Vol. 6 No. 3 SEPTEMBER 2012

FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

POWDER INJECTION MOULDING INTERNATIONAL

in this issue

MIM in Japan
Profiles: Epson Atmix, Hardex
Rapid prototyping for CIM

Published by Inovar Communications Ltd

www.pim-international.com

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Subscriptions
Powder Injection Moulding International is published on a quarterly basis. The annual subscription charge for four issues is £115.00 including shipping. Rates in € and US$ are available on application.

Design and production
Inovar Communications Ltd

Printed by
Cambrian Printers, Aberystwyth, United Kingdom

ISSN 1753-1497

Vol. 6, No. 3 September 2012

Powder Injection Moulding International

For the metal, ceramic and carbide injection moulding industries

The PIM industry prepares for PM2012 Yokohama

Welcome to the September 2012 issue of *PIM International*. The PM2012 World Congress and Exhibition, Yokohama, Japan, 14-18 October, is now just around the corner. This important event in the PM calendar has much to offer the PIM community and it is sure to be the year's essential meeting point for industry producers, suppliers and researchers.

As well as featuring a broad range of technical content, the PM2012 World Congress schedule includes a Special Interest Seminar (SIS1) that features six invited presentations reviewing global developments in PIM. In addition, the PM2012 exhibition not only features a number of PIM industry suppliers, but also some of Japan's leading PIM parts producers will be exhibiting examples of their products (page 37).

It is of course just 18 months since the devastating earthquake and tsunami hit the north east of Japan in March 2011. Japan's leading MIM powder and components producer, Epson Atmix Corporation, was badly affected by the disaster but as we report, the company worked tirelessly to restore production in difficult circumstances and is now in the middle of a major capacity expansion (page 45).

In this issue we also review the use of CIM components in Swiss luxury watches (page 51), developments in powder production and MIM processing at this summer's successful PowderMet 2012 conference held in Nashville (page 55), and an innovative rapid prototyping solution for CIM producers (page 65). Japan's A.L.M.T. Corp. additionally presents information on efficiency gains that can be achieved through the use of advanced ceramic coated molybdenum setters for sintering MIM parts (page 69).

Our technical papers in this issue present the latest innovations in MIM powder production, with Höganäs AB presenting an evaluation of coarser iron powders as a substitute for carbonyl iron (page 75) and Reading Alloys - Ametek presenting information on the production of a new plasma spheroidised (PS) titanium powder for MIM (page 78). We also present recent work on the processing and characterisation of porous NiTi alloy produced by MIM (page 81).

Nick Williams
Managing Director and Editor

Cover image
The Executive Dual Time watch by Ulysse Nardin, featuring black CIM zirconia top ring and pushers manufactured by Hardex, France (Courtesy Hardex)

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

Höganäs reports on the successful testing of a new coarse powder feedstock for MIM

Swedish iron powder producer Höganäs AB indicates that it has made major advances in the composition of material for the manufacture of Metal Injection Moulded components.

In the company's Interim Report for the period 1 January – 30 June 2012 it stated, "As a result of our work on accumulating know-how end to end in the value chain for these components, we have discovered unique alloys, which combined with the right binding agents, enable the use of coarser powders for MIM components. This makes it possible to produce larger components through injection moulding simultaneous with a significant reduction in cost compared to traditional, finer MIM powders."

Traditionally, stated Höganäs, the MIM process has been based on very fine metal powders, with particles as small as 20 microns. Such powders are very costly to produce, limiting their application to small, complex components where there is no more suitable technology.

Alongside its partner, Höganäs has successfully injection moulded and sintered coarser particles of 45 microns. This notably alters the costing, enabling the production of larger components (several hundred grams) using this technology.

Potential customers are participating with Höganäs in the development process. Testing is being conducted using Höganäs' powder mixes and feedstock. Through this development work, Höganäs has been able to build up critical knowledge of the manufacturing process, including equipment settings, tooling pressure and sintering parameters. The estimated value of the current market for MIM components, stated Höganäs, is $1 billion. The company's starting-point is that the result of current development work should make it possible for market value to double over time.

www.hoganas.com

Nippon Atomized Metal Powders expands capacity for ultrafine powders

Nippon Atomized Metal Powders Corp., a subsidiary of Nihon Seiko, with its headquarters in Chiba, Japan, has been a leading producer of water atomised non-ferrous and precious metal powders since the 1960's. The company succeeded in developing and producing a range of ultra fine metal powders by water atomisation technology around ten years ago.

The company reports that it has recently expanded production capacity of its latest generation of ultrafine atomised non-ferrous powders through the construction of a new plant in Tsukuba, where monthly melting capacity has been increased by 60% to 320 tonnes.

This is in addition to the capacity of 200 tonnes at its Chiba plant for metal and alloy powders used in bearings and structural parts, and also the expansion of production of atomised powders for magnetic materials at the Chiba site.

Nippon Atomized Metal Powders has seen considerable increase in demand for its ultrafine copper and precious metal powders having a particle size of less than 1 micron, for use in multi-layered ceramic capacitors and other electronic parts due to the surge in demand for smart phones and other mobile devices worldwide.

The company reported sales of Yen 4.901 billion for the financial year ended March 31, 2012.

www.atomize.co.jp

Ipsen's TITAN® DS vacuum debind and sintering furnace designed for energy-efficient MIM processing

Ipsen, Inc., headquartered in Cherry Valley, IL, USA, presented its new TITAN® DS vacuum debinding and sintering furnace at PowderMet 2012, June 10-13 2012, claiming that the company has made great strides in easing product maintenance and ensuring repeatability and precision.

Although Ipsen has already been in the sintering business for some time, the company states that it has recently partnered with one of the world's largest Metal Injection Moulded parts manufacturers to bring about some innovative changes that set the TITAN® DS apart.

"This furnace provides technical features optimised for MIM and PM applications with an unbeatable price-to-volume-ratio. And since it is based on Ipsen's TITAN® platform, which is bestselling and well-proven in the heat treatment and vacuum brazing industry, it comes with many of the same benefits," stated Ipsen.

The furnace operates at 50/60 Hz, works with any voltage and in more than 20 languages. The TITAN® DS fits into one standard global shipping container and ships within 10 weeks of the order with one-day installation.

The furnace's sintering process benefits include the ability to handle very high binder content in MIM applications and the company states that its batch processing is now competitive with continuous processing.

Thanks to the TITAN® DS's automated protection system, the company claims that binder build-up in pumps and piping is virtually eliminated. "This allows for a reduced door-to-door cycle time with easier maintenance and care. The accurate control of the gas flow carries evaporated binder out of the hot zone and eliminates cross contamination. Furthermore, the optimised hot zone design with additional front and rear heating elements provides for improved cleanliness and temperature uniformity for flawless part quality."

Ipsen's TITAN® DS vacuum debind and sintering furnace

Ipsen also claims that despite these benefits, the furnace does not sacrifice its energy efficiency, which it states is still 30% more efficient than comparable models. "This offers our customers a scalable solution to closely and cost-efficiently adapt their capacity planning to the quickly growing Powder Metallurgy and Metal Injection Moulding markets," stated the company.

www.ipsenusa.com

New atomiser for research and small scale production available from PSI

PSI, Hailsham, UK, has announced the introduction of a new atomiser in their HERMIGA mini atomiser range, the HERMIGA 75/5 EAC with hot gas.

A comprehensive list of options make it the most flexible, high performance research and small scale production atomiser available, states the company. The list of options provides ultimate control over material melting, atomisation, sprayform manipulation and powder collection.

The HERMIGA 75/5 EAC unit uses an advanced low flow close coupled die configuration specifically designed for research applications and allows the fitment of a hot gas atomisation system in a mini system for the first time.

The HERMIGA 75/3 and 75/5 models retain the high performance close-coupled die configuration as used in the production scale systems and can produce high quality spherical powders down to 10 microns median size. The powders experience rapid solidification with cooling rates in the region of 103 to 106 K/sec.

The smallest atomisers in the HERMIGA range have found favour in numerous research and development areas covering a wide range of applications from precious metals to solar energy and in geographic locations from Russia to India and USA to Japan.

The compact nature and range of options available for these machines, together with ease of cleaning, makes them the ideal tool for advanced metal powders research work, states PSI. The basic systems offer an induction heated crucible with 3 kg or 5 kg melt capacity, close-coupled nozzle assembly, inert gas atomisation capability (making them safe even with pyrophoric powders) and two stage powder collection.

The options available are quite comprehensive and have been selected to provide a wide range of research opportunities;

- Oxygen and moisture monitoring provide data to ensure optimum melt and atomisation purity.
- A gas supply rack accommodating up to five standard gas bottles. This provides minimal variation in atomising gas pressure and enables different gasses to be used during melting and atomisation.
- A melt additions hopper allows fine tuning of melt stock or final additions of relatively volatile components.
- Particle injection capability enables the production of spray-formed particulate metal matrix composites. Up to 30% by volume of the second phase can be introduced.
- Secondary gas injection at the nozzle can be specified, which provides additional cooling of the atomised powder.

PSI's HERMIGA 75 atomiser

Besides the atomisation options listed above, the atomisation chamber can be fitted with various manipulation and collection devices, including:

- Sprayform manipulators enable deposition of semi-solid droplets in billet or cylinder form providing materials with a homogeneous microstructure.
- Free jet melt spinning rigs are offered to provide further rapid solidification routes.
- Billet casting can be accommodated to afford direct casting of the molten material.
- Cryo-quench can be specified where materials need to be rapidly cooled to low temperatures.

Video monitoring of the melting and atomisation processes can be provided for permanent visual records. When combined with PSI's Glovebox/Classifier these atomisers offer one of the most comprehensive suite of powder metallurgy research tools available.

www.psiltd.co.uk

International MIM conference seeks technical papers

The programme committee for MIM2013, the International Conference on Injection Molding of Metals, Ceramics and Carbides, has issued a "Call for Presentations."

The focus of the technical programme is "Advances in Component Uniformity." Sponsored by the Metal Injection Molding Association (MIMA), a trade association of the Metal Powder Industries Federation (MPIF), and its affiliate APMI International, the conference will be held in Orlando, Florida, USA, March 4–6 2013, at the Hilton in the Walt Disney World Resort.

Technical programme co-chairmen Brian McBride, General Manager, Parmatech Corporation, 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 programme committee requests that abstracts be submitted by September 30, 2012, for consideration. Complete details on the conference, and on submitting abstracts, are available on the conference website. Contact Jim Adams at jadams@mpif.org for all enquiries relating to the conference technical programme.

www.mimaweb.org | www.mpif.org

Element 22 receives approval for Ti-MIM medical implants

Element 22 GmbH, based in Kiel, Germany, reports that one of its customers recently received approval to implant MIM medical parts in the United States. The approval is based in the 2011 ASTM standard for surgical titanium implants made by Metal Injection Moulding. A second standard is expected to follow by the end of 2012.

Element 22 is a fully integrated manufacturer, starting with titanium powder and manufacturing finished components. Currently, Element 22 uses spherical EIGA (Electrode Induction-melting Gas Atomisation) powders, but it is reported that it has also started looking at titanium powder made by alternative methods.

As well as manufacturing titanium implants and other medical components, Element 22 states that it also manufactures several titanium components for commercial aircraft.

Based on its existing MIM technology, Element 22 is developing further advanced titanium production technologies such as the production of foil or tubes, where the porosity can be adjusted from a solid helium-tight structure to open porosity. Such components or semi-finished products can be made from most titanium alloys.

www.element22.de

A Ti-MIM aerospace fastener manufactured by Element 22


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Kinetics offers new service for low volume MIM prototypes

There are a broad range of rapid prototyping technologies available today that are regularly used by product development engineers to create initial product concepts and to conduct early design reviews. Some of these technologies, for example stereolithography (SLA), can provide prototype plastic parts in as little as 24 hours and at very low cost.

One of the bigger challenges confronting product design engineers is producing prototypes that exhibit full-functionality including desired material performance, especially when metal alloys are being considered for the final product design. This is especially the case when new process technologies such as MIM are considered, or in cases where product designs are testing new performance boundaries. Here, the evaluation of 'production' prototypes can increase designer confidence that the design and process technology selections will work. However, 'production' prototypes generally equate to expensive tooling and long lead times where low volumes are required. Since the MIM process uses injection moulds similar to those required for plastic injection moulding, initial capital investments in tooling for production prototypes can be high.

For design engineers looking for low cost, quick-turn prototypes, US MIM producer Kinetics, based in Wilsonville, Oregon, USA, has developed an answer. The company offers a prototype process for customers who are planning to use MIM for production, but who want to conduct application design and performance assessments at a low initial investment.

Whilst the Kinetics process uses production feedstock, production injection moulding machines, production furnaces, and therefore, yields prototypes with production MIM material properties, the low-cost tooling for this process will generally cost the customer between $3,000 and $10,000 and will yield as many as 250 prototype parts. Design engineers can receive prototype MIM parts in as little as three to four weeks from the point of submitting electronic part data.

www.kinetics.com

Date set for the 2013 China PM Expo, Shanghai

The date for the next International Powder Metallurgy Exhibition and Conference (China PM Expo) to be held in Shanghai, has been set for April 26-28, 2013.

This successful and popular annual event will again be hosted by the China Machine Powder Metallurgy Association, the Powder Metallurgy Association of China Steel Association and the China Nonferrous Metal Powder Metallurgy Association. It is expected to once again attract international as well as Chinese exhibitors. A number of Powder Metallurgy and Metal Injection Moulding technology seminars are also planned.

www.cn-pmexpo.com

Parmatech Corp receives ISO 9001:2008 certification for its Rhode Island facility

ATW Companies, a leading US provider of custom manufactured metal components and services, has announced that its East Providence, Rhode Island based Parmatech Corporation subsidiary has received ISO 9001:2008 certification for its quality management system.

The three year certificate became effective July 23, 2012 and covers the manufacture of Powder Injection Moulded (PIM) products for a wide range of industrial applications.

Parmatech's Rhode Island facility received the ISO 9001:2008 certification following a rigorous audit conducted by TÜV Rheinland of North America, Inc., an accredited third-party registrar. Achievement of the certification, states Parmatech, demonstrates that its quality management system is based on such principles as strong customer focus, motivation and support of top management, use of a quality management process approach, and commitment to continual improvement.

"We are proud of achieving this valuable and internationally accepted quality management system certification," commented Brian McBride, General Manager at Parmatech. "Our customers now have yet another means of ensuring that they will be receiving consistent, high quality products and services."

Parmatech Corporation is a pioneer of Metal Injection Moulding (MIM) technology and has been a leading provider of custom manufactured MIM components and services since the 1970's.

The company's Rhode Island facility today operates eight moulding machines from the manufacturers Battenfeld and Nissei, ranging from 10 to 110 tons compacting force. Five furnaces are in operation, including batch and continuous systems.

The Rhode Island plant is Parmatech's second MIM facility, following its 2009 acquisition of MIM producer Proform from Morgan Ceramics. The company's original facility is based in Petaluma, California.

www.parmatech.com

Injection moulding machines at Parmatech's Rhode Island facility

Continuous and batch furnaces are operated at the Rhode Island facility

Slovakian PM producer Gevorkyan expands into MIM production

Gevorkyan s. r. o., a Slovakian Powder Metallurgy parts producer based in the town of Banska Bystrica, has recently enhanced its production capacity with an investment in a MIM production line. The facility uses injection moulding technology from Arburg Gmbh + Co KG, a debinding system from Lömi GmbH and a sintering furnace from Cremer Thermoprozessanlagen GmbH.

The company states that the primary materials processed are stainless and low alloy steels. Feedstock is sourced from PolyMIM GmbH.

Gevorkyan s. r. o. has been manufacturing a wide range of press and sinter PM parts, including bronze, bronze-iron and iron self-lubricating bearings, along with ferrous structural parts, since 1996. The company has also started the production of silver electrical contacts.

Sales in 2011 increased by 60% and in the coming years turnover is expected to enjoy three-digit annual growth. As a result, further investments are anticipated.

Gevorkyan s. r. o. was nominated as "Plant of the year 2011" in Slovakia and has benefited from recent external technology and production assessments. "Our company has gained a lot from the independent assessment, identifying our strengths and weaknesses," stated Artur Gevorkyan.

"The company clearly appreciated this, because of the enormous pace and commitment with which we work, such an objective expert view from the outside helped us better identify the challenges ahead".

www.gevorkyan.sk

The Gevorkyan s. r. o. MIM facility

MIM parts produced by Gevorkyan s. r. o.

The Gevorkyan s. r. o. MIM facility

2012 China MIM Symposium takes place in Kazuo; Chinese MIM committee formed

The first "China MIM Symposium" was held in Kazuo County, Chaoyang City, Liaoning Province, from 1-3 August 2012. The event, organised by the Powder Metallurgy Industry Technology Innovation Strategic Alliance, attracted more than 80 delegates from MIM enterprises, institutes and universities as well as representative from overseas MIM producers and suppliers.

Presentations covered all aspects of Powder Injection Moulding, including micro injection moulding, how injection moulding process parameters affect parts, and manufacturing methods. The injection moulding of parts for the automotive industry was also reviewed, along with examples of award-winning MIM products. Proceedings on the symposium have been published.

The importance of MIM technology as a rapidly growing manufacturing area was acknowledged by the participants, with the organisation of such seminars being regarded as extremely important to review progress, identify technology gaps, and to promote the development of the MIM business in China.

The establishment of a MIM Professional Committee took place in parallel with the event, the purpose of which is to pursue the development of international standards, to build cooperation between enterprises and governments as production, learning and research. Such a development, it was stated, will better serve the Chinese Metal Injection Moulding industry and contribute to its continuing rapid and positive development. Professor Qu Xuanhui was elected Chairman of the committee.

Speakers at the 2012 MIM Symposium

Delegates at China's 2012 MIM Symposium

A Chinese MIM committee was formed

KeyTec and ECN collaborate on components for extreme environments based on MIM technology

Dutch energy research centre ECN has developed an extremely thermo-shock proof material for nuclear fusion applications that is shaped using the injection moulding process. KeyTec, also based in The Netherlands, recently signed a license agreement with ECN enabling the company to produce and supply advanced products manufactured by this process, which offer high-melting points and high levels of wear resistance.

Tungsten and molybdenum are materials that withstand extreme heat load conditions. ECN states that its variant of the Metal Injection Moulding process for the production of tungsten components shows superior characteristics under extreme heat load conditions. The material is yttrium doped and, contrary to conventional materials, testing at an absorbed capacity of 1.13 GW/m^2 during 1000 cycles of 1 ms did not lead to any cracks developing in sample components.

In particular, states ECN, the ductility, fatigue resistance and hardness of the material has been optimised, as well as its recrystallisation properties. KeyTec and ECN aim to expand this development to a small range of other high-melting point materials that are difficult to process, as well as to precision metal components that require special properties.

Although this process was developed specifically for nuclear fusion applications, it is also highly suited for other applications involving high temperatures and shielding.

KeyTec states that the unique features of the obtained material are, to a large extent, determined by the use of MIM technology. The material is highly homogenous with a fine micro-structure that is similar in three dimensions, contrary to the direction-dependence of the conventionally processed material. The material is therefore relatively stress free and shows no signs of recrystallisation between 1200 and 1440°C, but remains stable up to and beyond 2000°C.

The technology, it is stated, is highly suited for a wide range of doping applications and for developing alloys, both of which are key for developing new materials.

Additionally, the Metal Injection Moulding process offers almost unlimited freedom of shaping, which omits the need for machining. For tungsten this is highly relevant, both for cost and performance.

ECN is the largest research institute in the Netherlands in the field of energy. It holds a strong international position in the fields of solar energy, wind energy, policy and strategy studies, biomass, energy efficiency and environmental research. KeyTec specialises in the development and supply of thermoforming and injection-moulding components for vacuum applications and is a well-known supplier of components in high-end materials.

For more information contact:
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Rudi Grootjans, email rudi.grootjans@keytec.nl.
www.ecn.nl / www.keytec.nl

Sandvik Osprey increases fine powder capacity by 60% with installation of new atomiser

Leading gas atomised metal powder producer Sandvik Osprey Ltd., based in South Wales, has completed the installation of its fifth and largest-ever fine powder atomising plant. The plant, which adds in excess of 60% to the company's already substantial capacity, has been commissioned successfully to plan and is now fully operational.

Planning for the new atomiser began in the first half of 2011 to meet the rapidly rising demand for high quality gas atomised fine metal powders. In addition to its new atomiser, the company has invested continually in downstream powder processing capacity and has more than doubled its sieving and classifying capacity in the last three years.

During this period of sustained growth, Sandvik Osprey's Powders Group has re-modelled its production operations on an enlarged site made possible by the acquisition of neighbouring land and buildings. In addition to plant installation and land acquisition, there has been a significant expansion of power and inert gas capacity to support present and future growth.

With final commissioning now complete the company is pleased to announce to existing and future customers that it is now taking orders to be fulfilled from the new plant, which is delivering product to the same high standard that its customers have come to expect.

Sandvik Osprey's Keith Murray stated, "This expansion is fully supported by Sandvik Materials Technology, who continue to invest in the Neath operation. This is good news for consumers in the ever-expanding MIM market and in other specialist technology sectors from coatings to additive manufacturing."

www.smt.sandvik.com/osprey

PowderMet 2013 Chicago: Call for Papers

A Call for Papers has been issued for the "2013 International Conference on Powder Metallurgy & Particulate Materials", June 24–27, Chicago, Illinois, USA.

Organised and sponsored by the Metal Powder Industries Federation (MPIF) in cooperation with APMI International, the event will include a comprehensive technical programme, poster programme, exhibition and special interest seminars. Over 200 worldwide industry experts are expected to present latest in PM and particulate materials. All abstracts are subject to review by the Technical Programme Committee and must be received by 26 October, 2012.

The event will also feature a 100 booth trade exhibition showcasing leading suppliers of powder metallurgy and particulate materials processing equipment, powders, and products.

www.mpif.org

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Ceramitec 2012 showcases PIM technology to a global audience

The Ceramitec 2012 exhibition took place in Munich, Germany, from 22-25 May 2012. The event, which featured a wide range of PIM equipment, materials and technology suppliers as well as suppliers to the wider ceramics industry, attracted more than 16,500 visitors and served as a high-profile showcase for PIM technology.

A focus for PIM at Ceramitec 2012 was the TASK (Technology Agency Structural Ceramics) stand, organised in cooperation with the Fraunhofer Alliance AdvanCer and featuring numerous PIM related technology suppliers. A series of presentations relating to PIM and PM also took place in a dedicated auditorium as part of Ceramitec's "Technology Days".

Michael Zins, Managing Director, TASK GmbH, commented after the event, "The fact that visitors responded well to the new offering of ceramic components, and posed specific questions about components from the sectors of manufacturing, medical and environmental technology, is a real highlight. Our co-exhibitors were delighted with the number and quality of the inquiries. Even before the end of Ceramitec 2012, we had inquiries about taking part in the event in 2015. We are firmly convinced that technical ceramics is a long-term enhancement to Ceramitec."

Powder Injection Moulding International first exhibited at Ceramitec in 2009. This year, with a larger stand that focused on the promotion of PIM technology, interest was very high with PIM producers, end-users and industry suppliers visiting the stand from around the world. Jon Craxford, *PIM International's* Sales Director commented, "With the record rise in visitor numbers and more than 1000 copies of *PIM International* distributed from our stand at Ceramitec, it is clear that interest in the technology has increased significantly in recent years. The event has certainly helped to increase awareness of both PIM technology and our publications."

www.ceramitec.de

PIM technology on show at Ceramitec

PIM International's exhibition stand

Technology seminars at Ceramitec 2012

PIM continues to attract interest for the production of Solid Oxide Fuel Cells (SOFCs)

Solid Oxide Fuel Cells (SOFCs) are a promising technology for high efficiency energy conversion, and Powder Injection Moulding (PIM) is being studied as a possible approach to reduce the fabrication costs of SOFC components that have intricate patterns and fine details.

Antonin Faes and his research colleagues at the University of Applied Science in Sion, Western Switzerland, plus researchers from HTceramix, SOFC Power and Ecole Polytechnique Federale Lausanne, recently presented the first published electrochemical test results of planar anode-supported SOFC's with fine details produced by PIM using 58 wt% nickel oxide (NiO) and 42 wt% yttria-stabilised zirconia (YSZ) powders. The results were presented at the 10th European SOFC Forum, held in Lucerne, Switzerland, June 26-29, 2012.

The researchers reported that the NiO/YSZ feedstock was used to produce the Powder Injection Moulded structured anode-support disks of 40 mm in diameter and 0.5 to 1 mm in thickness. The height and width of the fine structure is 0.5 mm as

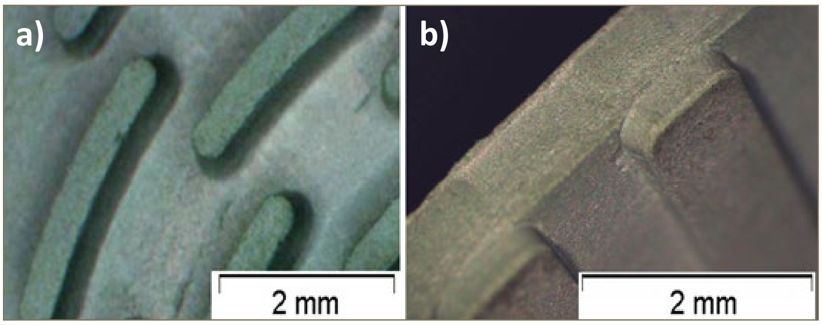


Fig. 1 Pictures of the structured NiO-YSZ anode-support with details of the (a) surface and (b) side of the green PIM parts. The width and height of the fine structured surface are 0.5 mm (left). Both scale bars are 2 mm long. (From paper by Antonin Faes, et.al., (University of Applied Science Western Switzerland, Sion) presented at the 10th European SOFC Forum, Lucerne, June 26-29, 2012, and published in the Proceedings.)

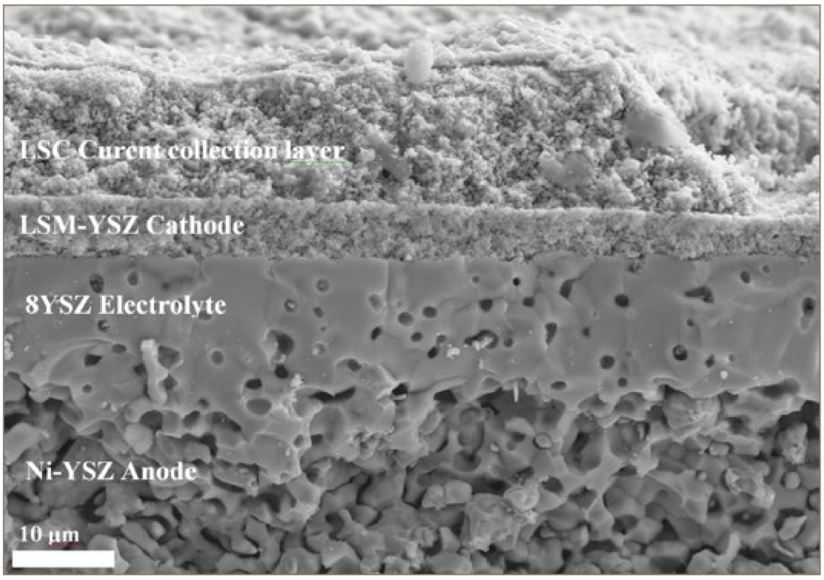


Fig.2 Scanning electron micrograph of the tested cell in reduced state. (From paper by Antonin Faes, et.al., (University of Applied Science Western Switzerland, Sion) presented at the 10th European SOFC Forum, Lucerne, June 26-29, 2012, and published in the Proceedings.)

can be seen in Fig. 1. In a previous study reported at the Euro PM2011 Conference held in Barcelona in October 2011, the researchers had presented results of PIM processing of the NiO-YSZ bars (5 x 5 x 40 mm), which showed a linear shrinkage of about 12 % and a porosity of ca 16% after sintering for one hour at 1450°C. After reduction (12 h at 800°C under hydrogen), the porosity increases to 36% and the room temperature electrical conductivity is higher than 4000 S cm^{-1}. These values are said to be comparable to anode supports produced by tape casting.

The fine structure of the PIM anode-supported SOFC will be in contact with the metal interconnect and will allow a proper gas distribution on the anode side. This structured anode-support will replace the gas diffusion layer (GDL) function, state the researchers. Saving a part of the single repeat element unit (SRU) will save multiple parts on the full stack. This will solve other compatibility problems of the GDL layer with the anode and interconnect. Finally, it will simplify the stack assembly and reduce the total cost of the 'hot box' zone.

The sequence of the thin cell layers is shown in Fig. 2. At the bottom of the SEM micrograph, the microstructure of the PIM anode-support in the reduced state after testing is clearly visible. The electrolyte layer is about 12 µm thick and the LSM-YSZ cathode is between 5 and 10 µm (depending on the location). On top of the cathode, the LSC-based current collection layer can be seen.

The researchers report that electrochemical testing was done with a lanthanum-strontium manganite (LSM)-YSZ cathode. The performance of the cell is said to be comparable to previous anode-supports at 0.45 W cm^{-2}, 0.6 V and 810°C. Long term galvanostatic testing shows a degradation rate of about 1.1 % per 1000 h, which is comparable to similar tests on tape cast cells. Electrical impedance spectroscopy (EIS) and energy dispersive X-ray spectroscopy (EDS) conclude that cathode degradation is due to Cr and S poisoning.

The results show that a structured anode support produced by PIM can meet the operational requirements of a commercial SOFC. Future work on the electrodes will improve the cell performance.

www.hevs.ch

PM-13 India: Call for Papers

The Powder Metallurgy Association of India (PMAI) has published the First Announcement and Call for Papers for its 13th International Conference and Exhibition, this time focused on 'Precision and Additive Manufacturing in Powder Metallurgy for Automotive and Engineering Industries'. The association has also announced details of its 39th Annual Technical meeting.

This year's event will take place from 7 to 9 February 2013 in Pune, India, and organisers have requested that abstracts of papers for oral presentations as well as posters should be submitted by email to kssamant@iitb.ac.in by 31 October 2012.

The technical programme will cover new and innovative PM materials, innovative processes, progress in modelling, simulation, analysis, characterisation and testing, and ultrafine and nano-materials.

MIM is once again expected to feature strongly in the conference programme, reflecting the growing interest in the technology in India.

www.pmai.in

Fraunhofer IKTS highlight advances in PIM at Euro PM2012

The Fraunhofer IKTS research centre in Dresden, Germany, has developed advanced ceramic materials produced by low pressure and high pressure Powder Injection Moulding (PIM) which will be on show at the IKTS booth at Euro PM2012 in Basel, Switzerland, September 16-19.

Included will be a PIM processed back-lit zirconia control knob with a translucent ceramic insert, complex shaped glass components having adjustable electrical resistance, and multifunctional ceramic/ceramic and ceramic/stainless steel composite parts made by 2-component injection moulding.

Fraunhofer IKTS will present a paper at EuroPM2012 on the use of PIM to produce conductive glass-carbon composites for electrical resistors. The technology for this new material is being developed as part of the 'GlasPIM' research project started in August 2011 and funded by the German Bundesministerium für Wirtschaft und Technologie (BMWi) in which Fraunhofer IKTS, INMATEC Technologies GmbH, Kläger Spritzguss GmbH and others are partners.

www.ikts.fraunhofer.de

NTN to commercialise injection moulded high-current amorphous core

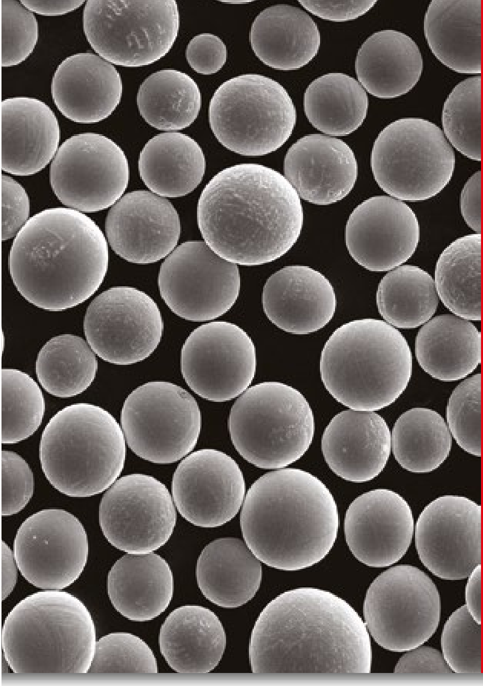
Japan's NTN Corporation has created an "Amorphous Core" by injection moulding a magnetic material comprising of a mixture of amorphous powder and resin.

The core of reactors and choke coils are generally made by press moulding magnetic ferrite. In recent years however, reactors installed in the storage devices of electric vehicles (EVs), hybrid vehicles (HEVs), solar power generators, and choke coils used in advanced medical equipment have required greater reliability under increasingly harsh conditions, including higher currents and faster drive frequencies, which has meant that conventional cores made of ferrite have become unsuitable.

The "Amorphous Core" has been developed by NTN group company Nippon Kagaku Yakin and delivers an inductance reduction ratio of just 30% under high current conditions with DC (direct current) superimposition characteristics. This high-reliability core shows no reduction in inductance at high frequency bands of 1000 kHz, and it is capable of reducing electrical signal noise at a high level of efficiency.

The core can also be made with injection moulding, which increases its design flexibility to meet the needs of coils with a greater variety of shapes and larger sizes. The coil is compact at approximately 1/8th the volume of conventional ferrite choke coils, which gives devices a more compact and lightweight design.

www.ntn.co.jp



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

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

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

HDH Powders: Greater Control of PSD

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

PS Powders: Free Flowing

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

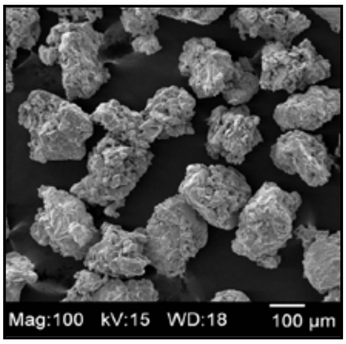
Powder Metallurgy Applications

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

Markets Served

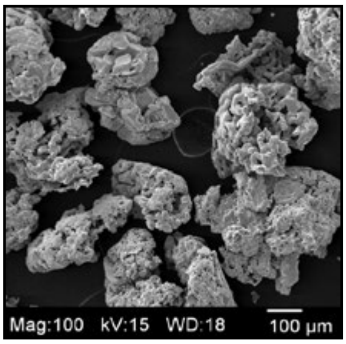
- Medical Device
- Electronics
- Thin Film (Sputtering Target)
- Net Shape
- Feedstock

Morphology: HDH Magnesium Reduced Titanium Sponge Powder



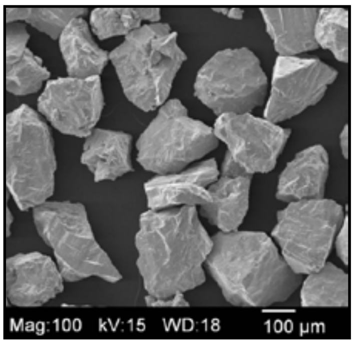
SEM image (x100) of HDH Magnesium Reduced Ti Sponge Powder, 70 mesh (212µm) x 100 mesh (150µm)

Morphology: HDH Sodium Reduced Titanium Sponge Powder



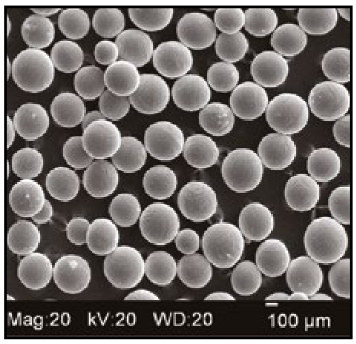
SEM image (x100) of HDH Sodium Reduced Ti Sponge Powder, 70 mesh (212µm) x 100 mesh (150µm)

Morphology: HDH CP Titanium Powder



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

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



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

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New head of Research & Development at H.C. Starck

H.C. Starck, Goslar, Germany, has announced that Dr. Michael Fooken will take over the research and development activities for the group.

Dr. Fooken has extensive experience in the field of research and development in the chemical industry and over the past 12 years has held various global positions within the Honeywell Group, including Head of Research for the Fine Chemicals division. Before that he spent many years in production as well as in research and development.

Dr. Fooken holds more than 20 patents in the fields of energy storage materials and inorganic salts. "As an experienced expert in the field of specialty chemicals, Dr. Fooken is an excellent addition to H.C. Starck, and we are very pleased to have him on board," stated Dr Andreas Meier, President and CEO of the company. "With his scientific expertise and management experience, he will successfully drive on further advancements in our research and development activities, and thus play an important role in the overall success of the entire company."

His predecessor, Dr. Gerhard Gille, is retiring on October 31, 2012. With more than 20 years at H.C. Starck, Dr. Gille played a key role in the company's growth, thanks to his new procedures for manufacturing reaction mixtures, customised components for carbides and cermets, as well as nanoscale hard material powders.

"Under the direction of Dr. Gille, many research projects were launched and carried out that made major contributions to H.C. Starck's global success. These include the magnesium reduction of highly capacitive tantalum and niobium powders, the development of ceramic cells for SOFC technology, and the development of cathode materials for lithium-ion batteries for electric cars, to name a few," added Dr Meier.

www.hcstarck.de

Hagen Symposium to focus on raw materials and application trends

The Hagen Symposium on Powder Metallurgy, organised by the Ausschuss für Pulvermetallurgie, is a key annual event in the German-speaking countries of Europe and brings together more than 250 delegates from industry, research, and academia over a two day period to review trends in PM technology and its products.

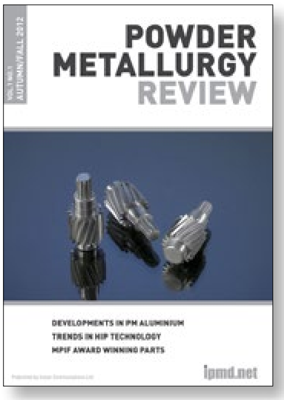
The 31st Hagen Symposium will take place as usual at the Town Hall in Hagen, November 29-30, 2012, and in addition to a full technical programme will feature more than 50 exhibitors.

The Skaupy Prize for 2012 will be presented to Dr Lorenz Sigl, Director of Research at Plansee SE, Reutte, Austria.

The organisers state that the key objective of the 2012 Symposium will be to review trends in a wide range of PM industry sectors, including ferrous and non-ferrous powder developments.

www.pulvermetallurgie.com

New magazine for the Powder Metallurgy industry to be launched



A new quarterly magazine for the Powder Metallurgy industry is to be launched this Autumn. *Powder Metallurgy Review* will be published by Inovar Communications Ltd.

Following the launch of the highly successful weekly *ipmd.net* e-newsletter in 2010, this new publication will be available in both print and digital formats and bring together highlights from the *ipmd.net* newsletter plus additional exclusive content.

Paul Whittaker, Editor of the new publication and the *ipmd.net* e-newsletter, stated, "This new high quality quarterly magazine will specifically focus on developments in the global press and sinter PM industry, including ferrous and non-ferrous components, hard materials, PM high alloy steels, PM superalloys, diamond tools and sintered magnets, as well as HIP/CIP and powder forging. We have launched the title in response to feedback from industry professionals, who, whilst appreciating the advantages of a weekly e-newsletter, also wish to have information on the PM industry in a format that they can save, either digitally or in print, for future reference."

"As with our sister publication *PIM International*, our aim is to not only provide relevant coverage of the industry and its many players, but to present the industry in a positive light, as the dynamic, modern and successful advanced metal forming process that it is."

The launch issue of *Powder Metallurgy Review* (Vol 1. No. 1 Autumn/Fall 2012) can be seen at our stand at the Euro PM2012 Conference and Exhibition, Basel, Switzerland (September 17-19) and the PM2012 World Congress and Exhibition, Yokohama, Japan (October 14-18). From 2013 *Powder Metallurgy Review* will be published four times a year (Vol. 2 Nos. 1-4).

In addition to distribution at key international PM events, *Powder Metallurgy Review* will be circulated by mail to PM industry professionals worldwide. It will also be available to download in PDF format from *www.ipmd.net* free-of-charge. Those wishing to ensure that they receive every issue in print format can purchase a subscription for £85 per year including worldwide shipping (ISSN ISSN 2050-9693).

For editorial enquiries, contact Paul Whittaker, email paul@inovar-communications.com. For advertising enquiries, contact Jon Craxford, Advertising Sales Director, jon@inovar-communications.com.

www.ipmd.net

'Handbook of Metal Injection Molding' published

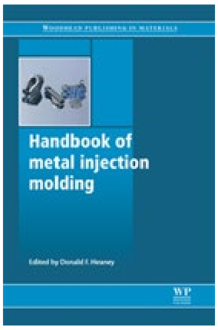
Metal Injection Moulding (MIM) is a rapidly advancing and changing metal forming technology capable of producing highly complex shaped components from a wide range of metal and alloy powders. With double-digit global MIM industry growth over the past decade the technology now find applications over a broad spectrum of end-user sectors ranging from medical, dental, electrical and electronic to automotive and aerospace.

With the many variations of MIM manufacturing technology available today, and the wide range of powder materials that have helped MIM to commercial success, it is timely and opportune to see the publication of a new and up-to-date reference book. Donald F. Heaney, the book's editor is a leading figure in the North American MIM industry and as President and CEO of Advanced Powder Products, Inc., Philipsburg, PA, and Director of the Center for Innovative Sintered Products at the Pennsylvania State University, he is in a good position to bring together the viewpoints and inputs from leading MIM experts from around the world. Heaney has divided the 586 page book into four main sections which are prefaced by a comprehensive market review outlining growth in the global MIM industry.

Part one of the main section of the book is a comprehensive review on 'Processing' which includes key criteria for designing MIM components, the manufacture and characterisation of metal and alloy powders used in MIM, MIM tooling and the essentials of the powder injection moulding process. Also covered in this section are debinding and sintering.

Part two covers the characterisation of MIM feedstock, modelling and simulation of the MIM process, the qualification of MIM parts, common defects found in different stages of the manufacturing process, and the important issue of the control of carbon content in MIM processing.

Part three of the book includes contributions from industry experts



on microMIM, 2-material and 2-colour PIM, and micro-porous MIM. Part four is devoted to specific material types used to produce MIM parts such as stainless steels, titanium, thermal management materials for electronics, soft magnetic materials, high-speed tool steels, and MIM of heavy metals, refractory metals and hard materials.

The new book will be a most useful reference tool for MIM researchers, producers and end-users alike.

'Handbook of Metal Injection Molding', Woodhead Publishing Ltd, Cambridge, UK. June 2012, 586 pages. £170 ($290). Edited by Donald F. Heaney.

www.woodheadpublishing.com

Arburg expands its PIM laboratory with additional testing and development facilities

In the spring of 2012 a new Powder Injection Moulding laboratory went into operation at German injection moulding machine maker Arburg GmbH + Co KG, based in Lossburg. The laboratory is used by in-house specialists in the processing of MIM and CIM materials for the further development of the process and for supporting customers in various aspects of the PIM process.

Services at Arburg for the PIM sector are now divided into two departments. In the company's Customer Centre, Powder Injection Moulding is presented in-depth on an Allrounder 270 S 400-70 specially adapted for the process. Associated upstream and downstream processes such as material preparation, debinding of the green compacts, sintering of the brown compacts and analysis of the finished parts are performed at the newly refurbished PIM laboratory in order to further enhance the process itself.

Marko Maetzig, who is responsible for applications development in the PIM team explains the capabilities of the new PIM laboratory. "For feedstock preparation in our new PIM laboratory, we use a kneading machine as well as a new shear roller extruder, which mixes and homogenises the material before bringing it into granulate form. We can consequently offer our customers a tailor-made feedstock."

"Using our systems, debinding can be performed either thermally, catalytically or using solvents. Our ceramics sintering furnace is fully automated and we can link the thermal debinding and sintering steps, feeding and removing the parts automatically via a transport system."

This system is complemented by further laboratory furnaces for tasks such as sintering injection moulded metal powder (MIM) parts. Moreover, a device for simultaneous thermal analysis is employed in order to determine weight losses and material behaviour during debinding as a function of temperature.

"Here, for example, the reactions that the materials undergo during debinding and the consequences of these reactions on the subsequent process can be identified," stated Maetzig.

Finally, the PIM specialists examine and analyse the samples in a targeted manner using optical microscopes and precision scales.

Hartmut Walcher, who is in charge of applications engineering consulting in the PIM department, describes the full scope of the service. "With our production area and the new laboratory, the infrastructure is in place in order to provide individual support to our customers throughout the entire PIM production process, from material selection through the production of sample parts. Whether it be prospective or existing customers, with our equipment we are in a position to provide comprehensive support and make entry into PIM processing simpler and safer."

www.arburg.com

At the new PIM laboratory, the Arburg specialists have all the facilities for preparing, debinding, sintering and comprehensively analysing MIM and CIM materials

Submitting News

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

MPIF 2012 award winning Metal Injection Moulded parts

Winning parts in the Metal Powder Industry Federation's (MPIF) 2012 Powder Metallurgy Design Excellence Awards competition were announced at the PowderMet 2012 International Conference on Powder Metallurgy and Particulate Materials, Nashville, June 10-13. MIM technology once again featured prominently amongst the award winners, with seven prizes being awarded to producers based in the USA and Asia.

Grand Prize Awards

Aerospace/Military

FloMet LLC, Deland, Florida, USA, won the grand prize in the aerospace/military category for a very complex 17-4 PH stainless steel rotor made by metal injection moulding (MIM) and used in a hand-emplaced munition device (Fig. 1).

The intricate design is demonstrated by its four holes on two perpendicular planes, two angled slots with square corners, and numerous internal and external radii, flats, slots, and cutouts. All of these features require very tight tolerances: 0.0025 inches to 0.005 inches. Moreover, the square bottom-hole could only be formed by MIM because prior attempts with other fabrication processes, including machining, proved unsuccessful.

It is estimated that the machined version of the part could cost as much as five times that of the MIM design. The rotor is made to a density of 7.5 g/cm^3 and has an ultimate tensile strength of 75,000 psi, yield strength of 25,000 psi, a six percent elongation, and 27 HRB hardness.

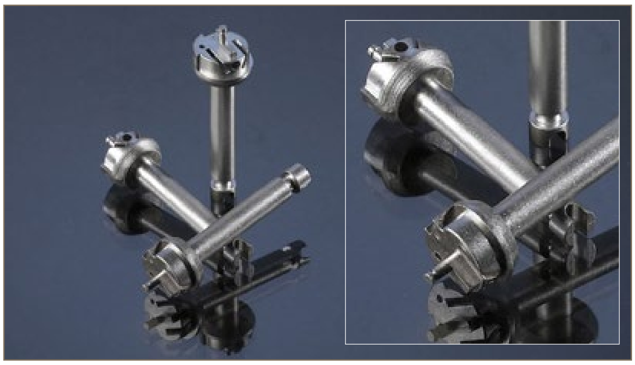


Fig. 1 A MIM stainless steel rotor for a military application manufactured by FloMet LLC, USA

Fig. 2 MIM components manufactured by Parmatech Corporation for use in a minimally invasive surgery device

Medical/Dental

Parmatech Corporation, Petaluma, California, USA, won the grand prize in the medical/dental category for a mechanical introducer device used in minimally invasive OB/GYN surgery (Fig. 2). Made for STD Med, Stoughton, Massachusetts, USA, the device contains five 17-4 PH stainless steel MIM parts: right and left cover, curved needle, curved needle linkage, and centre linkage. The covers have a complex three dimensional geometry incorporating assembly pins and slots for moving the internal parts that require smooth action for suturing.

Formed to a net shape requiring no machining, the parts are assembled by the customer who performs laser welding on the cover seam/joint. The parts are made to a density of 7.65 g/cm^3. Choosing the MIM process provided a 70% cost saving over equivalent machined parts.

Consumer

The grand prize in the consumer market segment category was won by Smith Metal Products, Lindstrom, Minnesota, USA, for a 17-4 PH stainless steel MIM top and bottom of an eyeglass-frame bridge made for Superfocus LLC, Van Nuys, California (Fig. 3). Featuring very thin walls, the parts form the bridge section over the nose, which also houses an actuator for changing the magnification level of the glasses. The as-sintered density is 7.6 g/cm^3.

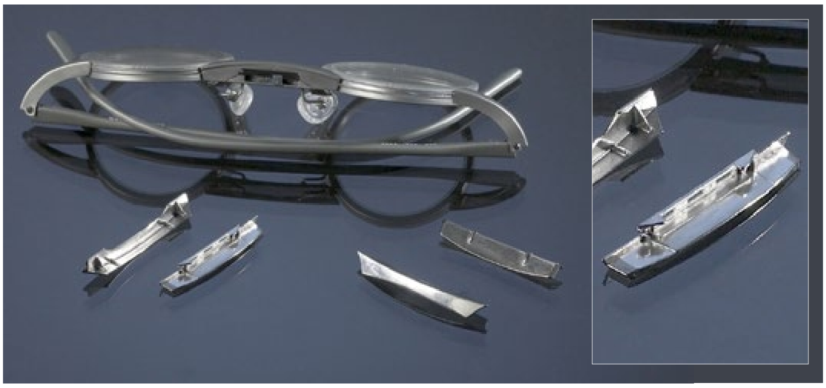


Fig. 3 MIM stainless steel components manufactured by Smith Metal Products and used in an adjustable focus eyeglass frame

Awards of Distinction

Automotive: Engine

SolidMicron Technologies Pte Ltd., Singapore, won the first award of distinction in the automotive—engine category for a 440C stainless steel MIM sealing seat used in a direct fuel injector assembly (Fig. 4).

Made for Magneti Marelli S.p.A. Powertrain, Bologna, Italy, the complex part has a multiple angled slot and top holes requiring precise tool design and fabrication. The near-net-shape part has a density range of 7.54 to 7.65 g/cm^3, a tensile strength of 232,000 psi, and yield strength of 203,000 psi.

The MIM process provided a 30%–40% cost saving over alternative manufacturing processes.

Fig. 4 MIM sealing seat parts used in a direct fuel injector assembly, manufactured by SolidMicron Technologies Pte Ltd.

Fig. 5 MIM 17-4 PH stainless steel parts used in a hand tool and manufactured by Megamet Solid Metals, Inc.

Recreation

Megamet Solid Metals, Inc., Earth City, Missouri, USA, won the award of distinction in the recreation category for a cable-tie hand tool assembly of six 17-4 PH stainless steel MIM parts (pinion, nosepiece, pawl gripper, insertable rack, cutoff cam, and short link) made for HellermannTyton, Milwaukee, Wisconsin, USA (Fig. 5).

Made to a density of 7.6 g/cm^3, the parts are produced to net shape with only four requiring minor secondary operations such as reaming and coining.

Industrial Motors/Controls & Hydraulics

Advanced Materials Technologies Pte. Ltd., Singapore, won the other award of distinction in the industrial motors/controls & hydraulics category for 316L and 440C stainless steel parts (catch X-Z datum, catch bias Z datum, front and rear support tabs) used in an industrial printer module (Fig. 6).

Achieving a yield strength rate of 234,000 psi, the 440C tabs support and align the print head module and are subjected to impact force during printing.

The 316L catches guide and feed paper to the printing module. Choosing the MIM process over casting and machining yielded a 40% cost saving.

Electrical/Electronic Components

Smith Metal Products, Lindstrom, Minnesota, USA, won the award of distinction in the electrical/electronic components category for a 17-4 PH stainless steel lever made for Methode Electronics, Harwood Heights, Illinois, USA.

The intricate MIM part is used in a latch ejector mechanism for pluggable gigabit Ethernet connections.

All images courtesy Metal Powder Industries Federation, www.mpif.org

Fig. 6 MIM stainless steel parts used in a printer module and manufactured by Advanced Materials Technologies Pte. Ltd.

Fig. 7 A MIM 17-4 PH stainless steel lever for ethernet hardware manufactured by Smith Metal Products

Induction-heated quick-sinter system
patent pending

FCT Anlagenbau GmbH

In the course of technical development in the area of sinter plants, quick-heating systems are demanded more and more often to make production of high-performance ceramics parts more economic. The focus is on designing the procedure's steps so that continuous and plannable production of high piece numbers is possible in short processing steps to achieve best economic efficiency.

FCT Anlagenbau, one of the leading providers of high-temperature plants, has now developed an innovative plant concept with which end-contour-near sinter parts that can be subjected to a brief heating or cooling cycle can be produced at large piece numbers. This plant concept was first presented to a specialist audience with great success at Ceramitec 2012 in Munich. The plant, for which a patent is pending, is available for test runs at the technical school of FCT.

The high-performance induction furnace FCI 600/150-100-SP was developed for production of MiM parts, parts of carbide, sinter parts of ceramics or for silicon infiltration of CFC components.

As compared to conventional plants, this trend-setting production concept convinces with its continuous multi-chamber system in module build that permits flexible adjustment. Production is possible in inert gas atmosphere and/or in vacuum operation. Quick heating rates by inductive heating permit short cycle times. Added to this are energy savings of about 30 percent - an important contribution in respect of sustainability. Lower life time costs are achieved by lower maintenance costs both in material effort and maintenance effort. An independent parts geometry of the products is possible by use of crucibles as carriers.

For more information please contact us.

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PM2012 World Congress Preview: Japan's MIM industry in the spotlight

The PM2012 Powder Metallurgy World Congress & Exhibition, to be held at the Pacifico Yokohama Convention Centre, Japan, October 14-18, promises to be the most important event of the year for the MIM industry. With industry leaders attending from around the world, and the participation of some of Japan's leading MIM parts producers in the exhibition, we report on the current status of MIM in Japan and preview what PM2012 has to offer for delegates and exhibition visitors alike.

Japan's MIM industry: An Overview

Asia continues to grow as the major force in the global MIM industry and the region now consumes more than 50% of global MIM grade powders. Within Asia, the industry is rapidly evolving thanks in large part to significant and ongoing capacity expansion, notably in China, where the number of MIM producers continues to grow and is conservatively estimated at 75. In terms of sales value, however, Japan's MIM industry remains the largest in Asia, with an estimated 30 companies generating sales of more than $150 million.

The impact of competition from lower-cost neighbouring countries has forced Japan's MIM producers to focus on higher value markets, where, for example, innovations in materials and processing offer a competitive advantage. Japan can today be considered to be at the cutting edge of a number of advanced MIM technologies, from microMIM to the MIM of titanium and superalloys, supported in large part by a number of internationally recognised research institutions.

With commercial MIM production starting in the late 1980's in Japan, the country also benefits from a significant "head start" on many of its neighbours. However the technology gap will inevitably continue to close over the remainder of the decade.

The Japan Powder Metallurgy Association (JPMA) has for many years collected and published detailed data on the growth of MIM in Japan, offering a unique insight into the development of the industry. The association's latest figures, which include 2011 component sales, show that Japan's MIM producers have started to recover ground that was lost as a result of the financial crisis, and later by the devastating earthquake and tsunami in north east Japan in March 2011.

Japanese MIM sales in 2011 reached 11.6 billion Yen, a 2.8% decrease on 2010 sales of 11,952 million Yen (Fig. 1). This dip can be directly attributed to the impact of the tsunami and the JPMA states that it is confident that Japan's MIM industry will enjoy a strong recovery in the coming years.

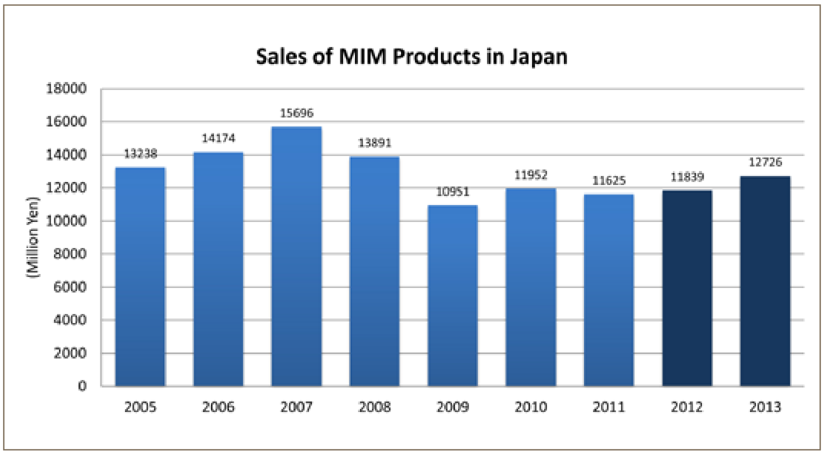


Fig. 1 Sales of MIM products in Japan, 2005-2013 (2012 and 2013 estimated). (Data courtesy JPMA)

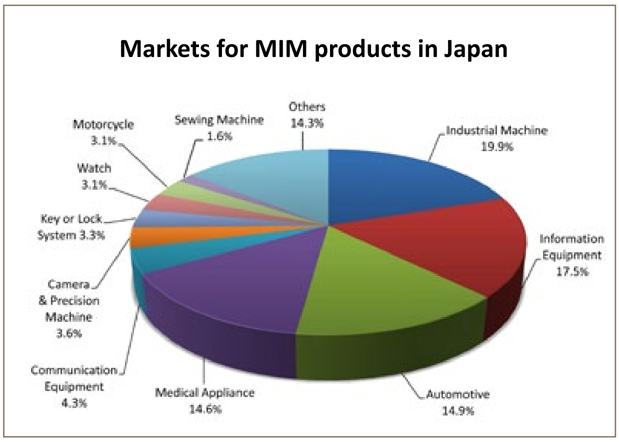


Fig. 2 Markets for MIM products in Japan, FY2011 (Data courtesy JPMA)

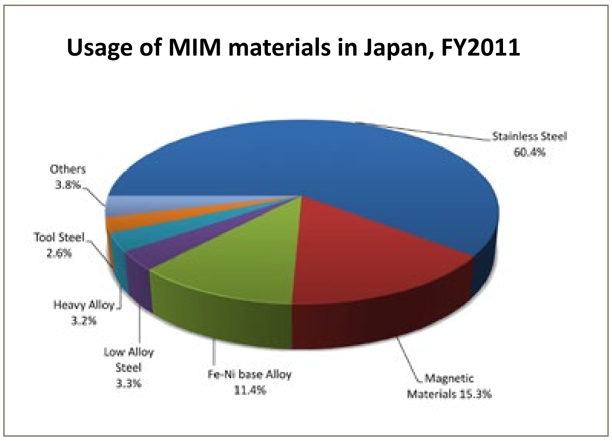


Fig. 3 Usage of MIM materials in Japan, FY2011 (Data courtesy JPMA)

The evolving market for MIM products

Japan's MIM industry supplies a diverse range of end-user markets, as can be seen in Fig. 2. Industrial machine parts accounted for 19.9% of sales (2010: 21.5%), parts for IT equipment accounted for 17.5% of sales (2010: 15.7%), automotive parts accounted for 14.9% of sales (2010: 15.6%), medical appliance parts accounted for 14.6% (2010: 14.7%), and the communications sector accounted for 4.3% (2010: 6.8%).

The JPMA states that the growth of the IT equipment sector can be attributed to an increase in the production of printer components from Permendur. This material has the highest saturation flux density among the soft magnetic materials. The magnetic characteristics of Permendur when processed by the MIM route are reported to be significantly better than those manufactured by alternative processes such as casting and rolling. The material's high resistivity leads to lower eddy current loss and a lower heating value.

Japanese MIM materials usage

Stainless steel continues to be the most widely used material in Japan's MIM industry, accounting in 2011 for 60.4% of production (2010: 64.1%). Together, stainless steels, magnetic materials and Fe-Ni alloys accounted for around 87% of production (Fig. 3).

As previously stated, magnetic materials have experienced the most significant increase in growth rising from 12.2% in 2011 to 15.3% in 2012.

PM2012 World Congress: Presenting a global perspective on the PIM industry

The PM2012 World Congress promises to offer those involved in the PIM industry an unrivalled insight into the current status and future prospects for the technology.

Special Interest Seminar 1: New Developments and Trends in Powder Injection Moulding
Thursday 18 October, 09.00-12.10

PM2012's Special Interest Seminar SIS1 features six invited presentations from academic and industrial experts that will provide a broad coverage of the possibilities of MIM and CIM technology, from its material selection to emerging market areas, integration of additional functions, miniaturisation and future R&D directions in simulation, optimisation, informatics and process-integrated quality control. The presentations as scheduled are:

- Development and Trends in North American PIM
  A. Bose, Materials Processing, Inc., R. M. German, San Diego State University (USA)
- New Developments and Trends in Metal Injection Moulding
  F. Petzoldt, Fraunhofer IFAM (Germany)
- Development and Trends of MIM in Taiwan
  K.-S. Hwang, National Taiwan University (Taiwan)
- New Developments and Trends in Korean PIM
  S. J. Park, Pohang University of Science and Technology (POSTECH), S. W. Lee, Pusan National University, Y.-S. Kwon, CetaTech, Inc. (Korea)
- A Review of the MIM Industry in China
  Y. Li, H. He, Central South University (China)
- Recent MIM Activity and Standardisation in Japan
  Y. Kato, Kato Professional Engineering Office (Japan)

In addition to this Special Interest Seminar, more than 35 papers in the technical programme address all aspects of PIM production, from innovative processing to new materials and applications.

For further information on the PM2012 World Congress and Exhibition visit: www.pm2012.jp

PM2012 exhibition attracts Japan's leading MIM parts producers

Following the success of Japan's MIM pavillion at the 2000 Powder Metallurgy World Congress in Kyoto, a number of Japan's leading MIM producers are once again using the PM World Congress as a platform from which to promote the MIM process. The following profiles reflect the continued optimism of Japan's MIM producers, as well as a willingness to meet and discuss the future development of the MIM industry with visiting producers from around the world.

CASTEM CO., LTD.

Hiroshima, Japan | Stand 2-15

The Castem Group is a leading Japanese manufacturer of MIM and CIM components, as well as products via the investment casting process and the FRP injection process. The company supplies a diverse range of industries, from medical devices to aerospace, rail, construction and precision devices.

Castem's President and CEO Mr. Takuo Toda outlined for *PIM International* the company's approach and future strategy for the development of the PIM industry. "Our company has succeeded in reducing the cost of PIM tooling thanks to a number of specialised operations. In the future, we wish to continue to meet the interests and needs of our customers through the experiences of producing a wider selection of lower volume products."

Mr. Toda continued, "In the business of MIM, we are trying to address the needs of customers by proposing high-value added products. We intend to grow as a comprehensive metal products manufacturer by further improving additional fabrication steps such as machining, assembly and so on."

Recent technology developments at Castem include a MIM tooth implant manufactured with a functionally gradient structure using pure titanium and 6Al-4V titanium alloy (Fig. 4). The surface is pure titanium formed as a porous layer, over a core of fully densified 6Al-4V titanium alloy. The company is also manufacturing aerospace turbine blades with CIM cores using the investment casting process.

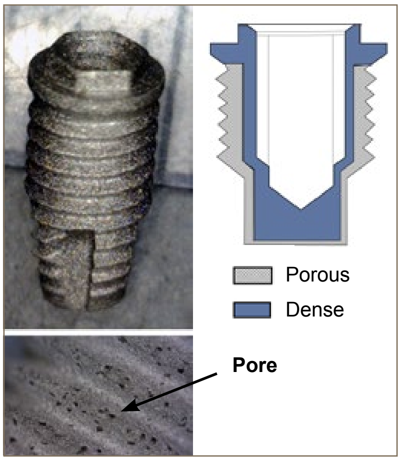


Fig. 4 A MIM tooth implant manufactured with a functionally gradient structure (Courtesy Castem Co., Ltd.)

Looking ahead to the PM2012 World Congress, Mr. Toda stated, "I have taken part in so many international conferences where many of the presentations have failed to address the needs of the industry. At PM2012, we are promoting the global standardisation of MIM. To grow, detailed and clear standards are very important, and we will re-state the continued importance of this at PM2012."

www.castem.co.jp

OSAKA YAKIN KOGYO CO., LTD.

Osaka, Japan | Stand 2-11

Osaka Yakin Kogyo was established in 1942 and celebrated its 70th anniversary in 2011. Throughout its existence, the company has strived to serve the needs of the heat treatment of metal by improving heat treatment technology, expansion of equipment, and promoting quality assurance activities.

Dr. Shuntaro Terauchi, President of Osaka Yakin Kogyo, explained, "Starting with our introduction of vacuum heat treatment technology in the 1970's, and later through our introduction of vacuum carburising furnaces and one of the largest vacuum heat treatment furnaces in Japan, we have made every effort to keep up with the trends of our industry. In the 1990's, we started manufacturing sintered parts by the then new process for metal parts called Metal Injection Moulding."

"In 2003 we introduced a gas atomisation unit to help us meet the needs of customers, including development of new MIM materials," Dr. Terauchi stated. "Today we provide MIM parts with improved mechanical properties by achieving high densities using pressure-assisted sintering. We also achieve higher reliability thanks to our microfocus X-ray inspection systems and microfocus X-ray CT (Computed Tomography) inspection systems. Our plant was certified according to ISO9001 in 2011."

"We have endeavoured to maintain and further enhance the quality of our products. We will continue to try contributing to production technologies and value-added products of customers. We hope you remember "Osaka Yakin Kogyo" as a company constantly pursuing metal materials and metallurgy."

Commenting on the PM2012 World Congress, Dr. Terauchi told *PIM International*, "PM2012 offers a good opportunity not only to understand the actual situation and trends of the MIM industry throughout the world, but also to disseminate information about Japan's MIM industry. I hope that PM2012 enables us, especially our young people, to build up a global network of connections through the world congress and its social events, resulting in further growth of the MIM industry."

www.osakayakin.co.jp

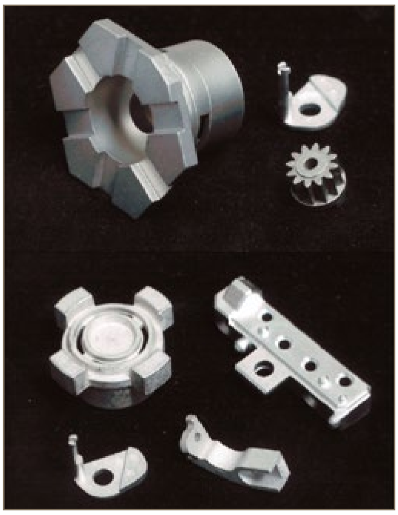


Fig. 5 A selection of MIM parts manufactured by Osaka Yakin Kogyo

Fig. 6 The EIGA gas atomiser at Osaka Yakin Kogyo

IWAKI DIECAST CO., LTD.

Miyagi, Japan | Stand 2-17

Iwaki Diecast was founded in 1968 as a precision die casting company and today manufactures die-cast components in zinc, aluminium and magnesium alloys. The company's metal injection moulding division, MOLDALLOY®, was founded in 1989. Today the company employs 300 people.

Commenting in the market outlook, Mr. Atsushi Kamada, Vice President of Iwaki Diecast, told *PIM International*, "Growth of the die-casting industry in Japan is not anticipated because of the shift to the sourcing of components closer to overseas production centres. In contrast, the MIM industry offers the prospect of steady growth in Japan in the coming years. Of course, recent growth was affected by the global financial crisis and the tsunami disaster. The Japanese MIM industry, however, was not seriously affected by these factors, and we regard this as a positive result of the fact that metal injection moulded parts are used by such a diverse range of industries."

Mr. Kamada added, "We are convinced that demand for MIM products in Japan will continue to grow, however there is still a very limited awareness of the technology and I believe that there are many untapped areas of application still to be discovered."

"We hope that at PM2012 we can see a deeper cooperation between MIM companies. There is a significant untapped market for MIM products, but it will be necessary to work hard to exploit the opportunities. The industry must, on an international basis, differentiate itself from competing technologies and improve awareness with end-users"

www.iwakidc.co.jp

Fig. 7 Metal injection moulded machine parts manufactured by Iwaki Diecast Co., Ltd.

TAISEI KOGYO CO., LTD.

Osaka, Japan | Stand 2-18

Taisei Kogyo, founded in 1972 with its head office in Osaka, is managed by its President, Dr. Shigeo Tanaka. Initially concentrating on plastic injection moulding, Taisei Kogyo has expanded over time and advanced its technology, now producing micro MIM parts, porous materials and various other products.

Dr. Tanaka told *PIM International*, "Taisei Kogyo has successfully established itself in the relatively new market of the increasingly popular MIM technology. While offering a large variety of production methods, our main focus lies in developing new applications for MIM technology, which is constantly supported by our dedicated research team."

"The obvious strengths of this popular technology have to be seen in the mass-production of geometrical components, by keeping the costs comparatively low," continued Dr. Tanaka. "This factor, paired with the large material selection and the avoidance of finishing procedures, makes MIM a very attractive moulding technology with a wide variety of applications such as medical technology, mechanical engineering, electrical engineering and so on."

Taisei Kogyo does not only apply MIM and micro-MIM as a production method but also porous MIM, which is used to produce porous materials with open and closed cells.

In comparison to other porous metals, which have been widely used in the past, Taisei has managed to develop a completely new class of materials, with a large specific surface area and other novel properties. One example of Taisei's innovative products is a porous metal paper, which is probably the thinnest metal sheet in the world right now.

"In the future you can expect Taisei Kogyo to constantly improve its technology and production methods to completely satisfy its customers," concluded Dr. Tanaka.

www.taisei-kogyo.com

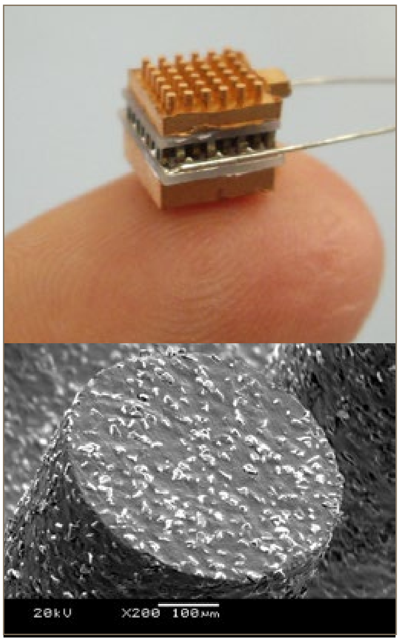


Fig. 8 A copper and diamond composite 'Super Heat Sink' produced by µMIM. The heat sink is made of a copper diamond composite with a micro-structured surface to improve thermal conductivity. Each pillar is 1500 µm high and 500 µm wide (Courtesy Taisei Kogyo Co., Ltd.)

NIPPON PISTON RING CO., LTD.

Saitama, Japan | Stand 2-14

Since Nippon Piston Ring was established in 1934, the company has manufactured and distributed a wide variety of engine components, becoming a technology leader in piston rings, cylinder liners, and valve train parts.

The company started manufacturing and selling metal injection moulded components in 1991. Mr. Teruo Takahashi, Director of Nippon Piston Ring, told *PIM International*, "We planned the production of a MIM automotive engine part quickly in the industry and we have produced injector parts and rocker arm parts for automobile engines and fuel injection systems for many years."

Since the development of metal injection moulded automotive components, Nippon Piston Ring has supplied a diverse range of markets with MIM products that include decorative clock fittings, parts for general machines and precision instruments. MIM materials processed include low alloy steels, stainless steel and soft magnetic materials, to name a just a few. The company states that it benefits from a high level of productivity thanks to a fast solvent-based system for the removal of the binder system.

Mr. Takahashi continued, "We will start the production of metal injection moulded orthodontic products this autumn as a step towards our entry into the medical field. Additionally, we intend to continue to develop new products and new materials to contribute to the development of the industry."

Commenting on the PM2012 World Congress, Mr. Takahashi stated, "I am very pleased that the PM2012 World Congress and Exhibition is taking place in Yokohama. Following the 1993 and 2000 World Congresses, this is the third to be held in Japan and I hope that the event will be a great success. The sharing of knowledge and research is essential for the development of the powder metallurgy and metal injection moulding industry."

www.npr.co.jp

Fig. 9 MIM injector parts for engine fuel control systems by Nippon Piston Ring

Fig. 10 MIM orthodontic parts manufactured by Nippon Piston Ring

EPSON ATMIX CORPORATION

Hachinohe, Japan | Stand 3-16

In October 1999, Atmix started its business as a producer of metal powder and metal injection moulded parts as a division of Japan's Seiko Epson Corporation.

Utilising its own ultra-fine powder, original binder technology, and high quality mass production systems, the company has consistently focused on the manufacture of MIM parts at a low cost.

The company today manufactures complex components for various industries, as well as for use within the Seiko Epson Corporation.

www.atmix.co.jp

Read our report on Epson Atmix Corporation on page 45 of this issue of PIM International

For full technical programme and exhibition information, visit the PM2012 World Congress website:

www.pm2012.jp

PM2012: Focus on competing technologies

A special interest seminar SIS3 at the PM2012 World Congress is dedicated to evaluating technologies that compete with PM and MIM, namely casting, forging, fine blanking and machining.

The status of such competition, state the organisers, is different in each region and with globalisation, economic considerations are no longer the only factor to consider. Technical competition to provide benefits for the customers has, for example, become more important than ever. In the session, representatives of the competitive technologies will explain their own technologies. In addition, PM and MIM producer GKN Sinter Metals presents its outlook on the challenge of competition.

- Recent Trend of Casting Technology and Applications
  K. Shimizu, Muroran Institute of Technology (Japan)
- The Latest Trend and Application Example of Forging Technology in Japan
  T. Ishikawa, Nagoya University (Japan)
- Fineblanking
  K. Hayashi, Committee on Fineblanking (Japan)
- New Technology for Functional Interface Creation Utilising Powder Jet Deposition
  T. Kuriyagawa, Tohoku University (Japan)
- PM in a Competitive Environment: New Materials, Related Processes and Improved Energy Consumption
  V. Arnhold, G. Kotthoff, R. Link, GKN Sinter Metals Engineering GmbH (Germany)

This session takes place on Monday, 15 October and is chaired by Y. Takeda (Höganäs Japan K.K., Japan) and C. Molins (AMES, S.A., Spain).

Powder Metallurgy World Congress PM2012 Yokohama, Japan 14-18 October 2012

PM2012横浜

14-18 October 2012

"Challenge for the next generation"

PM2012 in Yokohama Japan

Advanced technology and information for "Environment" and "Energy Saving" will be introduced.

Events

- ***Global Review (JPMA, MPIF, EPMA President)***
- ***Technical Program : Over 500 technical papers (Oral & Poster Sessions)***
  - ***Special Interest Seminar & Special Session Includes***
- ***Exhibition : 147 Booths (15 Countries 93 Companies)***
- ***JPMA Special Seminar (Environment & JPMA Awards)***
- ***Opening Ceremony, Welcome Reception, Farewell Party, etc.***

Organized by JPMA JSPM

Important Information

Registration

Late Registration

August 2 – October 1, 2012

▸▸▸**Further Information : www.pm2012.co.jp**

Epson Atmix Corporation: Major expansion underway after the devastation of the Great East Japan Earthquake

Epson Atmix Corporation, part of Japan's Seiko Epson Corporation, is the world's largest supplier of water atomised powder to the MIM industry, as well as being a leading Japanese producer of MIM parts. When the company's manufacturing plant, located in the Japanese coastal city of Hachinohe in the north east of Japan, was flooded by the devastating tsunami of March 2011, the impact was felt by MIM part producers worldwide. In the weeks and months after the tsunami, the staff and management at Epson Atmix worked tirelessly to restore production in challenging conditions, and the company is now in the process of completing a significant MIM powder capacity expansion.

On Friday March 11 2011, the port of Hachinohe was devastated by a tsunami that was triggered by the largest earthquake ever known to have hit Japan. Now known as the Great East Japan Earthquake, the tsunami that followed caused devastation along the north eastern coast of Japan. The wall of water that hit the Port of Hachinohe area around two hours after the earthquake was between 8-9 m high (26-29 ft). However, thanks to the geography of the area the port itself suffered a lesser tsunami of 5-6 m (16–19 ft).

The Epson Atmix powder and MIM plant, located close to the port of Hachinohe, was significantly damaged by flood waters. The tsunami swept overland and inundated the Epson Atmix site, putting more than half the buildings under a metre of seawater and debris. All power and communications were cut.

Fig. 1 A photograph taken from a roof at Epson Atmix during the tsunami

The immediate aftermath: Crisis management and recovery

By March 13, with all lifelines down, Akita Epson and the Seiko Epson Corp. Head Office delivered aid in the form of food, generators, heaters, fuel and other supplies. Employees of Epson Atmix were back at work within two days of the tsunami, digging the facility out of the mud and restoring transformers and production equipment to service.

The recovery teams used white boards to share information on progress being made on all aspects of the operation, from mud and debris removal to power restoration and equipment repair, at all times trying to keep employees as well-informed as possible.

As part of its risk management, Seiko Epson Corp. has in place a crisis management programme to handle major risks that could have a material impact on the management of the Group. The programme also outlines the swift initial response needed in the event of a devastating earthquake or other major disaster. The response would be led by the company's president.

Immediately after the March 11 disaster, Seiko Epson Corp. launched its Central Disaster Task Force, headed by the president, at its head office in accordance with the company's crisis management programme. The Task Force served as the administrative centre for the crisis management committee, which consisted of high-level managers with the specialised

Fig. 2 An artists preview of the new metal powder plant at Epson Atmix

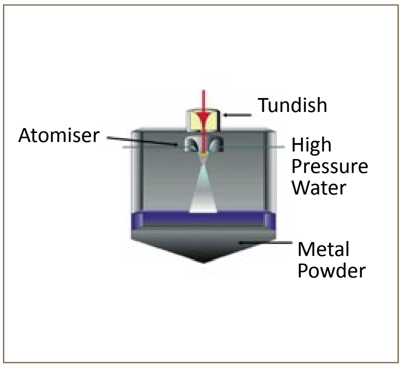


Fig. 4 Outline of the atomisation process at Epson Atmix

skills needed for the response, including human resources, general affairs, public relations, production planning, safety and environment. After gathering and analysing information on the damage in each of the affected regions and business sites, the Task Force decided the best plan of action and quickly moved to implement it.

Seiko Epson Corp. stated, "In the first stage of the response, we dedicated all resources to verifying and ensuring the safety of employees and their families. We then built an organisation aimed at sustaining and recovering operations at the damaged sites and began working with the related operations divisions and Group companies."

There was thankfully no loss of life amongst Epson Atmix employees.

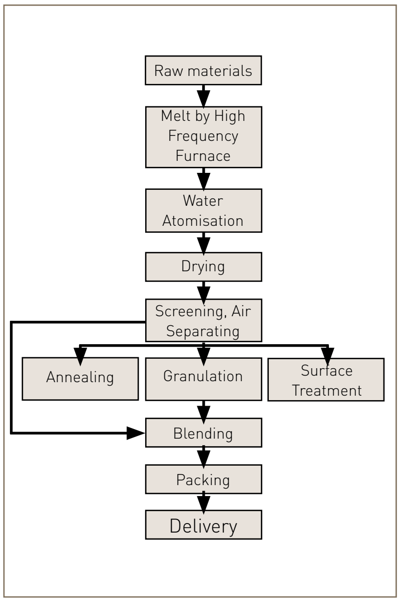


Fig. 3 Flow chart showing the complete powder production cycle at Atmix

The restoration of services

One of the biggest obstacles to the restoration of metal powder and MIM part production was the supply of electricity and industrial gas. Immediately after the disaster around 4.4 million households in northeastern Japan were left without electricity, and in many areas supplies were only able to be restored for limited periods, a situation which continued into the summer of 2011.

Power was initially restored to the Epson Atmix plant on March 31 by which time a heavily damaged electric furnace used in the production of metal powders had been repaired and could again be fired up. This enabled limited metal powder production to resume by April 15, a remarkable one month after the tsunami.

The production of MIM components and artificial quartz was able to commence as early as April 4, thanks in large part to the elevated location of the buildings housing the facilities for these products.

During this restoration period, Epson Atmix prioritised communication with its customers, aware of the impact that a shortage of its MIM grade powder would have on the ever growing global MIM industry. As soon as regional transportation services resumed, the company was able to ship powder from its warehouse that had been manufactured prior to the flooding. Alternative products were sourced where possible from external suppliers and new powder production was despatched as and when available.

'The new plant will triple Atmix's current production capacity in water-atomised superfine alloy and magnetic powder to approximately 10,000 tons per year'

Expansion of MIM grade powder production reflects optimism for the future of the MIM industry

On December 8 2011, just nine months after the tsunami, Seiko Epson Corp. announced a 3.2 billion Yen ($40 million) investment in new MIM and magnetic powder production facilities at Epson Atmix in Hachinohe. This dramatic expansion in capacity is a reflection of Epson Atmix's optimism for the continued growth of the international MIM industry.

The new plant will triple Atmix's current production capacity in water-atomised superfine alloy and magnetic powder to approximately 10,000 tons per year, enabling it to meet expanding demand from growing markets for goods such as smartphones and other high-performance mobile devices, automobiles, and medical equipment. The plant will have approximately 3,300 m^2 of factory floor space on a

20,000 m² site, and will begin operations in October 2013. It is expected that the plant will initially employ between 40-50 people. Construction of the plant was started in mid-June 2012.

Commenting at the time of the announcement, Mr. Satoshi Oguchi, President, Epson Atmix, stated, “We are very excited to announce the establishment of our new plant. We are determined to serve our customers’ growing demands for high quality magnetic powder and MIM powder, and this plant will help us to better serve their needs.”

The ongoing development of water atomised MIM powders

MIM grade superfine stainless steel powders have been produced in Hachinohe since 1982 when Pacific Metals Ltd (PAMCO) started high volume powder production using a newly developed high pressure water atomisation process. Epson Atmix took over powder production at the site in 1999, when Seiko Epson Corp. purchased the metal powder division of Pacific Metals Co., Ltd. The company’s superfine alloy powders have evolved into two main types according to the materials from which they are made and their uses: MIM grade alloy powders, mainly stainless steels, and magnetic powders.

The company produces these superfine alloy powders using a modified high-pressure water atomisation process. In this process, metal that has been melted in a high-frequency induction furnace is atomised by blasting it with pressurised water at up to 1000 kgf/cm² (Fig. 4). The atomised metal is then rapidly cooled, producing a powder with regularly-sized, micron-order particles, and uniform composition and characteristics.

Atmix states that particle size is directly related to water pressure during atomisation, with particle shape depending on the water jet angle. Exact control of the combination of these two criteria allows for the manufacture of spherical shaped powders specifically suited to MIM production.

Today, the company’s powders are manufactured using one of three atomisation methods. The F Method uses Atmix’s original powder production system, enabling it to efficiently produce fine powders ideally suited to MIM. The R method is an improved version of the F method, offering highly spherical powders with lower oxygen content. Particle sizes (D_{50}) range from 1 – 50 µm.

A final atomisation process used at Epson Atmix, called the Spinning Water Atomisation Process (SWAP™) method, combines gas and water atomisation to produce unique amorphous powders (see overleaf).

Following atomisation, powders are dried using a vacuum drying system. The powder is then screened using sieves capable of sieving down to 30 µm. Below this, an air separator system separates powders into the final product ranges.

Whilst the company has primarily been producing high-alloy steel fine powders such as stainless steels, it states that low-alloy fine powder for structural PM parts and automobile parts are also produced, with low oxygen content and finer particle sizes to improve sinterability.

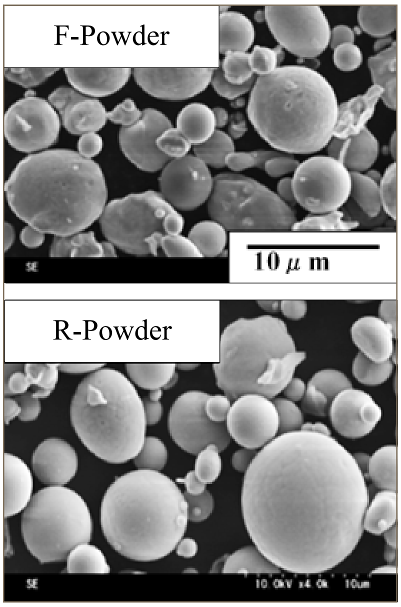


Fig. 5 Typical shapes of F and R powder grades

‘Atmix states that particle size is directly related to water pressure during atomisation, with particle shape depending on the water jet angle’

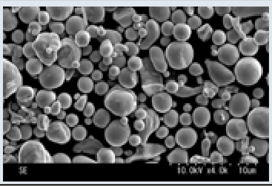
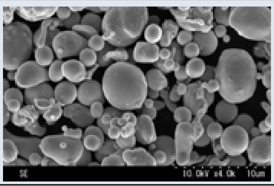
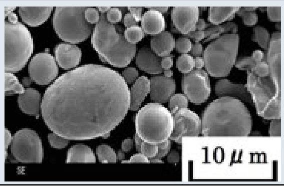
		PF-3F (Super Fine Powder)	PF-5F (Super Fine Powder)	PF-15F
Mean powder size (D_{50})	µm	2≈4	3≈5	7≈9
Tap density	g/cm³	3.6	3.9	4.3
Specific surface area	m²/g	0.66	0.57	0.25
Oxygen contents	ppm	5,000	4,000	3,500
Powder shape				 10 μ m

Table 1 Properties of 316L F grade Super Fine powder

MIM parts production at Epson Atmix

MIM parts production at Hachinohe dates back to 1989 when Pacific Metals Ltd established a facility to produce MIM stainless steel parts and soft magnetic parts. This facility was acquired by Seiko Epson in 1999 along with the powder production plant described above. The company's MIM parts plant was further expanded in 2003 following the merger of Atmix and Japan's Injex Corporation. Atmix then operated two separate MIM part plants until September 2007, when all the facilities and knowledge of Injex were transferred to Hachinohe.

Epson Atmix, as both a powder producer and a MIM parts maker, is in a unique position within the industry. The company believes this position brings positive benefits to the industry, for example by contributing to the growth of customers' business through the sharing of materials expertise and data relating to the Atmix powder range.

Today, a significant proportion of the company's MIM production is for use within the Seiko Epson Corp, with a core product being MIM yokes for dot matrix printers. The company currently operates 20 injection moulding machines, and uses its own original binder technology.

Outlook

The current investments in fine powder production capacity reflect Atmix's confidence in the continued growth of the MIM industry worldwide. Commenting on the outlook for the MIM industry in the next 5-10 years, Atmix's Ryo Numasawa stated, "We expect that the MIM manufacturers who will prosper will be those that have advanced technology, particularly in the areas of precise dimensional control. They must, of course, also possess high levels of cost competitiveness."

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Epson Atmix triples amorphous alloy powder production capacity

The global demand for amorphous alloy powders has been expanding at a rapid rate in recent years driven by the increasing demand for end products such as smart phones, notebook PC's, and electromagnetic shields for flat screen TVs. The amorphous magnetic alloy powders can be shaped into a variety of complex shapes such as inductors, choke coils, and reactors used to control voltages in electronic equipment.

As a result of this growing demand, Epson Atmix has announced that it began volume production at the beginning of August 2012 of amorphous alloy powders at a new plant in Hachinohe using its unique spinning water atomisation process (SWAP™).

The new SWAP™ plant represents an investment of approximately Yen 200 million ($2.55 million) and has a capacity to produce around 1000 tonnes/year of powder. It brings Epson Atmix's total capacity for amorphous alloy powders by SWAP™ to approximately 1500 tonnes, and makes the company one of the few in the world that can bulk produce such amorphous powders.

The SWAP™ technology (see illustration) used by Epson Atmix was first developed by the company in 2004. The SWAP™ process is used to manufacture amorphous (non-crystalline) alloy powder by atomising an alloy that has first been melted in a high-frequency induction furnace with the molten metal, then being atomised using high-pressure gas and cooling water. Super-cooling at rates of several hundred thousand degrees Celsius per second effect rapid solidification.

The resulting amorphous alloy powders have high magnetic flux densities and low energy loss in addition to excellent high-frequency characteristics. Amorphous alloys are lightweight and also have excellent electric and thermal conductivities, as well as high tensile strength.

The characteristics of such amorphous alloy powders make them extremely attractive as performance-enhancing, highly functional material powders that enable small, low-power voltage control components and that support high frequencies and large currents.

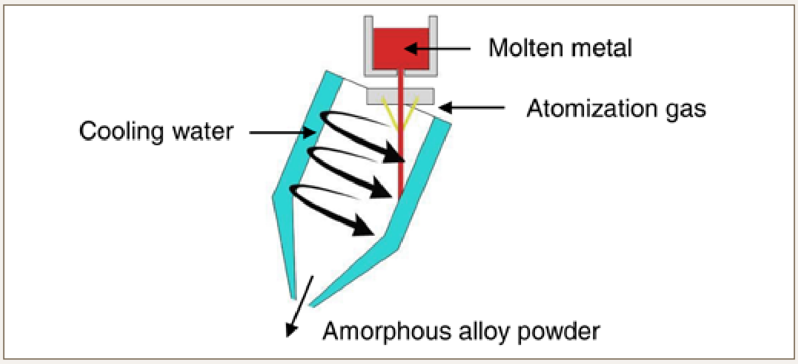

Fig. 6 The SWAP™ technology used by Epson Atmix was first developed by the company in 2004

Hardex: Expertise in Ceramic Injection Moulded components for Swiss luxury watches

Ceramic Injection Moulding continues to enjoy considerable success in the Swiss luxury watch industry thanks to the unique combination of technical and aesthetic qualities that the process is able to offer. Hardex, based near the French border with Switzerland, is part of a larger group of companies that has supplied components to the Swiss watch industry for more than 160 years. *PIM International* reports on the company's CIM activities and presents an overview of the use of CIM and MIM by the Swiss watch industry.

The luxury watch industry is dominated by Swiss manufacturers. With a near monopoly on this market, the famous phrase "Swiss Made" has become the essential component of any luxury watch. In 2011 the Swiss watch industry exported watches worth more than CHF 19.3 billion ($20 billion), a record rise of nearly 20% on the previous year, clearly showing that, at least within the luxury market, the global financial crisis has had little impact. As a percentage of units sold, luxury watches typically account for 1% of global sales, however in terms of value, they account for approximately 55%. A watch that is considered "accessible luxury" typically retails from $1,000, with "exclusive luxury" watches retailing from $10,000. Each luxury brand keeps a tight grip on product development, manufacturing, distribution, marketing and sales.

The combination of high fashion, advanced engineering and strong brand building has helped Swiss watch makers to dominate this market and create the most value in the global watch industry. Swiss luxury watch-makers, however, no longer strive to manufacture watches that are ever more accurate, for example. Instead, innovation today lies in such areas as design, materials and functionality. Of course as with all business, production efficiency and cost management are always important factors.

Fig. 1 The Executive Dual Time watch by Ulysse Nardin. Hardex manufactured the black CIM zirconia top ring and pushers

Since the 1980's, Ceramic Injection Moulding (CIM) has therefore enjoyed huge success in the Swiss watch industry, specifically because it has enabled watchmakers to differentiate themselves with new designs, to apply advanced, innovative and high performance materials in their products and, last but not least, to take advantage of the manufacturing efficiencies that the process is able to deliver.

The 1980's saw the launch of a number of exclusive CIM watch designs, including Rado's Integral in 1986 and soon afterward's IWC Schaffhausen's Da Vinci Chronograph Ceramic. Rado followed in 1990 with

Fig. 2 The rear case of the Easy Diver watch by Roger Dubuis

Fig. 5 Various CIM zirconia components manufactured by Hardex

its striking Ceramica designs. These early milestones for CIM were later superseded by the success of Chanel's J12 ceramic watches. The J12 range demonstrated more clearly than ever the potential of CIM not only as a desirable material from a technical perspective, but also as an aesthetically desirable product that is able to be used successfully as a material for luxury jewellery as much as for high-end watch making (see inset box, page 54).

CIM watches and watch components do, of course, have a number of unique appealing characteristics. They are extremely hard, with high resistance to heat, corrosion and scratches. They are inert, lighter than steel, have a silky smooth touch, and unlike metals, they feel comfortable against the skin and are never "cold to the touch" in winter.

Hardex's relationship with the Swiss watch industry

Through the history of its subsidiary Cheval Frères, France's IMI Group has been involved with the luxury watch making industry for 160 years. Based in Besançon, Franche-Comté, which borders the key watch making region of Switzerland, the group manufactures numerous components for high-end

Fig. 3 The Black Swan watch by Roger Dubuis featuring a CIM zirconia top ring and back ring manufactured by Hardex

Fig. 4 An example of a polished CIM component manufactured by Hardex

MIM and the Swiss watch industry

The Swiss watch industry has successfully leveraged the benefits that Metal Injection Moulding (MIM) processing offers for several decades.

The first MIM nickel-free 316L stainless steel watch cases for Swatch came off ETA Manufacture Horlogere Suisse's in-house MIM production line in Grenchen, Switzerland in October 1994. These watch cases, manufactured in one of the world's largest MIM facilities, made a significant contribution to the success of ETA's range of Swatch Irony watches.

The legendary DiaStar hardmetal watch case, based on tungsten carbide-cobalt mixtures, was developed by Rado Watches. Part of the ETA Manufacture Horlogere Suisse (Swatch Group) since 1983, it made its first appearance as far back as 1962 as a press and sintered case manufactured by Metallwerk Plansee in Reutte, Austria, although it did eventually convert to MIM as the shaping process in the mid-1980's. It had a number of innovative features such as its high scratch resistance thanks to the high hardness of the hardmetal case and subsequent coating, and its oval shape was unique for its time. The watch has enjoyed 50 years of uninterrupted success.

In 2005 a new range of the DiaStar, renamed Rado The Original, emerged under the slogan 'reinforced, rejuvenated, and enhanced'. It was barely altered from the original except that its scratch resistance properties had been enhanced even further due to the use of improved hardmetal grades. The injection moulded watch cases are polished with diamond powder, and in addition to the natural steel colour of WC-Co hardmetal, Rado offers coated cases.

With the continuing advances in MIM processing, the technology is set to remain a very attractive option for luxury watch manufacturers, particularly with the growing potential of materials, such as titanium, that can continue to satisfy the requirements for innovation, exclusivity and performance.

watch brands, as well as for leather goods, jewellery and the medical sector. Components range from watch faces to crowns, jewel bearings, pushers, levers and numerous other components used in watch movements.

Diversification has always been an important aspect of the business and in 1974, Cheval Frères started to design and manufacture laser machines for industrial applications such as cutting, welding, drilling and marking, leading to the establishment of the company Laser Cheval.

It was in the 1980's that Cheval Frères, following the watch making crisis of the time, diversified into the manufacture of ceramic components as a subcontractor to IBM. The expertise that was developed in this area eventually led to the formation of Hardex in 2006, drawing on more than 25 years of experience in the manufacture of ceramic products.

When Hardex was founded, it was decided that the company's expertise should not only be limited to machining and finishing of all types of ceramic products, but also to offer a complete production chain through the introduction of ceramic injection moulding.

Hardex's Manuel Debrosse told *PIM International*, "The objective was to access the emerging markets, not only for CIM technical products, but for zirconia components and products for consumer goods in the luxury market, such as pens, jewellery, etc."

CIM manufacturing at Hardex

Hardex today has a complete CIM production line that extends to a number of essential post-sintering and assembly operations. Debrosse stated, "Our CIM facility benefits greatly from other companies in the group; Cheval Frères for the machining and manufacture of components from stainless steel 316L, titanium and precious metals, and Laser Cheval for laser technology for engraving. We also have the technology in-house for fine and deep marking, for lacquer filling, and adding logos or trademarks by roughening ceramic products."

> *'the traditional requirement for high production volumes does not apply to the same degree with watch components for the luxury market'*

"Our R&D efforts are ongoing, which is natural considering that 80% of the products that we manufacture today did not exist in ceramic a few years ago. Thus, 10% of our staff is not only dedicated to continuously developing new products, but also the development of materials to safeguard both our independence and reliability."

For its CIM products, Hardex uses feedstock from external sources. "We use the world's leading feedstock supplier for the majority of our products, however we also evaluate other feedstock systems so that we have a full understanding of what is available," stated Debrosse.

Hardex has at its disposal various production methods, manufacturing small batches by machining, medium batches by CIM and mass production of simple shapes by dry pressing. "This flexibility allows us to serve the needs of a wide range of customers," stated Debrosse.

Unlike the majority of PIM applications, the traditional requirement for high production volumes does not apply to the same degree with watch components for the luxury market. Whereas in conventional CIM the cost is to a large extent in the tooling, with high-end CIM watch components the cost comes after sintering in the highly skilled and time-intensive grinding, polishing and quality inspection stages.

The perfect surface finish

For its CIM products for the luxury watch industry, Hardex is able to offer a wide variety of surface finishing operations, including, high-gloss polishing, brushed finishes, sandblasting, laser engraving and PVD coating.

The quality of CIM products that can be achieved for the luxury Swiss watch market can be seen in the Executive Dual Time watch by Ulysse Nardin (Fig. 1). Hardex manufactured the black ceramic top ring and pushers. Other product examples include components for the Easy Diver (Fig. 2) and Black Swan watches by Roger Dubuis (Fig. 3), the latter of which features a CIM top ring and back ring.

For such high-end luxury products, a perfect surface finish is essential and rigorous visual, dimensional and mechanical tests and inspections take place before components are released for shipment.

Applying specialist CIM knowledge to new markets

Commenting on the outlook for Hardex, Debrosse told *PIM International*, "The ambition for Hardex and the IMI Group is not only to manufacture individual components, but to provide finished products to our customers by integrating all the functions that are available through our various operations."

"We are confident that we can satisfy our clients' strong desire for creativity and innovation, thereby becoming an important player in Europe for products with both strong technical and aesthetic qualities that can be unique to each client."

"We also wish to develop partnerships within Europe, in the knowledge that we can all benefit from our experiences. We are open to commercial and technical cooperation that can help keep Europe at the forefront of the development and manufacture of high-value CIM components."

Hardex is also looking to apply its expertise in new markets. In the dental market, the company is, through the acquisition of Paris Implants, developing CIM-based angular monobloc self tapping dental implants.

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Milestones for CIM in Swiss watch manufacturing

IWC Schaffhausen's Da Vinci Chronograph Ceramic (1986) and Rado's striking black Ceramica model (1990) can be regarded milestones for CIM in the world of high-end Swiss watch design.

The success and popularity of ceramic watches reached new heights with the launch of Chanel's J12 ceramic watch (2005). Chanel produces the CIM parts for the J12 at its 8000 m² facility in the Swiss town of La Chaux-de-Fonds, where the movements are also assembled.

The use of CIM is by no means limited to luxury watches. A number of manufacturers in Asia are producing ceramic watches in high volumes for the mainstream market.*

The Chanel J12 in white ceramic

IWC Schaffhausen's Da Vinci Chronograph Ceramic

Rado's Ceramica watch

** Hardex is not associated with the Rado, Chanel and IWC watch brands*

MIM at PowderMet 2012: Advances in powder production, new materials and processing

The Metal Powder Industries Federation's PowderMet 2012 conference, held in Nashville, Tennessee, from 10-13 June, 2012 featured a number of presentations dedicated to developments in MIM grade powder production, materials and processing. Dr. David Whittaker reviews a selection of key papers for *Powder Injection Moulding International.*

Presentations on the latest developments in MIM technology again made important contributions to the programme for the MPIF's annual international conference, PowderMet 2012, held in Nashville, Tennessee, 10-13 June, 2012.

The focus of these presentations ranged from modelling and analytical studies aimed at enhancing understanding of the process most commonly applied to producing powders for MIM applications, close coupled gas atomisation, to considerations of the ever-growing spread of MIM material types and the benefits of post-processing operations, such as Hot Isostatic Pressing (HIP).

Close coupled gas atomisation of MIM powders

Close coupled gas atomisation is employed for the production of ultra-fine powder metals and alloys. In this method, the molten metal pours from a crucible that acts as a reservoir to control the flow rate of metal into the atomising chamber, which consists of a melt feed nozzle and gas manifold or die. The liquid metal is disrupted by the impact of high velocity jets of gas such as air, nitrogen or argon, just below the melt delivery nozzle exit tip, forming melt droplets, which subsequently solidify to form spherical particles. Due to the extremely high cooling rates during solidification, the powders have a refined microstructure and an improved homogeneity, providing superior structural and chemical properties.

There are many unknown issues regarding the process, because of its complexity, the deficiency of knowledge regarding gas and melt behaviour interactions and the manner in which the control parameters influence the overall process.

However, the application of numerical modelling methods, such as Computational Fluid Dynamics (CFD), is now beginning to underpin the scientific understanding of the process.

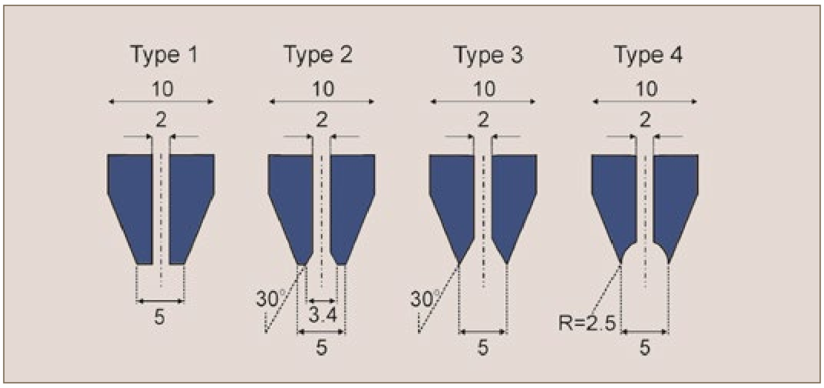

Fig. 1 Different melt delivery nozzle profile designs and dimensions (mm). From presentation by S Motaman et al. at PowderMet 2012 (Courtesy MPIF)

The effect of melt nozzle geometry of close-coupled gas atomisation

The gas flow pattern around the melt feed nozzle influences the liquid break up phenomenon and so it is important

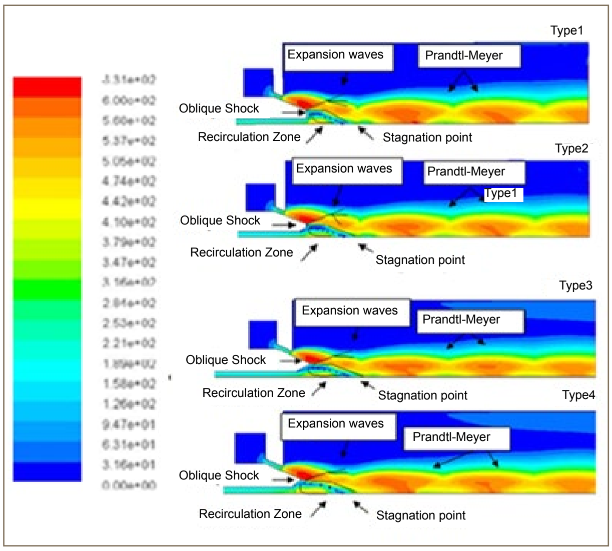

Fig. 2 Gas velocity contours for melt nozzle types 1, 2, 3 and 4 at 1 MPa gas pressure. From presentation by S Motaman et al. at PowderMet 2012 (Courtesy MPIF)

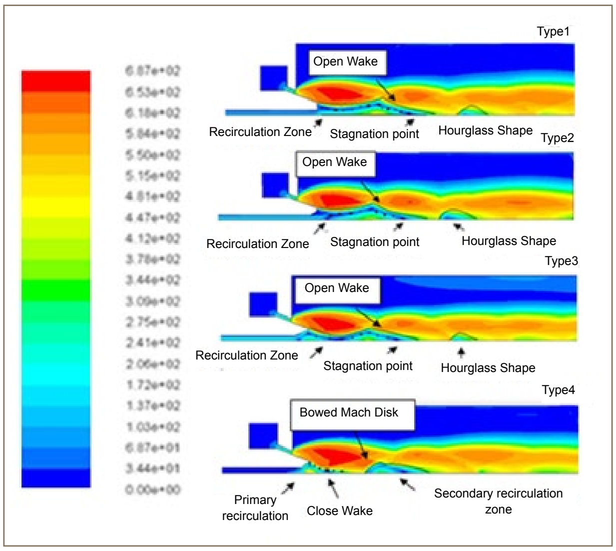

Fig. 4 Gas velocity contours for melt nozzle types 1, 2, 3 and 4 at a gas pressure of 3 MPa. From presentation by S Motaman et al. at PowderMet 2012 (Courtesy MPIF)

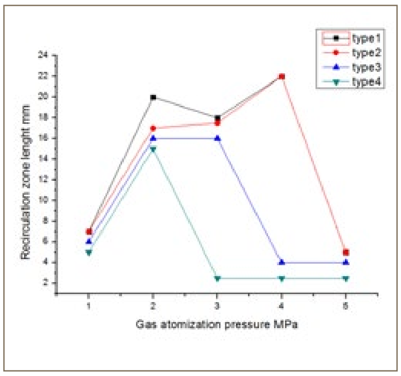

Fig. 3 Recirculation zone lengths from the nozzle tips at a range of gas pressures. From presentation by S Motaman et al. at PowderMet 2012 (Courtesy MPIF)

to determine the gas flow pattern in this region. One parameter that influences this is the internal profile of the melt delivery tip and this issue was the focus of CFD studies reported by Shahed Motaman, Andrew Mullis, Robert Cochrane and Duncan Borman (University of Leeds, UK).

The effect on the gas flow patterns of using four different internal design profiles for the melt tip of an annular slit close coupled gas atomiser (Fig. 1) has been modelled in this study.

Velocity flow field contours for the four nozzle profiles at an atomisation gas pressure of 1 MPa are shown in Fig. 2.

In all cases, a wake region occurs due to the separation of the supersonic flow from the melt nozzle's edge and is characterised by a recirculating subsonic gas flow. The far end of the wake region is determined by the position of the 'stagnation point', a point of maximum pressure and minimum velocity where the majority of the gas enters the wake region. In general, the higher the stagnation pressure, the larger the amount of gas entering the region.

At an atomisation gas pressure of 1 MPa, nozzle type 4 with the hemispherical internal shape had the smallest recirculation zone (Fig. 2). The recirculation zone length was increased for all nozzle types as the gas pressure was increased to 2 MPa (Fig. 3).

On increasing the gas inlet pressure, the structure of the flow field goes through a number of changes. The internal shocks caused by the expansion of the issuing gas move downstream. As they continue to move further downstream with increasing inlet pressure, the recompression shock that reflects from the internal sonic boundary moves far enough to move past this boundary and crosses with the rest of the recompression to form a mach disk. The formation of this disk cuts off the wake region from the surrounding flow, dramatically decreasing its size and strength. It is this phenomenon that is termed 'wake closure'. Fig. 4 demonstrates the phenomenon as, at a gas pressure of 3 MPa, the point of wake closure has been passed for nozzle type 4, but the wake remains open for the other three nozzle types.

Wake closure has been an area of intense study as it has been postulated that operation within this region may be beneficial in producing better powder products. One study, for instance, has reported that atomising just above wake closure pressure led to an increase of 42% in fine particle yield when compared to operation in open wake conditions.

At the transition pressure or wake closure pressure (WCP), the length of recirculation zone for all nozzles was affected and was truncated due to the transition from an open to closed wake condition. The study has demonstrated that the transition pressure or wake closure pressure (WCP) from an open to closed wake condition is highly dependent on the internal melt nozzle tip profile (Fig. 3). The WCP for nozzle types 1 and 2 was measured as 5 MPa while the WCPs for nozzle types 3 and 4 were observed at 4 MPa and 3 MPa, respectively.

The transition between open and closed wake is also thought to be a major cause of pulsation in the rate of delivery of melt to the nozzle during close coupled gas atomisation, and hence a contributor to the spread in the particle size distribution.

Investigation of the pulsation phenomenon in close-coupled gas atomisation

The University of Leeds group has therefore sought to supplement their numerical modelling studies by exploring whether a non-invasive technique could be developed as a means of characterising the performance of specific atomiser set-ups in terms of this transition from open to closed wake conditions. The results

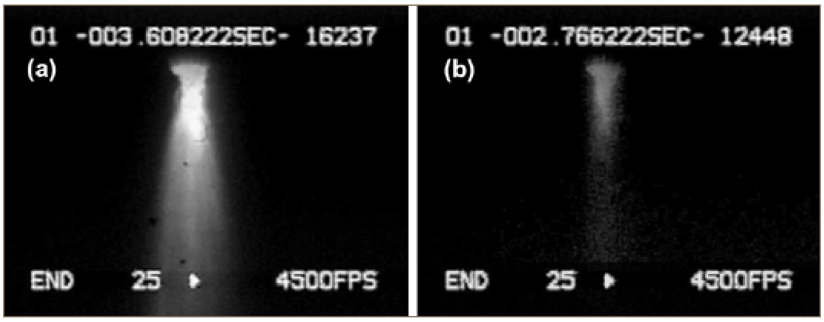

Fig. 5 Two frames from the high speed filming of the gas atomisation of NiAl melt at 1540°C. From presentation by A Mullis et al. at PowderMet 2012 (Courtesy MPIF)

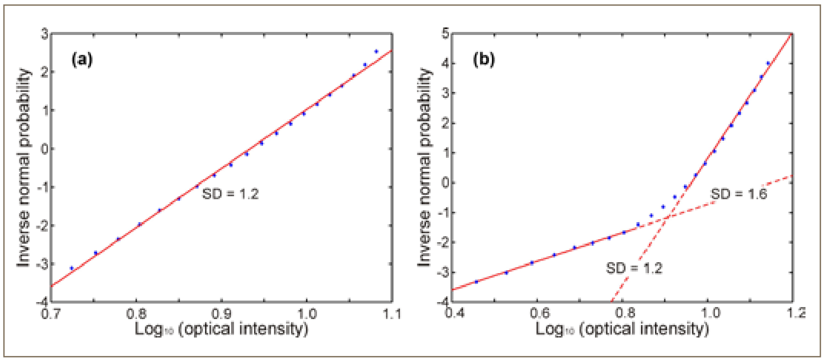

Fig. 6 Log-normal plots for an analogue (water) atomiser operating at a gas pressure of (a) 1.75MPa and (b) 3.5MPa. At low pressure, the intensity appears to follow a single log-normal distribution, but, at high pressure, two distributions appear to be present. From presentation by A Mullis et al. at PowderMet 2012 (Courtesy MPIF)

of these studies were reported in a separate paper from Andrew Mullis, Ian McCarthy and Robert Cochrane.

High speed photography (Fig. 5), coupled with sophisticated image analysis, has been used to study the low frequency pulsation in the volume of melt being instantaneously delivered to the melt nozzle.

The reported studies have shown that these fluctuations can be described statistically using the log-normal distribution. At low gas pressures, these fluctuations seem to follow a single distribution (Fig. 6a), whilst, at higher gas pressures, two distributions with quite different standard deviations appear to be present, with a high standard deviation being characteristic of low flow rates and a significantly lower standard deviation being observed at high flow rates (Fig. 6b).

For the particular atomisation geometry considered, this transition between a single and a dual distribution appears to occur around 3.0 MPa. At high gas pressure, the atomiser appears to spend 20-25% of its time in the high standard deviation state and to switch between the two states on time-scales of 0.05 – 0.01 seconds.

The authors have speculated that the low standard deviation state can be associated with an open wake condition and, conversely, that the high standard deviation state can be associated with a closed wake condition.

They have proposed that the developed methodology represents a simple, non-invasive technique for characterising the performance of gas atomisers that would be appropriate to off-line and potentially on-line monitoring. Off-line, the technique could be used to evaluate the effects of changes in atomiser configuration while, with appropriate automation, on-line monitoring would provide an additional tool for ensuring correct process operation and ultimately quality assurance of the product.

CFD study on fine powder production and optimisation in close-coupled gas atomisation

A second, broader-ranging CFD study, aimed at optimising close coupled atomiser design to improve yields of fine powders, was described in a paper from Guanghui Yu, Gregory Del Corso, James Scanlon and Ashish Patel (Carpenter Technology Corporation, USA).

Real scale models have been established on a prototype 300-lb experimental facility at Carpenter Technology Corporation's R&D Department.

To simplify the simulation work, isolated devices have been modelled separately. The devices, covered in

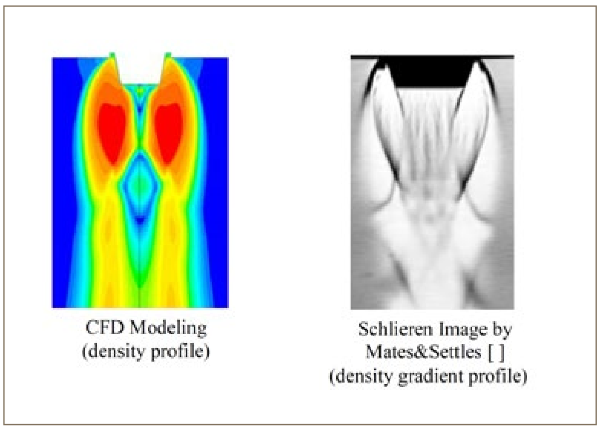

Fig. 7 Similarity between CFD modelling results and Schlieren image results of near field gas density variations. From presentation by G Yu et al. at PowderMet 2012 (Courtesy MPIF)

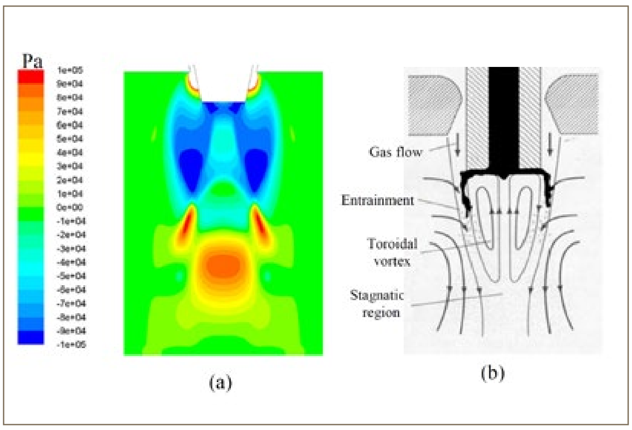

Fig. 8 (a) Near field pressure contour plot, (b) the prefilming operation of a close coupled nozzle. From presentation by G Yu et al. at PowderMet 2012 (Courtesy MPIF)

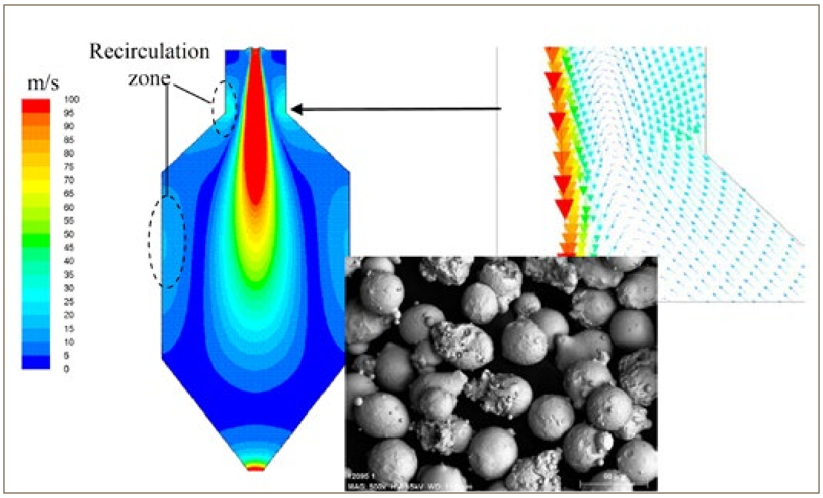

Fig. 9 Modelling results for velocity field in the collection chamber and SEM image showing super-fine satellites on fine particles. From presentation by G Yu et al. at PowderMet 2012 (Courtesy MPIF)

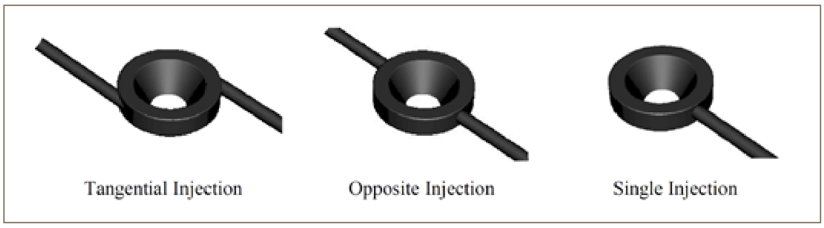

Fig. 10 Three types of gas manifold design modelled. From presentation by G Yu et al. at PowderMet 2012 (Courtesy MPIF)

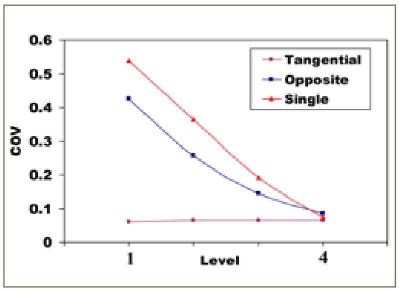

Fig. 11 Coefficient of variance (COV) of velocity for the three manifold designs. From presentation by G Yu et al. at PowderMet 2012 (Courtesy MPIF)

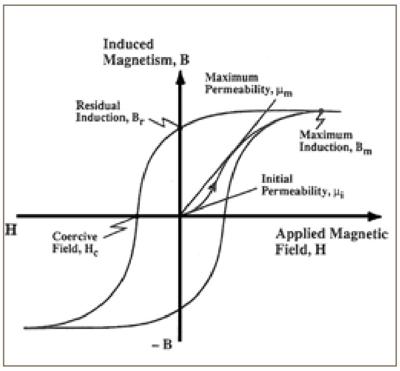

Fig. 13 Soft magnetic hysteresis curve. From presentation by M Bulger et al. at PowderMet 2012 (Courtesy MPIF)

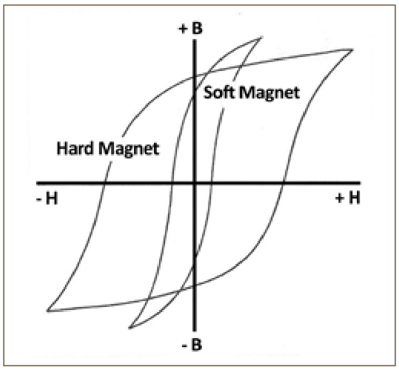

Fig. 12 Hysteresis curves for hard and soft magnetic materials. From presentation by M Bulger et al. at PowderMet 2012 (Courtesy MPIF)

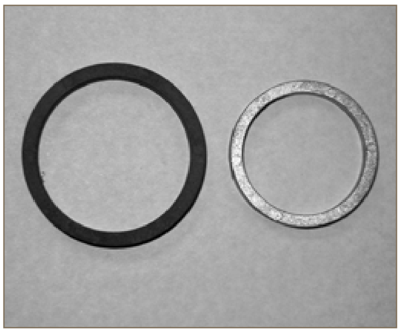

Fig. 14 Moulded (left) and as-sintered (right) toroids. From presentation by M Bulger et al. at PowderMet 2012 (Courtesy MPIF)

the reported work, include gas nozzle, collection chamber, and gas manifold.

Modelling results show that the near field density distribution from CFD simulation is consistent with high-speed imaging results (Fig. 7). Locations and profiles of sharp density gradients have convincing similarities. Pressure contours show that a gas-dynamics pressure punch is located immediately below the melt nozzle (Fig. 8). This punches a coherent melt stream into a low pressure and high speed toroidal zone to form film, and this is a critical criterion in characterising a close coupled nozzle.

The collection chamber model captures recirculation zones in the bulk area (Fig. 9). Uncontrolled gas recirculation can diminish powder yields, especially for fine powder. The recirculation gas speed is much higher than practical fluidisation velocities. Therefore, fine particles can be trapped in the back-flow and have sufficient time to meet other fine and ultra-fine particles and form satellites.

Gas manifold models have revealed that a tangential design (Fig. 10) has the best uniformity and highest speed and swirl in the plenum (Fig. 11). However, gas flow also tends to be uniform at the nozzle tip level for non-tangential designs.

The authors have concluded that this modelling work has created a good foundation for the optimisation of the atomising system and for the increase in fine powder yields. To put the effort in a broader context, more work could be undertaken in the areas of the use of atomisation gas with elevated temperature, secondary gas injection and de-recirculation gas injection, and powder trajectories in the near and far field.

MIM material developments

Processing of MIM Soft Magnetic Materials

Soft magnetic materials have emerged as a significant MIM material type and a paper from Matthew Bulger and Haorang Zhang (NetShape Technologies – MIM, USA) reported on a study aimed at optimising process conditions for three soft magnetic material alloys.

Soft magnetic alloys are materials that are relatively easily magnetised and demagnetised in the presence of an external magnetic field. They

Alloy	Theoretical density (g/cc)	Carbon Added?	Density (No HIP) (g/cc)	% theoretical	Density (w/ HIP) (g/cc)	% theoretical
Fe-50% Co	8.30	Yes	7.82	94.2%	8.12	97.8%
		No	7.84	94.4%	8.12	97.8%
Fe-3% Si	7.65	Yes	7.58	99.1%	7.56	98.8%
		No	7.56	98.8%	7.56	98.8%
Moly Permalloy (coarse powder	8.65	Yes	7.15	81.7%	7.44	85.0%
		No	7.23	82.6%	7.53	86.1%
Moly Permalloy (fine powder)	8.65	No	7.88	91.1%		

Table 1 Density results. From presentation by M Bulger et al. at PowderMet 2012 (Courtesy MPIF)

differ from hard (permanent) magnetic materials, which, once magnetised, require a substantial applied magnetic field to be demagnetised. The relative behaviours of the two material types are contrasted in the magnetic hysteresis curves in Fig. 12.

The typical hysteresis curve for soft magnetic materials (Fig. 13) identifies two parameters of key importance; the maximum permeability, μ_{max}, which defines how quickly the material is magnetised in the presence of an applied field, and B_{max}, which is a measure of how much magnetic flux per unit volume the material can deliver.

This study assessed the MIM processing of three soft magnetic alloys:-

- Fe-3%Si, a versatile alloy with good magnetic properties combined with relatively high resistivity.
- Fe-50%Co, an alloy with high B_{max}
- Molybdenum Permalloy (80%Ni, 4%Mo, balance Fe), an alloy with high permeability (μ_{max}). Two variants of this alloy were studied, one based on a coarse carbonyl nickel powder (d90 = 32.3 μm) and the second based on a finer carbonyl nickel powder (d90 = 13.9 μm).

Each material was assessed in the as-sintered condition, with and without a carbon black addition (aimed at reducing interstitial oxygen content after sintering), in the sintered + Hot Isostatic Pressed (HIP) condition and in the sintered + HIP + annealed condition. The test specimen used was a toroid ring (Fig. 14).

The achieved density levels after these treatments are summarised in Table 1. The Fe-50% Co and coarse

Condition	Carbon Added?	B_{max} (gauss)	μ_{max}
As-sintered	Yes	14.17	3270
As-sintered	No	14.53	3527
With HIP	Yes	13.36	2683
With HIP	No	13.98	2816
With HIP & anneal	Yes	13.98	3066
With HIP & anneal	No	14.01	2980

Table 2 Fe-3%Si magnetic properties. From presentation by M Bulger et al. at PowderMet 2012 (Courtesy MPIF)

Condition	Carbon Added?	B_{max} (gauss)	μ_{max}
As-sintered	Yes	19.33	3997
As-sintered	No	19.63	4940
With HIP	Yes	20.63	4400
With HIP	No	21.19	5553
With HIP & anneal	Yes	20.91	4860
With HIP & anneal	No	20.56	3603

Table 3 Fe-50%Co magnetic properties. From presentation by M Bulger et al. at PowderMet 2012 (Courtesy MPIF)

Ni Powder Used	Condition	Carbon Added?	B_{max} (gauss)	μ_{max}
Coarse	As-sintered	Yes	4267	25266
Coarse	As-sintered	No	4616	30450
Coarse	With HIP	Yes	4793	28590
Coarse	With HIP	No	4926	26470
Coarse	With HIP & anneal	Yes	4700	34010
Coarse	With HIP & anneal	No	4860	27160
Fine	As-sintered	No	6061	31540

Table 4 Moly Permalloy magnetic properties. From presentation by M Bulger et al. at PowderMet 2012 (Courtesy MPIF)

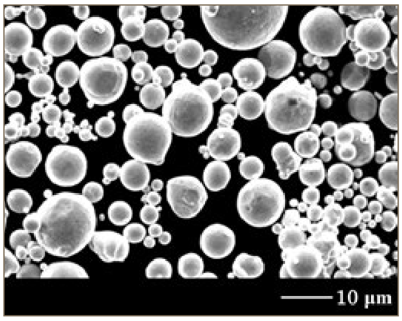

Fig. 15 SEM image of Rene95 powder. From presentation by H Miura et al. at PowderMet 2012 (Courtesy MPIF)

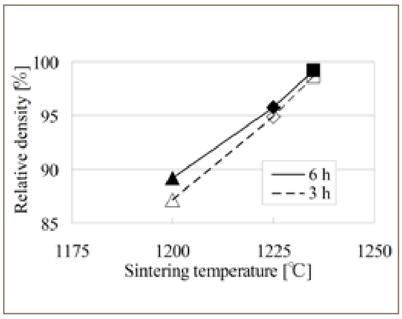

Fig. 16 Relationship between relative density and sintering conditions. From presentation by H Miura et al. at PowderMet 2012 (Courtesy MPIF)

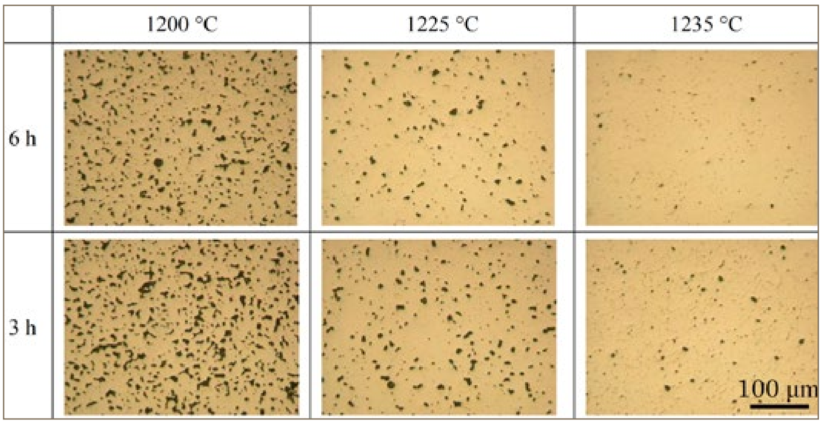

Fig. 17 Optical micrographs for each sintering condition. From presentation by H Miura et al. at PowderMet 2012 (Courtesy MPIF)

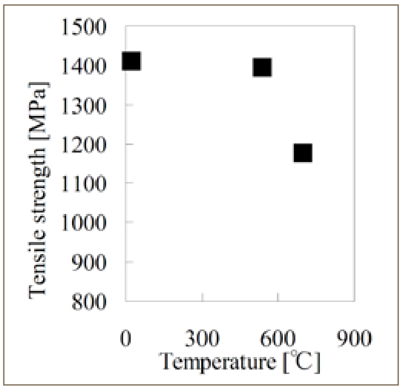

Fig. 18 Relationship between testing temperature and tensile strength for heat treated MIM Rene95. From presentation by H Miura et al. at PowderMet 2012 (Courtesy MPIF)

nickel powder Moly Permalloy densities were enhanced by HIP, while that of Fe-3% Si was not affected; for this alloy, as-sintered density was close enough to the theoretical density that HIP did not measurably improve it. The coarse nickel powder Moly Permalloy was still far from its theoretical density even after HIP, because the initial sintered density was so low (<85%) that interconnected porosity was present in the samples. There was a substantial increase in density (8.5% higher) when using the finer nickel powder for Moly Permalloy.

The magnetic results for the Fe-3%Si alloy are shown in Table 2. It was observed that the best properties were achieved with the simplest process (i.e. no carbon addition and as-sintered processing) with the use of HIP, annealing and carbon additions offering no improvement.

The results for the Fe-50%Co alloy, given in Table 3, showed that, for this alloy, HIP provided some improvement in magnetic properties (<10%), while annealing delivered no benefit. The addition of carbon black was detrimental to properties in both the as-sintered and HIP conditions.

For the Moly Permalloy material with a coarse Ni powder base (Table 4), the addition of carbon black again showed mixed results; there was no improvement in B_{max} for any group, but, for two of three groups, there was an improvement in maximum permeability. The use of HIP improved B_{max} but only within the 10% range. Annealing did not improve B_{max} but did increase permeability. The most significant influence of variations in processing parameters for the Moly Permalloy samples came with the use of the finer nickel powder base. B_{max} was improved by over 30%, and this result was consistent with the higher sintered densities seen with the finer powder base.

Overall the authors concluded that:-

- The addition of carbon black to reduce interstitial oxygen does not provide a general benefit to MIM soft magnetic materials and needs to be justified on a case-by-case basis.
- HIP did show improvements to magnetic properties when as-sintered MIM materials had appreciable residual closed porosity that could be eliminated by HIP.
- Annealing after HIP gave mixed results, with the majority showing a negative impact on magnetic properties.
- For the Moly Permalloy system, the most significant beneficial influence on magnetic properties came from the use of a finer nickel powder base.

Mechanical properties of injection moulded Superalloy (Rene95) compacts at high temperature

Nickel-based superalloys have excellent high temperature strength, corrosion and oxidation resistance and are widely used in both steam- and gas-turbines. Rene95, one such superalloy, has poor workability and is prone to segregation in casting. The manufacturing method used conventionally, therefore, comprises hot isostatic pressing followed by machining. This process, however, generally has a poor level of material utilisation. The potential use of MIM, as a net-shape approach to producing certain components from this material, has therefore become of increasing interest.

A paper from Hideshi Miura, Toshiko Osada, Shunsuke Morinaka, Hyungoo Kang and Fujio Tsumori (Kyushu University, Japan) reported the results of a study of the MIM processing of Rene95.

The MIM feedstock incorporated a Rene95 fully pre-alloyed gas atomised powder, of the composition shown in Table 5. An SEM image of this powder is shown in Fig. 15.

After MIM processing, samples were assessed in both the as-sintered condition and after a subsequent solution and ageing heat treatment.

Fig. 16 shows the resultant relationship between relative density and sintering temperature. At 1235°C sintering temperature, a relative density over 99% was achieved. It is known that Rene95 exhibits Super-solidus Liquid Phase Sintering characteristics and the authors postulated that this mechanism was responsible for the high sintered density obtained. Fig. 17 shows optical microstructures for each sintering condition. Porosity was reduced as sintering temperature and time were increased. It was also found that spheroidisation of the pores proceeded at longer sintering times.

Tensile properties were assessed both at room temperature and at elevated temperature. Fig. 18 reports the relationship between testing temperature and tensile strength for heat-treated samples. The measured tensile strength at 700°C was only reduced by around 16% from that at room temperature.

The tensile strength at 650°C of conventionally HIP processed Rene95 in the heat treated condition has been reported as 1500 MPa. So, although the measured tensile strength for the MIM processed material was somewhat lower than this level, the authors considered that further optimisation of MIM process conditions could close this gap.

Processing and properties of MIM AISI 4605 via master alloy routes

The leading supplier of gas atomised powders for MIM applications, Sandvik Osprey Ltd., has been developing the master alloy route for ferrous MIM grades for some time and a paper, presented by Toby Tingskog (Sandvik Osprey Ltd., USA) on behalf of his co-authors, Andrew Coleman, Keith Murray and Martin Kearns (Sandvik Osprey Ltd., UK) and Bob Sanford and Erainy Gonzales (TCK S.A., Dominican Republic) reported on a study of the approach for the processing of MIM AISI 4605.

AISI 4605 is a hardenable nickel low alloy steel (1.5-2.5% Ni, 0.5%max Mo, 0.5%max Si, 0.4-0.6%C) that can be used in either the as-sintered or heat treated condition and is becoming a more prominent MIM material for firearm component, general engineering and automotive applications. Published property data for as-sintered and heat treated MIM AISI 4605 are shown in Table 6.

Previous publications from these authors had demonstrated the benefits of using master alloy additions over the use of fully pre-alloyed materials for the MIM processing of the low alloy steel grades AISI 4140 and 4340 and had implied that the master alloy approach would also have benefits over conventional carbonyl iron powder + elemental blends. This latest study, however, was the first to report on a direct comparison between results obtained with the master alloy approach and the CIP + elemental blend approach. The powder "ingredients" used in the study were:

- A gas atomised 4605 master alloy at 5 times composition (i.e. 9-11% Ni, 1.5-2.0% Mo, 2.0%max Si).
- A gas atomised Fe38Mo ferro-alloy
- A gas atomised nickel powder
- Carbonyl iron powders at two different carbon contents – 0.016% and 0.83%.

In this paper, MA+CIP referred to parts produced using master alloy and carbonyl iron powder, whereas CIPB referred to parts produced from mixes of carbonyl iron powder, Fe38Mo and nickel.

For both MA+CIP and CIPB, feedstock was produced with carbon

Ni	Cr	Co	Al	Ti	Mo	W	Nb	Zr	Fe	Mn	B	Si	C
bal.	12.7	7.9	3.4	2.5	3.3	3.6	3.6	0.07	0.1	0.001	0.01	0.07	0.07

Table 5 Chemical composition of the Rene95 powder, wt%. From presentation by H Miura et al. at PowderMet 2012 (Courtesy MPIF)

4605	MPIF		BASF		German & Bose	
	AS	HT	AS	HT	AS	HT
% density					96	96
Density (g/cc)	7.5	7.5	7.55			
0.2% YS MPa *ksi*	207 *30*	1482 *215*	≥400 *58*	1500 *218*	205 *30*	1480 *215*
UTS MPa *ksi*	441 *64*	1655 *240*	≥600 *87*	1900 *276*	440 *64*	1655 *240*
%El	15	2	≥5	≥2	15	2
Hardness HRC	62 HRB	48 HRC	≥150 Hv10	≥55 HRC	62 HRB	48 HRC

Table 6 Published properties for MIM AISI 4605. From presentation by T Tingskog et al. at PowderMet 2012 (Courtesy MPIF)

	MA+CIP 90%-15µm	MA+CIP 90%-15µm	CIPB	MA+CIP (HC) 90%-15 µm	Carbonyl route CIPB (HC)
5x MA	Y	Y	N	Y	N
CIP	20%BC 60%HC	20%BC 60%HC	75%HC 21.5%BC	80%HC	Y
Ni	-	-	Y	-	Y
FeMo	-	-	Y	-	Y
%C	0.59	0.59	0.56	0.75	0.77
% Loading	61.80	57.87		56.93	56.93
% Shrinkage	17.4	20.0	20.66	20.66	20.66
Melt Flow Index	86.21	189.9		156.5	203.3

Table 7 CIP content and feedstock properties. From presentation by T Tingskog et al. at PowderMet 2012 (Courtesy MPIF)

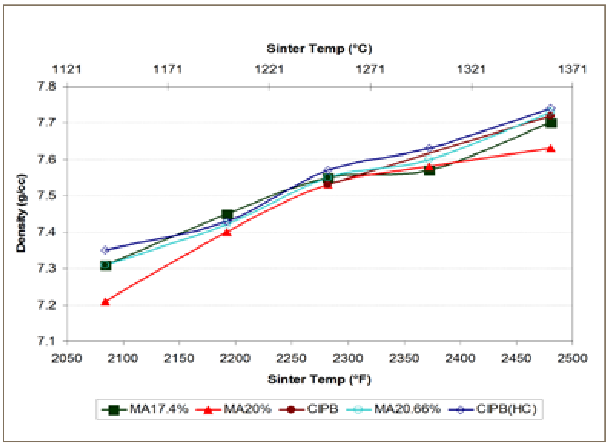

Fig. 19 Density of 4605 tensile test bars (solid) and 4605(HC) tensile test bars (hollow). From presentation by T Tingskog et al. at PowderMet 2012 (Courtesy MPIF)

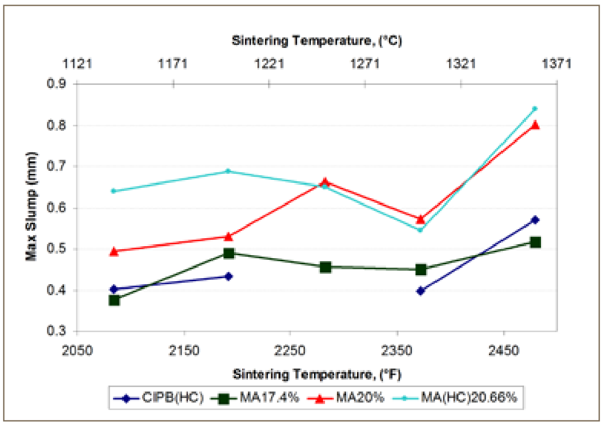

Fig. 20 Distortion as a function of sintering temperature. From presentation by T Tingskog et al. at PowderMet 2012 (Courtesy MPIF)

content meeting the 0.4-0.6% specification for AISI 4605, while a second batch was produced with a higher carbon content of ~0.75% (to assess the effect of carbon content on mechanical properties).

The fraction of each of the powders used in each feedstock along with the powder loading, shrinkage and Melt Flow Index (MFI) values are shown in Table 7. Feedstocks were prepared at different powder loadings using TCK's proprietary binder. 4605 MA+CIP feedstocks were prepared with 17.4% shrinkage and 20% shrinkage. For the higher carbon variant, the powder loading was reduced to 56.93% giving a shrinkage of 20.66%. The CIPB powder was prepared to the same specification. The latter shrinkage value was chosen to correspond closely to that of other commercial MIM feedstock.

The observed densification levels of the various feedstock types over a sintering temperature range from 1140°C to 1360°C are shown in Fig. 19. Of the MA+CIP feedstocks, the highest densities were observed for the MA+CIP17.4% blend. Comparable densities were produced with the MA+CIP17.4% and CIPB feedstocks.

Distortion levels during sintering were assessed on Charpy bars (Fig. 20). In most instances, the lowest distortion was observed at 1140°C. However, at this temperature full densification had not occurred. At higher sintering temperatures, the lowest distortion was typically observed

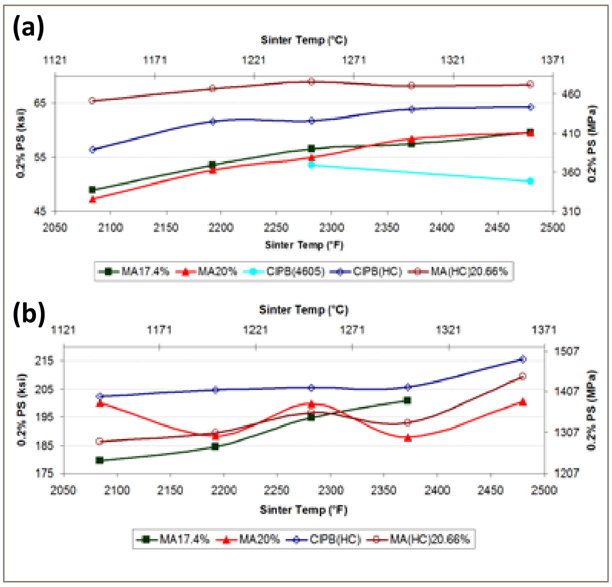

Fig. 21 0.2% Proof Stress values for (a) as-sintered and (b) heat treated MIM tensile test bars. Solid symbols denote 4605 feedstocks and hollow symbols denote HC grades. From presentation by T Tingskog et al. at PowderMet 2012 (Courtesy MPIF)

at 1300°C. The MA+CIP variants prepared with higher shrinkage factors of 20% and 20.66% (powder loadings of 57.87% and 56.93% respectively) exhibited higher distortion than the MA+CIP17.4% feedstock with a powder loading of 61.8%. Distortion results for the CIPB(HC) and MA+CIP17.4% feedstocks were comparable across the range of sintering temperatures.

Tensile test results were reported for both the as-sintered and heat treated conditions (the heat treatment cycle involved solution treatment for 60 minutes at 830°C, oil quenching, tempering for 1 hour at 200°C followed by air cooling). Proof stress results are shown in Fig. 21(a) and 21(b) respectively for the two conditions.

It is clear from the as-sintered results in Fig. 21(a) that the MA+CIP feedstocks achieved higher proof stresses than the CIPB feedstock. Compared with values reported elsewhere (shown in Table 6), the properties obtained for both the MA+CIP and CIPB feedstocks used in this study exceeded the minimum MPIF standard. However, only the MA+CIP feedstocks sintered at temperatures ≥1300°C achieved the minimum value of 400MPa reported by BASF.

For the higher carbon feedstocks, MA+CIP(HC) exhibited values 30-60MPa higher than CIPB(HC) across the range of sintering temperatures used in this study. The MA+CIP(HC) feedstock also exhibited values from 60 to 120 MPa higher than the MA+CIP17.4% feedstock.

The properties for the higher carbon grades in the heat treated condition, shown in Fig. 21(b), however, showed a reversal in the trend from the as-sintered results with the CIPB(HC) feedstock exhibiting a higher proof stress than the MA+CIP(HC) feedstock across the full sintering temperature range. At the time of submission of the paper, a similar comparison for the standard carbon feedstocks was not possible, as the results for the heat treated CIPB bars were not available.

However, the mechanical properties for the heat treated CIPB(HC) feedstock still did not exceed those reported elsewhere for MIM AISI 4605 and, therefore, the authors concluded that further work is needed to optimise the heat treatment cycle.

The effects of post-processing by Hot Isostatic Pressing (HIP)

A paper from Joseph Newkirk and Matthew Chott (Missouri Institute of Science and Technology, USA) and Phil McCalla and Bruce Dionne (Megamet Solid Metals, USA) reported on a study of the potential benefits of post-processing by HIP on the properties of another MIM low alloy steel grade that is growing in popularity, AISI 8740.

Condition	Yield Strength Ave (MPa)	Tensile S (MPa)	Elongation Ave (%)	Samples	Hardness (VHN-converted)
As-sintered	452±58.7	753±64.7	9±2.2	14	156
Heat Treated	1559±36	1723±65	2.3±0.6	23	487
HIP 2050	511±72.9	841±66.1	11±2.3	6	
HIP 2125	474±26.6	830±27.2	10±2.7	8	216
HIP 2050+HT	1675±36.8	1936±42.7	6±0.6	16	505
HIP 2125+HT	1674±50.4	1936±53.6	6±0.7	19	547

Table 8 Tensile and hardness test results for MIM 8740. From presentation by J Newkirk et al. at PowderMet 2012 (Courtesy MPIF)

PowderMet 2012 Nashville

MPIF President Matthew Bulger during his opening address

Drew Winter, WardsAuto World Magazine

The PowderMet Welcome Reception

Guests at the PowderMet 2012 Welcome Reception

PowderMet delegates enjoyed a night of country music at the Grand Ole Opry

The PowderMet 2012 exhibition

Condition	Charpy Ave (J)	Charpy s (J)	Samples
As-sintered	49	6.7	59
Heat Treated	14	3.4	29
HIP 2050	103	13.3	10
HIP 2150	103	9.3	10
HIP 2050 + HT	191	34.7	26
HIP 2125 + HT	189	25.3	24

Table 9 Unnotched Charpy test results for MIM 8740. s denotes Standard Deviation. From presentation by J Newkirk et al. at PowderMet 2012 (Courtesy MPIF)

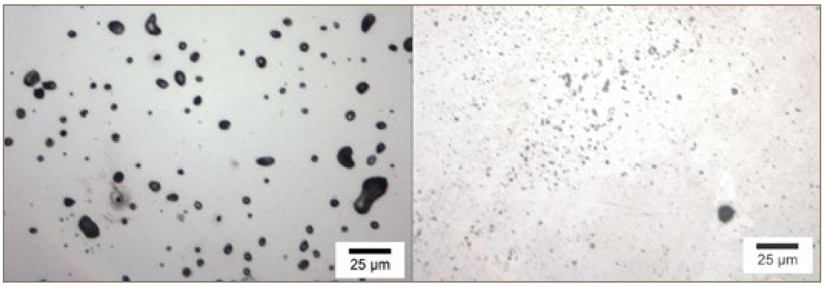

Fig. 22 Unetched optical micrographs of as-sintered MIM 8740 (left) and HIP+ quenched and tempered material (right). From presentation by J Newkirk et al. at PowderMet 2012 (Courtesy MPIF)

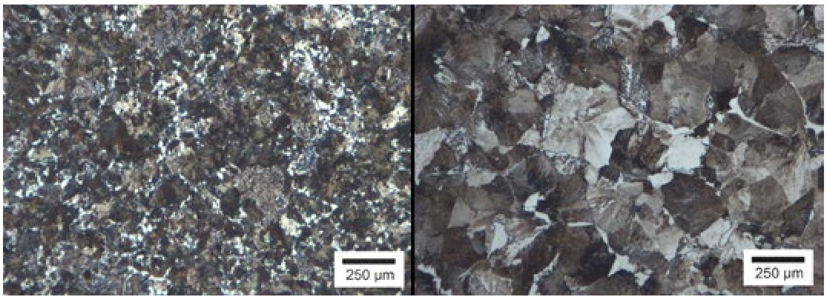

Fig. 23 Optical micrographs of as-sintered MIM 8740 (left) and HIP treated material (right). From presentation by J Newkirk et al. at PowderMet 2012 (Courtesy MPIF)

MIM AISI 8740 (~0.55%Ni, 0.5%Cr, 0.25% Mo) is a low alloy case hardening steel that can be used in the as-sintered condition or can be heat treated using either a through hardening or a case hardening cycle. The heat treatments applied in this study involved a through hardening cycle (austenitise at 885°C, oil quench, temper at 260°C for 1 hour).

Charpy impact and tensile test bars were processed by MIM and were then tested in six different conditions:-

- As-sintered
- Quenched and tempered
- HIP at 1121°C
- HIP at 1163°C
- HIP at 1121°C + Quench and Temper
- HIP at 1163°C + Quench and Temper

The results of the tensile tests are listed in Table 8. The as-sintered samples showed quite a good yield strength of 452 MPa, UTS of 753 MPa and 9% ductility. Quenching and tempering of the sintered samples increased the strength dramatically, as expected, but reduced the ductility to only 2%.

HIP increased strengths over the as-sintered values somewhat but did not come close to the quench and tempered values. There was little difference between the two HIP treatments. Along with the modest strength increase there was a significant increase in the ductility. This would be expected if the density was increased by HIP.

The tensile tests on the material that received both the HIP and the quench and temper treatment showed a dramatic increase in strength values coupled with excellent ductility. Again, there was little or no observable influence of the change in HIP temperature.

The unnotched Charpy test results are listed in Table 9. The as-sintered toughness values were good for MIM materials. Quenching and tempering to high strengths produced a significant drop in the toughness. As expected, the HIP treatments both produced a major increase in the Charpy values, but, again, with no distinction between the two treatment temperatures. The samples that were quenched and tempered after HIP showed an even higher toughness, averaging 190J.

As might have been anticipated, a major contributor to the property enhancements arising from the application of the HIP treatment was the increased densification created by the process. The samples prior to HIP had a density level around 94% of theoretical density, whereas, after HIP, this level rose to 99%. This comparison is illustrated by the two unetched micrographs in Fig. 22.

A somewhat more surprising observation in this study was that significant grain growth seemed to have occurred as a result of the HIP treatment, as evidenced by the micrographs of the as-sintered and HIPped microstructures shown in Fig. 23. This is particularly surprising as the sintering temperature employed in the MIM processing was, at 1325°C, significantly higher than either of the peak temperatures in HIP.

If the normal Hall-Petch relationship between strength and grain size applies, it would be predicted that the observed grain coarsening may have sacrificed around 50% of the potential strengthening effect from the HIP treatment.

The authors have concluded that this observation leaves open the possibility of further optimisation of processing to increase properties and reduce costs.

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Rapid Prototyping of high-performance ceramics opens new opportunities for the CIM industry

Whilst Rapid Prototyping has for a number of years enabled MIM producers to offer functional prototypes of components to their customers, the technology to enable the cost-effective low volume production of functional ceramic prototypes has until now remained out of reach. Dr. Johannes Homa, of Austria's Lithoz GmbH, outlines a novel Additive Manufacturing process that opens up new opportunities for CIM producers.

Additive Manufacturing technologies have gained considerable influence in both the plastic and metal manufacturing industries in recent years and they are now widely recognised as a viable manufacturing technology for these materials. The ceramic industry has, however, been reluctant to embrace Additive Manufacturing because the material properties and the accuracy of available ceramic Additive Manufacturing technologies were insufficient for the manufacturing of functional parts. To address this limitation, Lithoz GmbH has developed a unique process for the additive manufacturing of high-performance ceramics to a very high quality and with excellent dimensional accuracy.

The growth of technical ceramics

The technical ceramics sector has enjoyed strong growth over the last decade, with countless new applications in a diverse range of market sectors, with more applications being announced on a regular basis. Product designers and engineers now recognise many of the superior advantages of ceramic materials and are ever more willing to replace conventional materials with ceramic alternatives. Furthermore, aesthetic factors and the excellent biocompatibility of ceramics support the demand for ceramic materials, in addition to novel applications that require specific material properties.

Ceramic injection moulding (CIM) is a perfect mass production technology to fulfil the demand for technical ceramic parts. However, in many cases customers, particularly those who may not yet have used ceramics for their applications, may want to test a new material with a small number of prototype components. Moreover, the design of parts may have to be adjusted for the CIM process, which might result in a number of small design alterations. The problem of low volumes and design alternations is often a major obstacle in traditional ceramic forming techniques, especially in CIM.

Fig. 1 The CeraFab 7500 machine with Lihoz's Johannes Patzer (left) and Johannes Homa (right)

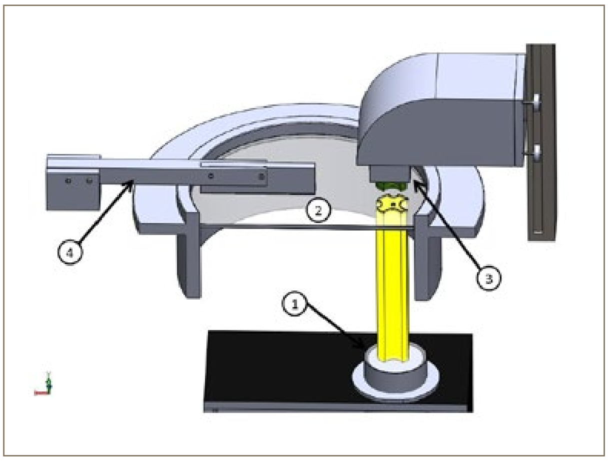

Fig. 2 Schematic showing the projection system (1), the slurry in a transparent vat (2), the building platform (3) and the coating knife (4)

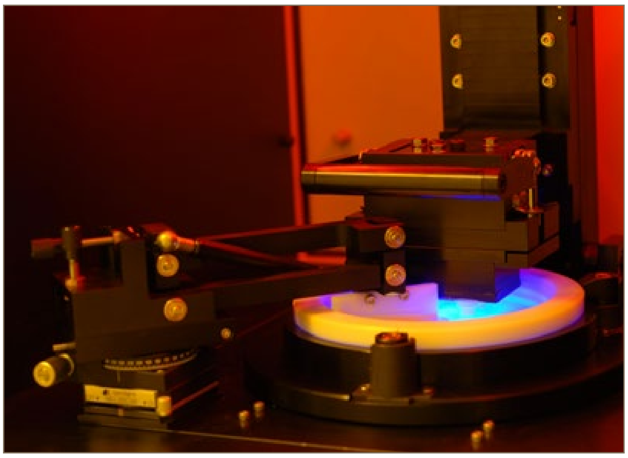

Fig. 3 The building chamber of the CeraFab 7500 during exposure

Additive Manufacturing's advantages

Additive Manufacturing technologies could solve this problem for the ceramic industry. Additive Manufacturing, also known as Rapid Prototyping or Layered Manufacturing, are forming methods that were developed from the late 1980's and are now well established for products manufactured from plastics and metals. A number of variations on the technology have evolved, but the core principle behind Additive Manufacturing is always the same.

Every process starts from a three-dimensional computer model which is then cut virtually into very thin layers. These layers are then "built" by the machine to form a three-dimensional object. The fabrication of the individual layers can be achieved in very different ways:

- melting powder with a laser (laser sintering or laser melting)
- curing photopolymers with light (stereolithography)
- bonding powder with a binder (3D-printing)
- extruding thermoplastic materials (fused deposition modelling).

Independent from these common layer fabrication methods, the result is always a real three-dimensional object which consists of hundreds of very thin layers.

The main advantage compared to CIM is the avoidance of tools. Since the part is directly "printed" from the computer aided design (CAD) data, no product specific tool is needed, which has a lot of advantages:

- no costs for a tool
- no cost for tool-alterations
- no limitations in geometry.

Additive Manufacturing technologies can therefore overcome two shortcomings of CIM, namely, the high costs incurred for single and small series production and the limitation in the design complexity. Specifically, the low costs for small quantities of parts could help the CIM industry to better support both new and established customers, as one of the main obstacles to the use of ceramic materials for the first time is the high initial cost of the tools, particularly if the part has a complex design.

'Additive Manufacturing technologies can provide the first test parts and design alterations can then be evaluated more cost effectively, as only the CAD-model has to be changed'

Existing CIM customers can also benefit from the flexibility that Additive Manufacturing technologies bring, for example if new products are evolving, or new ideas for novel solutions arise, it is often necessary to develop the geometry and make a number of design alternations. Additive Manufacturing technologies can provide the first test parts and design alterations can then be evaluated more cost effectively, as only the CAD-model has to be changed.

The main requirement when considering the use of Additive Manufacturing in the CIM industry is the very high quality of the finished ceramic parts. In the case of ceramic prototyping, the necessary quality simply could not have been achieved by existing technologies. If density, strength or tolerances do not meet the expected requirements, the prototype parts cannot be used for functional tests. This has made most of the existing ceramic Additive

Technical properties of the CeraFab 7500	
Lateral resolution	40 μm (635 dpi)
Building velocity	up to 100 slices per hour
Slice thickness	25 – 100 μm
Number of pixels (X, Y)	1920 x 1080
Building envelope (X, Y, Z)	76 mm x 43 mm x 150 mm

Table 1 Technical properties of the CeraFab 7500

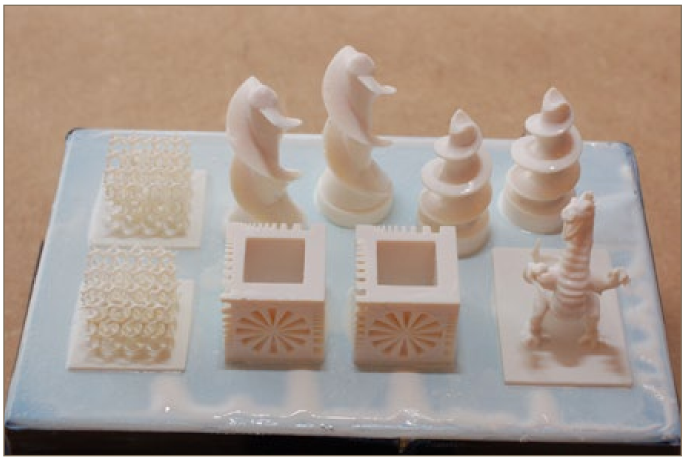

Fig. 4 Various "as formed" parts on the building platform prior to removal and sintering. The building process for these parts was 6 hours

Fig. 5 A sintered power turbine component for a heart-pump. The height of this part is 2 cm, however it will be downsized by a factor two for the final application

Manufacturing processes incompatible with the demands of the CIM industry, as the material properties did not meet the necessary criteria.

Together with the Vienna University of Technology, Austria, Lithoz has developed a novel technology which achieves very similar material properties to established high-volume precision ceramic forming processes such as CIM. Thus, the produced parts can be used for functional applications under real-world conditions.

A novel approach

Unlike most other ceramic Additive Manufacturing technologies, Lithoz has developed a slurry-based process. The slurry consists of a photopolymerisable monomer mixture filled with ceramic powders in typical concentrations between 75 and 85 wt%. The additional photoinitiator is chosen in accordance to the characteristics of the emitted wavelength of the LED-based projection system.

In order to ensure proper processability, the formulation has to be highly homogeneous, stable towards sedimentation of the fillers, and must exhibit a viscosity within the working window of the machine. The monomer mixture is polymerised by exposing it to light and thereby the liquid slurry is solidified. The solidified polymer acts as a binder and keeps the ceramic particles in shape. Blue LED's are used as a light source and the layer information is depicted via a projection system. The slurry is in a transparent vat and the light solidifies, slice by slice, the material from below. The building platform is moved upwards and the vat is rotated to re-cover the vat with new material using the coating knife. This procedure is repeated for every slice and the part is generated layer by layer. Fig. 2 shows the design of the machine and the relevant components.

After the build process is completed the parts are separated from the building platform and the residual slurry is removed by simple ultrasonification in a special cleaning fluid. After this, the process steps are similar to CIM. The part is thermally debound and then sintered. Since the part is "pulled out" of the material, very little material is used for the initial filling of the machine. This is especially useful when new materials are being developed. Compared to many other forming processes, where much material has to be used to get the process running, the minimum filling is as low as 10 ml.

The machine and materials

The currently available machine is the CeraFab 7500. The machine's specifications are shown in Table 1 and a detailed picture of the building chamber in can be seen in Fig. 3.

The commercially available material for this process is high purity alumina (purity higher than 99.99 %), however other ceramic materials are available upon request. The resulting alumina parts have a density of over 99.4 % of the theoretical density (> 3.96 g/cm^3) and a resulting biaxial-strength (piston on three balls) of over 500 MPa. The precision of the process is approximately 0.1 mm, with a minimum feature size of 0.15 mm. Typically the system can build 100 slices per hour. With a slice thickness of 50 µm, a building speed of 5 mm/h can be achieved.

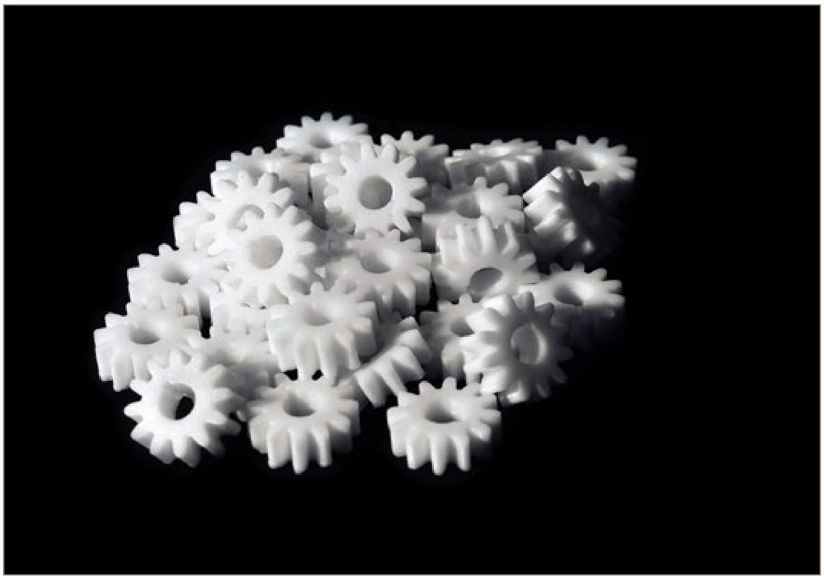

Fig. 6 High purity alumina gear wheels, as-sintered, with a diameter of 6 mm

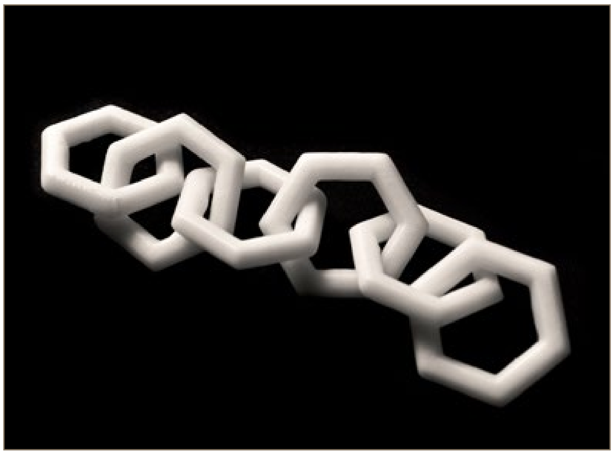

Fig. 7 High purity alumina chain

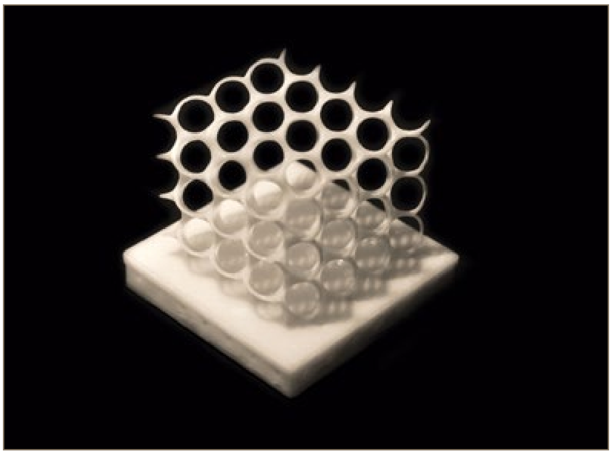

Fig. 8 Cell structures with a strut thickness of 200 µm

Applications

The material properties stated present the possibility to use parts produced by Additive Manufacturing as pre-series production samples of CIM components. Not only can the parts be used for functional testing, but design alterations can also be made without tool changes. If different designs are needed, these can be built in one building process without any additional cost. This gives more flexibility and speed in the fabrication of prototypes, and the "time to market" can be reduced dramatically.

Additive Manufacturing technologies can not only be used for the production of prototypes, but also for small series production. There are hardly any set-up costs and the tool-free parallel production enables the cost efficient fabrication of small to medium quantities.

The other significant advantage of Additive Manufacturing technologies is the production of highly complex parts which can not be produced using any other forming technique. There is no limitation for undercuts, hollow spaces or designed cell structures. This will lead in the future to a shift from "design for production" to "design for application". Currently, ceramic engineers are limited by the available forming techniques and have to consider these limitations in their designs.

A number of examples of the possibilities of this process are shown in Fig. 4. Here, the building platform is shown with various green parts after cleaning, all of which have been produced simultaneously. The cell-structure on the left hand side of the picture shows the possibilities for complex structures particularly well.

Applications for Additive Manufacturing technologies can be found in various industries, from automotive to jewellery and watches, as well as in mechanical engineering applications and medicine.

'Applications for Additive Manufacturing technologies can be found in various industries, from automotive to jewellery and watches, as well as in mechanical engineering applications and medicine'

One interesting ongoing project is the development of a heart-pump, conducted jointly by the Vienna University of Technology, the Medical University of Vienna and Lithoz. The sintered power turbine of the heart-pump is shown in Fig. 5. The part is not machined or finished and the accuracy and the fine detail resolution can also be seen in the picture. The height of the power turbine is currently 2 cm, but it will be downsized by a factor two for the real application. Other parts with high feature resolutions can be seen in Figs. 6-8.

Conclusion

Lithoz has developed an Additive Manufacturing technology for ceramic materials which produces parts with high densities (> 99.4 %), high strengths (> 500 MPa biaxial strength for alumina) and very high precision.

This is currently the only available Additive Manufacturing technology which enables the production of functional prototypes for the CIM industry. Not only can functional prototypes be fabricated, but also small series of components can be effectively produced by Additive Manufacturing.

Furthermore, highly complex parts that could not be fabricated with any other forming technique can now be produced. This new process opens up a number of new possibilities for the ceramic industry. The CIM industry in particular can benefit from a tool-free manufacturing solution. As such, the development of such a process should not be regarded as a challenge to CIM, but as a useful supplement to existing ceramic forming capabilities.

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Ceramic coated molybdenum setter plates for MIM offer increased capacity and energy efficiency

Ceramic coated molybdenum setters have been developed specifically for use as sintering bedplates in Metal Injection Moulded (MIM) parts production, including stainless steels, iron-nickel alloys, tungsten alloys and other powder materials. As Japan's A.L.M.T. Corp. explains, a variety of suitable ceramic powders can be used to coat the molybdenum setter to prevent the interaction between MIM parts and the setter, whilst also offering increased furnace capacity and energy savings.

Molybdenum is used as a structural material in vacuum and hydrogen atmosphere furnaces as the heater, reflector, supporting posts, and so forth, as well as in many industries as a refractory metal at high temperatures. A molybdenum setter is used for sintering and annealing in vacuum and hydrogen atmosphere furnaces. Molybdenum has superior properties to alumina, such as low specific heat, high thermal conductivity, and excellent bending strength at high temperatures, as shown in Table 1. The advantages of molybdenum are its high productivity. Thanks to its strength at high temperatures, the volume of the setter can be decreased, there is lower energy consumption, and the material offers excellent bending strength at high temperatures.

Improving deformation resistance

The creep resistance of the molybdenum plate is important when it is used as a setter. There are several types of molybdenum alloys that improve the resistance of a setter to deformation, compared with that of a pure molybdenum setter. In this study, we first evaluated the deflection of uncoated molybdenum plates. The deflection after 10 hours of heating at 1350°C when a load of 300 g was applied with a 100 mm span onto a plate of 20 mm width was measured, as shown in the left of Fig. 1. The right of Fig. 1 shows that pure molybdenum had the largest deflection and that a titanium-zirconium-molybdenum (TZM) plate was deflected by 6.0 mm for a plate of 1 mm thickness and by 1.9 mm for a plate of 1.5 mm thickness. In the case of TEM [3][4], a molybdenum alloy with a fibre structure that contains lanthanum oxide, which improves the strength at high temperatures, the deflections were only 2.0 mm and

	Molybdenum	Alumina
Specific heat	0.27 J/g K	0.80 J/g K
Thermal conductivity	138 W/m K	40 W/m K
Bending strength (at 1200°C)	400 MPa (full bend)	200 MPa

Table 1 Comparison between the characteristics of pure molybdenum and alumina [1][2]

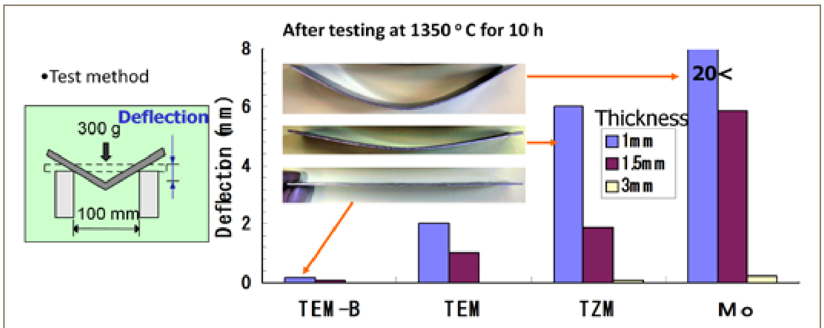

Fig. 1 Creep behavior of non-coated molybdenum plates. Sample dimensions: T x W20 x L120 mm. The length is in the rolling direction

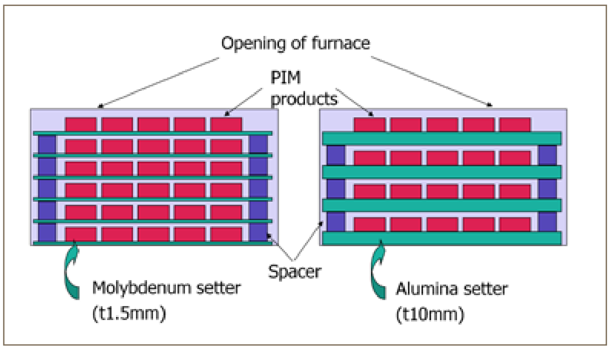

Fig. 2 Comparison between stacks used in molybdenum (TEM) and alumina setters

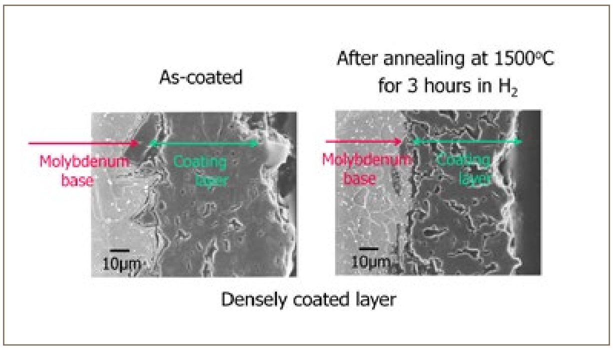

Fig. 4 SEM images of the cross section of a WA (Al_2O_3) coated layer

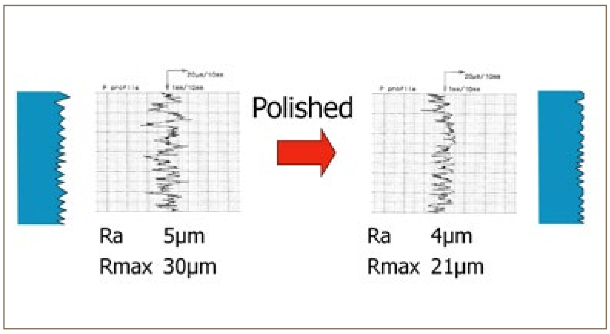

Fig. 3 Surface roughness of a WA-coated surface before and after polishing

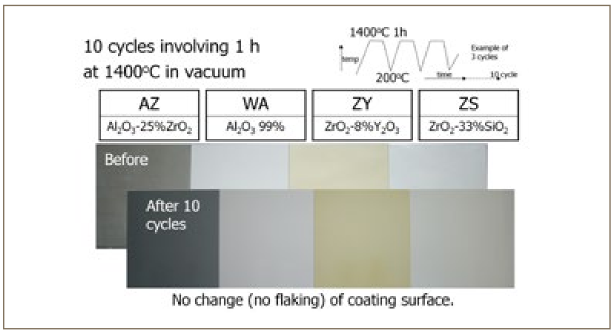

Fig. 5 Endurance test results of coated-molybdenum plates involving 1400°C heat cycles, TEM plate, dimensions T1 x W100 x L100 mm

1.0 mm for plate thicknesses of 1 mm and 1.5 mm, respectively. In addition, in the case of a fully recrystallised TEM plate, referred to as TEM-B "B" meaning recrystallised, the deflection was only 0.15 mm at the thickness of 1 mm. Consequently, TEM is recommended for use as a setter because it does not deform, as shown by its small deflection. Because the deflection is small, a coating on a TEM plate does not flake off easily.

Fig. 2 shows examples of the in-furnace stack used in molybdenum and alumina setters for sintering MIM and Powder Injection Moulded (PIM) parts. For a furnace with a 100 mm high opening, the alumina setter can only have a stack of four layers, although six layers can be stacked in a setter containing thin molybdenum plates with high strength at high temperatures and high resistance to thermal shock. Compared with the conventional alumina setter, the molybdenum setter has 50% higher capacity and 30% lower power consumption [5-7].

'Compared with the conventional alumina setter, the molybdenum setter has 50% higher capacity and 30% lower power consumption'

Coating materials

Four suitable coating materials for molybdenum are shown in Table 2. In particular, white alumina (WA, Al_2O_3 99%) is recommend as a coating. The coating can be selected on the basis of the sintering material to prevent it from adhering to the plate. In the case of molten metal sintering, a ZS (ZrO_2-33%SiO_2) coating may be the most suitable for preventing welding.

The coating layer can also be polished. The roughness of a surface with a WA coating is shown before and after polishing in Fig. 3. This result also shows that the coating layer does not flake off after sliding. Thus, it is also possible to form a smooth surface by polishing to remove the convex regions on the coating so that sticking does not easily occur. The convex regions on the coating result in the sintering body having concave regions. Also, a different rate of shrinkage occurs at the convex regions, which causes the sintered product to contain defects.

The coating layer was examined before and after annealing. The coating layer generally has a thickness of 50 to 100 µm. Cross sections of a coated layer before and after annealing are

Symbol	Composition of Coating	Coating Thickness	Temperature of use
AZ	Al_2O_3-25%ZrO_2	30-150 µm	up to 1700°C
WA *	Al_2O_3 99%		up to 1400°C
ZY	ZrO_2-8%Y_2O_3		
ZS**	ZrO_2-33%SiO_2		

** Recommended **Durability for use with molten metal*

Table 2 Compositions of various coatings on molybdenum plate.

shown in Fig. 4. Although shrinkage of the coating layer occurs during annealing, there is no evidence of the flaking of the coating layer according to the images obtained by microscopy. The results of an endurance test involving repeated heat cycles are shown in Fig. 5. Each heat cycle comprised an increase in temperature to 1400°C, which was maintained for 1 hour, followed by cooling to 200°C. No flaking was observed as a result of subjecting the plates to heat cycles.

It is important that the subliming sintering material does not adhere easily to the coating layer. The results when a ZS coating on a TEM plate and a noncoated TEM plate were used as sintering bedplates for a copper alloy sintering material are shown in Fig. 6. Noncoated molybdenum (TEM) cannot be used as a bedplate because the coagulating copper accumulates to form a three-dimensional structure. On the other hand, there is little adhesion between the copper and the ZS coated plate, and even if adhesion occurs, the plate can be removed immediately.

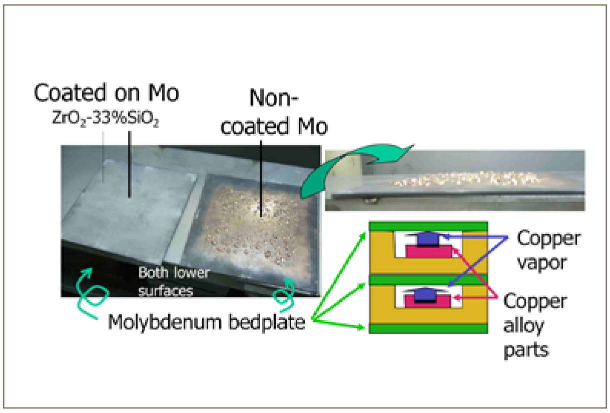

Fig. 6 ZS-coated TEM plate and noncoated plate used as sintering bedplates for copper PIM parts

Coating performance

The procedure used to evaluate peel strength is shown in Fig. 7, the results of which (data for exfoliation at the molybdenum/ceramic interface) are shown in Fig. 8. All annealing was performed at 1500°C for 1 hour. Each peel test was performed over an area of 83 mm^2 connecting the coating layer and the bolt, which were connected with an adhesive. Tension was applied by a crosshead with a speed of 0.3 mm/min. Flaking due to the adhesive was excluded from the data, and the amount of flaking that occurred at the interface was considered. The peel strength improved

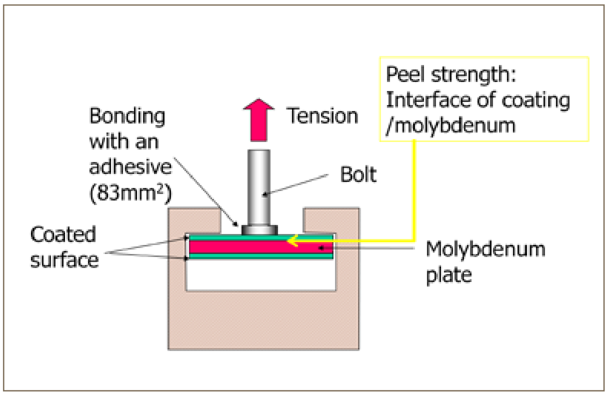

Fig. 7 Procedure used to evaluate peel strength

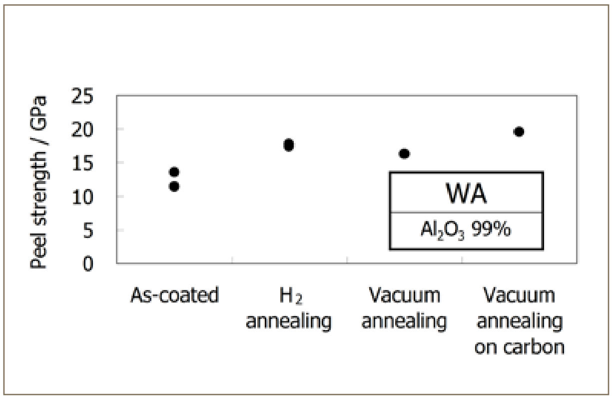

Fig. 8 Results of peel strength test. The data shown is for exfoliation at the molybdenum/ceramic interface

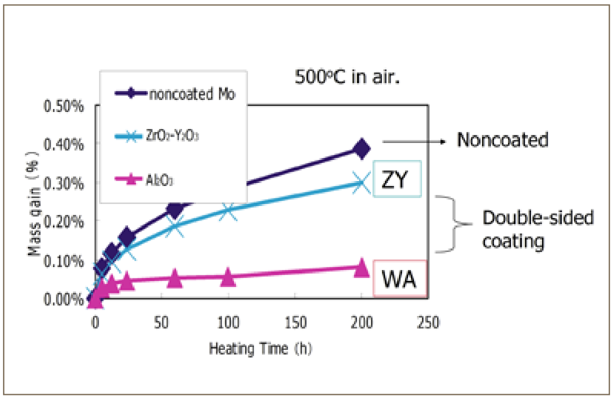

Fig. 9 Difference of oxidation mass gain between different coatings on molybdenum plates. The sample with the least mass gain has oxidation resistance. (Sample dimensions: T2 x W20 x L30 mm)

upon all types of annealing. The peel strength was 11 - 13 GPa for the WA coating, and annealing improved the peeling strength to up to about 20 GPa.

One of the processes in PIM, thermal debinding, in which the binder from the PIM products is removed, occasionally occurs by heating in air. Other thermal debinding processes take place in vacuum and hydrogen atmospheres. Vacuum and hydrogen atmospheres are also used for the atmosphere of the sintering process. However, because molybdenum is easily oxidised, hydrogen, an inert gas, or a vacuum atmosphere, is necessary when heating. Fig. 9 shows the oxidated mass gain in coated and non-coated samples heated in air at 500°C (T1 x W30 x L100 mm). Sublimation occurs following the oxidation of molybdenum when it is heated in air, and the mass of molybdenum decreases above sublimation temperature of MoO_3 of 700°C [8].

However, the weight increases upon heating at 500°C because it is less than the sublimation temperature of MoO_3. Although the shape of an annealed molybdenum plate in a setter is expected to be maintained, MoO_3 powder was formed from the molybdenum bulk at 500°C. Consequently some improvement in the resistance to oxidation was observed upon heating to 500°C, although the coated molybdenum alloy plate can not be used for the process of heating in air.

Conclusions

Ceramic coated molybdenum setters were developed for use as a sintering bedplate. We obtained the following results:

- Molybdenum has superior properties to alumina.
- Four suitable coating materials for molybdenum are: Al_2O_3-25%ZrO_2 , Al_2O_3 99%, ZrO_2-8%Y_2O_3, and ZrO_2-33%SiO_2
- Ceramic-coated molybdenum setters can be used in hydrogen and vacuum atmospheres.
- Oxidation of a coated molybdenum setter occurs in air.
- No flaking of coated layer occurs upon cyclic heating at 1400°C.
- A coated layer prevents the interaction between the sintering material and molybdenum (we recommend WA, Al_2O_3 99%).
- The molybdenum/ceramic interface has sufficient adhesion strength.

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[8] Japan Tungsten & Molybdenum Industries Association: Tungsten Molybdenum Technical Data 3rd edition, Japan Tungsten & Molybdenum Industries Association (2009) 102.

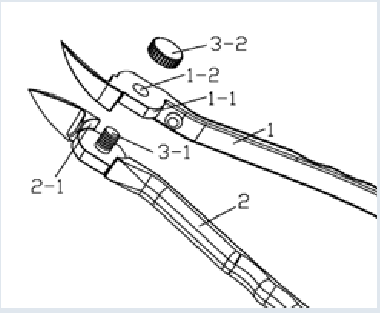
Global PIM Patents

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

CN101823246 (A)
PLIERS MANUFACTURED BY USING POWDER INJECTION MOULDING PROCESS
Publication date: 2010-09-08
Inventor(s): Yinliang Han, Hangzhou Fuxing Tools Co Ltd, China

The invention relates to pliers manufactured using a powder injection moulding process (PIM). The technical problem to be solved of the invention is to design a new plier assembling structure so as to solve the problems in conventional forging technology and bring convenience to assembly and maintenance of the pliers.

The pliers consist of an upper plier body (1), a lower plier body (2) and a connecting piece and are characterized in that: 1) the connecting piece is a bolt (3-1); one end of the bolt is positioned in the centre of an assembly cavity and integrated with the lower plier body (2), and the other end of the bolt is matched with a screw cap (3-2) by screwing; or one end of the bolt is integrated with the screw cap (3-2) and matched with a blind screw hole (2-2) at the bottom of an assembly cavity (2-1) of the lower plier body (2); and 2) the upper plier body (1) and the lower plier body (2) are manufactured by using the powder injection moulding process.



JP2010236042 (A)
METHOD OF JOINING METAL POWDER INJECTION MOLDINGS, AND METHOD OF PRODUCING METAL COMPOSITE SINTERED MATERIAL
Publication date: 2010-10-21
Inventor(s): Kimura Masahiro, Nippon Piston Ring Co Ltd

This patent identifies the problem to be solved as providing a method of bonding metal powder injection mouldings, by which a plurality of metal powder injection mouldings are bonded to produce a composite moulding. It also aims to provide a method of producing a metal composite sintered material having a high joining strength by sintering the composite moulding.

The metal composite sintered material is produced by applying a paste-like coating liquid, prepared by diluting metal powder of the same kind as the metal powder constituting at least one moulding, and a water-soluble glue-like substance with water to joint interfaces of a plurality of metal injection mouldings which are preferably subjected to debinding treatment. The metal injection mouldings adhere to one another to produce the composite moulding. The component is then sintered to form the final metal composite sintered material.

A starch glue-like substance is preferably used as the water-soluble glue-like substance. According to the invention, a defective joint due to deformation of the metal injection moulding can be avoided, while the deviation of joint face or the like occurring upon the handling operation can be prevented. High joining strength can be obtained and, moreover, a composite sintered material having high dimensional accuracy can be easily produced.

CN101691086 (A)
POWDER MICRO INJECTION MOULDING METHOD FOR CERAMIC SUBSTRATE OF PRINTING HEAD
Publication date: 2010-04-07
Inventor(s): Zhen Lu, et. al., Harbin Institute of Technology, China

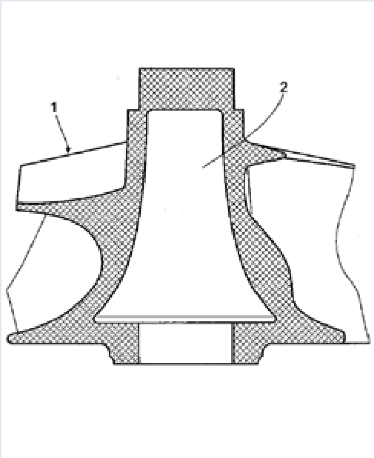
The invention relates to a powder micro injection moulding method for producing the ceramic substrate of a printing head. The method solves the problem of a complicated process, high cost and low processing efficiency in conventional processing.

The method comprises of selecting a suitable ceramic powder, preparing a binder, preparing a feedstock and adding the prepared feedstock into a micro injection moulder to mould the material. The charging barrel of the micro injection moulder is heated to a temperature of between 180 and 240°C, and the nozzle to a temperature of between 175 and 240°C. Injection pressure is maintained between 30 and 100MPa. The process involves thermal debinding, pre-sintering and sintering of the injection blank. The moulded blank is placed back into a high temperature sinter furnace for sintering in air atmosphere to produce the ceramic substrate, ensuring the sintering temperature is between 1,400 and 2,200°C. Heat is preserved for 1 to 3 hours. Post-processing, including grinding and polishing the ceramic substrate then takes place.

The ceramic substrate manufactured by adopting this method has excellent corrosion and wear resistance, and has improved service life.

WO2010115837 (A1)
METHOD FOR PRODUCING A TURBINE WHEEL FOR AN EXHAUST GAS TURBOCHARGER
Publication date: 2010-10-14
Inventor(s): Kern Andreas et. al., BASF SE, Germany

The invention relates to a method for producing a turbine wheel for an exhaust gas turbocharger by means of metal injection moulding, comprising the following steps: (a) providing a feedstock containing a metal powder and a binder material, (b) providing a tool, which comprises a negative mould of the turbine wheel to be produced, for metal injection moulding of the turbine wheel, (c) introducing a rotationally symmetrical core composed of the binder material into the negative mould of the tool provided in method step (b) and orienting the core so that the core is oriented symmetrically to the rotational axis of the turbine wheel to be produced, (d) producing a green body by means of metal injection moulding of the feedstock provided in method step (a) around the core, (e); performing a binder removal step to remove the binder from the green body to obtain a moulded body in the form of the turbine wheel, and (f) sintering the moulded body.



CN101623760 (A)
APPLICATION OF MICRO INJECTION MOULDING TECHNIQUE FOR PREPARING TUNGSTEN-BASE ALLOY PRODUCT AND MICRO INJECTION MOULDING METHOD OF TUNGSTEN-BASE ALLOY POWDER
Publication date: 2010-01-13
Inventor(s): Yeqi Hu, et. al., Xiamen University Of Technology, China

The invention discloses an application of micro injection moulding technique for preparing a tungsten-base alloy product, which can prepare a micro tungsten-base alloy part with a mass less than 0.5g and a relative density greater than 96%. The invention also discloses a micro injection moulding method of tungsten-base alloy powder, comprising steps of mixing, milling mixing, pelletizing, injection moulding, green-ware degreasing, sintering and the like.

The binder consists of wax, polyethylene, surface modifier and the like, wherein the modifier consists of stearic acid and organic naphthaline by a certain proportion, and the viscosity of the binder is dramatically reduced at a high shearing rate, thereby being favourable for filling.

The invention adopts two debinding steps of solvent debinding and heat debinding, wherein the first step of solvent debinding can remove wax of low melting point and partial surface modifier, and the second step of heat debinding mainly removes polymer of high melting point. The high-temperature insulation can remove the binder, sinter the green body, improve the intensity of the green body and ensure conformity of the green bodies.

CN101912888 (A)
MANUFACTURING METHOD OF DIE CORE OF WIRE-DRAWING DIE
Publication date: 2010-12-15
Inventor(S): Yang Liu et. al., Jiangyin Dongda New Material Res Institute, China

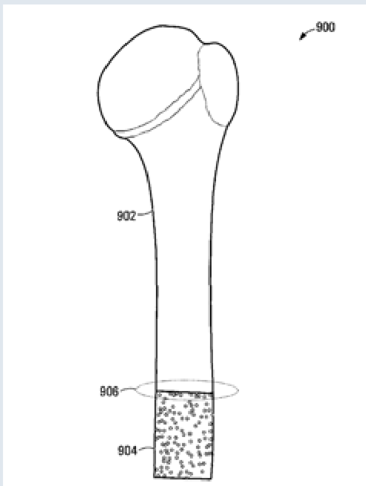
The invention relates to a manufacturing method of a die core of a wire-drawing die. The process comprises of mixing metal powder and binder to obtain a mixture that is then kneaded and pulverized. It is then added to a screw extruder for extruding and granulating to obtain material particles. The material particles are injection moulded to obtain a blank of the die core of the wire-drawing die.

The resulting blank is put into dichloromethane, gasoline or benzene, then placed into a vacuum debinding furnace for solvent debinding followed by a vacuum sintering furnace. The process then involves a low-pressure hot isostatic pressing treatment and annealing softening heat treatment in a sintering furnace, finally carrying out spalling, machining, grinding, quenching, tempering, burring and polishing treatment to obtain the die core of the wire-drawing die.

The density, hardness, flexural strength, compressive strength, wear resistance and corrosion resistance of the die core product prepared by the method of the invention are all improved.

WO2010124398 (A1)
A METHOD FOR CO-PROCESSING COMPONENTS IN A METAL INJECTION MOLDING PROCESS, AND COMPONENTS MADE VIA THE SAME
Publication date: 2010-11-04
Inventor(s): Julien Benoit et. al., Maetta Sciences Inc, Canada

This patent describes a method comprising moulding a first component from a first feedstock comprising a first material powder and a first binder, moulding a second component from a second feedstock comprising a second material powder and a second binder, placing the first component and the second component in physical communication with each other in order to form an assembled component, removing the first binder and the second binder from the assembled component and performing a sintering operation on the assembled component so as to bond the first component and the second component together.



An evaluation of coarser iron powders as a substitute for carbonyl iron in low alloyed MIM materials

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The effect of substituting carbonyl iron with a water atomised coarser iron powder (d50: 20-40 μm) was investigated regarding dimensional stability, mechanical properties and metallography, for an Fe-2Ni-0.5%C MIM feedstock. Three substitution levels (10, 20 and 30 weight%) as well as a control sample (0% coarse powder added) were mixed. Tensile and sharpy bars were injection moulded, debound in water and sintered in 76%N2/24%H2 at 1180°C. Main conclusions are that even if sintered density is affected (decreased) almost linearly with added coarse powder the effect on the micro structure counteracts this negative effect on mechanical properties resulting in unaffected ductility and slightly higher yield strength (with 30% added coarse powder) but decreased ultimate tensile strength and hardness. Dimensional stability in terms of slumping decreases with an increasing amount of coarse powder, but dimensional variations are improved (less variations with more coarse powder).

As the MIM industry matures, competition is driving suppliers to broaden their offerings. One of the trends in the MIM industry is to move to finer powders creating better surface finish, improved sinterability and more intricate geometries. The drawback is that finer powders often carry a price premium, which in turn means some applications are not made by MIM due to cost reasons. Conversely there are opportunities for lower cost options if a mouldable feedstock can be produced and mechanical properties can be met.

Even if there are opportunities outside of today's MIM market this paper explores the use of coarser iron powder as a substitution for carbonyl iron in a 4605 type of alloy (Fe-2Ni-0.5C).

Procedure

For this study three coarse powder addition levels were tested as well as a standard formulation to use as a control. The three levels were additions of 10%, 20% and 30% coarse powder (a water atomised iron powder with a d50 of 32 µm and d90 of 50 µm). The additions were straight substitutions for the standard carbonyl iron powder. The binder system is a production system utilising a water soluble minor component and acetal as the major binder. The solids loading was held constant for all feedstocks at 59% by volume.

Standard tensile bars and charpy bars were moulded from each of the four feedstocks. The moulded samples were then debound in water and dried. The parts were sintered in a high temperature pusher furnace under a 76% nitrogen, 24% hydrogen atmosphere at 1180°C set point. Slumping stability during sintering was tested by placing six charpy bars from each feedstock across a 2" span.

After sintering the slump samples were checked for the amount of bow and the data recorded. Length and width dimensions were taken from 15 samples per variable that were set on flat plates, to compare shrink variations in the different feedstocks. Sintered densities were checked via Archimedes' technique.

The tensile testing was done in accordance with EN10002-1 in a Zwick Z100 type testing machine. In addition to tensile testing, impact energy and apparent hardness were checked. The impact energy testing was performed on an Instron PW30 machine according to SS-EN-25754 test method. Hardness was measured per SS-EN ISO 6508-1:2006 and ISO4498-1:1990.

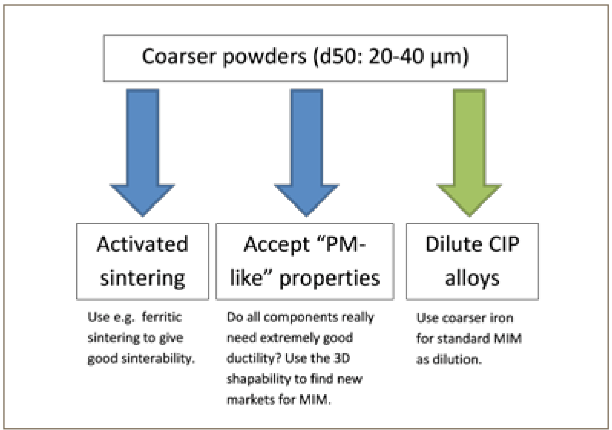

Fig. 1. Different paths to use coarser powder for MIM applications highlighting what the current paper is focusing on (green arrow)

Results and Discussion

Sintered density and dimensional stability

As expected the sintered density decreases almost linearly with an increasing amount of coarse material. At 30% addition the density drops little over 0.2 g/cm³. This is explained by the lower sintering activity. Still, 7.4 g/cm³ is a reasonable density for some applications. If the parts are HIPed anyway this might be enough to close pores.

The length follows the shrinkage logic; less shrinkage, longer parts. The differences are small but statistically significant. The

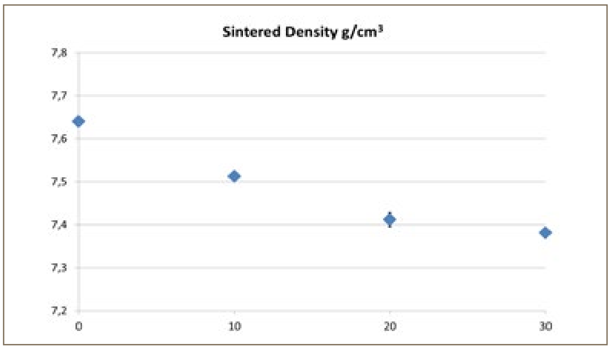

Fig. 2 Sintered density as a function of percentage added coarse powder. The error bars are displaying the 97.5% confidence interval of the data around the mean value (blue square)

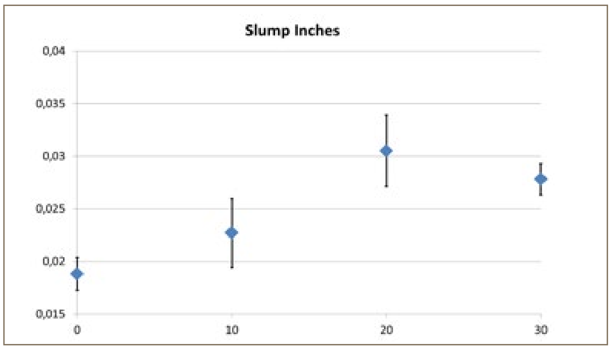

Fig. 5 Slumping of IE (sharpy) bars as a function of percentage added coarse powder. The error bars are displaying the 97.5% confidence interval of the data around the mean value (blue square)

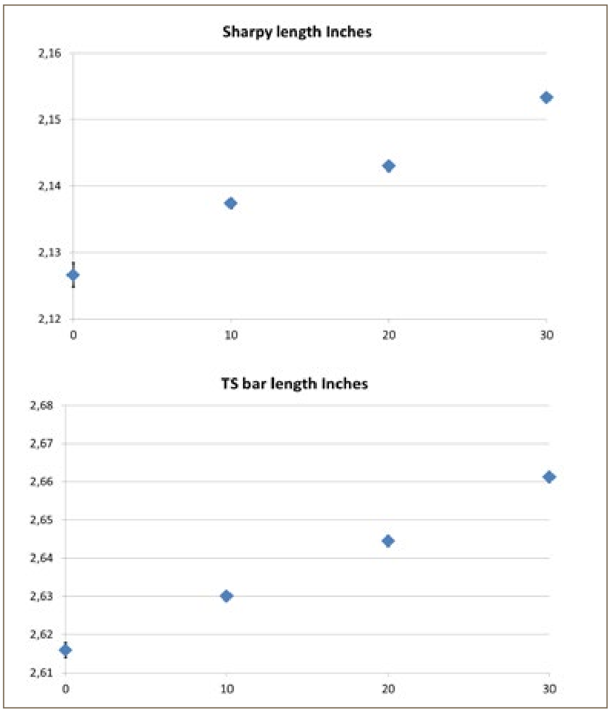

Fig. 3 Length of IE (Sharpy) bars and TS bars as a function of percentage added coarse powder. The error bars are displaying the 97.5% confidence interval of the data around the mean value (blue square)

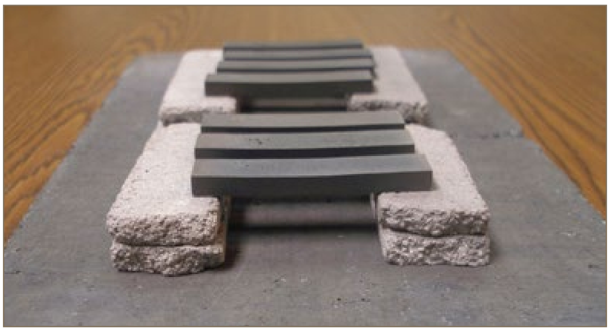

Fig. 4 Test configuration for measuring slumping on IE bars

Fraction coarse	C	O
0%	0.557	0.240
10%	0.428	0.147
20%	0.486	0.009
30%	0.498	0.009

Table 1 Carbon and oxygen in weight% of as sintered tensile bars

scatter of the length data for the parts with highest shrinkage (0% coarse added) is slightly higher, which is expected; higher absolute shrinkage will normally result in larger scatters of dimensions.

The slumping resistance is lower for parts with a higher amount of coarse powder. This is probably due to the lower sintering activity at lower temperatures for the coarse powder. As the backbone binder evaporates the carbonyl iron particles are sintering/have started to sinter slightly, ensuring a better slumping resistance.

Mechanical properties

The mechanical properties do not show the same almost linear behaviour as the dimensional changes and the density. The yield strength instead increases with the 30% added coarse powder, ductility (elongation) and impact energy are not significantly influenced at all, but the ultimate tensile strength and the hardness are decreased.

Integrative analysis

Mechanical properties are not affected proportionally to the decrease in density. This is explained by the microstructure. The larger pores will inhibit grain growth somewhat, which results in a more fine grained structure and in turn contributes to the strength (grain boundary hardening mechanism), see Fig. 7.

Moreover, the addition of coarse powder has significantly affected the presence and shape of oxides in the structure (grey dots in Fig. 7a). Fig. 7b has a "cleaner" ferritic matrix without oxides, which would promote ductility and impact strength. Oxygen analysis confirms this effect, see Table 1.

The differences in carbon content might also be one factor affecting the strength. If the carbon values in Table 1 are significant this can explain some of the increases in tensile strength on the 0% coarse material. This would imply less difference between 0% and 30% on the tensile properties.

Sources of errors and variations in data

Variations in carbon content might have influenced the variations and level of tensile properties. The carbon content has varied extremely in the microstructure (estimated from the pearlite fraction) between 0.3-0.7 weight%. A carbon controlled atmosphere would have made a difference in this case.

Also, the nickel content has varied which has been seen as nickel rich dots in the microstructure. The variations of nickel content might have been a result of segregation during mixing.

The 20% added coarse material is showing larger data variations than the other data groups. There is no explanation for this behaviour.

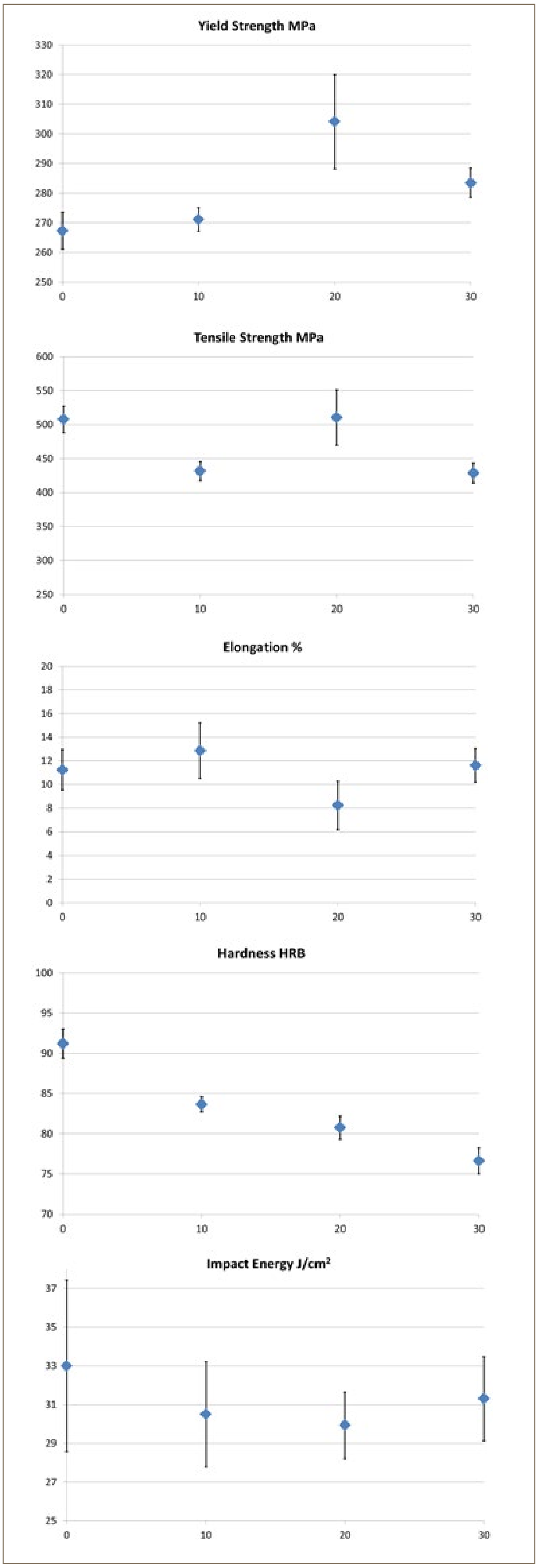

Fig. 6 Tensile properties, Hardness and Impact energy for varying content of coarse powder. The error bars are displaying the 97.5% confidence interval of the data around the mean value (blue square)

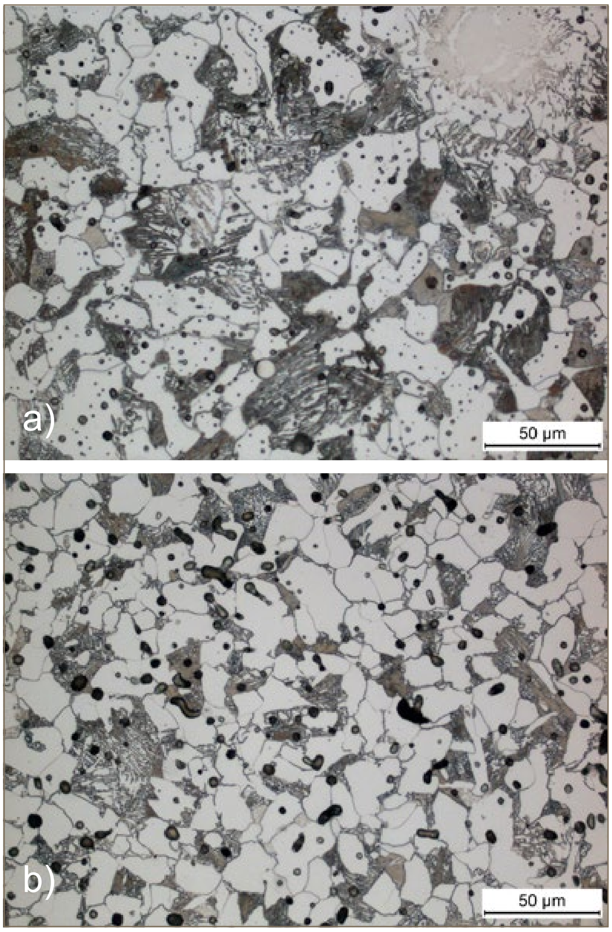

Fig. 7 a) 0% added coarse powder, etched in nital, b)30% added coarse powder, etched in nital. The structure is ferritic/pearlitic with small areas of martensite around the nickel-rich areas

Summary and conclusions

Compared to 100% CIP an addition of up to 30% coarse iron powder in this specific case with Fe-2Ni-0.5C sintered at 1180°C:

- Decreases sintered density up to around 0.2 g/cm^3
- Decreases slumping resistance
- Decreases ultimate tensile strength slightly in the as sintered state
- Decreases hardness more or less linearly
+ Decreases dimensional variations
+ Increases yield strength slightly in the as sintered state
+ Has no significant effect on impact energy
+ Has no significant effect on elongation

Coarse powder addition influences oxygen content (gives lower oxide content), which has a positive effect on ductility and impact strength. Larger pores inhibit grain growth somewhat, counteracting the negative effect of larger pores on the tensile properties.

"Diluting" CIP alloys with coarse iron powder (d50:20-40 µm) is possible and achieves reasonable mechanical properties and dimensional stability in the as sintered state.

Acknowledgements

Anna Larsson and Siv Olsson; Höganäs Sweden AB, MIM Department

Heike Grosser; Höganäs Sweden AB, Metallographic Department

Matt Bulger and Haorong Zhang; NetShape Technologies – MIM in Solon, Ohio, USA

Production of a new Plasma Spheroidised (PS) titanium powder

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Titanium metal injection moulding (MIM) has been successfully practiced for many years, however it still remains a niche application for several reasons. Titanium is known as the universal solvent and will readily absorb interstitial elements such as carbon, oxygen, nitrogen and hydrogen at elevated temperatures which in turn can significantly impact its mechanical properties. Therefore selection of the binder and debinder system is very important and can be costly if ultra low interstitial element limits are required. Another significant hurdle to the market expansion of titanium MIM is the relatively high cost of the fine spherical titanium powder used to produce the feedstock. There are two well established spherical titanium powder production routes. Ametek - Reading Alloys has been developing a new high volume, Plasma Spheroidisation (PS) titanium powder production route that offer both technical and productivity advantages over the existing manufacturing routes.

The mass production of today's titanium powders can be divided into two main powder morphology groups, spherical and non-spherical powders. The production of non-spherical titanium powders is dominated by the Hydride-Dehydride (HDH) process that offers the titanium PM industry a cost effective titanium powder [1-2]. The HDH titanium powder, however, has limited powder flow and tap density, which restricts its use in near net shape PM applications such as metal injection moulding (MIM) and additive (3D) manufacturing. The current spherical titanium powder production processes include the Plasma Rotating Electrode Process (PREP), Gas Atomisation (GA) and Plasma Atomisation (PA) processes; however, the price of these powders are significantly higher than the price of HDH powders [3].

A new spherical (hybrid) titanium powder manufacturing process has been developed by Ametek - Reading Alloys that combines both the HDH powder manufacturing process and a powder spheroidisation process to produce a wide range of cost effective spherical titanium powder products. This new Plasma Spheroidised (PS) titanium powder is a free flowing powder that exhibits no satellites and / or particle agglomerates and is also free from any entrapped insoluble gasses such as argon. This technology is also applicable to all of the other HDH produced metal powders, for example tantalum, niobium, vanadium, zirconium, hafnium and their alloys. This paper will describe the new Plasma Spheroidised (PS) titanium powder manufacturing route and compare the key powder characteristics against the other existing spherical titanium powder production processes.

Background

All spherical powder production methods incorporate a liquid phase (melting) process step which utilises the reduction in the surface area/volume ratio of a liquid droplet by surface tension forces to produce spherical powder particles. The hydride-dehydride and plasma spheroidisation processes are both well established high volume manufacturing processes which, when combined, convert

Fig. 1 Photographs of (left) dehydride vacuum furnace and (right) inert gas shielded ultrasonic screening equipment

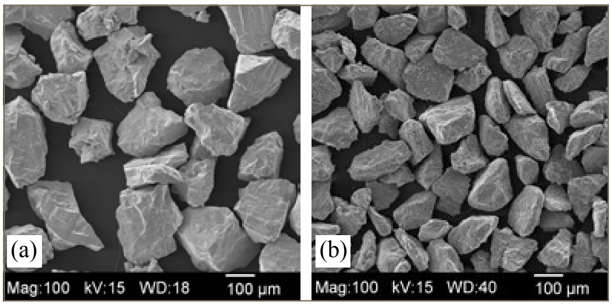

Fig. 2 Representative SEM images of HDH titanium powders: (a) Commercially Pure (CP) Ti, 150-250 µm (-60+100 mesh) and (b) Ti-6Al-4V, 75-212 µm (-70+200 mesh)

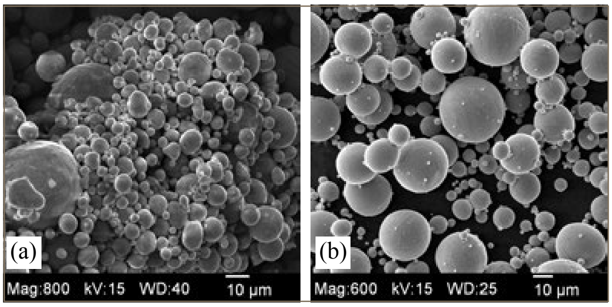

Fig. 3 Representative SEM images of spherical titanium powders: (a) GA Ti-6Al-4V, <45 µm (-325 mesh) and (b) PA CP Ti, <45 µm (-325 mesh)

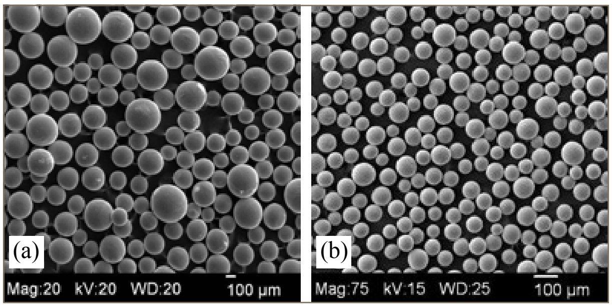

Fig. 4 Representative SEM images of Plasma Spheroidised (PS) powders: (a) CP Ti, 250-355 µm (-45+60 mesh) and (b) Ti-6Al-4V, 45-75 µm (-200+325 mesh)

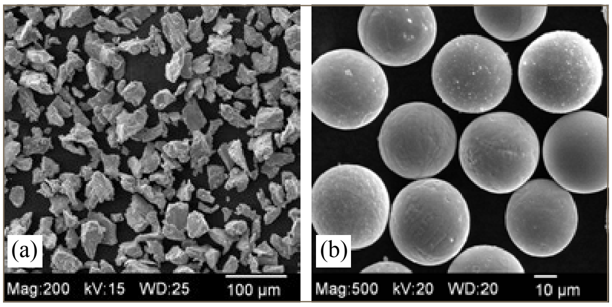

Fig. 5 Representative SEM images of CP Ti: (a) <45 µm HDH powder and (b) <45 µm PS powder

titanium feedstock first into irregular shaped powders and then into solid spherical powder particles by individually melting them through a high temperature plasma field.

The irregular shaped HDH titanium powders can be produced from a wide range of different raw materials and feedstocks, which include Commercially Pure (CP) titanium and titanium alloys, such as titanium-6wt% aluminum-4wt% vanadium (Ti-6Al-4V) [4]. The intermediate titanium (alloy) hydride raw material can be readily crushed and milled into a hydride powder using conventional powder sizing equipment and, more importantly, screened into the desired final particle size distribution (PSD) before the dehydride operation. Fig. 1 shows the typical equipment used in the production of HDH titanium powders. The hydride powder is then dehydrided under high vacuum and temperature to liberate the hydrogen and convert the titanium (alloy) hydride powder back into either HDH CP titanium powder or HDH Ti-6Al-4V powder as shown in Fig. 2.

MIM grade titanium feedstocks utilise spherical metal powders with a tight particle size distribution typically in the range of <45 µm (-325 mesh) [5-6]. Only the gas / plasma atomised processes are capable of producing <45 µm spherical titanium powders. The new PS titanium powder process is also capable of producing <45 um titanium powders by crushing and screening the hydride powder to the appropriate final PSD, unlike in the gas / plasma atomisation processes which screens the 'as atomised' powder to produce the final powder PSD, thus reducing yields and increasing powder production costs.

Production of <45 µm spherical titanium powders

The GA process relies on the disruption of a free falling molten stream of liquid titanium metal by the intersection with high pressure argon gas streams in order to produce droplets that continue to free fall and solidify. The production of a molten titanium stream can be achieved either by induction drip melting of a slowly rotating bar or any other conventional titanium melting process. These processes require the molten titanium to be dispensed through a refractory pour tube / nozzle in order to control the diameter and alignment of the molten titanium stream. The liquid titanium stream erodes the refractory and can contaminate the titanium powder with high levels of yttrium [7]. As the GA atomised powders free fall through the vertical atomising chamber the high gas turbulence can cause some un-solidified particles to fuse together. The most common particle interactions are either between larger un-solidified particles and smaller solidified particles, which can result in the formation of satellites and / or between clusters of smaller un-solidified particles which result in the formation of agglomerates as shown in Fig. 3a.

The PA process is comparable to the GA process, however the molten metal stream is replaced by a solid metallic wire feed system and the high pressure argon gas stream is replaced by DC argon plasma jets [8]. In the PA process the metallic wire is melted at the apex of three angled plasma jets and the aerodynamic drag from the argon gas flow results in the formation of liquid droplets which are slow cooled during their free fall in the argon filled atomising chamber (Fig. 3b). Typically, PA powders exhibit fewer satellites and agglomerates compared to GA powders due to the reduced gas turbulence, however both atomisation processes can exhibit a unique technical limitation resulting from the entrapment of argon gas between the powder satellites and / or agglomerates.

Some titanium MIM and additive (3D) manufactured components may require a subsequent Hot Isostatic Pressing (HIP) operation to ensure they are 100% dense in order to meet or exceed the corresponding ASTM standards for wrought titanium metal. The HIP process will close and seal all the residual porosity containing entrapped interstitial gases such as hydrogen, oxygen and nitrogen; however any residual porosity containing entrapped insoluble argon can not be closed during the HIP process, creating small residual argon filled pores, which can subsequently act as an internal stress raiser and lower fatigue strength. This limitation is particularly important with coarser GA titanium powders where the mean particle diameter is >75 µm [9].

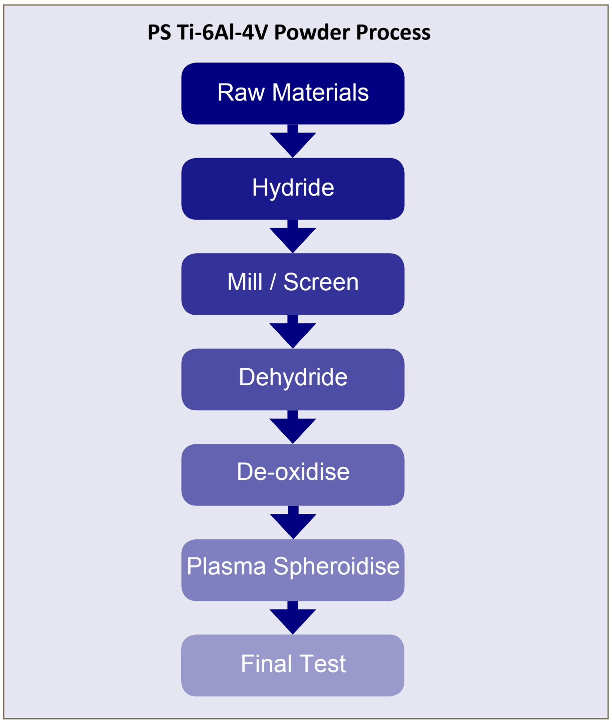

Fig. 6. Schematic flow diagram for the PS Ti-6Al-4V powder process

The PS titanium powder process combines two high volume powder manufacturing processes, i.e. the HDH and plasma powder processes. The HDH CP Ti or HDH Ti-6Al-4V powder feedstock is melted / spheroidised rapidly in a radio frequency induction coupled plasma field to produce highly spherical particles. The HDH powder is fed axially into the vertical argon plasma field resulting in extended residence times which can also refine out 'lighter' elements from the molten particles increasing the overall purity of the PS titanium powder. Also this process does not cause the molten particles to collide before solidification thus eliminating the formation of satellites and agglomerates, as shown in Fig. 4, and eliminates the possibility of any argon gas entrapment. In both the PS and PA processes the molten titanium never contacts any part of the production equipment before solidification, therefore eliminating any possible cross contamination. Fig. 5 shows SEM images of <45 µm commercially pure (CP) grade PS titanium powder before and after plasma spheroidisation.

As with all titanium powder production methods, the control of the interstitial gas levels and, in particular, the final oxygen content is a critical powder parameter. The GA and PA processes both utilise very high purity (low oxygen) raw materials, and then minimise oxygen pickup through each subsequent powder processing stage. The HDH powder process can utilise higher oxygen raw materials with the addition of a powder de-oxidation stage, which can be used to control and fine tune the desired final oxygen content to +/- 100 ppm from anywhere between 1,000-2,000 ppm in the PS Ti-6Al-4V powders [10]. This permits the use of a wider range of titanium feedstocks. Fig. 6 shows a schematic flow diagram for the PS titanium powder process for PS Ti-6Al-4V powder.

Summary

Today's commercially available <45 µm spherical titanium (alloy) powders are produced either by gas atomisation or plasma atomisation. The production of a new hydride Plasma Spheroidised (PS) titanium (alloy) powder addresses all of the critical titanium powder requirements for MIM and additive (3D) manufacturing, such as chemistry, roundness, absence of satellites and agglomerates resulting in a powder with high flow and packing densities. This new PS titanium powder also reduces internal powder porosity, and eliminates entrapped argon gas.

Another key titanium powder area is the supply chain management and manufacturing issues faced by all spherical titanium powder producers. The cost drivers in most manufacturing industries are similar; the cost of raw materials, the cost of manufacturing and the final yields. The PS titanium powder process is flexible in terms of both what raw materials and feedstocks can be used. It is also a high volume / cost effective powder manufacturing process resulting in higher final powder yields when compared to the other spherical titanium powder production processes.

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Processing and characterisation of porous NiTi alloy produced by metal injection moulding

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Porous NiTi parts were produced by metal injection moulding using three different feedstocks. All the feedstocks were formulated with a polyethylene glycol (PEG)/ polymethyl methacrylate (PMMA) binder system and elemental powders. The microstructure was characterised by X-ray diffraction (XRD) and scanning electron microscope (SEM) coupled with energy dispersive X-ray (EDX) analysis. After sintering at 1100°C for 2 h, the NiTi parts were porous with the average open cell porosity being 39.2, 38.4 and 40.9% for the three different feedstocks, respectively. The sintered parts made from these feedstocks revealed multiple phases: NiTi phase along with some Ti-rich, Ni-rich phases, oxides and carbides. The effect of TiH_2 powder and binder on the pore characteristics and the resultant mechanical properties of feedstock C sintered parts are discussed.

Because of the excellent mechanical and biocompatible properties, NiTi shape memory alloys (SMAs) are of great interest for applications such as actuators and medical implants [1-3]. Shape memory behaviour is based on a diffusionless and reversible phase transformation between low-temperature martensite and high-temperature austenite phases [4, 5]. Recently there has been an increasing interest in the porous NiTi alloy because of additional characteristics associated with porosity. Porous NiTi alloys find applications in bone implants [1, 2, 6, 7], energy absorption [8], light-weight actuators [9] and hydrogen isotope separation [10]. Biomedical applications remain the main target for porous NiTi due to the following properties: (i) good biocompatibility [11]; (ii) a combination of high strength, relatively low stiffness and high toughness [1, 12]; and (iii) shape-recovery behaviour facilitating implant insertion and ensuring good mechanical stability within the host tissue [12-14].

However, manufacturing of NiTi parts is limited by costly, multi-step processing and difficulty of machining. Metal injection moulding (MIM) has the potential to reduce manufacturing cost and to produce near-net shape parts directly from powder. Although titanium MIM has been practised since 1988, the research on NiTi shape memory alloys MIM is limited [15-21]. Among the limited reports on NiTi MIM, only very limited investigations were using elemental powders for feedstock preparation [15, 17, 19]. The use of elemental powders is however interesting from an economical point of view, since it avoids the delicate production of the pre-alloyed NiTi powder. Binder removal is a critical processing step and plays a central role in MIM part production in order to minimise contamination of the final products [22, 23]. In this sense, the debinding step must be carried out with great care so that there are as few contaminants as possible remaining.

The development of a suitable binder system is one of the main tasks in producing NiTi alloys by MIM with acceptably low level of contaminants. So far there are several binder systems for MIM NiTi in recent reports, such as the polyethylene glycol (PEG)/ polymethyl methacrylate (PMMA) system [18, 19, 24], the

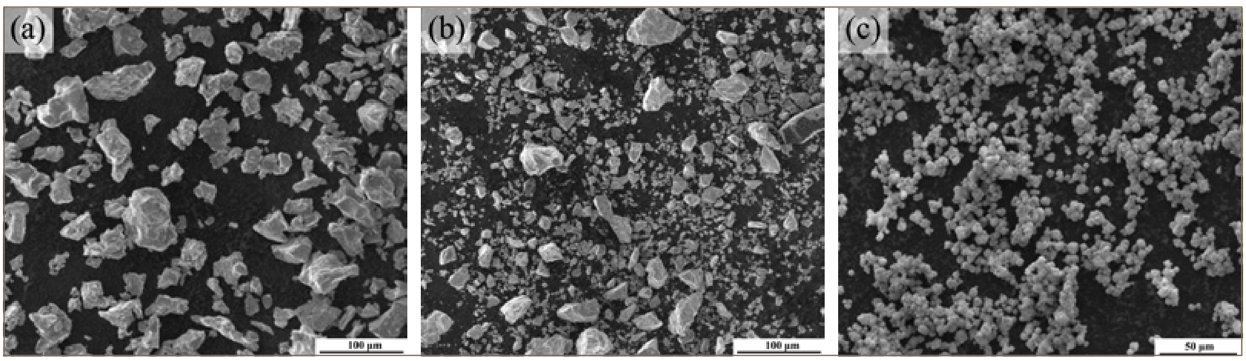

Fig. 1 Morphologies of elemental powders (a) Ti, (b) TiH_2 and (c) Ni

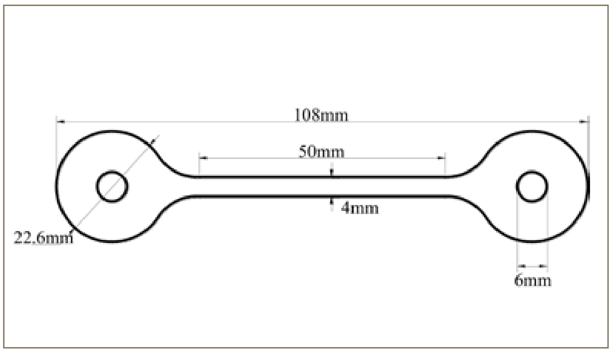

Fig. 2 Geometry of injection moulded tensile test bars: Thickness = 2 mm

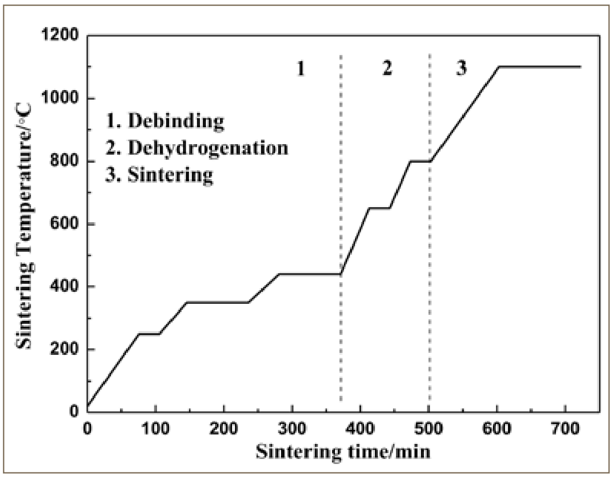

Fig. 3 Temperature profile for debinding and sintering under vacuum

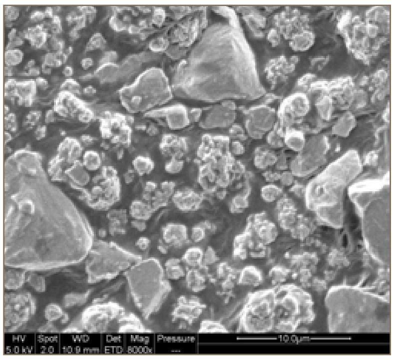

Fig. 4 SEM micrograph of Feedstock A

paraffin wax/polyethylene system [15, 17] and the polyamide wax/polyethylene system [21, 22]. Many problems are associated with using the wax-based systems, such as low green strength and very narrow processing windows. Plus, the wax-based binder system is flammable, carcinogenic and not environmentally friendly, which is the main drawback of wax-based binder systems. In contrast, PEG-based binders hold promise as a safe and ecological water-soluble binder system for MIM and it can be extracted at 40~60°C, which is a quite low temperature range [18, 19, 25]. PMMA decomposition can produce almost pure gaseous MMA monomer and leave no residue in vacuum and inert atmosphere, unlike other polyolefins [26-28]. The PEG based water soluble binder system has since been used widely with other ceramic and metallic powders, including titanium and its alloys [18, 19, 24, 25, 29-31]. This binder system is usually comprised of a major fraction of PEG as the primary binder component, a minor fraction of PMMA as the backbone polymer and a small proportion of stearic acid (SA) as the lubricant. The binder extraction is carried out in two

Powder	Particle size (μm)			O (wt.%)	C (wt.%)	N (wt.%)	H (wt.%)
	d_{10}	d_{50}	d_{90}				
HDH Ti	13.55	35.43	66.03	0.49	0.080	0.023	0.031
TiH_2	3.88	24.61	63.31	0.26	0.057	0.025	1.92
Ni	4.18	11.56	34.02	0.11	0.071	0.033	0.0025

Table 1 Characteristics of the starting elemental powders

Chemicals	Supplier	Density/ g•cm^{-3}	Average M_w/ g•mol^{-1}	Melting/ Softening point/°C
PEG	Minfeng, China	1.20	4000	58~61
PMMA	Bayer, Germany	1.19	1*10^6	190
SA	Kermel, China	0.87	284.5	70
BHT	Aladdin, USA	1.05	220	70~73

Table 2 Characteristics of the chemicals used in the binder system

stages. First, water leaching is used to remove the water soluble component – PEG. During this stage, the compacted metal powders are still held together by the water insoluble component – PMMA. Pyrolysis is then used to remove the remaining components. The second step is facilitated by the open-pore channels formed upon the removal of the PEG in the first debinding step [29].

This study therefore investigates the feasibility of MIM of NiTi. The feedstock was formulated using a PEG-based binder system and elemental powders. Two different titanium powders were used: pure titanium powder produced by the hydrogenation-dehydrogentation process (HDH), and titanium hydride (TiH_2) powder. The use of TiH_2 is to test whether it accelerates the sintering process [32-34].

Experimental

Materials

Table 1 summarises the particle size and impurity levels of the starting powders. The morphologies of the three powders are shown in scanning electron microscopy images (Fig. 1). The Ni/Ti and Ni/TiH_2 powders were weighed to have a nominal composition of 51 at.% Ni and 49 at.% Ti. The powders were gently ball-blended for 10 h using a powder rotator mixer. The ball-to-powder weight ratio was 3:1.

The characteristics of the chemicals used in the binder system are listed in Table 2. PEG was used as the primary binder component while PMMA, SA and butylated hydroxytoluene (BHT) were used as the backbone polymer, lubricant and antioxidant additive in the binder system, respectively. The compositions of the binder system used for preparing all the feedstocks consists of 87 wt.% PEG, 11 wt.% PMMA and 2 wt.% SA, with an extra 0.26 wt.% of BHT (based on PEG) added [35, 36]. The purpose of the BHT addition is to prevent PEG from thermal degradation during feedstock preparation.

Feedstock preparation

Mixing of metal powder and binder was undertaken in an oil heated mixer (XSM1/20-80). The charge of powder and binder materials follows a sequential procedure. First, PMMA was charged in the chamber at 195°C. Once the PMMA was melted, the temperature was reduced to 190°C [37], and the remaining PEG, SA and BHT were gradually added at a mixing speed of 40 rpm. Afterwards, the metal powders mixture was added to the chamber and allowed to mix with the molten binder for 1 hour until the mixing torque was stabilised, indicating homogeneity of the feedstocks. After solidification, all the feedstocks were crushed into normal pellets by a crusher. Feedstock A was prepared from elemental nickel and titanium mixture with the above prescribed binder, while Feedstock B was prepared from elemental nickel and TiH_2 powder. Both Feedstock A and Feedstock B had a solid loading of 60 vol.%, while Feedstock C was similar to Feedstock A except that its solid loading was 50 vol.%. All of the feedstocks were used for MIM.

MIM, debinding and sintering

Injection moulding was performed on a twin-screw HTF80X/1 machine (Haitian, China) using Feedstock A, B and C. Moulding parameters such as die and nozzle temperature were adjusted for each die and feedstock. Table 3 summarises the parameters used. Tensile test bars with 2 mm thickness were produced as per the MPIF Standards 35 and their geometry is shown in Fig. 2.

A typical two-step debinding process was performed on the MIM samples: water debinding and thermal debinding. Each sample was weighed with an accuracy of ± 0 .0001 g before and after debinding. To study the water debinding kinetics, the green parts were soaked in a 2.5 L distilled water bath at 60°C and dried for 12 h in a vacuum oven at 55°C [29]. After 20 hours and removal of PEG by water debinding [25], the brown parts were placed in a vacuum furnace (vacuum level: 2×10^{-3} Pa) where thermal debinding and sintering were conducted. The brown samples were placed on top of the alumina (Al_2O_3) support in order to avoid any impurity pickup from the supporting rack. The thermal debinding and sintering temperature profile is shown in Fig. 3, in which three stages can be distinguished. The first stage is designed to burn out the binder. The second stage is for the dehydrogenation of TiH_2 powders in Feedstock B, while sintering is performed at 1100°C for 2 h.

Testing and characterisation

Sintered density and open porosity of the sintered NiTi samples were measured by Archimedes methods. The largest pore size was measured with the bubble point method [38]. Microstructures of the debound and sintered samples were observed using an FEI Quanta 200F environmental scanning electron microscope (ESEM) equipped with an energy dispersive X-ray spectrometer (EDX). Phase constituents were identified with X-ray diffraction (Bruker D2 Phaser). Thermal gravimetric analysis (TGA) of Feedstock A was performed using a Shimadzu Thermogravimetric Analyzer TGA-50 with a heating rate of 10°C/min from room temperature to 600°C under flowing argon gas with 75 ml/min flow rate. The sintered NiTi samples were polished prior to optical microscopic examination. Room temperature tensile properties of the sintered NiTi samples made from Feedstock C were measured on an Instron 3367 universal testing machine with a cross-head speed of 0.5 mm/min.

Results and discussion

Solvent (water) debinding behaviour and TGA analysis

Fig. 4 shows the morphology of Feedstock A before water debinding (other feedstocks have very similar morphologies). The white particles represent the titanium and nickel powders, while the ribbons represent the binder. The binder fills the void space of the compact completely. Hardly any pores were observed in the feedstock. Apparently, the feedstock is homogeneous.

During extraction of the water-borne binder, the water molecules interact with and dissolve the PEG [24]. The objective of the water debinding is to create an interconnected channel throughout the powder compact so that the backbone binder components, PMMA in our case, can be removed through these channels when a second debinding (usually thermal debinding) is applied [39].

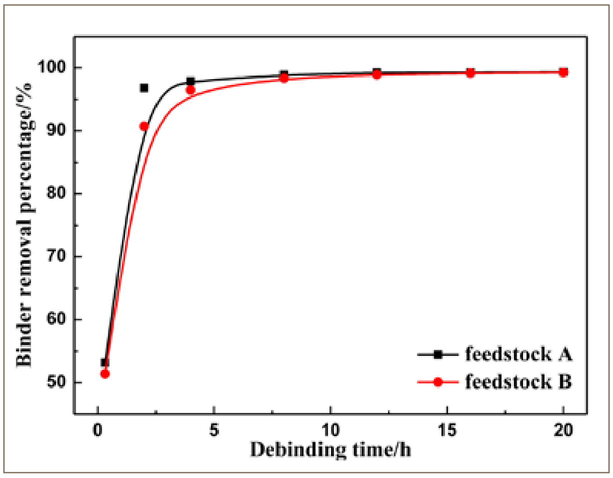

Fig. 5 Water debinding behaviour of PEG in the green parts made from Feedstock A and B. Debinding was carried out at 60°C

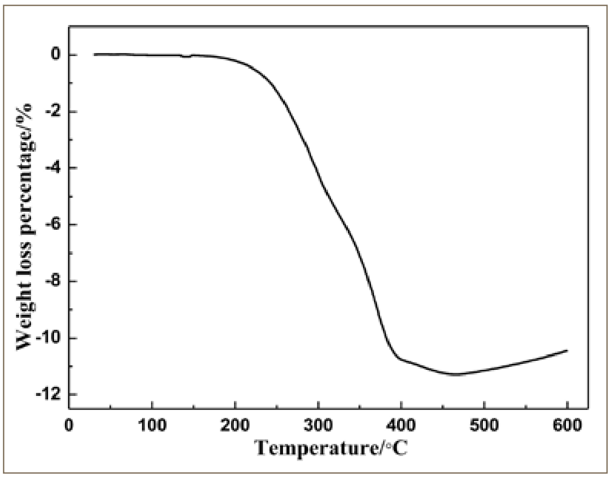

Fig. 6 TGA trace of Feedstock A

Parameter	Values
Nozzle temperature	180°C
Mould temperature	30°C
Injection pressure	75 MPa
Mass flow	30 cm^3/s

Table 3 Optimal parameters for metal injection moulding

PEG loss as a function of debinding time in water is shown in Fig. 5 for Feedstock A and B moulded parts both with 2 mm thickness at a debinding temperature of 60°C. From both curves a rapid removal rate was observed in the first few hours of debinding; afterwards the PEG removal rate levels off. During debinding, the pore channels gradually extend to the inner region of the specimen. Therefore, the transport path of the hydrated PEG complexes increases with debinding time, resulting in a slowdown of the debinding rate [24, 40].

As shown in Fig. 5 the binder removal rate of Feedstock A moulded parts is greater than that of Feedstock B compacts. For instance, after 2 hours of water debinding, the binder (PEG) removal percentages for Feedstock A moulded parts and Feedstock B moulded parts

Fig. 7 Injection moulded and as-sintered tensile bars made from Feedstock C

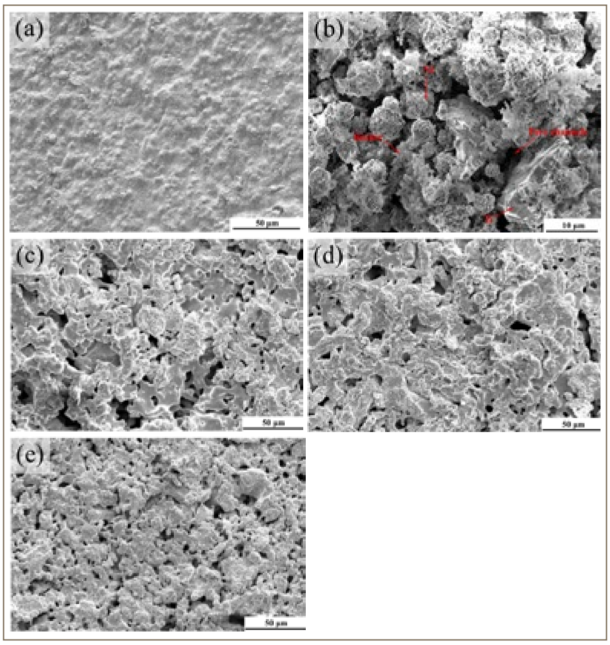

Fig. 8 SEM microimages showing microstructural evolution during processing (a) the MIM green part made from Feedstock C, (b) after water debinding of MIM parts made from Feedstock C, (c) as sintered parts made from Feedstock C, (d) as sintered parts made from Feedstock A and (e) as sintered parts made from Feedstock B. For all the cases, the samples were sintered at 1100°C

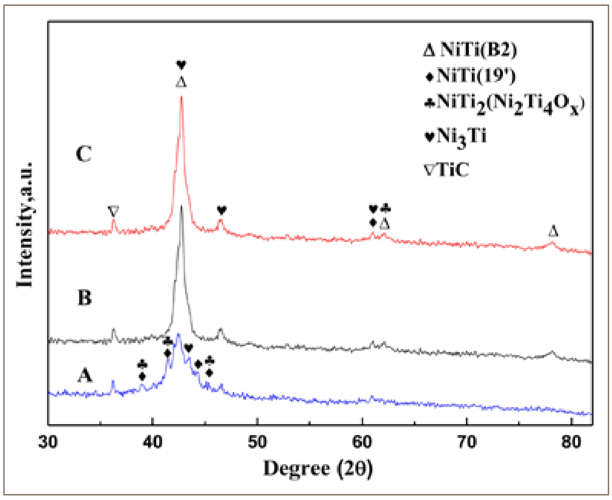

Fig. 9 X-ray patterns of the sintered samples made from the three feedstocks

were 96.8% and 90.7%, respectively. This is probably because the mean particle size of HDH-Ti powder is larger than that of TiH_2 as shown in the particle size distribution analysis. The main difference in these two feedstocks is the particle size of Ti and TiH_2, with the former particles being larger. Increasing particle size leads to a reduced packing density and hence an increased inter-particle space results in a faster debinding rate.

Fig. 6 shows the TGA trace for Feedstock A, indicating the binder burnout temperature ranges from 200°C to 440°C. As shown in Fig. 6, a maximum weight loss percentage of approximately 11.3% was observed, which corresponds to the total binder weight percentage in Feedstock A. The gradual weight gain after 450°C is attributed to the oxidation of titanium powder [41, 42].

Characterisation of debound and as-sintered samples

The injection moulded 'green' and as-sintered tensile bars from Feedstock C are shown in Fig. 7. No distortion or surface cracks were observed. Microstructural evolution from the injection moulded green samples to the final sintered samples made from Feedstock C is shown in SEM images (Fig. 8). In the green samples, Fig. 8(a), the binders (PEG/PMMA/SA) fill the interparticle spacing. After 20 hours of water debinding, the water soluble component PEG was completely removed, while PMMA ligaments held the powder particles as shown in Fig. 8(b). Some fine pore channels between powder particles were clearly observed. The pore channels facilitate the subsequent PMMA removal by thermal debinding [29]. Both thermal debinding and sintering were incorporated in to one step and conducted in the same vacuum furnace. Fig. 8 (c) presents the microstructure observed on the surfaces of the sintered NiTi samples made from Feedstock C. In comparison, the microstructures of the sintered NiTi samples made from Feedstock A and B are shown in Fig. 8 (d) and (e). It is observed that the average pore size of NiTi alloy samples sintered from Feedstock C is larger than that sintered from Feedstock B and A. Table 4 summarises the characteristics of the sintered porous NiTi samples. It can be seen that Feedstock B resulted in a smaller pore size while Feedstock A gave rise to the smallest lateral shrinkage. Comparing Feedstock A with Feedstock B, we found that TiH_2 in the feedstock plays an important role in the pore size and distribution. In addition to the particle size, the decomposition of TiH_2 would accelerate the reactive sintering densification process [34, 43-46], as a result of activated Ti powder surfaces exposed by dehydrogenation. In addition, the binder volume fractions in Feedstocks A and C also plays an important role in the porous microstructures of the sintered parts. A greater binder volume fraction leads to a higher open porosity and larger pore sizes in the final sintered parts. It needs to be pointed out that the other factors such as Kirkendall effect [47, 48], transient liquid phase [48] and phase transformation [49] also contribute to the pore formation.

The lateral shrinkage of the sintered samples made from Feedstock C is greater than that from Feedstock A. This is simply because Feedstock C contains a higher volume fraction of binder component. In comparison to Feedstock A, the sintered samples made from Feedstock B demonstrated greater shrinkage, although both feedstocks had an identical binder volume fraction of 60 vol.%. The shrinkage difference is thought to stem from the effect of TiH_2. Firstly, TiH_2 has a lower density (i.e. 3.9 g/cm³) than Ti (4.5 g/cm³). During dehydrogenation, the density increase may cause shrinkage. Secondly, the newly activated powder surfaces after dehydrogenation enhance densification [34, 43, 50], causing further shrinkage. Thirdly, the particle size of TiH_2 is smaller than that of Ti, which minimises the swelling phenomenon in the case of Feedstock B when compared with Feedstock A [51].

Fig. 9 shows the XRD patterns of the vacuum sintered NiTi alloys

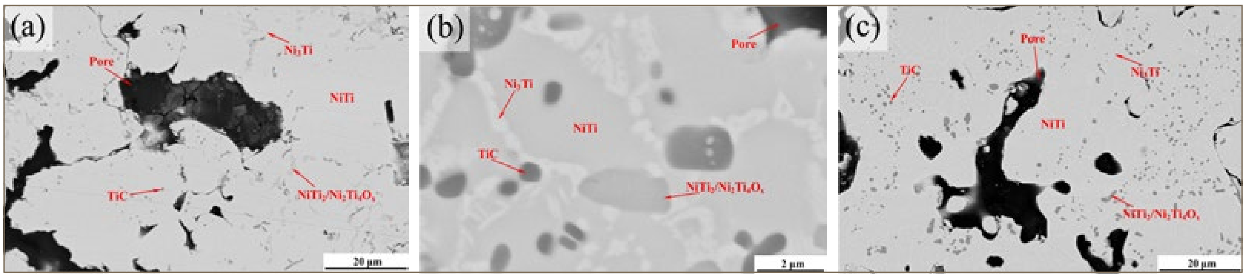

Fig. 10 Back-scattered electron (BSE) images of the sintered porous NiTi parts made from (a) Feedstock A, (b) Feedstock B and (c) Feedstock C

made from these three feedstocks. After sintering, in all cases, B2 (NiTi) phase dominates with a small fraction of B19' (NiTi) and several intermetallic phases present, such as $NiTi_2$ and Ni_3Ti. Some impurity phases such as TiC and $Ni_2Ti_4O_x$ were also observed. These minor and impurity phases are further confirmed by EDX analysis, Fig. 10. These observations are in good agreement with other reports [16, 18, 19, 21, 22].

Tensile properties

Fig. 11 illustrates a typical stress-strain curve for the MIM-NiTi alloy samples made from Feedstock C, where a non-linear elastic behaviour can be seen. This non-linear elasticity is attributed to the elastic buckling of cell walls or edges during the tensile test [52] or the superelastic effect of the NiTi phase, caused by a stress-induced austenite-to-martensite phase transformation [3, 53]. The strain to fracture of the sintered samples is 3.81% and the fracture strength is 89.9 MPa; both are lower than that reported in the literature for the pressureless sintered and HIPed NiTi [54-57]. This is caused by the larger porosity and insufficient densification of the NiTi parts in our study.

Fractography study is presented in Fig. 12 where a transgranular fracture is observed. Crack formation initiates from the pore-cell wall or pre-existing flaws and propagates rapidly due to the stress-concentrating effect of the pores [52].

Conclusions

Porous NiTi alloys are produced by MIM using elemental powder feedstocks followed by sintering at 1100°C for 2 h. The feedstocks are formulated with a PEG/PMMA binder system. The binder (PEG) removal rate of the feedstocks depends on the metal powder particle size; the larger the particle size the faster the debinding rate. All the sintered samples exhibit a porous structure with porosity of approximately 40 vol.%. Open porosity, pore size and lateral shrinkage also depend on the feedstocks. The use of TiH_2 powder has significant effect on the pore characteristics. Multi-phase constituents are observed in all the sintered samples, implying that complete alloying was not achieved and slight contamination occurred during processing. The porous sintered samples showed a limited fracture strain of 3.81% with the tensile strength of 89.9 MPa, which are typical for a porous structure.

Acknowledgements

The authors are grateful for the financial support from the University of Auckland through its Faculty Research Development Fund (FRDF) programme, and State Key Laboratory for Powder Metallurgy, Central South University, China for their travel grant. The technical assistance from Dr. Hao He, Central South University China is gratefully acknowledged. GC also wishes to acknowledge the scholarship support from the China Scholarships Council.

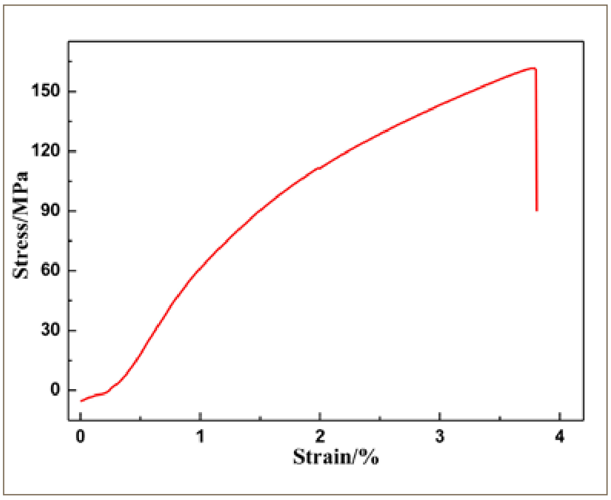

Fig. 11 A typical engineering stress-strain curve for the sintered samples made from Feedstock C

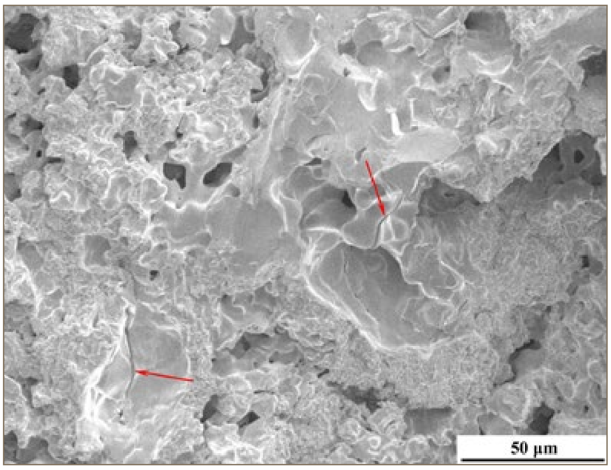

Fig. 12 SEM image of the fracture surface of the sintered tensile samples made from Feedstock C. The arrows indicate cracks occurring after the tensile test

Sample sintered from Feedstock	Lateral shrinkage/%	Density/ $g \cdot cm^{-3}$	Open porosity/%	Largest pore size/μm
A	5.43 ± 0.6	3.90 ± 0.02	39.2 ± 0.5	10.35 ± 0.7
B	10.36 ± 0.5	3.99 ± 0.03	38.4 ± 0.6	5.26 ± 0.5
C	11.28 ± 0.5	3.82 ± 0.05	40.9 ± 0.3	11.26 ± 0.9

Table 4 Characteristics of the sintered porous NiTi samples

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