules. Nitride ceramics, like silicon or aluminium nitride ceramics, are still at an early stage of application.

A comprehensive compilation of powders currently used for MicroPIM can be found in [6].

For micro replication gas-atomised powders are preferable as they ensure high powder filling rates and suitable feedstock viscosities because of their globular or spherical particle shape.

*'binder systems for Micro- and MacroPIM consist of the same ingredients, adaption to micro replication is mainly performed by variation of binder composition and powder/binder ratio'*

High shear rates, which can occur in micro-sized cavities, often cause separation of the individual alloying substances. Therefore, pre-alloyed powders are recommended for MicroPIM. Additionally, they show advantages with respect to dimensional details and surface quality as a result of a more homogenised feedstock.

**Binders**

Powder injection moulding of micro components is associated with stringent requirements regarding the selection of feedstock ingredients, binders as well as powders. Feedstocks for MicroPIM must, of course, be highly flowable to ensure rapid filling at high flow path-to-wall thickness ratios without the melt solidifying prematurely due to the high thermal conductivity of the feedstock.

On the other hand, and sometimes underestimated, is the need for high green strength for the safe demoulding of extremely complex components. Additionally, if demoulding is not successful, the cleaning of microstructured inserts is a difficult procedure bearing in mind the risk of damaging the micro-sized details.

The binder, mostly a polymer matrix material, plays a significant role in compounding as it has to provide both flowability and the green strength of the feedstock. Apart from these two factors, other criteria, such as the homogeneity of the feedstock, its storage stability and recyclability, simple and environmentally friendly debinding, and a controlled shrinkage behaviour, are of the same crucial importance in microdimensions as they are in conventional PIM technology [32, 33]. For commercial and scientific purposes, various organic substances, such as waxes (e.g. paraffin wax, microcrystalline wax, synthetic hydrocarbon wax, oxide polyethylene wax), thermoplastic materials (e.g. polyethylene (PE), polypropylene (PP), polyoxymethylene copolymer (POM), polyethylene glycol (PEG) as well as various additives stabilising the dispersion have to be combined in a suitable binder system [34, 35]. Generally speaking, binder systems for MicroPIM consist of the same basic ingredients as the ones for macroscopic applications, whereas the adaption to micro replication demands is mainly performed by variation of binder composition and powder/binder ratio.

## Equipment for Powder Injection Moulding

**Design of mould inserts and tools**

The right design of runner systems, gate dimensions, number of cavities, position of ejectors, etc. are clearly fundamental issues for the development of an effective MicroPIM fabrication chain. For this purpose the general design rules, for example as formulated in [36, 21] have to be adhered to at least as strictly as in macroscopic PIM. Additionally, uniquely microspecific aspects have to be considered, with the following being a selection of just the most important factors:

- there are higher surface-to-volume ratios
- there is faster cooling and freezing of the feedstocks
- the ratio of demoulding forces to microstructure cross section becomes more extreme

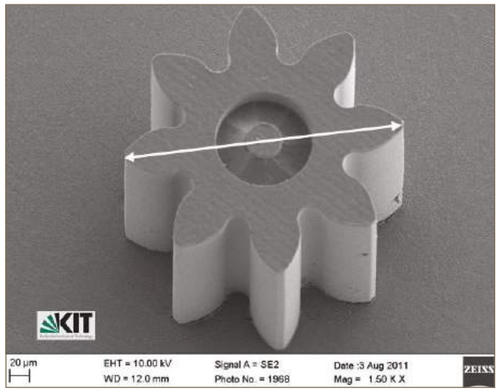



*Fig. 2 Ceramic ($ZrO_2$) micro gear wheel with an overall diameter of 275µm produced by using an X-Ray-LIGA mould insert*

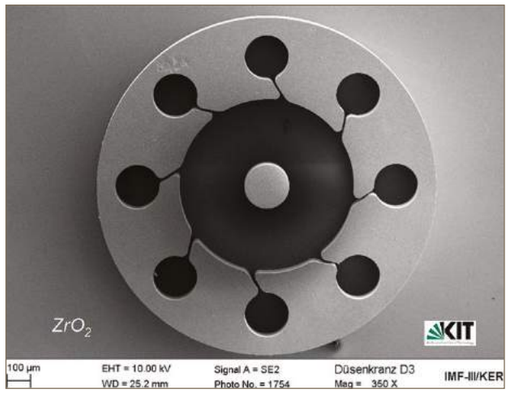



*Fig. 3 Ceramic ($ZrO_2$) micro turbine base plate produced by using an X-Ray-LIGA mould insert*

- there are tendentiously higher shear rates
- there are more abrupt variations of flow channels in square areas.

Qualitative guidelines and useful advice for the layout of MicroPIM tools can be found in [37, 38]. However, to develop optimal design solutions, considerable experience in MicroPIM is still necessary.

If a quantitative process analysis is envisaged, the question of modelling and simulation arises [39, 40]. At first sight, the general constraints are the same as for macroscopic PIM, especially the fact that commercial simulation programmes operate with single-phase material models only. This makes it rather difficult to predict phenomena such as powder/binder segregation [41, 42, 43, 44] or disproportionate formation of strands and folds satisfactorily. Additionally, typical PIM characteristics such as higher inertia, wall friction, yield points, etc. have to be implemented by using modified material models [45, 46] and further progress can be expected in the near future.

Nevertheless, simulation of mould filling based on commercially available software and carefully measured material data can already be used meaningfully for designing tools and runner systems [47, 48, 49].

| Manufacturer | Type | Clamping force [kN] | Injection volume [cm³] | Injection unit diameter |
|---|---|---|---|---|
| Cronoplast Sl / Rambaldi Co Srl | Babyplast 6/10P | 62 | ≤ 4 - 15 | pre-plastification + 1 plunger 10/12/14/16/18mm |
| Klöckner DESMA Schuhmaschinen GmbH | Formica-Plast 1/2 K | 10 | 0.01 - 0.2 | 1 plunger for pre-plastification 6mm + 1 injection plunger 3mm |
| Otto Männer Vertriebs GmbH | micro-män 50 | 50 | ≤ 0.4 | 1 screw 14/18mm + 1 plunger 4/6/8mm |
| Sodick Plustech Co. Ltd. | LD05EH2 | 50 | ≤ 2 | 1 screw 14mm + 1 plunger 8/12/16mm |
| Thermo Electron GmbH | HAAKE Minijet | mechani-cally locked | ≤ 5 | 1 plunger 12.5mm |
| Wittmann Battenfeld GmbH | MicroPower | 150 | ≤ 1.2 | 1 extruder 14mm + 1 plunger 5/8mm |

*Table 2 Compilation of micro injection moulding machines using piston-units currently available on the market*

In the medium and long-term, entirely new approaches based on multi-material models might be able to deliver all the features MicroPIM manufacturers demand [50, 51].

**Manufacturing of microstructured mould inserts**

Individual micro-components, as well as a wide variety of microstructured parts, cannot be fabricated by applying traditional manufacturing equipment and material parameters. For all replication processes, special micro-structured mould inserts have to be produced that are mounted within the tool. The methods to manufacture such microstructured mould inserts can be roughly divided into three classes:

- Subtracting techniques (micro-machining, etching, EDM, laser ablation, etc.)
- Generating techniques (electro-plating, STL, SLS, etc.)
- Combinations of subtracting and generating steps (LIGA, UV-LIGA, etc.)

An overview of established techniques and those currently under development is given in Table 1. It has to be emphasised that this data is based on appraised values only and depend strongly on the entire geometry of the microstructures, the particular replication material, and other process parameters. For each particular application an evaluation is necessary to establish which method is suitable and most effective. Further information can be found in [52, 53, 54, 55].

Although an obviously important topic, the detection and scientific investigation of wear and tear in micro-





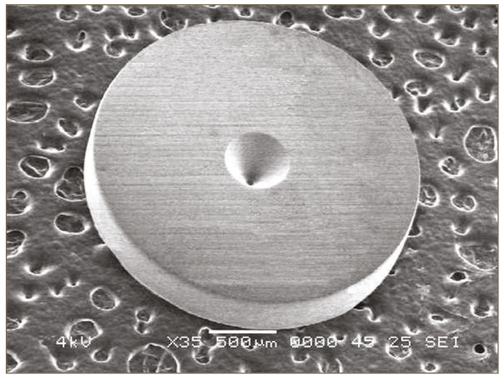



*Fig. 4 $Al_2O_3$ ink dot jetting component with smallest hole diameter 15µm (Courtesy SPT Roth Ltd., Switzerland)*

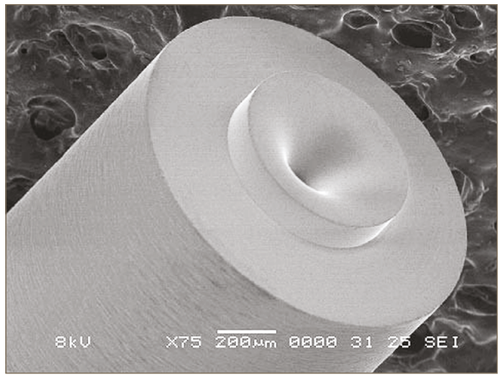



*Fig. 5 $Al_2O_3/ZrO_2$ ink dot jetting component, cylinder tube used for flow cytometry with smallest hole diameter 45 µm (Courtesy SPT Roth Ltd., Switzerland)*

structured mould inserts is reported only rarely [56, 57].

Compared to mould insert preparation, the production of the entire tool is much more conventional. In most cases a down-sizing of the methods well-understood from macroscopic injection moulding is sufficient to build up two or more plate tools, along with the utilisation of movable slides and the implementation of larger numbers of microstructured mould inserts - up to 32, and probably more in the near future.

In certain cases, for example in parts with very high aspect ratios, some microspecific features have to be added for the evacuation and variothermal temperature control of the tools. Evacuation of the cavities prior to injection avoids pressure defects (Diesel effects), especially in the case of blind-hole microstructures, where the microstructure orientation is perpendicular to the parting plane. Due to the small wall thicknesses and large surfaces of the microstructures, the feedstock temperature adapts to the wall temperature of the microstructure cavities within milliseconds. Heating of the mould inserts to temperatures near to the no-flow temperature ensures that complete mould filling is achieved [58] even for components with high aspect ratios. For defect-free demoulding, the core of the tool also

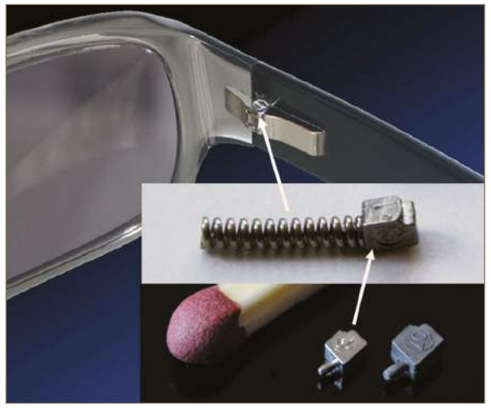


*Fig. 6 Component for spectacle hinges made of 316L steel, sintered and green body (bottom), equipped with a spring (middle), and mounted in the locking hinge. Weight of part 0.028g. Moulded in a 32-cavity tool at approx. 10 million parts per year (Courtesy of OBE GmbH, Germany)*

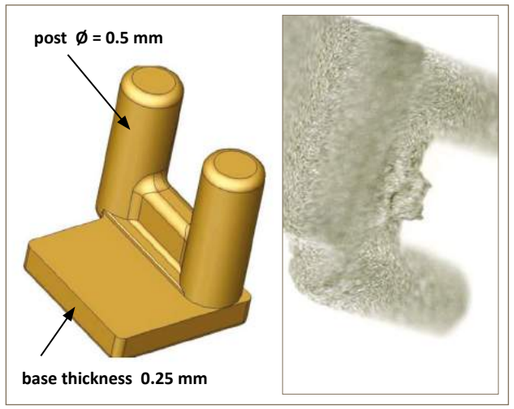



*Fig. 7 Electrical connector to be mounted in medical pacemakers. Weight of the part is only 0.007g. Material: MIM-Alloy 42 (also known as UNS K94100 or DIN 17745) plus subsequent Ni & Au plating (Courtesy of NetShape Technologies Inc., USA)*

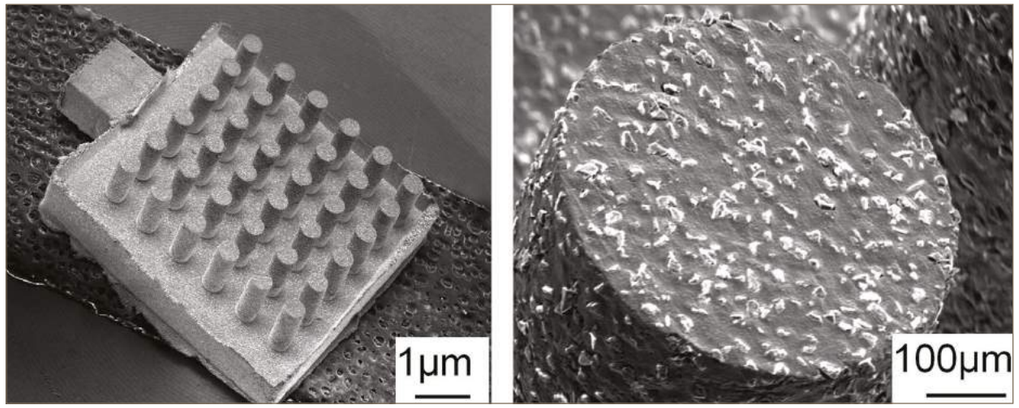



*Fig. 8 Micro electronics open up a wide field for MicroPIM applications, for example as heat sinks. Here an example made of Cu/Diamond material providing considerable high thermal conductivity at a CTE very near to silicon. Pillar diameter: 500µm, height: 1500µm (Courtesy of Kinki University and Taisei Kogyo Co., Ltd, Japan)*

has to be cooled down again after filling (variothermal temperisation).

**Suitable machinery for MicroPIM**

Of course, procurement of the right mould inserts and tools is not the only challenge when setting up a MicroPIM fabrication plant. Equally important is the right choice of injection moulding machine.

Here it is important to distinguish between units operating with reciprocating screws or with plunger injection. The first type represents the lower limit of conventional injection moulding machines, which is given by a screw diameter of 14mm and in certain cases down to 12mm but requiring special micro pellets. A further reduction of the screw diameter would lead either to unsuitable low cross sectional areas of the screw cores or winding depths too shallow to convey powder particles of conventional size. Obviously, both ways are unacceptable, especially for processing of PIM feedstocks.

To obtain even lower shot weights a further reduction in diameter is necessary. For this purpose injection plungers, better described as combinations of pre-plasticising screws and injection plungers, have been developed since the late 1990's. Such a configuration allows for sufficient plastification and homogenisation quality, whilst the low plunger diameters enable small shot volumes and at the same time maintains stroke ranges long enough to regulate the shot sizes very accurately. Examples of injection moulding machines specially developed to deliver the smallest shot volumes are shown in Fig. 1.

Disadvantages of plunger units, compared to reciprocating screws, are a certain leakage between injection piston and barrel and that the 'first in - first out' principle can not be assured. A compilation of piston-based micro injection moulding machines is given by Table 2.

The safe demoulding of extremely complex green bodies requires exact machine and tool control procedures which are inherently provided by electrically-driven machines. In the case of hydraulic units, throttle





| Material | Minimum lateral dimensions [µm] | Min. details [µm] | Aspect Ratio, salient structures | Aspect Ratio, sunken structures | Tolerances[b] [%] | Surface roughness[c] $R_{max}/R_a$ [µm] |
|---|---|---|---|---|---|---|
| Metals | 25 | 10 | 10[d] | > 10 | ≤ ±0.5 | 7/0.8 |
| Ceramics | <15 | < 3 | < 15 | 15 | ±0.1 - ±0.5 | < 3/0.2 |
| Polymers[e] | 10 | < 0.1 | 20 (200[f]) | 25 | ±0.05 | 0.05/0.04 |

*[a] = AR, corresponds to flow path-to-wall thickness ratio, [b] = reproducibility of nominal size, [c] = dependent on the type of mould insert, [d] = can be much higher in certain cases [71], [e] = for comparison, [f] = cliff profile*

*Table 3 Status of micro injection moulding, characterised by major parameters*

aggregates are sometimes used.

Peripheral equipment that has been well-tried in conventional injection moulding, for example automatic granule conditioning, feeding systems and all-automatic parts removal systems, as well as the entire quality assurance systems, have to be modified and adopted for micro-injection moulding use [59, 60].

## Debinding and sintering

From a certain point of view these steps differ least to the macroscopic procedures as described in [61, 21]. For example, debinding can be performed by thermal melting or wicking, pyrolysis, solvent extraction or catalytic degradation of the organic binder, or by a combination of these methods. It has to be taken into account that debinding times depend on the thickest section of the parts. In the case of single micro parts this often leads to shorter cycles, whereas in the case of microstructured parts, debinding times might be nearly as long as for macroscopic devices.

At a first glance the sintering of micro components should be easier to achieve due to their higher surface-to-volume ratio, and in many cases this fact actually leads to shorter cycle times. On the other hand, grain growth and the resulting anisotropic materials properties can be a considerable challenge in MicroPIM [62, 63, 64]. This is reinforced by the relatively small particle diameters, and consequently

*'The safe demoulding of extremely complex green bodies requires exact machine and tool control procedures which are inherently provided by electrical-driven machines'*

*Fig. 9 Small 316L hook used in stitch devices (Courtesy Taiwan Powder Technologies)*

high sintering activity, of typical MicroPIM powders. To avoid excessive grain growth, heating and cooling rates often have to be increased, unless there is a risk of warpage occurring.

A further crucial matter in macroscopic as well as in microscopic PIM is how to meet all the necessary dimension tolerances. Comprehensive literature is available for both applications leading to the conclusion that tolerances ranging from ±0.3% - ±0.5% are usually obtained without spending excessive time on process optimisation. As a result of thorough process optimisation, values of approximately ±0.1% for at least one dimension are possible [65, 66, 67, 68, 69, 70].

## Examples of MicroPIM in research and in commercial applications

The trend towards miniaturisation, as well as extensive research on MicroPIM, has been underway for many years and a significant state-of-the-art has been reached. It can be described quantitatively by key data as published in Table 3. This data is based on long-term R&D activities performed at Karlsruhe Institute of Technology (KIT), but has been cross-checked and approved by results gained at other institutions. It has to be mentioned, however, that these values depend strongly on the particular parameters, applied materials and other circumstances, therefore they should provide only a rough overview.

Besides these key data, observing microPIM components already on the market, or under development, is extremely informative. Concerning micro components made of ceramic materials, it has to be considered that ceramic powders are, in principle, better suited for MicroPIM. A higher replication performance in terms of smaller dimensional details and better surface qualities can therefore be achieved. Examples from the R&D sector are shown in Figs. 2 and 3.

MicroCIM, of course, is not only performed in research institutions. It has for many years found its way into industrial applications. Impressive examples are shown in Figs. 4 and 5, whereas examples for MicroMIM products are given in Figs. 6 - 9.





## Summary and outlook

Powder injection moulding is a technology that is highly suited to the medium or large series production of highly loadable micro components from metal alloys or ceramics. When applying micrometer or sub-micrometer powders, it is possible to fabricate precision components that require little, if any, finishing work. Sourcing of the finest powders, however, is still a challenge, especially when it comes to metals.

At present, a few trends can be observed regarding the ongoing development of MicroPIM. Some objectives, such as the reduction of failure rates, the inspection of green bodies, early detection of cracks, etc., are very similar to the challenges the PIM industry faces in macroscopic fabrication. There are, of course, R&D activities currently underway to enhance PIM techniques for the fabrication of microcomponents, for example the development of new machinery.

The promotion of MicroPIM to new end-user markets is also underway, and the increase in the number of materials used is opening up new fields of applications, for example to process metals such as tungsten or titanium. A further trend is the fabrication of multi-material parts by multi-component micro injection moulding, a topic that will be one of several to be covered in Part 2 of this review, to be published in the December 2011 issue of *PIM International*.

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## Author

Dr. Volker Piotter
Head of Process Development Unit
Karlsruhe Institute of Technology (KIT)
Institute for Applied Materials
Hermann-von-Helmholtz-Platz 1
Building 695
76344 Eggenstein-Leopoldshafen
Germany
Tel: +49 721 608 2 6463
Fax: +49 721 608 2 2095
Email: volker.piotter@kit.edu
www.iam.kit.edu/wpt

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# Indo-MIM: The world's largest capacity MIM producer sees no limits to the industry's expansion

Indo-US MIM Tec (P) Ltd, widely known as Indo-MIM, is today a major force in metal injection moulding. With the largest reported production capacity in the industry, the company's continued reinvestments have enabled it to enjoy a dramatic growth in sales to a wide variety of industry sectors. *PIM International's* Nick Williams reports on the story of the company to-date and interviews its President, John Gaspervich, about the factors behind the company's success, the outlook for MIM, and the challenges of doing business in India.

Bangalore, in Southern India, is one of the country's largest and most dynamic cities. As well as being known as India's 'silicon valley', it has attracted numerous high-tech and heavy industries, from aerospace and defence to telecommunications. The city is also home to one of the world's most successful MIM companies, Indo-MIM, which has its headquarters and two manufacturing sites here.

## The story of Indo-MIM

It was in 1997 that the precursor to Indo-MIM, AF Technologies India (P) Ltd., was formed through a 50/50 joint venture with one of North America's leading MIM producers, Advanced Forming Technologies (AFT), a company of Precision Castparts Corp.

In this joint venture, the new company benefitted from AFT's expertise in MIM processing that dated back to the company's formation in 1987. Technicians from India were sent to AFT's US plant for training, and a new 2000m$^2$ MIM production facility was commissioned in Hoskote, a suburb of Bangalore. MIM part production began in 1998 and ISO 9001 certification quickly followed.


*Fig. 1 Indo-MIM's Hoskote plant and headquarters, Bangalore*

In 2001 complete ownership of the venture was acquired from AFT and the company adopted its current name. Reflecting the company's international ambitions, a US sales and engineering office was opened in Ann Arbor, Michigan, in 2002 and by 2005 the company's MIM production facility had expanded to 10,000m$^2$.

Increases in moulding and sintering capacity followed and a year later, in 2006, a second manufacturing site was acquired in Doddaballapur, also in the Bangalore area and approximately 50km from Hoskote. This 13,000m$^2$ facility was designed specifically to address the demand for high volume MIM production and saw the introduction of the company's first continuous high temperature furnaces. Regular

*Fig. 2 Indo-MIM employs a staff of more than 1000 at its production facilities and engineering offices worldwide*

investments in production capacity and additional facilities followed, as well as an increasing network of regional sales offices in Europe and North America.

## Production facilities today

Today Indo-MIM has a staff of more than 1000 people and its MIM facilities cover more than 25,000m$^2$. Both facilities operate around the clock, seven days a week. The Hoskote facility serves as the company's headquarters and administrative centre, as well as manufacturing for low to medium volume MIM orders and selected finishing operations. The Doddaballapur facility is tailored towards the production of medium to high volume orders, as well as precision machining, heat treatment, assembly and plating operations.

Manoj Kabre, Vice President of Marketing at Indo-MIM, told *PIM International*, "As a strategic policy, we have been re-investing in anticipation of incoming orders. Given the captive tool room available in-house, new part addition has been at the rate of 15-20 parts every month, so around 150-180 parts every year. This has been made possible thanks to a dedicated product development and marketing team that continuously looks for new business in the market. Current spare capacity is at 10-15%, and the next stage of expansion is already underway."

In 2010 alone Indo-MIM increased production capacity by 40%. Injection moulding is currently undertaken using 76 Battenfeld machines, all adapted with proprietary enhancements. Each machine's output is continuously monitored using Statistical Process Control (SPC) tools. "We found Battenfeld technically amenable to our requirements and are very happy with their support. Both Indo-MIM and Battenfeld are as though 'made for each other'," commented Kabre.

Despite operating in a relatively low cost economy, there is an increasing awareness of the benefits of automation at Indo-MIM. "We have been increasingly using automation on our machines, although it is often difficult to justify investments in automation, considering the lower labour cost in India," stated Kabre. "However considering the depletion in the benefits of the labour cost in the coming years, we have plans to increase the participation of automation as well as robots at our plant. We have seen a clear advantage of automation in giving us higher consistency and control. There is of course the need to provide intensive training to our engineers for the best application of such technology."

Following solvent debinding, parts are processed in sintering facilities that include ten batch vacuum furnaces that average 1.3m$^3$ in capacity, manufactured by GM Enterprises and Seco Warwick, and two continuous furnaces manufactured by CM Furnaces Inc. The company has made significant recent investments in solvent debinding systems manufactured by Lömi GmbH, and a new continuous furnace is currently being installed by Cremer Thermoprozessanlagen GmbH, believed to be the world's largest MIM-Master system. Both the company's MIM facilities are certified to ISO 9001 and ISO/TS 16949.

## Materials

Indo-MIM produces all of its feedstock in-house using both batch and continuous compounding equipment. The mixing process and material characteristics, state Indo-MIM, are continuously monitored. "We use both water and gas atomised powders in our process. Almost all the key MIM powder producers worldwide are our suppliers, given the fact that our requirements are amongst of the largest in the world," stated Kabre.

*Fig. 3 Indo-MIM's toolmaking facility has the capacity to produce more than 30 new tools each month*

*Fig. 4 The company currently operates 76 Battenfeld injection moulding machines*





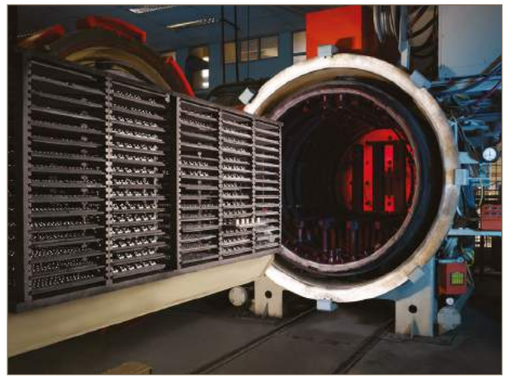

*Fig. 5 One of the large batch sintering furnaces at Indo-MIM*

*Fig. 6 A continuous furnace for the production of high volume parts*

Materials processed typically range from high strength and stainless steels to tool steels, magnetic materials, tungsten heavy alloys and superalloys. Commenting on future developments, Kabre stated, "Titanium MIM is in the pilot production stage at Indo-MIM and is expected to go into full production early next year. We have decided to pursue this market with an exclusive focus. In addition, we are now also geared up for ceramic injection moulding."

## Part production, sales and markets

Since 1998 Indo-MIM has developed more than 1500 MIM parts for a diverse range of end-user markets, broadly divided into the automotive, medical, consumer and industrial sectors.

"Every month we process 400 - 450 different part numbers and monthly production volumes, counting all these part varieties, are in the region of 10 million pieces. We introduced approximately 200 new parts in the past year, with typical annual volumes ranging from 50,000 to 10 million."

Customers are typically international blue-chip and Fortune 500 companies, with over 90% of production exported to more than 30 countries worldwide.

From 2001 sales of Rs.40 million (approx. $900,000) the company has consistently grown at about 20%+ year over year. In 2010 Indo-MIM managed a phenomenal jump in sales in excess of 40% coming off of a relatively flat year in 2009. As with many MIM producers, thanks to the diversity of markets that it serves and the cost savings that MIM offers, Indo-MIM was able to navigate the 2009 financial crisis with no drop in turnover. Sales growth in excess of 30% is anticipated in 2011.

The company is also aware of the huge potential of micro powder injection moulding (μPIM) and has started working towards the commercial production of microparts. "Micromolding does require exclusive machines, wherein we have made some small investments. We have selectively looked at this market based on customer requirements and are doing a detailed study. The technical challenges remain in the form of exclusive equipment requirements that do not rationalise with other commercial production at hand. However, we are open to additional investments in specialised machines for micro-moulding," commented Kabre.

## The advantages of in-house tooling facilities

Indo-MIM has invested heavily in its in-house tooling facility with a view to reducing product development times and cost. The company's tooling centre has the capability to design and manufacture up to 30 new tools each month, in addition to the maintenance of existing tooling.

As with the company's MIM processing equipment, much of the machinery for tooling manufacture is bought in from leading suppliers in Europe and Japan, including numerous CNC milling machines, modern EDM

*Fig. 7 A selection of MIM components for the automotive sector*

*Fig. 8 ISO Class 8 cleanroom facilities at Indo-MIM for the assembly of medical products*

*Fig. 9 A selection of MIM medical device components manufactured by Indo-MIM*

machines, hard milling equipment and precision grinding machinery that is operated in a temperature controlled environment. "We believe that this facility gives us a significant advantage when responding to the urgent requirements of customers and their markets," stated Kabre.

## Creating a leaner supply chain for customers

A key strategy of Indo-MIM has been to offer an extensive range of services in addition to MIM part production, helping to create a leaner supply chain for its customers. Finishing operations range from heat treatments to surface treatments such as plating, polishing and PVD coating.

The company has extensive precision machining operations that recently received AS9100 certification for their aerospace capabilities, and extensive use of SPC combined with a state of the art metrology laboratory helps the company's machining operations hold micron tolerances.

Assembly services are also a key part of Indo-MIM's offering to customers, ranging from basic sub-assemblies to the complete mechanical integration of products. The assembly areas include ISO Class 8 cleanroom facilities for the assembly of medical products, with ultrasonic washing and packing capabilities.

A more recent investment by Indo-MIM's owners was in a new investment casting facility, Gowri Ventures (P) Ltd., located in Tirupati, to the east of Bangalore. The 2,800m² facility started commercial production in 2010 and supplies fully finished investment castings of up to 2kg formed from carbon, alloy and stainless steels, as well as Ni and Co alloys.

Gowri Ventures, which recently attained AS9100 certification, shares technical sales staff with Indo-MIM, as well as the services of the group's machining and quality management facilities. Expansion into the vacuum casting of titanium and superalloy products is planned in due course.

## Philosophy and social responsibility

Those who have witnessed the growth of Indo-MIM in recent years are impressed not only by the scale of commitment to the technology that has resulted in such dramatic expansion, but also by the commitment of the employees who consistently demonstrate an enthusiasm and can- do mentality that, as one well known MIM materials supplier commented, 'makes a formidable combination of assets'.

With success the company has also retained a strong sense of social responsibility. Employees benefit from a high level of training and, as elsewhere in the industry, all precautions are taken to minimise the environmental impact of the company's activities.

A sense of social responsibility extends to outside of the factory gates, where the company does much for the local communities, including providing daily midday meals at local schools for more than 1000 children, as well as funding new operating theatres and medical laboratories at the Bangalore Baptist Hospital, an institution that is committed to serving the poorest in the community.

## Outlook

There appears to be no slowing down in the growth of Indo-MIM and the company looks set to continue its growth path through continued reinvestment in capacity and new technology. To keep feeding its lines of injection moulding machines, the company's sales team attend a full schedule of international exhibitions, from North America's SAE show to medical device and gun shows, totalling more than 25 events a year.

There is also a recognition that the company will need to establish manufacturing bases outside of India to better serve its growing international customer base.

## Contact

**Global Headquarters, India**
Global Headquarters, India
Indo-US MIM Tec Pvt.Ltd.
# 45 (P), KIADB Industrial Area
Hoskote,
Bangalore 562 114, India
www.indo-mim.com
Tel: +91 80- 27971418
Email: infohq@indo-mim.com

**In the United States**
214 Carnegie Center, Suite 104
Princeton, NJ 08540
Tel: +1 734 327 9842
Email: infous@indo-mim.com

**In Europe**
Indian Business Center
Oskar Lapp Str. 2
70565 Stuttgart-Vaihingen
Germany
Tel: +49 173 265 6067
E-mail: infoeu@indo-mim.com





# Interview with Indo-MIM President, John Gaspervich

*John Gaspervich has a long track record with MIM, from his early days as a founding member of Advanced Forming Technology (AFT) in Colorado, USA, to his current role as President of Indo-MIM, which he has held since 2008. In the following interview he discusses the factors behind Indo-MIM's success, the outlook for the technology, and the challenges of doing business in India.*

**The size and sophistication of MIM operations has grown considerably over the last decade, and the operations at Indo-MIM have also expanded dramatically. Was it the intention from the start for Indo-MIM to become the world's largest capacity MIM producer, and was such strong growth during the last decade anticipated?**
Our Chairman and CEO, Krishna Chivukula, set the goal back in 2002 that Indo-MIM would be the world's largest MIM producer someday. Of course at that time Indo-MIM was unknown and the young management team faced what seemed like insurmountable odds to achieve that. His leadership, confidence and commitment made the team believe they could achieve that goal, and they did.

**What do you believe are the fundamental reasons for Indo-MIM's dramatic growth and success to date?**
First and foremost I would credit every member of our company with sharing the same vision and drive that we want to be the leader in the industry. To become that, we all understand that we need to be focused like a laser on giving the customer exactly what they want as our first priority. Secondly, our management and sales team have been given wide latitude by our Chairman to make key business and investment decisions that allow us to support our first priority. Finally, it comes down to execution. Giving one hundred percent effort, day in and day out to satisfy the customer. Our entire team shares a personal commitment to meet our customer's needs.

**Can we expect the scale and speed of expansion at Indo-MIM to continue, and are manufacturing locations outside of India under consideration?**
Frankly speaking, going slow or thinking small is not part of our company culture. Growth, speed and agility is embedded in our DNA. So yes, I think the industry can expect we will continue our present course. Expansion outside of India is almost a necessity. We don't feel that we can fully service a global market from just one location.

**Indo-MIM has clearly made significant investments to address a desire from customers for more than just MIM component production. Do you see the requirement to offer finishing and assembly operations as something that the industry as a whole will have to address?**
Most definitely, supplying ready-to-assemble MIM parts is a customer expectation and thus essential for MIM parts producers. How a MIM supplier meets that expectation becomes a competitive advantage. Providing assembly services, simple or complex, I feel is less important for the MIM parts producer. However, our customers are always seeking ways to shrink their supply chain so having complete finishing and assembly capabilities are both strong core competencies of Indo-MIM.

**MIM has come a long way since the first commercial successes in the 1980's. What issues does the industry still need to address in order to ensure continued adoption by end-users, and to stay ahead of competing technologies?**
Having been in the MIM industry since that time, I have been engaged in, and witness to the industry's evolution. MIM clearly has earned respect as a viable manufacturing technology capable of making parts for very critical applications. With the wide variety of feedstock and equipment suppliers for MIM, the technology is now more accessible than ever to anyone having the wherewithal to try their hand at it. Therein lies the concern for the veteran producers who understand what it really takes to be a reliable supplier in the MIM industry. That's a long lead in to a short answer, which is, the MIM industry needs to maintain its credibility by generating and adopting strict material and product attribute standards so that user expectations are understood and met by every parts producer.

As far as competing technologies, MIM has generated its own market space over the past 25 years supported by the economics of the process. I don't feel it will lose any ground to the traditional metalworking techniques with which it competes. It will only continue to gain market share from machining, investment casting, Powder Metallurgy, stamping and so on as users understand how to make full use of what the technology can offer.

*Continued overleaf....*

Company profile: Indo-US MIM Tec

**What do you believe end-users are looking for when selecting a MIM supplier?**
Ultimately, trust. That you can deliver on their expectations. What generates this trust is a supplier who has the experience, attention to quality, resources and confidence to get the job done.

**When a potential user decides not to proceed with the production of a component via the MIM route, what in your experience are the main impediments? (Component cost, tooling cost, risk aversion, mechanical properties, technical obstacles?)**
We have bid on thousands of programs and not winning a project can be due to any or all of those issues. Assuming the product has been tooled up, it usually comes down to some sort of unidentified technical requirement that is found during product testing. We are sometimes pushing the envelope in the type of products we attempt. It usually comes down to a lack of time to solve those issues and instead the product launches with a traditional, albeit more costly metal forming route. We are disappointed when this happens but I think you aren't challenging yourself and the technology hard enough if you don't have a few failures.

**The continued growth of the MIM industry is inevitably tied to the availability of suitable powders. What concerns does Indo-MIM have about current powder supply capacity, about powder pricing, and what can a large MIM producer such as Indo-MIM do to manage its powder requirements?**
We are a very large consumer of MIM powders and our preferred suppliers have done a fantastic job to help us meet our requirements. In this case size does have its advantages. However, we have a large concern that the major powder producers are not investing in capacity fast enough to be in lock step with the industry. With no one giving guarantees up and down the supply chain, it's a tenuous situation that we just have to proactively manage. We would clearly like to see industry investment in atomising capacity and new technology that will drive up yields. Doing so will only strengthen the future growth of the industry. Our suppliers do get some confidence when they see the kind of investments we are making in MIM production capacity.

I am also now seeing more price instability in pre-alloyed powders driven by global strategic metals demand. This makes it tough for us to manage long-term contracts with our customers. That being said, powder cost as a percentage of part selling price for MIM parts is typically low. So I don't feel we (the MIM producers) are in as serious a situation compared to some other industries.

*'the MIM industry has done a fine job overall to continually develop standards. Early leaders in the industry had the foresight to begin this work...'*

**Do you think that existing standards provide adequate information on the physical and mechanical properties of MIM materials?**
I think the MIM industry has done a fine job overall to continually develop standards. Early leaders in the industry had the foresight to begin this work with MPIF and the creation of MIMA in 1987. There are many early industry pioneers that need to be thanked for their efforts to generate standards and build industry credibility.

I do feel the standards information available today is quite adequate for the majority of applications currently being developed for MIM products. However, there are a host of materials in use that are still yet to be characterized. Dynamic and high temperature properties are necessary for certain materials as well. I don't see this as a major impediment to industry growth but the standards work needs to continue at a steady pace.

**With regards to the marketing of MIM, does the industry as a whole need to do more to improve awareness, or are individual company's efforts succeeding in spreading the message?**
Given the relative age of the industry, I still come across many people in the engineering and manufacturing community who still have not heard of MIM. If they have, they have misconceptions about how the process works. I don't think this is for a lack of the industry and trade associations aggressively promoting the technology. Most of us in the industry really enjoy the opportunity to explain the technology and watch the amazement when you show someone a green part and a sintered part. To many people it seems like magic (it still is to me).

Conversely, I am very pleased to see that many products are now being designed for MIM up front. Given twenty-five years of industry evolution it may feel like awareness of MIM should be further along but investment casting has been around for thousands of years. Based on the same general lack of process understanding with investment casting, in relative terms I think the MIM industry is spreading the message well.

**What makes India a desirable place to locate a business such as Indo-MIM?**
India has given us some distinct advantages. The most obvious one allows us the ability to maintain a very large engineering team that gives us muscle none of our American or European competitors can match. This horsepower combined with affordable tooling and manufacturing resources has allowed us to capture market share that was previously just not there. In essence we really created our own market. This was mostly just out of necessity as we were small, unknown and ten years ago who would want to buy precision products from India of all places? But after development of well over one thousand unique MIM products we have become pretty good at what we do.

Our location has also presented many challenges in growing a high technology company. At times, India can still be a tough place to run a manufacturing business. You have to put up with bureaucratic issues and small nuisances that you just don't have to deal with in the west, or even China for that matter. You definitely learn to how improvise, adapt and overcome when running a business in India. Working in India teaches you things you cant learn anywhere else. I have gained a very deep respect for our Indian team and their fortitude to deal with all manner of problems thrown at them.

**India has a recognised tradition of metallurgical engineering and a strong record of R&D and manufacturing, particularly in the Powder Metallurgy sector. How easy is it to recruit and retain a workforce with the necessary skills for an operation such as Indo-MIM?**
MIM is a very unique process and it requires knowledge and skills in many disciplines that you just don't find in people unless they have worked in the industry. We have an extremely competent technical and management team that have been with Indo-MIM from the start. This team has become very adept at training and mentoring new engineering recruits right from university.

Besides MIM we teach them methodology like Six Sigma, Lean, 5's and Kaizen. They are taught in a classroom setting and also put to work in all parts of the process. After about two years we have an engineer who is pretty well versed in applying MIM technology. This recruiting and training process is continuous.

There is high competition for mechanical engineering resources in India because of the massive IT industry so retention can be a challenge. But we work hard to keep our top performers happy and those people in turn are very dedicated to our company. Our people like being a part of the winning team we have become.

*'We clearly see opportunity in MIM for the automotive and medical devices market growing over the next few years in India'*

**Over 90% of Indo-MIM's production is for export. Do you foresee an increase in the demand for MIM parts in India? If so, in which sectors do you see the markets for MIM developing?**
Historically we have had a difficult time competing with the traditional metal-working industries in India. Machining, casting, forging, PM, etc. are still all very low cost challengers but I think that is on the verge of tipping in MIM's favour in the next few years. Western companies in all sectors are increasing in number in India to supply the home market as well as for export. These companies are driving for much higher quality standards, which are making it tough for the low cost suppliers to survive. We clearly see opportunity in MIM for the automotive and medical devices market growing over the next few years in India.

**With rising labour and energy costs, can India remain a competitive location for a company such as Indo-MIM?**
I don't think that any company today can rely on using a labour cost advantage as their only business strategy and this has never been ours. Our labour cost advantage has given us the freedom to make large investments in facilities, equipment and technical capabilities. This has allowed us to rapidly grow our Indian operations to a size that gives us a dominant position in the MIM industry.

Our scale has gained us a global customer base in diverse markets, some of which will be better served in the future by adopting a local manufacturing strategy. We will continue to make investments in our India operations to gain efficiency and productivity improvements as our costs achieve parity with our western competitors.

**With Gowri Investment Castings, a unit of Indo-MIM, your company is able to offer its customers a far wider range of parts "under one umbrella". How do you see the ongoing development of the two processes, what are their advantages and disadvantages, and where do you see the greatest scope for technological innovation and efficiency?**
A key strength of Indo-MIM is the relationships we build with our customers. The immediate goal for the Gowri operation was the ability to service those customers who like doing business with us but had products where MIM did not make sense. Our view is that investment casting is a very mature technology. Finding and hiring the right technical talent is the key ingredient we need to add to continue to develop and expand our capabilities for this operation. We can certainly leverage our finishing capabilities between our two companies as a competitive advantage.

MIM on the other hand is a comparatively young technology and still evolving. I feel there is quite a bit of latitude for further improvements to be made in making larger part sizes and with general tolerance control. MIM has the capability to offer a much higher level of detail over investment casting so elimination of machining (especially in high volume programs) can drive a heavy product to MIM. Powder cost however will ultimately determine this break point and I don't think we are going to see any technological advancement that will significantly reduce powder costs from where they are today.

**Are there any other metal forming processes under consideration by Indo-MIM?**
Besides the considerable investments we have made in precision machining for aerospace, we do not have any immediate plans to add other metal forming operations. I can foresee where we could have interest in conventional PM, which is a complementary use of our sintering assets. We have also made small investments in equipment and people to expand into CIM. Personally, I am keeping a close eye on the various additive manufacturing processes, which I think are very exciting and will be the next frontier ■

# MIM at PowderMet 2011: Advances in the metal injection moulding of titanium

The Metal Powder Industries Federation's annual PowderMet conference once again proved to be an important event for the PIM industry. PowderMet 2011, held in San Francisco earlier this year, was attended by more than 750 delegates and attracted a large number of papers relating to titanium powder metallurgy. Dr David Whittaker reports for *PIM International* on a selection of key presentations relating to titanium MIM.

Six sessions on the advances in titanium powder metallurgy formed a thread that ran throughout the last two days of the MPIF's PowderMet 2011 Conference held on 20 and 21 May in San Francisco. The fact that two of these sessions were dedicated specifically to developments in the metal injection moulding (MIM) of titanium and titanium alloys emphasised the growing level of interest in this particular subject.

The papers in these two sessions focused on two broad themes:

- Material developments aimed at optimising the properties of MIM titanium products, with a special emphasis on the fatigue properties of Ti-6Al-4V.
- Process developments to enhance the use of titanium MIM products in biomedical, catalyst, photo-electrochemical reactor and solar cell applications.

**Metal Injection Moulding of Advanced Titanium Alloys**

In the first of these broad themes, a paper from Thomas Ebel, Orley Milagres Ferri, Wolfgang Limberg and Frank-Peter Schimansky (Helmhotz-Zentrum Geesthacht, Germany) covered developments in the MIM processing of the widely used Ti-6Al-4V alloy (wt %) and of the intermetallic Ti-45Al-5Nb-0.2B-0.2C (at %).

It has already been adequately demonstrated that Ti-6Al-4V can be processed by MIM to deliver mechanical properties near to those of wrought material. However, it has been recognised that MIM Ti-6Al-4V has relatively low fatigue strength compared to the standard wrought material because of its residual porosity and coarser microstructure.

Work at the Helmhotz-Zentrum Geesthacht has shown that, in MIM processing of powders produced by the crucible-free EIGA (Electrode Induction Gas Atomisation) technique (Fig. 1), the elemental addition of 0.5% boron results in a significant reduction in sintered grain size and a consequent dramatic improvement in fatigue performance.

During sintering, the boron addition reacts with titanium to form fine TiB particles that act as agents for grain boundary pinning, resulting in the significantly finer grain size, as shown in Table 1.

The measured property data in Table 2 show that (a) the fatigue endurance limit in 4-point bending was increased from 450 MPa to 640 MPa (comparable with wrought material) and, (b) the tensile properties achieved were consistent with the requirements of the ASTM B348 grade 23.

A further interesting outcome from this work was that the required properties of ASTM B348 grades can be

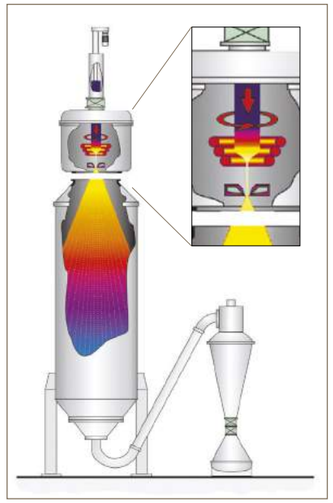

*Fig. 1 Schematic of the Electrode Induction Melting Gas Atomisation (EIGA) technique (Image courtesy ALD Vacuum Technologies GmbH)*

| Material | Porosity (%) | Grain size (µm) | O (µg/g) | N (µg/g) | C (µg/g) |
|---|---|---|---|---|---|
| Ti-6Al-4V, 1400°C (2552°F) | 3.3 | 247 | 1917 | 147 | 577 |
| Ti-6Al-4V, 1350°C (2462°F) | 3.6 | 148 | 2318 | 172 | 409 |
| Ti-6Al-4V-0.5B, 1400°C | 2.3 | 18 | 1960 | 164 | 390 |
| Ti-6Al-4V, 1350°C + HIP | n/a | 147 | 2308 | 177 | 480 |

*Table 1 Microstructural data for samples processed by MIM. From presentation by Thomas Ebel at PowderMet 2011 (Courtesy MPIF)*

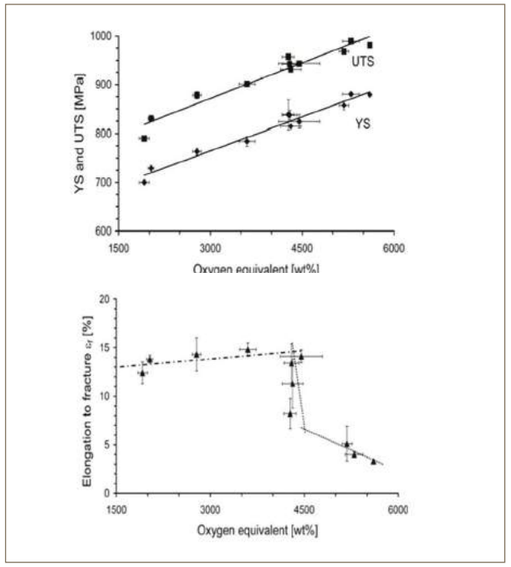


*Fig. 2 Dependence of strength and ductility of MIM Ti-6Al-4V on oxygen equivalent. From presentation by Thomas Ebel at PowderMet 2011 (Courtesy MPIF)*

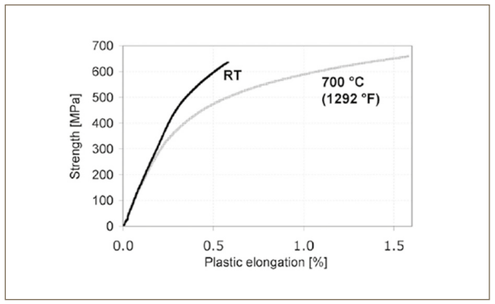


*Fig. 3 Tensile test results at room temperature and 700oC for Ti-45Al-5Nb-0.2B-0.2C (at %) processed by MIM. From presentation by Thomas Ebel at PowderMet 2011 (Courtesy MPIF)*

achieved at interstitial levels well above the compositional requirements of these grades. As shown in Fig. 2, no reduction in ductility is observed until an oxygen equivalent of over 0.4 wt% (4000 ppm) is reached, while strength increases linearly with oxygen equivalent. The oxygen equivalent is calculated as Oeq = O + 2N + 0.75C and takes into account the relative effects of all three interstitial elements.

The application of titanium aluminides has been constrained by the difficulties in shaping them and so the net-shape capabilities of MIM have attracted increasing interest for the processing of these materials. The work reported in this paper has shown that, by using a high-quality powder feedstock and by applying an optimised sintering process, MIM of Ti-45Al-5Nb-0.2B-0.2C can deliver a tensile strength of around 600 MPa at 700°C (Fig. 3), comparable to cast material.

**Dynamic Fracture Characteristics of Injection Moulded Ti Alloys Compacts**

The potential of an alternative compositional adjustment to enhance the fatigue properties of MIM Ti-6Al-4V was assessed in a paper from Hideshi Miura, Munehiro Noda and Hyungoo Kang (Kyushu University, Japan). An elemental addition of 4 wt % Cr was made. However, in contrast to the effects of the boron addition, no significant grain refinement was created (the average grain sizes of Ti-6Al-4V and of Ti-6Al-4V-4Cr both exceeded 100µm) and only a modest increase of 30 MPa in rotating bend fatigue endurance limit (Fig 4) resulted.

| Material | YS (MPa ($10^3$ psi)) | UTS (MPa ($10^3$ psi)) | $\varepsilon_f$ (%) | Endurance limit (MPa ($10^3$ psi)) |
|---|---|---|---|---|
| Ti-6Al-4V, 1400°C | 744 (108) | 852 (123) | 15 | n/a |
| Ti-6Al-4V, 1350°C | 720 (104) | 824 (119) | 13 | 450 (65) |
| Ti-6Al-4V-0.5B | 787 (114) | 902 (131) | 12 | 640 (93) |
| Ti-6Al-4V + HIP | 841 (122) | 937 (136) | 17 | 500 (72) |
| ASTM B348 grade 23 | >760 (110) | >825 (120) | >10 | n/a |
| ASTM B348 grade 5 | >828 (120) | >895 (130) | >10 | n/a |
| Typical range of wrought Ti-6Al-4V alloy | | | | 500 to 800 (72 to 116) |

*Table 2 Properties of samples processed by MIM. From presentation by Thomas Ebel at PowderMet 2011 (Courtesy MPIF)*





The authors concluded that the remaining large internal pores were the dominant factor influencing fatigue endurance and these should be eliminated (perhaps by a subsequent HIP treatment) to improve fatigue endurance.

**Controlling the interstitial content of MIM titanium products**

The important issue of controlling the interstitial content of MIM titanium products was returned to in a paper from Ying Shengjie (Dou Yee Technologies Pte. Ltd., Singapore). This paper reported on a development where a high purity titanium powder (0.17% O, 0.03%C, 0.03%N) was mixed with a "unique" polymer/wax-based low melting point binder, comprising constituents with melting points below 130°C. Moulded specimens were then subjected to a two-stage debinding process; solvent debinding in hexane for 10 hours, followed by thermal debinding in vacuum at less than $10^{-3}$ torr, followed by vacuum sintering at $10^{-6}$ torr and 1200°C for 3 hours.

During the MIM processing, the oxygen content rose to 0.248% and the carbon content to 0.052%. Based on the formula quoted in the paper from Ebel *et al*, the final oxygen equivalent was around 0.35%.

**Development of MIM Titanium Alloys for Implantable Biomedical Devices**

The theme of MIM process development for biomedical implant applications was introduced in a contribution from Alan Sago, Mark Broadley and John Eckert (Accellent Inc., USA). Accellent's developments have focussed on a broad range of implantable components, mainly in Ti-6Al-4V.

As an aid to targeting some of these applications, Accel-

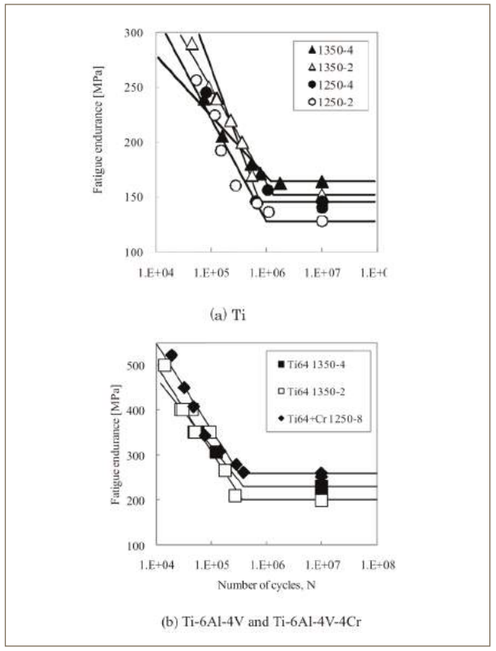


*Fig 4 – Fatigue endurance of MIM Ti (a), Ti-6Al-4V and Ti-6Al-4V-4Cr (b). From presentation by Hideshi Miura at PowderMet 2011 (Courtesy MPIF)*

| Test type | Test method | Test description | Results |
|---|---|---|---|
| Hemocompatibility | ISO 10993-4 | Human blood | Pass – non hemolytic |
| Cytotoxicity | ISO 10993-5 | ISO MEM Elution Cytotoxicity | Pass – non toxic |
| Sensitisation test | ISO 10993-10 | Kligman maximisation | Pass – non sensitiser |
| Irritation test | ISO 10993-10 | Intracutaneous reactivity test | Pass – non irritant |
| Systemic toxicity | ISO 10993-11 | Acute systemic injection | Pass – no adverse effects |
| Systemic toxicity | ---------- | 14 day sub-acute repeat dose intravenous toxicity | Pass – no adverse effects |
| Implantation | ASTM F763 | Implant – bone/4 weeks with histology | Pass |
| Implantation | ASTM F981 | Implant – bone/26 weeks with histology | Pass |
| Implantation | ISO 10993-6 | Implant – muscle/12 weeks with histology | Pass |

*Table 3 Biocompatibility testing of MIM Ti-6Al-4V. From presentation by Alan Sago at PowderMet 2011 (Courtesy Alan Sago, Accellent Inc.)*

lent have been involved in two areas of process development to extend MIM product capabilities.

Direct compression moulding has been developed as a process variant that can significantly extend MIM's size capability. This process can produce large sections up to 50-75 mm, is resistant to large void formation and incorporates a novel binder system that reduces debinding times and improves dimensional stability. The moulds are simpler and lower cost and consequently support lower piece count applications. The competitive capability of direct compression moulding is compared with a range of other processes, including conventional MIM, in Fig. 5.

MIM Forging is a hybrid process, where forging improves the performance capabilities of the MIM product and the use of a MIM preform reduces forging costs. Proof of concept of this process is claimed to be nearing completion and product applications are now under development.

To support the application engineering of its biomedical products, the company also carries out its own biocompatibility tests. Initial tests have been on MIM Ti-6Al-4V and ASTM F1472 wrought alloy control samples. Results have shown an acceptable level of biological response (Table 3).



**Processing of Titanium-based Materials from Titanium Hydride Powders: PIM and Tape Casting**

A paper from Efrain Carreno-Morelli and Jacques-Eric Bideaux (University of Applied Sciences Western Switzerland, Sion, Switzerland) presented a suite of new developments for the processing of titanium and titanium-nickel materials from titanium hydride powders.

Feedstocks for MIM were prepared with binders, comprising polyethylene, paraffin wax and stearic acid. Solvent debinding was carried out in heptane, followed by thermal debinding and dehydrogenation at 500°C in an argon atmosphere. Titanium MIM parts were then sintered at 1200°C, again in an argon atmosphere. The as-sintered UTS

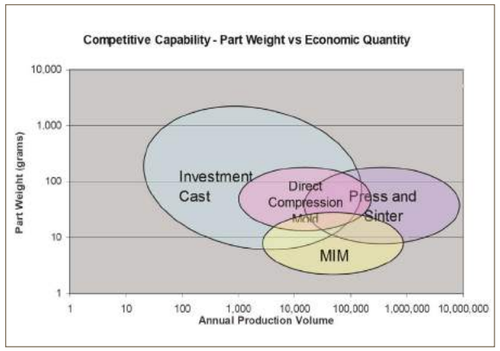


*Fig. 5 Competitive capability of direct compression moulding versus other processes. From presentation by Alan Sago at PowderMet 2011 (Courtesy Alan Sago, Accellent Inc.)*





(650 MPa) and elongation to fracture (15%) met the requirements for titanium grade 4. Watch bracelet segments (Fig. 6) were injection moulded, showing good shape retention and reproducibility.

Shape memory alloy titanium-nickel debound and dehydrogenated parts were sintered under argon at temperatures from 800°C to 1200°C to leave controlled porosity levels from 31 to 43%. Among the potential applications of porous titanium-nickel shape memory alloys is the fabrication of biomedical implants such as vertebral fusion devices or bone tissue scaffolds. Such applications are based on the materials' biocompatibility and low elastic modulus, which can be tuned to match that of human bone to avoid undesirable stress shielding problems. Where larger pore sizes are required to also facilitate osteo-integration, this could be achieved by using appropriate space holder additions such as NaCl or PMMA, which can be removed using solvent or thermal treatments.

By tape casting of slurries consisting of powder, solvent, plasticiser and binder to produce green tapes and then debinding and sintering, porous titanium thin sheets can be produced with 25% porosity (Figs. 7 and 8). Potential applications for such products include catalyst supports, photo-electrochemical reactors and organically modified solar cells.

**Titanium Powder Metallurgy – The merits of press-sinter and metal powder injection moulding**

Professor Randall German (San Diego State University, USA) reviewed the issues, which have driven interest in PM processing of titanium and titanium alloy powders, and the relative positions of conventional Press/Sinter PM and Metal Injection Moulding (MIM) in this area.

This presentation began by identifying the potential advantages of using titanium e.g. high strength to weight ratio, excellent corrosion and oxidation resistance, moderate temperature capability, biocompatibility. These advantages have ensured that titanium is an important material in the aerospace and chemical processing sectors and in certain specialist applications such as medical implants.

Overall, however, despite significant marketing hype, titanium has remained very much a niche material with sponge production amounting to only around 220,000 tonnes per year.

More extensive application of titanium has been limited by two major issues, the high raw material costs (e.g. mill products were quoted as selling for around $100 per kg) and costs and poor material efficiency of conventional manufacturing involving significant machining (the "fly-to-buy" ratios in aerospace titanium applications are often below 10%).

Much attention has been focused on the raft of new titanium powder production technologies that may offer lower-cost powders. However, Professor German argued strongly that this alone may not enable new titanium applications. There is a need for more material-efficient, net-shape forming methods, hence the interest in PM technologies.

In one guise or another, PM processing of titanium has been investigated for over 50 years. Initial publications in 1955 referred to the use of Spark Plasma Sintering (SPS), the use of HIP (Hot Isostatic Pressing) consolidation first appeared in 1972, work on Press/Sinter PM processing was first reported in 1977 and, more than a decade later (1988), MIM processing of titanium was first referred to.

Despite its being the "new kid on the block", reported work on MIM of titanium has, however, assumed a dominant

*Fig. 6 Titanium watch bracelets segments processed fromTiH2 based feedstock. From presentation by Efrain Carreno-Morelli at PowderMet 2011 (Courtesy MPIF)*

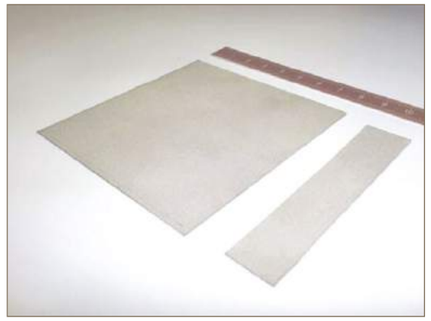

*Fig. 7 As-sintered thin sheets of porous titanium processed by tape casting. From presentation by Efrain Carreno-Morelli at PowderMet 2011 (Courtesy MPIF)*

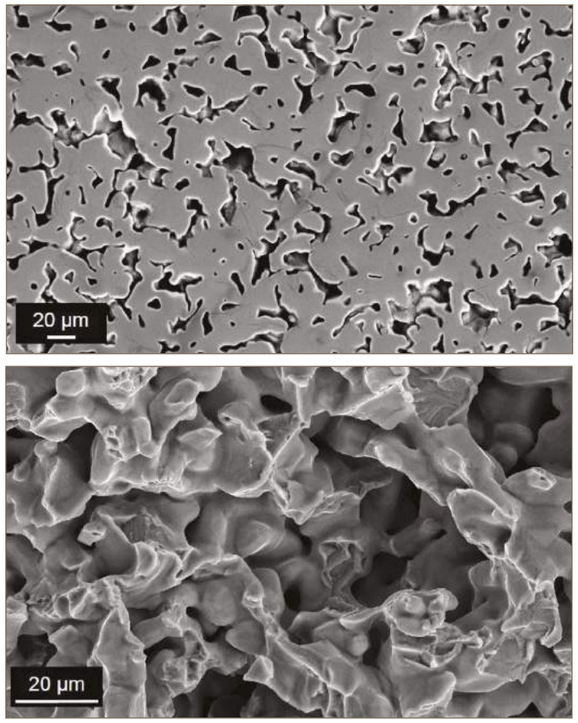


*Fig. 8 SEM observation of the porous structure of tape cast sintered titanium: polished cross section (top) and fracture surface (bottom). From presentation by Efrain Carreno-Morelli at PowderMet 2011 (Courtesy MPIF)*

position in the sintered titanium literature (Fig 9). Much knowledge on this process route has been generated in a shorter time than for the other PM technologies.

Professor German compared and contrasted the current status of MIM and Press/Sinter PM of titanium, in terms of a range of issues:

Powders

Powders are available from multiple vendors for both routes. Because of the requirement for finer powder fractions, MIM powders are relatively more costly.

Processing

Processing parameters for MIM production of titanium parts are understood and several MIM plants have relevant equipment installed and are producing titanium parts. On the other hand, although the required processing parameters for Press/Sinter titanium are understood, few PM shops are involved, products are mainly filters or briquettes and specialist equipment is generally lacking.

Impurities

The control of impurities, particularly of the interstitials O, C and N, is of great importance in developing optimum properties. Means of controlling impurity levels in MIM titanium products have now been well developed, whereas the equivalent control in Press/Sinter PM products is not yet available.

*'Means of controlling impurity levels in MIM titanium products have now been well developed'*

Microstructure

Microstructure is a very important consideration with titanium alloys. Both PM technologies offer good microstructural control, but residual porosity is, of course, higher for Press/Sinter than for MIM. For Press/Sinter PM, better density levels can be achieved with TiH2 powders and blended elemental additions and products can generally be densified with a subsequent HIP operation.

Properties

Properties of MIM titanium products are rated as "competitive" with wrought products, whereas the properties of Press/Sinter products are degraded by porosity and impurities.

Financial considerations

Costs of MIM titanium products, on a $/kg basis, are high but, despite high powder prices, markets are showing slow growth. It was claimed that there is no adequate information to benchmark the costs of Press/Sinter titanium products, but that substantial powder price reductions are required to stimulate prospects for increased market penetration.

For a combination of these reasons, MIM titanium has proved much more successful than Press/Sinter PM in establishing a range of high value applications. This is evidenced by the relative titanium powder usage figures in Fig 10. (The higher figure for HIP arises because many of the aerospace applications, e.g. engine casings, are large, heavy parts.)

Early MIM titanium applications were in non-stressed, often fashion, items, such as watch bands.

The second wave of applications took advantage of some of titanium's performance benefits (such as corrosion resistance or specific strength), e.g. surgical tools. These applications used standard alloys such as CP Ti or Ti-6Al-4V.

A third wave of MIM applications is now focusing on safety critical applications in aircraft, medical implant, dental, military and automotive markets. Special powder alloys are now being developed, e.g. Ti-7Fe-5Zr, Ti-6Al-5Nb.

## Author

Dr David Whittaker
DW Associates
231 Coalway Road
Wolverhampton, WV3 7NG, UK
Tel: +44 1902 338498
Email: david-dwa@blueyonder.co.uk

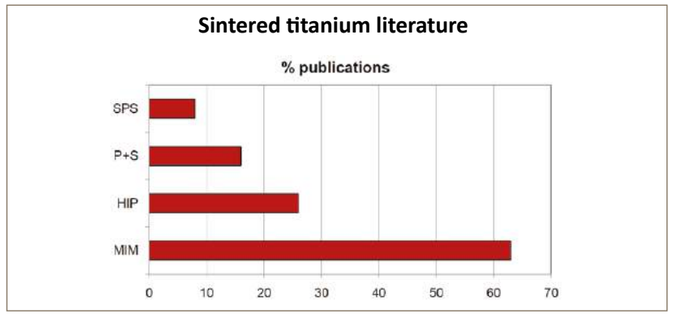


*Fig. 9 The relative contributions of the various PM technologies to the sintered titanium literature. (Courtesy Randall German, San Diego State University)*

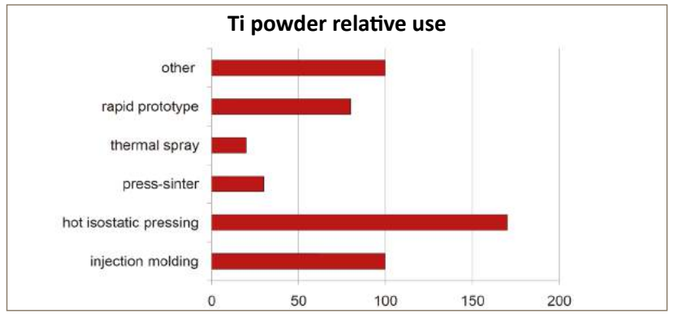


*Fig. 10 Relative use of titanium powder in various technologies. (Courtesy Randall German, San Diego State University)*

# MIM at PowderMet 2011: Advances in ferrous material grades for MIM applications

The PowderMet 2011 technical programme featured a number of presentations dedicated to the latest developments in ferrous material grades for metal injection moulding. From the sinter-hardening of MIM alloy steels, to tool steels and austenitic steels, the range of presentations highlighted both the diversity and continuing potential of MIM. Dr David Whittaker reviews a selection of key papers for *PIM International*.

A group of papers at the MPIF PowderMet 2011 conference, held in San Francisco, 18-21 May, focused on developments of a range of ferrous material grades tailored for Metal Injection Moulding (MIM) application.

**Ultra-high strength sinter-hardening PIM alloy steels**

A paper from Kuen-Shyang Hwang, Chen Hsu and Li-Hua Cheng (National Taiwan University, Taiwan) and Po-Han Chen (Taiwan Powder Technologies Co., Taiwan) reported on a five year development programme that has applied to MIM processing a concept that is well-known and well-established in press/sinter PM – sinter hardening of alloy steels.

Through careful selection of base powder, sintering parameters and the amounts and types of alloying additions, this group has developed a sinter-hardening MIM alloy, Fe-6Ni-0.8Cr-0.8Mo-0.4C, with a good combination of ultrahigh strength, high hardness and elongation.

With no quenching treatment, the sintered and tempered alloy has achieved an ultrahigh UTS of 1,900 MPa, a yield strength of 1,480 MPa, a hardness of 50HRC, an elongation of 7.6% and an impact energy of 65J.

Transmission Electron Microscopy (Fig. 2) and XRD analyses (Table 1) have shown that the matrix of the alloy consisted mainly of martensite with around 11% retained austenite. The martensite was mainly of the plate (twinned) type.

Dimensional control of the sintered and tempered Fe-6Ni-0.8Cr-0.8M0-0.4C was much better than that of quenched and tempered Fe-2Ni-0.5Mo-0.4C counterparts. The standard deviation of the 15 mm dimension of an industrial MIM product was reduced by

*Fig. 1 PowderMet 2011 once again proved to be an essential event for the PIM industry, attracting speakers and exhibitors from around the world (Courtesy MPIF)*

| Material | γ content (%) | Feritscope (%) |
|---|---|---|
| Sintered and tempered Fe-6Ni | 11.2 | 66.3 |

*Table 1 Amount of austenite in Fe-6Ni specimen, measured by X-ray diffractometry and amount of ferrite read from Feritscope. From presentation by Kuen-Shyang Hwang at PowderMet 2011 (Courtesy MPIF)*

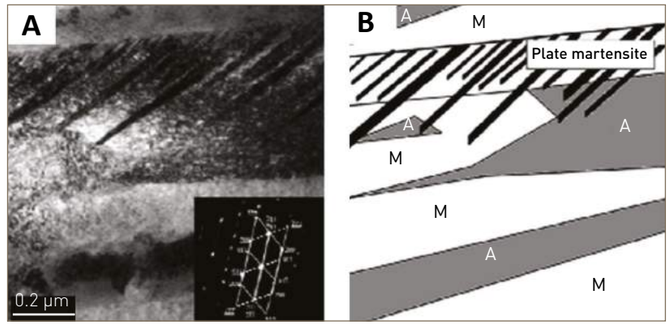


*Fig. 2 (a) Bright field and (b) schematic of tempered Fe-6Ni steel showing plate martensite, lath martensite and retained austenite. From presentation by Kuen-Shyang Hwang at PowderMet 2011 (Courtesy MPIF)*

| Dimension | Material | Length/ flatness (mm) | Standard deviation (mm) |
|---|---|---|---|
| 30mm of flat specimen | Sintered and temperedFe-6Ni | 25.26 | 0.012 |
| | Quenched and tempered Fe-2Ni | 25.24 | 0.016 |
| 15mm width between two walls | Sintered and tempered Fe-6Ni | 14.94 | 0.007 |
| | Quenched and tempered Fe-2Ni | 14.96 | 0.021 |
| Flatness deviation over 43mm width | Sintered and tempered Fe-6Ni | 0.174 | 0.013 |
| | Quenched and tempered Fe-2Ni | 0.229 | 0.047 |

*Table 2 Dimensions and standard deviations of the length and flatness of sinter-hardened and tempered Fe-6Ni and of quenched and tempered Fe-2Ni. From presentation by Kuen-Shyang Hwang at PowderMet 2011 (Courtesy MPIF)*

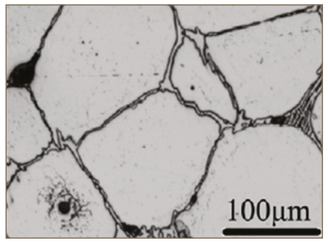


*Fig. 3 AISI D2 sintered at 1300°C for 90 minutes. From presentation by Kuan-Hong Lin at PowderMet 2011 (Courtesy MPIF)*

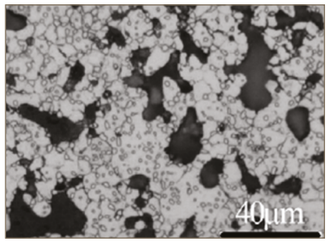


*Fig. 4 AISI D2 sintered at 1200°C for 90 minutes. From presentation by Kuan-Hong Lin at PowderMet 2011 (Courtesy MPIF)*

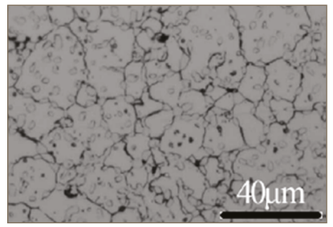


*Fig. 5 AISI D2 sintered at 1235°C for 150 minutes. From presentation by Kuan-Hong Lin at PowderMet 2011 (Courtesy MPIF)*

| Product | Hardness (HV30) | |
|---|---|---|
| | TDS | Measured |
| F75 | >260 | 268 |
| 316L | >120 | 127 |
| Nitronic 50 | >200 | 223 |
| 17-4PH | >320 | 334 |

*Table 3 As-sintered hardnesses of the various grades under comparison. From presentation by Stephanie Schneider at PowderMet 2011 (Courtesy MPIF)*

and electron probe micro-analysis assessments have shown that nickel, chromium and molybdenum are all uniformly distributed.

**Effect of sintering temperature on the microstructure and mechanical properties of MIM SKD11 component**

Kuan-Hong Lin and Yu-Xiang Sun (Tungnan University, Taiwan) and Yu-Chan Hsieh (National Taiwan University of Science and Technology, Taiwan) reported on the sintering response and mechanical properties of a MIM component produced from the tool steel, AISI D2.

The sintering of this type of powder is driven by the creation of a supersolidus liquid phase. As demonstrated in this study, there was a relatively narrow window for good sintering response. At high sintering temperatures above this window, grain coarsening occurred, carbide precipitates formed at grain boundaries (Fig. 3) and excessive liquid phase triggered distortion and slumping of the sintered specimen. On the other hand, at low sintering temperatures below the window, there was a lack of densification because of a deficiency of liquid phase (Fig. 4). The optimum sintering parameters were identified as being 150 minutes at 1235°C. Under these conditions, the sintered microstructure was close to full densification, grain size was not obviously changed and the precipitated $Cr_7C_3$ carbides were not enlarged and coalesced (Fig. 5).

The specimen sintered at 1235°C for 150 minutes had the highest hardness (Fig. 6) and therefore showed the optimum wear resistance (Fig. 7).

**Nitronic 50: An austenic steel with near perfect properties**

A paper by Stephanie Schneider (PolyMIM GmbH, Germany) focused on the dominant MIM material type, stainless steel.





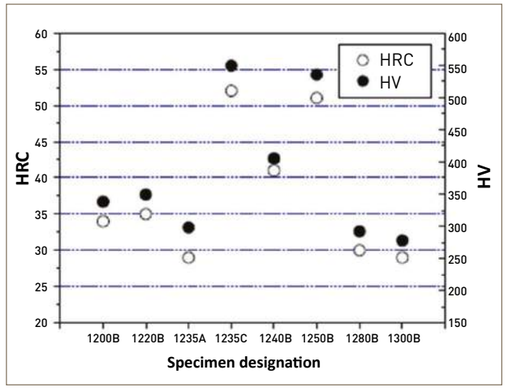


*Fig. 6 Surface hardness of AIS D2 specimens under various sintering conditions. From presentation by Kuan-Hong Lin at PowderMet 2011 (Courtesy MPIF)*

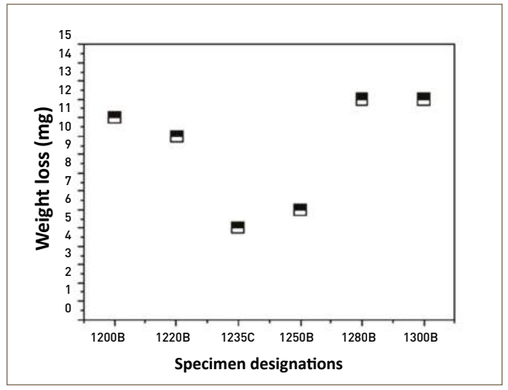


*Fig. 7 Weight loss in wear testing of the various sintered D2 specimens. From presentation by Kuan-Hong Lin at PowderMet 2011 (Courtesy MPIF)*

The two most commonly used stainless steels in MIM are the austenitic AISI 316L and the precipitation hardening AISI 17-4PH. Both of these grades offer certain advantages but also carry limitations. AISI 316L has excellent corrosion resistance and is non-magnetic (relevant to some applications), but has limited strength. On the other hand, AISI 17-4PH can provide higher strength but is magnetic and has limited corrosion resistance.

This paper reported on a stainless steel grade, Nitronic 50, that has been developed to offer excellent corrosion resistance, to be non-magnetic and to provide increased strength compared with AISI 316L. The compositional specification limits of Nitronic 50 are 20.5-23.5%Cr, 11.5-12.5%Ni, 4.0-6.0%Mn, 1.5-3.0%Mo, 0.1-0.3%V, <1.0%Si, <0.06%C, balance Fe.

The corrosion resistance, magnetic and tensile properties of Nitronic 50 were compared with those of 17-4PH, 316L and of ASTM F75, a cobalt-chromium steel, which has excellent biocompatibility, is used extensively for biomedical implants and has excellent corrosion resistance.

Nitronic 50 showed increased tensile strength and hardness (Table 3) compared with 316L, had good

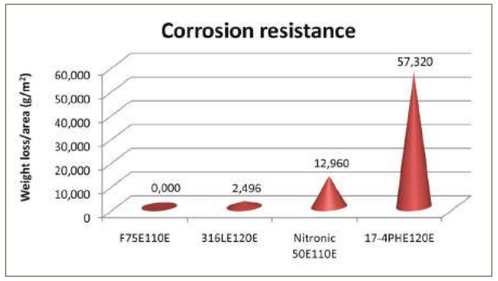


*Fig. 8 Comparison of the corrosion resistances of the grades evaluated. From presentation by Stephanie Schneider at PowderMet 2011 (Courtesy MPIF)*

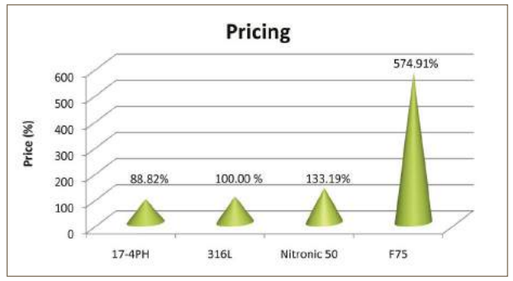


*Fig. 9 Comparative raw material prices for the grades evaluated (316L set as 100%). From presentation by Stephanie Schneider at PowderMet 2011 (Courtesy MPIF)*

corrosion resistance (Fig. 8) and, unlike 17-4PH was non-magnetic.

The paper also compared the raw material prices of the four grades evaluated (Fig. 9). Although Nitronic 50 has a material price around 33% higher than 316L, it is believed that its attractive combination of properties will make it viable for a broad range of applications. F75, as a material for high-end applications, has a significantly higher price than any of the other grades.

**Properties of 4340 and 4140 MIM parts made via prealloy and master alloy routes**

A paper from Keith Murray, Andrew Coleman and Martin Kearns (Sandvik Osprey Ltd. UK), Toby Tingskog (Sandvik Osprey Ltd., USA) and Bob Sanford and Erainey Gonzales (TCK S.A., Dominican Republic) concentrated on the low alloy steel grades, AISI 4140 and AISI 4340, and assessed the influence of particle size distribution on the processing response during MIM and on the resultant properties. As this subject is covered elsewhere in this issue, only a brief summary is given below.

The study compared 4140 and 4340 parts made using gas atomised pre-alloyed (PA) powders at 90%-16μm and 90%-22μm and a three-times concentration master-alloy (MA) powder (90%-22μm) blended with carbonyl iron powder (CIP).

It was demonstrated that both the PA and MA+CIP feedstocks could be successfully processed to deliver mechanical properties exceeding published values for MIM products. However, the process window for the 4140 MA+CIP product was significantly wider than that for the PA product, with sintering temperatures as low as 1100°C able to give acceptable properties compared with >1200°C for PA. This has been attributed to the earlier onset of sintering, promoted by the fine CIP addition, leading to earlier densification. For the MA+CIP materials, the sintering cycle must also be designed to achieve a homogeneous microstructure. The MA+CIP route offers advantages in terms of lower distortion for a fixed powder loading.

Higher than anticipated porosity levels in the 4340 alloy were claimed as having a deleterious effect on the reported mechanical properties and further work is ongoing to address this situation. Tempering at 250°C led to a doubling of strength of 4140 and around a 60% increase in strength for 4340.

Some MIM manufacturers, familiar with PA materials and with a good understanding of their behaviour, may choose not to have to carry out an additional blending operation with CIP. For those considering the change of material, however, this study has demonstrated that concerns over carbon control with the MA+CIP route are unfounded. For low alloy steels in general, there could be cost advantages in using MA as well as advantages in lower distortion and superior mechanical properties.



## Author

Dr David Whittaker
DW Associates, 231 Coalway Road, Wolverhampton, WV3 7NG, United Kingdom
Tel: +44 1902 338498
Email: david-dwa@blueyonder.co.uk





# PowderMet 2011 San Francisco

*More than 750 delegates attended the MPIF's International Conference on Powder Metallurgy & Particulate Materials, this year held in San Francisco, USA.*

With the PM industry having benefitted from effects of a broad manufacturing recovery in 2010, San Francisco turned out to be the perfect host for the PowderMet2011 and co-located Tungsten, Refractory and Hardmaterials conferences. More than 750 delegates, an increase of 25% over the attendance at the 2009 and 2010 conferences, made the most of their time in the "City by the Bay" by attending the highly diverse technical program, visiting the trade exhibition which featured the latest advances in equipment, materials, and services, and enjoying a unique "Escape to Alcatraz" social event.

At the Opening General Session, MPIF/APMI Executive Director/CEO C. James Trombino welcomed the group, and MPIF President Michael E. Lutheran, Royal Metal Powders, Inc., presented an overview of the State of the North American PM Industry. PowderMet2011 Technical Program Chairmen Iver E. Anderson, FAPMI, Ames Laboratory/Iowa State University, and Thomas W. Pelletiers, SCM Metal products, Inc., and Tungsten Technical Program Chairmen Animesh Bose, FAPMI, Materials Processing, Inc., Robert Dowding, U.S. Army Research Laboratory, and John L. Johnson, ATI Engineered Products, were collectively recognised for assembling the highly regarded program of technical sessions, special interest programs, and posters.

The Keynote Presentation, "Flawless Execution" by Afterburner, Inc., was a high-energy multimedia presentation about how fighter pilots operate in a combat environment utilising a simple 4-step approach - plan, brief, execute and debrief - and how companies within the PM industry can apply those lessons to our businesses.

*The next event in the series, PowderMet2012, will take place in Nashville, TN, USA, from June 10 -13, 2012. For more information contact MPIF.*

*The busy PM exhibition at PowderMet 2011 (Courtesy MPIF)*


*Delegates enjoy a visit to the infamous prison on Alcatraz (Courtesy MPIF)*

*MPIF / APMI 2011 award winners at PowderMet 2011 (Courtesy MPIF)*

*The busy PM exhibition at PowderMet 2011 (Courtesy MPIF)*

# Metal injection moulding of Inconel 713C for turbocharger applications

In response to growing interest in the manufacture of high performance turbocharger components via Metal Injection Moulding, PolyMIM GmbH based in Bad Sobernheim, Germany, has started the production of an Inconel 713C feedstock specifically targeted towards the turbocharger market. In the following article, Dr. Natalie Salk presents the company's most recent data on the processing and properties of tensile test bars processed using a water soluble binder system.

A turbocharger is a device designed to increase the air pressure within an internal combustion engine, allowing an enhanced quantity of fuel to be burned at each stroke. This significantly raises the power output of the engine and therefore the engine performance. The turbocharger is powered by a compressor driven turbine, which in turn is driven by the exhaust gases of the respective engine.

These extreme conditions require materials with excellent mechanical properties as well as good oxidation resistance at high temperatures. Here only the superalloys are applicable.

Superalloys [1] are high-performance nickel-base alloys that exhibit excellent mechanical strength and creep resistance at high temperatures, good surface stability, corrosion and oxidation resistance [1].

This material class is mainly produced by investment casting, direct solidification [2], powder metallurgy via hot isostatic pressing, or spray forming [3]. However, powder metallurgy, and here, metal injection moulding (MIM) technology still play a relatively minor role in the production of superalloys.


*Fig. 1 Inconel 713C green parts (left) and sintered tensile test parts (right)*

*'MIM technology still plays a relatively minor role in the production of superalloys'*

To overcome this and to exploit the advantages of MIM technology, such as, geometrical complexity, cost effective mass production and material variety, PolyMIM GmbH has developed a new feedstock based on the superalloy Inconel 713C.

In the following tests this material was injection moulded, debound and sintered, and material properties such as density, tensile strength at temperatures up to 1000°C and metallography were examined.

## Experimental procedure

A gas atomised pre-alloyed powder with a particle size of 85-22 µm was used. The powder was mixed on a twin

| Elements | Theoretical composition [%] | Powder analysis TDS [%] | Composition as sintered [%] |
|---|---|---|---|
| Fe | <2.5 | 1.80 | 2.06 |
| C | 0.08-0.2 | 0.12 | 0.112 |
| Si | <0.5 | 0.47 | 0.469 |
| Mn | <0.25 | 0.05 | 0.046 |
| P | <0.015 | -- | 0.00099 |
| S | <0.015 | -- | 0.0003 |
| B | 0.005-0.015 | 0.01 | 0.0145 |
| Cr | 12.0-14.0 | 13.0 | 12.83 |
| Mo | 3.8-5.2 | 4.3 | 3.85 |
| Ni | Bal. | Bal. | 71.26 |
| Al | 5.5-6.5 | 5.8 | 6.29 |
| Co | <1.0 | -- | 0.025 |
| Cu | <0.5 | 0.01 | 0.021 |
| Nb | 1.8-2.8 | 2.4 | 2.17 |
| Ti | 0.5-1.0 | 0.81 | 0.683 |
| Zr | 0.05-0.15 | 0.11 | 0.099 |

*Table 1 Summary of the theoretical composition of IN713C, the powder composition from the TDS and the measured sintered material*

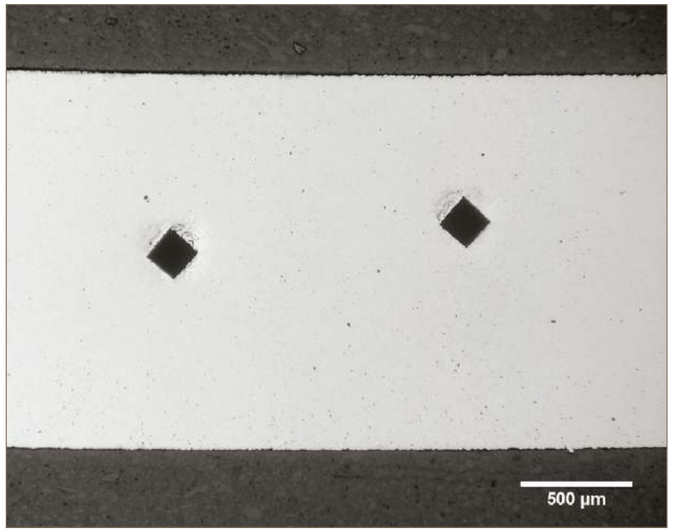


*Fig. 2 Micrograph of the Vickers indentation marks in the polished IN713C sample*

| Sample | $Rp_{0.2}$ [MPa] | $R_m$ [MPa] | A* [%] |
|---|---|---|---|
| IN713C as sintered | 829.5 | 1319.4 | 16.4 |

*Table 2 Tensile test results at room temperature, with $Rp_{0.2}$ as tensile strength at 0.2% non-proportional elongation, $R_m$ as ultimate tensile strength and A* as elongation*

*Fig. 3 Zwick hot tensile test machine located at the Fraunhofer IWU*

screw extruder with the water soluble binder system developed by PolyMIM GmbH. The material was injection moulded on an Arburg 320C machine into tensile test bars (Fig. 1).

The theoretical composition of the Inconel 713C, the composition of the powder analysis provided by the technical data sheet (TDS) of the powder supplier and the measured composition of the sintered test specimens are illustrated in Table 1. As can be seen, all measured values are within the theoretical ranges given for the Inconel 713C composition.

The tensile test bars were placed in a water bath for 24 hours, dried at 80°C for 2 hours and subsequently thermally debound at a heating rate of 300 K/h to 600°C. The holding time was 1 hour. Another ramp of 300 K/h led to the sintering temperature of 1310°C. The samples were held at this temperature for 1 hour, before being cooled at a rate of 900 K/h to room temperature. All process steps were performed under 300 L/h pure hydrogen.

## Characterisation of material properties

To characterise the material properties, the density of the as-sintered tensile test bars was determined. This was established as 7.856 g/cm$^3$. The hardness of the IN713C was measured by applying the Vickers method. Values of 355 HV10 were obtained for the as-sintered material. An image of the indentation marks is shown in Fig. 2.

In further experiments the tensile properties of all samples were measured using a proTens TWIN tensile test machine at room temperature. The results are given in Table 2.

In order to obtain information about the material properties at elevated temperatures, tensile tests were performed at temperatures of 650°C, 850°C and 1000°C. Fig. 3 illustrates the tensile test machine, manufactured by Zwick GmbH & Co. KG, used for hot tensile testing performed at Fraunhofer IWU.

The tensile test bars were eroded on two sides to obtain a flat surface. A picture of the test specimen can be seen in Fig. 4. This reduction in size was necessary to improve the fixation of the test samples in the tensile test machine and to enable the test bars to be broken. A broken sample and the modified fixation are shown in Fig. 5.

The results of the tensile tests at elevated temperatures are illustrated in Table 3; the corresponding graph is shown in Fig. 6.

At temperatures up to 650°C an exceptional strength of approximately 1000 MPa was obtained. As expected this value is reduced by increasing temperature to $R_m$ = 493 MPa for 850°C and to 170 MPa at 1000°C, respectively. Klöden et al. [4] measured the tensile strength at temperatures of 900°C on HIP IN713C samples. They obtained values of $Rp_{02}$ = 373 MPa and $R_m$ = 455 MPa. These values corroborate well with our measured values at 850°C. It is important to note that the tensile properties of [4] were measured by using HIP samples whereas our results

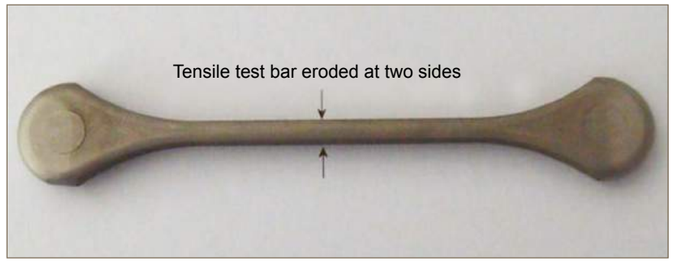


*Fig. 4 Hot tensile tests were only possible after reducing the thickness of the test bar to 2 mm by erosion*

*Fig. 5 Broken IN713C sample after the hot tensile test (left) and modified fixation (right)*

| Temperature [°C] | $Rp_{0.2}$ [MPa] | $R_m$ [MPa] |
|---|---|---|
| 650 | 720 | 997 |
| 850 | 340 | 493 |
| 1000 | 135 | 170 |

*Table 3 Results of the tensile tests at temperatures of 650°C, 850°C and 1000°C. The tensile tests were performed by applying the as-sintered samples*

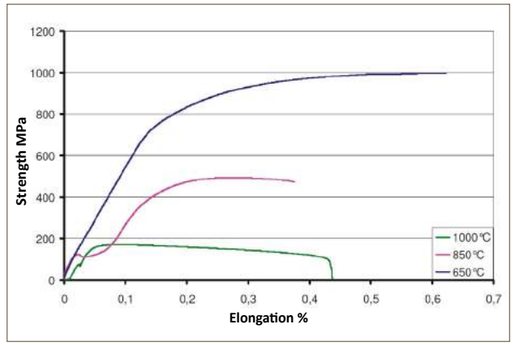


*Fig. 6 Graph of the determined tensile properties of IN713C at temperatures of 650°C, 850°C and 1000°C*

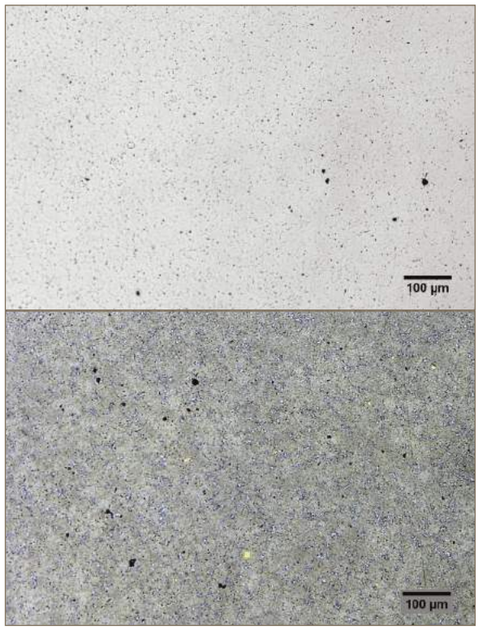


*Fig. 7 Micrographs of the polished (top) and etched (bottom) IN713C material; both pictures illustrate an equal magnification*

are based on as-sintered samples. If a HIPping step was to be performed on the Inconel 713C material presented here, the tensile properties would be further increased.

Micrographs of both an etched and un-etched surface of the IN713C are illustrated in Fig. 7. The overall surface structure of the material is homogeneous showing only a small residual porosity.

## Conclusions and outlook

Turbocharger materials such as the Inconel 713C examined here are widely used in industry. Automotive and aerospace applications in particular, have an increasing need for superalloy materials, since excellent material properties at high temperatures are required.

To illustrate the possibilities of metal injection moulding (MIM) technology on the mass production of complex shaped components, MIM processed IN713C materials have been presented and characterised. As-sintered IN713C tensile bars showed exceptional mechanical properties at temperatures up to 1000°C. A good microstructure as well as density was reached. The element analysis of the as-sintered composition was within the theoretical specifications of the Inconel 713C material.

It is predicted that the mechanical properties of hot isostatically pressed samples would result in further improvements to mechanical properties and this has to be evaluated in the near future.

*'the mechanical properties of hot isostatically pressed samples would result in further improvements to mechanical properties'*

## Contact

Dr. Natalie Salk
General Manager
PolyMIM GmbH
Am Gefach
55566 Bad Sobernheim
Germany
Phone: +49 6751 85769 380
Fax: +49 6751 85769 5380
Email: info@polymim.com

Stephanie Schneider
Head of Development
Phone: +49 6751-85769 162
Fax: +49 6751 85769 5162
Email: info@polymim.com

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# Sandvik Osprey: Expanding fine powder production to meet customer demand

Sandvik Osprey, a leader in the production of fine gas atomised powders for MIM, has over the last decade been steadily increasing capacity in an effort to keep up with spiralling demand from part producers. *PIM International* recently visited the company in Neath, South Wales, and spoke with Martin Kearns and Keith Murray about the company's latest expansion plans.

There is just a hint of déjà vu as we meet once more with Sandvik Osprey. Our last visit coincided with the looming recession in early 2009 and here again in 2011, the world is heading into a period of economic uncertainty. At that time Sandvik Osprey remained optimistic about the market outlook and, having weathered the storm successfully in 2009, the company's strong growth leading up to 2009 has been superseded by even higher growth rates in 2010/11.

Sales forecasters faced a difficult prospect in looking ahead to 2010 having to judge how the world would recover from a dramatic decline in business activity with the automotive sector down by typically >30% and consumer markets in a depressed state. "We were fortunate that at a time when certain sectors were contracting, other long term product developments were maturing to keep utilisation levels high," explains Martin Kearns, Powders Group Director. "In 2010, we budgeted for strong double digit growth on a flat 2009 but ultimately, sales growth far exceeded even our most optimistic expectations." He attributes the strong rebound to customers re-stocking, strong recovery in consumer confidence following stimulus packages in the automotive and other sectors and, critically, effective marketing efforts by the MIM industry have increased penetration into conventional metal forming territory. "The speed and extent of the recovery in the MIM market surprised most people and it was clear in Q2 2010 that we would have to accelerate our capital investment programme," he added.

Sandvik Osprey, part of Sandvik Materials Technology (SMT), a division of Sandvik AB, has occupied the same site in Neath since its foundation in 1974 and obtaining additional land has been an essential step towards increasing its capacity. In 2010, the company secured an adjacent site and buildings to increase its footprint by 60% (Fig. 1). The inherited buildings are ideal for housing support functions such as engineering and stores and this has allowed a reorganisation of operations on site which has improved work-flows and generated space to extend existing atomising and sizing process areas.

At the same time, long term infrastructure developments were initiated which have so far doubled the electricity supply to the site and provided up-rated water and gas services. Accomplishing these expansions with minimum disruption to the 24hr manufacturing operations proved highly challenging.

*'marketing efforts by the MIM industry have increased penetration into conventional metal forming territory'*

*Fig. 1 The new building at Sandvik Osprey*

*Fig. 2 The Sandvik Osprey team*

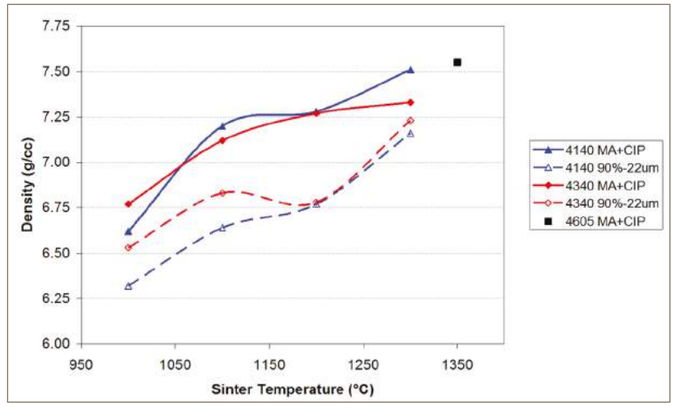


*Fig. 3 Pycnometric density data adjusted for errors between pycnometric tests and values determined by weighing & measuring Charpy bars*

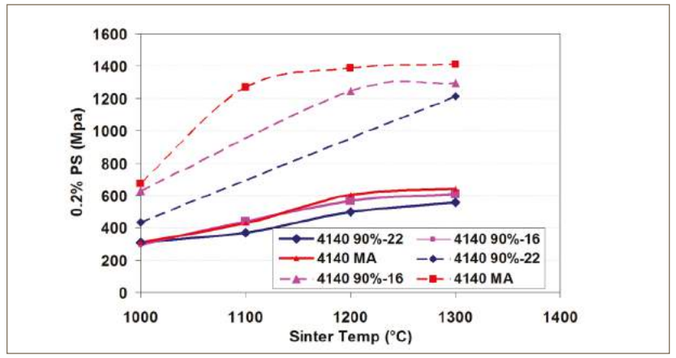


*Fig. 4 0.2% Proof Stress data for alloy 4140 vs. sintering temperature. Dotted lines indicate heat treated values and solid lines are as-sintered*

## New capacity increases approved for 2012

In 2010 a significant increase in fine powder capacity was achieved by conversion of a coarse powder atomiser to make fine powder using the company's proprietary high yield technology and installation of new sieves and classifiers for downstream processing. In parallel, the company recruited and trained a significant number of new personnel to allow full 6 x 24h production on all plants.

There has been no let-up in activity in 2011 with the installation of larger furnaces on fine powder plants, further recruitment and training, and expansion of the powder sizing area by 70% to accommodate extra equipment. Most significantly, the president of SMT, Jonas Gustavsson, has recently approved construction of what will be Sandvik Osprey's largest ever fine powder atomiser and work is well underway to realise this by Q2 2012.

Sandvik Osprey has a long history of building and commissioning atomising plant and while opportunities for efficiency improvements will be taken, the new plant, like previous 'generations', will be home-built to established specifications.

"We know that the last year in particular has been a testing period for the MIM industry in terms of powder supply," stated Dr. Kearns, "and we have worked hard to manage customers' demands and expectations while growing our capacity." The Powders Group has been loyal to established customers and has continued to satisfy some unique product requirements where there could otherwise have been serious consequences for individual customers. The company has also managed to maintain its efforts in new product development albeit the demand for existing products has been immense. Sandvik Osprey forecasts that demand will continue to outstrip supply until Q2 2012 when its new gas atomiser is fully commissioned. "We are looking forward to doing some more proactive selling at that point!" commented Keith Murray, Sales & Marketing Manager, EMEA.

The 2012 growth forecast for fine powder remains very strong, despite the current turmoil in global markets. The intensity of Osprey's expansion efforts in the last year has been a good learning exercise for the future, whatever it may bring.

*'Sandvik Osprey forecasts that demand will continue to outstrip supply until Q2 2012 when its new gas atomiser is fully commissioned'*

## Master alloy strategies

Sandvik Osprey's experience is that, in times of restricted availability, the popularity of master alloys (MA) tends to increase vs conventional prealloys (PA). Blending a 3x or 5x concentration MA with plentiful Carbonyl Iron Powder (CIP) can not only make Osprey powder go further, but can deliver real advantages in terms of mould filling, sintering behaviour and improved tolerances.

In recent years, the benefits of MA's for low alloy steel grades such as 4140, 4340 and 4605 have been examined. These grades are popular in applications including automotive, gun parts and general engineering components. A simple mixture of MA and CIP can simplify complex blends of raw materials leading to lower inventories, lower risk of human error, less likelihood of segregation and greater consistency in processing. While the use of MA's for stainless steels does not always work out cheaper than PA's, it is generally true for low alloy steels and, in both cases, overall cost/performance benefits can be realised. Previous work on stainless steel MA's [1,2] highlighted benefits including:

- Improved green strength
- Activation of sintering at lower temperatures
- Lower part distortion during sintering
- Higher sintered densities and consequently improved mechanical properties and
- Control of chemistry within a narrower range.

In recent collaborative studies [3-4], the sintering behaviour of MA + CIP (Sintez) has been compared with PA of 4140, 4340 and 4605. Feedstock batches made by TCK using their proprietary multi-component wax/polymer binder (61.8 vol% metal loading) were moulded and sintered by TCK to produce MIMA standard tensile test specimens.

Green parts were subject to a two stage debinding process and sintered in a nitrogen atmosphere. Sintering was carried out in the range 1000°C to 1300°C, with a holding time of 2h, followed by cooling in a nitrogen atmosphere. Austenitising was carried out for 1h. at 850°C in the case of 4140 and at 830°C in the case of 4340, followed by oil quenching, tempering for 1h at 250°C then air cooling.

Fig. 3 shows a summary of pycnometric density data for 4140 and 4340. An adjustment has been made from physical measurements to allow for interconnected porosity in samples sintered at low temperatures. The (3x) MA+CIP variants show the highest sintered densities across the full sintering temperature range, followed by the 90%-16µm and then 90%-22µm PA. A data point is also indicated for a 5x 4605 MA+CIP sample showing high density after sintering at 1350°C.

Fig. 4 shows the progression in 0.2% Proof Stress (0.2%PS) for both as-sintered and tempered 4140 samples with increasing sintering temperatures. For all variants, the 0.2%PS and UTS show plateau values around 1300°C. The MA variant achieved the highest strength levels across all sintering temperatures >1000°C in both the as-sintered and tempered conditions. After heat treatment, the MA variant exhibits a pronounced difference in behaviour vs. the PA version, with peak strength levels achieved at significantly lower sintering temperatures. Peak tensile strength levels in the heat treated condition are approximately double those achieved in the as-sintered condition. Fig. 5 presents the range of sintering conditions over which 4140 MA and PA variants exceed the MIMA standard mechanical property values. It shows that the fields for MA are broader than for PA. While satisfactory mechanical properties are achieved at low sintering temperatures, the MA variant only exhibits a homogeneous microstructure at temperatures above 1100°C. It is therefore important that this threshold is exceeded otherwise sintered parts may exhibit some hard spots and localised corrosion.

A similar trend in mechanical properties was observed for 4340 (as-sintered and tempered) vs. sintering temperature. The heat treated samples do not show a plateau in strength level for both the 90% 22µm PA and MA even up to a sintering temperature of 1300°C. This suggests that the sintering and/or heat treatment cycle are not fully optimised. Peak UTS in the heat treated condition is approximately 60% higher than as-sintered.

In the case of the 4140 alloy, it has been demonstrated that the use of a MA-based feedstock can extend the processing window substantially compared with PA. This is due to early onset of sintering conferred by the presence of fine CIP in the MA feedstock. For other alloys more data is being gathered, but a common picture is emerging of the benefits of MA's. Sandvik Osprey offers the widest range of MA's for MIM applications and is flexible in designing bespoke solutions for high volume applications.

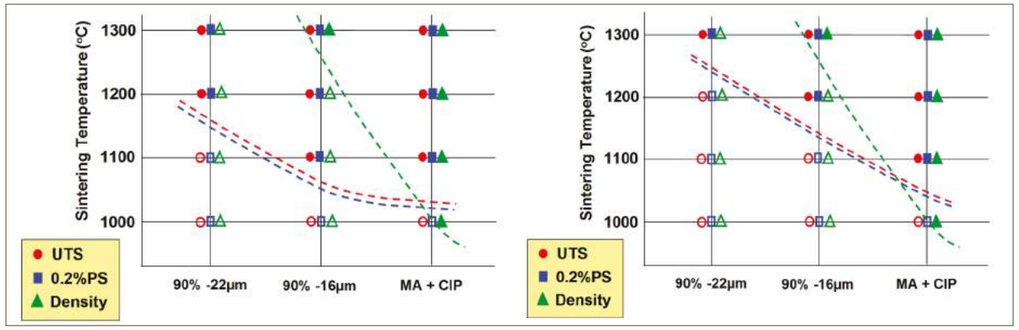


*Fig. 5 Matrix of conditions giving properties greater than; left) as-sintered and right) heat treated MPIF35 published values. Dotted lines indicate thresholds where published values are exceeded. Solid symbols denote combinations where properties exceed published values*

## Contact

Dr Martin Kearns
Sandvik Osprey Limited
Red Jacket Works, Milland Road
Neath SA11 1NJ, UK
Tel: +44 (0)1639 634121
Email: martin.kearns@sandvik.com

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# Improving MIM part quality through enhanced sintering atmosphere control

The production of consistently high quality sintered parts demands a process in which all parameters are closely controlled. Failure to control furnace atmospheres can, therefore, put final part quality at risk. A patented atmosphere control system, SINTERFLEX™, for controlling the C-Potential in sintering furnaces was specifically designed through cooperation between Höganäs AB and Linde Gas. In this article, Akin Malas gives details of the first application of this process to MIM part production, including the results of an industrial scale case study carried out at Megamet Solid Metals Inc.

Research teams from Höganäs AB, Sweden, and Linde Gas, Germany, initially developed a system for the processing of Powder Metallurgy components that helped to make the sintering process more consistent by controlling the carbon potential in furnaces.

The use of this technology has now been extended to undertake carbon control in the sintering of metal injection moulded parts, thereby improving product quality and consistency.

## Introduction

The SINTERFLEX™ system controls the gas composition, particularly its carbon enrichment flow rate, to maintain a reliable carbon potential in sintering furnaces. Knowledge of the oxygen partial pressure in the sintering zone tells the operator the actual conditions during sintering. It can be measured by an oxygen sensor, which determines whether the processed metal is oxidised or whether a metal oxide is reduced. The development of the system focused on two areas: a new furnace atmosphere to introduce carbon into the sintering zone which can be controllable and responsive to any changes; and a new measurement and monitoring system with an oxygen probe to control this carbon containing atmosphere.

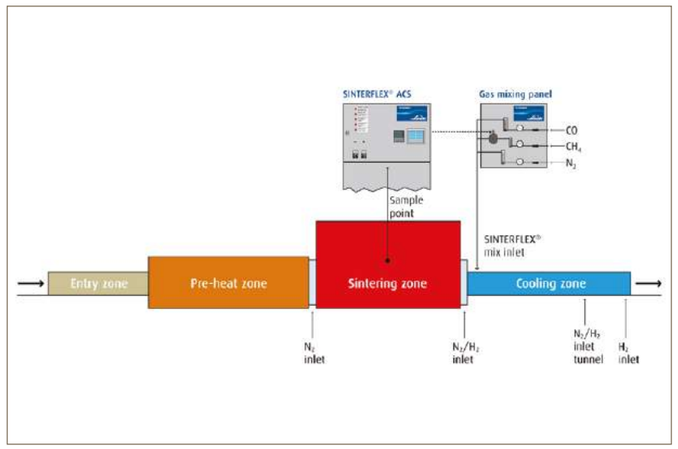



*Fig. 1 A schematic of the SINTERFLEX™ control system*

There are two conflicting requirements for atmospheres used in sinter hardening. Firstly, atmospheres must be very reducing, to ensure that alloy additions do not oxidise, and secondly they must have a carbon potential of around 0.45%. This cannot be achieved with conventional endothermic-type atmospheres. An atmosphere with a carbon monoxide (CO) content of about 2% would be ideal. This low level of CO would result in a very low carbon dioxide concentration at the correct carbon potential - too low to be measured by a conventional infra-red analyser.

In the SINTERFLEX™ system, the carbon potential is calculated from the CO and the oxygen level measured using an external oxygen probe. A schematic of the SINTERFLEX™ control system is shown in Fig. 1.

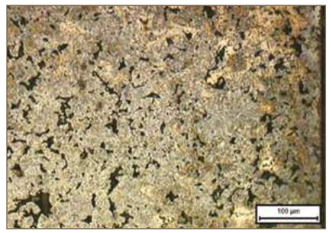


*Fig. 2 The microstructure of a chromium alloy part after sintering*


*Fig. 3 The three MIM parts used in the Megamet trial*

Using an atmosphere of nitrogen with 2% carbon monoxide, with propane as the carbon control gas, it was possible to sinter a chromium-alloyed powder to give a core carbon content of 0.38%, with a surface layer of about 100 µm at a maximum carbon content of 0.45%, an ideal target figure (see Fig. 2). This made the hardening step much easier and lowered overall product cost.

The control system was found to be robust and could rapidly counter the effects of simulated leaks and draughts. It was also effective in several other atmosphere systems including nitrogen/10% hydrogen, with and without a methane addition, and also with a nitrogen/2% carbon monoxide/2% hydrogen atmosphere.

## The challenge of carbon control when sintering MIM parts

Sintering of metal injection moulded parts can be even more challenging as the temperatures are higher. Conventional sintering furnaces use a hydrocarbon addition to maintain a carbon potential. However, in MIM furnace atmospheres this is usually not the case as it produces a high carbon potential leading to sooting and excessive carbide formation.

Carbon control and decarburisation have, therefore, typically been considered as a "common problem" in MIM sintering. MIM furnaces have sealed zones with internal doors, particularly for the pre-heating and sintering zones, making control more difficult.

It is quite common to specify a ±0.3% carbon variation after sintering. It would be a tenth of this in carburising applications, illustrating the size of the problem. Improved carbon control is therefore a major challenge in improving the properties of sintered steels.

## MIM sintering trials at Megamet

The Linde Gas team worked with Megamet Solid Metals Inc based in St Louis, USA, on the application of the SINTERFLEX™ technology it had developed for conventional furnaces to implement carbon control in high temperature MIM sintering furnaces.

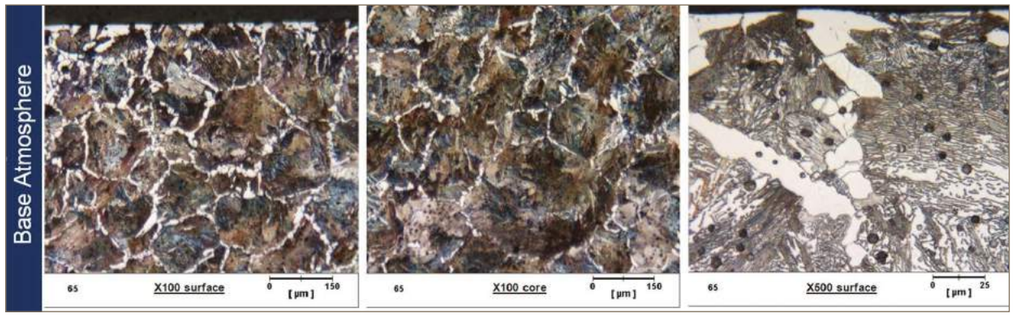



*Fig. 4 Microstructure of a part sintered using the base-line atmosphere*

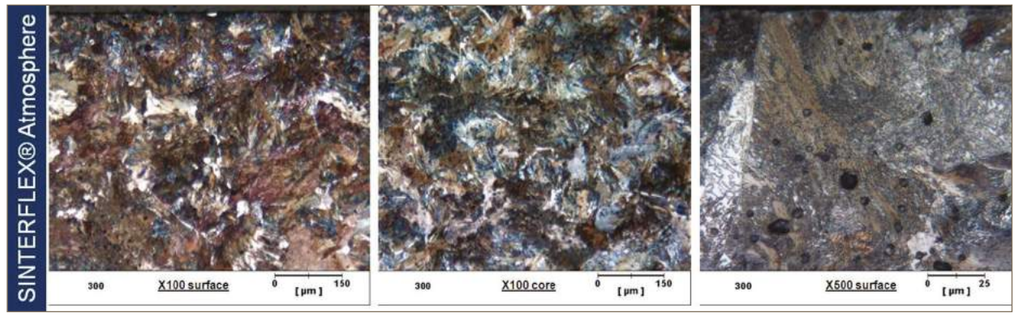



*Fig. 5 Microstructure of Part 1 sintered using a SINTERFLEX™ atmosphere*





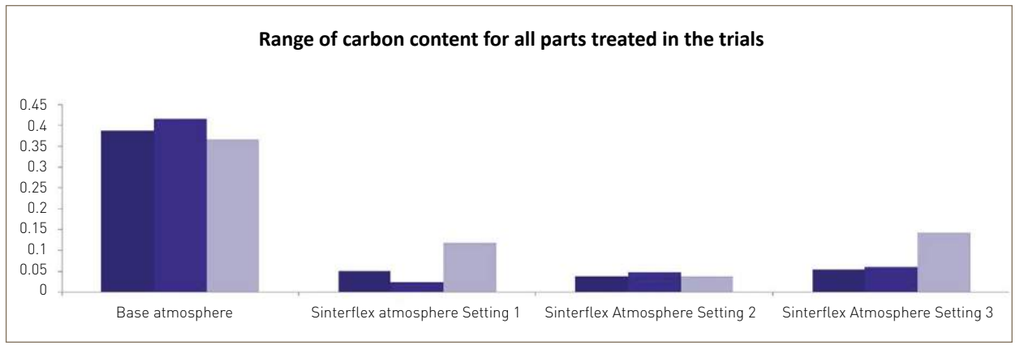



*Fig. 6 The range of carbon content for all parts treated in the trials*

Megamet had been experiencing decarburisation of parts with a specification of 0.4 to 0.6% carbon. The parts actually produced had a non-uniform carbon content within each furnace charging boat of between 0.1 to 0.4%. This was a particular problem for post mechanical and heat treatment operations leading to extra costs.

Three different components were tested during the demonstrations. The parts were manufactured using BASF Catamold® FN0205 feedstock, with the powder mixture being 0.4-0.6%C, 1.9-2.2%Ni and balance Iron. Three different components were tested during the demonstrations, as shown in Fig. 3. The parts were processed using a pusher-type MIM sintering furnace with a 20% hydrogen in nitrogen atmosphere. Total process time was 7.5 hours, with a 14 minute push each cycle.

In order to establish the base-line for the carbon control trials a SINTERFLEX™ controller was used to monitor the existing conditions. Base-line data showed erratic carbon content results ranging from 0.2 to 0.7% carbon both within and between boats. Fig. 4 shows the microstructure of a part treated using the base-line atmosphere. Decarburisation on the surface and in the core can be clearly seen as white structures.

The base-line furnace atmosphere was replaced with a carbon control mixture. The mixture contained nitrogen, hydrogen, carbon monoxide and propane for enrichment. The trials started with a CO addition of 2% in order to create the atmosphere for the carbon control. However, this addition actually increased the carbon content of the parts to 1 to 1.4% carbon on the surface and resulted in surface melting.

The tests continued with reduced CO levels. As predicted, the carbon potential was still higher than the base-line atmosphere carbon potential. SINTERFLEX™ had measured the carbon potential as 0.02% at 1280°C during the base-line atmosphere tests. For the atmosphere control trials the value was set to 0.05 - 0.07% depending on the requirements of each part. The results showed that uniform carbon contents of 0.5% (±0.05%) could be achieved in the parts.

Fig. 5 shows the microstructures after carbon control tests. Compared to Fig. 4 the decarburisation had been significantly improved, and almost eliminated in the core, due to higher carbon potentials in the lower temperature zones of the furnace.

## Trial summary

It was found that, as MIM sintering furnaces have other zones such as pre-heating and cooling where temperatures are above 750°C with the same atmosphere, carburisation and decarburisation occurs in these zones. By taking a reference point for the sampling it was possible to estimate the conditions in the other zones and optimise the overall process. Carbon potential measured in the high heat temperature zone could be used to control addition gases in order to optimise the process and improve part quality and consistency.

An overview comparison of base-line atmosphere results and the results during the SINTERFLEX™ trials is shown in Fig. 6.

*'It was possible to establish optimum furnace atmosphere conditions for all the parts tested and maintain carbon content within the specified 0.4 – 0.6% range'*

SINTERFLEX™ successfully controlled the carbon potential and helped achieve uniformity in the furnace atmosphere. It was possible to establish optimum furnace atmosphere conditions for all the parts tested and maintain carbon content within the specified 0.4 – 0.6% range, improve quality and reduce cost.

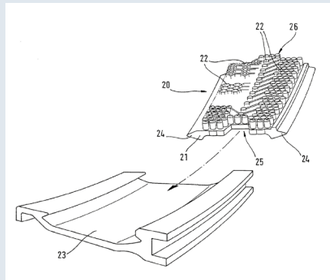
## Contact

Akin Malas
Head of Industry Segment
Heat Treatment & Electronics
Packaging
Linde AG
Linde Gas Division
Carl-von-Linde-Strasse 25
85716 Unterschleissheim
Germany
Tel: +49 (89) 31001 5549
E-mail: akin.malas@linde.com
www.heattreatment.linde.com

# 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.

**US 2009041610**
**GAS TURBINE HONEYCOMB SEAL**
**Publication date: 2009-02-12**
**Inventor(S): Meier Reinhold, MTU Aero Engines GmbH, Germany**



A honeycomb seal which is manufactured by powder metallurgical injection moulding is disclosed in this patent. The honeycomb seal is composed preferably of several segments. Each segment is embodied as a single piece and has a base element as well as honeycomb elements that are embodied as a single piece with the base element.
The present invention proposes for the first time that the honeycomb seal be manufactured by powder metallurgical injection moulding. This results in greater design freedom for the honeycomb seal. In addition, manufacturing costs are lower. The grinding and deburring processing steps are eliminated.

**JP 2009019264 (A)**
**SOFT MAGNETIC IRON-BASED SINTERED MEMBER**
**Publication date: 2009-01-29**
**Inventor(s): Takahashi Teruo et al, Nippon Piston Ring Co Ltd, Japan**
The aim of this invention is to provide a soft magnetic iron-based sintered member which is suitable as a material for an electromagnetic member of a needle valve, etc., of an electronically-controlled fuel injection apparatus. The iron-based sintered member contains, by mass, 8.0-15.0% of Cr and 0.5-7.0% of Si, or further contains 3.0% or less of Mo, 3.0% or less of Cu, and 3.0% or less of Al, the remnant being Fe and unavoidable impurities, and is produced by a metal powder injection moulding method followed by sintering.
Thus, an inexpensive soft magnetic iron-based sintered member having a high magnetic flux density, a high volume resistivity, and a high corrosion resistance is obtained, and an electro-magnetic member with a complicated shape can be produced with a high accuracy required and with a high density comparable with that of an ingot material.

**CN 101417337**
**METHOD FOR MANUFACTURING BEVEL GEAR**
**Publication date: 2009-04-29**
**Inventor(s): Qijun Xiang et al, Byd Co Ltd China**
Described in this patent is a manufacturing method of a bevel gear comprising the following steps: iron-based alloy powder and a binder are mixed at the temperature of 165 to 180C, the obtained mixture is moulded by injection to form bevel gear blank, degreasing treatment is carried out to the obtained bevel gear blank to remove the binder in the bevel gear blank, and then sintering is carried out so as to manufacture and obtain the bevel gear.
The final product has high density and good formability, therefore, the manufactured gear has high accuracy and good tooth surface smoothness, and follow-up machining is not needed even for the bevel gears with small size and complex shape, thus having simple manufacturing process.

**CN 101528619 (A) - POWDER INJECTION MOULDING METHOD OF GLASS AND GLASS-CERAMICS**
**Publication date: 2009-09-09**
**Inventor(s): Corning Inc, USA**
The present invention discloses a method for producing glass or glass ceramic articles by powder injection moulding of glass powder. The method includes mixing together, in a continuous mixing process, ingredients to form a mixture comprising a glass powder and a binder, where the ingredients include a glass powder in a relative amount sufficient to equal at least 50% by volume of the resulting mixture and a binder comprising a thermoplastic polymer, desirably a thermoplastic elastomer, and a wax; forming the mixture into a formed structure; and de-binding and sintering the formed structure.
The method involves mixing via a high intensity mixing process, desirably by mixing in a twin-screw extruder. The forming process may include pelletizing the mixture and injection moulding the pelletized mixture to form the formed structure. The ingredients of the mixture desirably comprise a glass powder in a relative amount suffi-

cient to equal at least 70% by volume of the resulting mixture. The glass powder desirably includes at least some glass particles having irregular shapes.

### WO 2009141100 (A1) METHOD FOR PRODUCING A COMPONENT FROM A COMPOSITE MATERIAL

**Publication date: 2009-11-26**
**Inventor(s): Adams Horst, et al, Alcan Tech & Man Ltd China**

In a method for producing a component from a composite material composed of metal and carbon nanotubes (CNT), a metal/CNT composite material is produced in the form of a powder. From said metal/CNT composite powder and a polymer, an injection-mouldable starting material is produced and processed in an injection moulding machine to produce a shaped article, the polymer is dissolved out of said shaped article, and the shaped article is sintered to produce the component part.

### CN 201311177 (Y) MULTI-STAGE CONTINUOUS SINTERING FURNACE

**Publication date: 2009-09-16**
**Inventor(s): Shukun Cao, Univ Jinan, China**

This relates to the technical field of metal powder injection moulding, in particular to a sintering furnace which comprises a furnace body, wherein the furnace body is provided with a degreasing stage, a sintering stage and a cooling stage which are distributed linearly from an inlet to an outlet. A furnace door is arranged at the inlet of the degreasing stage, at least a door is arranged between the degreasing stage and the sintering stage, and also between the sintering stage and the cooling stage, and a furnace door is arranged at the outlet of the cooling stage. The degreasing stage and the sintering stage are both provided with heating devices, the cooling stage is provided with a cooling device, and the degreasing stage, the sintering stage and the cooling stage are all provided with actuators. The sintering furnace adopts a linear structure which combines single degreasing, sintering, and cooling processes in traditional MIM procedure, thereby increasing product quality and reducing rejection rate during sintering process.

### CN 101579160 STAINLESS STEEL ZIPPER MANUFACTURED BY METAL INJECTION MOULDING AND PREPARATION METHOD THEREOF

**Publication date: 2009-11-18**
**Inventor(s): Shihua Liu, China**

The present invention provides a stainless steel zipper manufactured by metal injection moulding. The stainless steel zipper comprises a zipper fastener (1), a pull ring (2), a pull piece (3) and pull teeth (4). The stainless steel zipper consists of stainless steel powder and at least one binder. The preparation method of the stainless steel zipper manufactured by metal injection moulding comprises the following steps: 1, designing a mould; 2, selecting the metal powder; 3, selecting the binder; 4, preparing and compounding to uniform feeding material; 5, after injecting into a mould for heating at high temperature, cooling and forming; 6, cooling the binder for forming; and 7, sintering and forming the finished product. The stainless steel zipper manufactured by metal injection according to the invention has the advantages of suitability for manufacturing the zipper with random size, achieving the carving of complicated shape and three-dimensional forming characteristic, reducing the wastage, increasing the product performance, having fine surface, along with no easy generation of blistering, no oxidation, and no requirement of subsequent processing and auxiliary device.

### JP 2009144717 (A) MONOLITHIC, BI-METALLIC TURBINE BLADE DAMPER, AND METHOD OF MANUFACTURING THE DAMPER

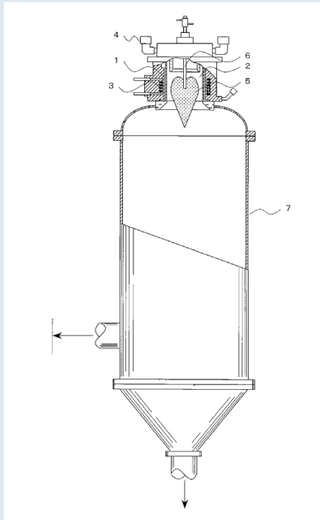
**Publication date: 2009-07-02**
**Inventor(s): Patrick D Keith et al, General Electric Co, USA**

The method of manufacturing a turbine damper by a metal injection moulding process is disclosed in this patent. The damper includes a base section and a wire section, and is formed of a nickel group or a cobalt group superalloy. The nickel group superalloy has a composition including 3.1-21.6% cobalt, 0-0.5% iron, 4.2-19.5% chromium, 1.4-7.80% aluminium, 0-5.00% titanium, 0-7.20% tantalum, 0-3.50% niobium, 0-9.50% tungsten, 0-5.40% rhenium, 0-10.00% molybdenum, 0.02-0.17% carbon, 0-1.55% hafnium, 0.004-0.030% boron, 0-0.09% zirconium, 0-0.01% yttrium, 0-1.00% manganese, 0-0.50% copper, 0-0.55% silicon, and residual part including nickel.

### JP 2009287105 (A) METHOD FOR PRODUCING SPHERICAL TITANIUM BASED POWDER

**Publication Date: 2009-12-10**
**Inventor(S): Abe Mariko et al, Hitachi Metals Ltd, Japan**

Described in this patent is a method for producing spherical titanium based powder where hydrogen-containing titanium based powder is passed through RF heat plasma flame, is subjected to spheroidizing treatment, and is thereafter subjected to dehydrogenation treatment. Further, in the method for producing spherical titanium based powder, the hydrogen-containing titanium based powder contains hydrogen by >=3 mass%.



# An approach to cost effective low alloyed materials for Metal Injection Moulding

**Jens Rassmus, Anna Larsson and Heike Grosser**
Höganäs AB, 263 83 Höganäs, Sweden

One of the growth limiting factors for the Metal Injection Moulding (MIM) industry is said to be material cost. The relative material cost is lower for a smaller part compared to a larger one. One way to decrease cost is to use coarser materials (larger particles), but normally larger particles do not sinter as well as small. This paper describes an alternative approach to enable the use of coarser materials by selecting alloys containing ferrite stabilising elements and sinter them in the ferritic phase.

## Introduction

When producing powder metallurgy (PM) parts via the MIM route the materials used are very fine with a particle median around 10 µm. Traditional press and sintering uses metal powders with a particle median from 50-100 µm and a sieve cut of 200-300 µm.

The traditional PM market is around 100 times larger in terms of volume compared to MIM.

The material (feedstock) price difference is in the order of 10 times higher for the MIM materials compared to PM materials. What is limiting the use of coarser materials for MIM? What would be the main driving force to use coarser materials?

The main driving forces to adapt to new technology are cost and/or performance. Since coarser materials have lower sintering activity, and less ability to fill thin sections, cost will be the major driver. Lower material cost will be important for larger components where the material represents a relatively high cost proportion compared to smaller components. Maybe a significant material cost reduction can expand the total market of MIM and thus compete with larger components produced by investment casting, forging and machining.

The technical feasibility of the coarser materials are determined by:

- mouldability
- debindability
- sinterability – or how well the material densifies during sintering
- dimensional stability.

In this introductory paper sinterability is studied for some different alloy systems, alloyed with ferrite stabilising elements.

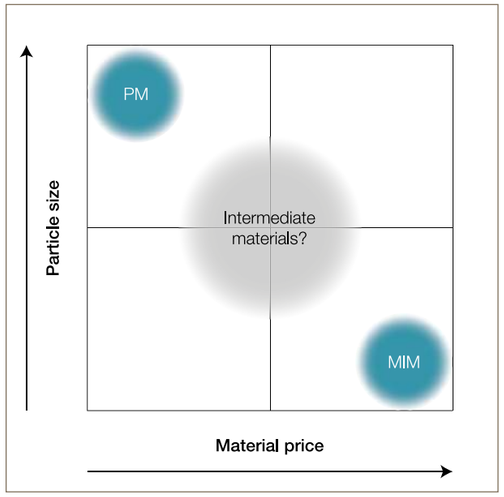



*Fig. 1 Relationship between particle size and material price for traditional press/sinter (PM) and MIM*

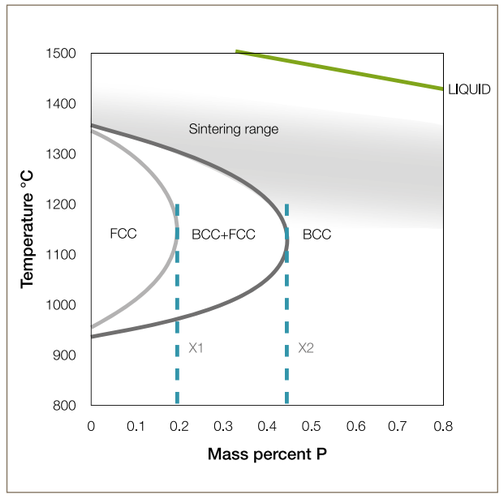



*Fig. 2 Fe-P-1.0%Mo phase diagram calculated by Thermocalc*





## Ferritic sintering

Sintering in the ferritic phase will speed up diffusion rates significantly [1,2], hence improve densification even for coarser materials. This implies that ferritic stainless steels are suitable candidates, but also low alloyed steels alloyed with Mo, Cr, P, Si etc. Three different alloy systems are investigated regarding densification behaviour, microstructure and tensile properties:

## Fe-Mo-P

The phase diagram for an essentially carbon free material with Fe, Mo and P is displayed in Fig. 2. Transition lines from FCC to BCC, BCC to FCC and BCC to liquid are drawn. The possible sintering range is shown as the shaded area in the BCC (ferritic) area. The compositions X1 and X2 define the maximum amount of phosphorous (or any other ferrite stabilizing element) which can be added to have a FCC or mixed phase (FCC+BCC) region during material heating or cooling. Over X2% P the material is fully ferritic in the whole temperature range.

As can be seen in Table 1 and Fig. 2 increasing Mo content will decrease the "need" for P in order to increase the ferritic area.

A series of material mixes containing a pre-alloyed water atomised Mo-steel, water atomised iron powder and $Fe_3P$ powder were prepared to be loosely consolidated in a TS pressing tool with

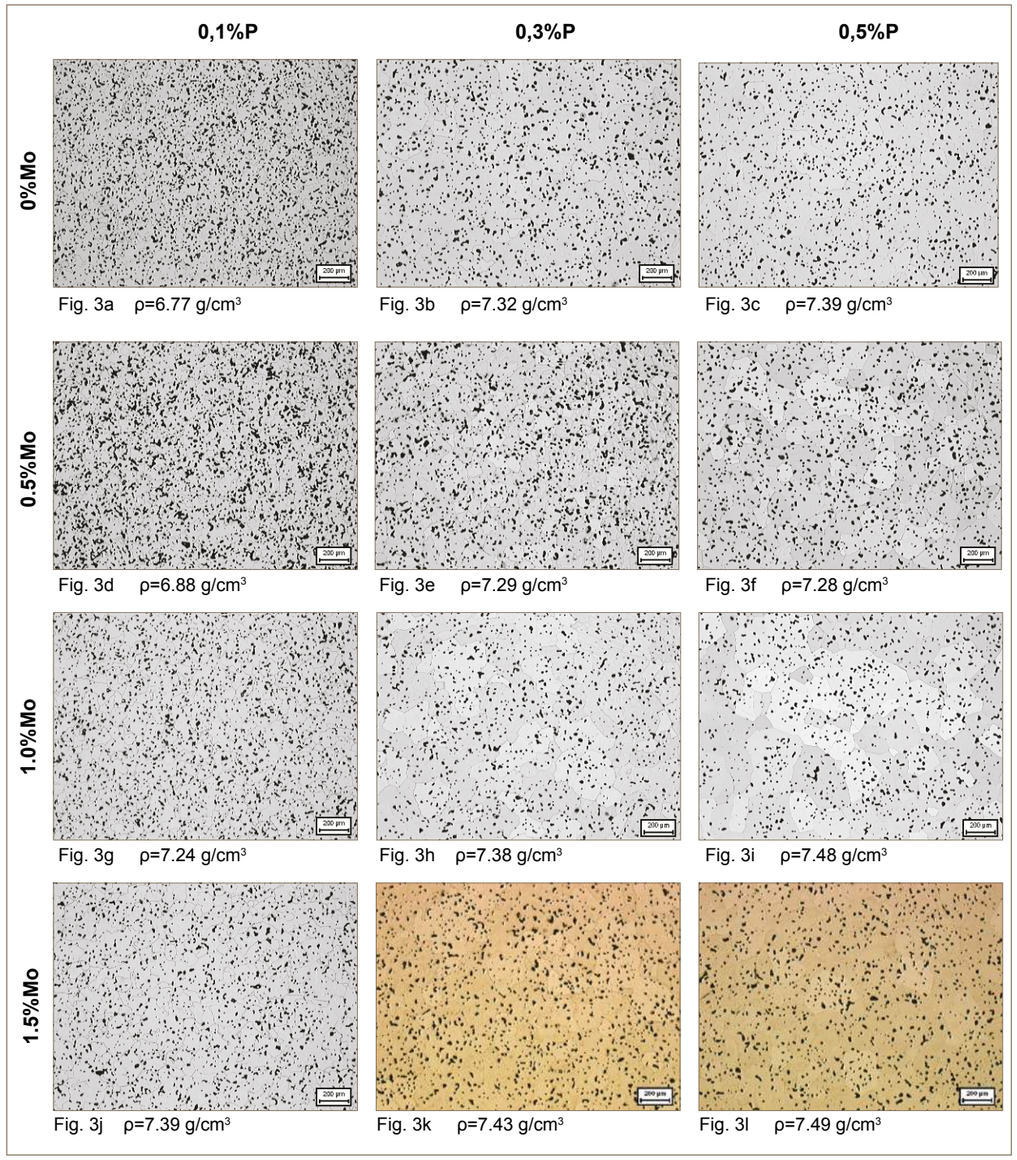



*Fig. 3(a-l) Fe-P-Mo sintered at 1400°C in 90/10 $N_2/H_2$ atmosphere for 1 hour. Micro structure and sintered density*

| Mo-content | X1 | X2 |
|---|---|---|
| 0 | 0.3 | 0.6 |
| 0.5 | 0.27 | 0.53 |
| 1.0 | 0.2 | 0.45 |
| 1.5 | 0.15 | 0.37 |

*Table 1 Maximum P contents in weight% for transition to FCC for different Mo-contents*

| | 0.1%P | 0.3%P | 0.5%P |
|---|---|---|---|
| 0%Mo | 5.42 | 6.31 | 7.27 |
| 0.5%Mo | 5.33 | 7.13 | 7.28 |
| 1.0%Mo | 5.50 | 7.26 | 7.33 |
| 1.5%Mo | 6.31 | 7.36 | 7.42 |

*Table 2 Sintered density g/cm³ of Fe-Mo-P materials sintered at 1350 °C in 90/10 $N_2/H_2$ for 2 hours*

| a) | | | |
|---|---|---|---|
| UTS/A | 0.1%P | 0.3%P | 0.5%P |
| 0%Mo | 250/10.7 | 377/25.2 | 466/11.9 |
| 0.5%Mo | 278/17.9 | 384/22.5 | 450/19.8 |
| 1.0%Mo | 331/23.9 | 395/24.1 | 458/24.1 |
| 1.5%Mo | 342/26.8 | 407/25.6 | 480/24.2 |
| b) | | | |
| UTS/A | 0.1%P | 0.3%P | 0.5%P |
| 0%Mo | 118/8.3 | 243/8.8 | 364/4.9 |
| 0.5%Mo | 116/6.9 | 333/16.5 | 450/14.5 |
| 1.0%Mo | 115/6.8 | 371/18.7 | 471/12.1 |
| 1.5%Mo | 191/8.2 | 403/22 | 494/20.1 |

*Table 3 Ultimate Tensile Strength, UTS, (MPa) and elongation, A, (%) for Fe-Mo-P material sintered at; a)1400°C in 90/10 $N_2/H_2$ atmosphere for 1 hour and b)1350°C for 2 hours*

150 MPa pressure (mixed with a high green strength lubricant). In this way the green components have close to tap density and resemble the density of an as-moulded component. The powders were sieved on -45 µm prior to mixing and had an approximate X50 between 30-40 µm.

The sintered results are displayed in Fig. 3 where the microstructure for each material together with density is shown

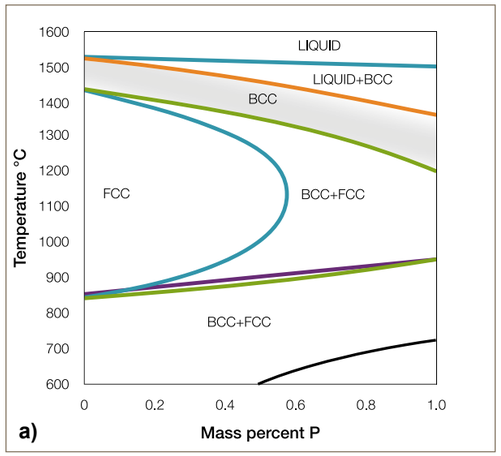
With increasing amount of ferrite stabilising elements an increased density can be seen. For compositions above X2 grain growth is evident in this fully ferritic material. When defocusing the images in a microscope it can be seen that the ferrite-austenite transformation has created a dual phase structure (with ferrite of different P-contents) in all materials below X2 and above X1 which is beneficial for the material toughness. Below X1 the structure has transformed totally to austenite and then ferrite again and shows a single phase structure with fine grains.

## Fe-Mo-Cu-P

Copper is a solid solution hardener although it is not as strong as carbon and phosphorus. The phase diagrams for Fe-3%Cu-P and Fe-1.5%Mo-3%Cu-P are shown in Fig. 4.

By adding Mo to Fe-3%Cu-0.5%P the austenitic region is pushed towards lower phosphorus contents (Figs. 4a and 4b).

Only alloying with Cu and P will yield a very narrow "sintering window" and the melting temperature (solidus) will be close at any



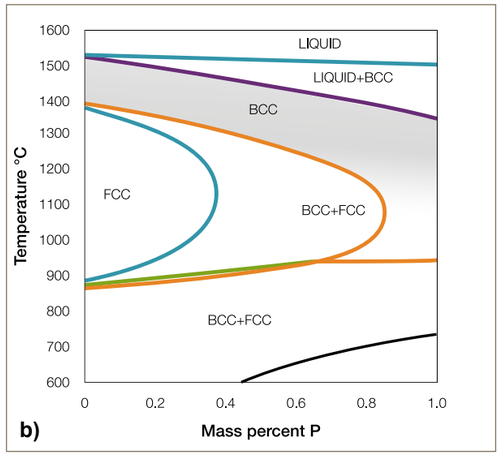




*Fig. 4 Phase diagrams in sintering range temperatures calculated by Thermocalc for a) Fe-P-3Cu and b)Fe-P-1.5Mo-3Cu. Shaded areas indicate the possible sintering ranges*

| | SD g/cm³ | UTS/A MPa/% |
|---|---|---|
| Fe-3Cu-0.5P | 7.41 | 556/9.6 |
| Fe-3Cu-0.5P (Cu3P) | 7.29 | 532/14 |
| Fe-1.5Mo-3Cu-0.5P | 7.44 | 604/4.7 |
| Fe-1.5Mo-3Cu-0.5P (Cu3P) | 7.34 | 551/6.3 |

*Table 4 Sintered properties, density, Ultimate Tensile Strength (UTS) and Elongation (A) of Fe-Mo-Cu-P sintered at 1400°C in 90/10 $N_2/H_2$ atmosphere for 1 hour*

sintering temperature in the BCC area, hence why it is favourable to alloy with Mo.

The same procedure as above with Fe-Mo-P was applied on the copper materials only the copper source was either 325 mesh standard PM copper or $Cu_3P$ powder.

The higher strength of the Cu-materials is a result of solid solution strengthening of the ferrite. Also some areas of bainite and martensite can be seen at higher resolution.





## Fe-Mo-Cr-P

Adding Cr, an additional ferrite stabilising element which also acts as a solid solution hardening element, is also one possible alternative metal system. In Fig. 6 the impact of and increased Mo-content from 0.5% to 2% is seen on an Fe-3Cr-Mo-P material.

## Discussion

The importance of ferritic sintering for the densification of coarser material is evident from the examples above. Compositions on the borderline or well below the transition line BCC-FCC exhibit significantly lower densities compared to those well in the BCC area. According to Fig. 2 a composition of 1%Mo and 0.1%P cuts the transition line around 1330°C. Sintering at 1350°C is clearly not enough (time/temperature) in the BCC area to densify the material. It exhibits a density of only 5.50 g/cm³ as can be seen in Table 2.

Passing through a phase transformation during cooling is beneficial for the metallurgical structure. The grain size

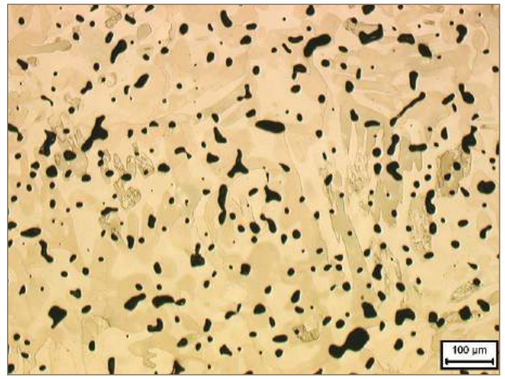


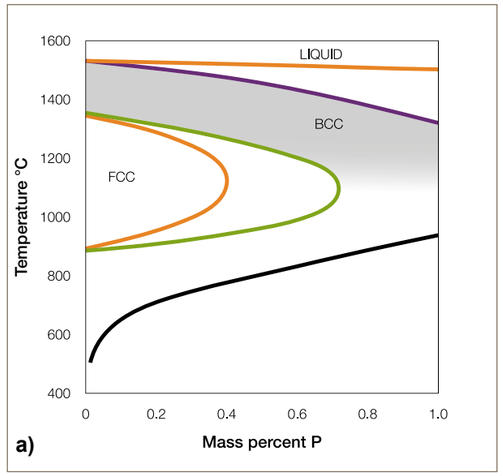

*Fig. 5 Microstructure of Fe-1.5Mo-3Cu-0.5P sintered at 1400°C in 90/10 $N_2/H_2$ atmosphere for 1 hour*

| | SD g/cm³ | UTS/A MPa/% |
|---|---|---|
| Fe-1.5Cr-0.3Mo | 5.89 | 171/11 |
| Fe-1.8Cr | 5.92 | 191/11 |
| Fe-3Cr-0.5Mo | 7.12 | 410/13 |
| Fe-1.5Cr-0.3Mo-0.3P | 7.44 | 428/25 |
| Fe-1.8Cr-0.3P | 7.34 | 415/24 |
| Fe-3Cr-0.5Mo-0.3P | 7.38 | 446/25 |

*Table 5 Sintered properties, density, Ultimate Tensile Strength (UTS) and Elongation (A) of Fe-Cr-Mo-P sintered at 1400°C in 90/10 $N_2/H_2$ atmosphere for 1 hour*

| a) | | |
|---|---|---|
| PM materials [3] sintered at 1120 °C | UTS MPa | A % |
| Fe-Cu-C | ≈500 | ≈3 |
| Distaloy™ materials | 600-900 | 1-3 |
| Astaloy™ materials | 600-900 | 1-3 |
| b) | | |
| -45 materials sintered at 1400°C | UTS MPa | A % |
| Fe-Mo-P | 330-480 | >20 |
| Fe-(Mo)-Cu-P | 500-600 | 4-14 |
| Fe-Mo-Cr-P | 400-450 | >20 |
| c) | | |
| Standard MIM materials [4] sintered at around 1250°C | UTS MPa | A % |
| Fe2Ni (no C) | >260 | >20 |
| Fe7Ni (no C) | >410 | >20 |
| FeCuNi | >600 | >3 |

*Table 6 Relation between Ultimate Tensile Strength (UTS) and elongation (a) in as sintered state for a) PM materials, b) -45 materials in current study, c) standard MIM materials*



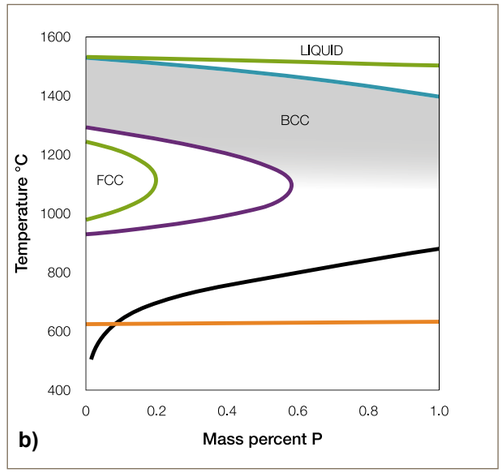




*Fig. 6 Phase diagrams for Fe-P-3Cr-0.5Mo (a) and Fe-P-3Cr-2Mo (b) calculated with Thermocalc. The possible sintering range is marked with a shaded area*

increases dramatically if the material is totally ferritic in the whole temperature range as can be seen from Fig. 3, which should have a negative effect on toughness.

Cu-addition helps to give a solid solution strengthening effect in the as sintered state, but combinations with Mo are more favourable due to the "thin" sintering window of the pure copper added materials. Variations in composition and impurity levels must be taken into consideration (Mn,C etc) and this might pull down the solidus-temperature. Copper addition makes the material more brittle (less ductile), but it seems the type of copper addition has some impact

Addition of Cr will also give a solid solution strengthening effect (but not as large as the Cu-addition) and less phosphorous is needed to achieve strength/ductility compared to the pure Fe-Mo-P materials.

Table 6 shows a comparison of standard PM materials and existing MIM materials (no carbon) in the as sintered state, made with the current study.

## Conclusions

By using alloys with Fe-Mo-P (Mo:1-2, P:0-0.5) adding Cu or Cr, and sintering between 1300-1400 °C:

- good sinterability and strength/ductility can be achieved using coarser materials (-45µm) if ferritic sintering is used, which makes these materials potential candidates for MIM applications.
- sintered properties in range with existing materials, based on carbonyl iron, can be achieved under certain conditions.

Higher strengths of around 600 MPa with these essentially carbon free materials will need a hardening or a secondary treatment.

## References

[1] Bergstrom R.T, Sintered austenitic-ferritic chromium-nickel steel alloy, US patent 3,620,690, filed Oct 7 1968

[2] Baum L.W, Wright M., Composition and process for metal injection molding, US patent 5,993,507, filed Dec 29 1997

[3] Höganäs Handbook, Höganäs iron and steel powder for sintered components, www.hoganas.com/pmc







# Bulk Metallic Glasses (BMG): A competing or complementary technology to Metal Injection Moulding?

**Frans Van Camp*, Leo Oelbrandt**
OCAS N.V., Pres. J. F. Kennedylaan 3, 9060 Zelzate, Belgium
*corresponding author. Email: frans.vancamp@ocas.be

The objective of this article is to present the technology of bulk metallic glasses, also called amorphous metals. Next to a specific structure, a BMG has a unique combination of mechanical and physical properties that can offer an added value to the parts producing industry. Based on extensive research, OCAS has identified that BMG can complement the MIM technology and decided to conduct a market feasibility assessment to acquire further insights into the industrialization potential for certain applications of this exciting technology.

## Technology

Most metals solidify into their lowest energy state structure, i.e. a crystalline lattice structure, within microseconds once they have reached a temperature below their melting point. These crystalline lattice structures are mostly polycrystalline, with grain structures of different shape and size.

Glass can be undercooled to below its melting temperature because of its slow crystallisation kinetics. The atoms do not form a crystalline lattice but keep their amorphous distribution.

Some metal alloys have a glass-like behaviour: if the melted alloy is cooled fast enough (hyper cooling) from the liquidus temperature to below the glass transition temperature it will freeze in its amorphous state, because below this glass transition temperature (Tg) the diffusion processes are too slow to rearrange the atoms into a crystalline phase (Fig. 1).

## History

In 1960, Klement, Willens and Duwez demonstrated at Caltech (Pasadena CA, USA) the vitrification of an Au-Si binary system using rapid quenching techniques. They used a cooling rate in the order of $10^6$ K/s [1].

At the end of the 1980's and beginning of the 1990's many new alloys were developed with slow crystallisation kinetics, which decreases the required cooling rate.

This evolution allowed the production of alloys with better so called glass formation properties, requiring lower quenching speeds and resulting in a higher critical casting diameter of the bulk metallic glasses (BMG) samples. It enabled the production of BMG parts using slightly adapted conventional casting techniques.

To achieve slow crystallisation kinetics, the following criteria need to be taken into account:

- Reduce the energetic advantage of forming a crystalline structure by using multi-component alloys (three or more alloying elements).
- A negative heat of mixing of the main alloying elements will increase the energy barrier at the liquid-solid interface.
- An atomic radius difference between the alloying elements (>12%).
- Use an alloy composition with a low melting point (close to a deep eutectic).

Peker and Johnson at Caltech discovered a composition called Vitreloy 1 ($Zr_{41}.2Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$) with a critical cooling rate of only 1 K/s. This alloy and variants are being commercialised today.

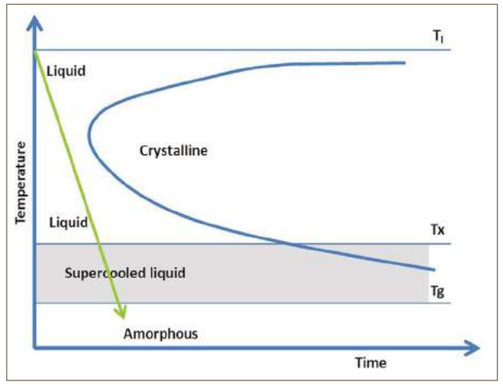



*Fig. 1 Time -Temperature diagram*

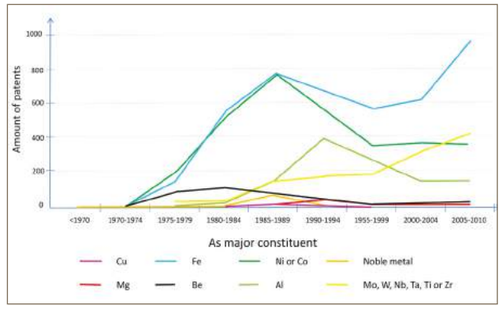



*Fig. 2 Amorphous alloy IP evolution*

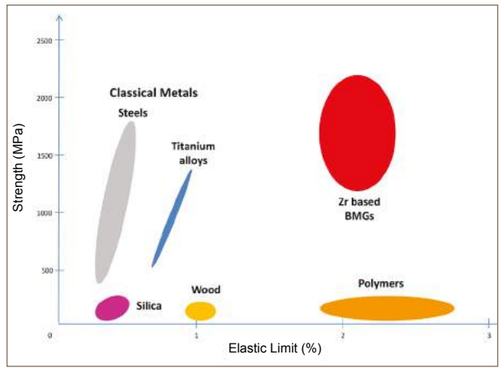



*Fig. 3 Strength versus elastic limit [4]*

More recently, due to the interesting properties and possible cost advantage potential, Fe-based BMG's are being studied intensively; the amount of generated IP on Fe-based BMG's is growing fast (Fig. 2).

## BMG properties

Because pure BMG's have no crystalline structure, no crystal defects and no grain boundaries they demonstrate quite interesting and unique mechanical, thermophysical and electromagnetic properties; the latter for Fe-based alloys. BMG's will, depending on the composition, have the following unique properties:

- **Mechanical [5]:**
  - Extremely good balance between strength and elastic limit (Fig. 3)
  - High hardness resulting in high abrasion and wear resistance
  - High specific strength: $\sigma_y / \rho$
  - High resilience per unit volume: $\sigma_y^2 / E$
- **Excellent corrosion resistance due to the lack of grain structure (depending on the composition)**
- **Processing:**
  - Real net shape casting: negligible shrinkage in the mould when die cast
  - Accurate surface finishing: the lack of grain structure results in a potentially high as cast surface polish
  - No porosity
- **Magnetic: (in the case of Fe-based alloys)**
  - Very low coercivity which leads to ultra-low energy losses (if used as a core material in electromagnetic devices)

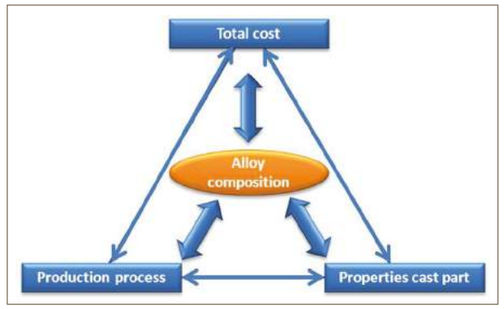



*Fig. 4 Industrial deployment*

  - High magnetic permeability
  - Higher resistivity (compared to electrical steel grades)

There are also some unattractive properties of BMG's:

  - Zero ductility in tension
  - The maximum service temperature of BMG is limited up to a percentage of the glass transition temperature Tg.

BMG technology presents many unique features, but for some applications it is not necessary to maximise all of them at the same time. In order to evolve to an industrial deployment, it is clear that there is some trade-off to be made among some of the variables, and that finding the right balance to fit a certain industrial application is crucial (Fig. 4).

The selected alloy composition will have a direct influence on the production process, robustness and the final cast product properties and, of course, on the total cost of the cast part.

## OCAS work on BMG's

OCAS has worked on BMG's for several years and performed research on iron and zirconium based alloys. OCAS opted to work with a beryllium free, zirconium based alloy. As OCAS is an applied research centre, it started working on BMG with a clear industrial application focus.

**Pre-alloy (feedstock) production on a lab-scale**

The raw materials composing the alloy are selected and the necessary amounts are weighed to form the target composition. Small volumes of pre-alloy (comparable to feedstock preparation in the case of MIM) can be prepared using an induction melter or levitation melter.

An induction melter melts the raw alloy materials with an induction coil. A levitation melter will also use induction to melt the materials, but it can do this container-less, meaning no melting crucible is used. This ensures a contamination free melting and the electromagnetic stirring ensures a homogeneous pre-alloy ready to be die-cast (Fig. 5). OCAS also has the possibility to melt larger volumes of pre-alloy of up to 50kg in a large vacuum oven.

**Advantages**

- No pollution of melt by crucible material (< 5 ppm in electrolytic iron)
- Homogeneity of alloys due to powerful electro-magnetic agitation
- Possibility to melt materials with high fusion temperature (>2000 °C)
- Fast throughput processing: multiple castings per hour possible (small batch sizes are possible).





For the (lab) casting, the materials are heated up to a temperature higher than the melting temperature, to assure homogenisation. This step can be performed under a protective gas atmosphere (e.g. argon) or vacuum. The molten material is kept in this state for a certain time before it is poured into a mould.

**Characterisation of cast samples**

The degree of amorphicity of the cast sample (part) has a major influence on a lot of mechanical (and in case of Fe-based BMG's also electromagnetic) characteristics. It is therefore of crucial importance to be able to determine the level of amorphicity of the produced parts. Different methods are used at OCAS:

- **X-ray diffraction (XRD):** This is a versatile, non-destructive technique that reveals detailed information about the crystallographic structure of materials. Investigation by conventional XRD of an amorphous sample displays broad halos without any sharp diffraction peaks typical for crystalline phases (Fig. 6a and 6b). In Fig. 6b the spectrum consists of a very broad peak (halo) with some superimposed Bragg peaks. The diffuse scattering (halo) shows the presence of a disordered or short range ordered phase, which corresponds to the amorphous matrix. The Bragg peaks are identified as crystalline phases.
- **Scanning electron microscope (SEM):** This can assure that the sample is not amorphous but the resolution is too low to detect the possible presence of nanocrystals (Fig. 7a and 7b).
- **Transmission electron microscopy + electron diffraction:** The bright-filed image exhibits a typical amorphous material with a salt and pepper like contrast (Fig. 8a and 8b).
- **Differential scanning calorimetry (DSC):** This is a thermo analytical technique in which the difference in the amount of heat required to maintain a sample and a reference material at the same prescribed temperature is measured as a function of temperature. With DSC, phase transitions but also glass transitions and crystallisation behavior can be evaluated. DSC measurements provide quantitative and qualitative information about physical and chemical changes that involve endothermic or exothermic processes or changes in heat capacity (Fig. 9).
- **Coercivity measurements:** This non-destructive technique is used to measure the amorphicity of Fe-based BMG's, and allows a quick measurement of relatively large volumes of the sample. Coercivity:
  - Hc is very sensitive to the sample's structure
  - Amorphous: Hc<=10A/m

*Fig. 5 Levitation melter (left) and samples of cast BMG R&D samples (right)*

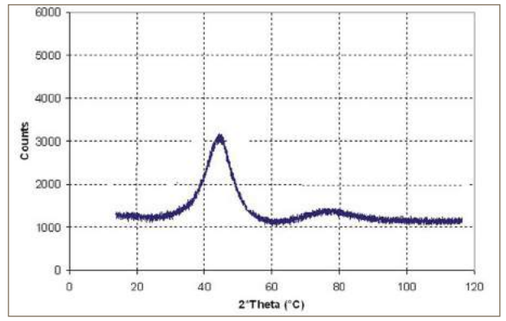



*Fig. 6a XRD graph of an amorphous sample*

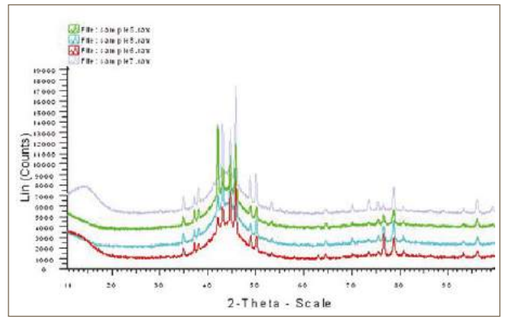



*Fig. 6b XRD graph of an amorphous sample with crystalline phase*

## Technology push: in the search for applications

For years amorphous ribbons (not bulk), obtained by high speed melt spinning, have been used in amorphous low-loss electrical transformers and residual current circuit breakers. Protective amorphous coatings have also been commercially available for many years.

Bulk metallic glasses were not discovered as a response to a specific market need. As is the case with many technological discoveries, there is an initial overhyped-response, and the technology's potential for immediate industrial deployment was overestimated. Typical for a technology push (a technology in search of an application) a lot of investments need to be made to mature the technology.

In scientific literature the different possible applications mentioned (tooling, springs, knives, clubs, sporting goods, MEMS, etc.) are essentially all based on the same technical BMG features: high strength & elasticity, and the lack of grain structure; and their main constraints: no plastic strain and limited casting thickness.

It was by studying the metal injection moulding market and technology that it became clear to OCAS that there are certain similarities and differences with the BMG technology. A further in depth investigation was necessary.

## BMG versus MIM

Both BMG and MIM are technologies that are mainly targeting the mass production of small (the majority smaller than 20 grams), complex metal parts. Both technologies are capable of producing these type of parts in a more economical and elegant way compared to other manufacturing methods (e.g. investment casting, machining, etc.).

An in depth study of the MIM technology and its applications revealed that BMG die-casting could be a complementary part

producing technology. BMG technology addresses some of the current challenges of the MIM technology such as dimensional stability, strength, straightness, surface finish and delivery time.

**Generic properties**

As stated in the previous paragraphs, in order to fully leverage all the beneficial properties of BMG, the cast part needs to be as amorphous as possible.


*Fig. 7a SEM picture of a fully amorphous sample*

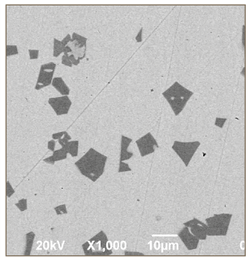



*Fig. 7b SEM picture of an amorphous sample with crystalline inclusions*


*Fig. 8a high resolution TEM image of the matrix*

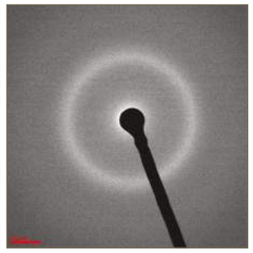


*Fig. 8b the corresponding electron diffraction pattern*

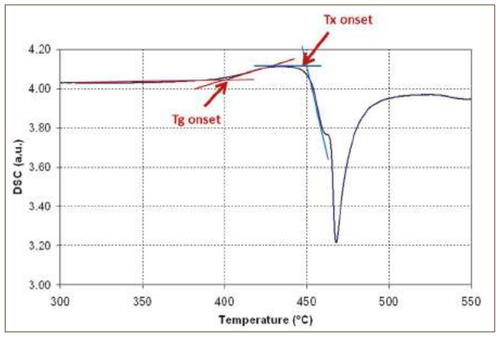



*Fig. 9 DSC curve*

| Feature | BMG Zr alloy | Stainless steel (17- 4 ph) |
|---|---|---|
| Hardness | 550 HV | 328 HV |
| Strength | 1000 MPa | 620 MPa |
| Density | 5.9 g/cm³ | 7.8 g/cm³ |
| Strength-to-weight ratio | 169 kNm/kg | 79 kNm/kg |
| Elastic deformation | up to 2% | 0.4% |

*Table 1 Mechanical properties; the figures for BMG are based on internal laboratory tests and literature*

Rapid quenching to obtain this amorphous structure can be achieved by an adapted die-casting process. Size and wall thickness of the cast parts will be limited if full amorphicity is required, although full amorphicity will probably not be a technical prerequisite for a lot of applications, and can best be verified by "in use testing" of the BMG part.

It is OCAS's belief that the other benefits of BMG die-casting such as real net shape casting and surface finishing could be of high value to the part producing industry. OCAS initiated a detailed technical and economical comparison between these two part manufacturing technologies, but realises that further in depth study together with the active participation of the MIM industry is necessary to maximise the result.

**The production process**

The BMG die-casting process is a single step part production process starting from a ready to use pre-alloy (feedstock). In comparison with the multi-stage production process of MIM, one can expect that the overall production yield of the BMG production process, once mastered and optimised, will therefore be higher.

Scrap or production waste (runners and cookies) can be recycled at any stage of the BMG production process, although possible alloy contamination needs to be monitored. This can also contribute to higher overall process efficiency compared to MIM.

No shrinkage occurs because of the amorphous nature of the alloy, this increases the dimensional accuracy of the final BMG part (e.g. in case of die-casting) and especially the repeatability of the dimensional accuracy over a produced batch. The dimensional tolerance for MIM parts is nominally between 0.3% and 0.5% [2], for BMG's it is expected to be less than 0.2%.

**Mechanical properties of a finished part (MIM: not heat treated)**

Table 1 shows the typical values of a zirconium BMG alloy and a stainless steel used for MIM.

**Design features**

As for part complexity, MIM is considered as the ideal choice for highly complicated structures, but certain constraints are still to be respected when it comes to part design. The following section details those bottlenecks where BMG technology could provide solutions or optimise manufacturability.

- For MIM parts, uniform wall thickness throughout the whole part helps avoid defects like distortions, voids, cracking etc. If variation is inevitable, gradual transition is recommended between the different thicknesses in order to mitigate the risk of errors and make the shrinkage during manufacturing more predictable. BMG has a negligible shrinkage, has no additional heat treatment (such as debinding and/or sintering) and so ensures better dimensional stability for the final part.
- Due to the high strength and elastic deformation limits, in combination with the absence of shrinkage during the casting process, BMG technology could ideally be suited to produce very thin casings for different electronic applications (e.g. mobile phone casings).
- In a stainless steel alloy, the chromium content is one of the determining anti-corrosion factors; this also counts for the stainless steel MIM alloys. During sintering the high vapour pressure of chromium can lead to its preferential evaporation towards the part surface and will diminish its resistivity to corrosion. The alloy composition (the composing elements) is the main factor that determines the corrosion properties of a BMG alloy; there are a number of BMG alloys that have excellent corrosion properties. BMG parts do not have a porous structure.
- Fillets and radii have pre-defined minimum dimensions for MIM parts since sharp corners cause stress concentration, can erode the mould and hinder smooth feedstock flow. Therefore the limit for corner and edge radii is around 0.05 mm [3]. Tighter limits or even sharp corners can be produced by BMG-technology, while moderating the negative consequences.
- During debinding and sintering the MIM part significantly shrinks, thus the larger the flat surface of the part, the lower the risk of distortion. In case of other shapes, extra fixtures or setters are necessary to support the part during sintering which can increase the cost of the part [3]. Since BMG die-casting will be a real net shape production process (with no debinding or sintering) no such supports are necessary.
- Reinforcements (webs and ribs) are necessary for certain MIM part geometries in order to increase strength and minimise shrinkage defects and distortions during manufacturing. Such design considerations are not necessary for BMG parts due to the nature of the production process.
- BMG technology allows the combination of glossy and mat surfaces on one part as cast. Moreover, surface texture patterns like knurls, serrations, logos and characters are also applicable without additional finishing steps, just as sharp edges are achievable as cast without grinding.
- The mould-surface structure is reproduced on a µ-scale onto the cast part (Fig. 10). From the measured value for Sa (= 3D Ra) one can conclude that on this sample the roughness was extremely low. Surface roughness measurements (3-D) were carried out with a Taylor Hobson CCI device. The technical characteristics of the equipment used are given in Table 2.

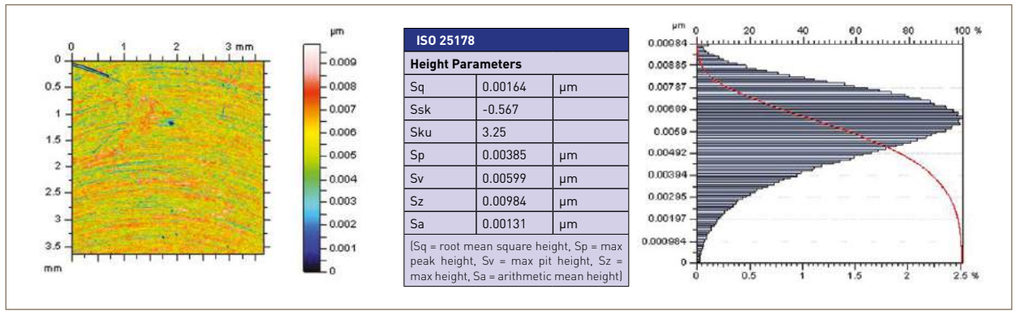



*Fig. 10 BMG surface roughness measurement results*

| | |
|---|---|
| Manufacturer | Taylor Hobson Cci |
| Configuration | Talysurf Cci |
| Measuring Method | Vertical Scanning Interferometry (VSI) |
| Interferometer Type | Cci |
| Vertical Range | 100µm |
| Vertical Resolution | 0.01nm |
| **Magnification Objectives:** | |
| Magnification Objective (M.O.) | 5* |
| Field Of View | 1 |
| Spatial Sampling | X: 3.6µm Y: 3.6µm |
| **Data Acquisition:** | |
| Measurement Array Size | 1024*1024 |
| Surface | 3.6*3.6 mm² |

*Table 2 Technical characteristics of equipment*

## Conclusion

OCAS has identified that BMG's can complement the MIM technology and decided to conduct a market feasibility assessment to acquire further insights into the industrialisation potential for certain applications of this exciting new technology. MIM companies that are interested to find out more about BMG technology and OCAS's BMG research are invited to make contact.

## References

[1] W. Klement et al. 'Non-crystalline Structure in Solidified Gold–Silicon Alloys', *Nature* (1960) 187, 869 – 870.

[2] 'What is MIM',Metal Injection Molding Association, http://www.mimaweb.org/About_MIM.htm, retrieved on 12/07/2011

[3] German, R.M., 'Designing for Metal Injection Moulding: A Guide for Designers and End-Users', *PIM International*, December 2008

[4] Mark Telford 'The case for bulk metallic glasses', *Materials Today*, March 2004.

[5] M.F. Ashby, A.L. Greer, 'Metallic glasses as structural materials' *Scripta Materialia* 09/2005

## About OCAS

OCAS is a market-oriented metal research centre founded in 1989, based in Belgium. OCAS is a joint venture between ArcelorMittal and the Flemish region.

The key areas investigated are metallurgy, surface functionalisation and steel applications. OCAS expertise concentrates on alloy and coating development, co-development of steel applications, processing and testing of metal based samples.

Not limited exclusively to steel, OCAS solutions and design teams look for ways to cross-fertilise materials, techniques, processes, characteristics and applications.

OCAS has access to the venture fund managed by OCAS Ventures. This fund invests in spin-offs of OCAS projects within the OCAS competence areas.

www.ocas.be

# Events Guide

## 2011

**MM Live - Micro Manufacturing**
September 27-29, Birmingham, UK
www.micromanu.com

**EuroPM2011 International Conference and Exhibition**
October 10-12, Barcelona, Spain
www.epma.com

**30th Hagen Symposium**
November 24-25, Hagen, Germany
www.pulvermetallurgie.com

**Powder Processing, Consolidation and Metallurgy of Titanium**
December 5-7, Brisbane Australia
www.materialsaustralia.com.au

## 2012

**MIM2012 International Conference on Injection Molding of Metals, Ceramics and Carbides**
San Diego, USA, March 19–21
www.mpif.org

**Ceramitec 2012**
May 22-15, Munich, Germany
www.ceramitec.de

**PowderMet 2012 International Conference on Powder Metallurgy & Particulate Materials**
June 10–13, Nashville, TN, USA
www.mpif.org

**EuroPM2012 International Conference and Exhibition**
September 17-19, Basel, Switzerland
www.epma.com

**Powder Metallurgy World Congress & Exhibition PM2012**
October 14-18, Yokohama, Japan
www.pm2012.jp



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